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CGA-329351

STUDY TITLE

Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

DATA REQUIREMENT

Environmental Fate Data Requirement
40 CFR 158
Subdivision N: Series 163-1

AUTHOR

5.1.2.e Woo

STUDY COMPLETED ON

December 5, 1995

PERFORMING LABORATORY

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LABORATORY PROJECT IDENTIFICATION

Agrisearch Project No. 12220

SPONSOR

Ciba-Geigy Study No. 60-95
Ciba Crop Protection
Ciba-Geigy Corporation
P.O. Box 18300
Greensboro, NC 27419

Volume 1 of 1 of Study

Total Pages - 284
(including page 167a)

STATEMENT OF NO DATA CONFIDENTIALITY CLAIM

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Section 10(d)(1)(A), (B), or (C).

Company Ciba Crop Protection, Ciba-Geigy Corporation

Company Representative 5.1.2.e Woo Date 12-7-95

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Title Signature

These data are the property of Ciba Crop Protection, Ciba-Geigy Corporation, and as such, are considered to be confidential for all purposes other than compliance with FIFRA Section 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality which may exist under any other statute or in any other country.

CERTIFICATION OF GOOD LABORATORY PRACTICE

Project No. 12220, "**Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils**", has been performed in compliance with EPA Good Laboratory Practice Standards (40 CFR: 160) by Agrisearch Incorporated. Soil clay mineralogy determination was not performed in compliance with US EPA GLPS. Test/reference substance characterization was conducted by Ciba Crop Protection, Ciba-Geigy Corporation, Greensboro, NC and these records are maintained by the sponsor.

Submitted By:

5.1.2.e Woo Director 12/5/95
Date

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Signature of Representative of Submitter/Sponsor

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CERTIFICATE OF AUTHENTICITY

This is an unaltered copy (except for changes required to comply with PR Notice 86-5) of the final report received from the following contract laboratory:

Agrisearch Incorporated
5734 Industry Lane
Frederick, MD 21701

Company: Ciba Crop Protection
Ciba-Geigy Corporation

Sponsor Representative: [redacted]

Signature: [redacted]

Date: 12/7/95

QUALITY ASSURANCE STATEMENT

Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

This project (Project Number 12220 was inspected/audited by Quality Assurance according to Agrisearch Standard Operating Procedures and EPA's Good Laboratory Practice Standards (40 CFR: 160) and all findings were reported to the study director and management:

Type of Inspection	Date Inspected/Audited	Date Reported
Test System Sampling, Sample Combustion	8/18/95	8/18/95
Raw Data Audit, Draft Report	8/10,11,15,31/95, 9/5,6,7/95, 9/28,29/95, 10/2,5,6/95	10/6/95
Final Report	11/17/95 12/5/95	11/17/95 12/5/95

Action has been taken in response to all items listed by Quality Assurance. It was concluded that the final report accurately reflects the raw data for this project.

5 1.2.e Woo

Quality Assurance Manager

Date: 12/5/95

KEY AGRISEARCH INCORPORATED STUDY PERSONNEL

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STUDY IDENTIFICATION

STUDY TITLE: Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

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SPONSOR STUDY NO: 60-95

STUDY INITIATION DATE: June 19, 1995

EXPERIMENTAL START DATE: July 18, 1995

EXPERIMENTAL TERMINATION DATE: September 18, 1995

STUDY COMPLETION DATE: December 5, 1995

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All original raw data, protocol, amendments, and a copy of the final report are stored in the archives at Agrisearch Incorporated. A certified copy of the raw data and the original final report will be provided to Ciba for archiving. Supporting records to be retained by Agrisearch Incorporated and not necessarily archived with the study data will include, but not necessarily be limited to, refrigerator and freezer temperature records and instrument calibration and maintenance records. Specimens shall be retained for quality assurance verification and approval by the sponsor to dispose, at which time the specimens shall be disposed of in accordance to Agrisearch standard operating procedures (or notification to transfer the specimens to the sponsor). Specimen disposition documentation shall be retained by Agrisearch Incorporated.

ABSTRACT

The rate of dissipation of a pesticide in the environment and in soils is influenced by numerous phenomena. This study was designed (APPENDIX A) according to US EPA Pesticide Assessment Guidelines (Reference 2) to predict the potential for CGA-329351 to move to and disperse in aquatic sites. Five soils varying in organic matter and texture were tested using the batch equilibrium technique (adsorption/desorption).

Analytical grade uniform phenyl-ring-labeled CGA-329351 [ϕ-^{14}C-CGA-329351] was prepared in aqueous 0.01M calcium ion solution at concentrations of 0.206, 0.513, 1.028, 5.145, and 10.217 μg/mL, added to soil, and allowed to equilibrate in the dark while shaking in a controlled temperature incubator at 25±1°C. After equilibration, the phases were separated by centrifugation with CGA-329351 concentrations determined by liquid scintillation counting (LSC) of the aqueous phases and by difference for the soil phases. The soil phases were next desorbed with 0.01M calcium ion solution free of CGA-329351 and concentrations determined by combustion of the soil and LSC of the aqueous phase following centrifugation. Soil characteristics were obtained. All data were evaluated using the Freundlich equation and values for K_{oc} (sorption coefficient), K_d (sorption constant) and n were determined.

The results demonstrated that the Freundlich isotherm was applicable to analysis of adsorption and desorption of CGA-329351. TLC and HPLC analysis demonstrated no degradation of CGA-329351 during this study. Logarithmic plots of data yielded a straight line for all five soils tested. CGA-329351 was determined to adsorb to all soil types and classified as having low to very high mobility with K_{oc} values between 25 and 1500. Values determined for K_d, K_{oc}, and n were as follows:

	ADSORPTION			DESORPTION		
SOIL TYPE	K_d	K_{oc}	n	K_d	K_{oc}	n
MS-Clay	7.6	570	0.690	18.4	1376	0.832
MD-Sand	0.1	20	1.121	2.8	790	1.009
CA-Sandy Loam	0.2	68	1.031	1.8	785	1.061
WA-Loam	1.3	86	0.957	2.9	195	1.048
AZ-Silty Clay Loam	2.3	1299	0.862	6.2	3539	0.963

INTRODUCTION

The objective of this study was to predict the potential for movement of CGA-329351 from the site of application to an aquatic site, and dispersion therein. This objective was achieved by determining the values of K_{oc} (sorption coefficient), K_d (sorption constant) and n using the Freundlich isotherm (Reference 1). Five soil types were tested in this study using the batch equilibrium technique (adsorption/desorption).

This study was performed to meet the US EPA requirements as listed in the US EPA Pesticide Assessment Guidelines, Subdivision N, Series 163-1 (Reference 2). This study was also conducted in accordance with the protocol (APPENDIX A); US EPA FIFRA Good Laboratory Practice Standards, 40 CFR 160 (Reference 3); the Data Reporting Guidelines (Reference 4); FIFRA Accelerated Reregistration, Phase 3 Technical Guidance (Reference 5); Pesticide Reregistration Rejection Rate Analysis (Reference 6); and applicable Agrisearch Incorporated Standard Operating Procedures. There were no circumstances that affected the quality or integrity of the data.

MATERIALS/METHODS

Test Soil

A total of five representative soil textures were used for this study - sand, sandy loam, loam, silty clay loam, and clay. Three of the soils (MD sand, CA sandy loam, and AZ silty clay loam) had an organic matter (OM) content of <1%. The pH of all five soils was within the range of 4 to 8. All five soils satisfy Arizona Department of Environmental Quality (ADEQ) standards of having an organic carbon (OC) content of 2.6% or less. Four of the five soils satisfy ADEQ standards of having a pH between 6.5 and 8.5±0.3. The sandy loam is the same soil used for the aerobic soil metabolism study and was collected from California; the clay was collected from Mississippi; the sand from Maryland; loam from Washington; and the silty clay loam from Arizona. All soils were collected from the soil surface (Horizon A). The source of the soils (location) is provided in APPENDIX C. All five soils were air dried at ambient laboratory temperatures and sieved through a 2 mm sieve prior to characterization and use in the study to assure uniform particle size. Air dried soils were maintained (stored) at ambient (room) temperature from receipt until use. A representative subsample of each soil was removed for soil characterization by AGVISE Laboratories (P.O. Box 510, Highway 15, Northwood, ND, 58267).

Soil characterization was performed by AGVISE Laboratories and the reports were submitted to Agrisearch Incorporated. The soil clay mineralogy was performed by the North Dakota State University, Fargo, ND. The cation exchange capacity was determined by summation of exchangeable Ca, Mg, Na, K, and H cations. The soil characteristics are presented in TABLE I.

The microbial viability of the five soil types used in this study was determined. Soils were evaluated using Standard Methods for the Examination of Water and Wastewater (Reference 7) spread plate technique. Dilutions of soil were incubated using plate count agar (total aerobic bacteria), rose bengal agar (total fungi), and actinomycete agar (total actinomycetes). Agar plates were incubated for 12 days at 25±1°C under aerobic conditions. Results of the microbial viability testing are presented in TABLE II.

Soil Microbial Biomass

A 500 g aliquot of each soil type was shipped to Ciba Crop Protection for biomass determination. Ciba-Geigy Corporation was responsible for all quality assurance and data generation and storage for this determination. The results of the biomass determination were submitted to Agrisearch Incorporated and are included in this report (TABLE I, APPENDIX C).

Test Water

The water used in this study was drinking water provided by Frederick County, Maryland. The drinking water was filtered, deionized, boiled, and distilled prior to use. This test water was used to prepare the 0.01M calcium ion solutions.

Silylation of Glassware

All glassware used in the study was silylated according to Agrisearch Incorporated Standard Operating Procedures. The procedures use a 5% solution of dichlorodimethylsilane in dichloromethane (DCM) to block all available binding sites on the glassware. After sitting for thirty minutes, the glassware is rinsed three times with methanol, three times with DCM, and oven baked at 100 to 110°C for two hours.

Test Substance

The radiolabeled test substance CGA-329351[a] was provided by Ciba-Geigy Corporation, Greensboro, NC. The test substance container was retained at Agrisearch Incorporated for the duration of the study.

Phenyl-^{14}C-CGA-329351, (0.811 mCi, 10 mg, Reference No. MSR-II-90, specific activity 81.1 µCi/mg, radiochemical purity 98.8%, expiration date November 1995) was received from Ciba-Geigy Corporation on June 6, 1995. The radioactive chemical was logged at Agrisearch Incorporated as number 95-041 (FIGURE 1). The chemical was stored frozen at approximately -20°C when not in use.

The entire received shipment of ^{14}C-CGA-329351 was completely dissolved in 10 mL of acetonitrile (1.0 mg/mL) to be used as a test substance stock solution. The acetonitrile was Burdick & Jackson UV spectroanalyzed grade. The radioactivity of the test substance solution was determined by liquid scintillation counting (LSC) of five replicate aliquots (APPENDIX D, page 166). The radioactivity in each aliquot was within 3% of the mean and the solution was considered homogeneous. The test substance stock solution was used to verify the radiopurity of the test substance and to demonstrate stability of the test substance at the test facility as required by US EPA FIFRA GLP Standard 40 CFR 160.105(e). Using two-dimensional thin layer chromatography (TLC), and high performance liquid chromatography (HPLC), the radiochemical purity of the test substance (as reported by Ciba-Geigy Corporation) was verified at Agrisearch to be >99% prior to study dosing. See APPENDIX B and FIGURES 3 and 4.

Reference Substances

Technical CGA-329351 (CGA-329351, Reference No. S95-1796, 100 mg) was received from Ciba-Geigy Corporation on May 31, 1995 with a stated purity of 97.3%. The shipment was logged at Agrisearch Incorporated as number 95-038 (FIGURE 1). The chemical was stored at refrigerator temperatures approximately 1 to 8°C when not in use.

Four additional analytical standards were received from Ciba-Geigy Corporation on May 31, 1995. These standards were to assist in characterization of possible CGA-329351 degradates by co-chromatography. The following standards were received:

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[a] CGA-329351 = (R)-2-[N-2,6-dimethylphenyl methoxyacetyl-amino]-propionic acid methyl ester

Compound ID	Lot No.	Quantity Received	Purity %	Expiration Date	Agrisearch Log No.
CGA-37734	BPM-I-8	≈50 mg	96.9	2/96	95-034
CGA-42447	MCO-I-2	≈50 mg	99.7	12/96	95-035
CGA-62826	BPM-I-4B	≈50 mg	>99.9	12/96	95-036
CGA-119857	GB-XLV-3	≈50 mg	>99.9	12/96	95-037

All four chemicals were logged at Agrisearch Incorporated and stored under refrigerator temperatures approximately 1 to 8°C when not in use.

Test and Reference Substance Documentation

The methods of synthesis, fabrication, and/or derivation of the test and reference substances are documented and retained by the sponsor as required by 40 CFR 160.105. Characterization of the test and reference substances was conducted by the sponsor and these records are retained by the sponsor. These records and archived samples are being maintained by the sponsor at Ciba-Geigy Corporation, Greensboro, NC.

Test Containers

Oak Ridge Teflon® FEP centrifuge tubes manufactured by Nalgene were obtained from VWR Scientific, Bridgeport, NJ (Cat. No. 21009-477) for use in this study. The 50 mL centrifuge tubes were entirely Teflon® including the screw cap closures. Prior to use, all centrifuge tubes were autoclaved for one hour at 121°C and 15 psig.

Test Solutions

The aqueous 0.01M calcium chloride solution was prepared by adding 7.75 g of calcium chloride dihydrate (Baker Chemical Co., Cat. No. 1332-01) to 4 liters of water and stirring until dissolved (1.94 g calcium chloride dihydrate per liter of water). The resultant solution (0.01M calcium ion concentration) was sterilized by filtration through a 0.2 micron membrane filter (Gelman Product No. 63025) into autoclaved glassware. The sterile calcium ion solution was covered with aluminum foil to maintain sterility. The pH of the $CaCl_2$ aqueous solution measured after preparation was 6.93 (APPENDIX D, pages 169 and 170).

For the preliminary phase, a 2.5 mL aliquot of the ^{14}C-CGA-329351 stock solution (1.0 mg/mL) was placed in a sterile 500 mL Erlenmeyer flask and the acetonitrile was evaporated under a stream of nitrogen. Next 250 mL of prepared sterile 0.01M calcium ion solution was added to dissolve the CGA-329351. Dissolution of the CGA-329351 was aided by the use of sonication for approximately five minutes. The final CGA-329351 concentration was 10.641 µg/mL. For the repeat preliminary phase, another ^{14}C-CGA-329351 solution was prepared in aqueous 0.01M calcium ion solution. The final CGA-329351 concentration of this solution was 10.390 µg/mL.

For the definitive phase, a 3.5 mL aliquot of the ^{14}C-CGA-329351 stock solution was placed into a sterile one liter Erlenmeyer flask and evaporated under a nitrogen stream. A 350 mL aliquot of prepared sterile 0.01M calcium ion solution was placed into the flask to dissolve the CGA-329351. Dissolution of the CGA-329351 was aided by brief sonication. Using sterile silylated Erlenmeyer flasks, a set of five dilutions of this prepared solution resulted in solutions for the definitive phase. The final concentrations for the definitive phase of the study were: 0.0, 0.206, 0.513, 1.028, 5.145, and 10.217 µg/mL.

All test solutions were prepared immediately prior to use. The actual test solution concentration, solubility and homogeneity of three aliquots were determined prior to use by LSC. The concentration of radioactivity in each LSC aliquot was within 3% of the respective mean concentration and the radioactivity was considered to be homogeneously dispersed in the test solution. The pH of each test solution was also measured prior to use. Aliquots of each of the test solutions were analyzed by HPLC and TLC.

Test System

The test system for this study was each capped Oak Ridge Teflon® centrifuge tube with soil, aqueous calcium ion solution and ^{14}C-CGA-329351. Each test system was identified by project number, soil type, replicate, concentration, date, and technician initials.

Solubility Determination

An estimate of the water solubility of ^{14}C-CGA-329351 was performed by placing approximately 0.5 mg (40.6 µCi) of ^{14}C-CGA-329351 (1.0 mg/mL acetonitrile) into duplicate aluminum foil covered silylated glass scintillation vials. The acetonitrile was evaporated and 2 mL of sterile, deionized distilled water was added to each vial with a maximum target concentration of 250 ppm. Each vial was sonicated for approximately five minutes, manually shaken for approximately 0.5 minutes, then placed into a laboratory incubator at 25±1°C. At the sampling times of 0, 3, 6, 24, 28, and

48 hours, the vials, except 0 time, were removed from the incubator, sonicated for approximately five minutes, then manually shaken for approximately 0.5 minutes, and centrifuged at approximately 2500 rpm for approximately fifteen minutes (APPENDIX D, page 167). Triplicate aliquots were removed for radioanalysis. Prior to placing the vials back into the incubator, the vials were sonicated for approximately five minutes then manually shaken for approximately 0.5 minutes. The solubility determination was performed for forty eight hours with pH determined at the final sampling time. The approximate solubility of CGA-329351 was 250 ppm in water at 25±1°C and pH 7.1 (APPENDIX D, Page 167a).

Preliminary Phase

Prior to the definitive test, each soil was tested to establish adsorption equilibrium time and the optimum ratio of soil to test solution for use in the definitive adsorption and desorption phases of the study. Additionally, the preliminary phase was used to determine if adsorption to the test container or cap occurred. The preliminary phase was performed in duplicate using an actual concentration of 10.641 $\mu g/mL$ of ^{14}C-CGA-329351 in aqueous 0.01M $CaCl_2$ solution (the highest concentration of test substance to be tested).

Duplicate 1 g samples of each air dried and sieved soil type were weighed into 50 mL centrifuge tubes (Oak Ridge Teflon® FEP). All tubes were capped with Teflon® screw cap lids. Twenty mL each of the test solution was transferred to the representative tube and capped. Replicate blank centrifuge tubes containing only the test solution, i.e., no soil, were included to detect any adsorption of the test substance to the centrifuge tubes. The soil/test solution tubes were placed in a laboratory HotPack incubator, and equilibrium determined by shaking in the dark using an Eberbach shaker at 175 to 200 rpm at a constant temperature of 25±1°C. Duplicate tubes of each soil/test solution were removed from the incubator at the approximate time intervals of 2, 4, 8, and 24 hours. The 0 time sampling was the analysis of the test solution directly. The blank tubes were removed at the 24 hours time interval. At each sampling time, the tubes were removed from the incubator, centrifuged at >1000 rpm for fifteen minutes to pellet the soil, and aliquots of the supernatant removed. Duplicate aliquots of 100 μL of the supernatant were removed and analyzed by LSC to determine the concentration of ^{14}C-CGA-329351 in solution. The radioactivity "lost" from the test solution (supernatant) was a measure of the test substance adsorption to soil. After removal of the aliquots for LSC analysis, the soil pellet was resuspended in the test solution by vortexing and manual shaking then returned to the incubator to continue shaking in the dark using an Eberbach shaker at 175 to 200 rpm at 25±1°C until the next time interval sampling. Equilibrium was obtained by 24 hours.

The adsorbed concentration was determined at the final equilibrium analysis (24 hours) by combustion of the pelleted soil. At the 24 hours time interval sampling, the centrifuge tubes were removed from the incubator and centrifuged as described above. The supernatant was decanted, retained in silylated liquid scintillation vials, and aliquots removed for LSC analysis. The total recovery of radioactivity (material balance) was determined for each soil/test solution at the last time interval. The sum of the radioactivity recovered in the test solution and the pellet as a proportion of the radioactivity applied was the material balance.

The stability of ^{14}C-CGA-329351 in aqueous 0.01M $CaCl_2$ solution at the end of the 24 hours equilibrium period was evaluated by the analysis of one of the replicate blank centrifuge tubes by HPLC. The chromatography of the blank centrifuge tube solution at the equilibrium time was compared with the 0 time chromatography (FIGURE 3).

Except for the MS clay soil, the amount of applied radioactivity bound to the soil during the preliminary phase was not the protocol specified 20 to 80% (APPENDIX D, page 179). Therefore, the preliminary phase of the study was repeated exactly as described above except that the ratio of solution to soil was changed. The clay soil and soil-less blanks were not repeated. Instead of 1 g soil to 20 mL of solution for each soil type, approximately 10 g of sandy loam and sand were used and 4 g of loam and silty clay loam were used per 20 mL of aqueous 0.01M calcium ion solution. These solution to soil ratios were 2:1 (highest protocol ratio) for sand and sandy loam and 5:1 for loam and silty clay loam. In the repeat preliminary phase of the study, the amount of applied radioactivity bound to the soil was 6.0 to 39.8% with the MS clay at 45.8 to 52.8% from the original preliminary testing (TABLE VII).

Based upon the rate of test substance adsorption to soil over time (estimated K_d as determined from $x/m = K_d C_e$, see CALCULATIONS section), an equilibrium adsorption time was selected for the definitive phase of the study. The criteria for selection of the soil/test solution ratio was based on the preliminary equilibrium constant (K_d) determination as follows:

Estimated K_d	Solution: Soil Ratio
≤ 1	2 mL:1 g
$>1 \quad \leq 10$	5 mL:1 g
$>10 \quad \leq 50$	20 mL:1 g
$>50 \quad \leq 400$	100 mL:1 g
>400	400 mL:1 g

Definitive Phase

Adsorption - Based upon the equilibrium time and test solution to soil ratios as determined in the repeat preliminary phase, the definitive adsorption phase of the study was performed. The selected solution to soil ratio based on the preliminary phase work was 20 mL solution to 10 g for the Maryland sand and California sandy loam soils, a ratio of 2 mL solution to 1 g soil. The Washington loam and Arizona silty clay loam soils were performed at 4 g soil to 20 mL of solution, a ratio of 5 mL to 1 g soil. The Mississippi clay soil was performed at 1 g soil to 20 mL of solution, a ratio of 20 mL to 1 g soil. Duplicate samples of air dried soil were weighed into centrifuge tubes for each test solution concentration. An appropriate volume of the test solution at 0.0, 0.206, 0.513, 1.028, 5.145, and 10.217 ppm was transferred to the appropriate centrifuge tubes and capped. The pH of the test solution was determined on a portion of each test solution and that portion was not added to any centrifuge tube. The 0.0 ppm concentration served as a control for determination of background radioactivity from undosed soil. Values obtained from these controls were used to demonstrate that the soil had not been previously exposed to radiocarbon. The entire sample set was shaken in the dark for the equilibrium time (4 hours) using an Eberbach shaker at 175 to 200 rpm in a constant temperature incubator at 25±1°C (HotPack incubator). At the equilibrium time, the tubes were removed from the incubator and centrifuged at >1000 rpm for fifteen minutes to pellet the soil. The supernatant was decanted, retained in silylated liquid scintillation vials, and triplicate 0.5 mL aliquots removed for LSC analysis. LSC analysis of the supernatant determined the equilibration concentrations (C_e) of CGA-329351 in all test solutions. The actual concentration of CGA-329351 adsorbed (x/m) was determined by difference with correction for the amount of radioactivity present in the interstitial water (water remaining with the soil samples).

The supernatant from one replicate at each concentration for each soil type was analyzed for pH determination (Corning 240 pH meter, data presented in TABLE IV). Additionally, the supernatant from one replicate per soil type at the highest concentration (10.217 ppm) was analyzed by TLC and HPLC.

Desorption - Desorption was performed on the soil samples following adsorption. Calcium chloride aqueous solution at a concentration of 0.01M without test substance was added to all centrifuge tubes containing soil pellets used for adsorption. The volume of 0.01M $CaCl_2$ solution added was the same as the volume used for the (definitive) adsorption phase. The soil pellet was then suspended by vortexing and manual shaking until dispersed. Soil samples at the 0.0 ppm level were also desorbed to demonstrate radiocarbon free calcium ion solution and no radiocarbon desorption from the control soils. The entire sample set was shaken in the dark for the equilibrium time (4 hours) using an Eberbach shaker at 175 to 200 rpm in a constant temperature incubator at 25±1°C. At the equilibrium time, the tubes are removed from the incubator, centrifuged at >1000 rpm for fifteen minutes to pellet the soil, and the supernatant was removed. Triplicate 1 mL aliquots of each supernatant were removed and analyzed by LSC to determine the equilibration concentrations (C_e) of CGA-329351. Desorption soil concentration (x/m) was determined by combustion of the residual soil with correction for the amount of radioactivity present in the interstitial water.

Additionally, the desorption supernatant from one replicate per soil type at the highest concentration (10.217 ppm) was analyzed by TLC and HPLC.

The material balance was calculated for every sample by adding the total dpm in the adsorption supernatant, plus the dpm in the desorption supernatant, plus the dpm in the soils combusted following desorption. The target recovery was ≥90% and ≤110%.

The soil pellet from the highest dose level (10.217 ppm) of each soil type was extracted by treating the soil with a 1:1 solution of 90:10 acetonitrile/water and sonicating (Branson 2210) for fifteen minutes with periodic ≈5 second vortexing (S/P Deluxe Mixer). The samples were then centrifuged at ≈1000 rpm for fifteen minutes to pellet the soil. The extract was decanted, and the soil was sonicated/centrifuged once more. The combined extract was radioassayed in triplicate (100 µL aliquots). The remaining soil pellet was air dried at ambient laboratory temperatures in a laboratory hood. Aliquots of the air-dried soil pellet were oxidized by combustion and the resulting radioactive carbon dioxide ($^{14}CO_2$) was analyzed by LSC. Additionally, an aliquot of the pooled extract was analyzed by TLC and HPLC to determine the amount of test substance remaining in the soil after desorption.

ANALYTICAL METHOD

Radiocarbon Analysis

All solutions were analyzed (preliminary phase in duplicate, definitive phase in triplicate) by direct LSC in appropriate scintillation fluor. Soils were oxidized in aliquots of 200 mg or greater (preliminary phase in duplicate, definitive phase in triplicate) except MS clay (50 mg or greater) using an R.J. Harvey Instruments Corporation Biological Materials Oxidizer (BMO). BMO generated $^{14}CO_2$ was trapped in Oxosol scintillation fluor and quantified by LSC. For the definitive phase, the machine efficiency of the oxidizer was determined before every series of soil combustions by oxidizing a known amount of radioactive standard. The machine combustion efficiencies were 95% or greater. The efficiency of the soil oxidation procedure was determined before every series of soil combustions using aliquots of each untreated soil type fortified with known amounts of radioactive standard (definitive phase only). For the preliminary phase, the oxidation efficiency was determined with CA sandy loam soil only. The amount of radioactivity recovered from each sample divided by the amount of radioactivity applied to the sample determined an oxidation efficiency factor (sample efficiency). For oxidation efficiencies <100%, the recoveries were adjusted. Sample combustion efficiencies were 93.94 to 98.26% (see combustions in APPENDIX D).

All quantification was performed using two channel counting for five minutes, or two sigma, with a Beckman Liquid Scintillation Spectrophotometer (LS3801, Irvine, CA). Obtained counts per minute (cpm) were converted to disintegrations per minute (dpm) using the external standard ratio and math package features of the instrument.

TLC Analysis

Aliquots of the radioactive test substance, one replicate of the 10.217 ppm adsorption and desorption solutions for each soil type, and the desorption soil extracts were analyzed by two dimensional thin-layer chromatography (TLC) using 20 x 20 cm glass plates precoated with silica gel (Merck - silica gel 60 F-254) at a thickness of 0.25 mm. All plates were scored at 15 cm above the origin in the first and second directions to provide a uniform solvent front.

Development of TLC plates was performed in the following specified solvent systems:

First Dimension:	chloroform/methanol/ammonium hydroxide/ water 80:30:4:2 (v:v:v:v),
Second Dimension:	chloroform/methanol/formic acid/water 75:20:4:2 (v:v:v).

Following sample application and air drying, plates were eluted to 15 cm above the origin. Following elution and air drying, plates were rotated 90° and eluted to 15 cm above the origin in the second dimension. After the second elution and air drying, nonradioactive standards were visualized by UV 254 nm light and their positions marked on the TLC plates with a soft lead pencil. TLC plates were evaluated using an AMBIS Radioanalytical Scanner (AMBIS Systems, Inc., San Diego, CA). The scanner provided a radioanalytical image of the TLC plate and quantified all radioactive areas. As an additional verification, 10% or more of the plates quantitated by the AMBIS were evaluated by scraping radioactive zones from the plate and analyzing by LSC.

HPLC Analysis

Samples of the radioactive test substance, all test solutions, one replicate of the 10.217 ppm adsorption and desorption solutions for each soil type, and the desorption soil extracts were chromatographed by HPLC on a YMC ODS-AQ (YMC Inc., Wilmington, NC) analytical column (250 x 4.6 mm) using the solvent system described below. The components separated during the elution were quantified using a RAMONA-5-LS Radioactivity Monitor. As an additional verification, 10% or more of the samples quantitated by the HPLC RAM were evaluated by collecting fractions from the HPLC and analyzing by LSC. Analytical standards were chromatographed each time samples were analyzed. The standards were diluted in acetonitrile-UV.

HPLC

Instrument:	Shimadzu SCL-6A HPLC System
Detector:	230 nm UV; RAM ^{14}C (RAMONA 5-LS)
Loop:	500 µL (nominal)
Column:	YMC ODS-AQ 250 x 4.6mm
Gradient Solvent System:	A = 0.0125% trifluoroacetic acid (TFA)/ acetonitrile 90:10 (v:v) B = acetonitrile

Gradient Elution:

Time Minutes	A %	B %
0	90	10
25	60	40
30	30	70
35	30	70
45	90	10
50	90	10
50	Stop	

Run Time:	35 Minutes
Flow Rate:	1 mL/min.
Sensitivity:	0.002 Absorbance
Chart Speed:	5 mm/min
Temperature:	Ambient, room temperature
Approximate Retention Time of ^{14}C-CGA-329351:	30 minutes

Detection Limits

Detection limits (APPENDIX D, page 165) were determined using methods detailed by Wang & Willis (Reference 8). Detection limits were calculated based on (1) a counting volume of 1 mL for desorption, 0.5 mL for adsorption, 0.1 mL for preliminary phase, (2) an average soil sample of 0.23 g for combustions (0.06 g clay), and, (3) a specific activity of ^{14}C-CGA-329351 of 180042 dpm/µg. For this study, the following detection limits were determined:

Soil Combustions	0.0011 ppm (0.0041 ppm, clay)
Preliminary Phase Solutions	0.0018 ppm,
Adsorption Solutions	0.0004 ppm,
Desorption Solutions	0.0002 ppm.

Calculations

The Freundlich equation (Reference 1) is commonly used to evaluate batch equilibrium data. The equation is an empirical relationship which may be expressed as:

$$x/m = K_d C_e^{1/n} \text{ or } \ln x/m = \ln K_d + 1/n \ln C_e$$

where x/m is the soil equilibrium concentration in µg/g, C_e is the aqueous phase equilibrium concentration in µg/mL, K_d is the Freundlich sorption constant and n is a constant.

The values of ln C_e versus ln x/m were plotted for adsorption and desorption. The constants K_d and n were determined from the slope (1/n) and intercept (ln K_d) of the resultant straight line by linear regression.

The sorption constant (K_d) was also expressed in terms of the soil organic carbon content using the following equation:

$$K_{oc} = (K_d \times 100) / \%\ \text{O.C.}$$

where K_{oc} is the sorption coefficient based on soil organic carbon content and % O.C. is the organic carbon content of the soil. The organic carbon content of the soil was calculated by dividing the organic matter content by 1.724 (Reference 9). See APPENDIX D, pages 191 and 192 for example calculations.

Statistical Methods

Mathematical methods of linear regression (used to calculate K_d and n values), means, percent variance, sums, and logs were used for data reduction. No other statistical methods were used.

Bias Contaminants

No experimental bias was expected in this study. Also, no contaminants were experienced during the study that were known to be capable of interfering with the purpose and conduct of the study.

RESULTS AND DISCUSSION

Soils

A total of five representative soil textures were used in this study. Soil characteristics, soil microbial biomass, and clay mineralogy obtained from AGVISE Laboratories, Ciba Crop Protection, and North Dakota State University, respectively, are presented in TABLE I (APPENDIX C). The soil order name and soil series name were obtained from N.C. Brady (Reference 10) and USDA soil maps, respectively. The five soils represented a range of soils with organic matter content of <1% (MD sand, CA sandy loam, AZ silty clay loam) to 2.6% (WA loam), and pH of 5.4 (MD sand) to 7.9 (AZ silty clay loam). The organic carbon (% organic matter/1.724) content for all soils was 1.5% (WA loam) or less. The microbial viability of each of the five soils was determined prior to use, or concomitantly, by spread plating technique (TABLE II). All soils showed varied, viable microbial populations.

Incubation Temperature

The temperature of the HotPack incubator was monitored continuously during all phases of the study. Continuous chart recordings of the incubation temperature showed 25±1°C. Additionally, at each sampling point of the preliminary phase and definitive phase, the temperature was manually recorded (TABLE III).

pH Determinations

Measurement of pH was performed on the prepared aqueous calcium chloride solution (6.93) following preparation. The stock solution prepared for the preliminary phase (pH 7.54 and 7.21, performed twice) as well as the solutions prepared for the definitive phase (pH 6.92 to 7.10) were assayed for pH. Following the adsorption phase of the definitive phase of the study, pH determinations were made on the decanted adsorption solutions. The MD sand soil significantly reduced the pH (5.50 to 5.82) but no other soil seemed to significantly affect the pH (range 6.39 to 7.25) (TABLE IV and APPENDIX D, pages 169, 170, and 190).

Preliminary Phase

Results of the preliminary phase testing are presented in TABLES V through VII. Data in TABLE V show that equilibrium in all soil types was reached rapidly. Solution equilibrium was achieved within 4 hours as demonstrated by only slight changes in the concentration of test substance in the subsequent aqueous sample. Therefore, the equilibrium shaking time selected for the definitive phase was 4 hours.

The 4 hour estimated K_d values shown in TABLE VI indicated the K_d of the clay soil would range between 10 and 50. The approximate sand and sandy loam 4 hour estimated soil K_d values were less than 1 and the loam and silty clay loam values were between 1 and 10. Based on these estimated values, a solution to soil ratio of 2:1 (20 mL solution to 10 g of soil) was selected for the Maryland sand and California sandy loam, 5:1 (20 mL solution to 4 g soil) was selected for Washington loam and Arizona silty clay loam, and 20:1 (20 mL solution to 1 g soil) was selected for the Mississippi clay.

Mass balance for the preliminary phase was determined by summation of the total radioactivity in the equilibration solution plus the total radioactivity in the combusted soils. This total activity recovered was divided by the total activity dosed to determine the mass balance. The mass balance obtained during the preliminary phase ranged from 89.8 to 103.2% (TABLE VII). The soil-less "blank" tubes demonstrated no adsorption of CGA-329351 with 99.8 to 99.9% accountability of radioactivity at the end of the 24 hour equilibrium period (APPENDIX D).

Definitive Phase

The mean measured concentrations for the duplicate samples for each chemical concentration in solution and in soil for the adsorption phase of the study are presented in TABLES VIII and IX. The mean measured concentrations for the duplicate samples for each chemical concentration in solution and in soil for the desorption phase of the study are presented in TABLES X and XI. The ppm values for each sample were determined by dividing the total dpm by the specific activity (SA = 180042 dpm/μg) and the sample mass. The total radioactivity balance in the definitive phase is presented in TABLE XII for each sample replicate of each soil type. The average radiocarbon balance for each soil type ranged from 98.2 to 101.4% of applied with individual replicates demonstrating a range of 97.5 to 102.9%.

Using the empirical Freundlich isotherm, values of n and K_d were determined from the adsorption data (TABLE XIII). The correlation coefficients for the adsorption phase ranged from 0.9918 to 0.9995. These high correlation coefficients demonstrate that the adsorption model was studied and that the Freundlich equation was applicable to the test system. The logarithmic plots of C_e (equilibrium solution concentration) versus x/m (equilibrium soil concentration) are presented as straight lines for the adsorption phase (FIGURE 2). Linear regression analysis used to calculate the Freundlich equation for adsorption is presented in TABLE XIII. The adsorption soil constants (K_d) ranged from 0.1 (MD sand) to 7.6 (MS clay) for the five soil textures. Calculation of the K_{oc} (adsorption coefficient based on organic carbon) yielded values of 20 (MD sand) to 1299 (AZ silty clay loam). These K_{oc} values placed CGA-329351 in the low to very high mobility class (K_{oc} = 0 - 2000, Reference 1).

Again, using the empirical Freundlich isotherm values of n and K_d were determined from the desorption data (TABLE XIV). The correlation coefficients obtained for the desorption phase were 0.9862 to 0.9997. The logarithmic plots of C_e versus x/m are presented as straight lines in FIGURE 2. The linear regression analysis of the desorption data used to calculate the Freundlich equation is presented in TABLE XIV. The desorption soil constants (K_d) were found to be 1.8 (CA sandy loam) to 18.4 (MS clay) for the five soil textures tested. Calculation of the K_{oc} (desorption coefficient based on organic carbon) yielded values of 195 (WA loam) to 3539 (AZ silty clay loam). These K_{oc} values placed CGA-329351 in the slight to medium mobility class (K_{oc} 150 - 5000, Reference 1).

Soil Extraction

Following completion of desorption of the definitive phase, the soil samples remaining at the highest tested concentration (10.217 ppm) were extracted. The extraction of the soil pellet was performed to determine the amount of test substance remaining in the soil following adsorption and desorption. Extractability varied ranging from 85.7 to 101.5% of the available radioactivity. Combustion of the non-extracted residue accounted for the remaining radiocarbon balance. Total balance (TABLE XV) for the residual soil, extraction, and combustion averaged 100.4% (range 90.8 to 109.7%).

HPLC

High performance liquid chromatography (HPLC) was used as the primary analytical technique to verify the percent CGA-329351 remaining in selected samples from this adsorption/desorption study. Prior to the start of the study, the radiochemical purity of ^{14}C-CGA-329351 (95-041) was verified to be >99% (FIGURE 3). Following the purity check, the percent of ^{14}C-CGA-329351 present in the 48 hour solutions from the solubility check were verified to be >99%. Samples from the blank and each soil type from the preliminary phase testing were 97.6 to 99.0% CGA-329351 following the preliminary phase (FIGURE 3 and TABLE XVI). TABLE XVI provides the quantitation results from the RAMONA radioactivity monitor (RAM) for analysis of the definitive phase dose solutions, adsorption solutions, desorption solutions, and the extractions of the soil remaining after desorption. In all cases the RAM quantitated CGA-329351 at 91.8% or greater (FIGURES 6, 8, and 10).

TLC

Two dimensional thin layer chromatography (TLC) was used to verify the percent CGA-329351 remaining in selected samples. Prior to start of the study, the radiochemical purity of ^{14}C-CGA-329351 (95-041) was verified to be >99%. The radioactivity from the 48 hour aqueous solubility check was >99% ^{14}C-CGA-329351. Samples from the soil-less blank and each soil type from the preliminary phase testing were 98.7 to 99.5% CGA-329351 following the preliminary phase (FIGURE 4, TABLE XVII). One replicate of the highest concentration for adsorption and desorption solutions from each soil type and aliquots of each post desorption soil extract were analyzed by two dimensional TLC. In all cases, the AMBIS scanner quantitated CGA-329351 at 97.7% or greater (TABLE XVII, FIGURES 5, 7, and 9).

Verification of AMBIS and RAM Quantitation

Selected samples were scraped and counted by LSC (TLC plate verification) or fraction collected and counted by LSC (HPLC verification) to verify the quantitation provided by the AMBIS scanner and the RAMONA RAM detector. The results of those analyses are presented in TABLE XVIII. The results support the AMBIS and RAM quantitations. No product, except CGA-329351 was observed at greater than 10% from the instrument (AMBIS or RAM) analysis or in the manual plate scraping and fraction collecting. The scraping of TLC plates yielded a 104.8% average radiocarbon recovery with 93.2% average CGA-329351 detection. The fraction collections from the HPLC resulted in a 94.4% average radiocarbon recovery with 97.3% average CGA-329351 detection. The manual plate scraping for TLC and fraction collections for HPLC are comparable to the AMBIS and RAM quantitations (see TABLE XIX).

Test and Reference Substance Stability

The stability of CGA-329351 at the testing facility and in the test system was demonstrated (FIGURES 3 to 11, TABLES XVI to XIX). All HPLC and TLC analyses verified CGA-329351 as the radioactive species present. No other product was observed. The stability of the test and reference substances at the testing facility was demonstrated by analysis of the substances at the beginning (HPLC, FIGURE 3 and TLC, FIGURE 4) and end of the study (HPLC, FIGURE 11). Those analyses qualitatively demonstrated the stability of the substances at the test facility.

CONCLUSIONS

The following conclusions resulted from the adsorption and desorption of ^{14}C-CGA-329351.

(1) CGA-329351 K_d values were 0.1 (sand), 0.2 (sandy loam), 1.3 (loam), 2.3 (silty clay loam), and 7.6 (clay) from adsorption.

(2) CGA-329351 K_d values were 1.8 (sandy loam), 2.8 (sand), 2.9 (loam), and 6.2 (silty clay loam), and 18.4 (clay) from desorption.

(3) CGA-329351 K_{oc} values were 20 (sand), 68 (sandy loam), 86 (loam), 570 (clay), and 1299 (silty clay loam) from adsorption.

(4) CGA-329351 K_{oc} values were 195 (loam), 785 (sandy loam), 790 (sand), 1376 (clay), and 3539 (silty clay loam) from desorption.

(5) CGA-329351 was classified as low to very high mobility class based on K_{oc} values from 25 to 1500.

(6) CGA-329351 was observed to be stable during the adsorption and desorption study using the batch equilibrium method.

TABLE I: SOIL CHARACTERIZATION - SOILS USED FOR ^{14}C-CGA-329351 ADSORPTION AND DESORPTION

SOURCE	LELAND MISSISSIPPI	BURTONSVILLE MARYLAND	SANGER CALIFORNIA	YAKIMA WASHINGTON	YUMA ARIZONA
Date Collected	6/2/94	6/8/95	5/22/95	5/30/94	6/7/95
Texture	Clay	Sand	Sandy Loam	Loam	Silty Clay Loam
Order Series Horizon	Inceptisol Sharkey A	Ultisol Sassafras A	Entisol Handford A	Mollisol Harwood A	Aridisol Gadsden A
% Sand % Silt % Clay	23 26 51	91 6 3	65 30 5	49 42 9	17 52 31
Clay Mineralogy*	Smectite Kaolinite Chlorite Illite	Kaolinite Chlorite Illite	Illite Kaolinite	Smectite Kaolinite Illite	Smectite Kaolinite Chlorite Illite
Organic Matter %	2.3	0.6	0.4	2.6	0.3
Organic Carbon %**	1.334	0.348	0.232	1.508	0.174
pH	7.0	5.4	6.3	7.0	7.9
Field Capacity % at 1/3 bar FMC	41.8	11.5	18.2	35.0	34.1
Cation Exchange Capacity (meq/100g)	37.8	4.1	8.0	22.7	50.4
Bulk Density (g/mL)	1.20	1.52	1.41	1.14	1.18
Soil Microbial Biomass *** mgC/Kg Soil	172.5	152.6	116.6	443.6	56.0

Soil characterization was performed by AGVISE Laboratories, Northwood, North Dakota.

* North Dakota State University, not performed in compliance with US EPA GLPS.
** Organic carbon % = organic matter %/1.724.
*** Performed at Ciba Crop Protection, Greensboro, NC.

TABLE II: SOIL MICROBIAL COUNTS FROM SOILS USED FOR ^{14}C-CGA-329351 ADSORPTION AND DESORPTION

SOIL TYPE	TOTAL COUNTS IN COLONY FORMING UNITS (CFU) PER GRAM SOIL AEROBIC BACTERIA	ACTINOMYCETES	FUNGI
Mississippi Clay	>3.0×10^{8}	1.2×10^{8}	3.3×10^{5}
Maryland Sand	8.9×10^{6}	4.0×10^{7}	3.1×10^{5}
California Sandy Loam	4.5×10^{7}	5.3×10^{7}	5.9×10^{4}
Washington Loam	1.6×10^{8}	1.9×10^{8}	2.8×10^{5}
Arizona Silty Clay Loam	3.1×10^{7}	8.8×10^{6}	3.0×10^{4}

Microbial counts determined using spread plating technique (Reference 7) with the following media:

plate count agar for total aerobic bacteria,
actinomycetes isolation agar for total actinomycetes,
rose bengal agar for total fungi.

All microbiological media was incubated under aerobic conditions at 25±1°C for 12 days.

TABLE III: INCUBATOR TEMPERATURE

TIME	DATE	SAMPLE INTERVAL	TEMPERATURE
Preliminary Phase			
9:30 AM	7/18/95	0 Time	24°C
11:30 AM	7/18/95	2 Hours	24°C
12:00 NOON	7/18/95		24°C
1:30 PM	7/18/95	4 Hours	25°C
1:45 PM	7/18/95		25°C
5:30 PM	7/18/95	8 Hours	25°C
5:45 PM	7/18/95		25°C
9:30 AM	7/19/95	24 Hours	24°C
Repeat Preliminary Phase			
9:25 AM	8/02/95	0 Time	25°C
11:30 AM	8/02/95	2 Hours	25°C
11:55 AM	8/02/95		25°C
1:25 PM	8/02/95	4 Hours	25°C
1:45 PM	8/02/95		25°C
5:25 PM	8/02/95	8 Hours	25°C
5:45 PM	8/02/95		25°C
9:25 AM	8/03/95	24 Hours	25°C
Definitive Phase			
Adsorption			
9:40 AM	8/17/95		25°C
1:40 PM	8/17/95	4 Hours	25°C
Desorption			
3:45 PM	8/17/95		25°C
7:45 PM	8/17/95	4 Hours	25°C

Continuous temperature chart recorder showed temperature was ±1°C during study periods.

TABLE IV: THE pH OF STOCK SOLUTIONS AND REMAINING SUPERNATANT FROM ADSORPTION SOLUTIONS FOR THE ADSORPTION AND DESORPTION OF ^{14}C-CGA-329351

SAMPLE	REP	pH MEASUREMENTS AT CONCENTRATION[a] 0.0	0.2	0.5	1.0	5.0	10.0
Definitive Stock Solutions[b]		7.05	7.04	7.10	7.01	6.92	7.03
MS Clay	1	7.25	7.24	7.16	7.10	7.10	7.10
MD Sand	1	5.82	5.73	5.64	5.60	5.52	5.50
CA Sandy Loam	1	6.39	6.42	6.40	6.41	6.43	6.46
WA Loam	1	7.11	7.05	7.00	7.00	7.01	7.02
AZ Silty Clay Loam	1	7.19	7.16	7.10	7.23	7.12	7.24

[a] Nominal solution ^{14}C-CGA-329351 concentration in μg/mL (ppm).

[b] Aliquot of dose stock solutions not added to soil tubes.

TABLE V: AVERAGE CONCENTRATION OF ^{14}C-CGA-329351 IN SOIL AND AQUEOUS SOLUTIONS DURING PRELIMINARY PHASE AT A WATER TO SOIL RATIO OF 20:1 (mL:g) - EXPRESSED AS ppm ^{14}C-CGA-329351

EQUILIBRIUM SHAKING TIME	REP	SOIL TYPE MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILTY CLAY LOAM
Solution (μg/mL)						
2 Hours	1	4.725	10.120	8.073	7.964	6.335
	2	4.717	10.277	7.976	7.937	6.333
	x̄	4.721	10.199	8.025	7.951	6.334
4 Hours	1	4.743	9.995	7.418	7.945	6.405
	2	4.732	10.024	7.386	7.988	6.450
	x̄	4.738	10.010	7.402	7.967	6.428
8 Hours	1	4.783	9.714	7.866	7.779	6.149
	2	4.684	9.846	7.593	7.842	6.615
	x̄	4.734	9.780	7.730	7.811	6.382
24 Hours	1	4.678	9.636	7.410	7.945	6.183
	2	4.546	9.860	7.466	7.962	6.184
	x̄	4.612	9.748	7.438	7.954	6.184
Detection Limit = 0.0018 ppm						
Soil (μg/g)						
24 Hours	1	78.36	1.23	5.03	11.53	20.61
	2	91.07	1.71	4.95	11.34	20.50
	x̄	84.72	1.47	4.99	11.44	20.56

Detection Limit = 0.0012 ppm
(0.0041 ppm MS clay)

TABLE VI: ESTIMATED K_d VALUES FROM PRELIMINARY PHASE ($x/m = K_dC_e$)*

EQUILIBRIUM SHAKING TIME	SOIL TYPE MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILTY CLAY LOAM
2 Hours	17.9	0.1	0.6	1.4	3.2
4 Hours	17.9	0.1	0.7	1.4	3.2
8 Hours	17.9	0.2	0.6	1.4	3.2
24 Hours	18.4	0.2	0.7	1.4	3.3

* Assumes 1/n = 1 in the Freundlich adsorption isotherm. Soil concentration (x/m) determined by oxidation of soils at 24 hours.

TABLE VII: RADIOCARBON BALANCE FOR PRELIMINARY PHASE - SOILS COMBUSTED AFTER 24 HOURS OF ADSORPTION SHAKING - BALANCE DETERMINED BY SUMMATION OF PERCENT OF DOSE* ON SOIL AND PERCENT OF DOSE IN SOLUTION FOR EACH REPLICATE OF EACH SOIL TYPE

SOIL TYPE	REP	TOTAL dpm** SOIL ($x\ 10^6$)	SOLUTION ($x\ 10^6$)	PERCENT OF DOSE SOIL	SOLUTION	TOTAL
Mississippi Clay	1	17.535	16.846	45.8	44.0	89.8
	2	20.232	16.368	52.8	42.7	95.5
Maryland Sand	1	2.231	34.699	6.0	92.8	98.8
	2	3.093	35.505	8.3	94.9	103.2
California Sandy Loam	1	9.123	26.682	24.4	71.3	95.7
	2	9.016	26.882	24.1	71.9	96.0
Washington Loam	1	8.322	28.608	22.2	76.5	98.7
	2	8.620	28.670	23.0	76.6	99.6
Arizona Silty Clay Loam	1	14.888	22.265	39.8	59.5	99.3
	2	14.791	22.267	39.5	59.5	99.0
Soil-Less Blank	1	NA	38.295	NA	99.9	99.9
	2	NA	38.246	NA	99.8	99.8

* Dose = 3.741×10^7 dpm per replicate (3.832×10^7 dpm/rep MS Clay and Blank).

** Determined by direct liquid scintillation counting of solutions and by oxidation of soils

NA = Not applicable

TABLE VIII: MEAN MEASURED CONCENTRATION (C_e) OF ^{14}C-CGA-329351 IN SOLUTION AT THE ADSORPTION PHASE OF THE STUDY

ADSORPTION TEST SOL. CONC.	REP	SOIL TYPE (µg/mL) MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILTY CLAY LOAM
0 µg/mL	1	0.000	0.000	0.000	0.000	0.000
	2	0.000	0.000	0.000	0.000	0.000
0.206	1	0.163	0.198	0.190	0.165	0.147
	2	0.165	0.198	0.189	0.161	0.148
0.513	1	0.404	0.497	0.475	0.410	0.376
	2	0.406	0.493	0.475	0.410	0.376
1.028	1	0.816	0.982	0.948	0.820	0.727
	2	0.799	0.987	0.949	0.820	0.731
5.145	1	3.012	4.988	4.807	3.989	3.273
	2	3.093	4.963	4.786	4.017	3.245
10.217	1	4.785	9.966	9.459	7.792	6.070
	2	4.960	10.005	9.525	7.820	6.174

Detection Limit = 0.0004 ppm.

TABLE IX: MEAN CONCENTRATION (x/m) OF ^{14}C-CGA-329351 IN SOIL AT EACH CONCENTRATION IN THE <u>ADSORPTION PHASE</u> OF THE STUDY - CALCULATED BY DIFFERENCE

ADSORPTION TEST SOL. CONC.	REP	SOIL TYPE (µg/g)[a]				
		MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILT CLAY LOAM
0 µg/mL	1	0.000	0.000	0.000	0.000	0.000
	2	0.000	0.000	0.000	0.000	0.000
0.206	1	0.729	0.014	0.030	0.202	0.277
	2	0.715	0.016	0.032	0.211	0.280
0.513	1	1.839	0.031	0.074	0.501	0.679
	2	1.837	0.038	0.074	0.502	0.669
1.028	1	3.993	0.091	0.158	0.986	1.395
	2	4.010	0.081	0.158	1.021	1.376
5.145	1	34.940	0.311	0.671	5.677	9.082
	2	36.324	0.357	0.697	5.291	8.984
10.217	1	94.062	0.496	1.486	11.599	19.847
	2	99.283	0.421	1.365	11.671	19.330

[a] Concentrations were calculated by subtraction of all solution dpm (decanted and interstitial) from total dose dpm.

TABLE X: MEAN MEASURED CONCENTRATION (C_e) OF ^{14}C-CGA-329351 IN SOLUTION AT THE DESORPTION PHASE OF THE STUDY

ADSORPTION TEST SOL. CONC.	REP	SOIL TYPE (µg/mL)				
		MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILTY CLAY LOAM
0 µg/mL	1	0.000	0.000	0.000	0.000	0.000
	2	0.000	0.000	0.000	0.000	0.000
0.206	1	0.029	0.004	0.007	0.023	0.026
	2	0.028	0.004	0.006	0.025	0.025
0.513	1	0.074	0.010	0.014	0.061	0.065
	2	0.072	0.007	0.014	0.062	0.064
1.028	1	0.148	0.019	0.026	0.116	0.137
	2	0.153	0.020	0.021	0.118	0.135
5.145	1	1.138	0.048	0.116	0.641	0.811
	2	1.130	0.076	0.105	0.635	0.816
10.217	1	2.039	0.122	0.327	1.424	1.604
	2	2.093	0.121	0.266	1.436	1.585

etection Limit = 0.0002 ppm

TABLE XI: MEAN MEASURED CONCENTRATION (x/m) OF ^{14}C-CGA-329351 IN SOIL AT THE DESORPTION PHASE OF THE STUDY

ADSORPTION TEST SOL. CONC.	REP	SOIL TYPE (µg/g)[a] MISSISSIPPI CLAY	MARYLAND SAND	CALIFORNIA SANDY LOAM	WASHINGTON LOAM	ARIZONA SILTY CLAY LOAM
0 µg/mL	1	0.000	0.000	0.000	0.000	0.000
	2	0.000	0.000	0.000	0.000	0.000
0.206	1	0.318	0.013	0.014	0.089	0.147
	2	0.316	0.013	0.013	0.084	0.155
0.513	1	0.761	0.025	0.033	0.203	0.343
	2	0.723	0.024	0.029	0.200	0.353
1.028	1	1.438	0.045	0.063	0.381	0.695
	2	1.736	0.044	0.065	0.383	0.688
5.145	1	14.889	0.208	0.273	1.904	4.506
	2	17.078	0.167	0.274	1.914	4.594
10.217	1	58.714	0.388	0.466	4.131	11.276
	2	63.956	0.329	0.496	4.232	11.424

Detection Limit = 0.0011 ppm (0.0041 ppm MS clay)

Concentrations are calculated by subtraction of solution dpm present in soil at time of combustion from total dpm (soil and solution).

TABLE XII: RADIOCARBON BALANCE FOR DEFINITIVE BATCH EQUILIBRIUM TESTING OF ^{14}C-CGA-329351

ADSORPTION TEST SOLUTION CONCENTRATION (µg/mL)	REP	PERCENT OF DOSE[a] SOIL	WATER ADS	WATER DES	TOTAL
Mississippi Clay Soil					
0.206	1	9.1	79.1	14.3	102.5
	2	8.7	80.3	13.9	102.9
0.513	1	8.8	78.8	14.4	102.0
	2	8.2	79.2	14.1	101.5
1.028	1	7.4	79.4	14.4	101.2
	2	9.6	77.7	14.9	102.2
5.145	1	17.7	58.5	22.1	98.3
	2	18.8	60.1	22.0	100.9
10.217	1	33.2	46.8	20.0	100.0
	2	33.1	48.5	20.5	102.1
			AVERAGE ± 1 SD =		101.4±1.4
Maryland Sand Soil					
0.206	1	3.2	96.5	2.2	101.9
	2	3.2	96.1	2.1	101.4
0.513	1	2.5	96.9	1.9	101.3
	2	2.4	96.2	1.4	100.0
1.028	1	2.2	95.5	1.9	99.6
	2	2.1	96.1	1.9	100.1
5.145	1	2.0	97.0	0.9	99.9
	2	1.7	96.5	1.5	99.7
10.217	1	1.9	97.5	1.2	100.6
	2	1.6	97.9	1.2	100.7
			AVERAGE ± 1 SD =		100.5±0.8
California Sandy Loam Soil					
0.206	1	3.5	92.6	3.2	99.3
	2	3.3	91.9	3.1	98.3
0.513	1	3.2	92.7	2.8	98.7
	2	2.9	92.7	2.7	98.3
1.028	1	3.1	92.2	2.5	97.8
	2	3.2	92.3	2.0	97.5
5.145	1	2.7	93.4	2.3	98.4
	2	2.7	93.0	2.0	97.7
10.217	1	2.3	92.6	3.2	98.1
	2	2.5	93.2	2.6	98.3
			AVERAGE ± 1 SD =		98.2±0.5

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[a] See page 42.

TABLE XII: RADIOCARBON BALANCE FOR DEFINITIVE BATCH EQUILIBRIUM
(CONTINUED) TESTING OF ^{14}C-CGA-329351

ADSORPTION TEST SOLUTION CONCENTRATION (µg/mL)	REP	PERCENT OF DOSE[a] SOIL	WATER ADS	WATER DES	TOTAL
Washington Loam Soil					
0.206	1	8.8	80.2	11.4	100.4
	2	8.6	78.4	12.2	99.2
0.513	1	8.2	79.9	12.0	100.1
	2	8.0	80.0	12.2	100.2
1.028	1	7.8	79.8	11.3	98.9
	2	7.6	79.7	11.5	98.8
5.145	1	7.5	77.5	12.5	97.5
	2	7.9	78.1	12.3	98.3
10.217	1	8.5	76.3	13.9	98.7
	2	8.5	76.5	14.1	99.1
				AVERAGE ± 1 SD =	99.1±0.9
Arizona Silty Clay Loam Soil					
0.206	1	15.0	71.7	12.7	99.4
	2	15.4	72.1	12.1	99.6
0.513	1	13.5	73.3	12.6	99.4
	2	14.1	73.3	12.4	99.8
1.028	1	14.6	70.8	13.3	98.7
	2	14.4	71.1	13.2	98.7
5.145	1	18.1	63.6	15.8	97.5
	2	18.9	63.1	15.9	97.9
10.217	1	23.1	59.4	15.7	98.2
	2	23.4	60.4	15.5	99.3
				AVERAGE ± 1 SD =	98.8±0.8

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[a] Percent of dose:
SOIL = radiocarbon remaining on soil following desorption.
WATER - ADS = radiocarbon remaining in adsorption calcium ion solution and not adsorbed to soil.
DES = radiocarbon desorbed from soil and quantified in the desorption calcium ion solution.

TABLE XIII: LINEAR REGRESSION ANALYSIS OF THE ADSORPTION DATA USING THE FREUNDLICH ISOTHERM ($\ln x/m = \ln K_d + 1/n \ln C_e$) FOR ^{14}C-CGA-329351

ADSORPTION TEST SOL. CONC. (μg/mL)	REP	MISSISSIPPI CLAY ln C_e	MISSISSIPPI CLAY ln x/m	MARYLAND SAND ln C_e	MARYLAND SAND ln x/m	CALIFORNIA SANDY LOAM ln C_e	CALIFORNIA SANDY LOAM ln x/m	WASHINGTON LOAM Water ln C_e	WASHINGTON LOAM Soil ln x/m	ARIZONA SILTY CLAY LOAM ln C_e	ARIZONA SILTY CLAY LOAM ln x/m
0.206	1	-1.817	-0.317	-1.618	-4.238	-1.659	-3.507	-1.803	-1.601	-1.915	-1.282
	2	-1.802	-0.336	-1.622	-4.148	-1.666	-3.430	-1.826	-1.555	-1.909	-1.273
0.513	1	-0.906	0.609	-0.699	-3.470	-0.743	-2.598	-0.892	-0.690	-0.978	-0.387
	2	-0.901	0.608	-0.706	-3.260	-0.744	-2.605	-0.890	-0.689	-0.979	-0.401
1.028	1	-0.203	1.385	-0.019	-2.395	-0.054	-1.844	-0.198	-0.014	-0.318	0.333
	2	-0.224	1.389	-0.013	-2.519	-0.053	-1.847	-0.199	0.020	-0.314	0.319
5.145	1	1.103	3.554	1.607	-1.167	1.570	-0.399	1.383	1.736	1.186	2.206
	2	1.129	3.592	1.602	-1.029	1.566	-0.361	1.391	1.666	1.177	2.195
10.217	1	1.566	4.544	2.299	-0.701	2.247	0.396	2.053	2.451	1.803	2.988
	2	1.601	4.598	2.303	-0.864	2.254	0.311	2.057	2.457	1.820	2.962
LINEAR REGRESSION ANALYSIS											
Correlation (r)			0.9922		0.9918		0.9994		0.9995		0.9984
Slope (1/n)			1.449		0.892		0.970		1.045		1.159
Intercept			2.029		-2.659		-1.852		0.266		0.815
n			0.690		1.121		1.031		0.957		0.862
K_d			7.6		0.1		0.2		1.3		2.3
K_{oc}			570		20		68		86		1299
% O.C.			1.334		0.348		0.232		1.508		0.174

% O.C. = % Organic Carbon = % Organic Matter / 1.724

K_{oc} = (K_d x 100) / % O.C.

Note: All natural log values were determined by computer spreadsheet and not the rounded numbers presented in TABLES VIII and IX (see APPENDIX D).

TABLE XIV: LINEAR REGRESSION ANALYSIS OF THE <u>DESORPTION DATA</u> USING THE FREUNDLICH ISOTHERM ($\ln x/m = \ln K_d + 1/n \ln C_e$) FOR ^{14}C-CGA-329351

ADSORPTION TEST SOL. CONC.		SOIL TYPE									
		MISSISSIPPI CLAY		MARYLAND SAND		CALIFORNIA SANDY LOAM		WASHINGTON LOAM		ARIZONA SILTY CLAY LOAM	
(µg/mL)	REP	$\ln C_e$	ln x/m	$\ln C_e$	ln x/m	$\ln C_e$	ln x/m	$\ln C_e$	ln x/m	$\ln C_e$	ln x/m
0.206	1	-3.530	-1.146	-5.418	-4.343	-5.019	-4.254	-3.752	-2.419	-3.645	-1.915
	2	-3.559	-1.151	-5.454	-4.337	-5.050	-4.324	-3.683	-2.479	-3.695	-1.867
0.513	1	-2.604	-0.273	-4.630	-3.683	-4.257	-3.421	-2.790	-1.594	-2.736	-1.070
	2	-2.628	-0.325	-4.963	-3.721	-4.297	-3.535	-2.774	-1.608	-2.755	-1.041
1.028	1	-1.908	0.363	-3.950	-3.105	-3.659	-2.769	-2.154	-0.966	-1.990	-0.364
	2	-1.880	0.551	-3.924	-3.130	-3.873	-2.733	-2.138	-0.959	-2.000	-0.375
5.145	1	0.129	2.701	-3.046	-1.571	-2.156	-1.297	-0.445	0.644	-0.209	1.505
	2	0.122	2.838	-2.574	-1.788	-2.252	-1.295	-0.455	0.649	-0.203	1.525
10.217	1	0.712	4.073	-2.106	-0.947	-1.117	-0.763	0.354	1.419	0.473	2.423
	2	0.738	4.158	-2.112	-1.111	-1.324	-0.701	0.362	1.443	0.461	2.436
LINEAR REGRESSION ANALYSIS											
Correlation (r)			0.9926		0.9862		0.9907		0.9997		0.9980
Slope (1/n)			1.202		0.991		0.942		0.954		1.038
Intercept			2.910		1.012		0.600		1.081		1.818
n			0.832		1.009		1.061		1.048		0.963
K_d			18.4		2.8		1.8		2.9		6.2
K_{oc}			1376		790		785		195		3539
% O.C.			1.334		0.348		0.232		1.508		0.174

% O.C. = % Organic Carbon = % Organic Matter / 1.724

K_{oc} = (K_d x 100) / % O.C.

Note: All natural log values were determined by computer spreadsheet and not the rounded numbers presented in TABLES X and XI (see APPENDIX D).

TABLE XV: EXTRACTION OF 10.217 PPM SAMPLE RESIDUAL SOILS REMAINING FOLLOWING ADSORPTION AND DESORPTION OF ^{14}C-CGA-329351

		PERCENT OF RADIOACTIVITY PRESENT[a]		
SAMPLE	REP	EXTRACT	RESIDUAL (NOT EXTRACTED)	TOTAL
MS Clay	1	90.6	10.8	101.4
	2	90.0	8.4	98.4
MD Sand	1	85.7	5.1	90.8
	2	89.0	5.2	94.2
CA Sandy Loam	1	86.7	6.3	93.0
	2	95.5	5.8	101.3
WA Loam	1	99.9	8.1	108.0
	2	101.5	8.2	109.7
AZ Silty Clay Loam	1	96.2	6.9	103.1
	2	96.9	6.7	103.6
			AVERAGE ± 1 SD =	100.4±6.3

[a] Percent of the total radioactivity present on soils following the definitive desorption phase of the adsorption and desorption of ^{14}C-CGA-329351.

TABLE XVI: RAMONA RADIOACTIVITY MONITOR QUANTITATION OF HPLC OF SELECTED SAMPLES FROM THE ADSORPTION AND DESORPTION STUDY OF ^{14}C-CGA-329351

SAMPLE	REP	PERCENT RADIOCARBON DETECTED AS CGA-329351			
		DOSE SOLUTION	ADSORPTION SOLUTION	DESORPTION SOLUTION	SOIL EXTRACTION
Purity Check		99.1			
48 Hour Aqueous Solubility	1	99.2			
	2	99.2			
Preliminary Phase (10.641 ppm)					
Blank	1		99.0		
MS Clay	1		97.6		
MD Sand	1		98.9		
CA Sandy Loam	1		98.7		
WA Loam	1		98.6		
AZ Silty Clay Loam	1		98.8		
Definitive Phase					
10.217 ppm Solution		97.5			
5.145 ppm Solution		97.4			
1.028 ppm Solution		97.2			
0.513 ppm Solution		98.3			
0.206 ppm Solution		97.9			
0.000 ppm Solution		0.0			
Test Soils[a]\Definitive Phase					
MS Clay	1				98.9
	2		91.8	95.3	99.8
MD Sand	1				93.2
	2		97.1	96.1	98.4
CA Sandy Loam	1				97.7
	2		98.9	98.8	96.0
WA Loam	1				97.4
	2		97.3	98.0	94.9
AZ Silty Clay Loam	1				97.3
	2		99.0	95.3	97.9

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[a] All samples from the highest test concentration (10.217 ppm initial dose).

TABLE XVII: AMBIS RADIOANALYTICAL SCANNER QUANTITATION OF TWO DIMENSIONAL TLC OF SELECTED SAMPLES FROM THE ADSORPTION AND DESORPTION STUDY OF ^{14}C-CGA-329351

SAMPLE	REP	PERCENT RADIOCARBON DETECTED AS CGA-329351 DOSE SOLUTION	ADSORPTION SOLUTION	DESORPTION SOLUTION	SOIL EXTRACTION
Purity Check		99.5			
48 Hour Aqueous Solubility	1	99.8			
	2	99.7			
Preliminary Phase (10.641 ppm)					
Blank	1		99.3		
MS Clay	1		98.7		
MD Sand	1		99.5		
CA Sandy Loam	1		99.4		
WA Loam	1		99.1		
AZ Silty Clay Loam	1		99.4		
Definitive Phase					
10.217 ppm Solution		99.2			
5.145 ppm Solution		99.3			
1.028 ppm Solution		99.8			
0.513 ppm Solution		99.2			
0.206 ppm Solution		98.7			
Test Soils[a] **Definitive Phase**					
MS Clay	1				99.8
	2		99.2	98.4	99.7
MD Sand	1				99.1
	2		99.6	98.2	99.3
CA Sandy Loam	1				99.9
	2		99.4	97.7	99.1
WA Loam	1				98.7
	2		99.2	98.5	98.9
AZ Silty Clay Loam	1				99.8
	2		99.4	98.7	99.8

[a] All samples from the highest test concentration (10.217 ppm initial dose).

TABLE XVIII: LIQUID SCINTILLATION COUNTING OF SCRAPINGS FROM TLC AND FRACTION COLLECTIONS FROM HPLC FOR VERIFICATION OF AMBIS AND RAMONA DETECTOR QUANTITATIONS

SAMPLE	APPLIED/ INJECTED DPM	RECOVERED DPM	CGA-329351 DPM	PERCENT TOTAL RECOVERY	PERCENT CGA-329351
2D-Thin Layer Chromatography					
Definitive Dose Solution 5.145 ppm	4632	4556	4364	98.4	95.8
Definitive Adsorption Solution AZ Silty Clay Loam Rep 2 10.217 ppm	11117	11777	10982	105.9	93.2
Post Desorption Soil Extraction MS Clay Soil Extract 10.217 ppm Rep 1	30929	33796	28249	109.3	83.6
			AVERAGE	104.5	90.9
High Performance Liquid Chromatography					
Definitive Dose Solution 1.028 ppm	46278	44754	43388	96.7	96.9
Definitive Desorption Solution MS Clay Rep 2 10.217 ppm	42401	37990	37501	90.1	98.7
Post Desorption Soil Extraction CA Sandy Loam Rep 1 10.217 ppm	34269	31862	30654	93.7	96.2
			AVERAGE	93.5	97.3

Percent Total Recovery = DPM recovered ÷ DPM applied/injected x 100
Percent CGA-329351 = DPM CGA-329351 ÷ DPM recovered x 100

TABLE XIX: DIRECT COMPARISON OF RESULTS FROM RAMONA-5-LS VS FRACTION COLLECTION FROM THE HPLC AND LSC OF TLC PLATE SCRAPINGS VS AMBIS RADIOANALYTICAL SCANNING

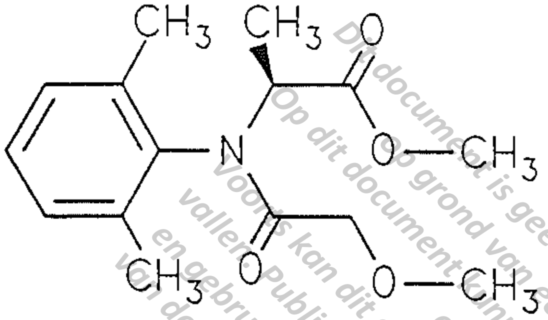
HPLC DATA

SAMPLE	OBSERVED PERCENT CGA-329351 RAMONA-5-LS	 FRACTION COLLECTION	HPLC COLUMN RECOVERY
Definitive Dose 1.028 ppm	97.2	96.9	96.7
Definitive Desorption Solution MS Clay 10.217 ppm Rep 2	95.3	98.7	90.1
CA Sandy Loam Soil Extraction 10.217 ppm Rep 1	97.7	96.2	93.7

TLC DATA

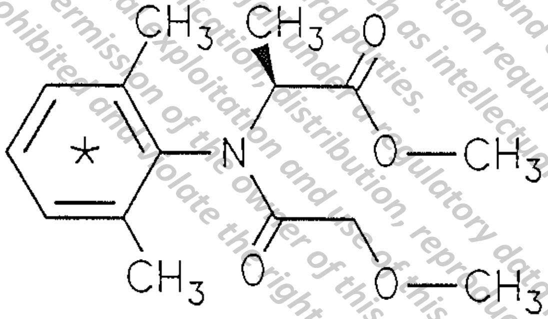
SAMPLE	OBSERVED PERCENT CGA-329351 AMBIS SCANNER	 SCRAPING LSC	TLC PLATE RECOVERY
Definitive Dose Solution 5.145 ppm	99.3	95.8	98.4
AZ Silty Clay Loam Adsorption Solution 10.217 ppm Rep 2	99.4	93.2	105.9
MS Clay Soil Extract 10.217 ppm Rep 1	99.8	83.6	109.3

FIGURE 1: STRUCTURE AND NOMENCLATURE OF CGA-329351



CGA-329351

CAS No.:	70630-17-0
Chemical Name:	(R)-2-[N-2,6-dimethylphenyl methoxyacetyl-amino]-propionic acid methyl ester
Molecular Weight:	279.34
Agrisearch Log No.:	95-038
Ciba Reference No.:	S95-1796
Quantity Received:	100 mg
Chemical Purity:	97.3% (Determined by Sponsor)
Reanalysis Date:	5/96
Water Solubility:	Approx. 250 ppm at 25°C pH 7.1 (Determined by Agrisearch)
Storage Condition:	Room Temperature



ϕ-^{14}C-CGA-329351

*:	Location of Radiolabel
Agrisearch Log No.:	95-041
Ciba Reference No.:	MRS-II-90
Quantity Received:	10.0 mg
Specific Activity:	81.1 µCi/mg
Radiochemical Purity:	98.8% (Determined by Sponsor)
Chemical Purity:	99.9% (Determined by Sponsor)
Expiration Date:	11/95
Storage Condition:	Freezer

FIGURE 2: FREUNDLICH ADSORPTION AND DESORPTION ISOTHERMS FOR THE FIVE TESTED AGRICULTURAL SOILS

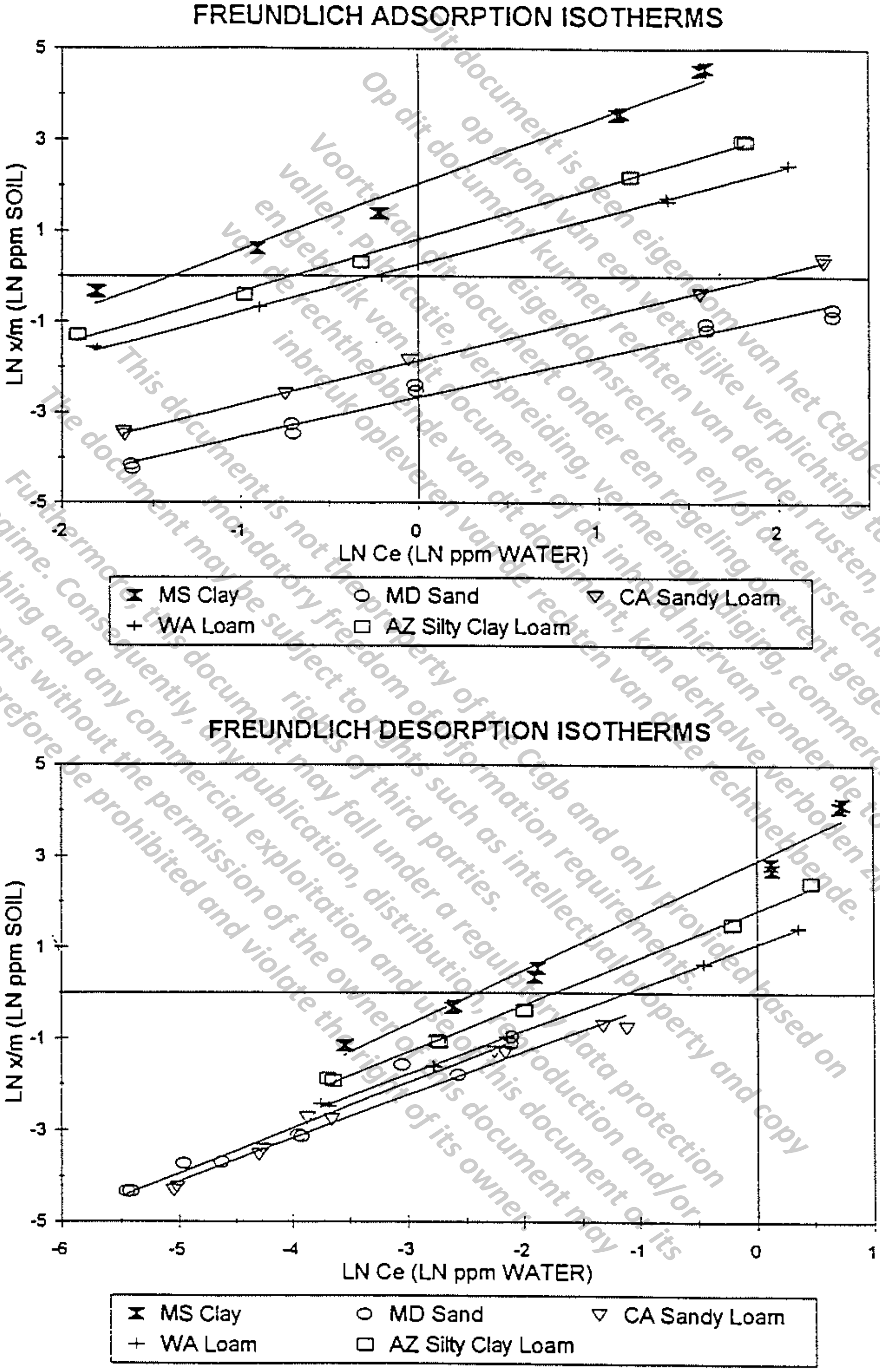
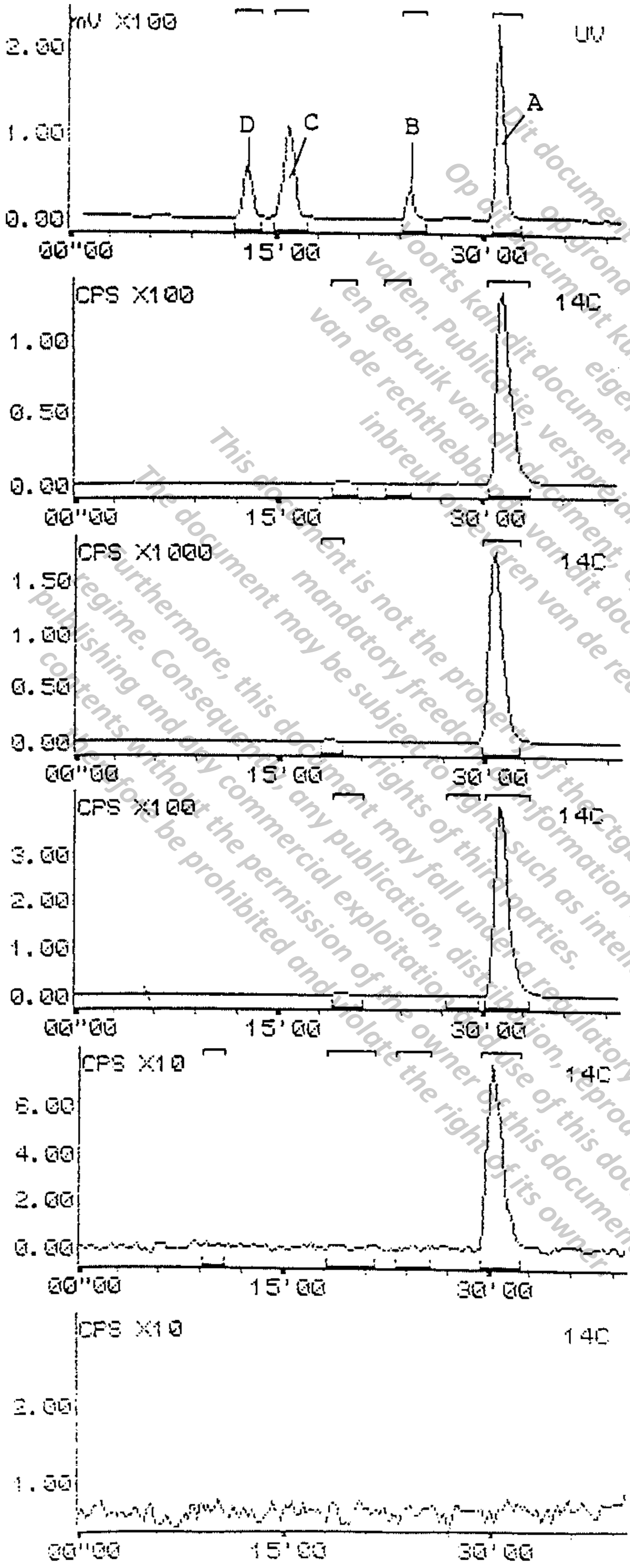


FIGURE 3: HPLC ANALYSIS OF CGA-329351 AND DOSE SOLUTIONS



UV 230 nm Visualization
A = CGA-329351 (95-038)
B = CGA-62826
C = CGA-42447/CGA-119857
D = CGA-37734

^{14}C-RAMONA DETECTION
Pre-Study Purity Check
99.1% CGA-329351
Date 7/11/95

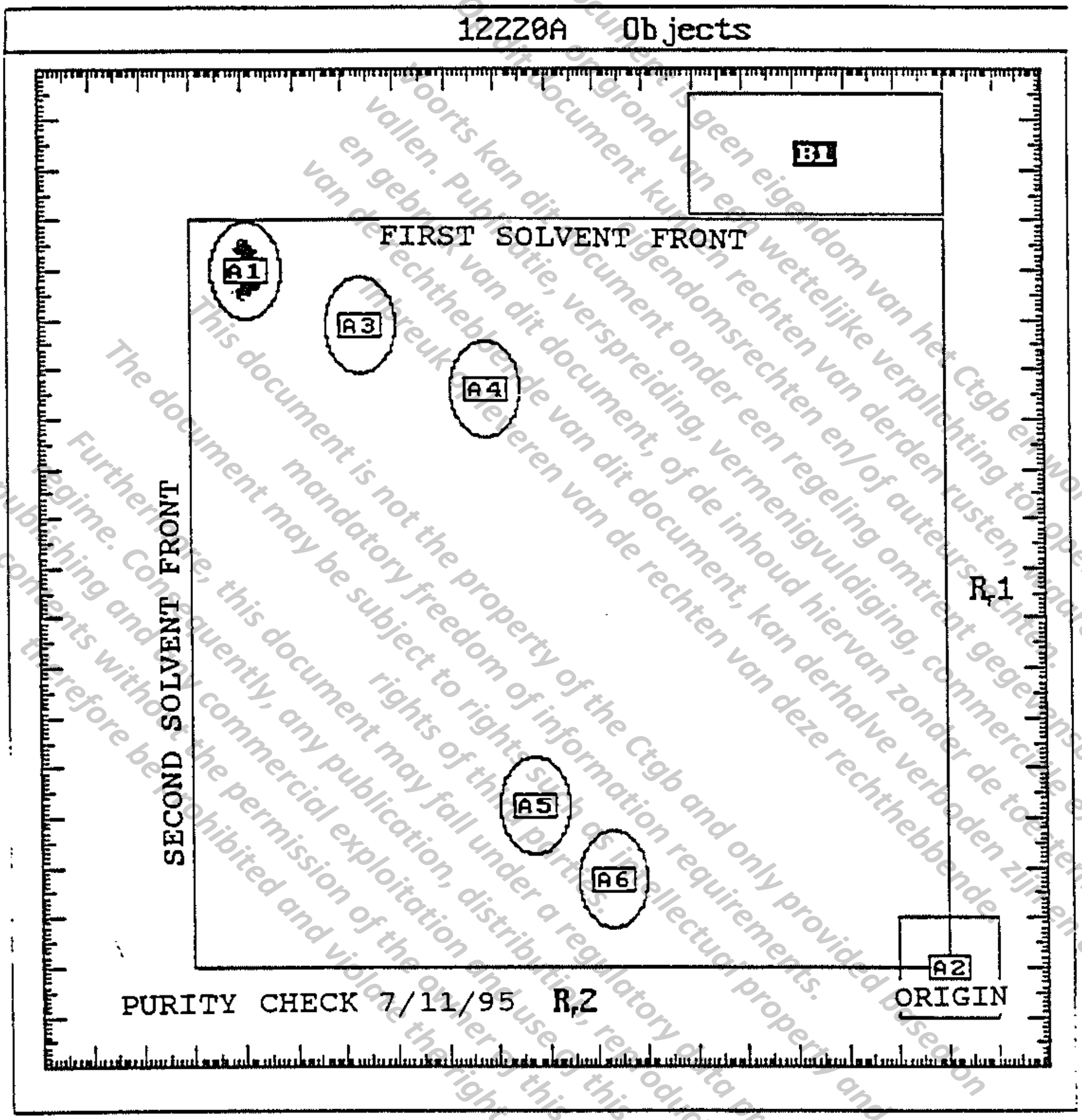
48 Hour Solubility
Rep 1 99.2% (Shown)
Rep 2 99.2%

Preliminary Phase (10.641 ppm)
24 Hour Soil-Less Blank 99.0%

Definitive Testing Dose Solutions
10.217 ppm 97.5% CGA-329351
5.145 ppm 97.4%
1.028 ppm 97.2% (Shown)
0.513 ppm 98.3%
0.206 ppm 97.9%

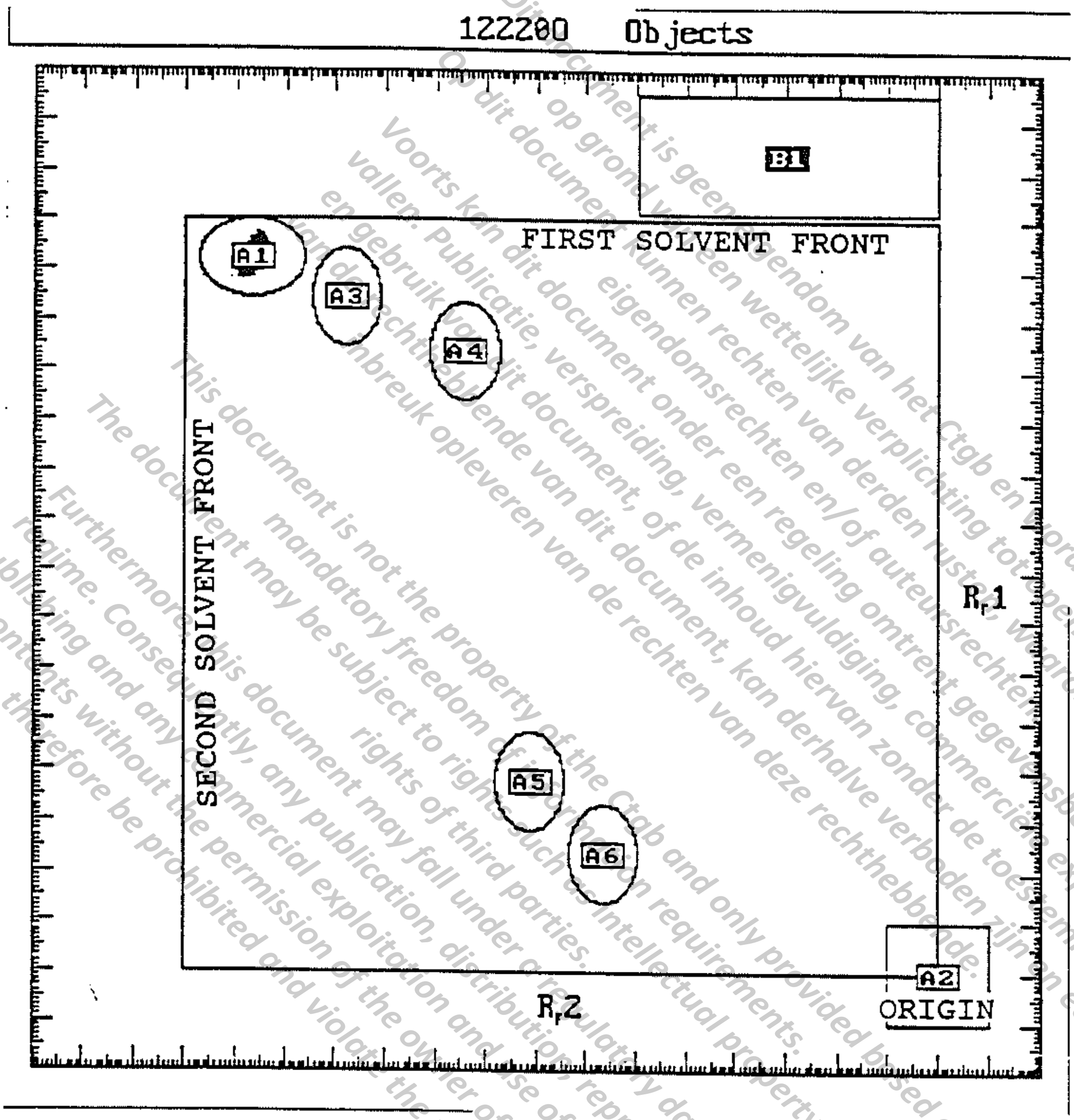
Definitive Dose Solution
0.0 ppm = 0.0% CGA-329351

FIGURE 4: TWO DIMENSIONAL TLC ANALYSIS OF CGA-329351



<u>Area A1 = CGA-329351</u>
Purity Check = 99.5% CGA-329351 (Shown)
48 Hour Aqueous Solubility = 99.8 and 99.7%
A2-A6 = Area of ≤0.4% Radiocarbon
B1 = Background

FIGURE 5: TWO DIMENSIONAL TLC ANALYSIS OF THE HIGHEST CONCENTRATION FROM ADSORPTION SOLUTIONS FOR EACH SOIL TYPE



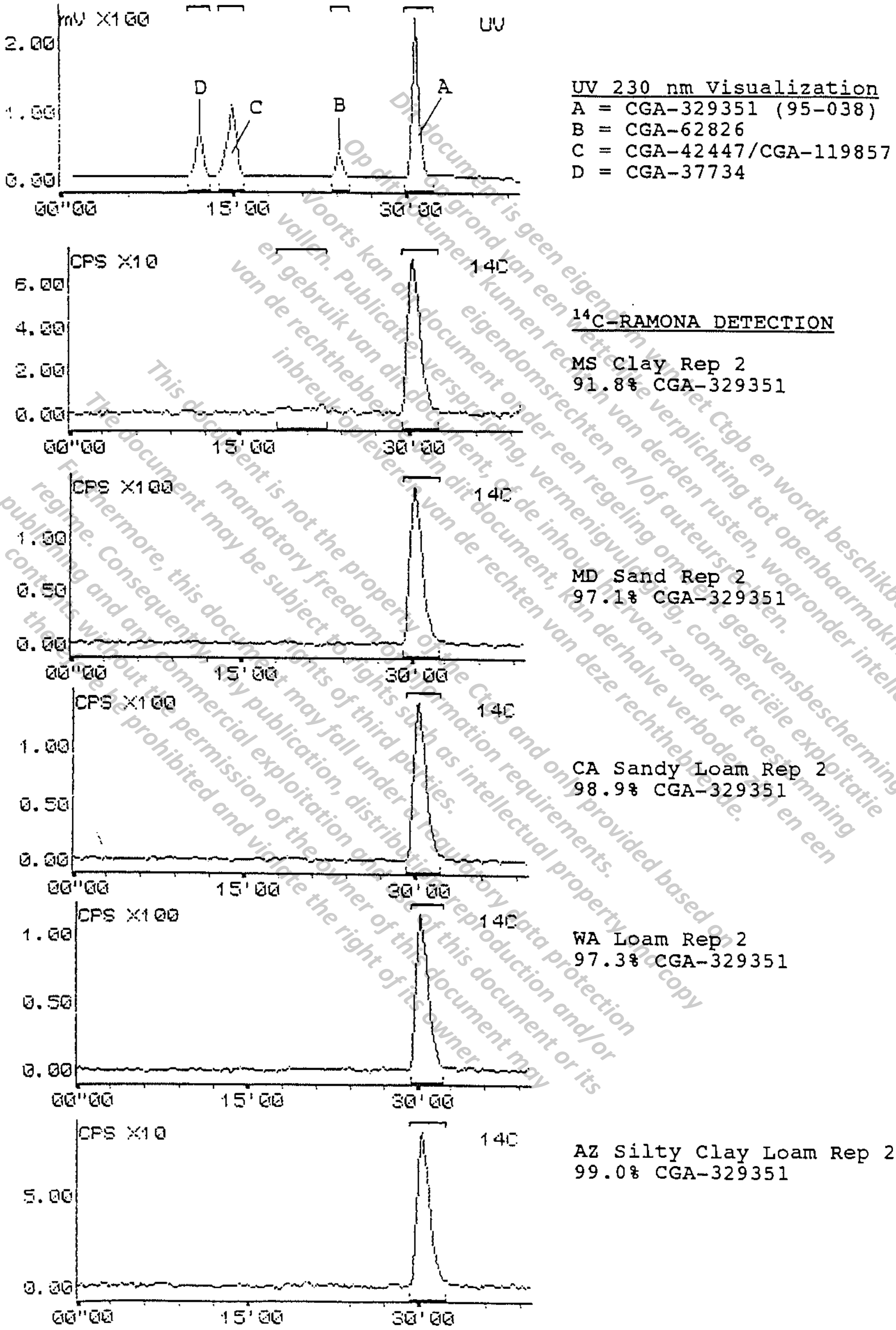
Area A1 = CGA-329351

10.217 ppm Adsorption Solutions

MS Clay(Shown)	Rep 2 = 99.2%	CGA-329351
MD Sand	Rep 2 = 99.6%	
CA Sandy Loam	Rep 2 = 99.4%	
WA Loam	Rep 2 = 99.2%	
AZ Silty Clay Loam	Rep 2 = 99.4%	

A2-A6 = Area of ≤0.5% Radiocarbon
B = Background

FIGURE 6: HPLC ANALYSIS OF THE HIGHEST CONCENTRATION FROM ADSORPTION SOLUTIONS FOR EACH SOIL TYPE



UV 230 nm Visualization
A = CGA-329351 (95-038)
B = CGA-62826
C = CGA-42447/CGA-119857
D = CGA-37734

^{14}C-RAMONA DETECTION

MS Clay Rep 2
91.8% CGA-329351

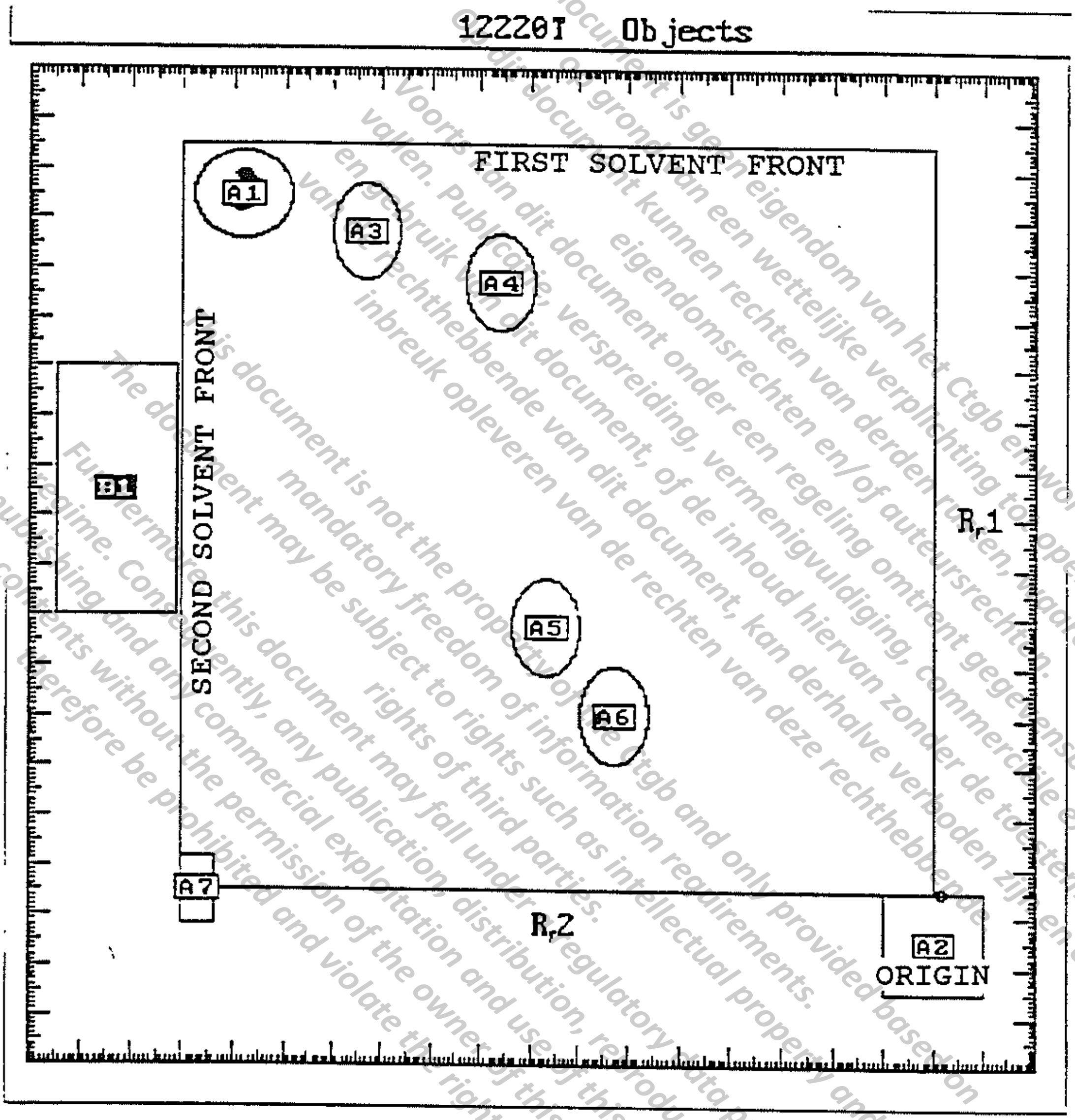
MD Sand Rep 2
97.1% CGA-329351

CA Sandy Loam Rep 2
98.9% CGA-329351

WA Loam Rep 2
97.3% CGA-329351

AZ Silty Clay Loam Rep 2
99.0% CGA-329351

FIGURE 7: TWO DIMENSIONAL TLC ANALYSIS OF THE HIGHEST CONCENTRATION FROM DESORPTION SOLUTIONS FOR EACH SOIL TYPE



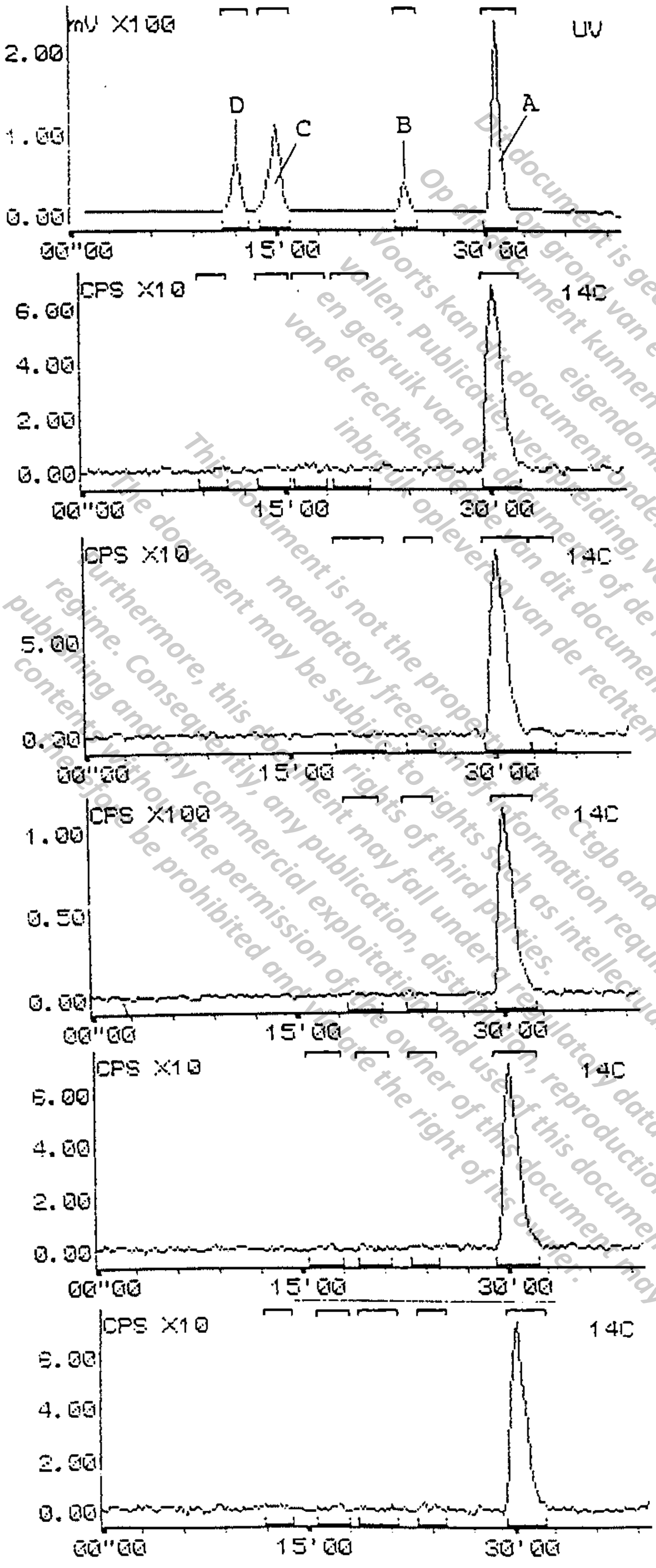
<u>Area A1 = CGA-329351</u>

10.217 ppm Desorption Solutions

MS Clay (Shown)	Rep 2 = 98.4%	CGA-329351
MD Sand	Rep 2 = 98.2%	
CA Sandy Loam	Rep 2 = 97.7%	
WA Loam	Rep 2 = 98.5%	
AZ Silty Clay Loam	Rep 2 = 98.7%	

A2-A7 = Area of ≤1.3% Radiocarbon
B = Background

FIGURE 8: HPLC ANALYSIS OF THE HIGHEST CONCENTRATION FROM DESORPTION SOLUTIONS FOR EACH SOIL TYPE



UV 230 nm Visualization
A = CGA-329351 (95-038)
B = CGA-62826
C = CGA-42447/CGA-119857
D = CGA-37734

^{14}C-RAMONA DETECTION
MS Clay Rep 2
95.3% CGA-329351

MD Sand Rep 2
96.1% CGA-329351

CA Sandy Loam Rep 2
98.8% CGA-329351

WA Loam Rep 2
98.0% CGA-329351

AZ Silty Clay Loam Rep 2
95.3% CGA-329351

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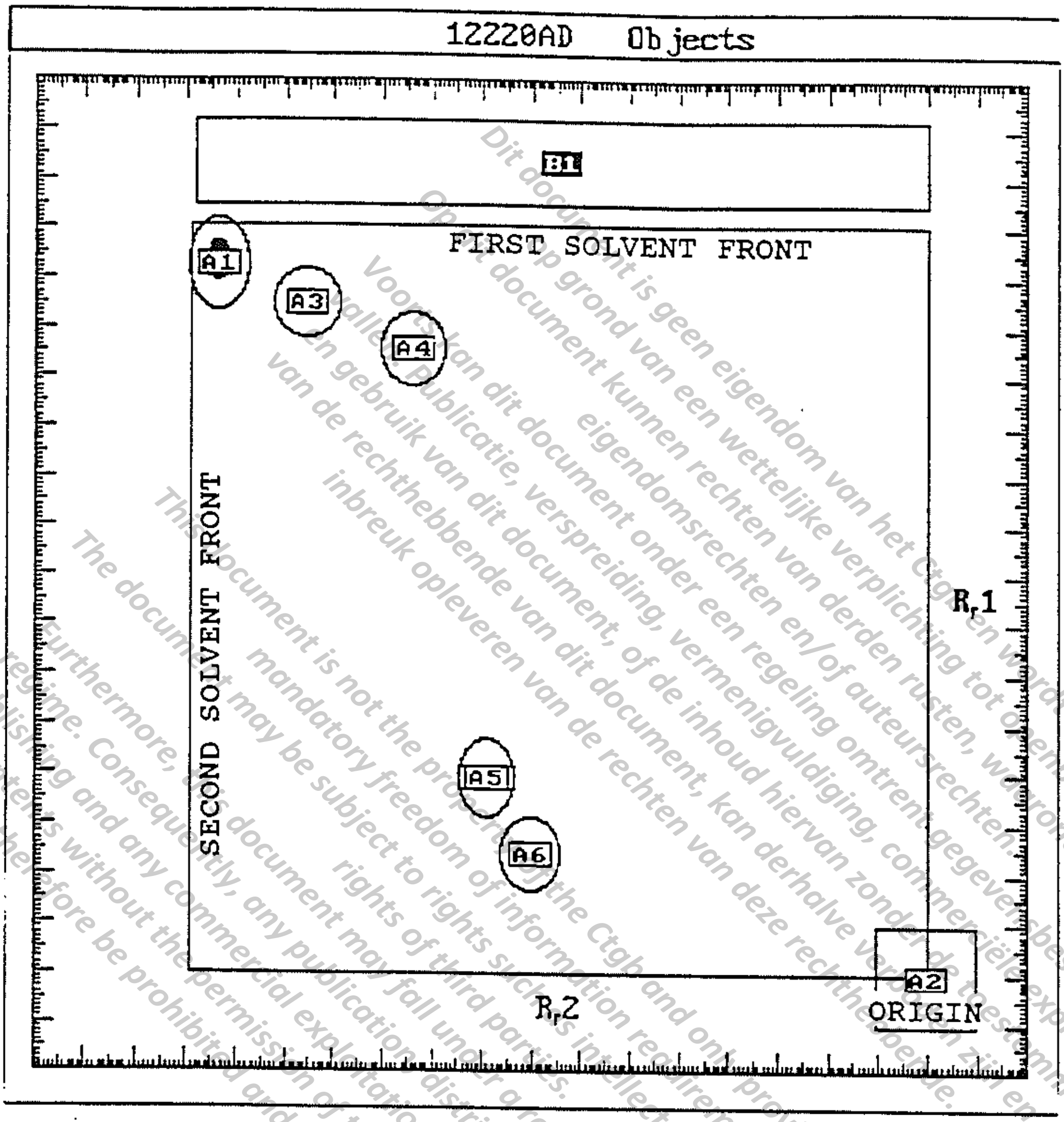
FIGURE 9: TWO DIMENSIONAL TLC ANALYSIS OF THE SOIL EXTRACTS FROM THE HIGHEST CONCENTRATION OF EACH SOIL TYPE FOLLOWING DESORPTION



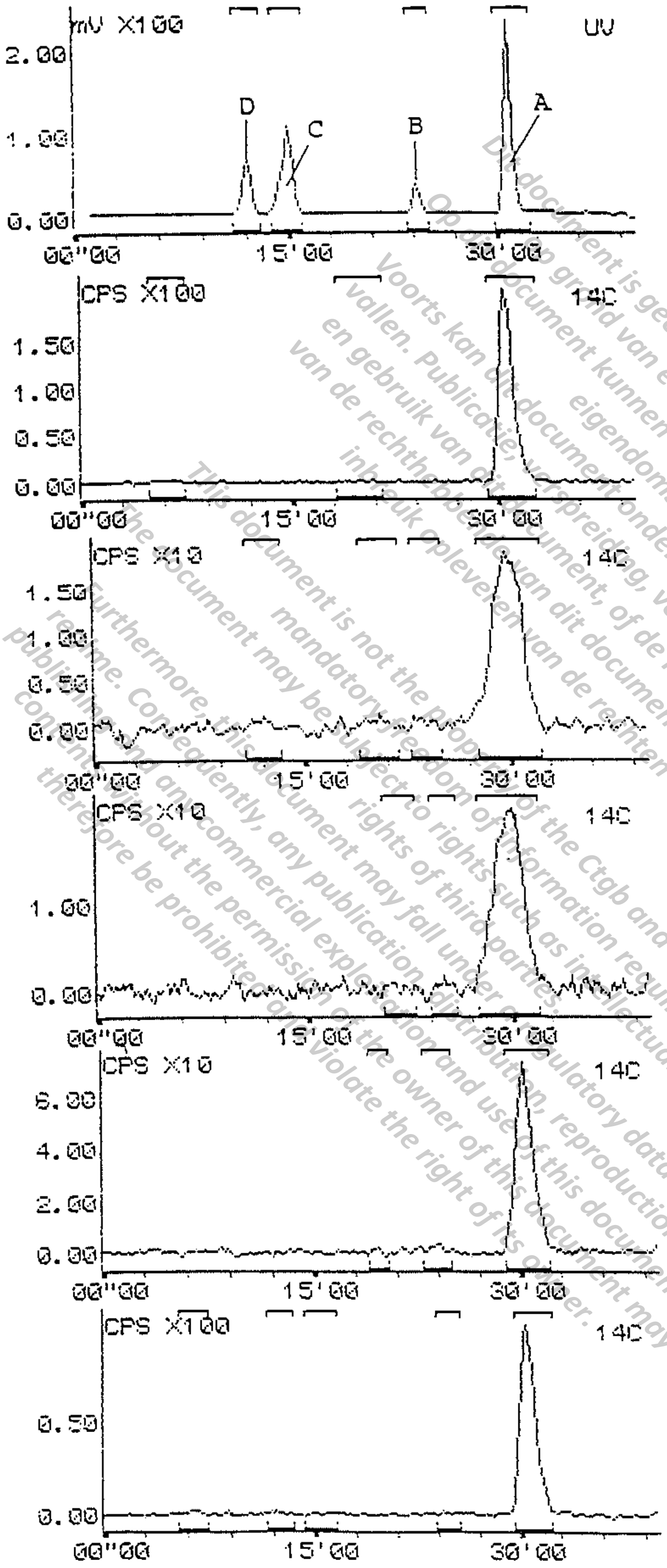
Area A1 = CGA-329351

10.217 ppm Post Desorption Soil Extracts

	Percent CGA-329351	
	Rep 1	Rep 2
MS Clay	99.8	99.7
MD Sand	99.1	99.3
CA Sandy Loam	99.9	99.1 (Shown)
WA Loam	98.7	98.9
AZ Silty Clay Loam	99.8	99.8

A2-A6 = Area of ≤0.9% Radiocarbon
B1 = Background

FIGURE 10: HPLC ANALYSIS OF THE SOIL EXTRACTS FROM THE HIGHEST CONCENTRATION OF EACH SOIL TYPE FOLLOWING DESORPTION



<u>UV 230 nm Visualization</u>

A = CGA-329351 (95-038)
B = CGA-62826
C = CGA-42447/CGA-119857
D = CGA-37734

<u>^{14}C-RAMONA DETECTION</u>

MS Clay
* Rep 1 98.9% CGA-329351
Rep 2 99.8%

MD Sand
* Rep 1 93.2% CGA-329351
Rep 2 98.4%

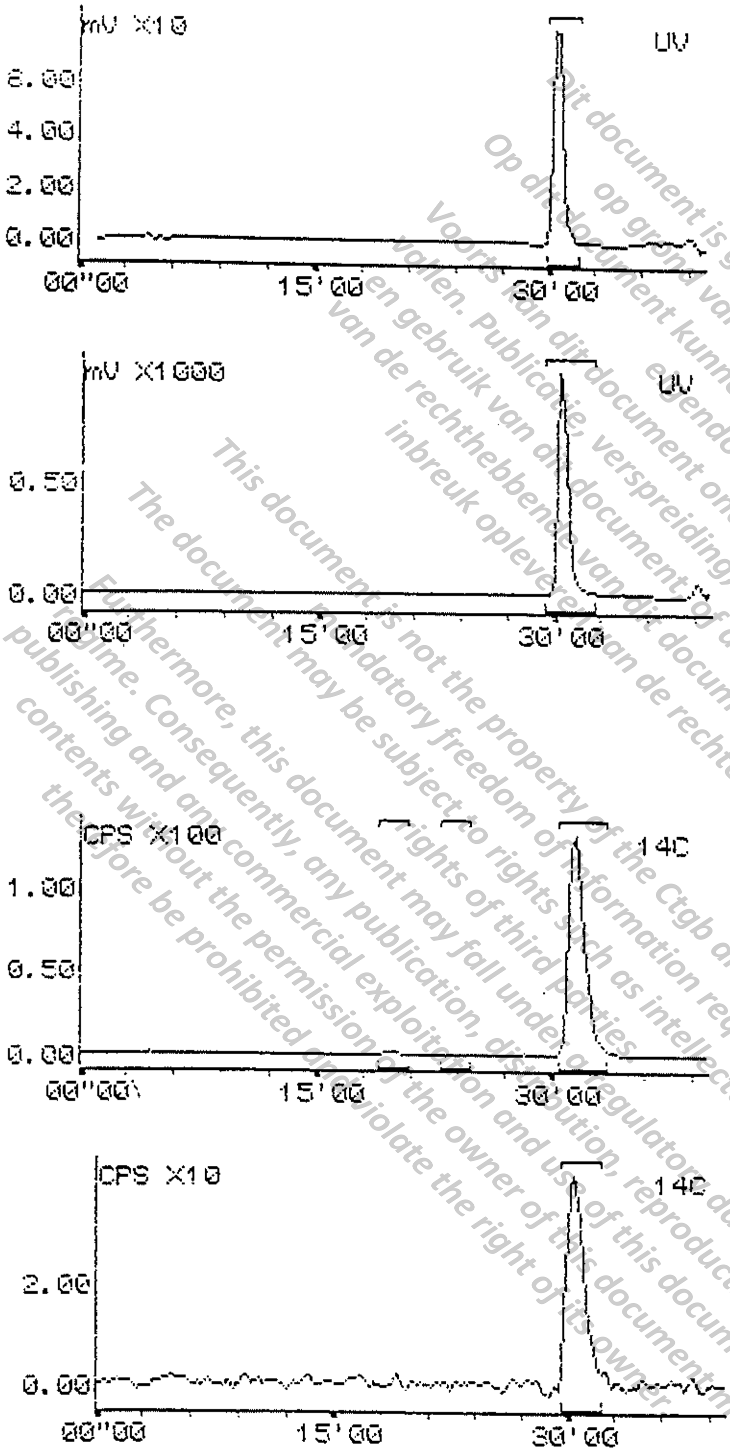
CA Sandy Loam
Rep 1 97.7% CGA-329351
* Rep 2 96.0%

WA Loam
* Rep 1 97.4% CGA-329351
Rep 2 94.9%

AZ Silty Clay Loam
* Rep 1 97.3% CGA-329351
Rep 2 97.9%

* Shown

FIGURE 11: STABILITY OF THE TEST AND REFERENCE SUBSTANCES AT AGRISEARCH INCORPORATED



<u>UV 230 nm Visualization</u>
CGA-329351 (95-038)
At Definitive Testing
(7/11/95)

At Study Completion
UV 230 nm
(8/25/95)

<u>RAMONA DETECTION</u>
^{14}C-CGA-329351 (95-041)
Prior to Study Start
99.1%
(7/11/95)

At Study Completion
99.2%
(8/25/95)

REFERENCES

(1) [redacted] and [redacted] and [redacted], "Measurement of Sorption Coefficients of Organic Chemicals And Their Use in Environmental Fate And Movement of Toxicants," AOAC Symposium Proceedings, 94th Annual Meeting, Washington, D.C., October 21-22, 1980.

(2) "Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate," U.S. Environmental Protection Agency, Office of Pesticide and Toxic Substances, Washington, DC, 20460; EPA 540/9-82-021, October 18, 1982. NTIS PB83-153973.

(3) U.S. Environmental Protection Agency, August 17, 1989. Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) Good Laboratory Practice Standards; Final Rule (40 CFR: 160). Federal Register, Vol. 54, No. 158: 34052-34074.

(4) U.S. Environmental Protection Agency, Data Reporting Guidelines, Environmental Fate Series 163-1, Leaching and Adsorption/Desorption Studies, Addendum 6, PB-88-161195, January 1988.

(5) FIFRA Accelerated Reregistration, Phase 3 Technical Guidance, EPA 540/09-90-078, December 1989.

(6) U.S. Environmental Protection Agency, Pesticide Reregistration Rejection Rate Analysis, Environmental Fate, EPA 738-R-93-010, September 1993.

(7) <u>Standard Methods for the Examination of Water and Wastewater</u>, 16th Edition, APHA, AWWA, WPCF, pp 866-869 (1985).

(8) Wang, C.H. and Willis, D.L. <u>Radiotracer Methodology in Biological Science</u>, Prentice-Hall, Inc., Englewood Cliffs, NJ, pp 186-190 (1965).

(9) "Methods of Soil Analysis, Part 2-Chemical and Microbiological Properties, Second Edition," Edited by A.L. Page, R.H. Miller, and D.R. Keeney, American Society of Agronomy, Inc., 1982.

(10) Brady, N.C. <u>The Nature and Properties of Soils 9th Edition</u>, Macmillan Publishing, pages 436-454, 1984.

APPENDIX A

Protocol and Deviations

PROTOCOL APPROVAL

STUDY TITLE: Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

TEST SUBSTANCE: ^{14}C-CGA-329351

AGRISEARCH PROJECT NO: 12220

CIBA STUDY NO: 60-95

EPA DATA REQUIREMENT: 40 CFR 158 Subdivision N: 163-1

PROPOSED EXPERIMENTAL START DATE: June 20, 1995

PROPOSED EXPERIMENTAL TERMINATION DATE: August 30, 1995

PROTOCOL ACCEPTANCE:

Sponsor:

[redacted] Sponsor Representative — 6/12/95 Date

Ciba Crop Protection
Ciba-Geigy Corporation
410 Swing Road
Greensboro, NC 27409
(910) 632-[redacted]
(910) 632-[redacted] FAX

Testing Facility:

[redacted] Study Director — 6/19/95 Date

Agrisearch Incorporated
5734 Industry Lane
Frederick, MD 21701
(301) [redacted]
(301) [redacted] FAX

OBJECTIVE

The objective of this study is to predict the potential for movement of CGA-329351 from the site of application to an aquatic site, and dispersion therein. The objective is achieved by determining the values of K_{oc} (sorption coefficient), K_d (adsorption constant) and n using the Freundlich isotherm (Reference 1). Five soil types will be tested in this study using batch equilibrium techniques (adsorption/desorption).

This study will be performed to meet the US EPA requirements as listed in the US EPA Pesticide Assessment Guidelines, Subdivision N, Series 163-1 (Reference 2). This study will also be conducted in accordance with this protocol; US EPA FIFRA Good Laboratory Practice Standards, 40 CFR 160 (Reference 3); the Data Reporting Guidelines (Reference 4); FIFRA Accelerated Reregistration, Phase 3 Technical Guidance (Reference 5); Pesticide Reregistration Rejection Rate Analysis (Reference 6); and applicable Agrisearch Incorporated standard operating procedures.

JUSTIFICATION FOR SELECTION OF TEST SYSTEM

Pesticide residues may move through the soil profile by leaching or be transported to aquatic environments by movement in surface water. This study is designed to predict the potential for leaching and movement of pesticides in terrestrial and aquatic systems by the use of the batch equilibrium technique with five soil types. The soils were chosen as recommended in the EPA Pesticide Assessment Guidelines, Subdivision N; 163-1 (Reference 2).

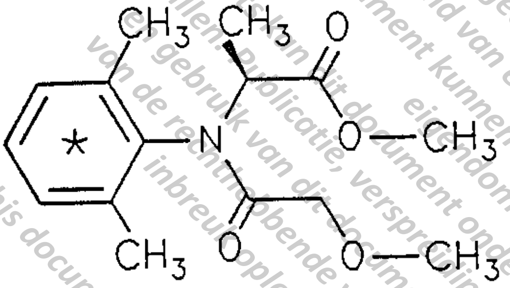
SUMMARY

^{14}C-Radiolabeled CGA-329351 in an aqueous 0.01M calcium chloride ($CaCl_2$, 0.01N Ca^{++}) solution is mixed with representative agricultural soils and allowed to equilibrate. The soil/test solution phases are separated by centrifugation and the concentration of ^{14}C-CGA-329351 remaining in the aqueous solution is determined by liquid scintillation counting (LSC). The adsorbed concentration is determined by difference. The soils are then desorbed with untreated aqueous 0.01M $CaCl_2$ solution and desorption concentrations are determined by LSC following centrifugation. Soil concentration following desorption is determined by combustion and subsequent LSC analysis. Adsorption and desorption isotherms are graphically presented. Soil characteristics are reported. Data are evaluated using the Freundlich equation, and n, K_d, and K_{oc} values are reported.

EXPERIMENTAL DESIGN

Test Substance

ϕ-^{14}C-CGA-329351



***Denotes site of radioactivity - uniformly labeled**

Chemical Name: (R)-2-[N-2,6-dimethylphenyl methoxyacetyl-amino]-propionic acid methyl ester
Supplier: Ciba Crop Protection, Ciba-Geigy Corporation
Reference No.: MSR-II-90

Radiochemical Purity: 98.8% determined by the Sponsor by TLC on 5/26/95. The radiochemical purity will be verified at/by Agrisearch Incorporated prior to use. The test substance will be analyzed via TLC (TABLE I) and/or HPLC (TABLE II) by Agrisearch Incorporated. The radiopurity value of the test substance must be greater than 95%.

Specific Activity: 81.1 µCi/mg (180,042 dpm/µg)
Chemical Purity: 99.9% determined (GC) by the Sponsor on 3/9/95
Expiration Date: 11/95
Water Solubility: Approximately 26 g/L at 22°C
CAS Number: 70630-17-0
Storage Conditions: Freezer
Safety: Material Safety Data Sheets (MSDS) are on file at Agrisearch Incorporated and Ciba Crop Protection.
Molecular Formula: $C_{15}H_{21}NO_4$
Molecular Weight: 279.34

Reference Substances

CGA-329351
Reference No.: S95-1796
Chemical Purity: 97.3% by GC (external standard)
Reassay Date: 5/96
Storage Conditions: Room Temperature

CGA-37734
Chemical Name: N-(2,6-dimethylphenyl)-2-hydroxyacetamide
CAS No.: 29183-14-0
Reference No.: BPM-I-8
Chemical Purity: 96.9% by GC (area distribution) on 2/2/94
Expiration Date: 2/96
Storage Conditions: Refrigeration

CGA-42447
Chemical Name: N-(2,6-dimethylphenyl)acetamide
CAS No.: 2198-53-0
Reference No.: MCO-I-2
Chemical Purity: 99.7% by GC (area distribution) on 12/12/94
Expiration Date: 12/96
Storage Conditions: Refrigeration

CGA-62826
Chemical Name: N-(2,6-dimethylphenyl)-N-(methoxyacetyl)-DL-alanine
CAS No.: 87764-37-2
Reference No.: BPM-I-4B
Chemical Purity: >99.9% by HPLC (area distribution) on 12/8/94
Expiration Date: 12/96
Storage Conditions: Refrigeration

CGA-119857
Chemical Name: Not Available
CAS No.: Not Assigned
Reference No.: GB-XLV-3
Chemical Purity: >99.9% by HPLC (area distribution) on 12/8/94
Expiration Date: 12/96
Storage Conditions: Refrigeration

Additional reference substances may be used in this study. Information pertinent to any additional reference substance will be included with this protocol via protocol amendment.

Test and Reference Substance Documentation

The methods of synthesis, fabrication and/or derivatization of the test and reference substances are documented and retained by the Sponsor. Characterization of the test and reference substances, as required by US EPA FIFRA GLP Standard 40 CFR 160.105, was conducted by the Sponsor and these records are retained by the Sponsor. Archived samples of the test and reference substances are being retained by the Sponsor. These records and archived samples are being maintained by the Sponsor at Ciba Crop Protection, Greensboro, NC.

The test and reference substances containers will be retained for at least the duration of the study. When the containers are emptied, and the study is completed, discontinued, or terminated, the containers will be disposed of according to standard Agrisearch Incorporated procedures. Any remaining test and/or reference substances will be retained by/at Agrisearch Incorporated, or authorization will be obtained from the Sponsor to return any remaining substances. Records will be maintained at Agrisearch Incorporated documenting use and disposal, if any, of test and/or reference substances.

Stability of Test and Reference Substances

The stability of the test and reference substances has been determined in accordance with US EPA FIFRA GLP Standards by the Sponsor. The Sponsor has provided in this protocol storage conditions under which the substances will be stored at Agrisearch Incorporated. Stability of the test and reference substances will be determined at/by Agrisearch Incorporated (test site), as required by US EPA FIFRA GLP Standard 40 CFR 160.105(e). Test and reference substances will be analyzed by TLC (TABLE I) and/or HPLC (TABLE II) by Agrisearch Incorporated at the beginning and end of the study, or about, thereby qualitatively determining the stability at the test facility (Agrisearch Incorporated).

Aqueous Solubility

The estimated water solubility of ^{14}C-CGA-329351 will be determined. Approximately 0.5 mg of ^{14}C-CGA-329351 will be placed into duplicate aluminum foil covered silylated glass vials. The solvent will be evaporated and 2 mL of sterile, deionized distilled water will be added for an approximate final concentration of 250 ppm. Each vial will be sonicated for approximately 5 minutes, manually shaken for approximately 0.5 minute, then placed into a laboratory incubator at 25±1°C. At the approximate sampling times of 0, 3, 6, 24, 28, and 48 hours, the vials, except 0 time, will be removed from the incubator, sonicated for approximately 5 minutes, then manually shaken for approximately 0.5 minute, and centrifuged at approximately 2500 rpm for approximately 15 minutes. Triplicate aliquots will be removed for radioanalysis. Prior to placing the vials back into the incubator, the vials will be sonicated for approximately 5 minutes then manually shaken for approximately 0.5 minute. The solubility determination will be performed for a minimum of 48 hours with a pH determination at the final sampling time.

Soils

A total of five representative soil textures will be used for this study; sand, sandy loam, silty clay loam, loam, and clay. At least one of the soils will have an organic matter (OM) content of <1%. The pH of all five soils will be within the range of 4 to 8. Three of the five soils will satisfy Arizona Department of Environmental Quality standards of having an organic carbon (OC) content of 2.6% or less and a pH between 6.5 and 8.5 ± 0.3. The sandy loam is the same soil used for the soil metabolism study and was collected from California. The silty clay loam was collected from Arizona, the sand from Maryland, the clay from Mississippi, and the loam from Washington. All soils were collected from the soil surface (horizon A). The source of the soils (location) will be documented in the raw data and reported in the final report. All five soils will be air dried at ambient laboratory temperatures and sieved through a 2 mm sieve prior to characterization and use in the study to assure uniform particle size. A representative subsample of each soil will be removed for soil characterization by AGVISE Laboratories (P.O. Box 510, Highway 15, Northwood, ND, 58267).

Soil characterization will be performed by AGVISE Laboratories and the report submitted to Agrisearch Incorporated. The soils will be characterized to include, but not limited to:

(a) textural composition (% sand, silt, and clay),
(b) USDA soil textural classification,
(c) percent organic matter (% OM),
(d) percent organic carbon (calculated as % OM/1.724),
(e) bulk density (disturbed),
(f) cation exchange capacity and extractable cations,
(g) field moisture capacity (FMC) at 1/3 bar,
(h) pH,
(i) clay mineralogy (X-ray diffraction).

The following information, if available, will be documented in the raw data:

(a) soil order name,
(b) soil series name (USDA),
(c) method for determining cation exchange capacity.

Identification of the Test System

The test system for this study is the soil, aqueous 0.01M calcium ion solution, and CGA-329351 in test tubes. Each test system is identified, at a minimum, by project number, soil type, replicate, concentration, date, and technician initials.

Bias/Contaminants

No experimental bias is expected in this study. Also, no contaminants are expected during the study that are known to be capable of interfering with the purpose and conduct of the study.

Silylation of Glassware

All glassware used in this study, e.g., volumetric flasks, pipettes, graduated cylinders, HPLC vials, scintillation vials, etc., will be silylated before use according to the pertinent Agrisearch Incorporated standard operating procedure.

Test Substance Preparation

Aqueous $CaCl_2$ Solution Preparation - Calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$, FW 147.02) will be dissolved in a quantity of deionized, distilled water, to yield a 0.01 M $CaCl_2$ solution (0.01N cationic solution based on Ca^{++} ion oxidation number of +2). The prepared 0.01M $CaCl_2$ solution will be filter sterilized using a 0.2 micron filter. The pH of the $CaCl_2$ aqueous solution will be measured after preparation.

Test Substance Stock Solution Preparation - A stock solution of the test substance will be prepared by dissolving the entire amount of test substance received by Agrisearch Incorporated in an organic solvent, e.g., acetonitrile, to obtain a suitable concentration. The solvent will be pesticide grade or better. The concentration of the test substance solution will be determined, at a minimum, in triplicate by liquid scintillation counting (LSC). If the radioactivity in each aliquot is within 3% of the mean, the solution will be considered homogeneous. This test substance stock solution will be used to verify the radiopurity of the test substance and to demonstrate stability of the test substance at the test facility as described previously in the Stability of Test And Reference Substances section of this protocol.

Test Solutions for Equilibrium Measurements - Test solutions of ^{14}C-CGA-329351 will be prepared using the aqueous 0.01M $CaCl_2$ stock solution prepared above. Aqueous 0.01M $CaCl_2$ solutions of ^{14}C-CGA-329351 may be diluted with non-labeled CGA-329351 to lower the specific activity of ^{14}C-CGA-329351. Test solutions will be prepared in silylated, or inert, containers at the target test solution concentrations of 0.2, 0.5, 1.0, 5.0, and 10.0 ppm (μg/mL) and may contain ≤1% (by volume) organic solvent. The aqueous 0.01M $CaCl_2$ solutions of ^{14}C-CGA-329351 will be maintained sterile by use of aseptic techniques and preparation of the solutions in sterile containers. Test solution containers are wrapped in aluminum foil, if transparent or translucent, to exclude light and stored, if need be, under refrigerated conditions when not in use. Test solutions should be prepared immediately prior to use. Other concentrations may be used in the study; however, any concentration used will be lower than the determined water solubility of the test substance. The water solubility of CGA-329351 will be determined prior to preparation of the test solutions. The actual test solution concentration, solubility and homogeneity, of at least three aliquots will be determined prior to use by LSC. If the concentration of radioactivity in each LSC aliquot is within 3% of the respective mean concentration, the radioactivity will be considered to be homogeneously dispersed in the test solution. The pH of each test solution will also be measured prior to use. Aliquots of each of the test solutions will be analyzed by TLC (TABLE I) and/or HPLC (TABLE II).

Soil Microbial Count

Microbial viability checks will be conducted prior to, or concomitantly, with the use of each of the five soil types. Spread plating techniques will be used to determine total bacteria, total fungi, and total actinomycetes, in accordance with pertinent Agrisearch Incorporated standard operating procedures.

Soil Microbial Biomass

A 500 g aliquot of each soil type will be shipped to Ciba Crop Protection for biomass determination. Ciba-Geigy Corporation will be responsible for all quality assurance and data generation and storage for this determination. The results of the biomass determination will be submitted to Agrisearch Incorporated for inclusion in the final report.

Preliminary Phase

A preliminary phase will be performed on each soil type to establish the adsorption equilibrium time and the optimum ratio of soil to test solution for use in the definitive adsorption and desorption phases of the study. The equilibrium time will be such that the absorbance of the test substance is within the linear range of the sorption isotherm (20 to 80%) of the applied test substance. The preliminary phase will be performed in duplicate with measured portions of the highest concentration of test substance to be tested (10 ppm ^{14}C-CGA-329351/aqueous 0.01M $CaCl_2$) being added to aliquots of each soil type in separate test tubes.

Duplicate (approximately 1 g) samples of each air dried soil type will be weighed into 50 mL centrifuge tubes (Oak Ridge Teflon® FEP). All tubes will be capped with Teflon® screw cap lids. Twenty mL of each of the test solutions is transferred to its respective tube and capped. Replicate blank centrifuge tubes containing only the test solution, i.e., no soil, will be included to detect any adsorption of the test substance to the centrifuge tubes. The soil/test solution tubes will be placed in an incubator, and equilibrium determined by shaking in the dark using an Eberbach shaker at 175 to 200 rpm at a constant temperature of 25 ± 1°C. Duplicate tubes of each soil/test solution will be removed from the incubator at the approximate time intervals of 0, 2, 4, 8, and 24 hours. The 0 time sampling will be the analysis of the test solution directly (see Test Solutions for Equilibrium Measurements section). The blank tubes will be removed at the 24 hours time interval, or when equilibrium time is established. At the sampling time, the tubes are removed from the incubator, centrifuged at greater than 1000 rpm for 15 minutes (or until the soil is firmly packed) to pellet the soil, and aliquots of the supernatant removed. Aliquots of at least 100 μL of the supernatant will be removed and analyzed by LSC to determine the concentration of ^{14}C-CGA-329351 in solution. The radioactivity "lost" from the test solution (supernatant) will be a measure of the test substance adsorption to soil. After removal of the aliquots for LSC analysis, the soil pellet will be resuspended in the test solution by vortexing and manual shaking then returned to the incubator to continue shaking in the dark using an Eberbach shaker at 175 to 200 rpm at 25±1°C until the next time interval. If equilibrium has not been obtained by 24 hours, shaking is continued for an additional 24 hour period until equilibrium is reached, but no longer than 72 hours.

The adsorbed concentration is determined at the final equilibrium analysis by combustion of the pelleted soil. At the last time interval, the tubes will be removed from the incubator and centrifuged as described above. The supernatant will be decanted, retained in silylated liquid scintillation vials, and aliquots removed for LSC analysis. The total recovery of radioactivity (material balance) will be determined for each soil/test solution at the last time interval. The sum of the radioactivity recovered in the test solution and the pellet as a proportion of the radioactivity applied will be the material balance.

The stability of ^{14}C-CGA-329351 in aqueous 0.01M $CaCl_2$ solution at the end of the equilibrium period will be evaluated by the analysis of one of the replicate blank centrifuge tubes by TLC (TABLE I) and/or HPLC (TABLE II). The chromatography of the blank centrifuge tube solution at the equilibrium time will be compared with radiochemical purity results.

Based upon the rate of test substance adsorption to soil over time (estimated K_d as determined from $x/m = K_d C_e$, see CALCULATIONS section), an equilibrium adsorption time will be selected for the definitive phase of the study. If the amount of applied radioactivity bound to the soil is not within 20% to 80% (linear portion of the isotherm) an additional experiment will be performed using the above procedure, except that the soil/test solution ratio will be modified. The criteria for selection of the soil/test solution ratio will be based on the preliminary equilibrium constant (K_d) determination as follows:

Estimated K_d	Solution: Soil Ratio
≤ 1	2 mL:1 g
$>1 \leq 10$	5 mL:1 g
$>10 \leq 50$	20 mL:1 g
$>50 \leq 400$	100 mL:1 g
>400	400 mL:1 g

Definitive Phase

Adsorption - Based upon the equilibrium time and test solution to soil ratios as determined in the preliminary phase, the definitive adsorption phase of the study is performed. Duplicate samples of air dried soil (soil weights based on above ratio determination and maximum volume of 50 mL solution) are weighed into centrifuge tubes for each test solution concentration. Appropriate volumes of the test solution at 0.0, 0.2, 0.5, 1, 5 and 10 ppm (or other concentrations based on the solubility) are transferred to the appropriate centrifuge tubes and capped. The 0.0 ppm concentration serves as a control for determination of background radioactivity from undosed soil. Values obtained from these controls are used to demonstrate that the soil has not been previously exposed to adiocarbon. The entire sample set is shaken in the dark for the equilibrium time using an Eberbach shaker at 175 to 200 rpm in a constant temperature incubator at 25±1°C. At the equilibrium time, the tubes are removed from the incubator, centrifuged at greater than 1000 rpm for 15 minutes (or until the soil is firmly packed) to pellet the soil, and aliquots of the supernatant removed. Aliquots of at least 100 μL of the supernatant will be removed and analyzed by LSC to determine the equilibration concentrations (C_e) of CGA-329351 in all solutions. The supernatant is decanted, retained in silylated liquid scintillation vials, and aliquots removed for LSC analysis. The actual concentration of CGA-329351 adsorbed (x/m) is determined by difference subtracting the radioactivity observed in the supernatant from that added to each tube prior to adsorption. The pH of one replicate per soil type per each test solution will be measured following decanting of the adsorption solution.

Additionally, the supernatant remaining after adsorption will be analyzed from one replicate per soil type at the highest concentration by TLC (TABLE I) and HPLC (TABLE II).

Desorption

Desorption is performed on the soil samples following adsorption. Calcium chloride aqueous solution at a concentration of 0.01M without test substance is added to all centrifuge tubes containing soil pellets used for adsorption. The volume of 0.01M $CaCl_2$ solution added will be the same as the volume used for the (definitive) adsorption phase. The soil pellet is then suspended by vortexing and manual shaking until dispersed. Soil samples at the 0.0 ppm level are also desorbed to demonstrate radiocarbon free calcium ion solution and no radiocarbon desorption from the control soils. The entire sample set is shaken in the dark for the equilibrium time using an Eberbach shaker at 175 to 200 rpm in a constant temperature incubator at 25±1°C. At the equilibrium time, the tubes are removed from the incubator, centrifuged at greater than 1000 rpm for 15 minutes (or until the soil is firmly packed) to pellet the soil, and aliquots of the supernatant removed. Aliquots of at least 100 µL of each supernatant solution are removed and analyzed by LSC to determine the equilibration concentrations (C_e,) of CGA-329351. Solutions remaining after desorption are decanted and analyzed by LSC. Desorption soil concentration (x/m) is determined by combustion of the residual soil with correction for the amount of radioactivity present in the solution remaining with each soil sample.

Additionally, the solution remaining after desorption will be analyzed from one replicate per soil type at the highest concentration by TLC (TABLE I) and HPLC (TABLE II).

The material balance will be calculated for every sample and will be determined by adding the total dpm in the adsorption solution, plus the dpm in the desorption solution, plus the dpm in the combusted soils. The target recovery will be ≥90% and ≤110%.

The soil pellet from the highest dose level of each soil type will be extracted by treating the soil with an approximate 1:1 mL/g solution of 90:10 acetonitrile/water and homogenized for approximately 15 minutes by sonication using a Branson sonicator. The samples will then be centrifuged at greater than 1000 rpm for 15 minutes to pellet the soil (or until the soil is firmly packed). The extract will be decanted, and the soil will be sonicated/centrifuged once more. The combined extract will be radioassayed in triplicate. The remaining soil pellet will be air dried at ambient laboratory temperatures in a laboratory hood. Aliquots of the air-dried soil pellet will be oxidized by combustion and the resulting radioactive carbon dioxide ($^{14}CO_2$) will be analyzed by LSC. Additionally, an aliquot of the pooled organic extract will be analyzed, if sufficient radioactivity is extracted (>1000 dpm/100 µL) by TLC (TABLE I) and HPLC (TABLE II) to determine the amount of test substance remaining in the soil after adsorption.

RADIOCARBON ANALYSIS

All solutions will be analyzed (preliminary test in duplicate, definitive in triplicate) by direct LSC in appropriate scintillation fluor. Soils are oxidized in aliquots of 200 mg or greater (preliminary test in duplicate, definitive in triplicate), using an R.J. Harvey Instruments Corporation Biological Materials Oxidizer (BMO). BMO generated $^{14}CO_2$ is trapped in appropriate scintillation fluor and quantified by LSC. The machine efficiency of the oxidizer will be determined before every series of soil combustions by oxidizing a known amount of radioactive standard. A machine combustion efficiency of less than 95% will not be accepted. The efficiency of the soil oxidation procedure will be determined before every series of soil combustions using aliquots of each untreated soil type fortified with known amounts of the combustion standard (definitive test only). For preliminary work, the soil oxidation efficiency will be determined using one soil only. The fortified soil aliquots will be oxidized by combustion and the resulting $^{14}CO_2$ will be counted by LSC. The amount of radioactivity recovered from each sample will be divided by the amount of radioactivity applied to the sample to determine an oxidation efficiency factor (sample efficiency). For soil sample oxidation efficiencies <100%, the recoveries will be adjusted. A sample combustion efficiency of less than 90% will not be accepted.

All quantification is performed using two channel counting for five minutes, or two sigma, with a Beckman Liquid Scintillation Spectrophotometer. Obtained counts per minute (cpm) are converted to disintegrations per minute (dpm) using the external standard ratio (H#) and math package features of the instrument.

TLC plates are scanned using a TLC scanner. Each chromatography plate is scanned by the analyzer for a minimum time to yield a usable image. All TLC scan data are stored on disks for marking regions of interest and retrieval of data from the computer.

Radiocarbon analysis will be performed using an AMBIS Radioanalytical Imaging System for TLC and a Shimadzu HPLC system with radioactivity monitor (RAM) for HPLC. At a minimum, 10% of the samples quantitated using the AMBIS Radioanalytical Imaging System will be confirmed by scraping the radioactive zones from the plate and analyzing by LSC. Also, at a minimum, 10% of the samples quantitated by the HPLC RAM will be confirmed by collecting fractions from the HPLC and subsequent analysis by LSC.

CALCULATIONS

The Freundlich equation (Reference 1) is applicable to the adsorption/ desorption of materials at intermediate concentrations and is commonly used to evaluate batch equilibrium data. The equation is an empirical relationship which may be expressed as:

$$C_{ads} = KC_e^{\,1/n}$$

where C_{ads} is the adsorbed concentration (µg), C_e is the solution equilibrium concentration (µg/mL) and K and n are constants. Another relationship which assumes that n equals one is the simple proportionality:

$$x/m = K_d\ C_e$$

where x/m is the adsorbed concentration (µg/g), C_e is the solution equilibrium concentration (µg/mL) and K_d is the distribution coefficient.

Both the adsorption and desorption phases are subjected to evaluation using the Freundlich equation. The adsorption coefficient K_d and the constant 1/n are calculated for each soil type. Additionally, K_{oc} (adsorption coefficient based on organic carbon) is determined from the following equation:

$$K_{oc} = (K_d \times 100)\ /\ \%\ O.C.$$

where: % O.C. = % Organic Carbon = % Organic Matter/1.724

STATISTICAL METHODS

Mathematical methods of linear regression (used to calculate K_d and n values), means, sums, and logs will be used for data reduction. No other statistical methods will be used.

RECORDS TO BE MAINTAINED

Analyst(s) will maintain records in which all procedures, weights, observations, etc., relevant to the study will be documented. Computer software program names and version numbers, if used, will also be recorded. Chromatograms, computer printouts, etc., will be clearly labeled and maintained by the analyst(s) for the duration of the study. Study records will be reviewed by the study director, the study monitor, and audited by the testing facility's QAU. Study records will be archived at the completion of the study.

REPORT

A final report will be issued in draft form after termination of the study. The final report will meet the reporting requirements under Subdivision N (160-5 and 163-1), PR 86-5, Data Reporting Guidelines, and GLPS (40 CFR Part 160.185), and will include, but not be limited to:

(1) complete description of test and reference substances,

(2) soil characterization,

(3) description of all procedures and analytical methods,

(4) concentration of radioactivity ($\mu g/g$ soil or $\mu g/mL$ solution) in all soils and equilibrium solutions,

(5) examples of calculations for n and K_d,

(6) detection limits, where appropriate,

(7) radiocarbon material balance for study phases (preliminary and definitive) will be calculated and reported,

(8) tabulated values of K_d (adsorption and desorption), n, and K_{oc},

(9) graphic presentation of adsorption and desorption isotherms,

(10) general information that will be reported will include: testing facility name and location, date of study initiation and completion, and the names and signatures of scientists responsible for the conduct and the study. Also included in the report will be a copy of the study protocol and any amendments,

(11) storage conditions of test substance,

(12) test system temperature data,

(13) Quality Assurance and US EPA GLPS Compliance Statements,

(14) location of storage of raw data, including the final report, and specimens.

After incorporation of the Sponsor's comments into the final report, the final report will be signed and a copy provided to the Sponsor.

ARCHIVES

At the completion of the study, all original raw data, including but not limited to, LSC counting data, data notebooks, TLC and HPLC chromatograms, documentation, records, and the original approved protocol and final report will be retained in the archives of Agrisearch Incorporated, 5734 Industry Lane, Frederick, Maryland 21701. A certified copy of the raw data and a final report will be provided to Ciba for storage in their archives. Supporting records to be retained by Agrisearch Incorporated and not necessarily archived with the study data will include, but not necessarily limited to, refrigerator and freezer temperature records and instrument calibration and maintenance records.

Specimens shall be retained by/at Agrisearch Incorporated for quality assurance verification and approval by the Sponsor to dispose, at which time the specimens shall be disposed of in accordance to pertinent Agrisearch Incorporated standard operating procedures.

QUALITY ASSURANCE

Agrisearch Incorporated has established a Quality Assurance Unit (QAU) as a commitment to performing laboratory studies in compliance with Good Laboratory Practice Standards (GLPS) as established by the Environmental Protection Agency. The independent QAU monitors each study in progress to assure conformance with applicable regulations.

All final reports issued by Agrisearch Incorporated are signed by the Quality Assurance Officer. This signature on the final report documents study examination by the QAU. The final report will include a signed statement including dates on which inspection and/or audits were performed and reports of findings were sent to Agrisearch Incorporated management and to the study director. Any deviations from the US EPA GLPS will be immediately reported to the study director and subsequently documented with the protocol. Additionally, each study may be subject to one or more unannounced in progress inspections. In progress inspections ensure that the study methods conform to GLPS and the protocol.

It is agreed that this is the official protocol for the conduct of this study. Any amendments will contain the reason for the amendment, be signed and dated by the sponsor representative and the study director and maintained with the protocol.

In the event that protocol modifications are required during the course of the study, the Sponsor's Representative will be contacted by phone for a verbal authorization. Any modification in the protocol and its justification will be documented in writing and approved by the study director and sponsor representative via protocol amendment.

REFERENCES

(1) [redacted]. and [redacted] and [redacted] "Measurement of Sorption Coefficients of Organic Chemicals and Their Use in Environmental Fate and Movement of Toxicants," AOAC Symposium Proceedings, 94th Annual Meeting, Washington, D.C., October 21-22, 1980.

(2) "Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate," U.S. Environmental Protection Agency, Office of Pesticide and Toxic Substances, Washington, DC, 20460; EPA 540/9-82-021, October 18, 1982. NTIS PB83-153973.

(3) U.S. Environmental Protection Agency, August 17, 1989. Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) Good Laboratory Practice Standards; Final Rule (40 CFR: 160). Federal Register, Vol. 54, No. 158: 34052-34074.

(4) U.S. Environmental Protection Agency, Data Reporting Guidelines, Environmental Fate Series 163-1, Leaching and Adsorption/Desorption Studies, Addendum 6, PB-88-161195, January 1988.

(5) FIFRA Accelerated Reregistration, Phase 3 Technical Guidance, EPA 540/09-90-078, December 1989.

(6) U.S. Environmental Protection Agency, Pesticide Reregistration Rejection Rate Analysis, Environmental Fate, EPA 738-R-93-010, September 1993.

TABLE I: THIN-LAYER CHROMATOGRAPHY (TLC)

Silica Gel Plates:

Precoated glass backed silica gel 60 F254 (Merck)
layer thickness of 0.25 mm

Candidate Solvent Systems:

(1) chloroform/methanol/formic acid/water 75:20:4:2 (v:v:v:v),

(2) chloroform/methanol/ammonium hydroxide/water 80:30:4:2 (v:v:v:v),

Visualization: UV - Light at 254 nm

TABLE II: HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC CONDITIONS FOR THE ANALYSIS OF CGA-329351

INSTRUMENT: Shimadzu SCL-6A Liquid Chromatograph

ANALYTICAL COLUMN: Zorbax ODS
    Length: 250 mm
    Diameter: 4.6 mm
    Particle Size: 5 microns

GUARD COLUMN: Zorbax ODS

MOBILE PHASE: Solvent 1: acetonitrile
    Solvent 2: 0.0125% trifluoroacetic acid

GRADIENT:

Time (min.)	Solvent 1 %	Solvent 2 %
0	10	90
25	50	50
30	70	30
40	100	0
45	100	0
50	10	90
Stop		

FLOW RATE: 1.0 mL/min

RUN TIME: 50 min

CHART SPEED: 5 mm/min

WAVELENGTH: 230 nm

AUFS: 0.002

DETECTORS: UV and Raytest RAMONA-5-LS Radioactive Detector (RAM) Solid Flow Cell

CGA-329351 APPROXIMATE RETENTION TIME: 28 min

All parameters may be modified to optimize the analysis of CGA-329351.

AGRISEARCH INCORPORATED
Log Of SOP/Protocol Deviations

Project No.: 12220

DESCRIPTION	DATE	INIT	STUDY DIRECTOR SIGNATURE
• Aliquots of soil for combustion were to be ≥ 200 mg. To retain sufficient clay soil to be able to extract the sample following desorption less than 200 mg was combusted. No negative impact on study.	9/26/95	5.1.2.e Woo	
• Calibration of the balance used on 7/31/95 to weigh preliminary study test system was insufficient to bracket the weight of those test systems.	10/9/95	5.1.2.e Woo	
• The protocol stated that non-labeled CGA-329351 would be stored at room temperature. However it was stored under refrigeration as were the other reference substances.	10/9/95	5.1.2.e Woo	
• A temporary summer employee who assisted in some parts of the preliminary testing failed to sign the Project Personnel Log.	11/10/95	5.1.2.e Woo	
The protocol states Agrisearch will receive the raw data from Agvise Labs for soil characterization, but in fact Agrisearch receives only their report.	11/17/95	5.1.2.e Woo	

AGI-L006-10/22/91

AGRISEARCH INCORPORATED
Project Personnel Log

Project No. 12220

Study Title Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

The Agrisearch Incorporated personnel listed below have participated in the technical activities of this study. Signature to this record indicates that the protocol and pertinent SOPs have been read and understood by all of the following personnel:

Name (Please Print)	Signature	Init	Date
[redacted]	[redacted]	[redacted]	6/19/95
[redacted]	[redacted]	[redacted]	6/20/95
[redacted]	[redacted]	[redacted]	7.14-9
[redacted]	[redacted]	[redacted]	7/14/95
[redacted]	[redacted]	[redacted]	8/17/95

5.1.2.e Woo

APPENDIX B

Test Substance Receipt Documents

Ciba Crop Protection

ciba

Ciba-Geigy Corporation
P.O. Box 18300
Greensboro, NC 27419-8300
Telephone 910 632 6000

June 1, 1995

5.1.2.e Woo
AgriSearch Inc.
5734 Industry Lane
Frederick, MD 21701

Dear Mr. 5.1.2.e Woo:

I am sending by Federal Express the following compounds for use in your research with regard to Adsorption/Desorption of ^{14}C-Metalaxyl and CGA-329351.

ϕ-^{14}C-CGA-48988
Reference Number: WFH-IX-65
10.0 mg = 0.732 mCi
Specific Activity - 73.2 µCi/mg
Chemical purity was 99.2% by GC (Ext. Std.) on 3/22/95
Radiochemical purity was 97.8% by TLC on 5/26/95
Expiration Date - 11/95
STORE IN FREEZER

The TLC systems used to determine the radiochemical purity were:
Chloroform:Methanol (95:5)
Toluene:Dioxane:Ligroine:Glacial Acetic Acid (4:2:2:1)

ϕ-^{14}C-CGA-329351
Reference Number: MSR-II-90
10.0 mg = 0.811 mCi
Specific Activity - 81.1 µCi/mg
Chemical purity was 99.9% by GC (Ext. Std.) on 3/9/95
Radiochemical purity was 98.8% by TLC on 5/26/95
Expiration Date - 11/95
STORE IN FREEZER

The TLC systems used to determine the radiochemical purity were:
Chloroform:Methanol (95:5)
Toluene:Dioxane:Ligroine:Glacial Acetic Acid (4:2:2:1)

All characterization of the above compounds was completed prior to shipment and included additionally, as a minimum, mass spectroscopy, and structure verification by cochromatography. Characterization raw data have been archived at Ciba-Geigy Corporation, Greensboro, NC.

License No.: MD-21-013-01

I request that no transfers be made to other parties and that you not use material remaining after the recommended expiration date with appropriate reanalysis. If you require more material, consideration will be given to replenish your supply.

Sincerely,

[redacted]

[redacted], Ph.D.
Chemical Synthesis
Biochemistry Resources Department

mae:3DPS0601

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-041 Date Received 6·6·95

Sponsor Ciba-Geigy Date Logged 6·6·95

Supplier Ciba-Geigy

CHEMICAL

Name/ID No. ϕ-^{14}C-CGA-329351

Lot/Batch No. MSR-II-90

Amount Received Per Supplier 10.0 mg = 0.811 mCi

Description chemical purity 99.9%
radiochemical purity 98.8% S.A.= 81.1 μCi/mg
expiration date 11·95

Shipped Via Fed Exp. 384 0796 373

Storage: Room Temp. Refrigerate [✓ Freezer]

[✓ Test Substance] Metabolite/Degradate Analytical Standard

Initials 5.1.2.e Woo

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

ciba

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Ciba Plant Protection
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba: 1-800-888-8372

CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: ϕ-14C-CGA 329351 CAS #: Not Assigned
Active Ingredient (%): ϕ-14C-CGA 329351
Chemical Name: Proprietary
Chemical Class: Fungicide (labled with carbon-14)

II. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
ϕ-14C-CGA 329351	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious give 1 or 2 glasses of water to drink, Never give anything by mouth to an unconscious person.
Eye Contact:	Immediately rinse eyes with a large amount of running water. Hold eyelids apart to rinse the entire surface of the eye and lids. Do not apply any medicating agents except on the advice of a physician.
Skin Contact:	Wash with plenty of soap and water, including hair and under fingernails. Do not apply any medicating agents except on the advice of a physician. Remove contaminated clothing and wash before reuse.
Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals. There are no medical conditions which are known to be aggravated by exposure to this material.

Symptoms of Acute Exposure
Contact with eyes can cause irreversible eye damage. A sensitizing (allergic) reaction may occur in sensitive individuals.

Acute Toxicity Studies
Oral LD_{50} (Rat) = 375 to 950 mg/kg
Dermal LD_{50} (Rat) = >2,000 mg.kg
Inhalation LC_{50} (Rat) = >2.3 mg/L - 4 Hrs.

Eye (Rabbit) = Corrosive
Skin (Rabbit) = Non-irritating
Sensitization (Guinea Pig) = Weak sensitizer

Chronic/Subchronic Toxicity Studies
Not Available

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.
Eye Contact:	To avoid eye contact, wear chemical safety glasses or goggles.
Skin Contact:	To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.
Inhalation:	To avoid breathing dust or vapors, wear NIOSH-approved chemical cartridge pesticide respirator or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Colorless to slightly yellow liquid.
Odor:	Not Available
Melting Point:	Not Available
Solubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

In Case of Spill or Leak
Wear specified protective clothing (Section V). For small spills, cover with an absorbent material, sweep up and place in approved chemical container. Wash spill area with water containing a strong detergent, absorb with absorbent material, sweep up and place in chemical container. Seal container and handle in an approved manner. Flush the spill area with water to remove any residue. Do not contaminate water supplies with wash water.

In Case of Fire
Use dry chemical, foam, or CO_2 extinguishing media. Wear full protective clothing and self-contained breathing apparatus. Evacuate nonessential personnel from the area to prevent human exposure to fire, smoke, fumes, or products of combustion. Prevent use of contaminated buildings, area, and equipment until decontaminated.

Disposal
Do not reuse product containers. Dispose of product containers, waste containers, and residues according to local, state, and federal health and environmental regulations.

Questions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
1-800-334-9481

Issued Date: 3/22/95
Revised Date:

Supersedes:

The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-038 Date Received 5·31·95

Sponsor Ciba Geigy Date Logged 5·31·95

Supplier Ciba Geigy

CHEMICAL

Name/ID No. CGA-329351

Lot/Batch No. S95-1796

Amount Received Per Supplier 100 mg

Description viscous amber liquid
purity 97.3%
reassay 5·96

Shipped Via UPS # 1Z 218 476 05 0235 6795

Storage: Room Temp. [✓ Refrigerate] Freezer

Test Substance Metabolite/Degradate [✓ Analytical Standard]

Initials [redacted]

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

ciba

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Crop Protection Division
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba-Geigy: 1-800-888-8372

CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: CGA-329351 Technical
CAS #: Not Assigned
Active Ingredient (%): CGA-329351
Chemical Name: Proprietary
Chemical Class: Fungicide

I. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
CGA-329351	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious, immediately give 1 or 2 glasses of water to drink. Never give anything by mouth to an unconscious person. Apply artificial respiration if necessary.
Eye Contact:	Immediately rinse eyes with a large amount of running water. Hold eyelids apart to rinse the entire surface of the eye and lids. Get medical attention.
Contact:	Wash with plenty of soap and water, including hair and under fingernails. Do not apply any medicating agents except on the advice of a physician. Remove contaminated clothing and wash before reuse.
Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals.

Symptoms of Acute Exposure
Contact with eyes can cause irreversible eye damage. A sensitizing (allergic) reaction may occur in sensitive individuals.

Acute Toxicity Studies
Oral LD_{50} (Rat) = 375 to 950 mg/kg
Dermal LD_{50} (Rat) = >2,000 mg/kg
Inhalation LC_{50} (Rat) = >2.3 mg/L - 4Hrs.
Eye (Rabbit) = Corrosive
Skin (Rabbit) = Non-irritating
Sensitization (Guinea Pig) = Weak sensitizer

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.
Eye Contact:	To avoid eye contact, wear chemical goggles or full-face shield.
Skin Contact:	To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.
Inhalation:	To avoid breathing dust or vapors, wear NIOSH-approved pesticide or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Not Available
Odor:	Not Available
Melting Point:	Not Available
Solubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

In Case of Spill or Leak

Wear specified protective clothing (Section V). For small spills, cover with an absorbent material, sweep up and place in approved chemical container. Wash spill area with water containing a strong detergent, absorb with absorbent material, sweep up and place in chemical container. Seal container and handle in an approved manner. Flush the spill area with water to remove any residue. Do not contaminate water supplies with wash water.

In Case of Fire

Use dry chemical, foam, or CO_2 extinguishing media. Wear full protective clothing and self-contained breathing apparatus. Evacuate nonessential personnel from the area to prevent human exposure to fire, smoke, fumes, or products of combustion. Prevent use of contaminated buildings, area, and equipment until decontaminated.

Disposal

Do not reuse product containers. Dispose of product containers, waste containers, and residues according to local, state, and federal health and environmental regulations.

Questions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
1-800-334-9481

Issued Date: 5/13/94
Revised Date: 3/18/95
Supersedes: 5/13/94

The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

Ciba Crop Protection

ciba

Ciba-Geigy Corporation
P.O. Box 18300
Greensboro, NC 27419-8300
Telephone 910 632 6000

May 24, 1995

Mr. 5.1.2.e Woo
AgriSearch, Inc.
5734 Industry Lane
Frederick, MD 21701

Dear Mr. 5.1.2.e Woo:

I am sending by Federal Express the following compounds for use in your research with regard to Protocols 59-95 and 60-95:

- CGA-37734
  Reference Number: BPM-I-8
  Approximately 50 mg
  Chemical purity: 96.9% by GC (area distribution) on 2/2/94
  Storage: Refrigerator
  Expiration Date: 2/96

- CGA-42447
  Reference Number: MCO-I-2
  Approximately 50 mg
  Chemical purity: 99.7% by GC (area distribution) on 12/12/94
  Storage: Refrigerator
  Expiration Date: 12/96

- CGA-62826
  Reference Number: BPM-I-4B
  Approximately 50 mg
  Chemical purity: >99.9% by HPLC (area distribution) on 12/8/94
  Storage: Refrigerator
  Expiration Date: 12/96

- CGA-119857
  Reference Number: GB-XLV-3
  Approximately 50 mg
  Chemical purity: >99.9% by HPLC (area distribution) on 12/8/94
  Storage: Refrigerator
  Expiration Date: 12/96

All characterization of the above compounds was completed prior to shipment and included additionally, as a minimum, elemental analysis, mass spectroscopy, nuclear magnetic resonance spectroscopy, and infrared spectroscopy. Characterization raw data have been archived at Ciba-Geigy Corporation, Greensboro, NC.

Sincerely,

[redacted]

[redacted], Ph.D.

Chemical Synthesis
Biochemistry Resources Department

3DS0524/ms

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-034 Date Received 5·31·95
Sponsor Ciba Geigy Date Logged 5·31·95
Supplier Ciba Geigy

CHEMICAL

Name/ID No. CGA-37734
Lot/Batch No. BPM-I-8

Amount Received Per Supplier ~ 50 mg
Description off-white solid
purity 96.9 %
expiration date 2·96

Shipped Via Fed Exp. 384 0793 887

Storage: Room Temp. [✓ Refrigerate] Freezer

Test Substance Metabolite/Degradate [✓ Analytical Standard]

Initials 5.1.2.e Woo

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

ciba

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Ciba Plant Protection
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba: 1-800-888-8372

CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: CGA 37734 CAS #: 29183-14-0
Active ingredient (%): CGA 37734
Chemical Name: N-(2,6-dimethylphenyl)-2-hydroxyacetamide
Chemical Class: CGA 48988 metabolite

II. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
CGA 37734	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious give 1 or 2 glasses of water to drink, Never give anything by mouth to an unconscious person.
Eye Contact:	Immediately rinse eyes with a large amount of running water. Hold eyelids apart to rinse the entire surface of the eye and lids. Do not apply any medicating agents except on the advice of a physician.
Skin Contact:	Wash with plenty of soap and water, including hair and under fingemails. Do not apply any medicating agents except on the advice of a physician. Remove contaminated clothing and wash before reuse.
Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals. There are no medical conditions which are known to be aggravated by exposure to this material.

Symptoms of Acute Exposure
Not Available

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.

Eye Contact:	To avoid eye contact, wear chemical safety glasses or goggles.
Skin Contact:	To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.
Inhalation:	To avoid breathing dust or vapors, wear NIOSH-approved chemical cartridge pesticide respirator or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Off-White Solid
Odor:	Not Available
Melting Point:	Not Available
Solubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

In Case of Spill or Leak
Wear specified protective clothing (Section V). For small spills, cover with an absorbent material, sweep up and place in approved chemical container. Wash spill area with water containing a strong detergent, absorb with absorbent material, sweep up and place in chemical container. Seal container and handle in an approved manner. Flush the spill area with water to remove any residue. Do not contaminate water supplies with wash water.

In Case of Fire
Use dry chemical, foam, or CO_2 extinguishing media. Wear full protective clothing and self-contained breathing apparatus. Evacuate nonessential personnel from the area to prevent human exposure to fire, smoke, fumes, or products of combustion. Prevent use of contaminated buildings, area, and equipment until decontaminated.

Disposal
Do not reuse product containers. Dispose of product containers, waste containers, and residues according to local, state, and federal health and environmental regulations.

Questions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
1-800-334-9481

Issued Date: 9/6/94
Revised Date:
Supersedes:

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-035 Date Received 5.31.95

Sponsor Ciba Geigy Date Logged 5.31.95

Supplier Ciba Geigy

CHEMICAL

Name/ID No. CGA-42447

Lot/Batch No. MCO-I-2

Amount Received Per Supplier ~50 mg

Description white solid

purity: 99.7%

expiration date 12.96

Shipped Via Fed Exp. 384 0793 887

Storage: Room Temp. [✓ Refrigerate] Freezer

Test Substance Metabolite/Degradate [✓ Analytical Standard]

Initials 5.1.2.e Woo

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Ciba Plant Protection
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba: 1-800-888-8372

CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: CGA 42447 CAS #: 2198-53-0
Active Ingredient (%): CGA 42447
Chemical Name: N-(2,6-dimethylphenyl)acetamide
Chemical Class: CGA 48988 metabolite

II. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
CGA 42447	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious give 1 or 2 glasses of water to drink. Never give anything by mouth to an unconscious person.
Eye Contact:	Immediately rinse eyes with a large amount of running water. Hold eyelids apart to rinse the entire surface of the eye and lids. Do not apply any medicating agents except on the advice of a physician.
Skin Contact:	Wash with plenty of soap and water, including hair and under fingernails. Do not apply any medicating agents except on the advice of a physician. Remove contaminated clothing and wash before reuse.
Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals. There are no medical conditions which are known to be aggravated by exposure to this material.

Symptoms of Acute Exposure
Not Available

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.

Eye Contact:	To avoid eye contact, wear chemical safety glasses or goggles.
Skin Contact:	To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.
Inhalation:	To avoid breathing dust or vapors, wear NIOSH-approved chemical cartridge pesticide respirator or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Off-White Solid
Odor:	Not Available
lting Point:	Not Available
ubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

In Case of Spill or Leak

Wear specified protective clothing (Section V). For small spills, cover with an absorbent material, sweep up and place in approved chemical container. Wash spill area with water containing a strong detergent, absorb with absorbent material, sweep up and place in chemical container. Seal container and handle in an approved manner. Flush the spill area with water to remove any residue. Do not contaminate water supplies with wash water.

In Case of Fire

Use dry chemical, foam, or CO_2 extinguishing media. Wear full protective clothing and self-contained breathing apparatus. Evacuate nonessential personnel from the area to prevent human exposure to fire, smoke, fumes, or products of combustion. Prevent use of contaminated buildings, area, and equipment until decontaminated.

Disposal

Do not reuse product containers. Dispose of product containers, waste containers, and residues according to local, state, and federal health and environmental regulations.

estions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
1-800-334-9481

Issued Date: 2/3/95
Revised Date:

Supersedes:

The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-036 Date Received 5·31·95

Sponsor Ciba Geigy Date Logged 5·31·95

Supplier Ciba Geigy

CHEMICAL

Name/ID No. CGA-62826

Lot/Batch No. BPM-I-4B

Amount Received Per Supplier ~50 mg.

Description off-white solid
purity >99.9%
expiration date 12·96

Shipped Via Fed Exp #384 0793 887

Storage: Room Temp. [✓ Refrigerate] Freezer

Test Substance Metabolite/Degradate [✓ Analytical Standard]

Initials 5.1.2.e Woo

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

ciba

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Ciba Plant Protection
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba: 1-800-888-8372
1-800-334-9481
CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: CGA 62826 CAS #: 87764-37-2
Active Ingredient (%): CGA 62826
Chemical Name: N-(2,6-dimethylphenyl)-N-(methoxyacetyl)-DL-alanine
Chemical Class: CGA 48988 metabolite

II. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
CGA 62826	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious give 1 or 2 glasses of water to drink. Never give anything by mouth to an unconscious person.
Eye Contact:	Immediately rinse eyes with a large amount of running water. Hold eyelids apart to rinse the entire surface of the eye and lids. Do not apply any medicating agents except on the advice of a physician.
Skin Contact:	Wash with plenty of soap and water, including hair and under fingemails. Do not apply any medicating agents except on the advice of a physician. Remove contaminated clothing and wash before reuse.
Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals. There are no medical conditions which are known to be aggravated by exposure to this material.

Symptoms of Acute Exposure
Not Available

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.

Eye Contact: To avoid eye contact, wear chemical safety glasses or goggles.

Skin Contact: To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.

Inhalation: To avoid breathing dust or vapors, wear NIOSH-approved chemical cartridge pesticide respirator or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Off-white Solid
Odor:	Not Available
Melting Point:	Not Available
Solubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

In Case of Spill or Leak

Wear specified protective clothing (Section V). For small spills, cover with an absorbent material, sweep up and place in approved chemical container. Wash spill area with water containing a strong detergent, absorb with absorbent material, sweep up and place in chemical container. Seal container and handle in an approved manner. Flush the spill area with water to remove any residue. Do not contaminate water supplies with wash water.

In Case of Fire

Use dry chemical, foam, or CO_2 extinguishing media. Wear full protective clothing and self-contained breathing apparatus. Evacuate nonessential personnel from the area to prevent human exposure to fire, smoke, fumes, or products of combustion. Prevent use of contaminated buildings, area, and equipment until decontaminated.

Disposal

Do not reuse product containers. Dispose of product containers, waste containers, and residues according to local, state, and federal health and environmental regulations.

Questions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
(919) 632-6000

Issued Date: 7/7/93
Revised Date:

Supersedes:

The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

AGRISEARCH INCORPORATED
Chemical Receipt Log

Agrisearch No. 95-037 Date Received 5·31·95

Sponsor Ciba Geigy Date Logged 5·31·95

Supplier Ciba Geigy

CHEMICAL

Name/ID No. CGA-119857

Lot/Batch No. GB·XLV·3

Amount Received Per Supplier ~50 mg

Description off·white solid
purity >99.9%
expiration date 12·96

Shipped Via Fed Exp. #384 0793 887

Storage: Room Temp. [✓ Refrigerate] Freezer

Test Substance Metabolite/Degradate [✓ Analytical Standard]

Initials 5.1.2.e Woo

Unless otherwise noted above: all material(s) were received intact and per shipping documentation.

AGI-L001-12/28/92

MATERIAL SAFETY DATA SHEET

EXPERIMENTAL COMPOUND

Ciba-Geigy Corporation
Ciba Plant Protection
Post Office Box 18300
Greensboro, NC 27419

In Case of Emergency, Call
Ciba: 1-800-888-8372
1-800-334-9481
CHEMTREC: 1-800-424-9300

I. MATERIAL IDENTIFICATION

Product Name: CGA 119857
CAS #: Not Assigned
Active Ingredient (%): CGA 119857
Chemical Name: Not Available
Chemical Class: CGA 48988 metabolite

II. HAZARDOUS INGREDIENTS

Material	OSHA PEL	ACGIH TLV	NTP/IARC/OSHA Carcinogen
CGA 119857	None	None	No

III. FIRST AID PROCEDURES

If poisoning is suspected, immediately contact a physician, the nearest hospital, or the nearest Poison Control Center. Tell the person contacted the complete product name, and the type and amount of exposure. Describe any symptoms and follow the advice given.

Ingestion:	Do not induce vomiting. If victim is fully conscious give 1 or 2 glasses of water to drink. Never give anything by mouth to an unconscious person.
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Inhalation:	Move victim from contaminated area to fresh air. Apply artificial respiration if necessary.

IV. HEALTH HAZARD INFORMATION

The health hazards associated with this material have not been fully evaluated. This material should be used only by persons trained in handling experimental chemicals. There are no medical conditions which are known to be aggravated by exposure to this material.

Symptoms of Acute Exposure
Not Available

V. PERSONAL PROTECTION AND PRECAUTIONS

Ingestion:	Store the material in a well-ventilated, secure area out of the reach of children and domestic animals. Do not store food, beverages, or tobacco products in the storage area. Prevent eating, drinking, tobacco usage, and cosmetic application in areas where there is a potential for exposure to the material. Always wash thoroughly after handling.

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Skin Contact:	To avoid skin contact, wear rubber gloves, rubber boots, long-sleeved shirt, long pants, and head covering.
Inhalation:	To avoid breathing dust or vapors, wear NIOSH-approved chemical cartridge pesticide respirator or supplied-air respirator.

VI. PHYSICAL HAZARD INFORMATION

Flash Point:	Not Available
Stability:	Stable
Hazardous Polymerization:	Will Not Occur
Decomposition Products:	None Known
Conditions to Avoid:	None Known
Appearance:	Off-white Solid
Odor:	Not Available
ting Point:	Not Available
Solubility in H_2O:	Not Available
Boiling Point:	Not Available

VII. ENVIRONMENTAL PROTECTION

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Disposal

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estions concerning the safe handling of this material should be referred to:

Ciba-Geigy Corporation
Environmental Safety & Compliance
(919) 632-6000

Issued Date: 7/7/93
Revised Date:
Supersedes:

The information and recommendations contained herein are based upon data believed to be correct. However, no guarantee or warranty of any kind, expressed or implied, is made with respect to the information contained herein.

APPENDIX C

Soil Characterization and Soil Microbial Biomass Determination

Characterization Chain of Custody

AGVISE

P.O. Box 510 / Highway 15 • Northwood, ND 58267
(701) 587 6010 FAX (701) 587 6013

Series 1. Soil Characterization - 500 g sample minimum pH, % organic matter, cation exchange capacity, potassium, calcium, magnesium, sodium, hydrogen, water holding capacity (1/3 and 15 bar), % sand silt clay, texture, % total nitrogen, phosphorus, bulk density, and soluble salts.

Series 2. Soil Characterization - 500 g sample minimum Same as Series 1. less 15 bar WHC, % total nitrogen, phosphorus, and soluble salts.

Series 3. Water Characterization - 500 ml sample minimum pH, calcium, magnesium, sodium, hardness, conductivity, sodium absorption ratio, total dissolved solids, and turbidity.

Series 4. Water Characterization - 500 ml sample minimum pH, calcium, magnesium, sodium, hardness, sodium absorption ratio.

SEND RESULTS TO:

AGRISEARCH INCORPORATED
5734 Industry Lane
Frederick, Maryland 21701

Contact: [redacted]
Phone: 301- [redacted]

BILL TO:

AGRISEARCH INCORPORATED
13456 OLD ANNAPOLIS ROAD
Mt Airy, MD 21771

Purchase Order # 3609

AGVISE Use Only
Date received 6-12-95
By [redacted]

Protocol / Study# 12219, 12220
Other Study Info:

REC.	COMMENTS	AGVISE LAB #	SPECIMEN I.D.	DEPTH	REQUESTED TEST(S)
		95-1394	WA loam	0-6"	Series 2 + clay type determination
		95-1395	MS clay	0-6"	Series 2 + clay type determination
		95-1396	CA sandy loam S95-012	0-6"	Series 2 + clay type determination
		95-1397	MD sand S95-013	0-6"	Series 2 + clay type determination
		95-1398	AZ silty clay loam S95-014	0-6"	Series 2 + clay type determination

COPY OF ORIGINAL
AGVISE Laboratories, Inc.
Initial [redacted] Date 6-12-95

METHOD OF SHIPMENT:
Shipment I.D.#(s)

Fed Express ☐
UPS ☐
USPS ☐
Other

Sent by: [redacted]
Date: 6/9/95

Other Instructions:

A copy of this COC will be mailed to you when specimens are received by Lab.

AGVISE
LABORATORIES

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

NORTHWOOD
NORTH DAKOTA
58267-0510

AGVISE Soil Characterization Report

Submitting firm = AGRISEARCH INC
Protocol or Study No = 12219, 12220 11/14/95
Sample ID. = MS CLAY 0-6"
Trial ID. = NA
Date Received = 6-12-95
Date Reported = 06-23-1995

AGVISE Lab No 95- 1395

Percent Sand 23
Percent Silt 26
Percent Clay 51
USDA Textural Class (hydrometer method) Clay

Bulk Density (disturbed) gm/cc 1.20
Cation Exchange Capacity (meq/100 g) 37.8

% Moisture at 1/3 Bar 41.8

Percent Organic Matter 2.3

pH 7.0

Base Saturation Data

Cation	Percent	ppm
Calcium	64.9	4900
Magnesium	23.4	1060
Sodium	0.5	40
Potassium	2.8	407
Hydrogen	8.5	32

These tests were completed in compliance of 40 CFR Part 160.

5.1.2.e Woo

6/23/95
Date

Soil Scientist

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58267-0510

AGVISE Soil Characterization Report

Submitting firm = AGRISEARCH INC
Protocol or Study No = 12219 , 12220 11/14/95
Sample ID. = MD SAND S95-013 0-6"
Trial ID. = NA
Date Received = 6-12-95
Date Reported = 06-22-1995

AGVISE Lab No	95- 1397
Percent Sand	91
Percent Silt	6
Percent Clay	3
USDA Textural Class (hydrometer method)	Sand
Bulk Density (disturbed) gm/cc	1.52
Cation Exchange Capacity (meq/100 g)	4.1
% Moisture at 1/3 Bar	11.5
Percent Organic Matter	0.6
pH	5.4

Base Saturation Data

Cation	Percent	ppm
Calcium	36.3	300
Magnesium	16.1	80
Sodium	1.7	16
Potassium	5.2	83
Hydrogen	40.7	17

These tests were completed in compliance of 40 CFR Part 160.

5.1.2.e Woo

Soil Scientist

6/22/95
Date

P O. Box 510. HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58257-0510

AGVISE Soil Characterization Report

Submitting firm = AGRISEARCH INC
Protocol or Study No = 12219, 12220 11/14/95
Sample ID. = CASANDY LOAMS95-012 0-6"
Trial ID. = NA
Date Received = 6-12-95
Date Reported = 06-22-1995

AGVISE Lab No	95- 1396
Percent Sand	65
Percent Silt	30
Percent Clay	5
USDA Textural Class (hydrometer method)	Sandy Loam
Bulk Density (disturbed) gm/cc	1.41
Cation Exchange Capacity (meq/100 g)	8.0
% Moisture at 1/3 Bar	18.2
Percent Organic Matter	0.4
pH	6.3

Base Saturation Data

Cation	Percent	ppm
Calcium	56.5	900
Magnesium	15.7	150
Sodium	0.8	15
Potassium	8.9	277
Hydrogen	18.1	14

These tests were completed in compliance of 40 CFR Part 160.

5.1.2.e Woo

Soil Scientist

6/22/95
Date

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58267-0510

AGVISE Soil Characterization Report

Submitting firm = AGRISEARCH INC
Protocol or Study No = 12219, 12220 11/14/95
Sample ID. = WA LOAM 0-6"
Trial ID. = NA
Date Received = 6-12-95
Date Reported = 06-22-1995

AGVISE Lab No	95- 1394
Percent Sand	49
Percent Silt	42
Percent Clay	9
USDA Textural Class (hydrometer method)	Loam
Bulk Density (disturbed) gm/cc	1.14
Cation Exchange Capacity (meq/100 g)	22.7
% Moisture at 1/3 Bar	35.0
Percent Organic Matter	2.6
pH	7.0

Base Saturation Data

Cation	Percent	ppm
Calcium	55.2	2500
Magnesium	19.9	540
Sodium	0.5	27
Potassium	10.7	944
Hydrogen	13.8	31

These tests were completed in compliance of 40 CFR Part 160.

5.1.2.e Woo

6/22/95
Date

Soil Scientist

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58267-0510

AGVISE Soil Characterization Report

Submitting firm = AGRISEARCH INC
Protocol or Study No = 12219, 12220 11/14/95
Sample ID. = AZ SILTY S95-014 0-6"
Trial ID. = NA
Date Received = 6-12-95
Date Reported = 06-22-1995

AGVISE Lab No 95- 1398

Percent Sand	17
Percent Silt	52
Percent Clay	31
USDA Textural Class (hydrometer method)	Silty Clay Loam
Bulk Density (disturbed) gm/cc	1.18
Cation Exchange Capacity (meq/100 g)	50.4
% Moisture at 1/3 Bar	34.1
Percent Organic Matter	0.3
pH	7.9

Base Saturation Data

Cation	Percent	ppm
Calcium	68.4	6900
Magnesium	15.5	940
Sodium	9.4	1094
Potassium	2.8	557
Hydrogen	3.8	19

These tests were completed in compliance of 40 CFR Part 160.

5.1.2.e Woo

Soil Scientist

6/22/95
Date

METHODS SUMMARY FOR SOIL ANALYSIS

Page 1 of 2

TESTING LABORATORY: AGVISE Laboratories, Inc.
Box 510, Hwy. 15
Northwood, ND 58267
(701) 587-6010

The following is a summary of methods used by AGVISE Laboratories in the determination of soil physical characteristics, chemical properties and water relations. Some or all of these methods are used based upon protocol requirements.

CHEMICAL PROPERTIES

Carbonates - Determined by gravimetric loss of carbon dioxide (SOP# NUT.02.14).

Cation exchange capacity - Determined by summing the cations with hydrogen (SOP# NUT.02.03). The cations of Magnesium, Potassium, Calcium, and Sodium are determined by extraction with 1.0 N ammonium acetate (SOP# NUT.02.12). Hydrogen is determined by measuring the pH of the soil in Adams-Evans Buffer Solution (SOP# NUT.02.11).

Nitrogen, % Total - Determined by the Kjeldahl method (SOP# NUT.02.15).

Organic carbon % - Determined by the Walkley-Black procedure (SOP# NUT.02.04).

Organic matter % - Determined by the Walkley-Black procedure (SOP# NUT.02.09) in soils with less than 10% organic matter. Determined by the loss of weight on ignition procedure (SOP# NUT.02.04) in soils with 10% or more organic matter.

pH - Determined with a pH electrode in a 1:1 soil:water suspension (SOP# NUT.02.05) except when specified by state regulations to use a saturated paste (SOP# NUT.02.39).

Phosphorus - Determined by the Olsen method (SOP# NUT.02.07).

Soluble salts - Determined using a conductivity meter in a 1:1 soil:water suspension (SOP# NUT.02.19).

PHYSICAL PROPERTIES

% Gravel - Determined by dry sieving and weighing the fraction over 2mm (SOP# NUT.02.16).

% Sand, silt, and clay - Determined by hydrometer method (SOP# NUT.02.06) or sand determined by wet sieving and clay determined by hydrometer (SOP# NUT.02.47).

Sand particle size - Determined by weighing fractions obtained by wet sieving (SOP# NUT.02.32).

Bulk density - Disturbed bulk density is determined by weighing a known volume of dried and ground soil (SOP NUT#.02.10). Core or non-disturbed bulk density is determined by weighing a known volume of an intact, dried soil core (SOP# NUT.02.02).

WATER HOLDING CAPACITY AND WATER RELATIONS

Moisture % - Determined by gravimetric loss upon drying (SOP# NUT.02.36).

Saturated hydraulic conductivity - Determined by using the constant head method and measuring the rate of flow of water through a saturated soil column (SOP NUT.02.34).

Water infiltration rate - Determined by using the constant head method and measuring the length of time from water application to production of a leachate from a soil column (SOP# NUT.02.35).

Water holding capacity - Determined by measuring the moisture remaining when saturated soil is placed under 1/3 bar pressure (SOP NUT.02.08).

Water holding capacity - Determined by measuring the moisture remaining when saturated soil is placed under 15 bar pressure (SOP# NUT.02.13).

All of the above methods are detailed in the current analytical SOPs used by AGVISE Laboratories' Characterization testing laboratory.

APPROVED BY
ANALYTICAL INVESTIGATOR: 5.1.2.e Woo ____________ 4/19/93
5.1.2.e Woo, Soil Scientist Date

4-19-93

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58267-0510

The following personnel have been duly trained to perform Soil and Water Characterization under 40 CFR Part 160 Good Laboratory Practice Standards.

1995

5.1.2.e Woo - Laboratory Technician
5.1.2.e Woo - GLP Receiving Clerk/Data Entry Operator
5.1.2.e Woo - Analytical Investigator/Soil Scientist
5.1.2.e Woo - Senior Laboratory Technician
5.1.2.e Woo - GLP Data Entry Operator
5.1.2.e Woo - Laboratory Technician
5.1.2.e Woo - GLP Receiving Clerk Supervisor/Data Entry Operator
5.1.2.e Woo - Laboratory Technician
5.1.2.e Woo - Laboratory Technician
5.1.2.e Woo Laboratory Manager for Soil/Water Characterization
5.1.2.e Woo - Laboratory Technician
5.1.2.e Woo - Receiving GLP Receiving Clerk
5.1.2.e Woo - Laboratory Technician

Quality Assurance

5.1.2.e Woo - Quality Assurance Officer
5.1.2.e Woo - Quality Assurance Specialist
5.1.2.e Woo - Quality Assurance Specialist

6/16/95

P.O. Box 510, HWY 15
PH. (701) 587-6010
FAX: (701) 587-6013

AGVISE
LABORATORIES

NORTHWOOD
NORTH DAKOTA
58267-0510

August 1, 1995

5.1.2.e Woo
AGRISEARCH INCORPORATED
5734 Industry Lane
Frederick, MD 21701

Dear 5.1.2.e Woo

Enclosed are the results of the clay scans you requested.

Specimen ID - WA Loam AGVISE #95-1394
Clay minerals: Poorly crystallized smectite; kaolinite, and illite.
Other minerals: Quartz, feldspar, goethite.
Specimen ID - MS Clay AGVISE #95-1395
Clay minerals: Smectite, kaolinite, chlorite, and illite. Smectite confirmed by glycollation treatment.
Other minerals: Quartz, feldspar.
Specimen ID - CA Sandy Loam S95-012 AGVISE #95-1396
Clay minerals: Illite dominant; kaolinite. No evidence of smectite found by glycollation treatment.
Other minerals: Quartz, goethite and feldspar.
Specimen ID - MD Sand S95-013 AGVISE #95-1397
Clay minerals: Kaolinite, chlorite, and illite. Gibbsite suggested by peak at 18.3° 2θ.
Other minerals: Quartz and feldspar.
Specimen ID - AZ Silty clay loam S95-014 AGVISE #95-1398
Clay minerals: Smectite(confirmed by glycollation treatment), kaolinite, chlorite and illite. Glycollation treatment suggests the presence of a disordered interlayered clay mineral, such as smectite-illite.
Other minerals: Quartz, feldspar and calcite.

The scans were run on clay-size particles (<2 μm) oriented using the Millipore filter transfer method(Moore, D. M. and R. C. Reynolds, Jr. 1989 X-ray diffraction and the identification and analysis of clay minerals. Oxford University Press, New York). A Phillips automated diffractometer was used with CuK-alpha radiation to analyze the slides. Phases were identified by comparison with known scans, as well as by computer search-match procedures which employ the ICDD Powder Diffraction File. X-ray scans are for qualitative purposes only.

Analytical Testing & Field Research

The samples were received at AGVISE Laboratories, Northwood, ND, dried and prepared for analysis by grinding to a 1 mm size. The X-ray diffraction was done at North Dakota State University in Fargo, ND.

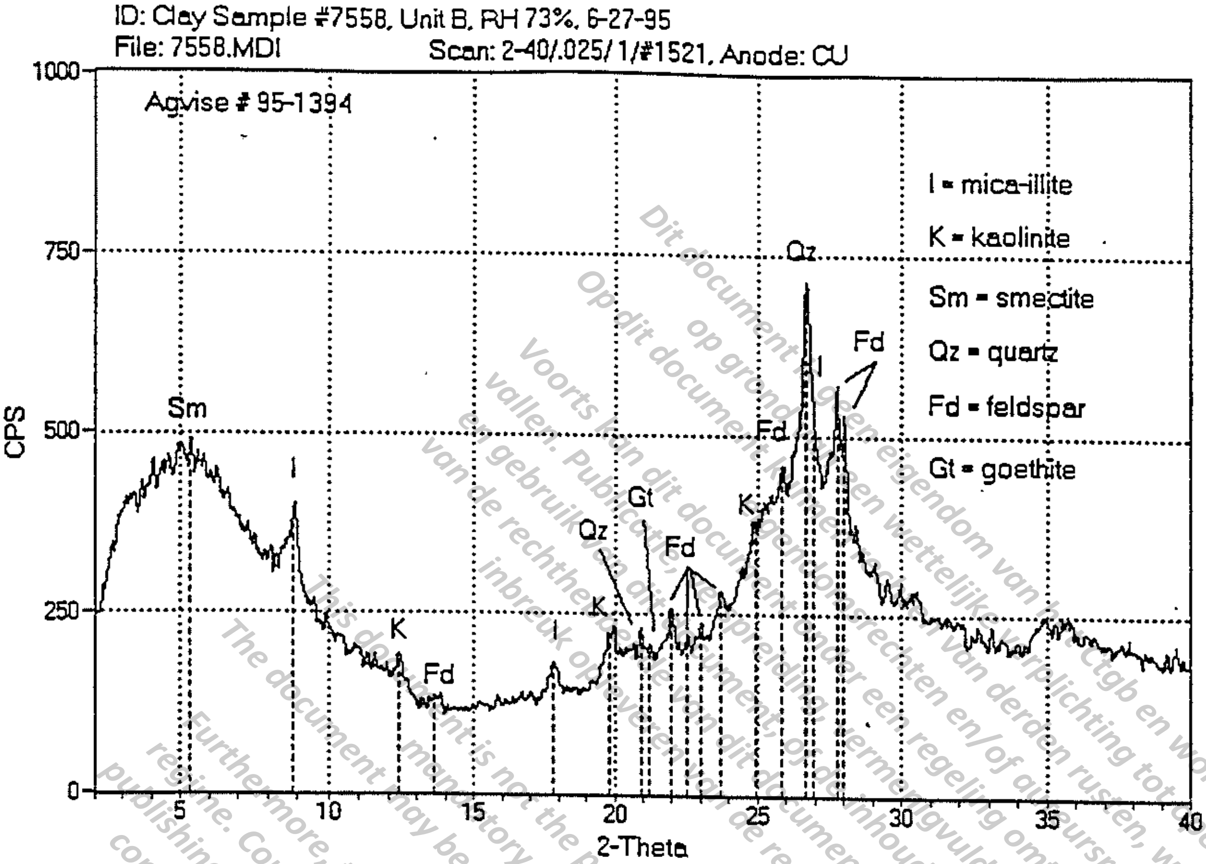
If you have any questions, please call.

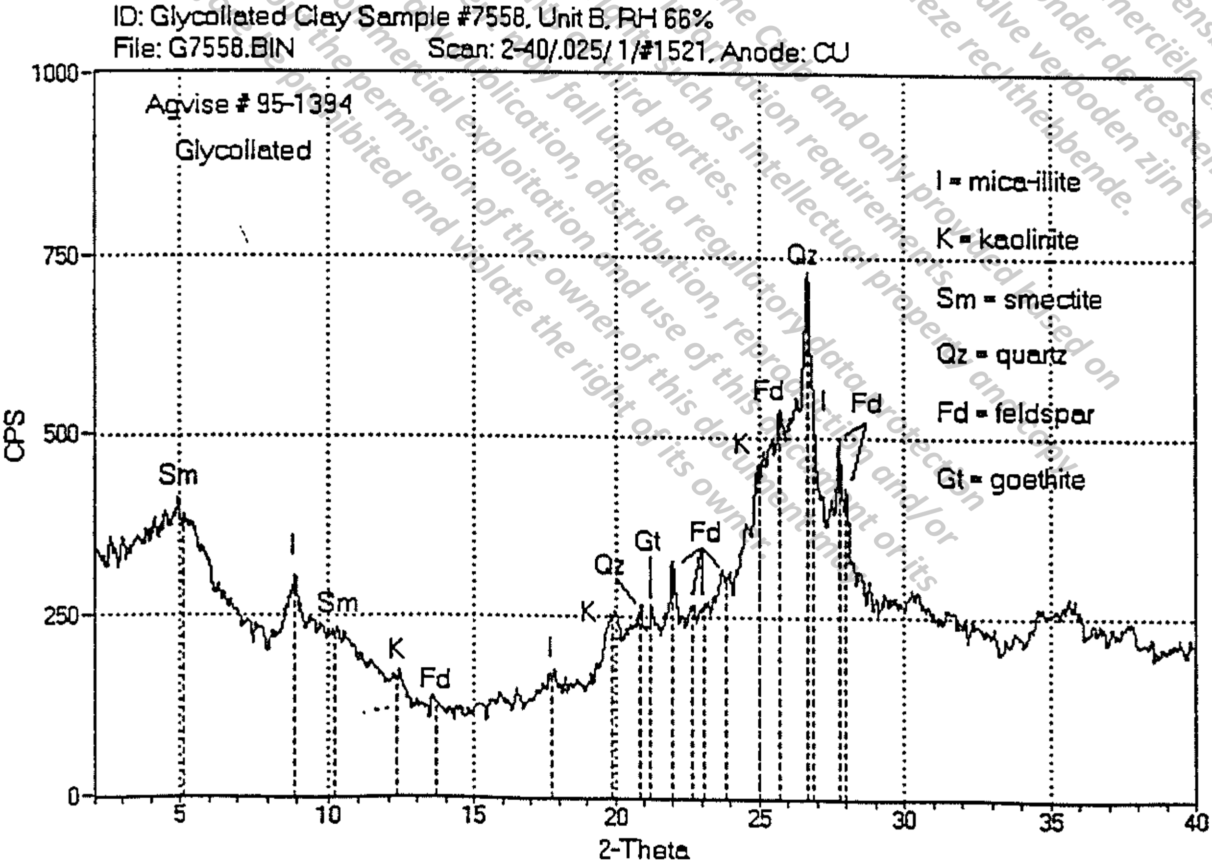
Sincerely,

5.1.2.e Woo

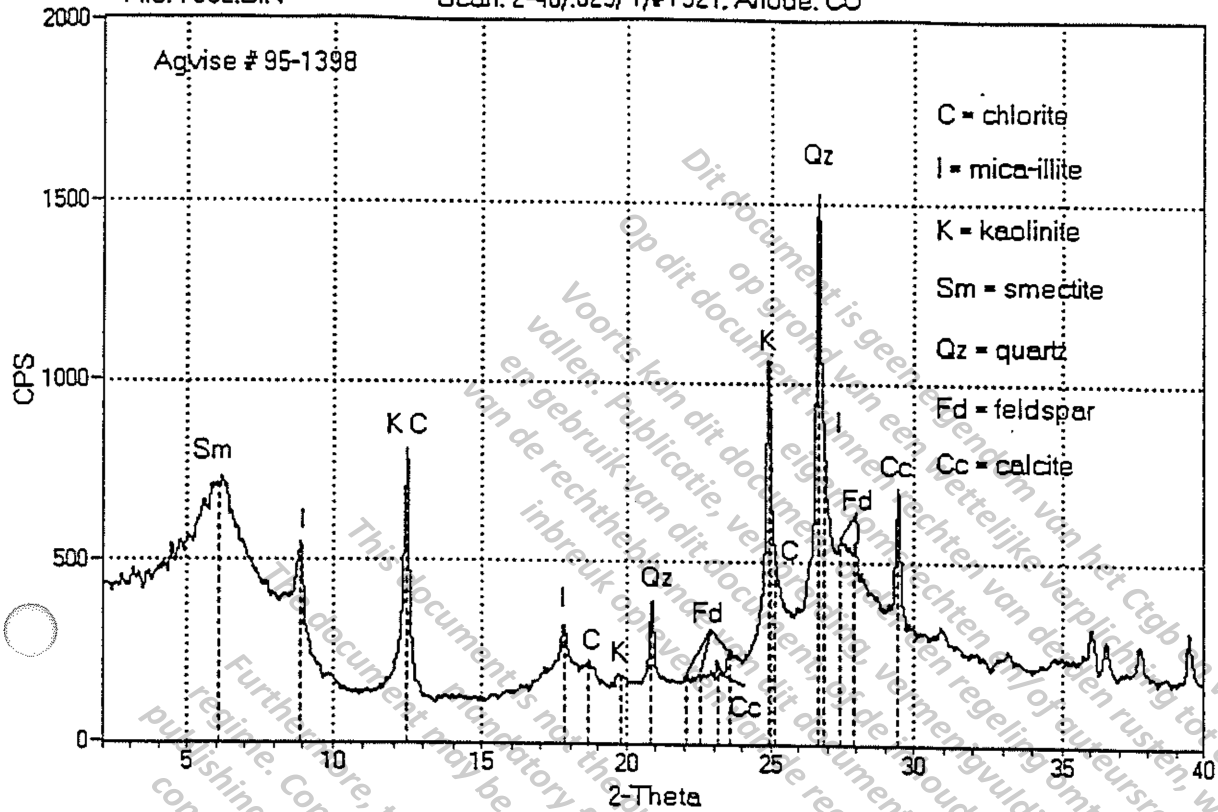
Soil Scientist

Enc

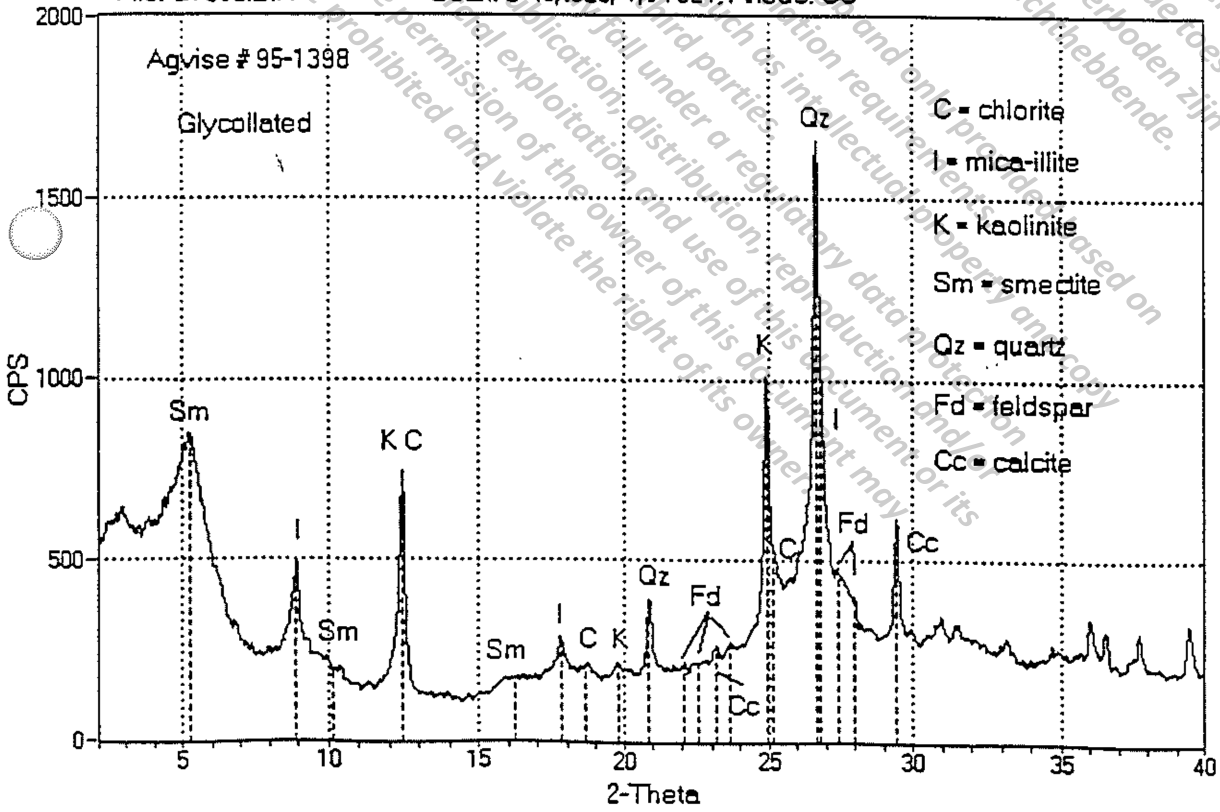

ID: Clay Sample #7558, Unit B, RH 73%, 6-27-95
File: 7558.MDI
Scan: 2-40/.025/1/#1521, Anode: CU
Agvise # 95-1394
I = mica-illite
K = kaolinite
Sm = smectite
Qz = quartz
Fd = feldspar
Gt = goethite
Sm
I
K
Fd
I
Qz
K
Gt
Fd
K
Fd
Qz
I
Fd
CPS
1000
750
500
250
0
5
10
15
20
25
30
35
40
2-Theta


ID: Glycollated Clay Sample #7558, Unit B, RH 66%
File: G7558.BIN
Scan: 2-40/.025/1/#1521, Anode: CU
Agvise # 95-1394
Glycollated
I = mica-illite
K = kaolinite
Sm = smectite
Qz = quartz
Fd = feldspar
Gt = goethite
Sm
I
Sm
K
Fd
I
K
Qz
Gt
Fd
K
Fd
Qz
I
Fd
CPS
1000
750
500
250
0
5
10
15
20
25
30
35
40
2-Theta

ID: Clay Sample #7562, Unit B, RH 70%, 7-4-95
File: 7562.BIN Scan: 2-40/.025/1/#1521, Anode: CU

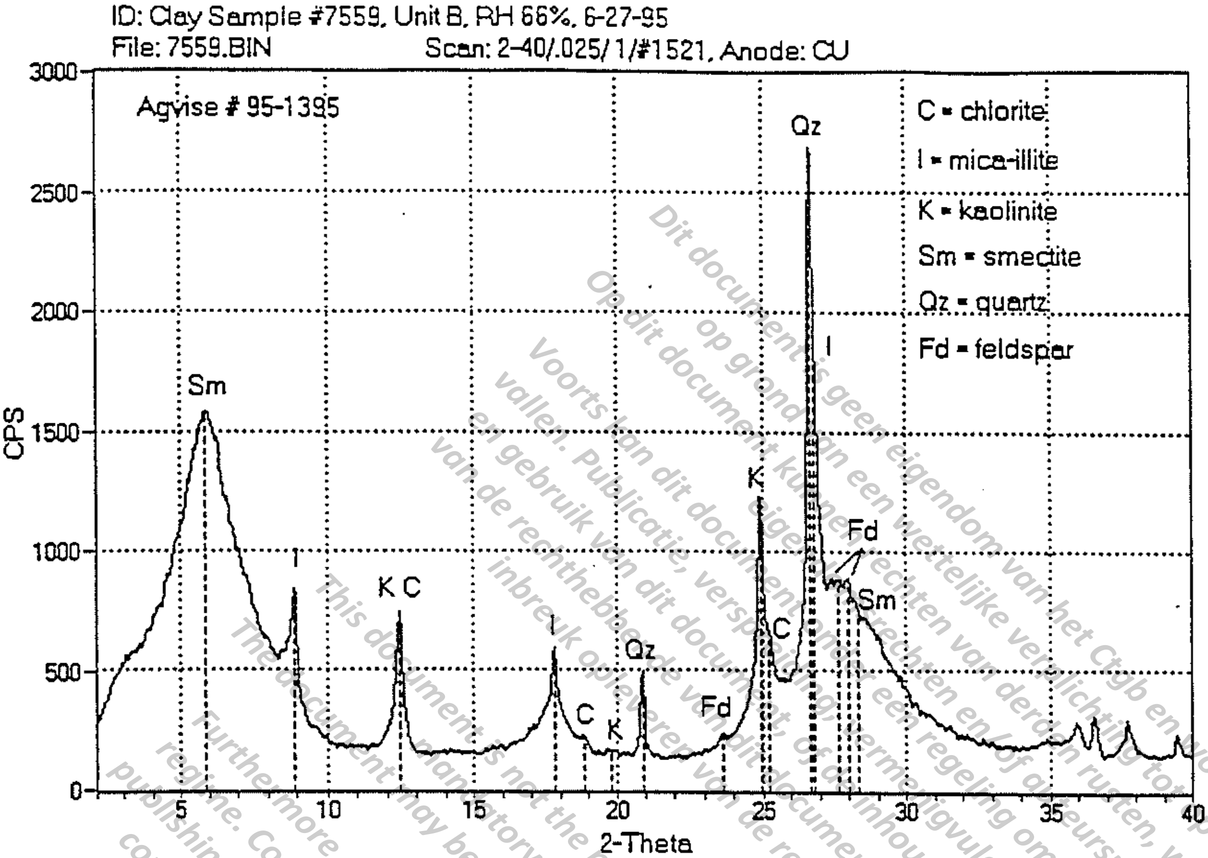


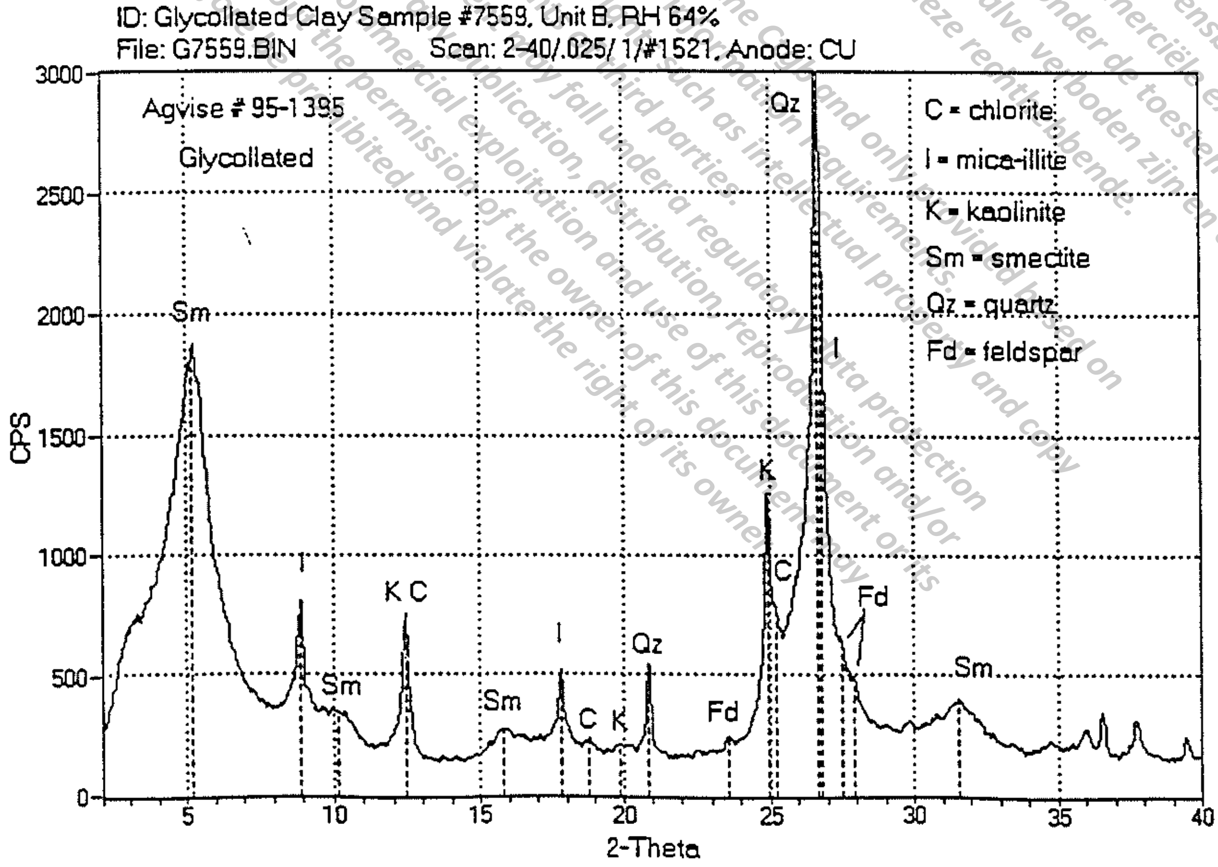
ID: Glycollated Clay Sample #7562, Unit B, RH 62%
File: G7562.BIN Scan: 2-40/.025/1/#1521, Anode: CU



```
====================== North Dakota State University ====================
Jade: Peak Listing
                                                    Fri Jul 21 1995 @5:58p
==========================================================================
File: 7562.BIN> Clay Sample #7562, Unit B, RH 70%, 7-4-95

----------- Scan Parameters: --------------------- Search Parameters: ---------
      Radiation   = CU_1.54059          ||   Filter length(pts)   = 9
      Scan Range = 2- 40                ||   Noise level(sigmas)  = 5.0
      Step Size   = .025                ||   Intensity cutoff(%)  = .5-100
      Count Time = 1 sec.               ||   2-Theta Zero (degs)  = 0
-------------------------------------------------------------------------------

      Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
      -------------   -----------------   -----------------------------
 #    2Theta      d      2Theta      d      Bkgrd   Peak    I%     Area    I%    FWHM*
-------------------------------------------------------------------------------------
 1:    6.084  14.5145    6.084  14.5145    439    275   23.7     461  100.0   1.509
 2:    8.895   9.9330    8.895   9.9330    320    231   19.9      60   13.0   0.234
 3:   12.432   7.1144   12.432   7.1144    114    698   60.2     242   52.5   0.312
 4:   17.793   4.9809   17.793   4.9809    140    179   15.4     172   37.3   0.865
 5:   18.650   4.7540   18.650   4.7540    151     75    6.5      38    8.2   0.456
 6:   19.826   4.4745   19.826   4.4745    157     33    2.8       8    1.7   0.218
 7:   20.897   4.2475   20.888   4.2494    159    231   19.9      54   11.7   0.2
 8:   22.044   4.0291   22.044   4.0291    170     23    2.0       2    0.4   0.0
 9:   22.491   3.9500   22.491   3.9500    176     13    1.1       1    0.2   0.100
10:   23.116   3.8445   23.116   3.8445    191     36    3.1       3    0.7   0.075
11:   23.571   3.7714   23.571   3.7714    207     51    4.4      11    2.4   0.194
12:   24.911   3.5715   24.911   3.5715    296    766   66.1     245   53.1   0.288
13:   25.127   3.5413   25.127   3.5413    307    248   21.4     103   22.3   0.374
14:   26.653   3.3419   26.653   3.3419    369   1159  100.0     400   86.8   0.311
15:   26.849   3.3179   26.850   3.3178    360    514   44.3     324   70.3   0.567
16:   27.450   3.2466   27.452   3.2463    360    215   18.6     310   67.2   1.298
17:   27.942   3.1906   27.942   3.1906    341    223   19.2     289   62.7   1.166
18:   29.438   3.0317   29.438   3.0317    349    363   31.3      70   15.2   0.174
-------------------------------------------------------------------------------------

=====================================================================================
File: G7562.BIN> Glycollated Clay Sample #7562, Unit B, RH 62%

      Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
      -------------   -----------------   -----------------------------
 #    2Theta      d      2Theta      d      Bkgrd   Peak    I%     Area    I%    FWHM*
-------------------------------------------------------------------------------------
 1:    5.215  16.9306    5.215  16.9306    423    425   32.2     560   74.5   1.186
 2:    8.925   9.9005    8.919   9.9070    201    293   22.2     177   23.5   0.5
 3:   10.132   8.7236   10.132   8.7236    181     16    1.2       1    0.1   0.0
 4:   12.411   7.1262   12.411   7.1262    126    620   47.0     192   25.5   0.279
 5:   16.230   5.4568   16.230   5.4568    156     20    1.5       1    0.1   0.045
 6:   17.790   4.9816   17.790   4.9816    163    123    9.3      67    8.9   0.490
 7:   18.671   4.7487   18.671   4.7487    167     40    3.0      24    3.2   0.540
 8:   19.792   4.4820   19.792   4.4820    175     47    3.6      15    2.0   0.287
 9:   20.897   4.2475   20.888   4.2494    159    231   17.5      54    7.2   0.210
10:   22.039   4.0300   22.039   4.0300    192     31    2.3       4    0.5   0.116
11:   22.507   3.9472   22.507   3.9472    206     15    1.1       1    0.1   0.060
12:   23.118   3.8443   23.118   3.8443    224     42    3.2       8    1.1   0.171
13:   23.645   3.7598   23.645   3.7598    250     25    1.9       1    0.1   0.036
14:   24.901   3.5728   24.908   3.5719    376    630   47.7     155   20.6   0.221
15:   25.146   3.5386   25.148   3.5383    394    144   10.9      44    5.9   0.275
16:   26.650   3.3422   26.656   3.3416    340   1320  100.0     752  100.0   0.513
17:   26.824   3.3209   26.825   3.3209    368    644   48.8     267   35.5   0.373
18:   27.400   3.2525   27.400   3.2524    343    141   10.7     132   17.6   0.843
19:   27.996   3.1845   27.996   3.1845    387     13    1.0       1    0.1   0.100
20:   29.424   3.0331   29.424   3.0331    279    337   25.5      86   11.4   0.230
-------------------------------------------------------------------------------------
   * Intensity values are based on counts per second.
```

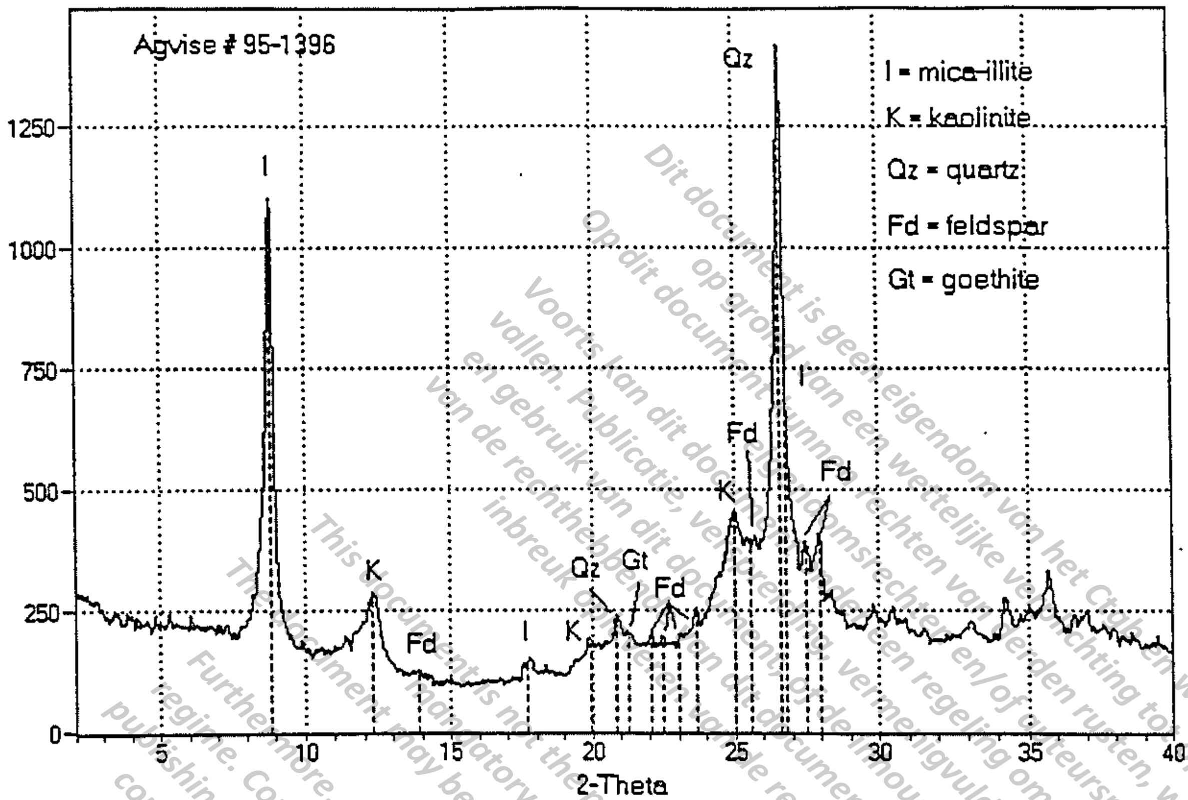
```
====================== North Dakota State University =====================
Jade: Peak Listing                                    Tue Jul 25 1995 @9:30pm
==========================================================================
File: 7558.MDI> Clay Sample #7558, Unit B, RH 73%, 6-27-95

----------- Scan Parameters: ------------------- Search Parameters: ---------
     Radiation  = CU_1.54059          ||   Filter length(pts)   = 9
     Scan Range = 2- 40               ||   Noise level(sigmas)  = 5.0
     Step Size  = .025                ||   Intensity cutoff(%)  = .5-100
     Count Time = 1 sec.              ||   2-Theta Zero (degs)  = 0
------------------------------------------------------------------------------

      Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
      -------------   -----------------   -----------------------------
  #   2Theta      d     2Theta      d     Bkgrd  Peak    I%    Area    I%   FWHM*
------------------------------------------------------------------------------
  1:   5.305 16.6463   5.302 16.6540    377   102  26.8     208  74.3  1.835
  2:   8.875  9.9560   8.874  9.9572    278   125  32.8      50  17.9  0.360
  3:  12.399  7.1330  12.394  7.1362    150    43  11.3      10   3.6  0.209
  4:  13.624  6.4944  13.637  6.4881    111    23   6.0       9   3.2  0.352
  5:  17.801  4.9787  17.807  4.9770    129    54  14.2     ·24   8.6  0.400
  6:  19.851  4.4689  19.851  4.4689    156    65  17.1      34  12.1  0.471
  7:  20.922  4.2426  20.925  4.2420    178    54  14.2      13   4.6  0.217
  8:  21.201  4.1874  21.203  4.1869    184    26   6.8       4   1.4  0.138
  9:  21.973  4.0419  21.967  4.0431    194    66  17.3      15   5.4  0.205
 10:  22.486  3.9509  22.486  3.9509    196    30   7.9       4   1.4  0.120
 11:  23.001  3.8636  22.999  3.8639    215    27   7.1       3   1.1  0.100
 12:  23.699  3.7513  23.696  3.7517    246    39  10.2       7   2.5  0.162
 13:  24.909  3.5717  24.909  3.5717    367    13   3.4       1   0.4  0.100
 14:  25.822  3.4475  25.822  3.4475    411    47  12.3       6   2.1  0.115
 15:  26.675  3.3391  26.675  3.3391    333   381 100.0     280 100.0  0.661
 16:  26.915  3.3099  26.915  3.3099    341   200  52.5     148  52.9  0.666
 17:  27.760  3.2111  27.760  3.2111    350   219  57.5     125  44.6  0.514
 18:  27.998  3.1843  27.998  3.1843    340   188  49.3      53  18.9  0.254
------------------------------------------------------------------------------

==============================================================================
File: G7558.BIN> Glycollated Clay Sample #7558, Unit B, RH 66%
      Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
      -------------   -----------------   -----------------------------
  #   2Theta      d     2Theta      d     Bkgrd  Peak    I%    Area    I%   FWHM*
------------------------------------------------------------------------------
  1:   5.086 17.3597   5.086 17.3597    286   105  25.9     166  47.0  1.423
  2:   8.883  9.9472   8.883  9.9472    203   100  24.7      48  13.6  0.432
  3:  10.249  8.6239  10.249  8.6239    206    24   5.9       2   0.6  0.075
  4:  12.309  7.1852  12.309  7.1852    151    12   3.0       1   0.3  0.100
  5:  13.656  6.4794  13.656  6.4794    109    21   5.2       8   2.3  0.343
  6:  17.743  4.9948  17.743  4.9948    139    33   8.1      11   3.1  0.300
  7:  19.896  4.4589  19.896  4.4589    206    43  10.6      16   4.5  0.335
  8:  20.855  4.2561  20.856  4.2558    207    59  14.6      15   4.2  0.229
  9:  21.227  4.1823  21.223  4.1830    219    47  11.6       8   2.3  0.153
 10:  21.988  4.0392  21.988  4.0392    236    93  23.0      21   5.9  0.203
 11:  22.654  3.9219  22.650  3.9226    242    25   6.2       5   1.4  0.180
 12:  23.052  3.8551  23.052  3.8551    253    16   4.0       1   0.3  0.056
 13:  23.812  3.7338  23.812  3.7338    278    32   7.9       6   1.7  0.169
 14:  25.041  3.5532  25.041  3.5532    443    14   3.5       1   0.3  0.100
 15:  25.725  3.4603  25.724  3.4604    334   204  50.4     353 100.0  1.557
 16:  26.646  3.3428  26.646  3.3428    324   405 100.0     295  83.6  0.656
 17:  26.846  3.3183  26.844  3.3185    348   202  49.9     109  30.9  0.486
 18:  27.751  3.2121  27.755  3.2116    349   150  37.0      35   9.9  0.210
 19:  28.002  3.1838  28.002  3.1838    334    95  23.5      20   5.7  0.189
------------------------------------------------------------------------------
   * Intensity values are based on counts per second.
```


ID: Clay Sample #7559, Unit B, RH 66%, 6-27-95
File: 7559.BIN
Scan: 2-40/.025/1/#1521, Anode: CU
Agvise # 95-1395
C = chlorite
I = mica-illite
K = kaolinite
Sm = smectite
Qz = quartz
Fd = feldspar
Sm
I
K C
I
C
K
Qz
Fd
K
C
Qz
I
Fd
Sm
CPS
3000
2500
2000
1500
1000
500
0
5
10
15
20
25
30
35
40
2-Theta

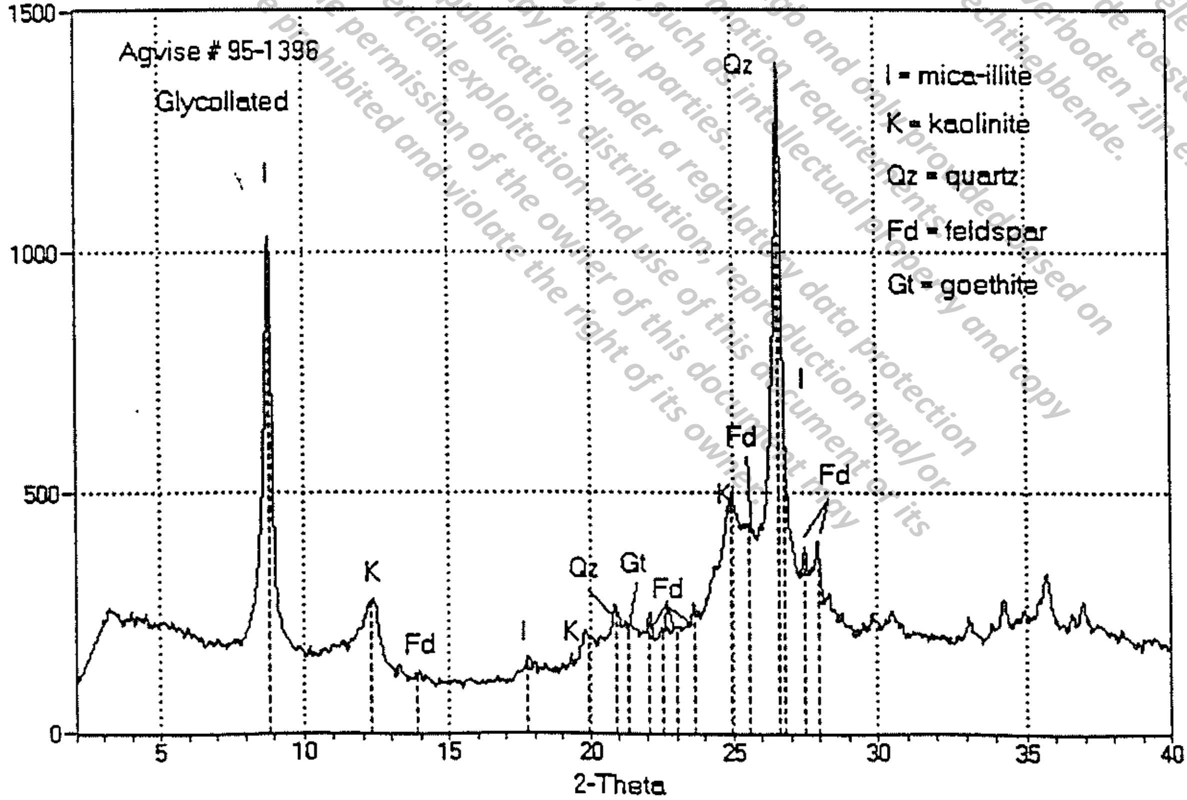

ID: Glycollated Clay Sample #7559, Unit B, RH 64%
File: G7559.BIN
Scan: 2-40/.025/1/#1521, Anode: CU
Agvise # 95-1395
Glycollated
C = chlorite
I = mica-illite
K = kaolinite
Sm = smectite
Qz = quartz
Fd = feldspar
Sm
I
Sm
K C
Sm
I
C
K
Qz
Fd
K
C
Qz
I
Fd
Sm
CPS
3000
2500
2000
1500
1000
500
0
5
10
15
20
25
30
35
40
2-Theta

```
======================== North Dakota State University =======================
Jade: Peak Listing
                                                     Fri Jul 21 1995 @3:06pr
=============================================================================
File: 7559.BIN> Clay Sample #7559, Unit B, RH 66%, 6-27-95

----------- Scan Parameters: --------------------- Search Parameters: ---------
     Radiation  = CU_1.54059       ||   Filter length(pts)   = 9
     Scan Range = 2- 40            ||   Noise level(sigmas) = 5.0
     Step Size  = .025             ||   Intensity cutoff(%) = .5-100
     Count Time = 1 sec.           ||   2-Theta Zero (degs) = 0
------------------------------------------------------------------------------

     Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
     -------------- ------------------- ---------------------------------
 #   2Theta     d     2Theta     d     Bkgrd   Peak    I%    Area    I%   FWHM*
------------------------------------------------------------------------------
 1:   5.903  14.9608   5.903  14.9608   657     919   42.6   1726 100.0  1.690
 2:   8.898   9.9307   8.898   9.9307   465     382   17.7     82   4.8  0.193
 3:  12.427   7.1169  12.427   7.1169   155     588   27.3    224  13.0  0.343
 4:  17.815   4.9747  17.815   4.9747   156     440   20.4    322  18.7  0.659
 5:  18.846   4.7050  18.846   4.7050   210      30    1.4      2   0.1  0.060
 6:  19.805   4.4792  19.805   4.4792   142      27    1.3     11   0.6  0.367
 7:  20.900   4.2469  20.900   4.2469   137     358   16.6     74   4.3  0.186
 8:  23.599   3.7670  23.594   3.7678   213      34    1.6      5   0.3  0.132
 9:  24.913   3.5712  24.913   3.5712   369     863   40.0    317  18.4  0.331
10:  25.224   3.5279  25.224   3.5278   403     246   11.4     76   4.4  0.278
11:  26.678   3.3387  26.678   3.3387   542    2155  100.0    800  46.3  0.334
12:  26.834   3.3197  26.834   3.3197   539    1283   59.5    639  37.0  0.448
13:  27.600   3.2293  27.599   3.2294   483     404   18.7    812  47.0  1.809
14:  27.950   3.1897  27.946   3.1901   451     440   20.4    775  44.9  1.585
15:  28.298   3.1512  28.292   3.1519   489     261   12.1    577  33.4  1.990


==============================================================================
File: G7559.BIN> Glycollated Clay Sample #7559, Unit B, RH 64%

     Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
     -------------- ------------------- ---------------------------------
 #   2Theta     d     2Theta     d     Bkgrd   Peak    I%    Area    I%   FWHM*
------------------------------------------------------------------------------
 1:   5.199  16.9848   5.199  16.9848   582    1293   42.9   1890  96.0  1.316
 2:   8.911   9.9156   8.911   9.9156   318     488   16.2    210  10.7  0.387
 3:  10.132   8.7236  10.132   8.7236   318      38    1.3     10   0.5  0.237
 4:  12.407   7.1282  12.407   7.1282   170     585   19.4    204  10.4  0.314
 5:  15.799   5.6046  15.802   5.6036   181      97    3.2     87   4.4  0.807
 6:  17.806   4.9774  17.806   4.9774   208     309   10.3    109   5.5  0.317
 7:  18.812   4.7135  18.812   4.7135   191      49    1.6     14   0.7  0.257
 8:  19.866   4.4657  19.866   4.4657   166      51    1.7     26   1.3  0.459
 9:  20.891   4.2488  20.891   4.2488   162     386   12.8    101   5.1  0.235
10:  23.562   3.7727  23.562   3.7727   213      34    1.1      5   0.3  0.132
11:  24.917   3.5706  24.917   3.5706   547     712   23.6    178   9.0  0.225
12:  25.221   3.5282  25.221   3.5282   610     159    5.3     35   1.8  0.198
13:  26.663   3.3407  26.663   3.3407   402    3014  100.0   1968 100.0  0.588
14:  26.824   3.3210  26.824   3.3209   507    1708   56.7    829  42.1  0.437
15:  27.498   3.2411  27.498   3.2411   638       5    0.2      1   0.1  0.100
16:  27.919   3.1932  27.919   3.1932   482      10    0.3      1   0.1  0.100
17:  31.600   2.8290  31.600   2.8291   244     167    5.5    189   9.6  1.019
------------------------------------------------------------------------------
 * Intensity values are based on counts per second.
```

ID: Clay Sample #7560, Unit B, RH 63%, 6-29-95
File: 7560.BIN Scan: 2-40/.025/1/#1521, Anode: CU

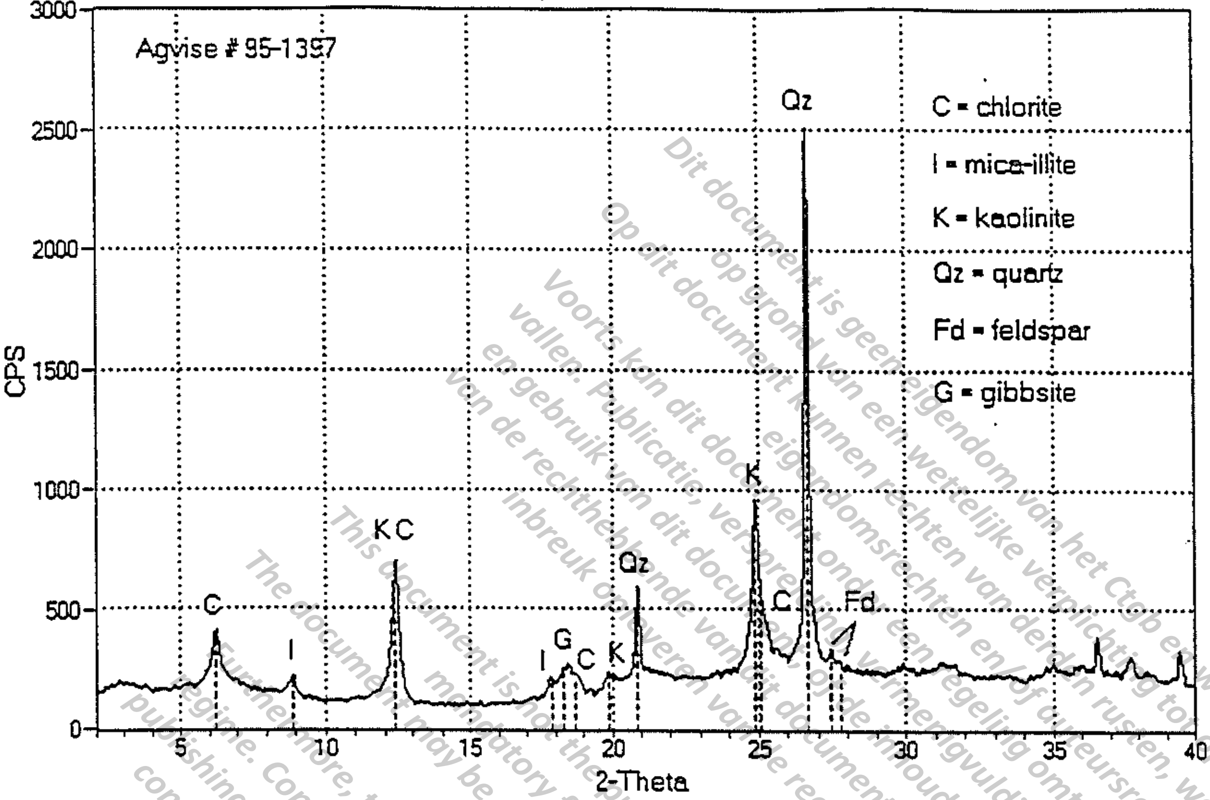


ID: Glycollated Clay Sample #7560, Unit B, RH 50%
File: G7560.BIN Scan: 2-40/.025/1/#1521, Anode: CU

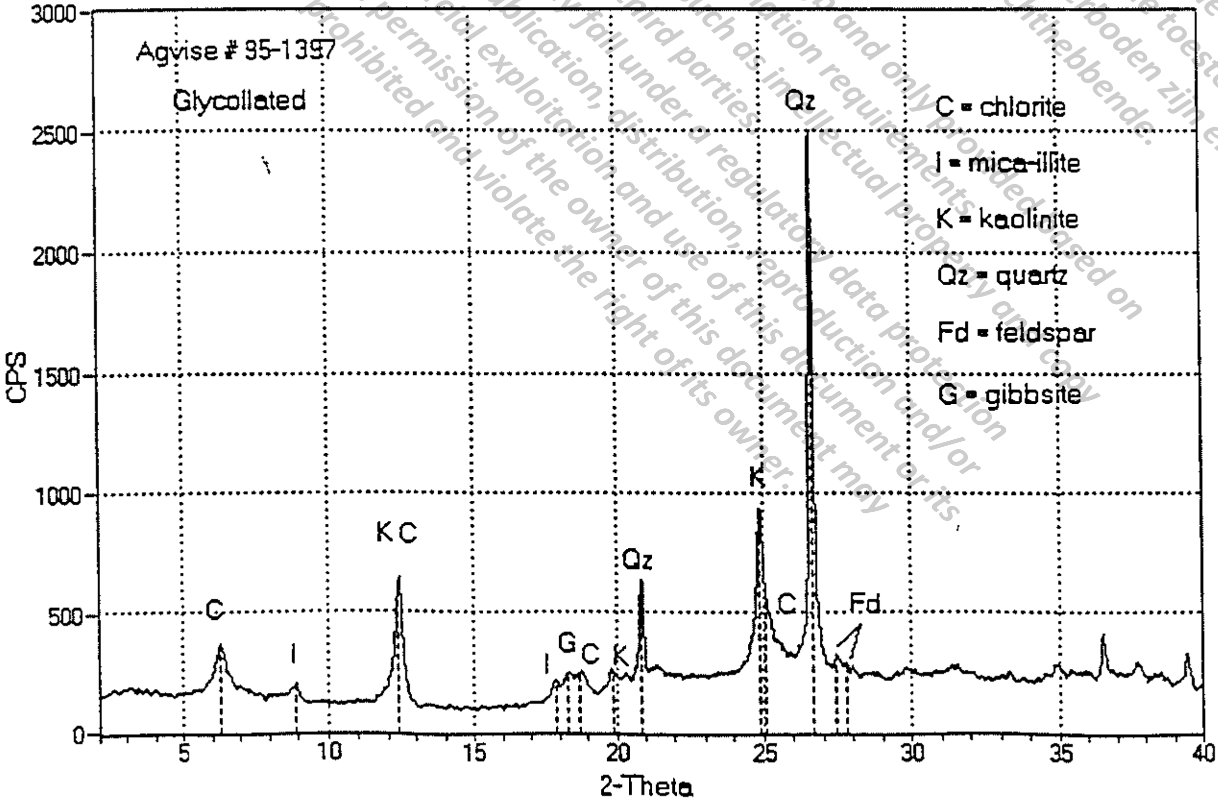


```
Jade: Peak Listing                                          Tue Jul 25 1995 @9:14pm
=====================================================================================
File: 7560.BIN> Clay Sample #7560, Unit B, RH 63%, 6-29-95

----------- Scan Parameters: --------------------- Search Parameters: ----------
      Radiation   = CU_1.54059           ||   Filter length(pts)   = 9
      Scan Range  = 2- 40                ||   Noise level(sigmas)  = 5.0
      Step Size   = .025                 ||   Intensity cutoff(%)  = .5-100
      Count Time  = 1 sec.               ||   2-Theta Zero (degs)  = 0
-------------------------------------------------------------------------------------

      Peak-Position    Centroid-Position    Peak & Area are without Bkgrd
      --------------   -----------------   -------------------------------
 #    2Theta      d      2Theta      d      Bkgrd   Peak   I%      Area    I%    FWHM*
-------------------------------------------------------------------------------------
 1:    8.831  10.0050     8.831  10.0050    172     929   82.7     403   75.6   0.390
 2:   12.312   7.1833    12.312   7.1833    132     153   13.6     118   22.1   0.694
 3:   13.905   6.3638    13.905   6.3638    108      20    1.8       2    0.4   0.090
 4:   17.697   5.0076    17.697   5.0076    109      34    3.0      14    2.6   0.371
 5:   19.948   4.4475    19.939   4.4495    151      42    3.7      17    3.2   0.364
 6:   20.896   4.2477    20.896   4.2477    154      87    7.7      51    9.6   0.528
 7:   21.284   4.1711    21.284   4.1711    168      32    2.8       8    1.5   0.225
 8:   22.050   4.0280    22.044   4.0291    176      37    3.3       5    0.9   0.122
 9:   22.446   3.9578    22.446   3.9578    177      22    2.0       2    0.4   0.082
10:   23.024   3.8597    23.024   3.8597    192      17    1.5       1    0.2   0.053
11:   23.597   3.7673    23.597   3.7673    216      37    3.3       5    0.9   0.122
12:   24.983   3.5614    24.983   3.5614    318     132   11.8      93   17.4   0.634
13:   25.581   3.4794    25.581   3.4794    276     128   11.4     331   62.1   2.327
14:   26.624   3.3455    26.624   3.3455    295    1123  100.0     533  100.0   0.427
15:   26.817   3.3218    26.817   3.3218    328     343   30.5     133   25.0   0.349
16:   27.499   3.2409    27.495   3.2414    320      73    6.5      18    3.4   0.222
17:   27.973   3.1871    27.956   3.1890    305     103    9.2      20    3.8   0.175


File: G7560.BIN> Glycollated Clay Sample #7560, Unit B, RH 50%

      Peak-Position    Centroid-Position    Peak & Area are without Bkgrd
      --------------   -----------------   -------------------------------
 #    2Theta      d      2Theta      d      Bkgrd   Peak   I%      Area    I%    FWHM*
-------------------------------------------------------------------------------------
 1:    8.826  10.0111     8.826  10.0111    166     867   79.7     367   69.8   0.381
 2:   12.308   7.1857    12.308   7.1857    131     141   13.0     111   21.1   0.709
 3:   13.896   6.3679    13.896   6.3679    106      18    1.7       4    0.8   0.200
 4:   17.761   4.9898    17.761   4.9898    106      52    4.8      20    3.8   0.346
 5:   19.942   4.4487    19.942   4.4487    166      35    3.2      12    2.3   0.309
 6:   20.931   4.2408    20.931   4.2408    161      96    8.8      85   16.2   0.797
 7:   21.367   4.1551    21.367   4.1551    175      46    4.2      28    5.3   0.548
 8:   22.049   4.0281    22.038   4.0301    189      58    5.3      10    1.9   0.155
 9:   22.548   3.9401    22.548   3.9401    196      21    1.9       2    0.4   0.086
10:   22.974   3.8680    22.974   3.8680    207      15    1.4       1    0.2   0.060
11:   23.629   3.7622    23.629   3.7622    230      39    3.6       5    1.0   0.115
12:   24.926   3.5693    24.926   3.5693    342     157   14.4     112   21.3   0.642
13:   25.548   3.4839    25.548   3.4839    282     159   14.6     375   71.3   2.123
14:   26.609   3.3473    26.609   3.3473    304    1088  100.0     526  100.0   0.435
15:   26.824   3.3210    26.824   3.3209    341     264   24.3      79   15.0   0.269
16:   27.499   3.2409    27.495   3.2414    320      73    6.7      18    3.4   0.222
17:   27.952   3.1895    27.952   3.1895    283     115   10.6      26    4.9   0.203
-------------------------------------------------------------------------------------
 * Intensity values are based on counts per second.
```

ID: Clay Sample #7561, Unit B, RH 53%, 6-29-95
File: 7561.BIN Scan: 2-40/.025/ 1/#1521, Anode: CU



ID: Glycollated Clay Sample #7561, Unit B, RH 42%
File: G7561.BIN Scan: 2-40/.025/ 1/#1521, Anode: CU



```
======================== North Dakota State University ======================
Jade: Peak Listing                                    Fri Jul 21 1995 @5:43p
=============================================================================
File: 7561.BIN> Clay Sample #7561, Unit B, RH 59%, 6-29-95

----------- Scan Parameters: -------------------- Search Parameters: ---------
     Radiation   = CU_1.54059      ||  Filter length(pts)   = 9
     Scan Range  = 2-.40           ||  Noise level(sigmas)  = 5.0
     Step Size   = .025            ||  Intensity cutoff(%)  = .5-100
     Count Time  = 1 sec.          ||  2-Theta Zero (degs)  = 0
-----------------------------------------------------------------------------

     Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
     -------------   -----------------   -----------------------------
 #   2Theta     d     2Theta     d       Bkgrd   Peak    I%     Area    I%    FWHM*
-----------------------------------------------------------------------------------
 1:   6.233  14.1693   6.233  14.1693     154     259   11.6     151   30.6   0.525
 2:   8.899   9.9293   8.899   9.9293     130      94    4.2      29    5.9   0.278
 3:  12.389   7.1388  12.389   7.1388     102     598   26.7     239   48.5   0.360
 4:  17.867   4.9605  17.867   4.9605     123      94    4.2      37    7.5   0.354
 5:  18.335   4.8349  18.335   4.8349     126     128    5.7     127   25.8   0.893
 6:  18.738   4.7318  18.738   4.7318     132      94    4.2      45    9.1   0.431
 7:  19.892   4.4599  19.892   4.4599     171      70    3.1      20    4.1   0.257
 8:  20.883   4.2504  20.883   4.2504     176     417   18.6     171   34.7   0.369
 9:  24.894   3.5739  24.894   3.5739     256     705   31.5     280   56.8   0.357
10:  25.061   3.5504  25.061   3.5504     261     339   15.1     154   31.2   0.409
11:  26.648   3.3425  26.648   3.3425     274    2240  100.0     493  100.0   0.198
12:  27.450   3.2467  27.450   3.2467     257      80    3.6      30    6.1   0.337
13:  27.750   3.2121  27.750   3.2121     278       8    0.4       1    0.2   0.100
-----------------------------------------------------------------------------------


=============================================================================
File: G7561.BIN> Glycollated Clay Sample #7561, Unit B, RH 42%

     Peak-Position   Centroid-Position   Peak & Area are without Bkgrd
     -------------   -----------------   -----------------------------
 #   2Theta     d     2Theta     d       Bkgrd   Peak    I%     Area    I%    FWHM*
-----------------------------------------------------------------------------------
 1:   6.295  14.0291   6.295  14.0291     143     229   10.2     148   30.0   0.582
 2:   8.912   9.9151   8.912   9.9151     123      81    3.6      29    5.9   0.322
 3:  12.391   7.1375  12.391   7.1375     104     540   24.1     226   45.8   0.377
 4:  17.857   4.9633  17.857   4.9633     123      94    4.2      37    7.5   0.354
 5:  18.335   4.8349  18.335   4.8349     126     128    5.7     127   25.8   0.893
 6:  18.752   4.7282  18.747   4.7295     150     101    4.5      68   13.8   0.606
 7:  19.892   4.4599  19.892   4.4599     189      74    3.3      22    4.5   0.268
 8:  20.874   4.2523  20.870   4.2530     193     443   19.8     157   31.8   0.319
 9:  24.898   3.5732  24.897   3.5735     284     648   28.9     274   55.6   0.381
10:  25.074   3.5486  25.075   3.5485     266     305   13.6     214   43.4   0.631
11:  26.642   3.3432  26.642   3.3432     274    2240  100.0     493  100.0   0.198
12:  27.460   3.2455  27.460   3.2455     257      80    3.6      30    6.1   0.338
13:  27.755   3.2116  27.755   3.2116     261      39    1.7       5    1.0   0.115
-----------------------------------------------------------------------------------
  * Intensity values are based on counts per second.
```

Sharkey *series*

The Sharkey series consists of level to gently sloping, poorly drained, clayey soils that formed in fine-textured sediments of the slack-water areas along the Mississippi River. The surface soil is a dark grayish-brown clay, which overlies a very dark gray to dark gray, clayey subsoil. The Sharkey soils shrink when dry and form cracks from 1 to 5 inches wide and several feet deep. The Sharkey soils are medium acid to neutral.

Sharkey soils are associated with the Tunica, Alligator, Mhoon, and Dowling soils, mainly in the western part of the county. They occupy about one-third of the county. Sharkey soils differ from the Tunica soils in being finer textured to a greater depth and in being more poorly drained. They are darker colored than the Alligator soils. They have deep, clayey profiles; whereas the Mhoon soils are stratified with coarser textured material. They differ from the Dowling soils in being level to gently sloping rather than depressional.

The native vegetation on the Sharkey soils consists of American elm, sugarberry, bitter pecan, green ash, willow oak, Nuttall oak, overcup oak, other oaks and gums, and an undergrowth of vines and canes. More than half of the total acreage has been cleared and is planted to cotton, soybeans, and small grains. Because of poor drainage and clayey texture, use of the Sharkey soils for farming is limited.

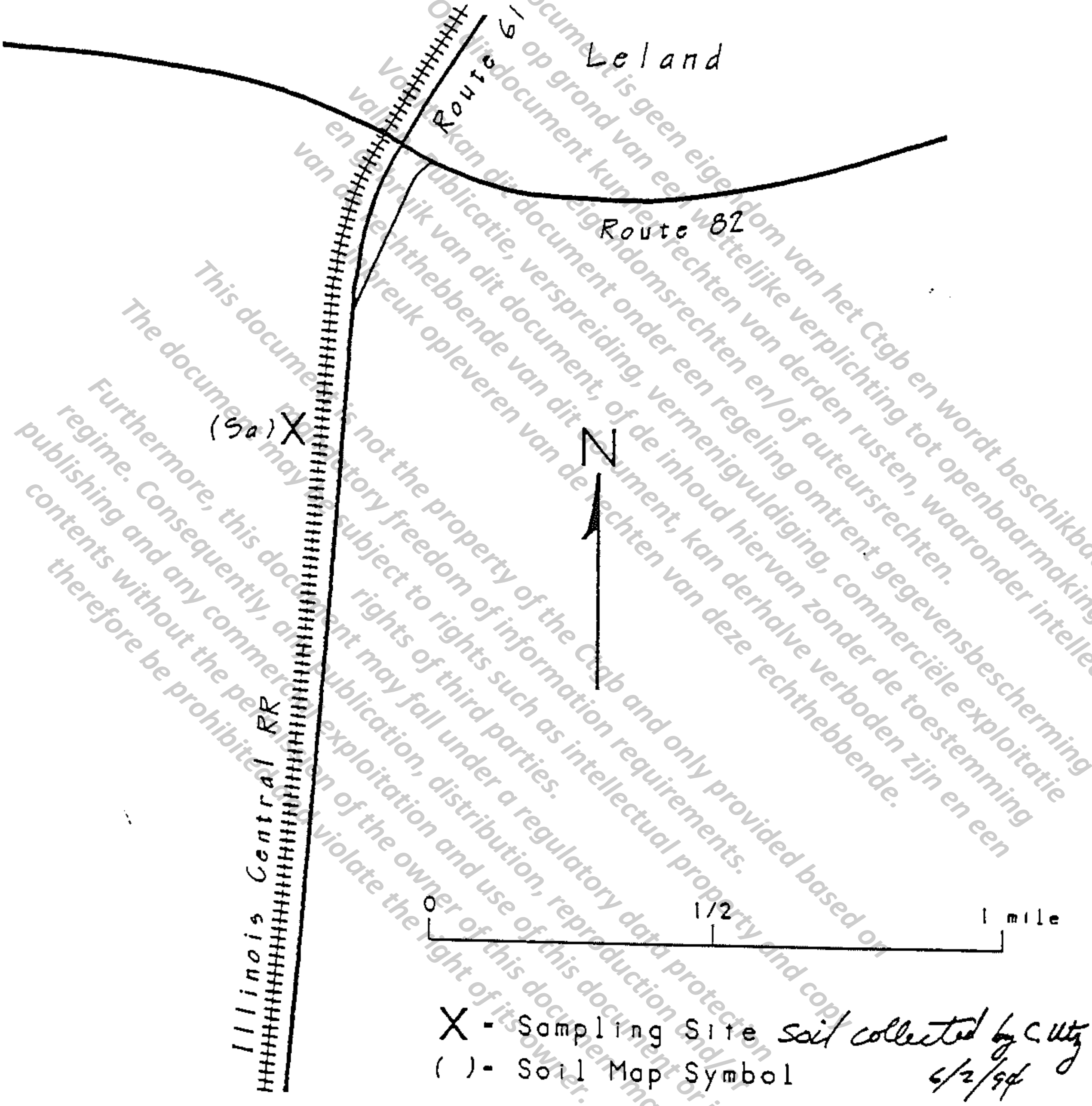
Sharkey clay, level phase (0 to ½ percent slopes) (**Sa**).—This soil is on broad flats or in slightly depressed areas. Water from the surrounding higher areas collects and ponds on it. Infiltration and the rate of runoff are very slow.

Present use and management.—This is the second most extensive soil of the Sharkey series. Most of the acreage is now wooded. If the soil is adequately drained, cotton, rice, soybeans, grasses, and legumes can be grown. Drainage can be provided by row arrangement, by V-type and W-type ditches, and by adequate outlets to remove water. Capability unit 19 (IIIw-11).

SOURCE: USDA SCS Soil Survey Map 1961

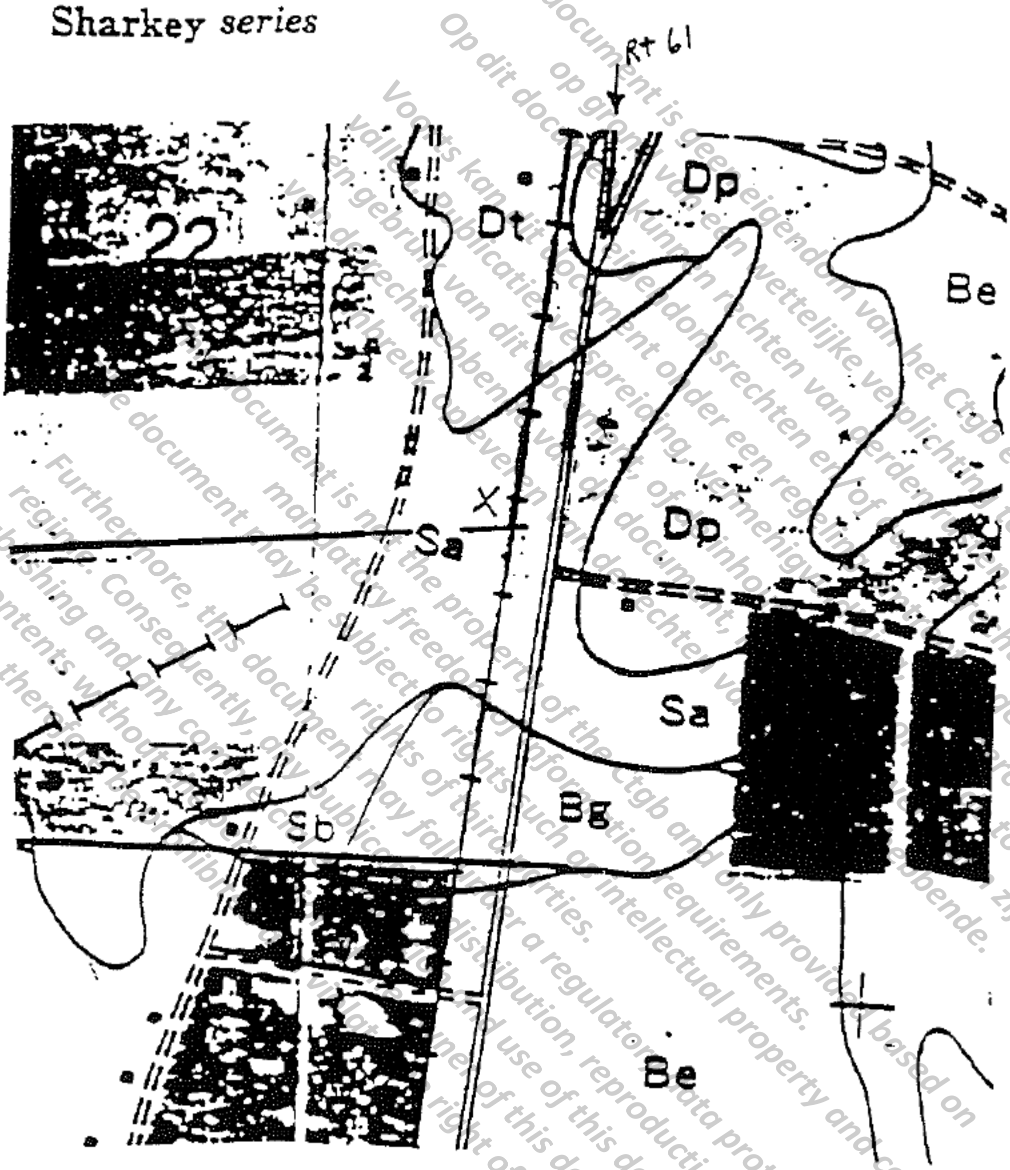
Route 61
Leland, MS

Clay Soil
Sharkey Series
Washington County, MS



soil collected by C. Utz
6/2/94

Sharkey *series*



Source: USDA SCS Soil Survey Map 1961
X = Sampling site soil collected 6/2/94 by C. Litz.

[redacted]

AGRISEARCH INCORPORATED
[✓ SOIL]/SEDIMENT/WATER COLLECTION

COLLECTION

TYPE Sand Soil

Date Collected 6/8/95 8PM

SERIES Sassafras

Collected By [redacted]

SOURCE

Collection Site Riding Stable Road

City Burtonsville

County Montgomery State MD

Depth 0-6"

Horizon A

Quantity Collected 25 ([✓ pounds], gallons)

RECEIPT

LAB NUMBER S-95-013

STORAGE Room Temp. [✓ Refrigeration] Freezer (Circle One)

Date 6/9/95 8:30AM (overnight on truck) Initials [redacted]

SIEVED [✓ Yes] No (Circle one) Sieve Size 2 (mesh, [✓ mm])

Date 6/9/95 Initials [redacted]

AGI-L153-11-9-94

Sassafras series

The Sassafras series consists of deep, well-drained, moderately coarse textured to medium textured soils that developed from old outwash material and probably some silty material deposited by wind. These soils are underlain by sands and clayey sands that grade at some depth to waterworn gravel. They are in the eastern part of the county on the Coastal Plain and generally are associated with the Chillum, Beltsville, and Rumford soils.

Profile of Sassafras loam, 3 to 8 percent slopes, moderately eroded, in a forest of oak, maple, dogwood, and hickory, on the horticultural farm of the University of Maryland on Cherry Hill Road:

Surface soil—

A_{00} 2 to ½ inch, loose hardwood leaves.

A_0 ½ to 0 inch, very thin layer of fibrous organic matter; lacking in many places.

A_1 0 to ½ inch, very dark gray (N 3/0) loam; moderate, fine and medium, crumb structure; very friable when moist; very uneven in thickness, filling vertical cracks and large root channels to a depth of about 2 inches; strongly acid; abrupt, irregular boundary.

A_{21} ½ to 3 inches, yellowish-brown (10YR 5/4), heavy loam to light silt loam; moderate, medium, granular structure; very friable when moist; roots abundant; very porous; wormholes abundant; strongly acid; diffuse, smooth boundary.

A_{22} 3 to 7 inches, yellowish-brown (10YR 5/4), heavy loam to light silt loam; moderate, medium, subangular blocky structure; friable when moist; roots abundant; porous; a few rounded gravel fragments; strongly acid; clear, smooth boundary.

Subsoil—

B_1 7 to 13 inches, dark yellowish-brown (10YR 4/4) silt loam; moderate, medium and coarse, subangular blocky structure; friable when removed moist, but firm in place; roots fairly plentiful; moderately porous; strongly acid; clear, smooth boundary.

B_{21} 13 to 22 inches, dark-brown (7.5YR 4/4) silty clay loam; moderate to strong, medium and coarse, subangular blocky structure; slightly firmer than B_1 horizon but friable when removed moist; roots rather few; some worm channels; very strongly acid; gradual, smooth boundary.

B_{22} 22 to 30 inches, dark-brown (7.5YR 4/4) silty clay loam; moderate, coarse, blocky structure; firm in place, friable when removed moist; some fine roots; no worm channels; abundant clayskins; very strongly acid; very abrupt, smooth boundary.

Substratum—

D_1 30 to 40 inches, variegated yellowish-brown and dark-brown (10YR 5/6 and 4/3), gritty sandy clay loam; dark yellowish brown (10YR 4/4) when crushed; moderate, medium and coarse, subangular blocky structure; slightly cemented; firm when moist and very hard when dry; many cracks that have clayskins on surfaces; very strongly acid; gradual, wavy boundary.

D_2 40 to 50 inches, strong-brown (7.5YR 5/6), gritty sandy clay loam, that grades with depth to loose coarse sand; very weak, coarse, blocky structure; a few thin clayskins; very strongly acid; gradual, wavy boundary.

D_3 50 to 66 inches, dark-brown (7.5YR 4/4), slightly clayey coarse sand; loose; structureless.

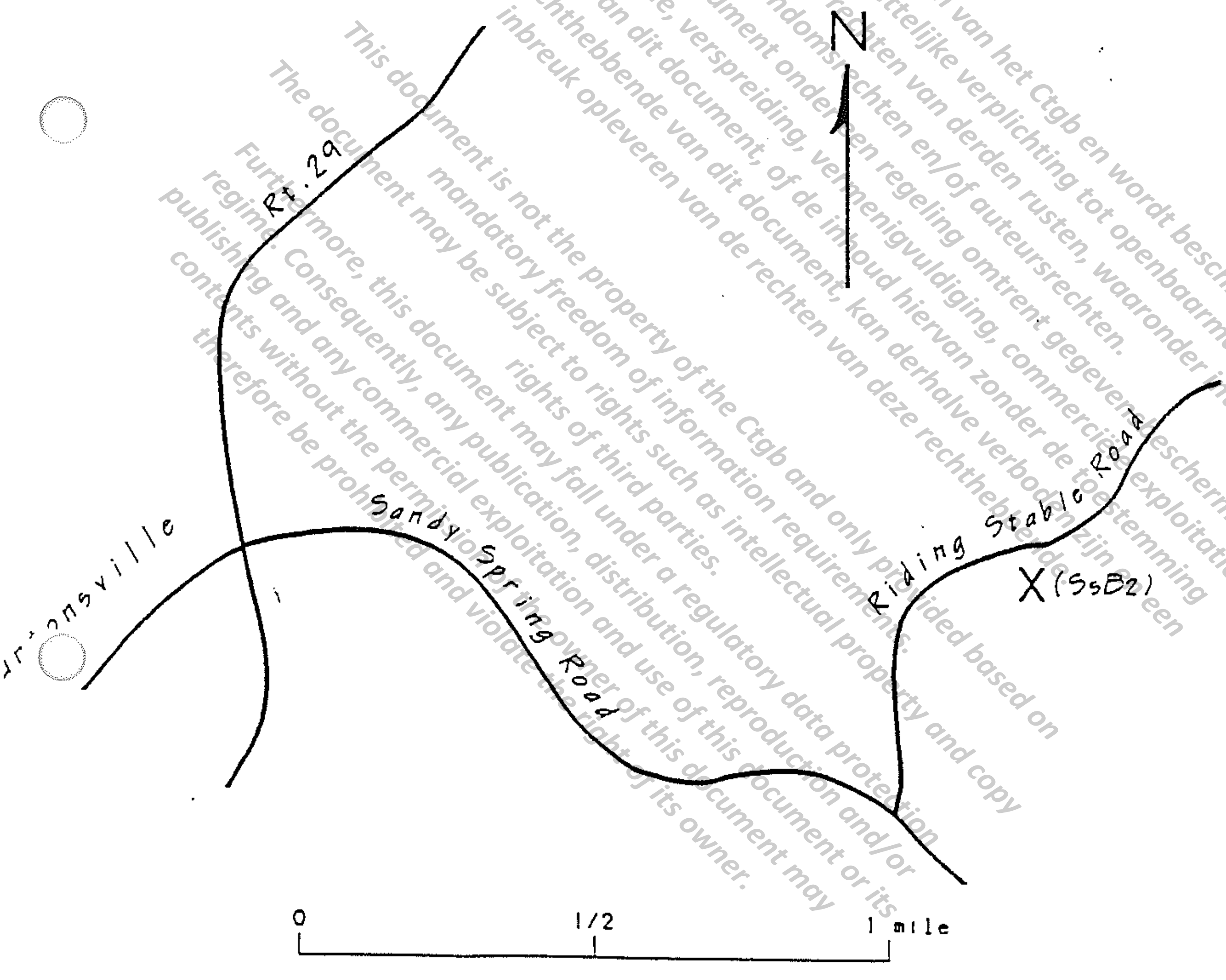
D_4 66 to 72 inches +, brownish-yellow (10YR 6/4), coarse sand that grades to coarse, waterworn gravel.

Sassafras sandy loam, 3 to 8 percent slopes, moderately eroded (Ss32).—The profile of this soil is like that described in the representative profile, except that it is sandy to the depth of the plow layer. Because of sandiness and the hazard of further erosion, this soil is in capability unit IIe-5.

Source: USDA SCS Soil Survey Map 1987

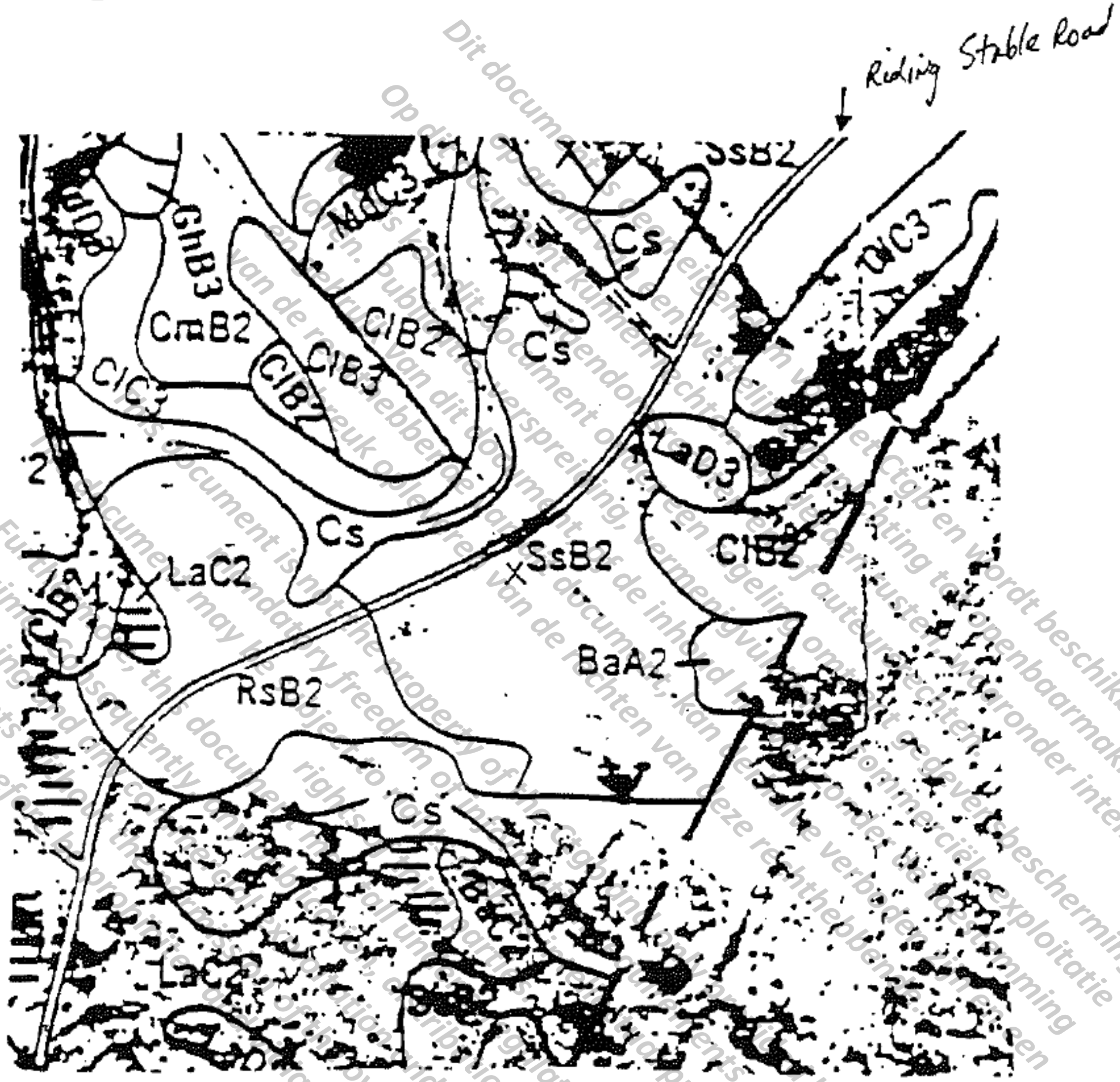
Warfield/Spring Lake Farm
15620 Riding Stable Road
Burtonsville, MD

Sand Soil
Sassafras Series
Montgomery County, MD



X - Sampling Site Soil collected 6/8/95
( )- Soil Map Symbol

Sassafras series



Source: USDA SCS Soil Survey Map 1987

X = Sampling site soil collected 6/8/95

5.1.2.e Woo

CHAIN OF CUSTODY FORM

TO BE COMPLETED BY THE SENDER

Sponsor Study No.: 248-94 Contract Lab Study No.: 12213 /63 5-6/94

Date/Time Sampled 5/30/94 13:30pm Sample Type: Soil

Description of Sampling Procedure:
20lb of sample Taken from a field at
12413 Wide Hollow Road, Yakima, WA 98908
Sampled from 0-6 inches

Sampled By [redacted] [redacted]
Signature Printed or Typed Name

Date/Time Stored: / Stored By:
Storage Conditions:

Shipped/Transported By:
Signature Printed or Typed Name
Company Name & Address:

Was ice/dry ice used? Y/ [✓ N]. If Y. ice/ dry ice and amount used lbs.

Name of Commercial Shipper: Federal Express
Waybill No.:

Date Shipped/Transported: 5/30/94 Number of Samples: 1
Shipped/Transported To (Name & Address): [redacted]
Agrisearch Incorporated
5734 Industry Lane
Frederick, MD 21701

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT
DATE 9/7/94
BY [redacted]

****

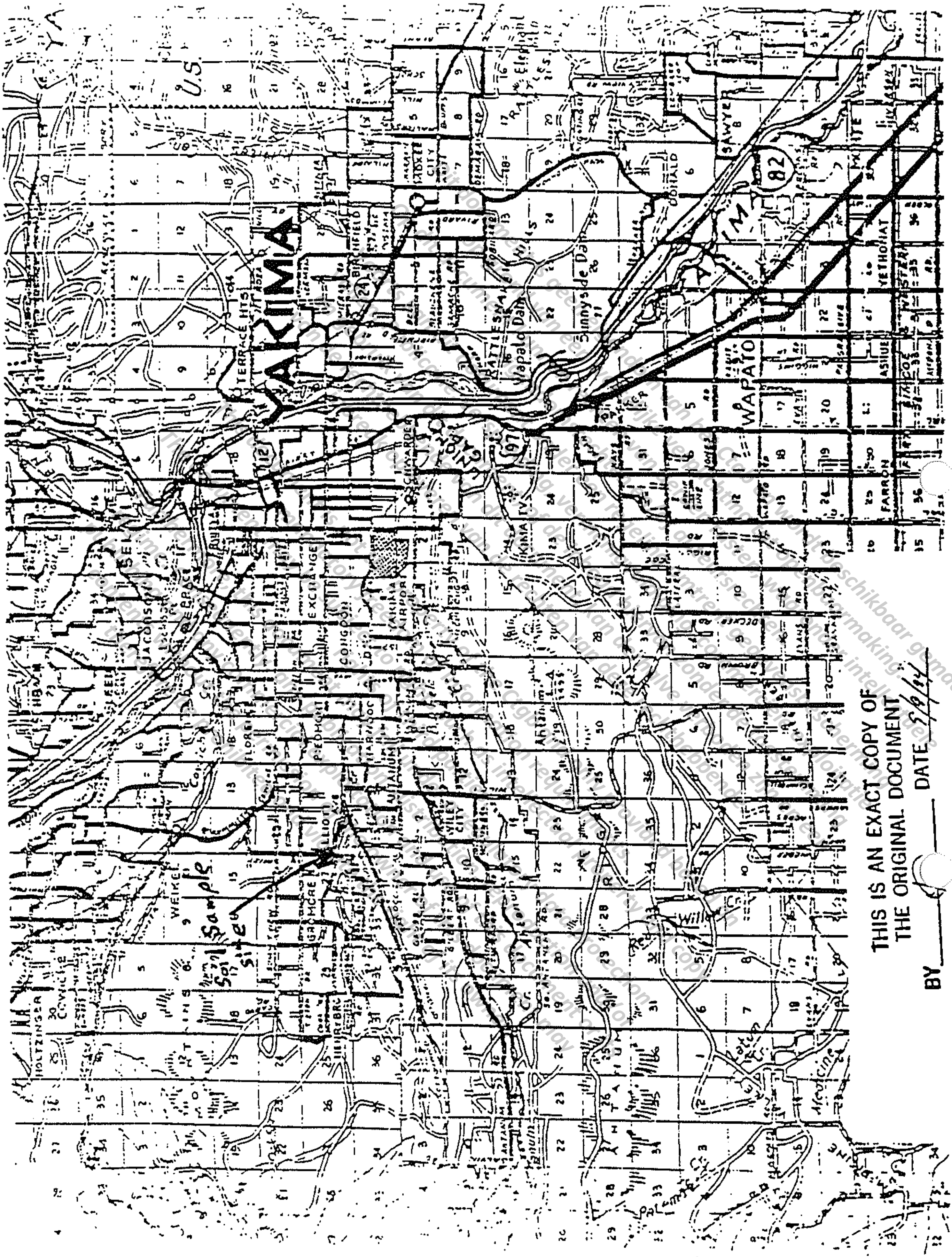
TO BE COMPLETED BY THE RECEIVER

Received By: [redacted] [redacted]
Signature Printed or Typed Name

Company Name & Address: AGRISEARCH INCORPORATED
5734 Industry Lane
Frederick, MD 21701

Date Received: 6/1/94
Condition Received:
good

Please maintain a copy of this form through all phases of the corresponding study which utilizes this sample matrix. Please return a verified photocopy of this form to the sender.



THIS IS AN EXACT COPY OF
THE ORIGINAL DOCUMENT
BY ______ DATE 5/9/84

5.1.2.e Woo

Soil Sample

History of field where sample was taken:

1987 to present – garden

prior to 1987 – grass and alfalfa hay

There are Commercial orchards 0.5 mile to the West and 0.25 mile to the north of the sample site.

I do not think any pesticides have been applied to this site since 1987 and probably not before that.

THIS IS AN EXACT COPY OF
THE ORIGINAL DOCUMENT
BY [redacted] DATE 9/9/94

USDA Soil Series
Harwood loam series
per [redacted] 11/15/95

AGRISEARCH INCORPORATED
[✓ SOIL]/SEDIMENT/WATER COLLECTION

COLLECTION

TYPE Soil – sandy loam Date Collected 5/22/95

SERIES Hanford [redacted] 11/14/95 Collected By Ciba Geigy ([redacted])

SOURCE

Collection Site Ciba Crop Protection's Western Research Station

City Sanger

County Fresno State Ca.

Depth 0-6"

Horizon A

Quantity ~~Collected~~ ~70 Kg. (pounds, gallons)

(S) Rec'd 10/6/95

Fed. Exp. 005 5317 135

RECEIPT

LAB NUMBER S- 95-012

STORAGE Room Temp. [✓ Refrigeration] Freezer
(Circle One)

Date 5/24/95 Initials [redacted]

SIEVED [✓ Yes] No (Circle one) - Sieve Size 2 mm (mesh, mm)

Date 5-24-95 Initials [redacted]

AGI-L153-11-9-94

Ciba Plant Protection

ciba

Ciba-Geigy Corporation
P.O. Box 158
17915 E. Annadale
Sanger CA 93654
(209) 875-6075

May 22, 1995

[redacted]
Agri Search Inc.
5734 Industry Lane
Frederick, MD 21701

Dear Mr. [redacted]

The attached soil sample was collected at Ciba Crop Protection's Western Research Station. A map of the research station and a soil analysis for soil collected in the adjacent area is included for your general information.

If you need any additional information concerning this soil sample, please feel free to give me a call.

Sincerely,

[redacted]

[redacted] Ph.D.
[redacted]

cc: [redacted]

Ciba Plant Protection

ciba

Ciba-Geigy Corporation
P.O. Box 158
17915 E. Annadale
Sanger, CA 93654
(209) 875-6075

Chain of Custody

A soil sample (0-6” sandy loam) weighing approximately 7.0 kg was collected on May 22, 1995 from Ciba Crop Protection's Western Research Station, Sanger, CA

The soil sample was shipped to Bill Stare of Agri Search Inc., by Federal Express on May 22, 1995.

Shipper: Name: [redacted] (printed)

Signature:

Date Shipped: 5-22-95

Recipient: Name: [redacted] (printed)

Signature:

Company: Agrisearch Inc.

Date Received: 5/24/95

CHAIN OF CUSTODY
AGRISEARCH INCORPORATED
5734 INDUSTRY LANE
FREDERICK, MD 21701

Project No.: NA Sample Type: Soil

Time Sampled: 8:00 AM By: [redacted] Date: 6/7/95

Date Stored: NA By: Condition: NA

Date Shipped: 6/7/95 No. Samples Shipped: 1

Shipped To: (Name & Address): Agrisearch Incorporated
5734 Industry Lane
Frederick, MD 21701
(301) 662-2203
ATTENTION: [redacted]

Shipped By: [redacted]
Signature Print Name

Company Name: Univ. of Ariz / Yuma Agri Center

Address: 6425 W 8th St Yuma AZ 85364

Phone: 520 782 3836

Amt. Dry Ice/Container: NA Commercial Shipper: Federal Express

Waybill No.: 9524258144

Received By: [redacted]
Signature Print Name

Company Name: Agrisearch Incorporated

Address: 5734 Industry Lane, Frederick, MD 21701

Date Received: 6·8·95 Condition: good

Please maintain a copy of this form through all phases of the study. Return completed form to sender.

AGI-L036-9/28/94

AGRISEARCH INCORPORATED
[✓ SOIL]/SEDIMENT/WATER COLLECTION

COLLECTION

TYPE Soil

Date Collected 6/7/95

SERIES Gadsden

Collected By 5.1.2.e Woo

SOURCE

Collection Site Univ. of Ariz / Yuma Agri. Center
6425 W 8th St. / Section 23 Township 8S Range 24W
UTM Zone 11, 715422E 36226004 N
using GPS

City Yuma

County Yuma

State AZ

Depth 0-6"

Horizon A

Quantity Collected 30 (pounds, gallons)

RECEIPT

LAB NUMBER S-95-014

STORAGE Room Temp. [✓ Refrigeration] Freezer
~~(Circle One)~~

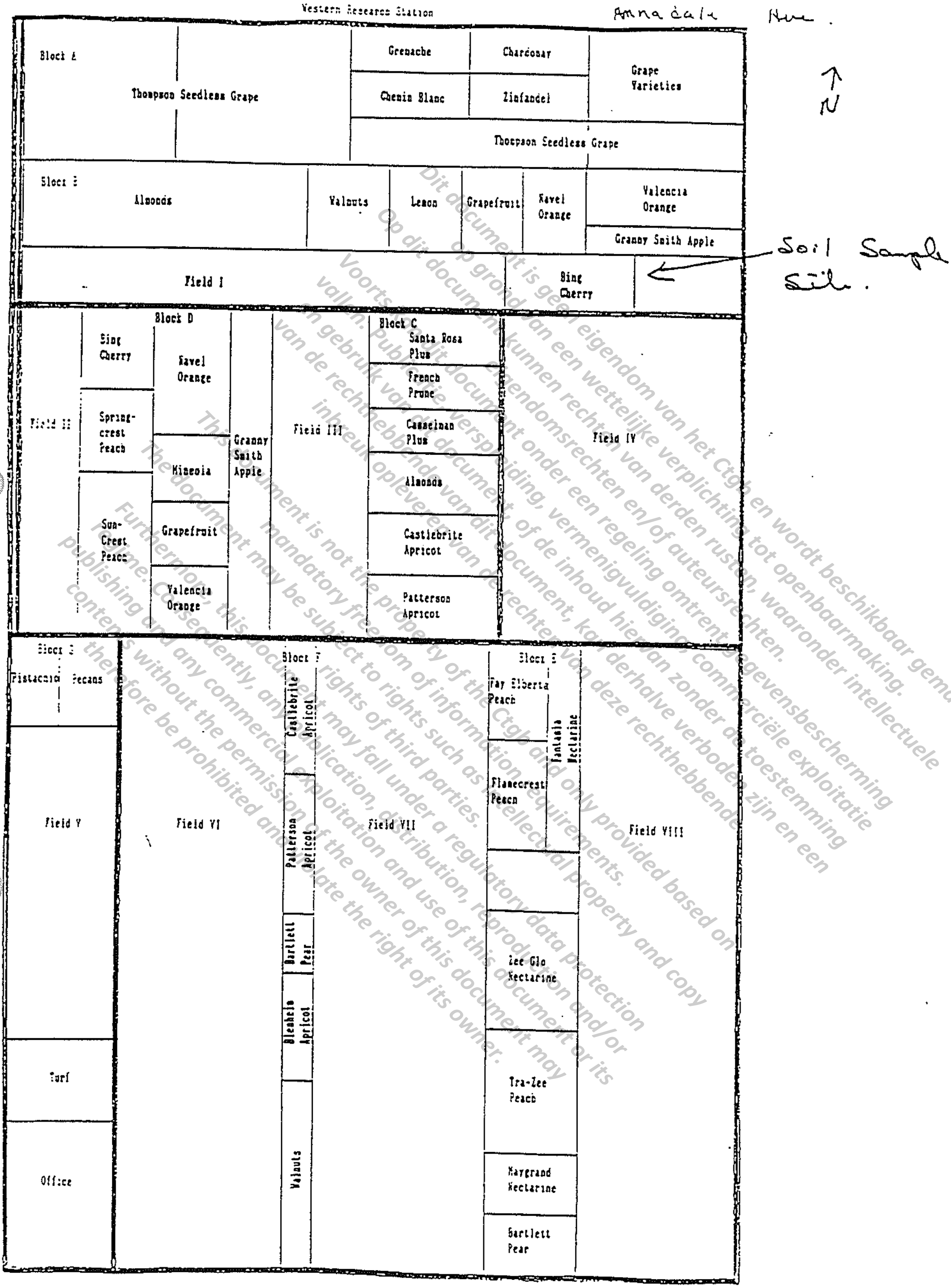
Date 6/8/95 Initials [redacted]

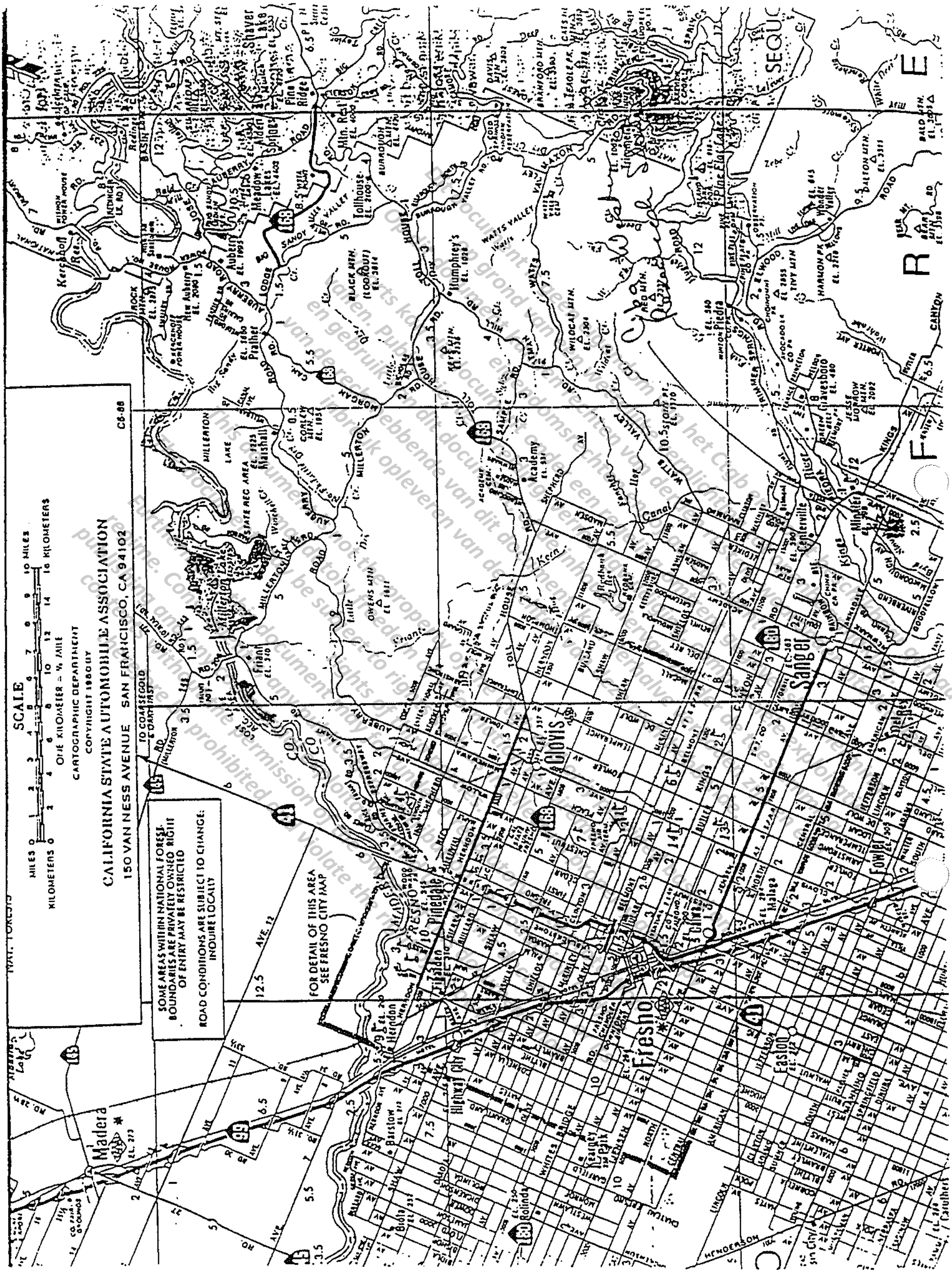
SIEVED [✓ Yes] No
(Circle one)

Sieve Size 2 (mesh, [✓ mm])

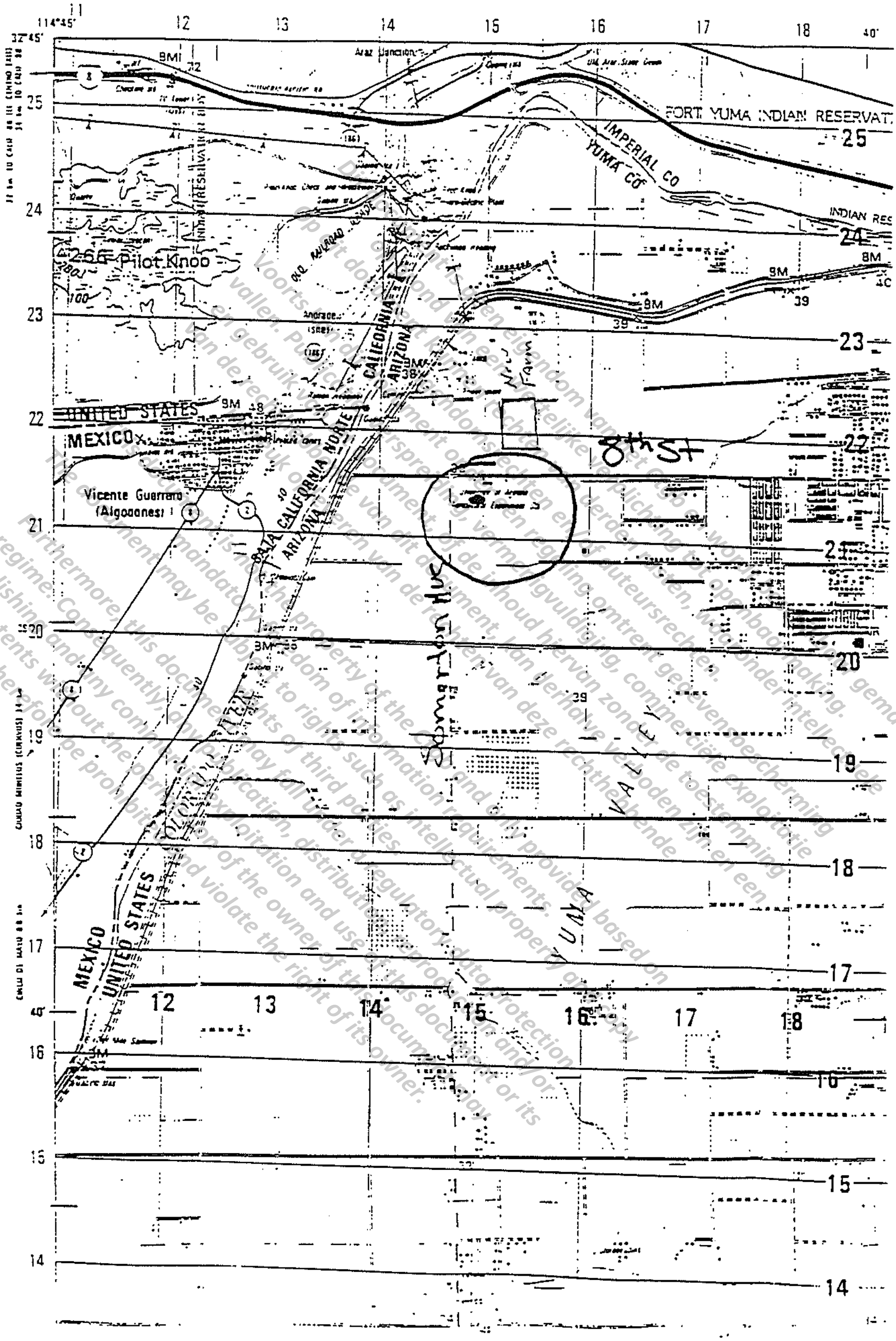
Date 6/8/95 Initials [redacted]

AGI-L153-11-9-94


Western Research Station
Annadale Ave.
N
Block A
Thompson Seedless Grape
Grenache
Chardonay
Chenin Blanc
Zinfandel
Grape Varieties
Thompson Seedless Grape
Block B
Almonds
Walnuts
Lemon
Grapefruit
Navel Orange
Valencia Orange
Granny Smith Apple
Field I
Bing Cherry
Soil Sample Site.
Field II
Bing Cherry
Spring-crest Peach
Sun-Crest Peach
Block D
Navel Orange
Mineola
Grapefruit
Valencia Orange
Granny Smith Apple
Field III
Block C
Santa Rosa Plum
French Prune
Casselman Plum
Almonds
Castlebrite Apricot
Patterson Apricot
Field IV
Block E
Pistachio
Pecans
Field V
Turf
Office
Field VI
Block F
Castlebrite Apricot
Patterson Apricot
Bartlett Pear
Blenheim Apricot
Walnuts
Field VII
Block G
Fay Elberta Peach
Fantasia Nectarine
Flamecrest Peach
Lee Glo Nectarine
Tra-Zee Peach
Maygrand Nectarine
Bartlett Pear
Field VIII

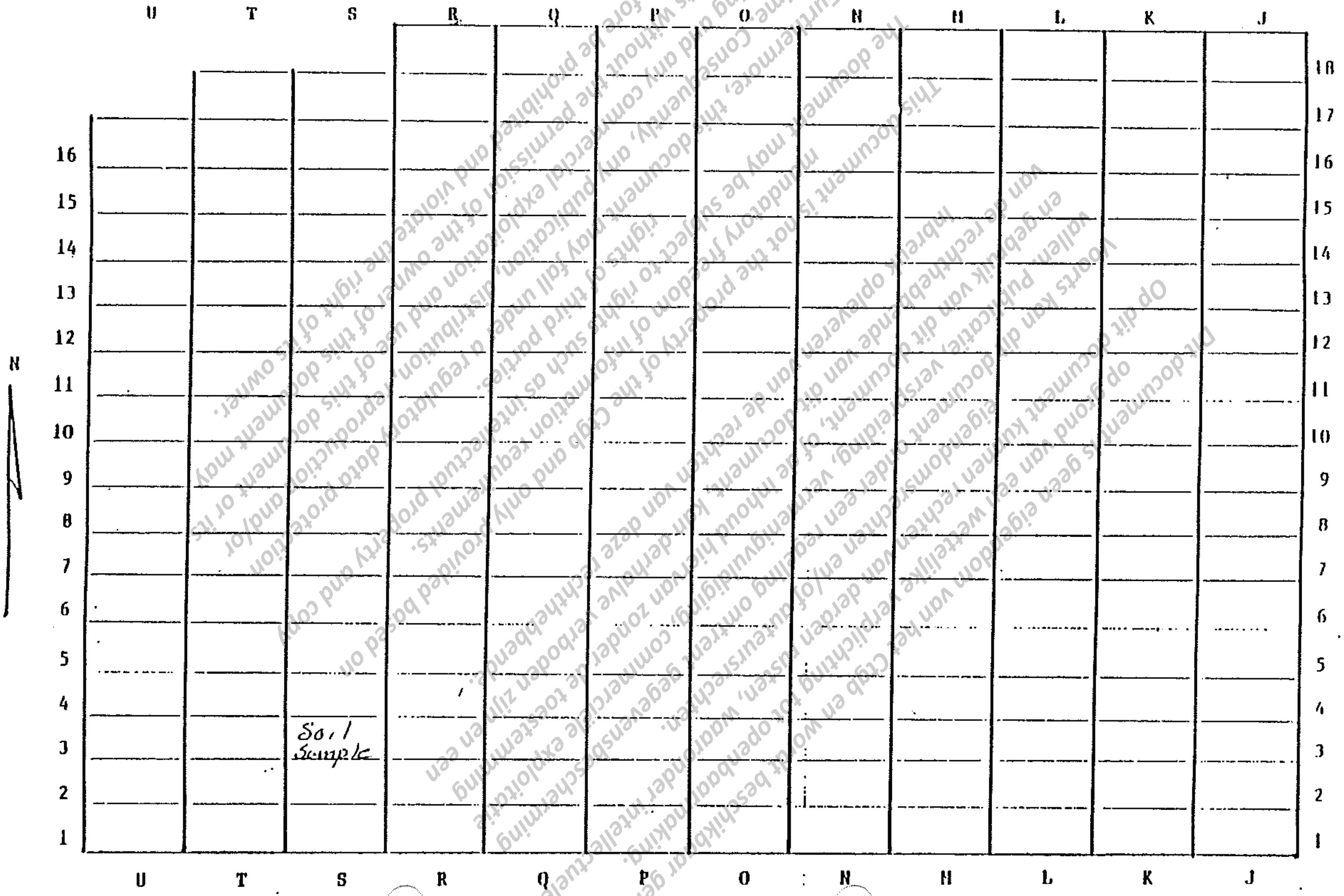

SCALE
MILES
KILOMETERS
ONE KILOMETER = 5/8 MILE
CARTOGRAPHIC DEPARTMENT
COPYRIGHT 1980 BY
CALIFORNIA STATE AUTOMOBILE ASSOCIATION
150 VAN NESS AVENUE SAN FRANCISCO, CA 94102
C6-88
SOME AREAS WITHIN NATIONAL FOREST BOUNDARIES ARE PRIVATELY OWNED. RIGHT OF ENTRY MAY BE RESTRICTED
ROAD CONDITIONS ARE SUBJECT TO CHANGE. INQUIRE LOCALLY
FOR DETAIL OF THIS AREA SEE FRESNO CITY MAP
Madera
Fresno
Clovis
Sanger
Fowler
Pinedale
Herndon
Friant
Prather
Auberry
Academy
Centerville
Minkler
Kerckhoff Res.
Shaver Lake
Millerton Lake
Tollhouse
Easton
Kearney Park
Biola
Highway City
Malaga
Piedra
Humphrey's
SEQUOIA
F O R E S T

ARIZONA 1:50,000



FIELD NUMBERING SYSTEM

UNIVERSITY OF ARIZONA - YUMA VALLEY AG CENTER
(NEW FARM)



Gadsden series

The Gadsden series consists of deep, well drained soils on flood plains and low terraces. These soils formed in mixed fine-textured alluvium. Slope is less than 1 percent. Mean annual precipitation ranges from 2 to 4 inches, and mean annual air temperature ranges from 72 to 76 degrees F.

Gadsden soils are similar to Holtville, Glenbar, and Kofa soils. They are near Glenbar, Holtville, Kofa, Indio, and Ripley soils. Glenbar soils have a fine-silty control section. Holtville soils have a clayey over loamy control section. Kofa soils have sand at a depth of 20 to 40 inches. Indio soils have a coarse-silty control section. Ripley soils have a coarse-silty over sandy control section.

Typical pedon of Gadsden clay, about 2 miles north of Somerton; about 1,500 feet east and 1,300 feet south of the northwest corner of sec. 27, T. 9 S., R. 24 W.:

Ap1—0 to 10 inches; brown (10YR 5/3) clay, dark brown (10YR 3/3) moist; massive; very hard, firm, very sticky and very plastic; few fine roots; few fine tubular pores; few fine interstitial pores; strongly effervescent; moderately alkaline; clear smooth boundary.

AGRISEARCH INCORPORATED

5734 Industry Lane
Frederick, MD 21701
(301) 662-2203
(301) 694-6765 FAX

June 9, 1995

Ms. [redacted]
Ciba Crop Protection
Ciba-Geigy Corporation
P.O. Box 18300
Greensboro, NC 27419

Dear [redacted]

Enclosed are 5 various soil types for biomass determination as requested by Ms. [redacted] These 5 soils are to be used for the upcoming adsorption/desorption testing with metalaxyl and CGA-329351. Please send me your data report as soon as available. Thank you.

Sincerely,

[redacted]

Enclosed

WCS/vs

US Mail

CHAIN OF CUSTODY
AGRISEARCH INCORPORATED
5734 INDUSTRY LANE
FREDERICK, MD 21701

Project No.: 12219/12220
Sample Type: Soils (500g)

Time Sampled: NA
By:
Date: NA

Date Stored: NA
By:
Condition: Ambient

Date Shipped: 6/9/95
No. Samples Shipped: 5

Shipped To: (Name & Address): 5.1.2.e Woo
CIBA CROP Protection
PO Box 18300
Greensboro NC 27419

MS clay
WA loam
S-95-012 sandy loam CA
S95-013 sand MD
S95-014 silty clay loam AZ

Shipped By: 5.1.2.e Woo
Signature
Print Name

Company Name: Agrisearch Incorporated
Address: 5734 Industry Lane Frederick, MD 21701
Phone: (301) 662-2203
Amt. Dry Ice/Container: NA
Commercial Shipper: USPS
Waybill No.: First Class Mail

Received By:
Signature
Print Name

Company Name:
Address:
Date Received:
Condition:

Please maintain a copy of this form through all phases of the study. Return completed form to sender.

AGI-L036-9/28/94

Ciba-Geigy Corporation
Ciba Crop Protection
Quality Assurance Unit

QUALITY ASSURANCE STATEMENT

Study Title: Adsorption/Desorption of ^{14}C-CGA-329351 by the Batch Equilibrium Method on Representative Agricultural Soils

Study Director: 5.1.2.e Woo Agrisearch, Inc.

Agrisearch Project No.: 12220

Ciba Study No.: 60-95

Pursuant to Good Laboratory Practice Standards, this statement verifies that this study was inspected and/or audited and the findings reported to the study director and management by the Ciba Crop Protection Quality Assurance Unit on the dates listed below.

INSPECTION/AUDIT TYPE	INSPECTION/AUDIT DATES	REPORTING DATE
In-Progress Inspection	6/29/95	6/29/95
Data Audit	7/26, 27, 31/95	8/1/95

Prepared by: 5.1.2 e Woo Date: 8/1/95
Quality Assurance Auditor

CIBA, GREENSBORO, NC
BIOMASS ANALYSIS
MISSISSIPPI CLAY
PAGE 1 OF 2

CIBA STUDY NUMBER: 59-95 / 60-95
CONTRACT LABORATORY STUDY NUMBER: 12219 / 12220
STUDY PARTICIPANTS: 5.1.2.e Woo
DATE: July 14, 1995

SOIL IDENTIFICATION: COLLECTED 6/2/94
SOIL TYPE: MISSISSIPPI CLAY

PROCEDURE FOR GRAPHICAL REPRESENTATION OF CO_2 EFFLUX RATE VERSUS HOURS OF INCUBATION AND CALCULATION OF BIOMASS C (mg C/kg SOIL)

1. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed. This graph will usually show a negative slope (CO_2 consumption by microbes) followed by a positive slope (CO_2 production due to microbial generation). If there is no negative slope use the first data point as a representation of the mineralization rate best representing the original biomass.

   INTERVAL: 1

2. Plot the glucose concentration versus the CO_2 efflux rate using Excel version 5.0 for the hour from number 1. Choose the glucose concentration corresponding to the maximal respiration rate. The maximal respiration rate can be described as the inflection point which represents the lowest concentration of glucose on the plateau. See the Excel graph for the initial test.

3. Apply the glucose concentration from number 2 to replicate soil samples.

   GLUCOSE CONCENTRATION: **0.02 G**

4. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed as in number 1.

   INTERVAL: 1

5. Calculate the total microbial biomass C (C) for the soil using the equation:

$$X = 40.04\ Y + 0.37$$

where: X = biomass, mg C per dry weight of soil used
Y = mL CO_2/hr

ex. if the mL CO_2/hr = 0.0934 for 25 g dry weight of soil

$$X = (40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37$$

$$X = \left[\frac{(40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37}{25\ \text{g soil}}\right] X\ 1000\ {}^{g}\!/\!{}_{kg}$$

$$X = 164.389\ \text{mg C/kg soil}$$

CALCULATED BIOMASS (mg C/kg SOIL)

mL CO2 EFFLUX	g DRY SOIL	BIOMASS (mg C/kg SOIL)
R1: 0.0737	18.79	176.74
R2: 0.0876	18.80	206.25
R3: 0.0538	18.78	134.41

AVERAGE BIOMASS: 172.47

These tests have been completed in compliance with 40 CFR Part 160.

5.1.2.e Woo 8/11/95
CHEMIST III DATE

CIBA, GREENSBORO, NC
BIOMASS ANALYSIS
MARYLAND SAND
PAGE 1 OF 2

CIBA STUDY NUMBER: 59-95, 60-95
CONTRACT LABORATORY STUDY NUMBER: 12219 / 12220
STUDY PARTICIPANTS: [redacted]
DATE: JULY 5, 1995

SOIL IDENTIFICATION: S-95-013, COLLECTED 6/8/95
SOIL TYPE: MARYLAND SAND

PROCEDURE FOR GRAPHICAL REPRESENTATION OF CO_2 EFFLUX RATE VERSUS HOURS OF INCUBATION AND CALCULATION OF BIOMASS C (mg C/kg SOIL)

1. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed. This graph will usually show a negative slope (CO_2 consumption by microbes) followed by a positive slope (CO_2 production due to microbial generation). If there is no negative slope use the first data point as a representation of the mineralization rate best representing the original biomass.

   INTERVAL: 4

2. Plot the glucose concentration versus the CO_2 efflux rate using Excel version 5.0 for the hour from number 1. Choose the glucose concentration corresponding to the maximal respiration rate. The maximal respiration rate can be described as the inflection point which represents the lowest concentration of glucose on the plateau. See the Excel graph for the initial test.

3. Apply the glucose concentration from number 2 to replicate soil samples.

   GLUCOSE CONCENTRATION: 0.02 G

4. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed as in number 1.

   INTERVAL: 3

5. Calculate the total microbial biomass C (C) for the soil using the equation:

$$X = 40.04\ Y + 0.37$$

where: X = biomass, mg C per dry weight of soil used
Y = mL CO_2/hr

ex. if the mL CO_2/hr = 0.0934 for 25 g dry weight of soil

$$X = (40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37$$

$$X = \left[\frac{(40.04)(0.0934\ \text{mL}\ CO_2\ /\ \text{hr}) + 0.37}{25\ \text{g soil}}\right] X\ 1000\ {}^{\text{g}}\!/_{\text{kg}}$$

$$X = 164.389\ \text{mg C/kg soil}$$

CALCULATED BIOMASS (mg C/kg SOIL)

mL CO2 EFFLUX	g DRY SOIL	BIOMASS (mg C/kg SOIL)
R1: 0.0796	22.81	155.95
R2: 0.0780	22.81	153.14
R3: 0.0755	22.81	148.75

AVERAGE BIOMASS: 152.61

These tests have been completed in compliance with 40 CFR Part 160.

5.1.2.e Woo

CHEMIST III

8/11/95
DATE

CIBA, GREENSBORO
BIOMASS ANALYSIS
CALIFORNIA SANDY LOAM
PAGE 1 OF 2

CIBA STUDY NUMBER: 59-95 / 60-95
CONTRACT LABORATORY STUDY NUMBER: 12219 / 12220
STUDY PARTICIPANTS: 5.1.2.e Woo
DATE: July 14, 1995

SOIL IDENTIFICATION: 5-95-012, COLLECTED 5/22/95
SOIL TYPE: CALIFORNIA SANDY LOAM

PROCEDURE FOR GRAPHICAL REPRESENTATION OF CO_2 EFFLUX RATE VERSUS HOURS OF INCUBATION AND CALCULATION OF BIOMASS C (mg C/kg SOIL)

1. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed. This graph will usually show a negative slope (CO_2 consumption by microbes) followed by a positive slope (CO_2 production due to microbial generation). If there is no negative slope use the first data point as a representation of the mineralization rate best representing the original biomass.

   INTERVAL: __3__

2. Plot the glucose concentration versus the CO_2 efflux rate using Excel version 5.0 for the hour from number 1. Choose the glucose concentration corresponding to the maximal respiration rate. The maximal respiration rate can be described as the inflection point which represents the lowest concentration of glucose on the plateau. See the Excel graph for the initial test.

3. Apply the glucose concentration from number 2 to replicate soil samples.

   GLUCOSE CONCENTRATION: __0.02 G__

4. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed as in number 1.

   INTERVAL: __2__

CIBA, GREENSBORO
BIOMASS ANALYSIS
CALIFORNIA SANDY LOAM
PAGE 2 OF 2

5. Calculate the total microbial biomass C (C) for the soil using the equation:

$$X = 40.04\ Y + 0.37$$

where: X = biomass, mg C per dry weight of soil used
Y = mL CO_2/hr

ex. if the mL CO_2/hr = 0.0934 for 25 g dry weight of soil

$$X = (40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37$$

$$X = \left[\frac{(40.04)(0.0934\ \text{mL}\ CO_2\ /\ \text{hr}) + 0.37}{25\ \text{g soil}}\right] X\ 1000\ {}^{\text{g}}\!/_{\text{kg}}$$

$$X = 164.389\ \text{mg C/kg soil}$$

CALCULATED BIOMASS (mg C/kg SOIL)

mL CO2 EFFLUX	g DRY SOIL	BIOMASS (mg C/kg SOIL)
R1: 0.0539	21.67	116.67
R2: 0.0525	21.68	114.03
R3: 0.0552	21.68	119.01

AVERAGE BIOMASS: 116.57

These tests have been completed in compliance with 40 CFR Part 160.

5.1.2.e Woo
CHEMIST III

8/11/95
DATE

CIBA STUDY NUMBER: 59-95 / 60-95
CONTRACT LABORATORY STUDY NUMBER: 12219 / 12220
STUDY PARTICIPANTS: [redacted]
DATE: JULY 5, 1995

SOIL IDENTIFICATION: 5/30/94
SOIL TYPE: WASHINGTON LOAM

PROCEDURE FOR GRAPHICAL REPRESENTATION OF CO_2 EFFLUX RATE VERSUS HOURS OF INCUBATION AND CALCULATION OF BIOMASS C (mg C/kg SOIL)

1. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed. This graph will usually show a negative slope (CO_2 consumption by microbes) followed by a positive slope (CO_2 production due to microbial generation). If there is no negative slope use the first data point as a representation of the mineralization rate best representing the original biomass.

   INTERVAL:_1__

2. Plot the glucose concentration versus the CO_2 efflux rate using Excel version 5.0 for the hour from number 1. Choose the glucose concentration corresponding to the maximal respiration rate. The maximal respiration rate can be described as the inflection point which represents the lowest concentration of glucose on the plateau. See the Excel graph for the initial test.

3. Apply the glucose concentration from number 2 to replicate soil samples.

   GLUCOSE CONCENTRATION: _0.02 G____

4. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed as in number 1.

   INTERVAL:_1__

5. Calculate the total microbial biomass C (C) for the soil using the equation:

$$X = 40.04\ Y + 0.37$$

where: X = biomass, mg C per dry weight of soil used
Y = mL CO_2/hr

ex. if the mL CO_2/hr = 0.0934 for 25 g dry weight of soil

$$X = (40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37$$

$$X = \left[\frac{(40.04)(0.0934\ \text{mL}\ CO_2\ /\ \text{hr}) + 0.37}{25\ \text{g soil}}\right] X\ 1000\ {}^{g}\!/_{kg}$$

$$X = 164.389\ \text{mg C/kg soil}$$

CALCULATED BIOMASS (mg C/kg SOIL)

mL CO2 EFFLUX	g DRY SOIL	BIOMASS (mg C/kg SOIL)
R1: 0.2206	19.87	463.15
R2: 0.1846	19.87	390.61
R3: 0.2275	19.87	477.06

AVERAGE BIOMASS: ~~443.83~~ 443.61 corr. Avg. 5.1.2.e Woo 11/15/95

These tests have been completed in compliance with 40 CFR Part 160.

5.1.2.e Woo

CHEMIST III

8/11/95
DATE

CIBA STUDY NUMBER: 59-95 / 60-95
CONTRACT LABORATORY STUDY NUMBER: 12219 / 12220
STUDY PARTICIPANTS: [redacted]
DATE: JULY 18, 1995

SOIL IDENTIFICATION: S-95-014 , COLLECTED 6/7/95
SOIL TYPE: ARIZONA SILTY CLAY LOAM

PROCEDURE FOR GRAPHICAL REPRESENTATION OF CO_2 EFFLUX RATE VERSUS HOURS OF INCUBATION AND CALCULATION OF BIOMASS C (mg C/kg SOIL)

1. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed. This graph will usually show a negative slope (CO_2 consumption by microbes) followed by a positive slope (CO_2 production due to microbial generation). If there is no negative slope use the first data point as a representation of the mineralization rate best representing the original biomass.

   INTERVAL:_2__

2. Plot the glucose concentration versus the CO_2 efflux rate using Excel version 5.0 for the hour from number 1. Choose the glucose concentration corresponding to the maximal respiration rate. The maximal respiration rate can be described as the inflection point which represents the lowest concentration of glucose on the plateau. See the Excel graph for the initial test.

3. Apply the glucose concentration from number 2 to replicate soil samples.

   GLUCOSE CONCENTRATION: __0.1 G__

4. Use the graph of the CO_2 rate-mg/hr and the spreadsheet data from the Oxymax instrument to determine the hour where the lowest CO_2 rate is effluxed as in number 1.

   INTERVAL:_1__

5. Calculate the total microbial biomass C (C) for the soil using the equation:

$$X = 40.04\ Y + 0.37$$

where: X = biomass, mg C per dry weight of soil used
Y = mL CO_2/hr

ex. if the mL CO_2/hr = 0.0934 for 25 g dry weight of soil

$$X = (40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37$$

$$X = \left[\frac{(40.04)(0.0934\ \text{mL}\ CO_2/\text{hr}) + 0.37}{25\ \text{g soil}}\right] X\ 1000\ \text{g}/\text{kg}$$

$$X = 164.389\ \text{mg C/kg soil}$$

CALCULATED BIOMASS (mg C/kg SOIL)

mL CO2 EFFLUX	mg DRY SOIL	BIOMASS (mg C/kg SOIL)
R1: 0.0177	20.84	51.76
R2: 0.0211	20.84	58.29
R3: 0.0209	20.84	57.91

AVERAGE BIOMASS: 55.99

These tests have been completed in compliance with 40 CFR Part 160.

5.1.2.e Woo

CHEMIST III

8/11/95
DATE

APPENDIX D

Representative Data and Example Calculations

<u>DETECTION LIMITS</u>

Determination of Radioactivity Analysis Sensitivity

Sample Size: Solutions	=	adsorption	0.5 mL
		desorption	1.0 mL
		preliminary (Rf)	0.1 mL
Soil	=	combustions	0.23 g
		clay combustions	0.06 g

Typical Background: solutions 32 dpm,
combustion 44 dpm

Sample Counting Time: 5 min.

Background Counting Time: 5 min.

Lowest acceptable gross count rate (LAGCR; 2 x background): 64 dpm

Lowest acceptable net count rate (LANCR = LAGCR - background): 32 dpm

Greatest percent probable error =

$$\frac{0.675\sqrt{\dfrac{\text{LAGCR}}{\text{Sample Count Time}}+\dfrac{\text{Background}}{\text{Background Count Time}}}}{\text{LANCR}} \times 100\%$$

= 9.2% for solutions
= 9.8% for combustions

Minimum sensitivity =

$$\text{LANCR} \div \underset{(\text{dpm}/\mu\text{g})}{\text{specific activity}} \div \underset{(\text{g})}{\text{average sample size}}$$

= 32 dpm ÷ 180042 dpm/μg ÷	0.1 mL	= 0.0018 ppm	solutions (Rf)
32	0.5 mL	0.0004 ppm	adsorption
32	1.0 mL	0.0002 ppm	desorption
44	0.21 g	0.0012 ppm	preliminary combustion
44	0.23 g	0.0011 ppm	combustion soils
44	0.06 g	0.0041 ppm	combustion clay

Wang, C.H. & Willis, D.L. <u>Radiotracer Methodology in Biological Science</u>. Prentice-Hall, Inc., Englewood Cliffs, New Jersey, pp. 186-190 (1965).

ADSORPTION/DESORPTION OF CGA-329351

RADIOCARBON CHECK / DOSE CHECK
SPECIFIC ACTIVITY
CGA-329351 #95-041 180042 DPM/μg = 81.1 μCi/mg

SAMPLE	LS3801 DPM	DPM/mL	TOTAL DPM	TOTAL μCi	TOTAL mg	%VAR
95-~~040~~ 041	379717	189858500	1872593000	843.5	10.40	1.4
	377880	188940000				0.9
corr #	363531	181765500				-2.9
5.1.2.e Woo	373986	186993000				-0.1
11/14/95	377479	188739500				0.8

DOSE SOLUTION IN 10mL UV-ACN
95-041 SOLUTION = 1.040 mg/mL

7.14.95 Gr.

Spreadsheet Program
Quattro Pro Version 1.0

SOLUBILITY CHECK FOR CGA-329351

S.A.= 81.1 µCi/mg
180042 DPM/µg

APPROX DOSE 40.6µCi/2mL WATER 90132 DPM/2µL [redacted] 11/15/95

SAMPLE REP ID	LS3801 DPM	DPM/mL	AVERAGE DPM/mL	PPM	% OF THEORY
2µL ALIQUOTS OF WATER AFTER SONIC, SHAKE, CENTRIFUGE					
OTIME 1	92582	46291000	46584667	258.7	103.4
	94150	47075000			
	92776	46388000			
2	94734	47367000	47124333	261.7	104.6
	92899	46449500			
	95113	47556500	AVERAGE	260.2	
@25°C for 3 hours					
1	95108	47554000	46907167	260.5	104.1
	94615	47307500			
	91720	45860000			
2	90215	45107500	45871333	254.8	101.8
	90009	45004500			
	95004	47502000	AVERAGE	257.7	
@25°C for 6 hours					
1	90052	45026000	46116167	256.1	102.3
	93519	46759500			
	93126	46563000			
2	94021	47010500	46865667	260.3	104.0
	89926	44963000			
	97247	48623500	AVERAGE	258.2	
@25°C for 24 hours					
1	93904	46952000	46232167	256.8	102.6
	91790	45895000			
	91699	45849500			
2	91953	45976500	46873333	260.3	104.0
	96822	48411000			
	92465	46232500	AVERAGE	258.6	
@25°C for 28 hours					
1	94792	47396000	46472833	258.1	103.1
	90850	45425000			
	93195	46597500			
2	90513	45256500	45921667	255.1	101.9
	92294	46147000			
	92723	46361500	AVERAGE	256.6	
@25°C for 48 hours					
1	88306	44153000	45122167	250.6	100.1
	91692	45846000			
	90735	45367500			
2	93463	46731500	46364000	257.5	102.9
	92867	46433500			
	91854	45927000	AVERAGE	254.1	
		STUDY AVERAGE		257.6	

AGRISEARCH INCORPORATED

DATA MEMO

Date 7/11/95 Subject Aqueous Solubility

Project No. 12220 Signature [redacted]

#95-041 ø - ^{14}C-CGA-329351 10 mg

MSR-II-90 SA = 81.1 µCi/mg

= 180042 dpm/µg

1) Duplicate aluminum foil covered silylated glass scintillation vials.

2) 500 µL 95-041 added to each (1 mL pipette) - 10:45 AM

3) ACN evaporated off using gentle stream of N_2

4) 2 mL sterile deionized distilled water (6/26/95) added to each vial - 11:00 AM

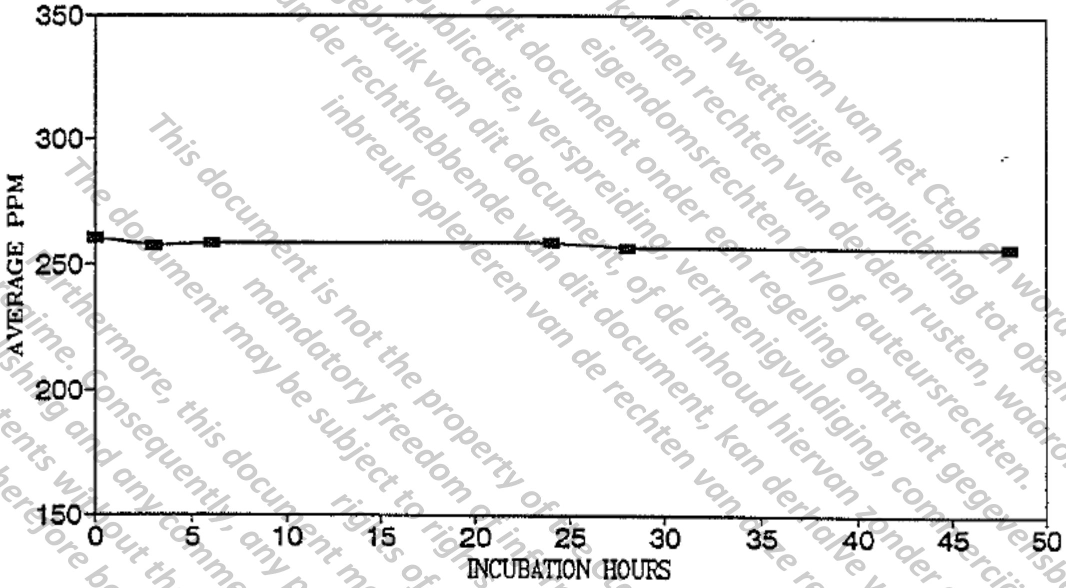
5) Hand shaken 0.5 min, sonicated 5 min, centrifuged ~2500 rpm 15 min.
3×2 µL each rep for LSC.

6) At each sampling removed vials from incubator (Lab-Line Humidity) 25±1°C sonicated ~5 min, hand shaken ~0.5 min, centrifuged ~2500 rpm ~15 min then 3×2 µL for LSC. Prior to return to incubator vials sonicated approx 5 ~~approx 5~~ min 7/11/95 and hand shaken ~0.5 min.

	Date	Time	Initial
⊖ Time	7/11/95	11:00AM	[redacted]
3 hours	↓	2:00PM	[redacted]
6 hours		5:00PM	[redacted]
24 hours	7/12/95	11:00AM	[redacted]
28 hours	7/12/95	3:05PM	[redacted]
48 hours	7/13/95	11:00AM	[redacted]

AGI-L007b-9/17/91

Corning 240 pH meter: Rep1 7.09
Rep2 7.12 [redacted] 7/13/95



CGA-329351 WATER SOLUBILITY

25°C 7.11pH

7.14.95

AGRISEARCH INCORPORATED

Dose Preparation - Rangefinder Repeat

Study No. 12220

Agrisearch No. 95-041 Test Material CGA-327351

Concentration 1mg/μL

(Received 1.75mL① ~~μL~~ for Study from Standard Storage)

4 soils in duplicate 1 g. + 20 mL solution each.

~~2 blanks at 20 mL solution each.~~ not repeated [redacted] 8/2/95

requires ~~200~~ 1750① mL buffer at 10 ppm = ~~2~~ 17.5① mg

(2) ~~(1)~~ placed ~~200~~ 1750① mL Ca^{2+} solution in sterile 500 mL Erlenmeyer flask
(1) ~~(2)~~② added 1.75mL① ~~μL~~ of 95-041. Pipettor ID 1mL pip
(3) mixed well--aliquoted 5 ~~3~~ x 0.1 mL for dose check (LSC) (~3646 dp)
(4) added 20 mL/replicate to start rangefinder
(5) shaking began at 9:25 Am on Eberbach reciprocal shaker at 175-200 rpm 4cm throw in Hotpack incubator @ 25°C

① corr. vol. # , # [redacted] 8/2/95

② corr. order.
ACN evaporated off under N_2 stream [redacted] 8/2/95

pH Check corning pH meter ④ 8.2.95

pH 7.21 Dose Soln 8.2.95

[redacted] 8/2/95
Initials Date

AGI-L019-9/22/92

AGRISEARCH INCORPORATED

Dose Preparation - Rangefinder

Study No. 12220

Agrisearch No. 95-041 Test Material CGA-329351

Concentration 1mg/mL

(Received 7.5mL ~~µL~~ ① for Study from Standard Storage)

5 ~~4~~ ① soils in duplicate 1 g. + 20 mL solution each.

2 blanks at 20 mL solution each.

requires ~~200~~ 250 ① mL buffer at 10 ppm = ~~2~~ 2.5 ① mg

(2) ~~(1)~~ ③ placed ~~200~~ 250 ① mL Ca^{2+} solution in sterile 500 mL Erlenmeyer flask
(1) ~~(2)~~ added 2.5 mL ② ~~µL~~ ① of 95-041. Pipettor ID 1 mL pipet
(3) mixed well--aliquoted 3 x 0.1 mL for dose check (LSC)
(4) added 20 mL/replicate to start rangefinder
(5) shaking began at 9:30 am on Eberbach reciprocal shaker at 175-200 rpm 4cm throw in ~~Hotpack incubator~~ Laboratory [redacted] 7/18/95

① correct # soils, # mL, # mg [redacted] 7/18/95
② ACN evap off under N_2 stream [redacted] 7/18/95
③ correct order [redacted] 7/18/95

[redacted] 7/18/95
Initials Date

pH checks Corning 240 meter

Ca ion soln 6.93 7.18.95

Dose soln 7.54 7.18.95 [redacted]

AGI-L019-9/22/92

AGRISEARCH INCORPORATED

```
PRELIMINARY PHASE
-----------------------------------------------------------------
STUDY NO: 12220                    SAMPLE SIZE: 20mL
         S.A: 180042 DPM/µg        DOSE:    38317680 DPM/20mL
-----------------------------------------------------------------
SAMPLE     ALIQLS3801    DPM/ml     ppm      MEAN     %
I.D.   REP  mL DPM                           ppm     DOSE
-----------------------------------------------------------------
TEST SOLUTION CONCENTRATION                                  %VAR
0 TIME     0.1 192717   1927170   10.704   10.641   100.0      0.6
           0.1 192318   1923180   10.682                       0.4
           0.1 190942   1909420   10.605                      -0.3
           0.1 190759   1907590   10.595                      -0.4
           0.1 191206   1912060   10.620                      -0.2

          TOTAL DOSE DPM
          ============
       AVG DPM/mL        1915884

       TOTAL DPM/20mL 38317680
                      =========
```

7.27.95

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PRELIMINARY PHASE

STUDY NO: 12220 S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/ml	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
WA LOAM									
2 Hour	1	0.1	178363	1783630	1779490	35589800	9.907	9.884	92.9
		0.1	177535	1775350			9.861		
	2	0.1	176097	1760970	1766840	35336800	9.781	9.813	92.2
		0.1	177271	1772710			9.846		
4 Hour	1	0.1	177484	1774840	1773270	35465400	9.858	9.849	92.6
		0.1	177170	1771700			9.840		
	2	0.1	175835	1758350	1759440	35188800	9.766	9.772	91.8
		0.1	176053	1760530			9.778		
8 Hour	1	0.1	177625	1776250	1780045	35600900	9.866	9.887	92.9
		0.1	178384	1783840			9.908		
	2	0.1	177178	1771780	1774925	35498500	9.841	9.858	92.6
		0.1	177807	1778070			9.876		
24 Hour	1	0.1	179210	1792100	1802335	36046700	9.954	10.011	94.1
		0.1	181257	1812570			10.067		
	2	0.1	177060	1770600	1778100	35562000	9.834	9.876	92.8
		0.1	178560	1785600			9.918		
BLANK									
24 Hour	1	0.1	190868	1908680	1914755	38295100	10.601	10.635	99.9
		0.1	192083	1920830			10.669		
	2	0.1	191181	1911810	1912315	38246300	10.619	10.621	99.8
		0.1	191282	1912820			10.624		

7-27-95 [redacted]

$$ppm = \frac{DPM/mL}{180042\ DPM/\mu g}$$

$$MEAN\ ppm = \frac{MEAN\ DPM/mL}{180042\ DPM/\mu g}$$

$$\%\ DOSE = \frac{TOTAL\ DPM}{DOSE\ 38317680\ DPM} \times 100$$

[redacted] /26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

STUDY NO: 12220
S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
CA SANDY LOAM									
2 Hour	1	0.1	187890	1878900	1884645	37692900	10.436	10.468	98.4
		0.1	189039	1890390			10.500		
	2	0.1	189412	1894120	1889725	37794500	10.520	10.496	98.6
		0.1	188533	1885330			10.472		
4 Hour	1	0.1	189206	1892060	1896230	37924600	10.509	10.532	99.0
		0.1	190040	1900400			10.555		
	2	0.1	190441	1904410	1901140	38022800	10.578	10.559	99.2
		0.1	189787	1897870			10.541		
8 Hour	1	0.1	188849	1888490	1890075	37801500	10.489	10.498	98.7
		0.1	189166	1891660			10.507		
	2	0.1	190152	1901520	1899550	37991000	10.562	10.551	99.1
		0.1	189758	1897580			10.540		
24 Hour	1	0.1	185999	1859990	1878785	37575700	10.331	10.435	98.1
		0.1	189758	1897580			10.540		
	2	0.1	191841	1918410	1899535	37990700	10.655	10.551	99.1
		0.1	188066	1880660			10.446		

8.11.95 [redacted]

$$ppm = \frac{DPM/mL}{180042\ DPM/\mu g}$$

$$MEAN\ ppm = \frac{MEAN\ DPM/mL}{180042\ DPM/\mu g}$$

$$\%\ DOSE = \frac{TOTAL\ DPM}{DOSE} \times 100$$

[redacted] 9/26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

STUDY NO: 12220
S.A: 180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
MS CLAY									
2 Hour	1	0.1	84426	844260	850780	17015600	4.689	4.725	44.4
		0.1	85730	857300			4.762		
	2	0.1	84332	843320	849275	16985500	4.684	4.717	44.3
		0.1	85523	855230			4.750		
4 Hour	1	0.1	85893	858930	853885	17077700	4.771	4.743	44.6
		0.1	84884	848840			4.715		
	2	0.1	85214	852140	851895	17037900	4.733	4.732	44.5
		0.1	85165	851650			4.730		
8 Hour	1	0.1	86058	860580	861170	17223400	4.780	4.783	44.9
		0.1	86176	861760			4.786		
	2	0.1	84012	840120	843325	16866500	4.666	4.684	44.0
		0.1	84653	846530			4.702		
24 Hour	1	0.1	83896	838960	842315	16846300	4.660	4.678	44.0
		0.1	84567	845670			4.697		
	2	0.1	81932	819320	818400	16368000	4.551	4.546	42.7
		0.1	81748	817480			4.540		

8-17-95 [redacted]

$$\text{ppm} = \frac{\text{DPM/mL}}{180042\ \text{dpm}/\mu g}$$

$$\text{mean ppm} = \frac{\text{mean DPM/mL}}{180042\ \text{dpm}/\mu g}$$

$$\%\text{Dose} = \frac{\text{TOTAL DPM}}{\text{Dose } 38317680\ \text{DPM}} \times 100$$

[redacted] 7/26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

STUDY NO: 12220
S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
MD SAND									
2 Hour	1	0.1	190600	1906000	1891505	37830100	10.586	10.506	98.7
		0.1	187701	1877010			10.425		
	2	0.1	194331	1943310	1925790	38515800	10.794	10.696	100.5
		0.1	190827	1908270			10.599		
4 Hour	1	0.1	191719	1917190	1918230	38364600	10.649	10.654	100.1
		0.1	191927	1919270			10.660		
	2	0.1	191134	1911340	1909910	38198200	10.616	10.608	99.7
		0.1	190848	1908480			10.600		
8 Hour	1	0.1	190687	1906870	1917570	38351400	10.591	10.651	100.1
		0.1	192827	1928270			10.710		
	2	0.1	191132	1911320	1915540	38310800	10.616	10.639	100.0
		0.1	191976	1919760			10.663		
24 Hour	1	0.1	189513	1895130	1905370	38107400	10.526	10.583	99.5
		0.1	191561	1915610			10.640		
	2	0.1	190293	1902930	1903070	38061400	10.569	10.570	99.3
		0.1	190321	1903210			10.571		

7·27·95 [redacted]

$$\text{ppm} = \frac{\text{DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\text{MEAN ppm} = \frac{\text{MEAN DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\%\ \text{DOSE} = \frac{\text{TOTAL DPM}}{\text{DOSE}} \times 100$$

[redacted] /26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

STUDY NO: 12220
S.A: 180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
AZ SILTY CLAY LOAM									
2 Hour	1	0.1	160655	1606550	1611130	32222600	8.923	8.949	84.1
		0.1	161571	1615710			8.974		
	2	0.1	159235	1592350	1588760	31775200	8.844	8.824	82.9
		0.1	158517	1585170			8.804		
4 Hour	1	0.1	161275	1612750	1601435	32028700	8.958	8.895	83.6
		0.1	159012	1590120			8.832		
	2	0.1	157214	1572140	1571445	31428900	8.732	8.728	82.0
		0.1	157075	1570750			8.724		
8 Hour	1	0.1	159424	1594240	1602405	32048100	8.855	8.900	83.6
		0.1	161057	1610570			8.946		
	2	0.1	158742	1587420	1585850	31717000	8.817	8.808	82.8
		0.1	158428	1584280			8.800		
24 Hour	1	0.1	159206	1592060	1589910	31798200	8.843	8.831	83.0
		0.1	158776	1587760			8.819		
	2	0.1	157358	1573580	1576125	31522500	8.740	8.754	82.3
		0.1	157867	1578670			8.768		

7.27.95 [redacted]

$$\text{ppm} = \frac{\text{DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\text{MEAN}_{\text{ppm}} = \frac{\text{MEAN DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\%\ \text{DOSE} = \frac{\text{TOTAL DPM}}{\text{DOSE}} \times 100$$

[redacted] 1/26/95

PRELIMINARY PHASE AGRISEARCH INCORPORATED

SAMPLE TIME: 24 HOURS
STUDY NO: 12220
S.A: 180042 DPM/μg
SAMPLE SIZE: 20mL
DOSE: 38317680 DPM/20mL

ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANT

SAMPLE	REP	TARE (g)	DRY SOIL+T. (g)	WET SOIL+T. (g)	WET SOIL (g)	DRY SOIL (g)	WATER (g)	% MOIST
MS Clay	1	35.722	36.965	38.269	2.547	1.243	1.304	51.20
	2	32.725	33.959	35.313	2.588	1.234	1.354	52.32
MD Sand	1	34.852	35.817	36.369	1.517	0.965	0.552	36.39
	2	33.360	34.604	35.233	1.873	1.244	0.629	33.58
CA Sandy Loam	1	33.123	34.350	35.081	1.958	1.227	0.731	37.33
	2	32.932	33.959	34.609	1.677	1.027	0.650	38.76
WA Loam	1	34.794	35.710	36.505	1.711	0.916	0.795	46.46
	2	36.076	37.108	38.009	1.933	1.032	0.901	46.61
AZ Silty Clay Loam	1	35.766	36.779	37.637	1.871	1.013	0.858	45.86
	2	35.060	36.175	37.066	2.006	1.115	0.891	44.42

SOIL COMBUSTIONS

SAMPLE	REP	BMO ALIQ.g	LS3801 DPM	DPM/g	MEAN DPM/g	TOTAL DPM	DPM ABS	% DOSE ABS
SOIL EFF =		0.9500 *						
MS Clay	1	0.213	1472866	7278887	7316058	18634000	17535621	45.8
		0.201	1404083	7353229				
	2	0.207	1581342	8041496	8245907	21340406	20232293	52.8
		0.200	1605542	8450318				
MD Sand	1	0.206	141698	724066	661681	1003770	0	0.0
		0.203	115573	599296				
	2	0.208	125510	635179	590748	1106471	0	0.0
		0.208	107951	546317				
CA Sandy Loam	1	0.209	165887	835502	777852	1523034	149642	0.4
		0.201	137521	720202				
	2	0.209	176762	890275	870687	1460142	225444	0.6
		0.200	161707	851099				
WA Loam	1	0.205	396092	2033872	2122672	3631892	2199035	5.7
		0.207	434881	2211472				
	2	0.208	438083	2217045	2145085	4146449	2544381	6.6
		0.204	401767	2073125				
AZ Silty Clay Loam	1	0.207	777831	3955453	3900673	7298160	5934017	15.5
		0.212	774554	3845893				
	2	0.213	702553	3472009	3704724	7431676	6027348	15.7
		0.212	792991	3937438				

* Used to correct LS3801 DPM to total DPM/g
(DPM/g = LS3801 DPM/SOIL EFF/BMO ALIQ)

8.17.95 [redacted]

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

SAMPLE TIME: 24 HOURS
STUDY NO: 12220
S.A: 180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 38317680 DPM

SAMPLE	REP	DPM ABS.	MEAN DPM/ml	WATER g	DRY SOIL	DPM/g DRY SOIL	ppm SOIL	ppm WATER	Kd EST. [s]/[w]
MS Clay	1	17535621	842315	1.304	1.243	14107499	78.36	4.678	16.75
	2	20232293	818400	1.354	1.234	16395699	91.07	4.546	20.03
MD Sand	1	0	1905370	0.552	0.965	0	0.00	10.583	0.00
	2	0	1903070	0.629	1.244	0	0.00	10.570	0.00
CA Sandy Loam	1	149642	1878785	0.731	1.227	121958	0.68	10.435	0.06
	2	225444	1899535	0.650	1.027	219517	1.22	10.551	0.12
WA Loam	1	2199035	1802335	0.795	0.916	2400694	13.33	10.011	1.33
	2	2544381	1778100	0.901	1.032	2465485	13.69	9.876	1.39
AZ Silty Clay Loam	1	5934017	1589910	0.858	1.013	5857865	32.54	8.831	3.68
	2	6027348	1576125	0.891	1.115	5405694	30.02	8.754	3.43

8-17-95

$$\text{ppm Soil} = \frac{\text{DPM/g Dry Soil}}{180042\ \text{DPM}/\mu g}$$

$$\text{ppm Water} = \frac{\text{MEAN DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\text{Kd EST} = \frac{[S]}{[W]} = \frac{\text{ppm soil}}{\text{ppm Water}}$$

9/26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE 24 HOURS RADIOCARBON BALANCE

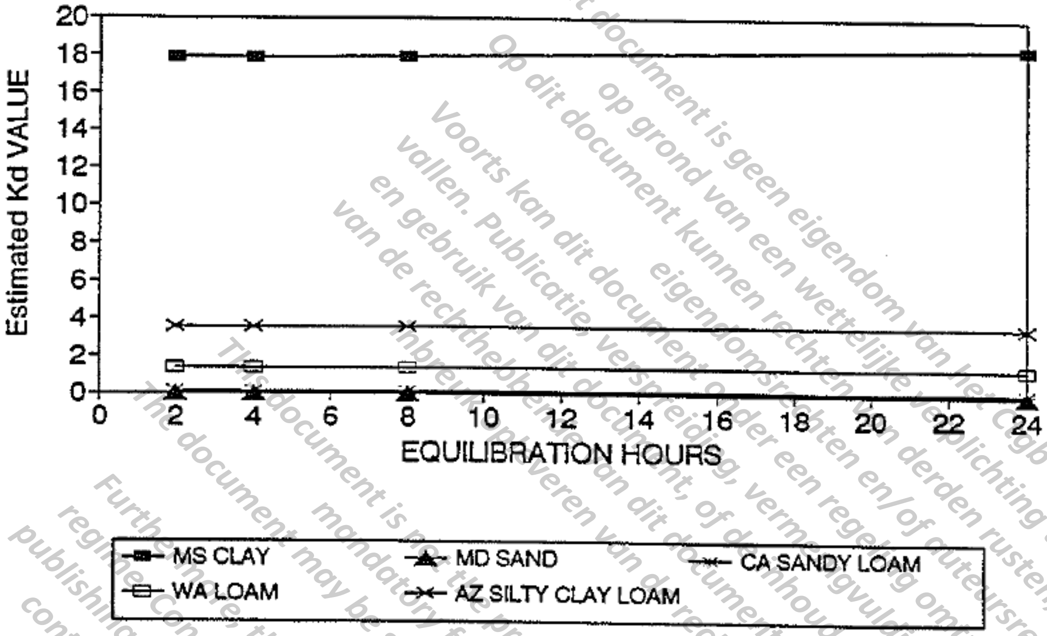
STUDY NO: 12220 SAMPLE SIZE: 20mL
S.A: 180042 DPM/µg DOSE: 38317680 DPM/20mL

SAMPLE I.D.	REP	=====TOTAL DPM===== SOLUTION	SOIL	=====% DOSE===== SOLUTION	SOIL	TOTAL %DOSE
MS Clay	1	16846300	17535621	44.0	45.8	89.8
	2	16368000	20232293	42.7	52.8	95.5
MD Sand	1	38107400	0	99.5	0.0	99.5
	2	38061400	0	99.3	0.0	99.3
CA Sandy Loam	1	37575700	149642	98.1	0.4	98.5
	2	37990700	225444	99.1	0.6	99.7
WA Loam	1	36046700	2199035	94.1	5.7	99.8
	2	35562000	2544381	92.8	6.6	99.4
AZ Silty Clay Loam	1	31798200	5934017	83.0	15.5	98.5
	2	31522500	6027348	82.3	15.7	98.0
					AVERAGE %	97.8
BLANK *	1	38295100	NA	99.9	NA	99.9
	2	38246300	NA	99.8	NA	99.8

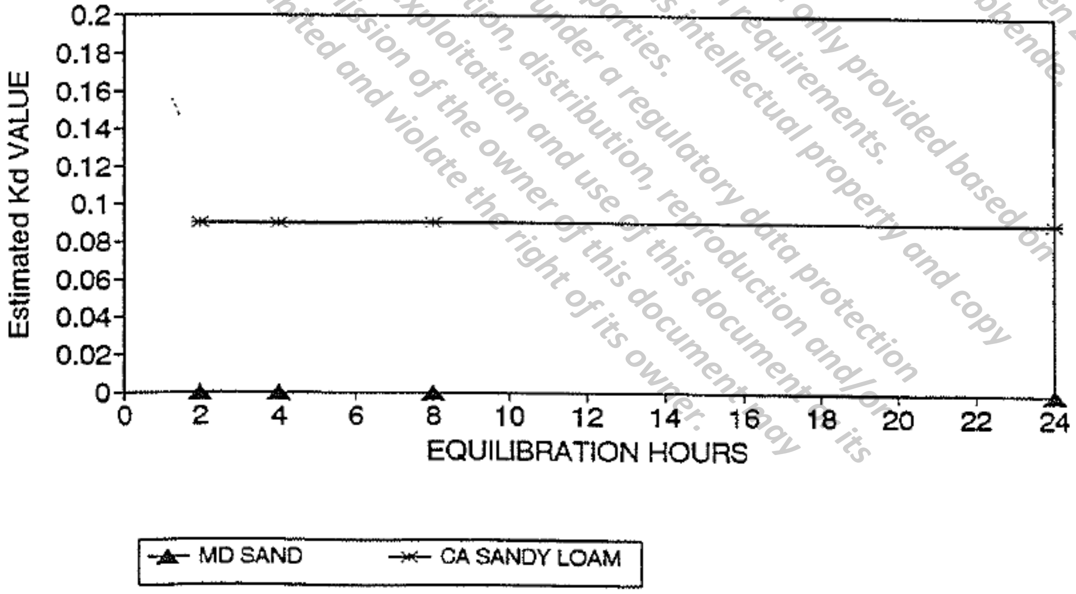
* SOIL-LESS BLANK
NA = NOT APPLICABLE

8·17·95

^{14}C-CGA-329351
RANGEFINDER ESTIMATED Kd'S



Expanded Y-Scale for
MD Sand and CA Sandy Loam



8·17·95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE: REPEAT RANGEFINDER

SAMPLE TIME: 24 HOURS
STUDY NO: 12220
S.A: 180042 DPM/μg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM/20mL

SAMPLE	REP	TARE (g)	DRY SOIL+T. (g)	WET SOIL+T. (g)	WET SOIL (g)	DRY SOIL (g)	WATER (g)	% MOIST
ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM,CENTRIFUGATION,DECANT								
MD Sand	1	35.440	45.490	48.628	13.188	10.050	3.138	23.79
	2	35.782	45.802	48.977	13.195	10.020	3.175	24.06
CA Sandy Loam	1	35.306	45.387	50.043	14.737	10.081	4.656	31.59
	2	40.376	50.495	55.385	15.009	10.119	4.890	32.58
WA Loam	1	35.410	39.420	41.842	6.432	4.010	2.422	37.66
	2	35.818	40.040	42.432	6.614	4.222	2.392	36.17
AZ Silty Clay Loam	1	34.968	38.980	41.519	6.551	4.012	2.539	38.76
	2	36.047	40.055	42.630	6.583	4.008	2.575	39.12

SOIL COMBUSTIONS

SAMPLE	REP	BMO ALIQ.g	LS3801 DPM	DPM/g	MEAN DPM/g	TOTAL DPM	DPM ABS	% DOSE ABS
SOIL EFF = 0.9563 *								
MD Sand	1	0.237	131079	578332	582025	7675747	2231427	6.0
		0.237	132753	585718				
	2	0.231	147091	665835	661556	8729225	3092790	8.3
		0.201	126343	657276				
CA Sandy Loam	1	0.237	223776	987320	1040576	15334966	9123490	24.4
		0.220	230134	1093832				
	2	0.227	220403	1015276	1038624	15588712	9016014	24.1
		0.205	208197	1061972				
WA Loam	1	0.201	357378	1859192	1832530	11786835	8322418	22.2
		0.219	378214	1805868				
	2	0.200	347874	1818798	1821754	12049081	8620185	23.0
		0.216	376925	1824710				
AZ Silty Clay Loam	1	0.215	558960	2718535	2704062	17714309	14887780	39.8
		0.211	542720	2689589				
	2	0.211	544358	2697707	2682366	17658014	14791099	39.5
		0.233	594279	2667025				

* Used to correct LS3801 DPM to total DPM/g
(DPM/g = LS3801 DPM/SOIL EFF/BMO ALIQ)

8.10.95

AGRISEARCH INCORPORATED
PRELIMINARY PHASE: REPEAT RANGEFINDER

STUDY NO: 12220
S.A: 180042 DPM/μg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
AZ SILTY CLAY LOAM									
2 Hour	1	0.1	115023	1150230	1140570	22811400	6.389	6.335	61.0
		0.1	113091	1130910			6.281		
	2	0.1	113894	1138940	1140230	22804600	6.326	6.333	61.0
		0.1	114152	1141520			6.340		
4 Hour	1	0.1	114291	1142910	1153100	23062000	6.348	6.405	61.6
		0.1	116329	1163290			6.461		
	2	0.1	118385	1183850	1161255	23225100	6.575	6.450	62.1
		0.1	113866	1138660			6.324		
8 Hour	1	0.1	110675	1106750	1107055	22141100	6.147	6.149	59.2
		0.1	110736	1107360			6.151		
	2	0.1	124467	1244670	1190975	23819500	6.913	6.615	63.7
		0.1	113728	1137280			6.317		
24 Hour	1	0.1	112067	1120670	1113245	22264900	6.224	6.183	59.5
		0.1	110582	1105820			6.142		
	2	0.1	111731	1117310	1113365	22267300	6.206	6.184	59.5
		0.1	110942	1109420			6.162		

8-10-95 [redacted]

$$ppm = \frac{DPM/mL}{180042\ DPM/\mu g}$$

$$MEAN\ ppm = \frac{MEAN\ DPM/mL}{180042\ DPM/\mu g}$$

$$\%\ DOSE = \frac{TOTAL\ DPM}{DOSE} \times 100$$

[redacted] 9/26/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE: REPEAT RANGEFINDER

STUDY NO: 12220
S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/ml	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
WA LOAM									
2 Hour	1	0.1	144463	1444630	1433935	28678700	8.024	7.964	76.7
		0.1	142324	1423240			7.905		
	2	0.1	143118	1431180	1429015	28580300	7.949	7.937	76.4
		0.1	142685	1426850			7.925		
4 Hour	1	0.1	142886	1428860	1430390	28607800	7.936	7.945	76.5
		0.1	143192	1431920			7.953		
	2	0.1	144877	1448770	1438170	28763400	8.047	7.988	76.9
		0.1	142757	1427570			7.929		
8 Hour	1	0.1	139780	1397800	1400595	28011900	7.764	7.779	74.9
		0.1	140339	1403390			7.795		
	2	0.1	141278	1412780	1411890	28237800	7.847	7.842	75.5
		0.1	141100	1411000			7.837		
24 Hour	1	0.1	143121	1431210	1430395	28607900	7.949	7.945	76.5
		0.1	142958	1429580			7.940		
	2	0.1	143108	1431080	1433485	28669700	7.949	7.962	76.6
		0.1	143589	1435890			7.975		

8.10.95 [redacted]

$$\text{ppm} = \frac{\text{DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\text{MEAN ppm} = \frac{\text{MEAN DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\%\ \text{DOSE} = \frac{\text{TOTAL DPM}}{\text{DOSE}} \times 100$$

[redacted] 9/26/95

AGRISEARCH INCORPORATED
PRELIMINARY PHASE: REPEAT RANGEFINDER

STUDY NO: 12220
S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
CA SANDY LOAM									
2 Hour	1	0.1	146463	1464630	1453560	29071200	8.135	8.073	77.7
		0.1	144249	1442490			8.012		
	2	0.1	146258	1462580	1436015	28720300	8.124	7.976	76.8
		0.1	140945	1409450			7.828		
4 Hour	1	0.1	128197	1281970	1335595	26711900	7.120	7.418	71.4
		0.1	138922	1389220			7.716		
	2	0.1	133311	1333110	1329780	26595600	7.404	7.386	71.1
		0.1	132645	1326450			7.367		
8 Hour	1	0.1	146490	1464900	1416145	28322900	8.136	7.866	75.7
		0.1	136739	1367390			7.595		
	2	0.1	138510	1385100	1367100	27342000	7.693	7.593	73.1
		0.1	134910	1349100			7.493		
24 Hour	1	0.1	138432	1384320	1334080	26681600	7.689	7.410	71.3
		0.1	128384	1283840			7.131		
	2	0.1	132293	1322930	1344110	26882200	7.348	7.466	71.9
		0.1	136529	1365290			7.583		

8.10.95 [redacted]

$$ppm = \frac{DPM/mL}{180042\ DPM/mL}$$

$$MEAN\ ppm = \frac{MEAN\ DPM/mL}{180042\ DPM/\mu g}$$

$$\%\ Dose = \frac{TOTAL\ DPM}{DOSE} \times 100$$

[redacted] 9/26/95

AGRISEARCH INCORPORATED
PRELIMINARY PHASE: REPEAT RANGEFINDER

STUDY NO: 12220
S.A:180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM/20mL

SAMPLE I.D.	REP	ALIQ mL	LS3801 DPM	DPM/mL	MEAN DPM/mL	TOTAL DPM	ppm	MEAN ppm	% DOSE
MD SAND									
2 Hour	1	0.1	183194	1831940	1822085	36441700	10.175	10.120	97.4
		0.1	181223	1812230			10.066		
	2	0.1	188743	1887430	1850365	37007300	10.483	10.277	98.9
		0.1	181330	1813300			10.072		
4 Hour	1	0.1	181235	1812350	1799485	35989700	10.066	9.995	96.2
		0.1	178662	1786620			9.923		
	2	0.1	180258	1802580	1804820	36096400	10.012	10.024	96.5
		0.1	180706	1807060			10.037		
8 Hour	1	0.1	174878	1748780	1748915	34978300	9.713	9.714	93.5
		0.1	174905	1749050			9.715		
	2	0.1	177126	1771260	1772645	35452900	9.838	9.846	94.8
		0.1	177403	1774030			9.853		
24 Hour	1	0.1	173300	1733000	1734965	34699300	9.626	9.636	92.8
		0.1	173693	1736930			9.647		
	2	0.1	177304	1773040	1775255	35505100	9.848	9.860	94.9
		0.1	177747	1777470			9.873		

8-10-95 [redacted]

$$ppm = \frac{DPM/mL}{180042\ DPM/\mu g}$$

$$MEAN\ ppm = \frac{MEAN\ DPM/mL}{180042\ DPM/\mu g}$$

$$\%\ DOSE = \frac{TOTAL\ DPM}{DOSE} \times 100$$

[redacted] 9/26/95

AGRISEARCH INCORPORATED

```
PRELIMINARY PHASE
-----------------------------------------------------------------
STUDY NO: 12220                     SAMPLE SIZE: 20mL
          S.A: 180042 DPM/µg        DOSE:    37411080 DPM/20mL
-----------------------------------------------------------------
SAMPLE      ALIQLS3801    DPM/ml     ppm      MEAN       %
I.D.    REP  mL DPM                           ppm       DOSE
-----------------------------------------------------------------
TEST SOLUTION CONCENTRATION                                   %VAR
0 TIME       0.1 186792  1867920  10.375   10.390   100.0     -0.1
             0.1 187465  1874650  10.412                       0.2
             0.1 187099  1870990  10.392                       0.0
             0.1 186661  1866610  10.368                      -0.2
             0.1 187260  1872600  10.401                       0.1

         TOTAL DOSE DPM
         ============
      AVG DPM/mL        1870554

      TOTAL DPM/20mL 37411080
                     =========
```

8-10-95

AGRISEARCH INCORPORATED
PRELIMINARY PHASE: REPEAT RANGEFINDER

SAMPLE TIME: 24 HOURS
STUDY NO: 12220
S.A: 180042 DPM/µg

SAMPLE SIZE: 20mL
DOSE: 37411080 DPM

SAMPLE	REP	DPM ABS.	MEAN DPM/ml	WATER g	DRY SOIL g	DPM/g DRY SOIL	ppm SOIL	ppm WATER	Kd EST. [s]/[w]
MD Sand	1	2231427	1734965	3.138	10.050	222033	1.23	9.636	0.13
	2	3092790	1775255	3.175	10.020	308662	1.71	9.860	0.17
CA Sandy Loam	1	9123490	1334080	4.656	10.081	905018	5.03	7.410	0.68
	2	9016014	1344110	4.890	10.119	890998	4.95	7.466	0.66
WA Loam	1	8322418	1430395	2.422	4.010	2075416	11.53	7.945	1.45
	2	8620185	1433485	2.392	4.222	2041730	11.34	7.962	1.42
AZ Silty Clay Loam	1	14887780	1113245	2.539	4.012	3710813	20.61	6.183	3.33
	2	14791099	1113365	2.575	4.008	3690394	20.50	6.184	3.31

8.10.95 [redacted]

$$\text{ppm SOIL} = \frac{\text{DPM/g DRY SOIL}}{180042\ \text{DPM}/\mu g}$$

$$\text{ppm WATER} = \frac{\text{MEAN DPM/mL}}{180042\ \text{DPM}/\mu g}$$

$$\text{Kd Est} = \frac{[S]}{[W]} = \frac{\text{ppm soil}}{\text{ppm water}}$$

[redacted] /24/95

AGRISEARCH INCORPORATED

PRELIMINARY PHASE

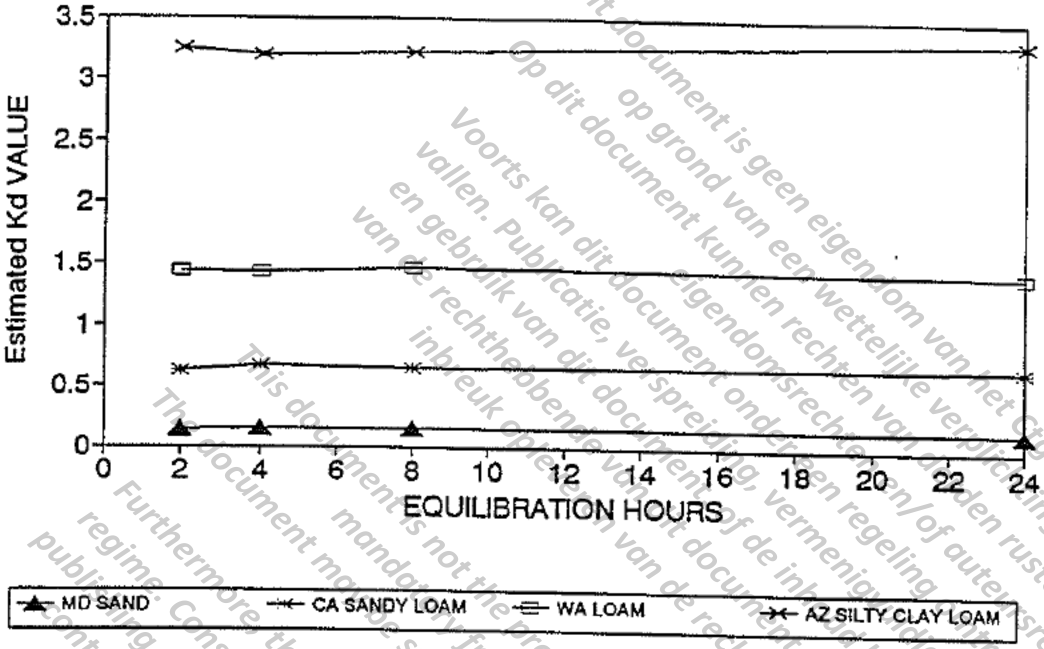
24 HOURS RADIOCARBON BALANCE

STUDY NO: 12220 SAMPLE SIZE: 20mL
S.A: 180042 DPM/µg DOSE: 37411080 DPM/20mL

SAMPLE I.D.	REP	=====TOTAL DPM===== SOLUTION	SOIL	======% DOSE===== SOLUTION	SOIL	TOTAL %DOSE
MD Sand	1	34699300	2231427	92.8	6.0	98.7
	2	35505100	3092790	94.9	8.3	103.2
CA Sandy Loam	1	26681600	9123490	71.3	24.4	95.7
	2	26882200	9016014	71.9	24.1	96.0
WA Loam	1	28607900	8322418	76.5	22.2	98.7
	2	28669700	8620185	76.6	23.0	99.7
AZ Silty Clay Loam	1	22264900	14887780	59.5	39.8	99.3
	2	22267300	14791099	59.5	39.5	99.1
					AVERAGE %	98.8

8.10.95

^{14}C-CGA-329351
RANGEFINDER ESTIMATED Kd'S



repeat rangefinder
8.10.95

AGRISEARCH INCORPORATED

DATA MEMO

Date 8/17/95 Subject pH Measurements

Project No. 12220 Signature [redacted]

Corning 240 pH Meter

Definitive Dose Solutions ①

µg/mL	0.0	0.2	0.5	1.0	5.0	10.0
pH	7.05	7.04	7.10	7.01	6.92	7.03

8.17.95 [redacted]

Adsorption Solution (post adsorption mixing)

	0.0	0.2	0.5	1.0	5.0	10.0
~~MS clay Rep.1~~	~~6.91~~	~~6.62~~	~~6.72~~	~~6.96~~	~~6.94~~	~~6.97~~
~~MD sand Rep.1~~	~~5.69~~	~~5.44~~	~~5.37~~	~~5.46~~	~~5.64~~	
~~CA sandy loam Rep.1~~	~~5.60~~	~~5.56~~	~~5.57~~	~~5.66~~	~~5.93~~	
~~WA loam Rep.1~~	~~5.61~~	~~5.60~~	~~5.65~~	~~5.7[illegible]~~		
~~AZ silty clay loam Rep.1~~	~~6.05~~	~~5.92~~	~~6.00~~	~~5.99~~	~~7.47~~	~~7.46~~
MS Clay Rep.1	7.25	7.24	7.16	7.10	7.10	7.10
MD Sand Rep.1	5.92	5.72	5.64	5.60	5.52	5.50
CA Sandy Loam Rep.1	6.39	6.42	6.40	6.41	6.43	6.46
WA Loam Rep.1	7.11	7.05	7.00	7.00	7.01	7.02
AZ Silty Clay Loam Rep.1	7.19	7.16	7.10	7.23	7.12	7.24

*

8/17/95

① prior to addition to soil — subsample not added to any soil tube [redacted] 8/17/95

AGI-L007b-9/17/91

* Insufficient pH probe recovery time all pH measurements repeated [redacted] 8/17/95

Example Calculations
Definitive Phase Adsorption

(A) **PPM** = $\frac{\text{Avg dpm/mL}}{\text{180042 dpm/}\mu\text{g}}$
WATER

Example: MS Clay 0.20 ppm REP 1

$$\frac{\text{29273 DPM/mL}}{\text{180042 DPM }\mu\text{g}} = 0.163 \text{ ppm}$$

(B) CARRY OVER DPM = WATER (g) * (1 mL/1 g) * AVG DPM/mL

Example: Ms Clay 0.20 ppm REP 1

0.971 * 29273 = 28424 DPM

(C) DPM FOR DESORPTION = DPM DOSED - TOTAL DPM ADSORPTION SOLUTION

Example: Ms Clay 0.20 ppm REP 1

740253 - 585453 = 154800 dpm

(D) **PPM SOIL** = DPM FOR DESORPTION / DRY SOIL g / 180042 DPM/μg

Example: MS Clay 0.20 ppm REP 1

154800 / 1.180 / 180042 = 0.729
ppm Soil

Example Calculations
Definitive Phase Desorption

(A) **CARRY OVER / mL** = ADSORPTION AVG DPM/ML / 20 mL SAMPLE VOLUME * Water(g)
Example: MS Clay 0.20 ppm REP 1

29273 / 20 * 0.971 = 1421 dpm/mL

(B) **DPM/mL - CARRYOVR** = AVG DPM/mL - CARRY OVER DPM/mL
Example: Ms Clay 0.20 ppm REP 1

6698 - 1421 = 5277 DPM

(C) **PPM WATER** = (DPM/mL - CARRYOVR) / 180042 DPM/µg
Example: Ms Clay 0.20 ppm REP 1

5277 / 180042 = 0.029 ppm

(D) DESORPTION SOIL
CARRY OVER DPM = WATER (g) * DESORPTION DPM/mL
Example: MS Clay 0.20 ppm REP 1

1.091 * 6698 = 7308 CARRY OVER DPM

(E) **DESORPTION SOIL**
PPM Soil = (AVG TOTAL DPM - D) / DRY SOIL g/ 180042 DPM/µg
Example: MS Clay 0.20 ppm REP 1

(74875 - 7308) / 1.180 / 180042 = 0.318 PPM SOIL

(F) Percent of Dose

Soil =
(SOIL PPM * DESORPTION DRY g * 180042 DPM/µg) / DPM DOSED * 100
Example: MS Clay 0.20 ppm REP 1

(0.318 * 1.180 * 180042) / 740253 * 100 = 9.1 % OF DOSE

Water
Adsorption = AVG DPM/mL * 20 mL / DPM DOSED * 100
Example: MS Clay 0.20 ppm REP 1

29273 * 20/740253 * 100 = 79.1 % OF DOSE

Desorption = (AVG DPM/mL - Carryovr) * 20/DOSE DPM * 100
Example: MS Clay 0.20 ppm REP 1

(6698 - 1421) * 20/740253 * 100 = 14.3% OF DOSE

TOTAL PERCENT OF DOSE = SOIL + WATER ADSORPTION + WATER DESORPTION
9.1 + 79.1 + 14.3 = 102.5% OF DOSE

AGRISEARCH INCORPORATED

DEFINITIVE CGA-329351 ADSORPTION/DESORPTION 12220

DOSE CALCULATIONS
SPECIFIC ACTIVITY 180042 DPM/µg
ALIQUOT = 500 µL

NOMINAL µg/mL	LS3801 DPM	DPM/mL	AVG DPM/mL	ACTUAL µg/mL	%VAR
0.2	18333	36666	37013	0.206	-0.94
	18528	37056			0.12
	18658	37316			0.82
0.5	46263	92526	92349	0.513	0.19
	46219	92438			0.10
	46041	92082			-0.29
1.0	92486	184972	185071	1.028	-0.05
	92606	185212			0.08
	92514	185028			-0.02
5.0	470291	940582	926324	5.145	1.54
	459285	918570			-0.84
	459910	919820			-0.70
10.0	915158	1830316	1839559	10.217	-0.50
	922611	1845222			0.31
	921569	1843138			0.19

9.7.95

AGRISEARCH INCORPORATED 12220
MS CLAY

DEFINITIVE
S.A 180042 DPM/µg

SAMPLE PPM	REP	ADSORPTION TUBE + DRY SOIL g	 TUBE TARE g	 TUBE + WET SOIL g	 DRY SOIL g	 WATER (g)	% MOIST.	DESORPTION TUBE + WET SOIL g	 WATER (g)	 DRY SOIL g
ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANTING										
0.0	1	37.501	36.316	38.525	1.185	1.024	46.36	38.667	1.166	1.185
	2	36.476	35.241	37.554	1.235	1.078	46.61	37.691	1.215	1.235
0.2	1	41.986	40.806	42.957	1.180	0.971	45.14	43.077	1.091	1.180
	2	36.446	35.310	37.348	1.136	0.902	44.26	37.525	1.079	1.136
0.5	1	35.957	34.774	36.999	1.183	1.042	46.83	37.074	1.117	1.183
	2	36.666	35.503	37.629	1.163	0.963	45.30	37.756	1.090	1.163
1.0	1	36.401	35.340	37.263	1.061	0.862	44.83	37.428	1.027	1.061
	2	36.874	35.733	37.812	1.141	0.938	45.12	38.026	1.152	1.141
5.0	1	36.444	35.223	37.480	1.221	1.036	45.90	37.651	1.207	1.221
	2	35.672	34.542	36.622	1.130	0.950	45.67	36.818	1.146	1.130
10.0	1	35.633	34.478	36.619	1.155	0.986	46.05	36.769	1.136	1.155
	2	35.967	34.908	36.895	1.059	0.928	46.70	37.046	1.079	1.059

8.28.95 5.1.2e Woo

AGRISEARCH INCORPORATED 12220

DEFINITIVE ADSORPTION

MS CLAY

S.A. 180042 DPM/µg

SAMPLE VOL: mL 20

SAMPLE PPM	REP	DPM DOSED	ALIQ mL	LS3801 DPM	DPM/mL	AVG DPM/mL	TOTAL DPM	PPM WATER
0.0	1	0	0.5	14	28	11	213	0.000
			0.5	2	4			
			0.5	0	0			
	2	0	0.5	0	0	6	120	0.000
			0.5	0	0			
			0.5	9	18			
0.2	1	740253	0.5	14638	29276	29273	585453	0.163
			0.5	14691	29382			
			0.5	14580	29160			
	2	740253	0.5	14803	29606	29703	594053	0.165
			0.5	14882	29764			
			0.5	14869	29738			
0.5	1	1846973	0.5	36156	72312	72763	1455267	0.404
			0.5	36319	72638			
			0.5	36670	73340			
	2	1846973	0.5	36503	73006	73112	1462240	0.406
			0.5	36495	72990			
			0.5	36670	73340			
1.0	1	3701413	0.5	73280	146560	146931	2938613	0.816
			0.5	73521	147042			
			0.5	73595	147190			
	2	3701413	0.5	72078	144156	143878	2877560	0.799
			0.5	72003	144006			
			0.5	71736	143472			
5.0	1	18526480	0.5	270122	540244	542283	10845667	3.012
			0.5	270966	541932			
			0.5	272337	544674			
	2	18526480	0.5	277644	555288	556823	11136453	3.093
			0.5	279144	558288			
			0.5	278446	556892			
10.0	1	36791173	0.5	431354	862708	861559	17231187	4.785
			0.5	430580	861160			
			0.5	430405	860810			
	2	36791173	0.5	444949	889898	893069	17861373	4.960
			0.5	446061	892122			
			0.5	448593	897186			

SAMPLE PPM	REP	DPM NOT ADSORBED	CARRY OVER DPM		DPM FOR DESORPT	PPM SOIL
0.0	1	213	11	CALCULATE BY	-213	-0.001
	2	120	6	DIFFERENCE	-120	-0.001
0.2	1	585453	28424		154800	0.729
	2	594053	26792		146200	0.715
0.5	1	1455267	75819		391707	1.839
	2	1462240	70407		384733	1.837
1.0	1	2938613	126654		762800	3.993
	2	2877560	134958		823853	4.010
5.0	1	10845667	561806		7680813	34.940
	2	11136453	528982		7390027	36.324
10.0	1	17231187	849498		19559987	94.062
	2	17861373	828768		18929800	99.283

8-25-95 5.1.2.e Woo

AGRISEARCH INCORPORATED 12220
DEFINITIVE DESORPTION MS CLAY
S.A: 180042 DPM/µg SAMPLE VOL: 20mL 1 mL ALIQ

SAMPLE PPM	REP	DPM FOR DESORB	LS3801 DPM	DPM/mL	AVG DPM/mL	CARRY OVER /mL	DPM/mL-CARRYOVR	PPM WATER
0.0	1	0	0	0	0	1	0	0.000
			0	0				
			0	0				
	2	0	0	0	0	0	0	0.000
			0	0				
			1	0				
0.2	1	154800	6695	6695	6698	1421	5277	0.029
			6692	6692				
			6708	6708				
	2	146200	6518	6518	6467	1340	5128	0.028
			6595	6595				
			6289	6289				
0.5	1	391707	17273	17273	17112	3791	13321	0.074
			17014	17014				
			17050	17050				
	2	384733	16385	16385	16525	3520	13005	0.072
			16565	16565				
			16626	16626				
1.0	1	762800	32059	32059	33059	6333	26726	0.148
			33056	33056				
			34062	34062				
	2	823853	34900	34900	34231	6748	27483	0.153
			34199	34199				
			33595	33595				
5.0	1	7680813	233054	233054	232996	28090	204905	1.138
			231290	231290				
			234643	234643				
	2	7390027	228105	228105	229882	26449	203433	1.130
			233799	233799				
			227743	227743				
10.0	1	19559987	401478	401478	409492	42475	367017	2.039
			417669	417669				
			409329	409329				
	2	18929800	418328	418328	418211	41438	376773	2.093
			420456	420456				
			415850	415850				

835.95 [redacted]

DEFINITIVE DESORPTION
COMBUSTIONS
S.A: 180042 DPM/μg

MS CLAY

SAMPLE PPM	REP	BMO ALIQ. g	LS3801 DPM	DPM/g	WET SOIL g	AVG TOT DPM	CARRY OVER DPM	PPM SOIL
		SOIL EFF	0.9826 *	MACHINE EFF	0.9878			
0.0	1	0.051	37	0	2.351	0	0	0.000
		0.065	0	0				
		0.054	0	0				
	2	0.056	0	0	2.45	0	0	0.000
		0.067	0	0				
		0.063	0	0				
0.2	1	0.070	2294	33352	2.271	74875	7308	0.318
		0.060	1932	32771				
		0.051	1643	32787				
	2	0.056	1809	32876	2.215	71643	6978	0.316
		0.055	1772	32789				
		0.067	2065	31367				
0.5	1	0.055	4529	83805	2.3	181180	19114	0.761
		0.054	4112	77498				
		0.059	4349	75019				
	2	0.062	4758	78102	2.253	169365	18013	0.723
		0.058	4169	73154				
		0.066	4816	74263				
1.0	1	0.054	7448	140371	2.088	308665	33952	1.438
		0.055	8439	156157				
		0.058	8375	146956				
	2	0.072	11654	164731	2.293	395982	39434	1.736
		0.054	8366	157673				
		0.057	10959	195672				
5.0	1	0.071	78371	1123386	2.428	3554238	281226	14.889
		0.056	93468	1698661				
		0.063	97157	1569515				
	2	0.056	92105	1673891	2.276	3737889	263445	17.078
		0.051	80817	1612740				
		0.063	101538	1640287				
10.0	1	0.064	353521	5621693	2.291	12674611	465183	58.714
		0.054	290565	5476227				
		0.063	340410	5499126				
	2	0.062	363289	5963380	2.138	12645309	451250	63.956
		0.068	383811	5744344				
		0.051	302470	6035928				

* DPM/g = LS3801 DPM/BMO ALIQ./SOIL EFF

5.1.2.e Woo [redacted] 3/14/95

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                  AGRISEARCH INCORPORATED          12220
                        MS CLAY
      S.A: DPM/µg  180042        SAMPLE VOL:   mL    20
--------------------------------------------------------------------
SAMPLE                 ====SOIL=====          =====WATER=====
PPM     REP             ppm    LN ppm            ppm    LN ppm
--------------------------------------------------------------------

DEFINITIVE ADSORPTION

  0.2   1              0.729  -0.317             0.163  -1.817
        2              0.715  -0.336             0.165  -1.802
  0.5   1              1.839   0.609             0.404  -0.906
        2              1.837   0.608             0.406  -0.901
  1.0   1              3.993   1.385             0.816  -0.203
        2              4.010   1.389             0.799  -0.224
  5.0   1             34.940   3.554             3.012   1.103
        2             36.324   3.592             3.093   1.129
 10.0   1             94.062   4.544             4.785   1.566
        2             99.283   4.598             4.960   1.601
LINEAR REGRESSION ANALYSIS
      Regression Output:
Constant               2.02853
Std Err of Y Est       0.25551          CORRELATION        0.9922
R Squared              0.98446          SLOPE (1/n)         1.449
No. of Observation          10          INTERCEPT           2.029
Degrees of Freedom           8                   n          0.690
                                                 Kd           7.6
X Coefficient(s)       1.44893                   Koc          570
Std Err of Coef.       0.06437                   % O.C.     1.334
--------------------------------------------------------------------
SAMPLE      ====SOIL=====  =====WATER====    ====PERCENT OF DOSE=====
PPM     REP   ppm    LN ppm   ppm   LN ppm     SOIL    ---WATER--- TOTAL
                                                         ADS   DES
--------------------------------------------------------------------
DEFINITIVE DESORPTION
  0.2   1    0.318  -1.146  0.029  -3.530      9.1    79.1  14.3 102.5
        2    0.316  -1.151  0.028  -3.559      8.7    80.3  13.9 102.8
  0.5   1    0.761  -0.273  0.074  -2.604      8.8    78.8  14.4 102.0
        2    0.723  -0.325  0.072  -2.628      8.2    79.2  14.1 101.4
  1.0   1    1.438   0.363  0.148  -1.908      7.4    79.4  14.4 101.3
        2    1.736   0.551  0.153  -1.880      9.6    77.7  14.9 102.2
  5.0   1   14.889   2.701  1.138   0.129     17.7    58.5  22.1  98.3
        2   17.078   2.838  1.130   0.122     18.8    60.1  22.0 100.8
 10.0   1   58.714   4.073  2.039   0.712     33.2    46.8  20.0 100.0
        2   63.956   4.158  2.093   0.738     33.1    48.5  20.5 102.2
LINEAR REGRESSION ANALYSIS                                        ======
      Regression Output:                              AVG % DOSE  101.4
Constant                2.9102                                    ======
Std Err of Y Est       0.26685          CORRELATION        0.9926
R Squared              0.98523          SLOPE (1/n)         1.202
No. of Observation          10          INTERCEPT           2.910
Degrees of Freedom           8                   n          0.832
                                                 Kd          18.4
X Coefficient(s)       1.20187                   Koc         1376
Std Err of Coef.       0.05202                   % O.C.     1.334
--------------------------------------------------------------------
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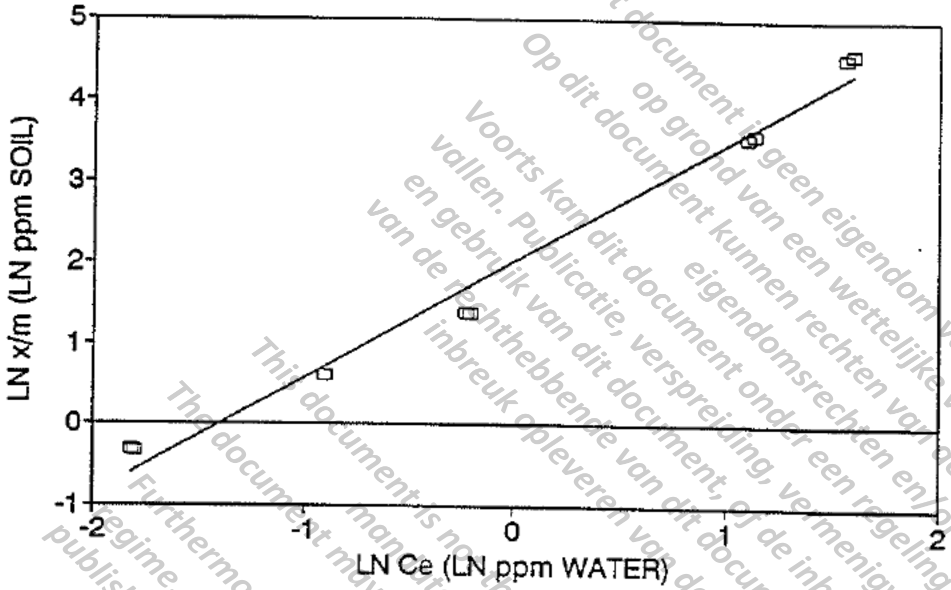
9.7.95

ADSORPTION ISOTHERM
MISSISSIPPI CLAY



8·25·95

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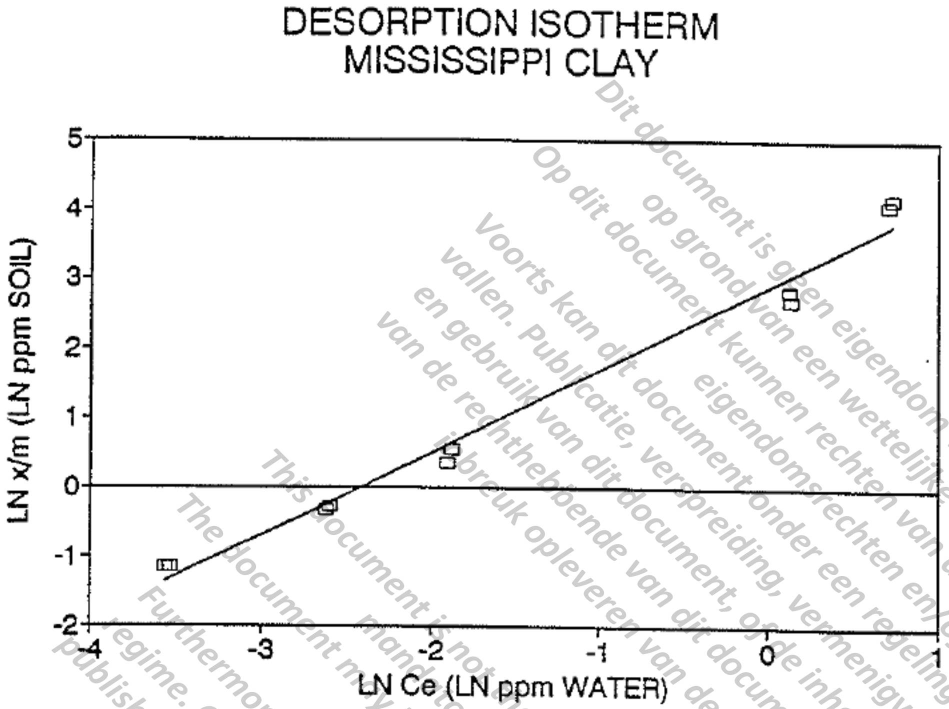

8.25.95 [redacted]

AGRISEARCH INCORPORATED 12220

DEFINITIVE MD SAND

S.A 180042 DPM/µg

SAMPLE PPM	REP	ADSORPTION TUBE + DRY SOIL	 TUBE TARE	 TUBE + WET SOIL	 DRY SOIL	 WATER (g)	% MOIST.	DESORPTION TUBE + WET SOIL	 WATER (g)	 DRY SOIL
		g	g	g	g			g		g
ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANTING										
0.0	1	46.261	36.153	49.518	10.108	3.257	24.37	49.762	3.501	10.108
	2	46.252	36.116	49.679	10.136	3.427	25.27	49.610	3.358	10.136
0.2	1	45.200	35.134	48.204	10.066	3.004	22.98	48.366	3.166	10.066
	2	43.905	33.804	46.859	10.101	2.954	22.63	47.018	3.113	10.101
0.5	1	45.875	35.749	48.929	10.126	3.054	23.17	48.946	3.071	10.126
	2	45.570	35.423	48.613	10.147	3.043	23.07	48.461	2.891	10.147
1.0	1	43.754	33.600	46.753	10.154	2.999	22.80	46.733	2.979	10.154
	2	45.037	34.966	47.958	10.071	2.921	22.48	47.981	2.944	10.071
5.0	1	45.839	35.768	48.950	10.071	3.111	23.60	48.713	2.874	10.071
	2	44.647	34.431	47.675	10.216	3.028	22.86	47.645	2.998	10.216
10.0	1	44.058	33.940	47.083	10.118	3.025	23.02	46.987	2.929	10.118
	2	46.165	36.079	49.235	10.086	3.070	23.34	49.029	2.864	10.086

8.28.95 [redacted]

DEFINITIVE ADSORPTION AGRISEARCH INCORPORATED 12220
MD SAND
S.A. 180042 DPM/µg SAMPLE VOL: mL 20

SAMPLE PPM	REP	DPM DOSED	ALIQ mL	LS3801 DPM	DPM/mL	AVG DPM/mL	TOTAL DPM	PPM WATER
0.0	1	0	0.5	5	0	0	0	0.000
			0.5	0	0			
			0.5	2	0			
	2	0	0.5	1	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
0.2	1	740253	0.5	17697	35394	35705	714093	0.198
			0.5	17882	35764			
			0.5	17978	35956			
	2	740253	0.5	17662	35324	35577	711533	0.198
			0.5	17813	35626			
			0.5	17890	35780			
0.5	1	1846973	0.5	45149	90298	89513	1790253	0.497
			0.5	44409	88818			
			0.5	44711	89422			
	2	1846973	0.5	44631	89262	88843	1776853	0.493
			0.5	43746	87492			
			0.5	44887	89774			
1.0	1	3701413	0.5	89419	178838	176733	3534667	0.982
			0.5	88470	176940			
			0.5	87211	174422			
	2	3701413	0.5	89004	178008	177772	3555440	0.987
			0.5	88958	177916			
			0.5	88696	177392			
5.0	1	18526480	0.5	448649	897298	898113	17962267	4.988
			0.5	448898	897796			
			0.5	449623	899246			
	2	18526480	0.5	444854	889708	893465	17869293	4.963
			0.5	447896	895792			
			0.5	447447	894894			
10.0	1	36791173	0.5	900480	1800960	1794357	35887147	9.966
			0.5	889604	1779208			
			0.5	901452	1802904			
	2	36791173	0.5	902147	1804294	1801305	36026107	10.005
			0.5	898570	1797140			
			0.5	901241	1802482			

SAMPLE PPM	REP	DPM NOT ADSORBED	CARRY OVER DPM		DPM FOR DESORPT	PPM SOIL
0.0	1	0	0	CALCULATE BY DIFFERENCE	0	0.000
	2	0	0		0	0.000
0.2	1	714093	107257		26160	0.014
	2	711533	105093		28720	0.016
0.5	1	1790253	273372		56720	0.031
	2	1776853	270348		70120	0.038
1.0	1	3534667	530023		166747	0.091
	2	3555440	519272		145973	0.081
5.0	1	17962267	2794031		564213	0.311
	2	17869293	2705411		657187	0.357
10.0	1	35887147	5427931		904027	0.496
	2	36026107	5530007		765067	0.421

5.1.2.e Woo [redacted] 9/14/95

AGRISEARCH INCORPORATED 12220

DEFINITIVE DESORPTION
MD SAND
S.A: 180042 DPM/μg
SAMPLE VOL: 20mL
1 mL ALIQ

SAMPLE PPM	REP	DPM FOR DESORB	LS3801 DPM	DPM/mL	AVG DPM/mL	CARRY OVER /mL	DPM/mL-CARRYOVR	PPM WATER
0.0	1	0	0	0	0	1	0	0.000
			0	0				
			0	0				
	2	0	0	0	0	0	0	0.000
			0	0				
			0	0				
0.2	1	26160	6002	6002	6161	5363	798	0.004
			6316	6316				
			6166	6166				
	2	28720	6097	6097	6025	5255	770	0.004
			5984	5984				
			5994	5994				
0.5	1	56720	15559	15559	15425	13669	1756	0.010
			15444	15444				
			15271	15271				
	2	70120	15060	15060	14776	13517	1259	0.007
			14216	14216				
			15053	15053				
1.0	1	166747	28628	28628	29966	26501	3465	0.019
			31091	31091				
			30180	30180				
	2	145973	29496	29496	29520	25964	3557	0.020
			30259	30259				
			28806	28806				
5.0	1	564213	148231	148231	148263	139702	8561	0.048
			147120	147120				
			149438	149438				
	2	657187	150648	150648	148996	135271	13725	0.076
			151267	151267				
			145073	145073				
10.0	1	904027	291435	291435	293312	271397	21915	0.122
			278653	278653				
			309847	309847				
	2	765067	303697	303697	298277	276500	21777	0.121
			299842	299842				
			291293	291293				

8.25.95

[redacted] 5.1.2.e Woo

DEFINITIVE DESORPTION
COMBUSTIONS

MD SAND

S.A: 180042 DPM/μg

SAMPLE PPM	REP	BMO ALIQ. g	LSC DPM	DPM/g	WET SOIL g	AVG TOT DPM	CARRY OVER DPM	PPM SOIL
		SOIL EFF	0.9467 *	MACHINE EFF		0.9661		
0.0	1	0.253	0	0	13.609	347	0	0.000
		0.252	10	0				
		0.235	17	76				
	2	0.237	0	0	13.494	335	0	0.000
		0.244	0	0				
		0.213	15	74				
0.2	1	0.263	708	2844	13.232	43072	19507	0.013
		0.232	782	3561				
		0.264	840	3361				
	2	0.209	678	3427	13.214	42524	18756	0.013
		0.257	722	2968				
		0.244	753	3260				
0.5	1	0.219	1543	7443	13.197	93224	47369	0.025
		0.243	1560	6781				
		0.218	1438	6968				
	2	0.206	1289	6610	13.038	86958	42718	0.024
		0.212	1270	6328				
		0.235	1573	7071				
1.0	1	0.245	3036	13090	13.133	171254	89270	0.045
		0.222	2913	13861				
		0.229	2638	12169				
	2	0.235	2822	12685	13.015	166157	86908	0.044
		0.214	2576	12716				
		0.204	2491	12899				
5.0	1	0.232	12675	57712	12.945	802927	426108	0.208
		0.224	14106	66522				
		0.209	12236	61844				
	2	0.229	11428	52716	13.214	754269	446690	0.167
		0.223	11756	55688				
		0.236	14039	62839				
10.0	1	0.248	27603	117574	13.047	1565780	859110	0.388
		0.208	24940	126660				
		0.256	28063	115798				
	2	0.224	24371	114930	12.95	1452043	854266	0.329
		0.211	23098	115638				
		0.254	25443	105814				

* DPM/g = LS3801 DPM/BMO ALIQ./SOIL EFF

8.25.95

5.1.2.e Woo

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                    AGRISEARCH INCORPORATED          12220
                              MD SAND
      S.A: DPM/µg  180042          SAMPLE VOL:   mL    20
--------------------------------------------------------------------
SAMPLE                 ====SOIL=====           =====WATER=====
PPM    REP              ppm   LN ppm             ppm   LN ppm
--------------------------------------------------------------------

DEFINITIVE ADSORPTION

  0.2   1              0.014 -4.238            0.198 -1.618
        2              0.016 -4.148            0.198 -1.622
  0.5   1              0.031 -3.470            0.497 -0.699
        2              0.038 -3.260            0.493 -0.706
  1.0   1              0.091 -2.395            0.982 -0.019
        2              0.081 -2.519            0.987 -0.013
  5.0   1              0.311 -1.167            4.988  1.607
        2              0.357 -1.029            4.963  1.602
 10.0   1              0.496 -0.701            9.966  2.299
        2              0.421 -0.864           10.005  2.303
LINEAR REGRESSION ANALYSIS
      Regression Output:
Constant              -2.6589
Std Err of Y Est      0.18638         CORRELATION        0.9918
R Squared             0.98363         SLOPE (1/n)         0.892
No. of Observation         10         INTERCEPT          -2.659
Degrees of Freedom          8                n            1.121
                                             Kd             0.1
X Coefficient(s)      0.89186                Koc             20
Std Err of Coef.      0.04068                % O.C.       0.348
----------------------------------------------------------------------
SAMPLE       ====SOIL=====   =====WATER====    ====PERCENT OF DOSE=====
PPM     REP    ppm   LN ppm    ppm   LN ppm     SOIL   ---WATER--- TOTAL
                                                        ADS   DES
----------------------------------------------------------------------
DEFINITIVE DESORPTION
 0.2    1    0.013 -4.343  0.004 -5.418    3.2    96.5  2.2 101.8
        2    0.013 -4.337  0.004 -5.454    3.2    96.1  2.1 101.4
 0.5    1    0.025 -3.683  0.010 -4.630    2.5    96.9  1.9 101.3
        2    0.024 -3.721  0.007 -4.963    2.4    96.2  1.4 100.0
 1.0    1    0.045 -3.105  0.019 -3.950    2.2    95.5  1.9  99.6
        2    0.044 -3.130  0.020 -3.924    2.1    96.1  1.9 100.1
 5.0    1    0.208 -1.571  0.048 -3.046    2.0    97.0  0.9  99.9
        2    0.167 -1.788  0.076 -2.574    1.7    96.5  1.5  99.6
10.0    1    0.388 -0.947  0.122 -2.106    1.9    97.5  1.2 100.7
        2    0.329 -1.111  0.121 -2.112    1.6    97.9  1.2 100.7
LINEAR REGRESSION ANALYSIS                                      ======
      Regression Output:                          AVG % DOSE    100.5
Constant              1.01164                                   ======
Std Err of Y Est      0.22995         CORRELATION        0.9862
R Squared             0.97251         SLOPE (1/n)         0.991
No. of Observation         10         INTERCEPT           1.012
Degrees of Freedom          8                n            1.009
                                             Kd             2.8
X Coefficient(s)      0.99146                Koc            790
Std Err of Coef.      0.05894                % O.C.       0.348
----------------------------------------------------------------------
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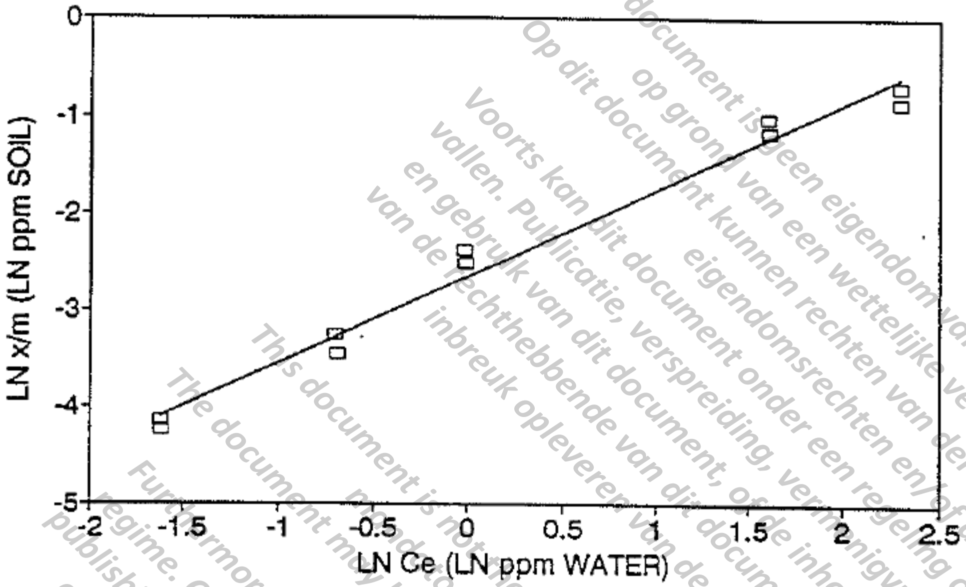
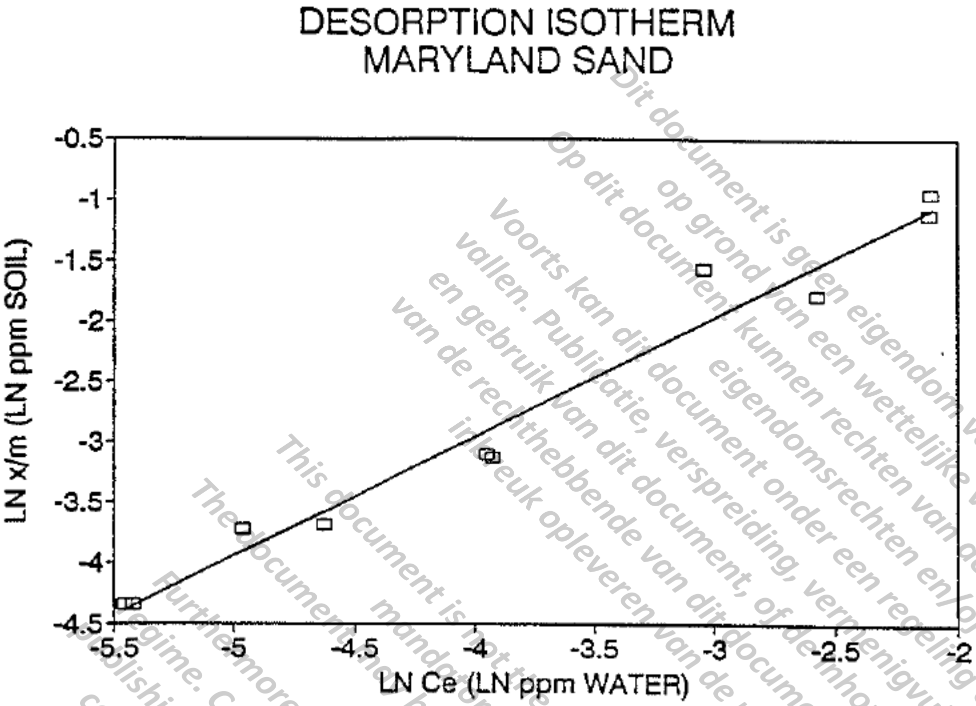
9-7-95

ADSORPTION ISOTHERM
MARYLAND SAND



8· 5.1.2.e Woo 95


DESORPTION ISOTHERM
MARYLAND SAND
LN x/m (LN ppm SOIL)
-0.5
-1
-1.5
-2
-2.5
-3
-3.5
-4
-4.5
-5.5
-5
-4.5
-4
-3.5
-3
-2.5
-2
LN Ce (LN ppm WATER)

[redacted]

DEFINITIVE
S.A 180042 DPM/µg

AGRISEARCH INCORPORATED
CA SANDY LOAM

12220

SAMPLE PPM	REP	ADSORPTION TUBE + DRY SOIL (g)	TUBE TARE (g)	TUBE + WET SOIL (g)	DRY SOIL (g)	WATER (g)	% MOIST.	DESORPTION TUBE + WET SOIL (g)	WATER (g)	DRY SOIL (g)
ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANTING										
0.0	1	46.517	36.334	49.928	10.183	3.411	25.09	49.841	3.324	10.183
	2	45.610	35.490	49.006	10.120	3.396	25.13	48.883	3.273	10.120
0.2	1	42.734	32.583	46.097	10.151	3.363	24.89	45.973	3.239	10.151
	2	45.396	35.165	48.731	10.231	3.335	24.58	48.560	3.164	10.231
0.5	1	45.203	35.141	48.537	10.062	3.334	24.89	48.295	3.092	10.062
	2	46.225	36.045	49.603	10.180	3.378	24.92	49.963	3.738	10.180
1.0	1	45.983	35.864	49.346	10.119	3.363	24.94	49.072	3.089	10.119
	2	45.236	35.199	48.611	10.037	3.375	25.16	48.363	3.127	10.037
5.0	1	45.829	35.751	49.150	10.078	3.321	24.79	49.043	3.214	10.078
	2	45.536	35.234	48.967	10.302	3.431	24.98	48.779	3.243	10.302
10.0	1	45.706	35.505	49.056	10.201	3.350	24.72	48.968	3.262	10.201
	2	43.167	33.025	46.486	10.142	3.319	24.66	46.408	3.241	10.142

8.28.95 [redacted]

DEFINITIVE ADSORPTION
CA SANDY LOAM
S.A. 180042 DPM/µg
SAMPLE VOL: mL 20

SAMPLE PPM	REP	DPM DOSED	ALIQ mL	LS3801 DPM	DPM/mL	AVG DPM/mL	TOTAL DPM	PPM WATER
0.0	1	0	0.5	0	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
	2	0	0.5	3	6	2	40	0.000
			0.5	0	0			
			0.5	0	0			
0.2	1	740253	0.5	16965	33930	34272	685440	0.190
			0.5	17248	34496			
			0.5	17195	34390			
	2	740253	0.5	16895	33790	34029	680573	0.189
			0.5	17107	34214			
			0.5	17041	34082			
0.5	1	1846973	0.5	43335	86670	85609	1712173	0.475
			0.5	42711	85422			
			0.5	42367	84734			
	2	1846973	0.5	42892	85784	85579	1711573	0.475
			0.5	42819	85638			
			0.5	42657	85314			
1.0	1	3701413	0.5	84450	168900	170657	3413147	0.948
			0.5	85217	170434			
			0.5	86319	172638			
	2	3701413	0.5	85049	170098	170825	3416493	0.949
			0.5	85508	171016			
			0.5	85680	171360			
5.0	1	18526480	0.5	432232	864464	865430	17308600	4.807
			0.5	431140	862280			
			0.5	434773	869546			
	2	18526480	0.5	429353	858706	861674	17233480	4.786
			0.5	432044	864088			
			0.5	431114	862228			
10.0	1	36791173	0.5	851812	1703624	1703104	34062080	9.459
			0.5	854656	1709312			
			0.5	848188	1696376			
	2	36791173	0.5	854372	1708744	1714920	34298400	9.525
			0.5	865427	1730854			
			0.5	852581	1705162			

SAMPLE PPM	REP	DPM NOT ADSORBED	CARRY OVER DPM		DPM FOR DESORPT	PPM SOIL
0.0	1	0	0	CALCULATE BY	0	0.000
	2	40	7	DIFFERENCE	0	0.000
0.2	1	685440	115257		54813	0.030
	2	680573	113486		59680	0.032
0.5	1	1712173	285419		134800	0.074
	2	1711573	289085		135400	0.074
1.0	1	3413147	573921		288267	0.158
	2	3416493	576533		284920	0.158
5.0	1	17308600	2874093		1217880	0.671
	2	17233480	2956403		1293000	0.697
10.0	1	34062080	5705398		2729093	1.486
	2	34298400	5691819		2492773	1.365

8-25-95 5.1.2.e Woo

AGRISEARCH INCORPORATED 12220

DEFINITIVE DESORPTION CA SANDY LOAM

S.A: 180042 DPM/µg SAMPLE VOL: 20mL 1 mL ALIQ

SAMPLE PPM	REP	DPM FOR DESORB	LS3801 DPM	DPM/mL	AVG DPM/mL	CARRY OVER /mL	DPM/mL- CARRYOVR	PPM WATER
0.0	1	0	0	0	0	0	0	0.000
			0	0				
			0	0				
	2	0	0	0	0	0	0	0.000
			0	0				
			0	0				
0.2	1	54813	6562	6562	6953	5763	1190	0.007
			7322	7322				
			6975	6975				
	2	59680	7003	7003	6828	5674	1154	0.006
			6998	6998				
			6484	6484				
0.5	1	134800	16247	16247	16820	14271	2549	0.014
			17285	17285				
			16929	16929				
	2	135400	17333	17333	16905	14454	2450	0.014
			16821	16821				
			16560	16560				
1.0	1	288267	31900	31900	33334	28696	4638	0.026
			34662	34662				
			33440	33440				
	2	284920	35049	35049	32570	28827	3744	0.021
			30504	30504				
			32158	32158				
5.0	1	1217880	163270	163270	164559	143705	20854	0.116
			164627	164627				
			165780	165780				
	2	1293000	172001	172001	166752	147820	18932	0.105
			161740	161740				
			166516	166516				
10.0	1	2729093	357332	357332	344189	285270	58919	0.327
			318146	318146				
			357089	357089				
	2	2492773	339493	339493	332511	284591	47920	0.266
			333078	333078				
			324963	324963				

8-25-95 [redacted] 5.1 2.e Woo

DEFINITIVE DESORPTION
COMBUSTIONS
S.A: 180042 DPM/µg

CA SANDY LOAM

SAMPLE PPM	REP	BMO ALIQ. g	LS3801 DPM	DPM/g	WET SOIL g	AVG TOT DPM	CARRY OVER DPM	PPM SOIL
		SOIL EFF	0.9567 *	MACHINE EFF		0.9620		
0.0	1	0.226	0	0	13.507	0	0	0.000
		0.247	0	0				
		0.245	0	0				
	2	0.206	0	0	13.393	0	0	0.000
		0.233	0	0				
		0.246	0	0				
0.2	1	0.232	822	3704	13.39	48481	22521	0.014
		0.254	877	3609				
		0.235	798	3549				
	2	0.247	813	3441	13.395	46010	21605	0.013
		0.215	725	3525				
		0.226	722	3339				
0.5	1	0.253	1954	8073	13.154	111223	52008	0.033
		0.259	2249	9077				
		0.237	1863	8217				
	2	0.215	1747	8493	13.918	116642	63190	0.029
		0.225	1811	8413				
		0.255	2009	8235				
1.0	1	0.228	3472	15918	13.208	217247	102969	0.063
		0.258	3735	15132				
		0.231	4043	18295				
	2	0.221	3417	16162	13.164	219329	101847	0.065
		0.245	4316	18414				
		0.216	3184	15408				
5.0	1	0.233	16702	74928	13.292	1024711	528893	0.273
		0.237	18955	83600				
		0.246	17121	72749				
	2	0.229	15942	72768	13.545	1048957	540778	0.274
		0.218	18207	87300				
		0.224	15485	72260				
10.0	1	0.234	32464	145017	13.463	1979480	1122745	0.466
		0.218	30242	145006				
		0.243	35120	151071				
	2	0.215	32253	156806	13.383	1983202	1077669	0.496
		0.259	34828	140560				
		0.214	30136	147199				

* DPM/g = LS3801 DPM/BMO ALIQ./SOIL EFF

9.7.95

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                    AGRISEARCH INCORPORATED          12220
                        CA SANDY LOAM
       S.A: DPM/µg  180042          SAMPLE VOL:   mL    20
-------------------------------------------------------------------
SAMPLE                ====SOIL=====          =====WATER=====
PPM    REP              ppm   LN ppm             ppm    LN ppm
-------------------------------------------------------------------

DEFINITIVE ADSORPTION

  0.2  1                0.030 -3.507             0.190 -1.659
       2                0.032 -3.430             0.189 -1.666
  0.5  1                0.074 -2.598             0.475 -0.743
       2                0.074 -2.605             0.475 -0.744
  1.0  1                0.158 -1.844             0.948 -0.054
       2                0.158 -1.847             0.949 -0.053
  5.0  1                0.671 -0.399             4.807  1.570
       2                0.697 -0.361             4.786  1.566
 10.0  1                1.486  0.396             9.459  2.247
       2                1.365  0.311             9.525  2.254
LINEAR REGRESSION ANALYSIS
      Regression Output:
Constant               -1.852
Std Err of Y Est      0.05334          CORRELATION       0.9994
R Squared             0.99885          SLOPE (1/n)        0.970
No. of Observation         10          INTERCEPT         -1.852
Degrees of Freedom          8                n            1.031
                                             Kd             0.2
X Coefficient(s)      0.96989                Koc             68
Std Err of Coef.      0.01165                % O.C.       0.232
-------------------------------------------------------------------
SAMPLE       ====SOIL=====  =====WATER====  ====PERCENT OF DOSE=====
PPM    REP    ppm   LN ppm   ppm  LN ppm     SOIL   ---WATER--- TOTAL
                                                     ADS    DES
-------------------------------------------------------------------
DEFINITIVE DESORPTION
  0.2  1     0.014  -4.254  0.007 -5.019      3.5    92.6   3.2  99.3
       2     0.013  -4.324  0.006 -5.050      3.3    91.9   3.1  98.4
  0.5  1     0.033  -3.421  0.014 -4.257      3.2    92.7   2.8  98.7
       2     0.029  -3.535  0.014 -4.297      2.9    92.7   2.7  98.2
  1.0  1     0.063  -2.769  0.026 -3.659      3.1    92.2   2.5  97.8
       2     0.065  -2.733  0.021 -3.873      3.2    92.3   2.0  97.5
  5.0  1     0.273  -1.297  0.116 -2.156      2.7    93.4   2.3  98.4
       2     0.274  -1.295  0.105 -2.252      2.7    93.0   2.0  97.8
 10.0  1     0.466  -0.763  0.327 -1.117      2.3    92.6   3.2  98.1
       2     0.496  -0.701  0.266 -1.324      2.5    93.2   2.6  98.3
LINEAR REGRESSION ANALYSIS                                        =====
      Regression Output:                         AVG % DOSE       98.2
Constant              0.60019                                     =====
Std Err of Y Est      0.20169          CORRELATION       0.9907
R Squared             0.98151          SLOPE (1/n)        0.942
No. of Observation         10          INTERCEPT          0.600
Degrees of Freedom          8                n            1.061
                                             Kd             1.8
X Coefficient(s)      0.94212                Koc            785
Std Err of Coef.      0.04572                % O.C.       0.232
-------------------------------------------------------------------
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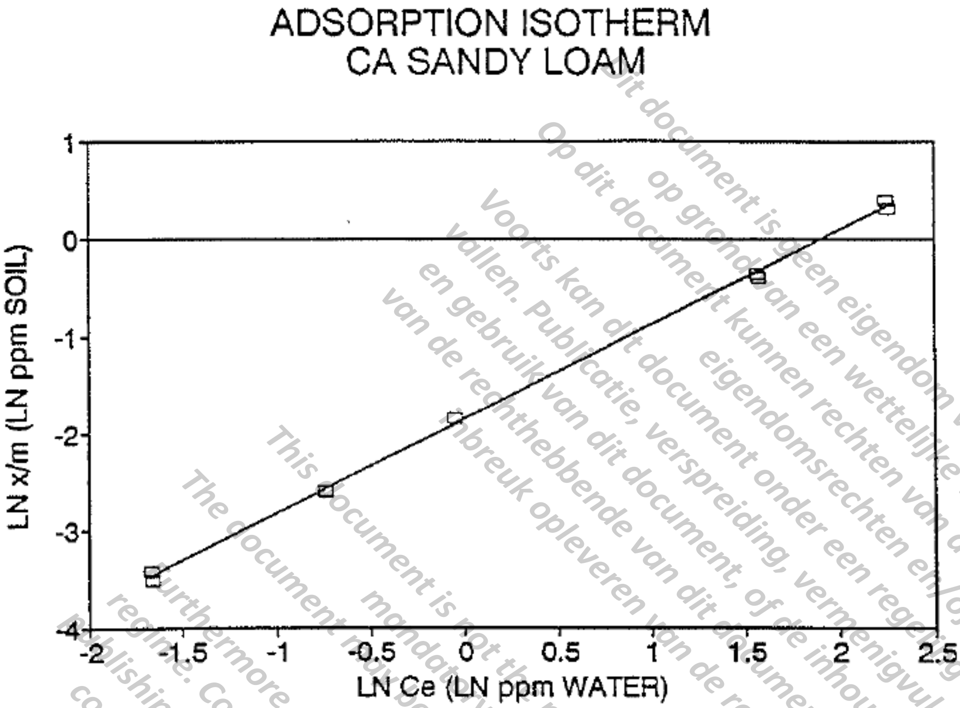

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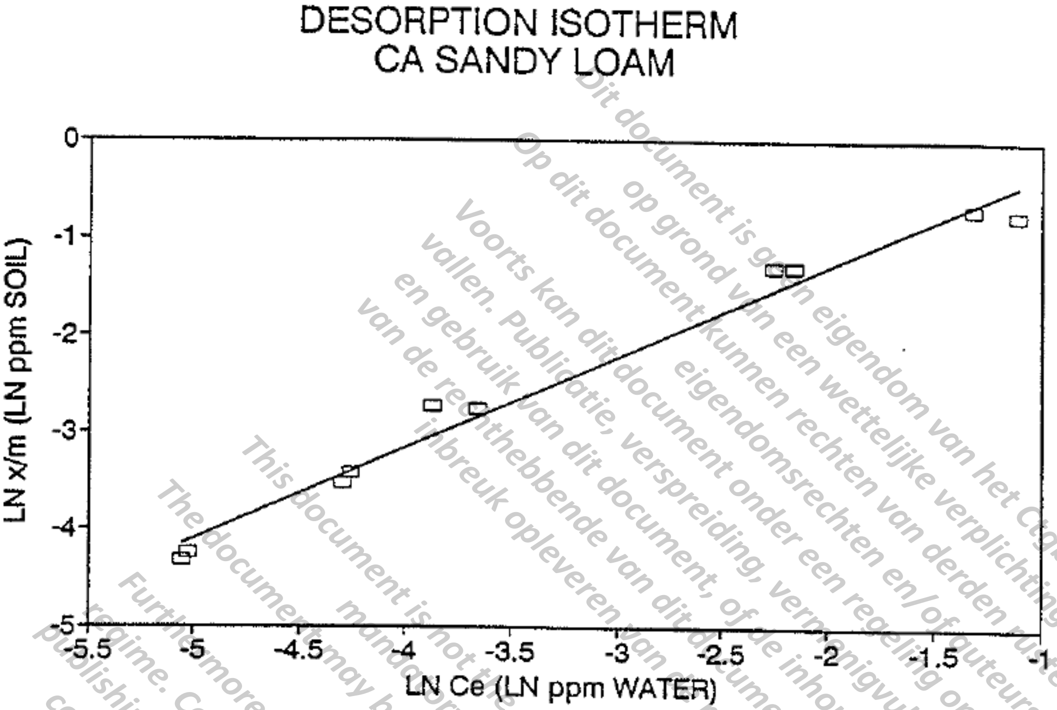

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AGRISEARCH INCORPORATED
WA LOAM

12220

DEFINITIVE
S.A 180042 DPM/µg

ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANTING

SAMPLE PPM	REP	ADSORPTION TUBE + DRY SOIL g	TUBE TARE g	TUBE + WET SOIL g	DRY SOIL g	WATER (g)	% MOIST.	DESORPTION TUBE + WET SOIL g	WATER (g)	DRY SOIL g
0.0	1	39.833	35.679	42.016	4.154	2.183	34.45	42.086	2.253	4.154
	2	40.005	35.970	42.133	4.035	2.128	34.53	42.174	2.169	4.035
0.2	1	39.012	34.971	41.228	4.041	2.216	35.42	41.052	2.040	4.041
	2	38.845	34.631	41.066	4.214	2.221	34.51	40.925	2.080	4.214
0.5	1	39.942	35.827	42.186	4.115	2.244	35.29	41.992	2.050	4.115
	2	40.047	35.964	42.194	4.083	2.147	34.46	42.076	2.029	4.083
1.0	1	39.962	35.754	42.162	4.208	2.200	34.33	42.059	2.097	4.208
	2	39.837	35.757	42.032	4.080	2.195	34.98	41.857	2.020	4.080
5.0	1	39.363	35.289	41.521	4.074	2.158	34.63	41.441	2.078	4.074
	2	38.985	34.721	41.242	4.264	2.257	34.61	41.129	2.144	4.264
10.0	1	40.731	36.548	43.001	4.183	2.270	35.18	42.974	2.243	4.183
	2	36.353	32.245	38.561	4.108	2.208	34.96	38.523	2.170	4.108

8.31.95

DEFINITIVE ADSORPTION
WA LOAM
S.A. 180042 DPM/μg
SAMPLE VOL: mL 20

SAMPLE PPM	REP	DPM DOSED	ALIQ mL	LSC DPM	DPM/mL	AVG DPM/mL	TOTAL DPM	PPM WATER
0.0	1	0	0.5	0	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
	2	0	0.5	0	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
0.2	1	740253	0.5	14931	29862	29673	593453	0.165
			0.5	14860	29720			
			0.5	14718	29436			
	2	740253	0.5	14153	28306	29001	580027	0.161
			0.5	14619	29238			
			0.5	14730	29460			
0.5	1	1846973	0.5	36963	73926	73772	1475440	0.410
			0.5	37041	74082			
			0.5	36654	73308			
	2	1846973	0.5	36812	73624	73899	1477973	0.410
			0.5	36976	73952			
			0.5	37060	74120			
1.0	1	3701413	0.5	73882	147764	147721	2954427	0.820
			0.5	73607	147214			
			0.5	74093	148186			
	2	3701413	0.5	73698	147396	147587	2951747	0.820
			0.5	73847	147694			
			0.5	73836	147672			
5.0	1	18526480	0.5	367335	734670	718132	14362640	3.989
			0.5	366258	732516			
			0.5	343605	687210			
	2	18526480	0.5	360272	720544	723232	14464640	4.017
			0.5	362434	724868			
			0.5	362142	724284			
10.0	1	36791173	0.5	701122	1402244	1402802	28056040	7.792
			0.5	699477	1398954			
			0.5	703604	1407208			
	2	36791173	0.5	703336	1406672	1407968	28159360	7.820
			0.5	705778	1411556			
			0.5	702838	1405676			

SAMPLE PPM	REP	DPM NOT ADSORBED	CARRY OVER DPM		DPM FOR DESORPT	PPM SOIL
0.0	1	0	0	CALCULATE BY	0	0.000
	2	0	0	DIFFERENCE	0	0.000
0.2	1	593453	65755		146800	0.202
	2	580027	64412		160227	0.211
0.5	1	1475440	165544		371533	0.501
	2	1477973	158660		369000	0.502
1.0	1	2954427	324987		746987	0.986
	2	2951747	323954		749667	1.021
5.0	1	14362640	1549729		4163840	5.677
	2	14464640	1632335		4061840	5.291
10.0	1	28056040	3184361		8735133	11.599
	2	28159360	3108793		8631813	11.671

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AGRISEARCH INCORPORATED

DEFINITIVE DESORPTION
WA LOAM

S.A: 180042 DPM/µg
SAMPLE VOL: 20mL
1 mL ALIQ

SAMPLE PPM	REP	DPM FOR DESORB	LSC DPM	DPM/mL	AVG DPM/mL	CARRY OVER /mL	DPM/mL-CARRYOVR	PPM WATER
0.0	1	0	0	0	0	0	0	0.000
			0	0				
			0	0				
	2	0	0	0	0	0	0	0.000
			0	0				
			2	0				
0.2	1	146800	7537	7537	7512	3288	4224	0.023
			7533	7533				
			7465	7465				
	2	160227	7776	7776	7749	3221	4529	0.025
			7934	7934				
			7538	7538				
0.5	1	371533	19185	19185	19330	8277	11053	0.061
			19020	19020				
			19786	19786				
	2	369000	19228	19228	19172	7933	11239	0.062
			18832	18832				
			19455	19455				
1.0	1	746987	37266	37266	37129	16249	20880	0.116
			37670	37670				
			36451	36451				
	2	749667	38368	38368	37420	16198	21223	0.118
			37602	37602				
			36291	36291				
5.0	1	4163840	193127	193127	192868	77486	115381	0.641
			194835	194835				
			190641	190641				
	2	4061840	196357	196357	195877	81617	114260	0.635
			197170	197170				
			194104	194104				
10.0	1	8735133	421923	421923	415674	159218	256456	1.424
			406573	406573				
			418527	418527				
	2	8631813	413344	413344	414055	155440	258616	1.436
			417361	417361				
			411461	411461				

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DEFINITIVE DESORPTION
COMBUSTIONS

WA LOAM

S.A: 180042 DPM/µg

SOIL EFF 0.9394 * MACHINE EFF 0.9779

SAMPLE PPM	REP	BMO ALIQ. g	LSC DPM	DPM/g	WET SOIL g	AVG TOT DPM	CARRY OVER DPM	PPM SOIL
0.0	1	0.209	0	0	6.407	0	0	0.000
		0.245	0	0				
		0.253	0	0				
0.0	2	0.232	0	0	6.204	0	0	0.000
		0.242	0	0				
		0.229	0	0				
0.2	1	0.234	2931	13333	6.081	80102	15324	0.089
		0.234	2794	12710				
		0.202	2557	13475				
	2	0.233	2705	12358	6.294	79715	16119	0.084
		0.212	2600	13055				
		0.229	2707	12583				
0.5	1	0.215	6256	30974	6.165	190170	39627	0.203
		0.246	7191	31116				
		0.203	5807	30450				
	2	0.236	6880	31032	6.112	186066	38899	0.200
		0.248	7128	30595				
		0.256	7143	29701				
1.0	1	0.246	13799	59710	6.305	366212	77860	0.381
		0.213	11378	56862				
		0.218	11812	57677				
	2	0.214	11899	59188	6.100	357264	75589	0.383
		0.224	12417	59007				
		0.227	12264	57510				
5.0	1	0.228	63691	297356	6.152	1797252	400779	1.904
		0.218	58526	285776				
		0.214	58963	293291				
	2	0.225	63322	299575	6.408	1889618	419960	1.914
		0.205	57055	296260				
		0.246	66746	288818				
10.0	1	0.253	149081	627242	6.426	4043758	932358	4.131
		0.228	134104	626095				
		0.250	149019	634505				
	2	0.226	133674	629610	6.278	4028410	898500	4.232
		0.252	150846	637187				
		0.228	140984	658216				

* DPM/g = LSC DPM/BMO ALIQ./SOIL EFF

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                   AGRISEARCH INCORPORATED          12220
                              WA LOAM
      S.A: DPM/µg  180042         SAMPLE VOL:   mL    20
-------------------------------------------------------------------
SAMPLE             ====SOIL=====          =====WATER=====
PPM    REP           ppm   LN ppm           ppm   LN ppm
-------------------------------------------------------------------

DEFINITIVE ADSORPTION

  0.2  1            0.202 -1.601          0.165 -1.803
       2            0.211 -1.555          0.161 -1.826
  0.5  1            0.501 -0.690          0.410 -0.892
       2            0.502 -0.689          0.410 -0.890
  1.0  1            0.986 -0.014          0.820 -0.198
       2            1.021  0.020          0.820 -0.199
  5.0  1            5.677  1.736          3.989  1.383
       2            5.291  1.666          4.017  1.391
 10.0  1           11.599  2.451          7.792  2.053
       2           11.671  2.457          7.820  2.057
LINEAR REGRESSION ANALYSIS
     Regression Output:
Constant                0.26574
Std Err of Y Est        0.05316        CORRELATION       0.9995
R Squared               0.99899        SLOPE (1/n)        1.045
No. of Observation           10        INTERCEPT          0.266
Degrees of Freedom            8               n           0.957
                                              Kd            1.3
X Coefficient(s)        1.04512               Koc           ·86
Std Err of Coef.        0.01176               % O.C.      1.508
-------------------------------------------------------------------
SAMPLE        ====SOIL=====  =====WATER====   ====PERCENT OF DOSE=====
PPM     REP   ppm    LN ppm   ppm   LN ppm     SOIL  ---WATER---  TOTAL
                                                     ADS    DES
-------------------------------------------------------------------
DEFINITIVE DESORPTION
 0.2   1     0.089  -2.419  0.023 -3.752      8.8   80.2  11.4  100.3
       2     0.084  -2.479  0.025 -3.683      8.6   78.4  12.2   99.2
 0.5   1     0.203  -1.594  0.061 -2.790      8.2   79.9  12.0  100.0
       2     0.200  -1.608  0.062 -2.774      8.0   80.0  12.2  100.2
 1.0   1     0.381  -0.966  0.116 -2.154      7.8   79.8  11.3   98.9
       2     0.383  -0.959  0.118 -2.138      7.6   79.7  11.5   98.8
 5.0   1     1.904   0.644  0.641 -0.445      7.5   77.5  12.5   97.5
       2     1.914   0.649  0.635 -0.455      7.9   78.1  12.3   98.3
10.0   1     4.131   1.419  1.424  0.354      8.5   76.3  13.9   98.7
       2     4.232   1.443  1.436  0.362      8.5   76.5  14.1   99.1
LINEAR REGRESSION ANALYSIS                                      ======
     Regression Output:                          AVG % DOSE     99.1
Constant                1.08054                                 ======
Std Err of Y Est        0.03726        CORRELATION       0.9997
R Squared               0.99946        SLOPE (1/n)        0.954
No. of Observation           10        INTERCEPT          1.081
Degrees of Freedom            8               n           1.048
                                              Kd            2.9
X Coefficient(s)        0.95419               Koc           195
Std Err of Coef.        0.00786               % O.C.      1.508
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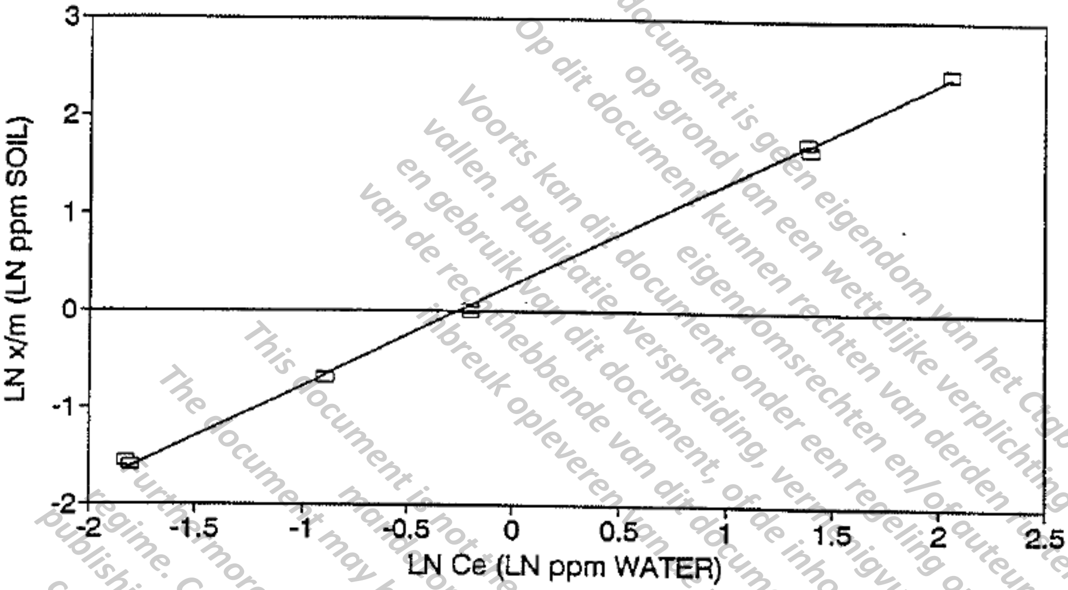
ADSORPTION ISOTHERM
WA LOAM



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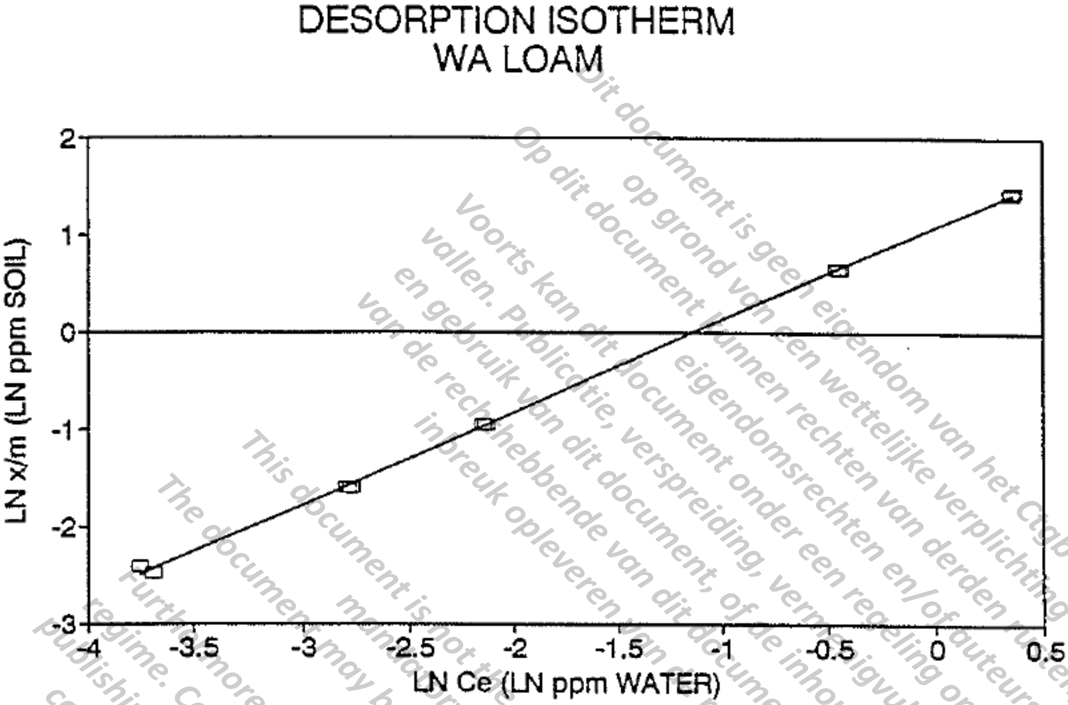

DESORPTION ISOTHERM
WA LOAM

8.31.95

DEFINITIVE
S.A 180042 DPM/μg

AGRISEARCH INCORPORATED
AZ SILTY CLAY LOAM

		ADSORPTION						DESORPTION		
SAMPLE PPM	REP	TUBE + DRY SOIL g	TUBE TARE g	TUBE + WET SOIL g	DRY SOIL g	WATER (g)	% MOIST.	TUBE + WET SOIL g	WATER (g)	DRY SOIL g
ACTUAL TEST SYSTEM WEIGHTS PRE/POST EQUILIBRIUM, CENTRIFUGATION, DECANTING										
0.0	1	39.683	35.456	41.882	4.227	2.199	34.22	42.096	2.413	4.227
	2	39.843	35.720	42.045	4.123	2.202	34.81	42.194	2.351	4.123
0.2	1	39.745	35.551	41.857	4.194	2.112	33.49	42.055	2.310	4.194
	2	38.785	34.691	40.952	4.094	2.167	34.61	41.085	2.300	4.094
0.5	1	39.008	34.974	41.114	4.034	2.106	34.30	41.100	2.092	4.034
	2	36.753	32.655	38.841	4.098	2.088	33.75	39.071	2.318	4.098
1.0	1	38.769	34.460	40.976	4.309	2.207	33.87	41.145	2.376	4.309
	2	39.859	35.542	42.071	4.317	2.212	33.88	42.212	2.353	4.317
5.0	1	39.954	35.831	42.056	4.123	2.102	33.77	42.239	2.285	4.123
	2	39.606	35.376	41.803	4.230	2.197	34.18	42.002	2.396	4.230
10.0	1	39.474	35.295	41.560	4.179	2.086	33.30	41.901	2.427	4.179
	2	39.213	35.030	41.426	4.183	2.213	34.60	41.576	2.363	4.183

8.28.95 [redacted] 5.1.2.e Woo

AGRISEARCH INCORPORATED 12220

DEFINITIVE ADSORPTION AZ SILTY CLAY LOAM

S.A. 180042 DPM/µg SAMPLE VOL: mL 20

SAMPLE PPM	REP	DPM DOSED	ALIQ mL	LS3801 DPM	DPM/mL	AVG DPM/mL	TOTAL DPM	PPM WATER
0.0	1	0	0.5	0	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
	2	0	0.5	0	0	0	0	0.000
			0.5	0	0			
			0.5	0	0			
0.2	1	740253	0.5	13263	26526	26536	530720	0.147
			0.5	13283	26566			
			0.5	13258	26516			
	2	740253	0.5	13370	26740	26692	533840	0.148
			0.5	13300	26600			
			0.5	13368	26736			
0.5	1	1846973	0.5	33807	67614	67683	1353653	0.376
			0.5	33849	67698			
			0.5	33868	67736			
	2	1846973	0.5	33891	67782	67652	1353040	0.376
			0.5	33814	67628			
			0.5	33773	67546			
1.0	1	3701413	0.5	65300	130600	130971	2619413	0.727
			0.5	65492	130984			
			0.5	65664	131328			
	2	3701413	0.5	65986	131972	131580	2631600	0.731
			0.5	65682	131364			
			0.5	65702	131404			
5.0	1	18526480	0.5	297479	594958	589229	11784587	3.273
			0.5	294402	588804			
			0.5	291963	583926			
	2	18526480	0.5	291192	582384	584207	11684147	3.245
			0.5	290947	581894			
			0.5	294172	588344			
10.0	1	36791173	0.5	544163	1088326	1092907	21858133	6.070
			0.5	548645	1097290			
			0.5	546552	1093104			
	2	36791173	0.5	554114	1108228	1111660	22233200	6.174
			0.5	562765	1125530			
			0.5	550611	1101222			

SAMPLE PPM	REP	DPM NOT ADSORBED	CARRY OVER -DPM-		DPM FOR DESORPT	PPM SOIL
0.0	1	0	0	CALCULATE BY DIFFERENCE	0	0.000
	2	0	0		0	0.000
0.2	1	530720	56044		209533	0.277
	2	533840	57842		206413	0.280
0.5	1	1353653	142540		493320	0.679
	2	1353040	141257		493933	0.669
1.0	1	2619413	289052		1082000	1.395
	2	2631600	291055		1069813	1.376
5.0	1	11784587	1238560		6741893	9.082
	2	11684147	1283504		6842333	8.984
10.0	1	21858133	2279803		14933040	19.847
	2	22233200	2460104		14557973	19.330

8.25.95 5.1.2.e Woo

DEFINITIVE DESORPTION AGRISEARCH INCORPORATED 12220
AZ SILTY CLAY LOAM
S.A: 180042 DPM/µg SAMPLE VOL: 20mL 1 mL ALIQ

SAMPLE PPM	REP	DPM FOR DESORB	LS3801 DPM	DPM/mL	AVG DPM/mL	CARRY OVER	DPM/mL-CARRYOVR	PPM WATER
						/mL		
0.0	1	0	0	0	0	0	0	0.000
			0	0				
			0	0				
	2	0	0	0	0	0	0	0.000
			0	0				
			5	0				
0.2	1	209533	7673	7673	7506	2802	4704	0.026
			7451	7451				
			7394	7394				
	2	206413	7329	7329	7367	2892	4475	0.025
			7410	7410				
			7363	7363				
0.5	1	493320	18602	18602	18795	7127	11668	0.065
			19066	19066				
			18716	18716				
	2	493933	18596	18596	18510	7063	11447	0.064
			18828	18828				
			18105	18105				
1.0	1	1082000	39300	39300	39052	14453	24600	0.137
			38465	38465				
			39392	39392				
	2	1069813	38670	38670	38916	14553	24364	0.135
			39521	39521				
			38558	38558				
5.0	1	6741893	208703	208703	207974	61928	146046	0.811
			204185	204185				
			211033	211033				
	2	6842333	216182	216182	211164	64175	146989	0.816
			209899	209899				
			207412	207412				
10.0	1	14933040	399528	399528	402840	113990	288850	1.604
			398294	398294				
			410699	410699				
	2	14557973	409615	409615	408389	123005	285383	1.585
			397837	397837				
			417714	417714				

[redacted] 5.1.2 e Woo

1/14/95

AGRISEARCH INCORPORATED 12220

DEFINITIVE DESORPTION
COMBUSTIONS

AZ SILTY CLAY LOAM

S.A: 180042 DPM/µg

SOIL EFF 0.9486 * MACHINE EFF 0.9686

SAMPLE PPM	REP	BMO ALIQ. g	LS3801 DPM	DPM/g	WET SOIL g	AVG TOT DPM	CARRY OVER DPM	PPM SOIL
0.0	1	0.207	0	0	6.64	0	0	0.000
		0.214	0	0				
		0.218	0	0				
	2	0.225	0	0	6.474	0	0	0.000
		0.253	0	0				
		0.208	0	0				
0.2	1	0.258	4814	19670	6.504	128565	17339	0.147
		0.215	3926	19250				
		0.237	4582	20381				
	2	0.231	4398	20071	6.394	130907	16945	0.155
		0.244	4573	19757				
		0.255	5223	21592				
0.5	1	0.257	11138	45687	6.126	288421	39318	0.343
		0.212	9503	47255				
		0.222	10172	48303				
	2	0.257	11531	47299	6.416	303413	42905	0.353
		0.215	9668	47404				
		0.229	10246	47167				
1.0	1	0.217	19231	93424	6.685	631896	92788	0.695
		0.232	20729	94191				
		0.234	21300	95958				
	2	0.223	19509	92225	6.670	625984	91570	0.688
		0.209	18715	94398				
		0.202	18190	94929				
5.0	1	0.253	143523	598025	6.408	3819791	475220	4.506
		0.217	122658	595874				
		0.211	118970	594393				
	2	0.214	118824	585341	6.626	4004773	505950	4.594
		0.251	143108	601047				
		0.220	130812	626821				
10.0	1	0.258	359287	1468048	6.606	9461785	977693	11.276
		0.224	304654	1433762				
		0.243	321582	1395095				
	2	0.233	320343	1449365	6.546	9568898	965022	11.424
		0.228	325232	1503754				
		0.221	300259	1432261				

* DPM/g = LS3801 DPM/BMO ALIQ./SOIL EFF

9.7.95 [redacted]

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                    AGRISEARCH INCORPORATED            12220
                    AZ SILTY CLAY LOAM
          S.A: DPM/µg  180042           SAMPLE VOL:   mL    20
-------------------------------------------------------------------
SAMPLE                ====SOIL=====          =====WATER=====
PPM     REP            ppm    LN ppm          ppm    LN ppm
-------------------------------------------------------------------

DEFINITIVE ADSORPTION

  0.2   1              0.277 -1.282          0.147 -1.915
        2              0.280 -1.273          0.148 -1.909
  0.5   1              0.679 -0.387          0.376 -0.978
        2              0.669 -0.401          0.376 -0.979
  1.0   1              1.395  0.333          0.727 -0.318
        2              1.376  0.319          0.731 -0.314
  5.0   1              9.082  2.206          3.273  1.186
        2              8.984  2.195          3.245  1.177
 10.0   1             19.847  2.988          6.070  1.803
        2             19.330  2.962          6.174  1.820
LINEAR REGRESSION ANALYSIS
      Regression Output:
Constant                0.81547
Std Err of Y Est        0.10053       CORRELATION          0.9984
R Squared               0.99681       SLOPE (1/n)           1.159
No. of Observation         10         INTERCEPT             0.815
Degrees of Freedom          8                n              0.862
                                             Kd               2.3
X Coefficient(s)        1.15947              Koc             1299
Std Err of Coef.        0.02321              % O.C.         0.174
-------------------------------------------------------------------
SAMPLE     ====SOIL=====   =====WATER====    ====PERCENT OF DOSE=====
PPM    REP   ppm   LN ppm   ppm   LN ppm      SOIL   ---WATER---  TOTAL
                                                      ADS    DES
-------------------------------------------------------------------
DEFINITIVE DESORPTION
  0.2  1    0.147  -1.915  0.026 -3.645       15.0   71.7  12.7   99.4
       2    0.155  -1.867  0.025 -3.695       15.4   72.1  12.1   99.6
  0.5  1    0.343  -1.070  0.065 -2.736       13.5   73.3  12.6   99.4
       2    0.353  -1.041  0.064 -2.755       14.1   73.3  12.4   99.8
  1.0  1    0.695  -0.364  0.137 -1.990       14.6   70.8  13.3   98.6
       2    0.688  -0.375  0.135 -2.000       14.4   71.1  13.2   98.7
  5.0  1    4.506   1.505  0.811 -0.209       18.1   63.6  15.8   97.4
       2    4.594   1.525  0.816 -0.203       18.9   63.1  15.9   97.8
 10.0  1   11.276   2.423  1.604  0.473       23.1   59.4  15.7   98.2
       2   11.424   2.436  1.585  0.461       23.4   60.4  15.5   99.3
LINEAR REGRESSION ANALYSIS                                       ======
      Regression Output:                           AVG % DOSE    98.8
Constant                1.81791                                  ======
Std Err of Y Est        0.11334       CORRELATION          0.9980
R Squared               0.99603       SLOPE (1/n)           1.038
No. of Observation         10         INTERCEPT             1.818
Degrees of Freedom          8                n              0.963
                                             Kd               6.2
X Coefficient(s)        1.03815              Koc             3539
Std Err of Coef.        0.02317              % O.C.         0.174
-------------------------------------------------------------------
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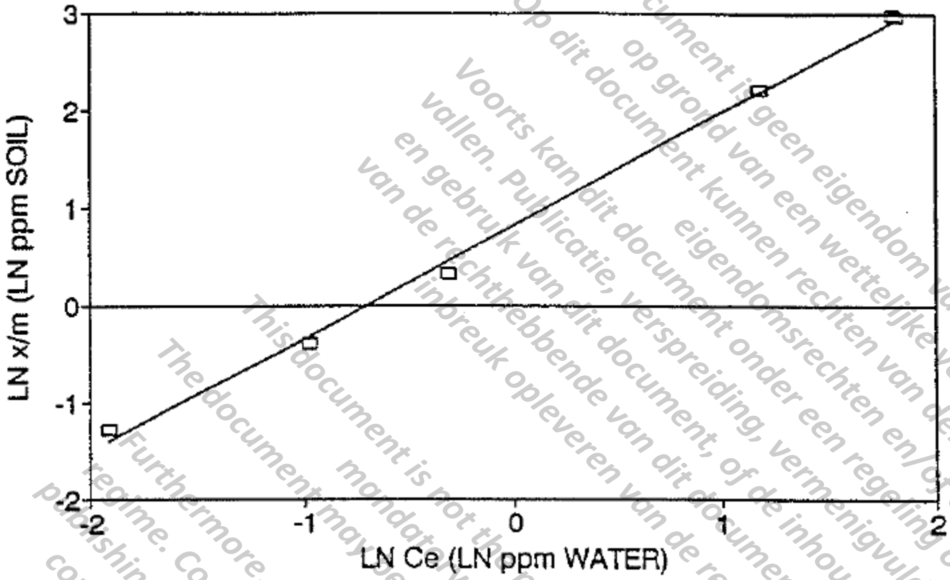
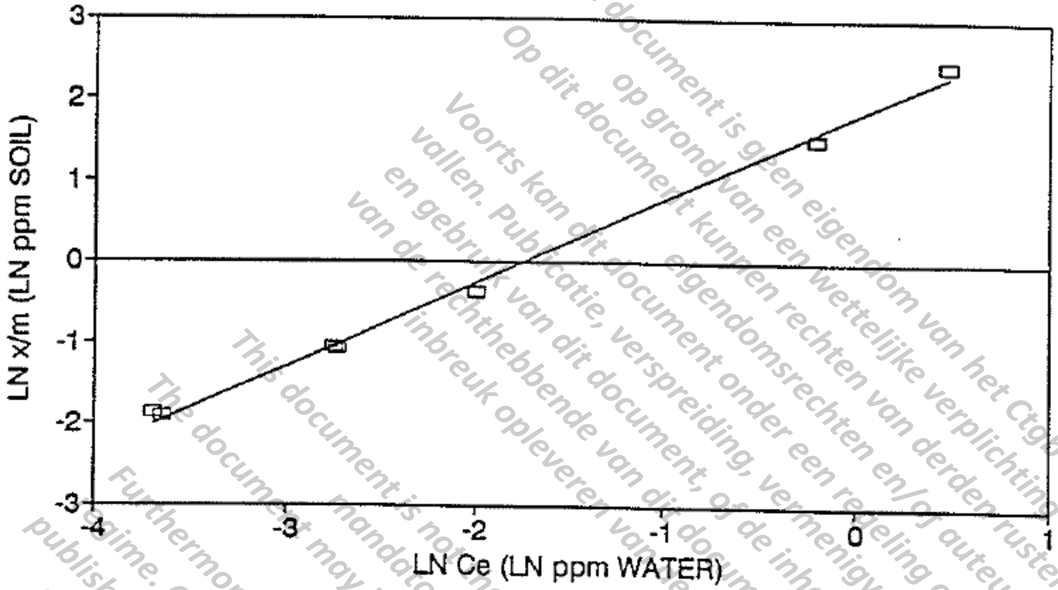
9.7.95

ADSORPTION ISOTHERM
AZ SILTY CLAY LOAM



8.25.95 [redacted]

DESORPTION ISOTHERM
AZ SILTY CLAY LOAM



9.7.95

AGRISEARCH INCORPORATED

POST DESORPTION 10.0 ppm SOIL EXTRACTIONS

STUDY NO: 12220

S.A: 180042 DPM/µg

TOTAL DOSE DPM: 36791173

SAMPLE	REP	TARE (g)	---POST DESORPION(1)---- GROSS WT --(g)--	NET WT (g)	TOTAL DPM FOR EXT.*	-----POST EXTRACTION------- WET SOIL GROSS WT (g)	DRY SOIL GROSS WT (g)	DRY SOIL NET WT (g)
MS Clay	1	34.478	36.560	2.082	11518350	36.440	35.527	1.049
	2	34.908	36.820	1.912	11308620	36.713	35.857	0.949
MD Sand	1	33.940	46.248	12.308	1477092	45.527	43.511	9.571
	2	36.079	48.284	12.205	1368509	47.550	45.567	9.488
CA Sandy Loam	1	33.505	48.065	14.560	2140773	47.271	45.028	11.523
	2	33.025	45.444	12.419	1840349	44.625	42.427	9.402
WA Loam	1	36.548	42.148	5.600	3523972	41.907	40.174	3.626
	2	32.245	37.791	5.546	3558707	37.621	35.845	3.600
AZ Silty Clay Loam	1	35.295	41.153	5.858	8390423	40.812	38.987	3.692
	2	35.030	40.845	5.815	8500327	40.635	38.724	3.694

(1) WEIGHT FOLLOWING POST DESORPTION COMBUSTIONS PRIOR TO EXTRACTION

* TOTAL DPM AVAILABLE FOR EXTRACTION AS DETERMINED BY POST DESORPTION COMBUSTIONS

9.18.95 [redacted]

AGRISEARCH INCORPORATED

POST DESORPTION 10.0 ppm SOIL EXTRACTIONS

STUDY NO: 12220
S.A: 180042 DPM/µg

ALIQUOT: 100µL
TOTAL DOSE DPM: 36791173

SAMPLE	REP	LSC DPM	AVERAGE DPM/mL	TOTAL VOL. (mL)	TOTAL DPM EXT'D	EXTRACTION EFF.(%)*	% DOSE DPM EXT'D
MS Clay	1	266539 259654 256540	2609110	4	10436440	90.6%	28.4%
	2	256629 254928 251997	2545180	4	10180720	90.0%	27.7%
MD Sand	1	5290 5278 5256	52747	24	1265920	85.7%	3.4%
	2	5049 5070 5108	50757	24	1218160	89.0%	3.3%
CA Sandy Loam	1	6682 6563 6646	66303	28	1856493	86.7%	5.0%
	2	6356 6300 6171	62757	28	1757187	95.5%	4.8%
WA Loam	1	29829 28859 29292	293267	12	3519200	99.9%	9.6%
	2	29941 30068 30281	300967	12	3611600	101.5%	9.8%
AZ Silty Clay Loam	1	67430 67028 67272	672433	12	8069200	96.2%	21.9%
	2	68188 68060 69607	686183	12	8234200	96.9%	22.4%

*TOTAL DPM EXTRACTED / TOTAL DPM AVAILABLE FOR EXTRACTION

9.18.95

AGRISEARCH INCORPORATED

POST DESORPTION 10.0 ppm SOIL EXTRACTIONS

STUDY NO: 12220
S.A: 180042 DPM/μg
TOTAL DOSE DPM: 36791173

POST EXTRACTION, AIR DRIED SOIL COMBUSTIONS

SAMPLE	REP	BMO ALIQUOT (g)	LSC DPM	DPM/g	MEAN DPM/g	TOTAL DPM	% POST DESORP. DPM	% DOSE DPM
MS Clay		Soil Eff =		0.9506				
	1	0.084	94079	1178212	1186550	1244691	10.8%	3.4%
		0.085	99163	1227272				
		0.077	84479	1154166				
	2	0.070	61919	930542	1006481	955151	8.4%	2.6%
		0.085	87459	1082420				
		0.077	57467 *	785124				
MD Sand		Soil Eff =		0.9737				
	1	0.257	2129	8508	7890	75516	5.1%	0.2%
		0.222	1663	7693				
		0.242	1760	7469				
	2	0.203	1537	7776	7531	71455	5.2%	0.2%
		0.239	1747	7507				
		0.212	1509	7310				
CA Sandy Loam		Soil Eff =		0.9671				
	1	0.209	2334	11548	11678	134564	6.3%	0.4%
		0.203	2269	11558				
		0.217	2503	11928				
	2	0.225	2567	11798	11411	107285	5.8%	0.3%
		0.206	2238	11234				
		0.226	2448	11201				
WA Loam		Soil Eff =		0.9766				
	1	0.210	15704	76572	78384	284221	8.1%	0.8%
		0.207	16711	82663				
		0.218	16163	75918				
	2	0.205	16304	81436	80820	290951	8.2%	0.8%
		0.226	17465	79130				
		0.223	17835	81893				
AZ Silty Clay Loam		Soil Eff =		0.9799				
	1	0.212	32541	156646	156298	577052	6.9%	1.6%
		0.214	31710	151219				
		0.224	35345	161029				
	2	0.228	35057	156915	155162	573167	6.7%	1.6%
		0.222	32330	148620				
		0.221	34638	159950				

[redacted] 11/15/95

AGRISEARCH INCORPORATED

PROJECT: 12220
FRACTION COLLECTION
MATRIX: DOSE SOLUTION 1.0 ppm

DATE/INIT: [redacted] 8/21/95
INJECTION VOL: 250uL

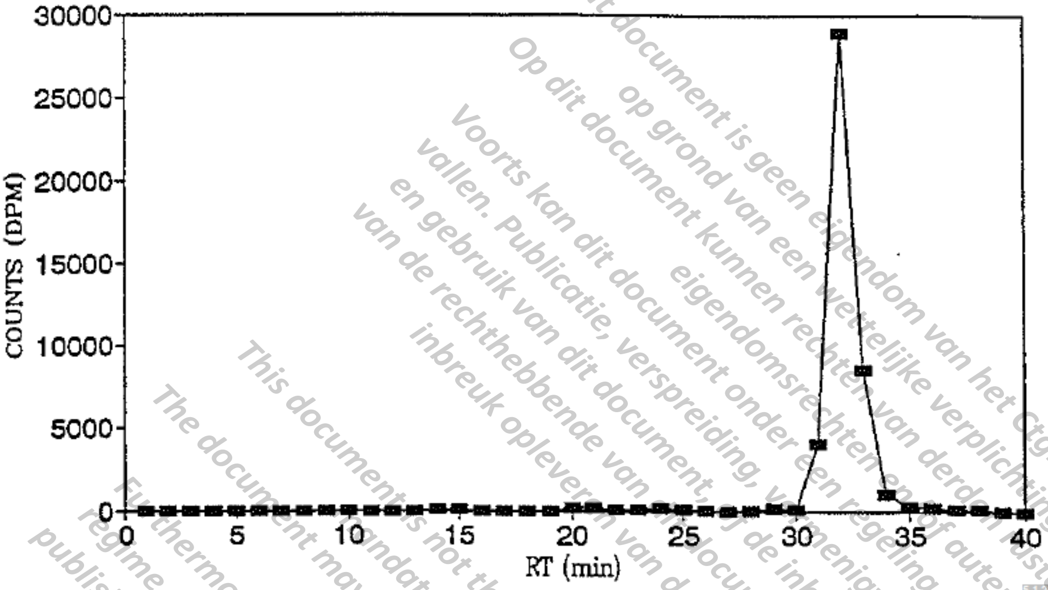
SOLVENT A:0.0125%TFA/ACN-UV 90/10 v/v
B: ACN

TIME MIN	COUNTS DPM	COUNTS BACKGROUND CORRECTED		
1	0	0		
2	0	0		
3	0	0		
4	0	0		
5	3	0		
6	10	0		
7	15	0		
8	26	0		
9	36	36	?	
10	34	34		0.2%
11	29	0		
12	31	0		
13	43	43		
14	72	72	95-034	
15	88	88		0.6%
16	45	45		
17	29	0		
18	28	0		
19	25	0		
20	203	203		
21	240	240	UNKNOWN 1	
22	81	81		1.2%
23	80	80		
24	127	127		
25	79	79	95-036	
26	42	42		0.7%
27	28	0		
28	60	60	UNKNOWN 2	
29	136	136		0.4%
30	111	111		
31	4099	4099		
32	28971	28971	CGA-329351	
33	8530	8530		96.9%
34	1019	1019		
35	261	261		
36	195	195		
37	88	88		
38	77	77		
39	37	37		
40	18	0		
41	16	0		
42	19	0		
43	10	0		
44	8	0		
TOTAL:	45049	44754		

ALQ.100 uL :	18645	R1
	19012	R2
	18895	R3
AVG:	18851	
DPM PER 250ul*	46278	
*CORR for pipettor calib of 101.8%		
RECOVERY:	97.3%	

12220 HPLC FRACTION COLLECTION

DOSE SOLUTION 1.0ppm



[redacted] 1/21/95

AGRISEARCH INCORPORATED

PROJECT: 12220 DEFINITIVE
FRACTION COLLECTION
MATRIX: CA SANDY LOAM 10ppm REP 1
SOIL EXTRACTION

DATE/INIT: [redacted] 11/15/95
INJECTION VOL: 100uL

SOLVEN A: 0.0125% TFA/ACN-UV 90/10 v/v
B: ACN

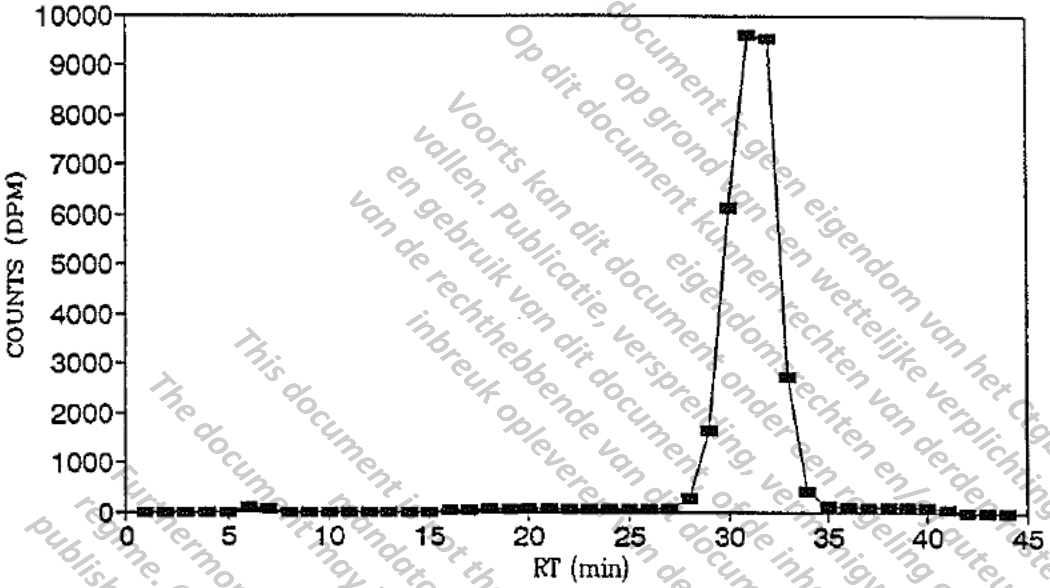
TIME MIN	COUNTS DPM	COUNTS BACKGROUND CORRECTED	
1	11	0	
2	3	0	
3	4	0	
4	3	0	
5	7	0	
6	85	85	UNKNOWN 1
7	84	84	0.5%
8	31	0	
9	11	0	
10	12	0	
11	8	0	
12	12	0	
13	12	0	
14	24	0	
15	24	0	
16	34	34	
17	40	40	95-035
18	70	70	0.6%
19	62	62	
20	74	74	
21	78	78	UNKNOWN 2
22	50	50	0.6%
23	46	46	
24	47	47	
25	51	51	95-036
26	50	50	0.8%
27	66	66	
28	259	259	
29	1639	1639	
30	6143	6143	
31	9616	9616	
32	9521	9521	CGA-329351
33	2726	2726	96.2%
34	411	411	
35	137	137	
36	107	107	
37	95	95	
38	117	117	
39	120	120	UNKNOWN 4
40	91	91	1.2%
41	43	43	
42	26	0	
43	22	0	
44	26	0	
TOTAL:	32098	31862	

ALQ.100 uL :	6877	R1
	6828	R2
	6673	R3
AVG:	6793	
DPM PER 500ul*	34269	
RECOVERY:	93.7%	

*CORR for pipettor calib of 99.1%

12220 HPLC FRACTION COLLECTION

CA SANDY LOAM 10ppm REP 1 SOIL EXTRACT



8.28.95- [redacted] 5.1.2.e Woo

AGRISEARCH INCORPORATED

PROJECT: 12220
FRACTION COLLECTION
MATRIX: DEF DESORPTION SOL'N
MS CLAY 10ppm REP 2

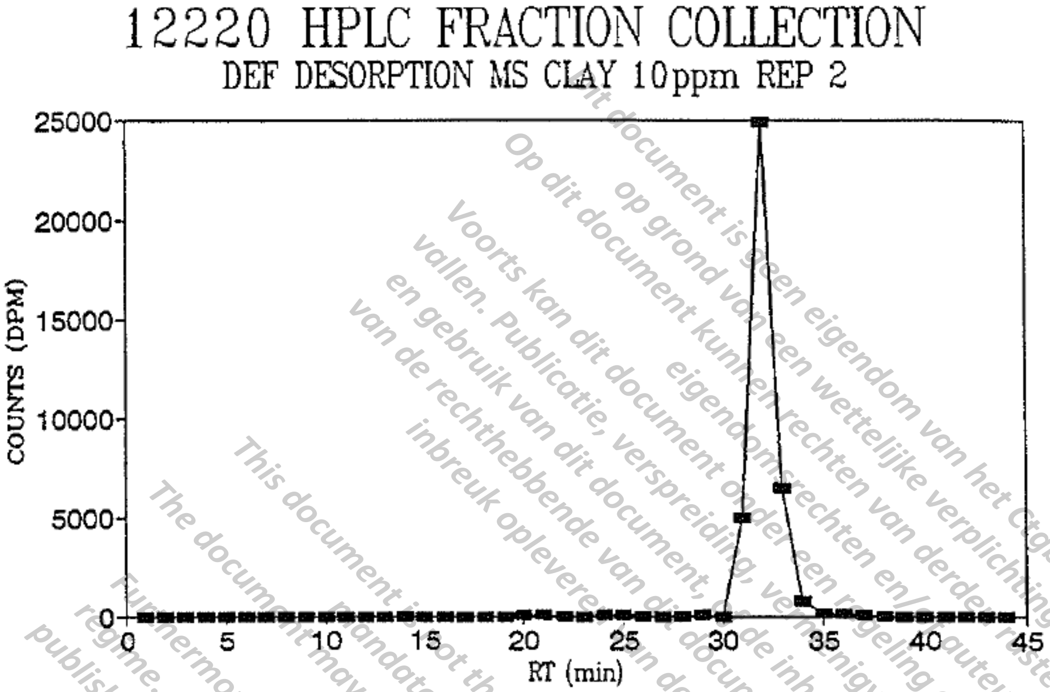
DATE/INIT: [redacted] 11/15/95
INJECTION VOL: 100uL

SOLVEN A:0.0125% TFA/ACN-UV 90/10 v/v
B: ACN

TIME MIN	COUNTS DPM	COUNTS BACKGROUND CORRECTED		
1	0	0		
2	0	0		
3	0	0		
4	0	0		
5	0	0		
6	2	0		
7	1	0		
8	1	0		
9	7	0		
10	10	0		
11	5	0		
12	9	0		
13	22	0	95-034	0.1%
14	35	35		
15	26	0		
16	3	0		
17	0	0		
18	17	0		
19	10	0		
20	79	79	UNKNOWN 2	0.5%
21	80	80		
22	35	35		
23	28	0	95-036	0.4%
24	56	56		
25	59	59		
26	36	36		
27	15	0		
28	36	36	UNKNOWN 3	0.3%
29	73	73		
30	22	0		
31	4976	4976	CGA-329351	98.7%
32	24938	24938		
33	6426	6426		
34	749	749		
35	202	202		
36	121	121		
37	55	55		
38	34	34		
39	13	0		
40	6	0		
41	0	0		
42	1	0		
43	0	0		
44	0	0		
TOTAL:	38188	37990		

ALQ.100 uL:	41586	R1
	43344	R2
	42656	R3
AVG:	42529	
DPM PER 100ul*	42401	
RECOVERY:	90.1%	

*CORR for pipettor calib of 100.3%



[redacted] 5.1.2 e Wob /21/95

AGRISEARCH INCORPORATED

PROJECT: 12220 TLC PLATE SCRAPES MS CLAY 10.0 ppm REP.1
MATRIX: POST DESORPTION EXTRACT SPOTTING VOL: 10uL

AREA	LSC DPM	DPM MINUS BKG
1	28443	28247
2	119	0
3	85	0
4	109	0
5	61	0
6	50	0
I	1311	1115
II	2926	2730
III	591	395
IV	1505	1309
BACKGROUND	196	
BLANK	16	
TOTAL:	35396	33796

MS CLAY 10.0 ppm REP.1
LSC ANALYSIS OF THREE
10 µL ALIQUOTS: 32151
(PIPETTOR 73646) 30031
30606

AVERAGE COUNTS PER 10µL: 30929

% RECOVERY: 109.3%

SS1 = CHCL3/MeOH/NH4OH/WATER 80:30:4:2 v:v:v:v
SS2 = CHCL3/MeOH/FA/WATER 75:20:4:2 v:v:v:v

9.18.95

```
                    AGRISEARCH INCORPORATED

PROJECT:   12220 TLC PLATE SCRAPES   DEFINITIVE DOSE SOLUTION
MATRIX:     5.0 PPM SOLUTION         SPOTTING VOL:5uL
-----------------------------------------------------------------
              COUNTS              COUNTS  DPM MINUS
               CPM2     %EFF      * DPM      BKG
-----------------------------------------------------------------
     1         4141     94.59     4378       4364        95.8 %PARENT
     2           31     94.59       33         19
     3           18     94.65       19          5
     4           25     94.70       26         13
     5           16     94.59       17          3
     6           21     94.70       22          8
     I           57     94.11       61         47   COUNTS PER
    II           40     94.32       42         29          5uL:   4632
   III           46     94.25       49         35
    IV           44     94.37       47         33
BACKGROUND       13     94.58       14             % RECOVERY:   98.4
  BLANK          13     94.73       14

-----------------------------------------------------------------
    TOTAL:     4452               4707       4556

    * = CPM2/%EFF TO CORRECT FOR DPM
    SS1 = CHCL3/MeOH/NH4OH/WATER 80:30:4:2 v:v:v:v
    SS2 = CHCL3/MeOH/FA/WATER 75:20:4:2 v:v:v:v
```

[redacted] 1/15/95

AGRISEARCH INCORPORATED

PROJECT: 12220 TLC PLATE SCRAPES AZ S.C. LOAM ADSORP. SOLUTION
MATRIX: 10 PPM SOLUTION REP 2 SPOTTING VOL:10uL

AREA	COUNTS CPM2	%EFF	COUNTS * DPM	DPM MINUS BKG
1	10398	94.48	11006	10982
2	71	94.63	75	52
3	23	94.64	24	1
4	41	94.61	43	20
5	81	94.67	86	62
6	24	94.64	25	2
I	484	94.12	514	491
II	41	94.27	43	20
III	82	94.17	87	64
IV	100	94.29	106	83
BACKGROUND	22	94.54	23	
BLANK	16	94.70	17	
TOTAL:	11367		12033	11777

93.2 %PARENT

COUNTS PER 10uL: 11117

% RECOVERY: 105.9

* = CPM2/%EFF TO CORRECT FOR DPM
SS1 = CHCL3/MeOH/NH4OH/WATER 80:30:4:2 v:v:v:v
SS2 = CHCL3/MeOH/FA/WATER 75:20:4:2 v:v:v:v

[redacted] 11/13/95

APPENDIX E

References

Test Protocols for Environmental Fate & Movement of Toxicants

Proceedings of a Symposium
Association of Official Analytical Chemists
94th Annual Meeting
October 21, 22, 1980
Washington, DC

MEASUREMENT OF SORPTION COEFFICIENTS OF ORGANIC CHEMICALS AND THEIR USE IN ENVIRONMENTAL FATE ANALYSIS

5.1.2.e Woo, 5.1.2.e Woo, and 5.1.2.e Woo

Residue/Environmental/Metabolism Research
Agricultural Products Department
Dow Chemical U.S.A.
Midland, MI 48640

Sorption of organic chemicals to soil or sediment is a major factor in the processes that control transport of chemicals in the environment, such as volatility, leaching, and runoff. Therefore, sorption measurements must be made in such a way that they can be related to these processes.

In general, for neutral organic compounds sorption can be standardized on the organic carbon content of the soil, which is the single most important soil property affecting sorption. Thus, a single value ($\underline{K}_{oc}$) is obtained which can provide a basis for relating the inherent sorptivity of chemicals and which is more easily used in hazard evaluation schemes. In addition, this value is highly interrelated with other physical measurements, i.e., water solubility, octanol:water ratio, and retention time on HPLC, such that reasonable estimates of K_{oc} values can often be obtained from simple correlations of properties.

Methods of determination of sorption constants, important aspects of experimental design, and application of sorption measurements to hazard evaluation are discussed.

Introduction

Characterization of the sorption properties of organic chemicals to soils and sediments is essential for understanding the migratory tendency of chemicals in the air, water, and soil compartments of the environment. Partitioning of a chemical between sorbed and solution phases will affect various transport processes that act on the chemical in the environment. Volatility from water and soil, leaching through the soil profile, concentrations in water, and runoff from land surfaces into natural waters represent mechanisms where sorption is directly related to the transport process.

In order to describe these processes such that the environmental fate of chemicals can be assessed, a standard method for measurement of sorption must be developed. Such a measurement would presumably provide a value or set of values that could be easily incorporated into appropriate mathematical models for prediction of environmental hazard. At the same time, a uniform data base characterizing sorption properties of organic chemicals would be developed which would be useful for ranking and comparing chemicals.

Principles of sorption of chemicals to soils and sediments have been studied extensively in the past and are discussed in detail by Hamaker (1) and Pionke and Chesters (2). It becomes immediately apparent that one is dealing with complex intermolecular interactions at a variety of surfaces. Hydrophobic bonding, van der Walls - London forces, charge transfer, ion exchange, and chemisorption represent primary interactions associated with sorption. The type of interaction which predominates will depend on the nature of the chemical. It would seem difficult, at best, to develop a standard method of measurement which would describe these relationships. However, if one restricts the approach to a general class which represents those chemicals of primary environmental importance, a generalized characterization of the sorption process can be obtained. Therefore, the principles of sorption measurement discussed in this paper will apply mainly to neutral organic compounds where the primary interaction with the soil or sediment is considered to be hydrophobic in nature.

Sorption Equilibrium Expressions

The partitioning of a chemical between the sorbed and solution phases is typically defined in terms of an equilibrium expression. Many equations have been used to express this relationship; however, historically the Freundlich equation has been used most commonly in dealing with soils. This equation is expressed as

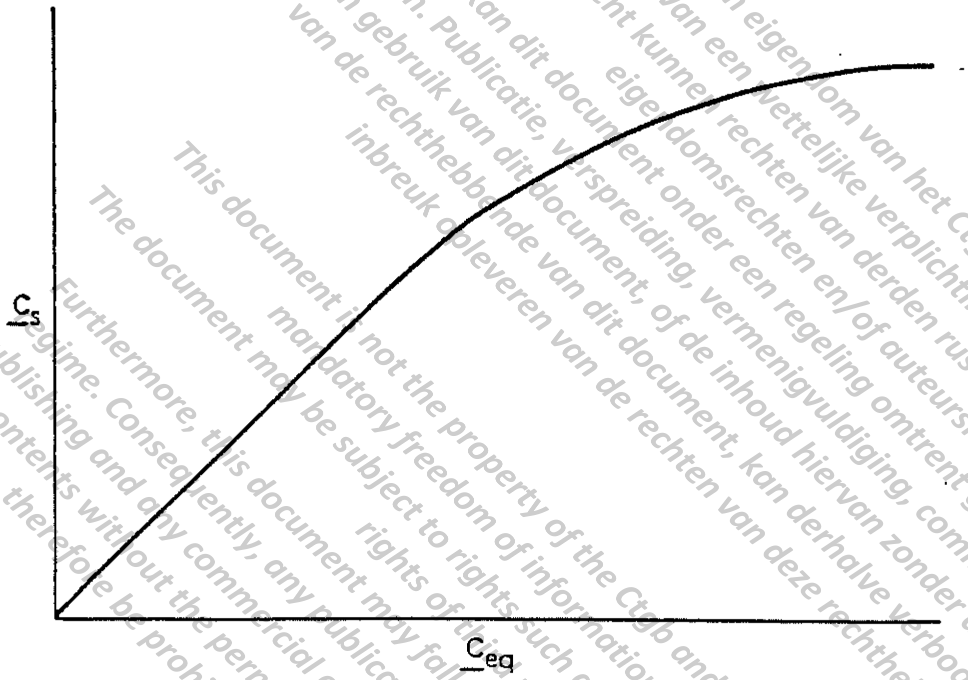
$$\underline{C}_s = \underline{K}_d \underline{C}_{eq}^{1/n} \qquad (\underline{1})$$

where:

$\underline{C}_s$ = sorbed chemical, µg/g sorbate
$\underline{C}_{eq}$ = equilibrium solution concentration, µg/g
$\underline{K}_d$ = sorption coefficient
$1/n$ = empirical exponent.

The Freundlich equation is strictly empirical in nature with an exponential term (1/n) which takes into account non-linearity in the sorption isotherm (Figure 1) which occurs at higher chemical concentrations. As concentration increases, sorption sites begin to become saturated such that increasing the solution concentration does not increase the amount of chemical in the sorbed state proportionally.

FIGURE 1. Typical sorption isotherm for chemical-soil interaction



$$C_s = K_d C_{eq}^{1/n}$$

At lower concentrations the value of 1/n becomes unity and the sorption isotherm becomes linear. This usually is the case at typical chemical loadings encountered in the environment where sorption can be characterized by

$$C_s = K_d C_{eq} \qquad (2)$$

Karickhoff et al. (3) have determined that if the equilibrium solution phase concentration is kept below 10^{-5}M or below one-half of the solute water solubility, whichever is lower, this expression generally holds. In considering sorption at these concentration levels, an equilibrium partition coefficient independent of concentration is obtained. This type of coefficient is mathematically simpler to incorporate into hazard assessment models.

It is true that for certain environmental situations (landfills, spills, etc.) the Freundlich approach may be necessary to characterize sorption. Also, chemicals that interact with soil/sediment surfaces through mechanisms other than hydrophobic interaction may exhibit non-linear sorption isotherms. However, a large number of chemicals can be characterized by the simpler form of the equation which provides a more useful way to describe sorption at typical environmental loadings.

Organic Carbon Contribution to Sorption

Of the variety of soil factors that can influence sorption, organic carbon content has been shown to be the single most important soil property related to the sorption of neutral organic compounds. This effect was first shown by Goring (4,5) in the 1960s and has been developed extensively in the literature since that time (1,6-12). Soils or sediments with higher amounts of organic carbon have proportionately higher capacities for sorption. Sorption can therefore be expressed in terms of organic carbon content where

$$\underline{K}_{oc} = \frac{\mu g\ \text{chemical/g organic carbon}}{\mu g\ \text{chemical/g water}} \qquad (\underline{3})$$

and

$$\underline{K}_{oc} = \frac{\underline{K}_d \times 100}{\%\ \text{organic carbon}} \qquad (\underline{4})$$

A property which is independent of other soil properties and which is unique to the chemical is obtained. An example of this effect is given for nitrapyrin in Table 1. Although some variability exists in the $\underline{K}_{oc}$ value (coefficient of variation = 20%), an average value is obtained which can be used to represent the sorption properties of the chemical. In general, variations of 20 to 50% of the mean K_{oc} values can be anticipated. Initially this may seem like a large variation, but it is actually small with respect to the range over which $\underline{K}_{oc}$ values are observed for different chemicals. This range is from zero to 10^6. As chemicals increase in non-polar character, the hydrophobic interaction with organic carbon increases and $\underline{K}_{oc}$ values increase. Table 2 presents $\underline{K}_{oc}$ values for several compounds.

TABLE 1. Sorption of Nitrapyrin by Several Soils Differing in Content of Organic Carbon (Initial Water Concentration of Nitrapyrin was 1 ppm)

Soil	Organic Carbon, %	K_D	K_{OC}
1	0.081	0.397	491
2	0.62	3.18	514
3	0.86	5.39	627
4	0.97	4.43	457
5	1.45	9.09	627
6	3.80	15.9	417
7	5.76	44.0	764
8	21.7	132.4	611
	Mean	26.9	564
	SD	44.9	114
	CV	167%	20%

TABLE 2. Sorption Constants for Several Chemicals

Chemical	K_{OC}
Dicamba	0
Cis-1,3-dichloropropene	26
Ethylene dibromide	32
Monuron	83
DBCP	130
Atrazine	170
EPTC	280
Nitrapyrin	560
Lindane	1,300
Trifluralin	3,900
Parathion	10,000
Chloropyrifos	14,000
Pyrene	63,000
DDT	240,000
Dibenz[a,h]anthracene	1,670,000

The $\underline{K}_{OC}$ value then represents "a number" which can be used in mathematical models to characterize the innate sorption properties of a chemical. Or, more simply, it may be used for the purpose of ranking and comparing chemicals with respect to this property. Chemicals can be grouped into classes which may be related to some aspect of environmental behavior which is associated with sorption. This will be discussed later.

Sorption Measurement - Experimental Considerations

Establishment of a standard method to measure sorption must center around a general philosophy of what one is attempting to measure, the desired accuracy, and the factors that can critically affect the results of the experiment. The philosophy of the suggested approach has already been addressed. Experimentally, this is translated into characterizing sorption at environmentally expected concentrations in a representative number of soils. Emphasis should be placed on numbers of soils rather than on different concentrations in one soil. It is usually true that errors arising from concentration effects at low concentrations are generally less than the variation existing between different solids in determining $\underline{K}_{OC}$ values. In general, at least 4-7 soils or sediments of varying organic carbon content should be studied in order to obtain the best possible value for $\underline{K}_{OC}$.

Critical factors which may influence the outcome of the experiment are handling of soils/sediments, preparation of samples, water-to-solids ratios, equilibration times, mass balance effects, and centrifugation conditions. These will each be discussed in turn.

Soil/Sediment Handling

In general, it is preferable to study sorption with soils or sediments which are as close to their natural state as possible. Soils are typically collected from the top six inches of the soil profile and screened through a 2 mm sieve before storage at 4°C when not in use. This is typically done at the moisture level at time of collection unless the soil is too wet for sieving and must be partially dried. If such soil preparations are not possible, air-dried soils are acceptable; however, rewetting of the organic layers of the soil can be a slow process and longer experimental equilibration times may be required.

Sediments from aquatic systems should not be air-dried or frozen before use. Samples are collected with a suitable grab or coring device and, if not used immediately, can be stored at 4°C for up to 10 days. Care must be taken when working with anoxic sediments to strictly exclude oxygen during the test.

Soils and sediments should be characterized with respect to organic carbon content, pH, cation and anion exchange capacities, particle size or textural analysis, and moisture content or conversely solids content when sediments are used. Sediments should also be characterized as to their re-dox potential. Since organic carbon is in most cases used to characterize sorption, care should be taken with this measurement to obtain the best value possible. The preferred method is wet combustion of the sample and gravimetric determination of carbon dioxide released.

Sample Preparation

Samples are generally prepared by dissolving the chemical in water which is then added to the solids. Initial concentrations in solution should not exceed 1 ppm or one-half the water solubility. With compounds of very low water solubility, it is sometimes difficult to prepare an aqueous solution; then the chemical may be added in a co-solvent of acetonitrile or other suitable solvent provided the co-solvent is kept below 0.2% by volume.

Containers for the experiment should be selected of material which has minimal surface interaction with the chemical. Glass is usually suitable; however, other materials may suffice. Centrifuge tubes are preferred since the samples must be centrifuged before analysis. It is important that the container be sealed during the experiment to prevent loss of chemicals with high Henry's law constants from solution. This can be a cause of substantial error in the measured $\underline{K}_d$ value.

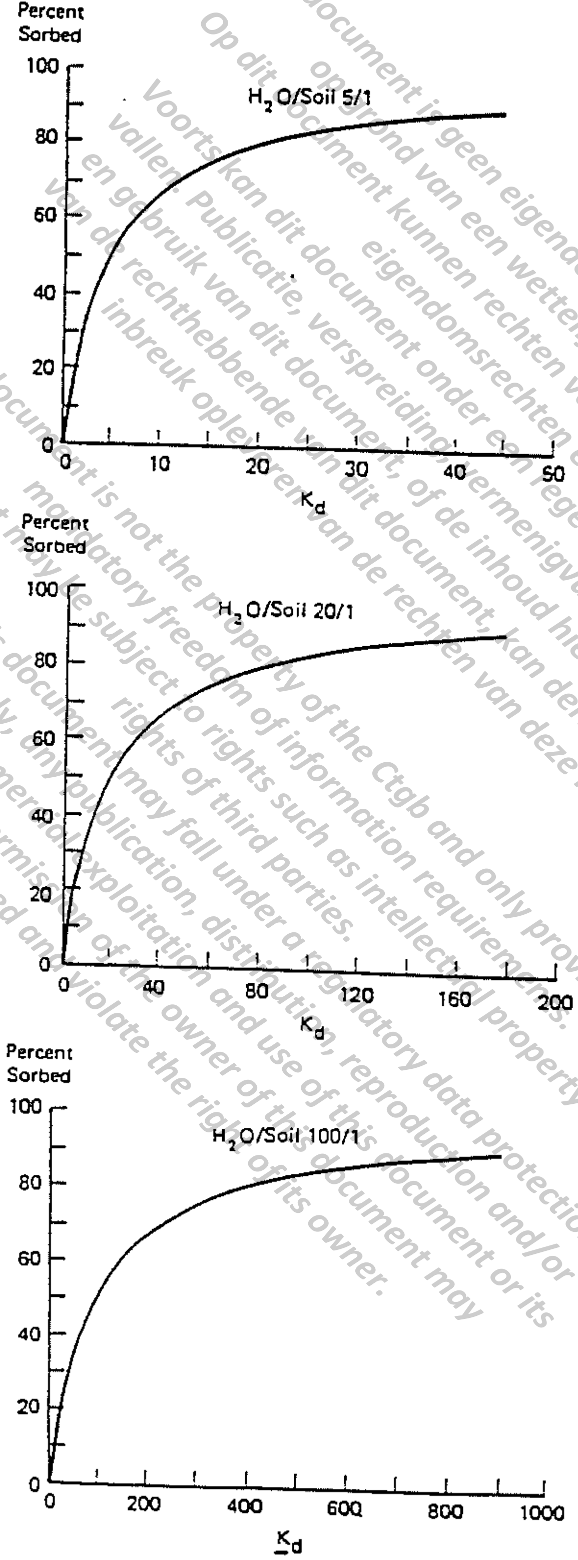
In addition, it is important to prepare appropriate controls for the experiment. Samples containing no soil should be prepared to measure effects of the container or the solution concentration. Likewise, samples containing no chemical should be prepared to detect the possibility of interferring contaminants in the analytical method.

Water-to-Solids Ratio

Selection of appropriate water to soil ratios for sorption studies will depend on the sorption coefficient ($\underline{K}_d$) and the relative degree of sorption desired. The degree of sorption of chemical from solution will determine the statistical accuracy of the measurement based on the form of the sorption equation and the limit of the analytical methodology in detecting the concentration of the chemical in solution.

If $\underline{K}_d$ is plotted vs percent sorbed at fixed water/solids ratios, the range over which reliable measurements are made can be seen. Such plots are shown in Figure 2 for 5/1, 20/1, and 100/1 water/solids ratios. Two aspects of such plots become apparent; First, at a given ratio, as percent sorbed increases, a small change in this value can result in a rather large change in $\underline{K}_d$. This is particularly true above 80% sorbed. Second, as $\underline{K}_d$ becomes very small, percent sorbed decreases rapidly such that measurements in these cases need to be made at as low as possible H_2O/solids ratio.

FIGURE 2. Relationship between percentage sorbed and $\underline{K}_d$



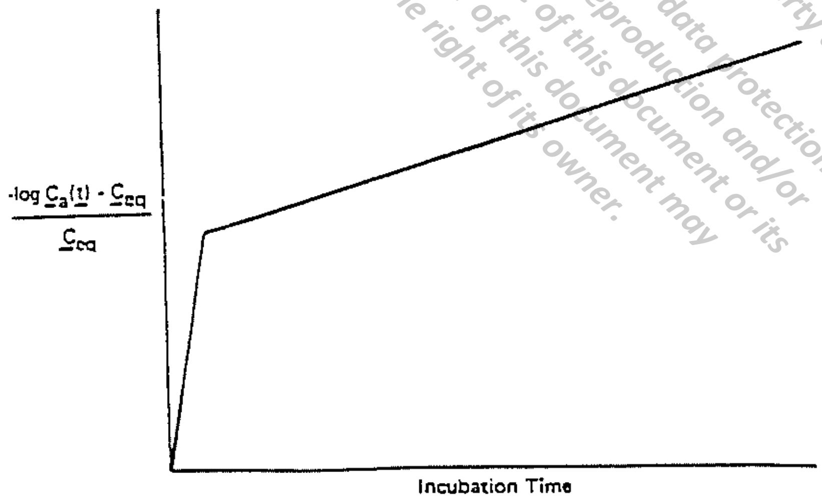
Therefore, in general, it is most desirable to stay below 80% and above 20% sorbed. This is possible by selection of the appropriate water-to-soil ratio based on an estimate of the value of $\underline{K}_d$ either by preliminary studies or by established estimation techniques.

Ideally, sorption studies can be done at any water/solids ratio, but in practice it is perhaps useful to standardize on a few fixed ratios, as long as the percent sorbed falls within 20-80%. It is recommended that 5:1, 20:1, or 100:1 be used. These ratios will cover the 20-80% sorption ranges for $\underline{K}_d$ values of approximately 1-20, 4-80, and 20-400, respectively.

Equilibration Time

The time required to reach equilibrium is highly variable, depending on the chemical and the sorbant as well as the ratio of water to solids. Hance (13) in studying the sorption of four herbicides found equilibrium was attained in 24 hours or less with one exception; LaFleur (14) in studying sorption equilibrium with selected pesticides in model soils found equilibrium to be reached between 10^4 and 10^5 minutes with substrates containing organic matter. Karickhoff (15) and Leistra and Dekkers (16) as well as others have shown that sorption is at least a two-step process with a fast step and a slow step. The slow step was observed to take more than 24 hr to reach equilibrium. If the adsorption kinetics are treated as a simple first order reversible mechanism, a plot of $-\log(\underline{C}_a(\underline{t}) - \underline{C}_{eq})/\underline{C}_{eq}$ vs time should be linear, where $\underline{C}_a(\underline{t})$ equals the solution phase concentration at time, $\underline{t}$, and $\underline{C}_{eq}$ equals the solution phase concentration at equilibrium. The type of plot obtained, however, is generally bi-phasic, as shown in Figure 3, indicating a multiple rate process.

FIGURE 3. Sorption approach to equilibrium assuming simple reversible first order kinetics



It is important then in determining the sorption coefficient, K_d, that enough time be allowed for equilibrium. Experimentally, this can be accomplished by taking samples over time until no change in the solution and solids concentration is detected. In addition, samples should be well mixed or shaken during incubation to facilitate equilibration of the system. If equilibrium is not attained, the value of the sorption coefficient will be underestimated.

Mass Balance Determination

Another cause of error often encountered in sorption experiments is failure to perform a mass balance on the material added to the system. Often, only the solution phase is analyzed and the amount of sorbed chemical is equated to the decreased amount of chemical in the solution phase. Physical loss of the chemical, sorption to the container surfaces, and degradation of the chemical during incubation can all account for decrease in the solution concentration of the chemical in addition to sorption. The sorption coefficient can be substantially overestimated if one of these mechanisms contributes significantly to chemical loss from solution.

The chemical concentration in the solution and on the solids need both be measured by validated analytical methods. Frequently, radioactive labeled chemicals are used which can make quantitation of material simpler. However, analysis for the chemical itself in each phase is necessary rather than just determination of total radioactivity. With highly sorbed compounds, it is possible that a radioactive impurity which is weakly sorbed and initially present in very low amounts can represent a significant amount of the radioactivity remaining in solution. Analytical methods involving unlabeled material must demonstrate reasonable recoveries (generally >90%) and must be sensitive to interferences which affect the determination.

Centrifugation Techniques

Samples must be centrifuged under conditions sufficient to remove most particulate matter from solution. Generally, removal of particles greater than 0.1 micron in radius is adequate. This is particularly important for highly sorbed chemicals since significant error could result if a substantial amount of chemical measured in the solution phase was sorbed to particles not removed by centrifugation.

Centrifugation conditions will vary from one instrument to another but can be calculated from Stoke's law. An equation derived to calculate the time to remove special particles 0.1 micron in radius from solution is given below.

$$\underline{t} = \frac{2.22 \times 10^{10}}{(\mathrm{rpm})^2} \ln \underline{R}_b/\underline{R}_t$$

where:

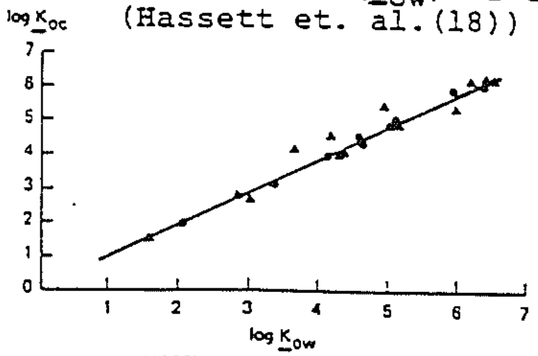
$\underline{t}$ = seconds
rpm = revolutions per minute
$\underline{R}_t$ = distance from center of centrifuge rotor to top of solution in tube
$\underline{R}_b$ = distance from center of centrifuge rotor to bottom of tube

This equation assumes a particle density of 2.65 g/cm^3 and a viscosity for water at 25°C of 8.95 x 10^{-3} g/sec-cm. Since particles are not necessarily spherical, it is generally advisable to double calculated times to assure adequate centrifugation.

Correlation of Sorption with Other Physical Properties

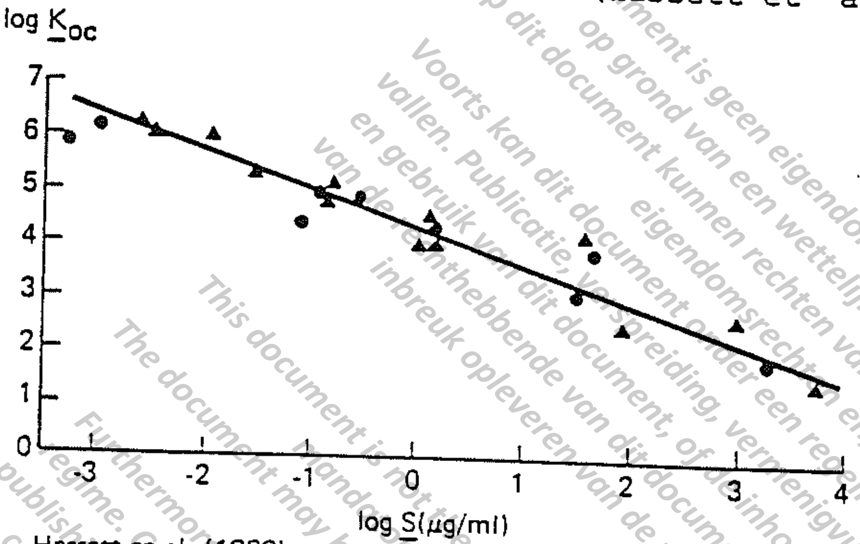
As has been previously discussed, the degree to which a chemical partitions from water to organic carbon in soil or sediment is related to the hydrophobic nature of the molecule. The more hydrophobic a molecule is, the greater tendency it has to partition from aqueous to organic media. The $\underline{K}_{oc}$ value is a measure of this chemical property in soils. In addition, other laboratory measurements are also expressions of this property. Partitioning between water and n-octanol ($\underline{K}_{ow}$), the retention time on a reverse-phase high performance liquid chromatographic column ($\underline{R}_t$), and the water solubility ($\underline{W}_s$) of a chemical are related to the basic hydrophobic nature of the molecule. As such, these coefficients measure the same property except in different media, and are therefore related to one another. Correlations between various combinations of these partition coefficients have appeared in the literature. Briggs (17) initially demonstrated the relationship between $\underline{K}_{oc}$ and $\underline{K}_{ow}$. Since then the correlation between these parameters has been extensively demonstrated (3,18-20). A plot taken from the work of Hassett et al. (18) who measured $\underline{K}_{oc}$ and $\underline{K}_{ow}$ values for 15 compounds is shown in Figure 4. Likewise, water solubility has been correlated with $\underline{K}_{oc}$ (3,18-21) for hydrophobic chemicals. Figure 5 shows

FIGURE 4. Relationship between $\underline{K}_{oc}$ and octanol-water partition coefficient ($\underline{K}_{ow}$) of energy-related organic pollutants (Hassett et. al.(18))



Hassett et al. (1980)

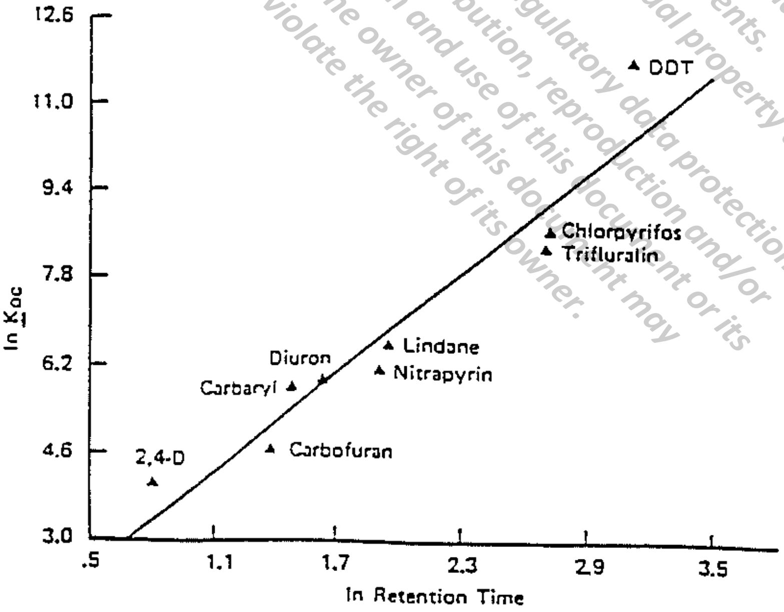
FIGURE 5. Relationship between $\underline{K}_{oc}$ and water solubility($\underline{s}$) of energy-related organic pollutants (Hassett et al.(18))



Hassett et al. (1980)

this correlation, also taken from Hassett et al. More recently, retention time on HPLC has been shown by McCall et al. (22) and Swann et al. (20,23) to be highly correlated with $\underline{K}_{oc}$ values (Figure 6). Again, the HPLC column packing material is a long chain hydrocarbon which presents a hydrophobic regime to chemicals chromatographed.

FIGURE 6. Correlation of soil sorption constant with HPLC retention time for nine organic chemicals



These correlations are useful for several reasons. In conducting a sorption experiment it is helpful to have an approximate value for the $\underline{K}_{oc}$ value before starting the experiment in order to properly design the study. In particular, selection of appropriate water-to-solids ratio is facilitated such that preliminary range finding experiments are not needed. In addition, in some instances an estimated value which is generally accurate within a factor of 2-3 may suffice for the intended interpretation of the influence of sorption in a particular evaluation of the chemical which would preclude conducting the experiment.

Conversely, once a measured value of $\underline{K}_{oc}$ is obtained, the value can be used to estimate the other partition coefficients. Octanol/water ratio has been correlated with bioconcentration factors in fish (19,20-26), therefore by combining the relationships one can relate soil sorption constants to bioconcentration factors. Equations for these relationships with $\underline{K}_{oc}$ are given below.

$$\ln \underline{W}s \text{ (ppm)} = 1.7288 \ln \underline{K}_{oc} - 0.01 \text{ (MP-25)} + 15.1621$$
$$\ln \underline{K}_{oc} = \ln \underline{K}_{ow} - 0.7301$$
$$\ln \underline{K}_{oc} = (\ln \text{BCF})\ 1.935 + 2.951$$

In the first equation the melting point term (MP-25) was a contribution to the understanding of these relationships by Yalkowski et al. (27,28). This addition takes into account the entropy involved in dissolution of solid materials and aids in linearizing the correlation with water solubility.

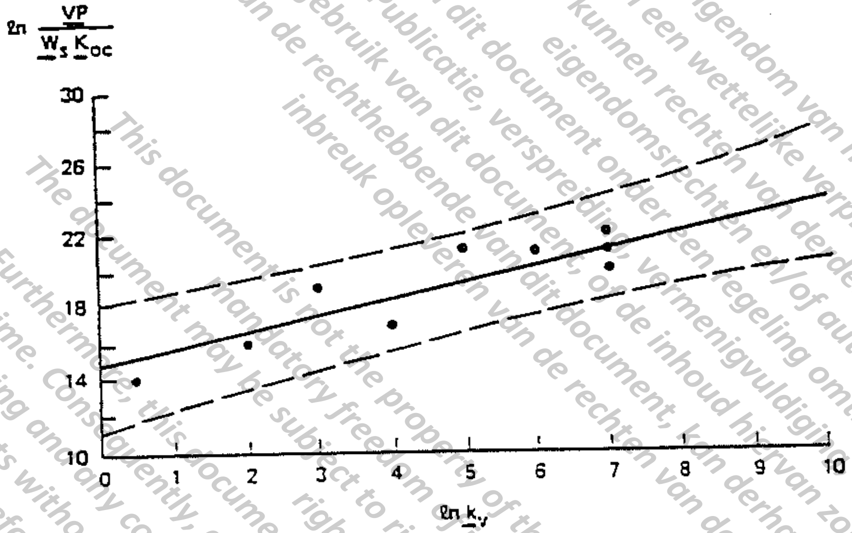
Data Interpretation

Knowledge of the sorption characteristics of chemicals can be useful in understanding the tendency of a chemical to move through the environment. Several examples will be given relating $\underline{K}_{oc}$ to volatility from soil, leaching through soil, and estimation of overall environmental partitioning of chemicals.

The volatility of a chemical from soil is related to its vapor pressure, Henry's law constant, and degree to which it is sorbed to the soil. Two chemicals with the same tendency to volatize from aqueous solution may exhibit different degrees of volatility if solids are added to the system or if the chemical is placed on soil, depending on their relative $\underline{K}_{oc}$ values. The higher a $\underline{K}_{oc}$ value, the stronger the interaction with solids and in general a greater effect on attenuating the volatility potential of a chemical is observed. Although volatility is a complex phenomena controlled by several rate processes, Swann et al. (29) have shown a simple relationship which includes the influences or sorption utilizing the $\underline{K}_{oc}$ value. A plot of initial rate of volatility loss of several pesticides from soil versus a function of the vapor pressure, water solubility, and $\underline{K}_{oc}$ value shown in

Figure 7 demonstrates this relationship. Therefore, from these physical properties an indication of the volatility potential of chemicals from soil can be estimated.

FIGURE 7. Linear regression line for the correlation of physical properties of chemicals to the rate of volatilization



The mobility of a chemical through soil can be directly related to its sorption properties. The relationship that exists between sorption and leaching has been clearly demonstrated in the literature (6-10,16,22,30). When critical soil parameters are known, sorption theory combined with differential equations describing mass flow can predict exact distribution profiles of a chemical in laboratory soil column experiments.

Hamaker (30) has shown that simple chromatographic theory can be used to relate sorption characteristics of chemicals through soil. The following relationship has been derived:

$$\underline{R} = \frac{1}{\theta^{2/3}(1-\underline{K}_{oc}(\%\ O.C./100)\underline{d}_s) + \underline{K}_{oc}(\%\ O.C./100)\underline{d}_s} \qquad (\underline{5})$$

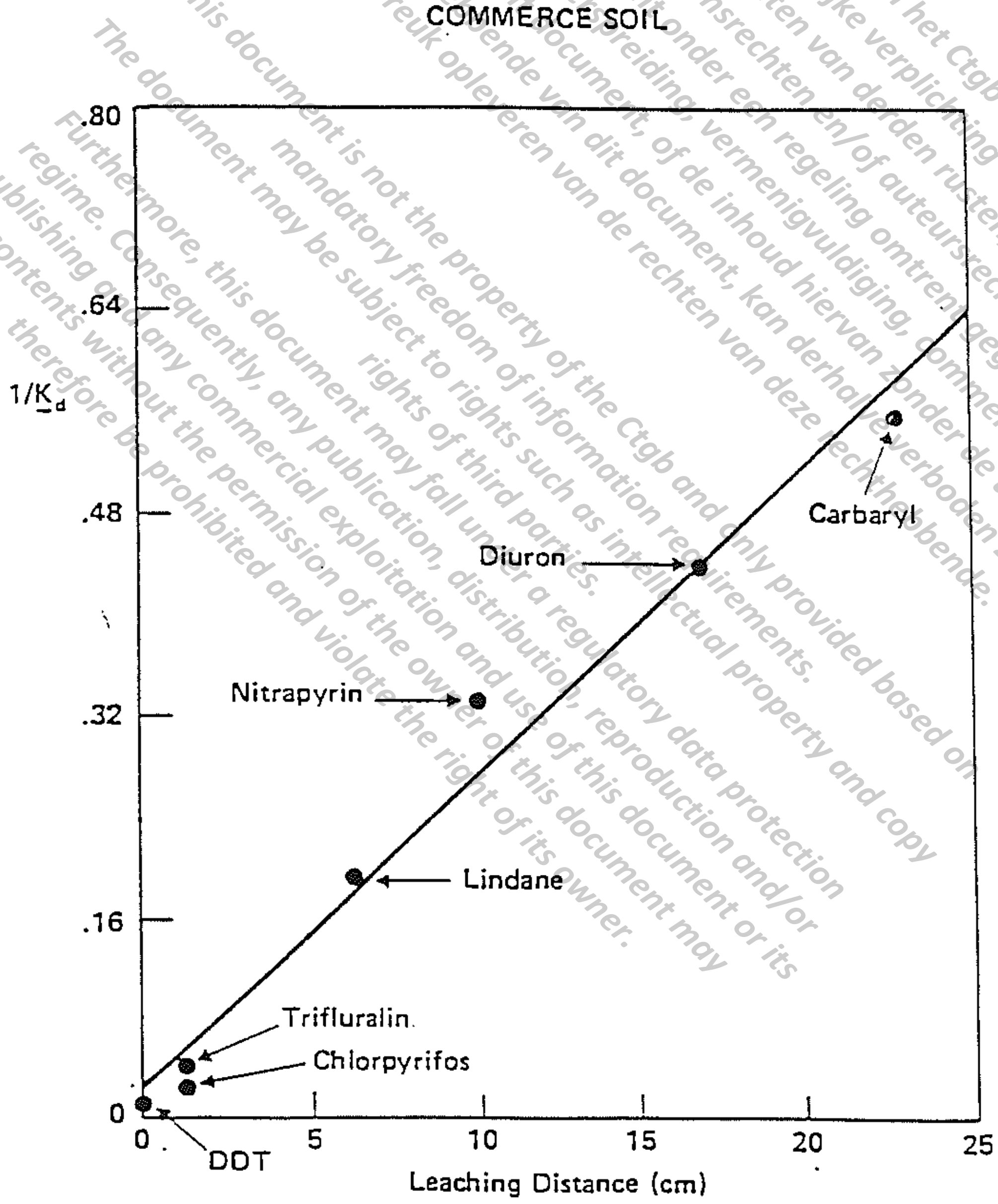
where

$\underline{R}$ = $\frac{\text{cm moved by chemical}}{\text{cm of water entering}}$ the soil

θ = pore fraction of the soil
$\underline{d}_s$ = bulk density of soil solids
$\underline{K}_{oc}$ = sorption constant
%O.C. = percent organic carbon

The distance moved by the chemical refers to the position of the maximum concentration peak, such that peak spreading is not considered. However, it is a useful relationship for assessing the relative leaching potential of chemicals in soil. McCall et al. (22,31) have recently demonstrated this expression in laboratory studies conducted with soil columns. For a soil containing a given amount of organic carbon, the equation can be expressed in terms of $\underline{K}_d$ and then the distance a chemical will move for a given amount of water entering the column will be proportional to $1/\underline{K}_d$. A plot of $1/\underline{K}_d$ vs cm moved for seven chemicals upon addition of 20 cm of water to a soil column is shown in Figure 8.

FIGURE 8. Inverse relationship of leaching distance with soil sorption coefficient, $\underline{K}_d$



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This relationship will be applicable for neutral organic compounds whose sorption can be characterized by a $\underline{K}_{oc}$ value. For a given amount of organic carbon a $\underline{K}_d$ value can be calculated and related to leachability of the chemical in a particular soil. In a more general sense, K_{oc} values can be used to rank and compare chemicals with respect to their leaching potential for the purpose of general evaluation. A proposed ranking system is shown in Table 3.

TABLE 3. Classification of Soil Mobility Potential of Chemicals from HPLC Retention Times

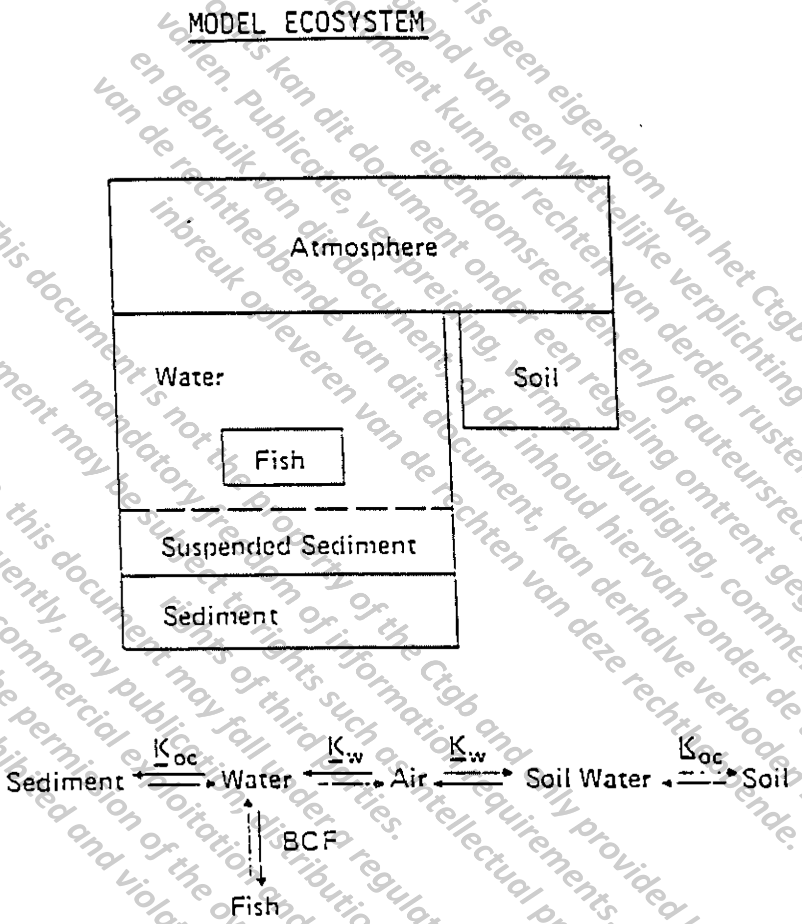
$\underline{K}_{oc}$	Mobility class
0-50	very high
50-150	high
150-500	medium
500-2000	low
2000-5000	slight
>5000	immobile

This work indicates that $\underline{K}_{oc}$ values alone can provide a reliable estimate of a chemical's mobility in soil without performing demonstrative tests such as column leaching or soil TLC studies. The results of these methods are highly dependent on the conditions under which they are conducted and are therefore very difficult to standardize. $\underline{K}_{oc}$ values are easily converted to $\underline{K}_d$ values which can then be plugged into models descriptive of leaching behavior in the environment.

The tendency of a chemical to distribute among various compartments of the environment will be related to its physical properties and partition coefficients. The soil sorption constant, $\underline{K}_{oc}$, will be an integral factor affecting environmental distribution patterns. Its overall influence on environmental behavior can only be evaluated in perspective with other partitioning processes together with transformation mechanisms in a collective type of model. Sophisticated models, such as EXAMS, deal with dynamic systems and describe kinetic processes related to rates of migration and transformation. Simple equilibrium models such as those described by MacKay (32) and McCall et al. (33) can evaluate the overall environmental distribution potential of a chemical between various compartments. These types of models are useful in giving insight into tendencies of chemicals to move to various compartments.

As an example, if the model ecosystem (33) shown in Figure 9 is considered, several chemicals representing a wide range of sorption properties as well as other properties can be evaluated,

FIGURE 9. Model ecosystem (33)



Atmosphere	1000m x 1000m x 10Km = 10^{10} m^3
Water	1000m x 300m x 10m = 3 x 10^6 m^3
Soil	1000m x 700m x .076m = 5.4 x 10^4 m^3
Sediment	1000m x 300m x .05m = 1.5 x 10^4 m^3
Suspended Sediment	10 ppm in water = 15m^3
Fish	1 ppm in water = 3m^3
Soil Organic Carbon	= 2%
Sediment Organic Carbon	= 4%

assuming complete equilibrium exists throughout the system. Physical properties of seven chemicals are given in Table 4 and their distribution patterns are shown in Table 5. A chemical such as DDT which has a very high sorption constant tends to reside primarily in the soil and sediment; however, tetrachlorobiphenyl which also has a relatively large $\underline{K}_{OC}$ value tends to go to the air because its potential to volatize is effectively larger than its

TABLE 4. Physical Properties and Partition Coefficients

Chemical	MW	WS(ppm)	$\underline{V}p$(mmHg)	$\underline{K}_w$	$\underline{K}_{OC}$	BCF
DDT	354.5	0.0017	1.9×10^{-7}	470	150,000	61,600
Tetrachlorobiphenyl	290	0.017	4.9×10^{-4}	2	32,500*	72,950
Lindane	290.8	0.15	3.2×10^{-5}	300	1,300	325
Chlorpyrifos	351	1.2	1.9×10^{-5}	3340	6,100	470
Nitrapyrin	230.9	40	2.8×10^{-5}	1150	560	82*
2,4-D	221	900	6.0×10^{-7}	1.3×10^{8}	60	3*
1,3-Dichloropropene	111	2700	25	18	68*	3*

*Estimated properties.

TABLE 5. Chemical Distribution Using Model Ecosystem

Chemical	DDT	Tetrachlorobiphenyl	Lindane	Chlorpyrifos	Nitrapyrin	2,4-D	1,3-Dichloropropene
in							
air	4.72	97.9	83.2	12.8	65.4	0.0025	99.4
water	0.65	0.065	7.48	12.8	22.5	93.8	0.54
soil	44.8	0.96	4.41	35.3	5.78	3.14	0.02
sediment	49.8	1.06	4.86	39.1	6.31	3.06	0.02
suspended sediment	0.05	0.001	0.004	0.04	0.006	0.003	1.8×10^{-5}
fish	0.04	0.0048	0.0024	0.0060	0.0078	0.0003	1.7×10^{-6}

potential to become sorbed. Chemicals like 2,4-D and lindane which have vastly different sorption constants have similar amounts of chemical in the soil and sediment compartments of this model system. In the case of lindane, the chemical tends to partition into air, whereas with 2,4-D, the water compartment predominates.

In models of this simple type, or in more complex models, the validity of the predictions is based in the reliability of the parameters input. In the case of sorption constants, a $\underline{K_{OC}}$ value which is independent of soil parameters fits into these types of evaluation mechanisms quite adequately.

Summary

The sorption of chemicals to soil and sediments represents an important environmental process which affects the migratory behavior of a chemical in the various compartments of the environment. In order to characterize chemical transport with various models developed to evaluate environmental fate a solid data base must be established. Standard principles and techniques must be followed in order to arrive at data which are meaningful and useful to the modeling effort.

The development of the $\underline{K_{OC}}$ concept to characterize the chemical's sorption properties can be useful in reaching this goal. Attention to proper experimental methods is required to minimize variations in the data and to make the data suitable for hazard evaluation schemes. It is realized that a correlation with organic carbon may not apply for all chemicals, that cation exchange capacity, pH, or some other soil/sediment property may influence sorption. However, for the large class of neutral organic compounds which are often subjects of environmental concern, the relationship generally applies. Acceptability of this value to represent sorption will depend on the accuracy required in its usage.

Site specific evaluations requiring detailed information regarding the nature of the sorbent interaction may require additional information on an individual basis. However, in the application of models to evaluate chemicals regarding their expected environmental hazard in a general format, $\underline{K_{OC}}$ values can adequately describe the general sorption properties of a chemical.

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STANDARD METHODS

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c. Sterility controls: Check sterility of medium and dilution water blanks by pouring control plates for each series of samples. Prepare additional controls to determine contamination of plates, pipets, and room air.

4. Incubation

See Section 907.7.

5. Counting, Recording, Computing, and Reporting

See Sections 907.8 and 907.9.

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907 B. Spread Plate Method

1. Laboratory Apparatus

a. Glass rods: Bend 4-mm-diam fire-polished glass rods, 200 mm in length, 45° about 40 mm from one end. Sterilize before using.

b. Pipet, glass, 1.1 mL, with tempered, rounded tip. Do not use disposable plastic pipets.

c. Turntable (optional).*

d. Incubator or drying oven, set at 42°C, or laminar-flow hood.

---

*Fischer Scientific, hand operated, No. 08-758 or Lab-Line motor driven, No. 1580, or equivalent.

2. Media

See 907.6. If R2A agar is used best results are obtained at 28°C with 7 d incubation.

3. Preparation of Plates

Pour 15 mL of the desired medium into sterile 100 × 15 or 90 × 15 petri dishes; let agar solidify. Predry plates inverted so that there is a 2- to 3-g water loss overnight with lids on. See Figure 907:2, Table 907:I, or Figure 907:3. Use predried plates immediately after drying. For predrying and using plates the same day, pour 25 mL agar into petri dish and dry in a laminar-flow hood at room temperature (24 to 26°C) with the lid off to obtain the desired 2- to 3-g weight loss. See Figure 907:3.

4. Procedure

Prepare sample dilutions as directed in 907A.2.

a. Glass rod: Pipet 0.1 or 0.5 mL sample onto surface of predried agar plate. Using a sterile bent glass rod, distribute inoculum over surface of the medium by rotating the dish by hand or on a turntable. Let inoculum be absorbed completely into the medium before incubating.

b. Pipet: Pipet desired sample volume (0.1, 0.5 mL) onto the surface of the predried agar plate while dish is being rotated on a turntable. Slowly release sample from pipet while making one to-and-fro motion, starting at center of the plate and stopping 0.5 cm from the plate edge before returning to the center. Lightly touch the pipet to the plate surface. Let inoculum be absorbed completely by the medium before incubating.

5. Incubation

See 907.7.

6. Counting, Recording, Computing, and Reporting

See 907.8 and 907.9.

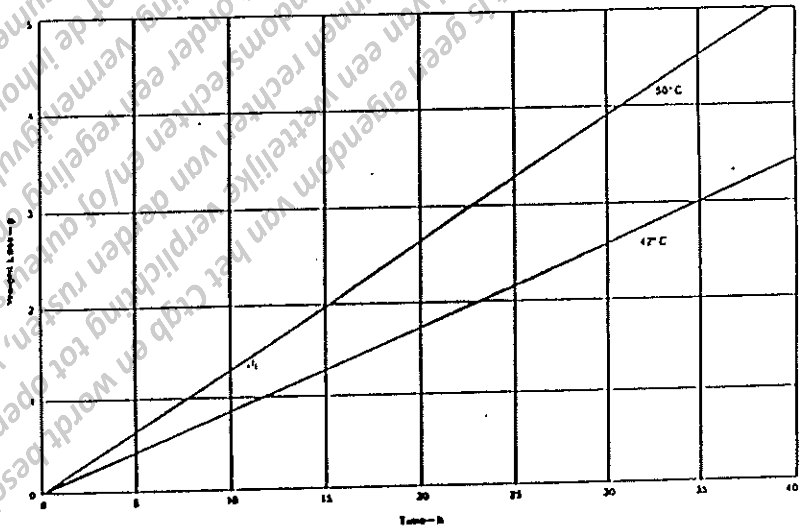

Figure 907:2. Drying weight loss of 15-mL agar plates stored separately, inverted with lids on. SOURCE: Unpublished data. Water Purification Lab., Chicago Dep. Water.

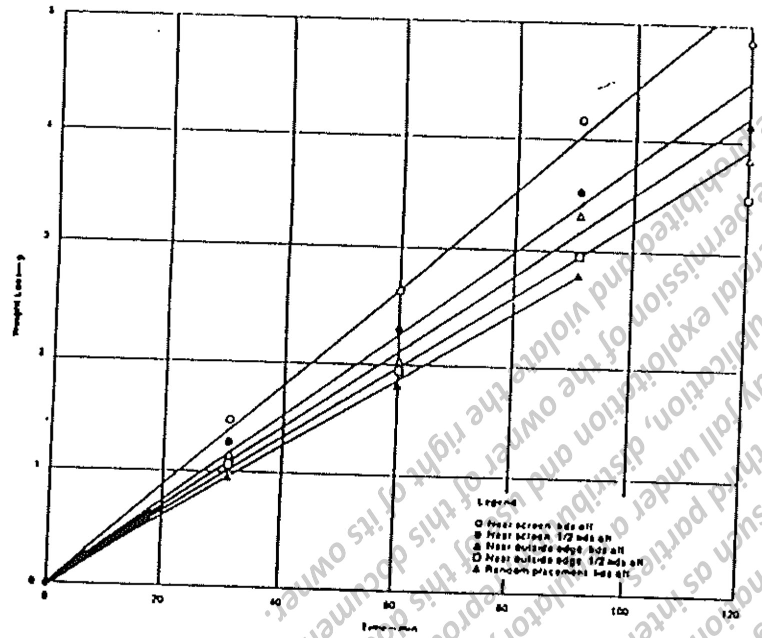

Figure 907:3. Weight loss of 25-mL agar plates (100 × 15 mm) dried separately in a laminar-flow hood at room temperature (24 to 26°C), relative humidity (30 to 33%), and air velocity 0.6 m/s. SOURCE: Unpublished data, Alberta Environmental Centre, Vegreville, Alta.

TABLE 907:I. EFFECT OF TEMPERATURE OF DRYING ON WEIGHT LOSS OF 15 ML AGAR PLATES STORED SEPARATELY*

Temp. °C	Time for Plates to Lose 1 to 4 g of Water (Avg. for 5 Plates) h — Plates Inverted with Lids On				Plates Inverted with Lids Removed			
	1 g	2 g	3 g	4 g	1 g	2 g	3 g	4 g
24	32	64	95	125	3.7	7.0	10.5	14.0
37	17	35	51	67	1.7	3.5	5.3	7.0
50	6	12	18	24	0.7	1.3	1.9	2.7
60	4	8	12	16	—	—	—	—

* Referenced in Canada Centre for Inland Waters Manual, Burlington, Ont.

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907 C. Membrane Filter Method

1. Laboratory Apparatus

See Section 909A.1.

2. Media

See 907.6. Use m-HPC agar, or alternatively R2A agar.

3. Preparation of Plates

Dispense 5-mL portions of sterile medium into 50- × 9-mm petri dishes. Let solidify at room temperature. Prepared plates may be stored inverted up to 1 week in a refrigerator in a plastic bag or tight container.

4. Sample Size

The volume to be filtered will vary with the sample. Select a maximum sample size to give 20 to 200 CFU per filter.

5. Procedure

Filter appropriate volume through a sterile 47-mm, 0.45-µm, gridded membrane filter, under partial vacuum. Rinse funnel with three 20- to 30-mL portions of sterile dilution water. Place filter on agar in petri dish.

6. Incubation

Place dishes in close fitting box containing moistened paper towels. Incubate at 35 ± 0.5°C for 48 h if using m-HPC agar, or longer if using R2A medium. Duplicate plates may be incubated for other time and temperature conditions as desired.

7. Counting, Recording, Computing, and Reporting

See 907.8 and 907.9. Report as CFU/mL, membrane filter method, time, medium.

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CHAPTER NINE

Analysis of Data in Radioactivity Measurements

In analyzing the apparent counting data observed in the assay of radioactive samples, two basic considerations are of primary importance. First, how statistically reliable are the data; second, what relation exists between the apparent counting rate of the sample and the actual disintegration rate? It is necessary to subject radioactivity measurement data to statistical analysis, because radioactive decay is a strictly random phenomenon. Needless to say, rigorous statistical treatment is especially essential when low levels of radioactivity are being assayed. A number of general references to the application of statistical methods to radioactivity measurement are given in the chapter bibliography; of these, Jarrett's (12) is the most comprehensive. Herberg's recent papers are particularly important in liquid scintillation counting (7, 8).

Several phenomena prevent all the sample disintegrations from being detected or, on the other hand, lead to the counting of events not related to sample disintegrations. Correction factors for these interferences must be applied to the apparent counting data if one wants to cross-compare measurement data of samples obtained under different circumstances. Many of these factors can be standardized from assay to assay, thus avoiding the necessity of making corrections, but the principle underlying this standardization must be understood.

Radiotracer experiments are, of course, subject to the typical errors of all chemical operations: weighing, volume measurements, the chemical (and radiochemical) purity of reagents used, and general competence of laboratory technique. Such errors are not considered at length in this chapter, but they cannot be ignored in practice.

A. STATISTICAL CONSIDERATIONS

1. Error Probability

As has been discussed in Chapter 2, radioactive decay is a random phenomenon. This can be readily demonstrated by making repeated measurements of the activity of a long-lived nuclide, each for the same duration of time (see Experiment VII). The results will not be identical, but rather varied over a range of values with a clustering near the center of the range. If a sufficiently large number of such measurements are made and the data plotted, a curve such as that shown in Figure 9-1 will result. This is the familiar *normal distribution curve*, which for most radioactivity measurements is a close approximation of the Poisson distribution curve.

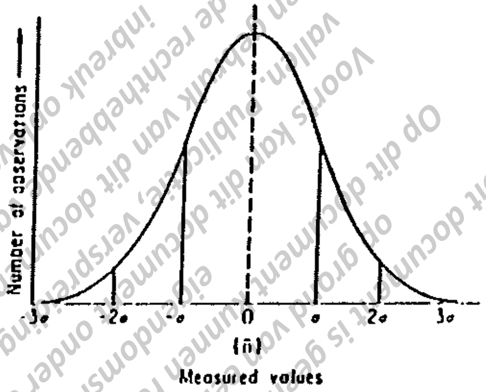

Figure 9-1. A normal distribution curve.

Because of this situation, one obviously cannot know the "true" value of the result of a given counting operation. From the Poisson distribution equation, it can, however, be shown that a mean value ($\bar{n}$) of a given counting is the best estimate of the "true" value when large numbers of determinations are involved. The accuracy of any individual measurement (n) is gauged by the magnitude of its deviation (Δ) from the mean value, or

$$\Delta = n - \bar{n} \tag{9-1}$$

On the plot in Figure 9-1, the vertical dashed line through the apex of the curve represents the $\bar{n}$ value and is arbitrarily labeled zero.

Note that the distribution of positive and negative deviations from the mean value of any given magnitude are of equal occurrence and that the occurrence of small deviations is more probable than that of large ones. The probability of any deviation ("error") being as large or larger than a given value may be calculated. The basic "error" unit used is the standard deviation (σ). For practical purposes, the *standard deviation* can be defined as the square root of the mean value, or $\sigma = \sqrt{\bar{n}}$. Only 31.7 per cent of the values on a normal curve are as large or larger than $\pm 1\sigma$. All other error units are expressed in terms of the standard deviation. Table 9-1 lists the names and values of the common measures of error probability.

TABLE 9-1

Measures of error probability

Name of Error	Deviation / Standard Deviation (Δ/σ)	Probability of an Error Exceeding Such a Deviation
Probable error	0.6745	0.5000
Standard error (deviation), one sigma	1.0000	0.3173
90% error	1.6449	0.1000
95% error	1.9600	0.0500
Two sigma	2.0000	0.0455
99% error	2.5758	0.0100
Three sigma	3.0000	0.0027

The foregoing discussion dealt with repeated measurements on the same sample, but in radiotracer assay it is common to make only one or two activity determinations per sample. In such cases there is no $\bar{n}$ value from which σ may be calculated. Because of the nature of the Poisson distribution, however, the standard deviation (σ) of a single determination of magnitude n may be taken as $\sqrt{n}$, that is, the square root of the total number of counts collected. A related unit, the *relative standard deviation* (or relative σ) is defined as $\sqrt{n} \times 100/n$, or $100/\sqrt{n}$. Thus, the larger the value of n (number of counts collected), the smaller the relative standard deviation. To realize a given relative standard deviation for a given counting operation it is necessary to continue counting only until sufficient counts are collected.

Radiotracer measurements are usually made to have an accuracy equivalent to 1 per cent standard deviation. In order to achieve this specification, it is necessary to collect 10,000 counts from the radioactive sample in question, since when $100/\sqrt{n} = 1$ per cent, $n = 10{,}000$. This is irrespective of the length of time required to collect this number of counts. From this single assay one could then state that there is a 68.3 per cent probability that the true total count was 10,000 ± 100 counts, or (from Table 9-1) a 95 per cent probability that the total count was 10,000 ± 196 counts.

2. Contribution of Background Error

The 10,000 counts collected from the foregoing sample represent a combination of two factors, namely, sample activity and background. The standard deviation of this measurement is therefore actually a combination of the errors of each separate factor. When the ratio of gross sample counting rate (counts per unit time) to background counting rate is high (over ten-fold), the standard deviation of the net sample radioactivity can be calculated simply by taking the square root of the gross counting result. As the ratio becomes smaller, however, the contribution of the background error to the error of the gross sample counts becomes increasingly significant. Furthermore, it is unlikely that the same counting duration will be used for both the sample and the background determination. It is necessary under these circumstances to consider the standard deviations of the counting rates of the sample and the background separately, before the standard deviation of the net sample counting rate can be defined. This is true since the net sample counting rate is derived from the difference of two independent counting operations, that is, gross sample counts (sample plus background) and background.

The standard deviation of the net sample counting rate (σ_s) is equal to the square root of the sum of the squares of the standard deviations of the gross sample count rate (σ_g) and the background count rate (σ_b), or

$$\sigma_s = \sqrt{\sigma_g^2 + \sigma_b^2} \tag{9-2}$$

But the best estimate for the standard deviation of a rate is the square root of the counting rate divided by the counting time ($\sqrt{r/t}$). Thus, $\sqrt{r_g/t_g}$ may be substituted for σ_g and $\sqrt{r_b/t_b}$ may be substituted for σ_b in Equation 9-2 to yield

$$\sigma_s = \sqrt{\frac{r_g}{t_g} + \frac{r_b}{t_b}} \tag{9-3}$$

As an example of the effect of background error on the standard deviation of the net sample count rate, suppose that a sample is assayed for 40 min and 10,000 counts collected. The background is measured for 10 min and a total of 1000 counts registered. Thus, the gross sample count rate is 250 cpm, the background is 100 cpm, and the net sample activity is found to be 150 cpm. Ignoring the contribution of background error and calculating the standard deviation as the square root of the gross sample counts, the result could be reported as 150 ± 2.5 cpm. Using Equation

9-3, which takes background error into account, the results would be as follows:

$$\sigma_n = \sqrt{\frac{250}{40} + \frac{100}{10}} = 4.0$$

Thus, the more accurate expression of the counting data would be 150 ± 4.0 cpm. Such error differences assume great importance in certain types of radiotracer experiments and cannot be taken lightly.

3. Requirements of Counting Time for Sample and Background

As indicated previously, in a sample having a counting rate sufficiently high in comparison to the background counting rate, one can readily collect enough counts to insure an acceptable relative standard deviation of the counting operation without worrying about the contribution of the background to the over-all counting process. Such is not the case when one encounters a sample with a low counting rate. If the counting rate of the sample is only fourfold that of the background, it is necessary to count the sample and the background over defined durations of time so that the relative standard deviation of each of the two counting operations is of comparable magnitude. To this end, the ratio of background counting time (t_b) to gross sample counting time (t_g) is related to the ratio of the counting rates of the background (R_b) and the sample (R_g) as follows:

$$\frac{t_g}{t_b} = \sqrt{\frac{R_g}{R_b}} \tag{9-4}$$

Obviously, when the ratio of R_g/R_b approaches unity, in order to realize a respectable relative standard deviation of the net sample counting rate, one must count the sample as well as the background over a long period of time. The requirements of counting time for various ratios of R_g/R_b are given in Table 9-2. To avoid lengthy counting operations, it is, of course, essential to design a radiotracer experiment in such a way that the counting rate of the final counting sample is considerably higher, preferably tenfold or more, than that of the background radiation. By the same token, it is important to avoid "technical" contamination in a radiotracer laboratory from the careless handling or avoidable spillage of small amounts of radioactive material. Although such technical contaminations do not constitute a health hazard, they will undoubtedly raise the background observable in radiation detectors, and hence, lengthen the required counting time.

Browning (1) used the foregoing relation as a base for charts determining optimum distribution of counting time. Freedman and Anderson (5)

TABLE 9-2

Counting times required for a given standard error of net activity with a background counting rate of 30 cpm [from Calvin, et al. (2, p. 288)]

Ratio of Sample Counting rate to Background Counting Rate (R_g/R_b)	Standard Error in Net Counting Rate (R_n)[*]							
	1%		2%		5%		10%	
	Background Counting Time [in min[†] (t_b)]	Sample Counting Time [in min (t_g)]	Background Counting Time [in min (t_b)]	Sample Counting Time [in min. (t_g)]	Background Counting Time [in min. (t_b)]	Sample Counting Time [in min (t_g)]	Background Counting Time [in min (t_b)]	Sample Counting Time [in min (t_g)]
1.1	68,300	71,100	17,100	18,000	2,730	2,870	683	717
1.2	17,500	19,100	4,360	4,780	698	764	175	191
1.4	4,550	5,380	1,140	1,350	182	215	46	54
1.7	1,570	2,050	393	513	63	82	16	21
2.0	804	1,140	201	284	32	46	8	11
2.5	383	606	96	152	15	24	4	6
3.0	225	395	57	99	9	16	2	4
3.5	153	286	38	72	6	11	2	3
4.0	111	222	28	56	4	8	1	2
5.0	68	151	17	38	3	6	1	2
6.0	46	113	12	28	2	5	1	1
7.0	34	89	8	22	1	4	1	1
8.0	26	74	7	18	1	3	1	1
9.0	21	62	5	16	1	3	1	1
10.0	17	54	4	14	1	2	1	1

[*] Net counting rate (R_n) = sample counting rate (R_g) − background counting rate (R_b).

[†] Short times are rounded off to nearest minute greater than zero.

and Hughes (9) discuss the problem of counting time in the assay of very low levels of activity.

4. Rejection of Abnormal Data

Thus far, error based on the randomness of radioactive decay has been the primary consideration. Other factors, such as variation in sample preparation procedures, errors in measuring quantities, contamination of the detector, or mislabeling and confusion of samples, also contribute to errors in radioactivity measurement data. As a result, when repeated assays of similar samples are made under seemingly identical circumstances, one occasionally finds that one measurement differs from the others by a large amount. Since the number of observations is usually limited, one abnormal value can introduce considerable error into the over-all average, which is used as the best estimate of the "true" value. Various criteria for rejecting such suspected data are in use (10, 18), but most investigators prefer *Chauvenet's criterion*; namely, an observation should be rejected if the probability of its occurrence is equal to, or less than, ½N, where N represents the number of observations involved. This relation is more conveniently used in terms of the ratio of the deviation of the suspected value to the standard deviation of the series of observations $[(n - \bar{n})/\sigma]$. Table 9-3 lists values of $(n - \bar{n})/\sigma$ for various numbers of observations (N). If the calculated value of $(n - \bar{n})/\sigma$ for a suspected observation exceeds that given in Table 9-3 for the appropriate number of observations, the value is discarded.

For simplicity, some investigators reject suspected values which deviate from the mean of a series by more than 2σ or 3σ. The probabilities of the occurrence of such deviations are 4.5 per cent and 0.27 per cent, respectively.

TABLE 9-3

Values for use with the Chauvenet criterion

Number of Observations (N)	Ratio of the Individual Deviation to the Standard Deviation $[(n - \bar{n})/\sigma]$	Number of Observations (N)	Ratio of the Individual Deviation to the Standard Deviation $[(n - \bar{n})/\sigma]$
2	1.15	15	2.13
3	1.38	20	2.24
4	1.54	25	2.33
5	1.65	30	2.40
6	1.73	35	2.45
7	1.80	40	2.50
8	1.86	50	2.58
9	1.91	75	2.71
10	1.96	100	2.81
12	2.04	200	3.02

B. CORRECTION FACTORS IN RADIOTRACER ASSAY

In ordinary radioactivity assay encountered in radiotracer experiments, the observed counting rate is not equal to the true disintegration rate of the sample, but is nevertheless related to it. A number of factors affect the relation between absolute disintegration rate and the observed counting rate. Some tend to increase the observed counting rate, but most tend to decrease it in relation to the disintegration rate. Of these factors, some are inherent in the detectors, others arise from the nature of the counting sample, and still others are related to the sample-to-detector arrangement. Of the many recognized factors, only a few are important in connection with radiotracer experimentation. Figure 9-2 illustrates those that are relatively more important in relation to an end-window G-M detector. The factors to be described in detail are largely of importance in relative beta measurements, but some apply equally to gamma assay. Even though only relative measurements are being made,

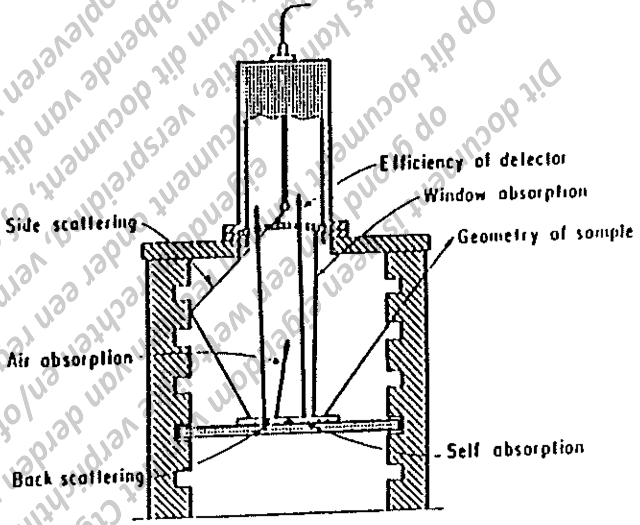

Figure 9-2. Factors in G-M radiotracer assay.

it is necessary to understand the origin and the effect of these factors and the corrections to be made for them.

1. Background

The background count rate registered on a radiation counter can come from such varied sources as cosmic radiation, natural radioactivity in the vicinity, artificial radioactivity (fallout), nearby X-ray generators, and thermal or other circuit noise. Several means are employed to reduce the background counting rate for various counter assemblies. Detector shielding with lead or iron is most widely utilized. In addition, coincidence circuitry and photomultiplier cooling in liquid scintillation counters, and anticoincidence circuitry in special low background G-M counters are exceptionally effective in reducing background count, in some cases to the level of 10 counts per hour (44). Regardless of such background reduction, the gross sample count rate (m_s) always includes some background count rate (m_b). The net observed counting rate (m_o) due to sample activity alone is thus,

$$m_o = m_s - m_b \tag{9-5}$$

Scales (41) has discussed in detail the problem of determining background counts in liquid scintillation counting, where the complication of quenching also occurs.

By ignoring some minor factors, the net observed counting rate can now be related to the absolute disintegration rate (D) of a sample as follows:

$$m_o = DE f_\tau f_b f_w f_s \tag{9-6}$$

where E represents the correction for geometry and detector efficiency, f_τ stands for the coincidence loss factor, f_b is the backscatter factor, f_w represents the factor for window and air absorption, and f_s stands for the self-absorption factor. In gamma ray measurements, the latter three factors may usually be ignored.

2. Geometry

Since sample radiation is emitted equally in all directions, the placement of the detector with respect to the sample is clearly a primary limiting factor on the percentage of disintegrations that will be detected. If Ω represents the solid angle subtended by the sensitive volume of the detector, then $\Omega/4\pi$ equals the fraction of the disintegrations that would be directed toward that sensitive volume. Figure 9-3 indicates this relationship and defines the terms of d, r, and α. The value of $\Omega/4\pi$ for an ideal point source may be calculated as follows:

$$\frac{\Omega}{4\pi} = \frac{1}{2}(1 - \cos\alpha) = \frac{1}{2}\left(1 - \frac{d}{\sqrt{d^2 + r^2}}\right) \tag{9-7}$$

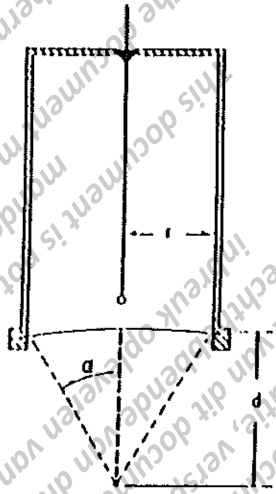

Figure 9-3. Geometry of an end-window G-M detector.

In practice a counting sample is spread over an often considerable area and is an extended source, rather than a point source. It is generally recognized that the size of the source should be as small as possible and should by no means approach the size of the detector window.

Since it is extremely difficult to calculate accurately the solid angle for extended sources, for comparative measurements it is strongly recommended that counting samples be prepared of uniform size and distribution and that they be placed at a reproducibly identical distance from the detector. A sample holding rack with several shelves usually provides for this latter feature. Where desired, the relative values of $\Omega/4\pi$ (shelf ratios) for the various shelf positions of a sample holder can be determined as indicated in Experiment 1, Section B-5.

The value of Ω for G-M and proportional end-window detectors is inherently less than 2π, and frequently less than 1π (27). For G-M and proportional windowless detectors, where the sample is introduced into the detector, the solid angle approximates 2π. In counting with liquid scintillation detectors and internal-sample ion chambers, the geometry involved approaches 4π. An arrangement in which two windowless G-M detectors are placed face to face and the sample suspended in the center (a "4π counter") is widely used to standardize beta sources. A common method for greatly increasing the geometrical efficiency of NaI (Tl) crystal scintillation detectors is the employment of "well" crystals, where the counting sample (usually liquid in a vial) can be inserted deep into the crystal. In this case, sample volume is limited, since geometrical efficiency decreases sharply as the sample volume reaches and exceeds the well capacity (22, 31, 47).

3. Detector Efficiency

The factor E in Equation 9-6 is made up of the geometry factor $\Omega/4\pi$ and the intrinsic efficiency factor (f_i) of the given detector for the radiation from a specific nuclide. Factor f_r represents the fraction of radiation within the sensitive volume of a detector that is actually detected. For G-M detectors the values of f_r are 100 per cent, about 96 per cent, and 1 per cent for alpha, beta, and gamma radiation, respectively. With a NaI(Tl) crystal scintillation detector, the gamma detection efficiency varies from 10-60 per cent depending on the photon energy. Excessively high counting rates can, however, lead to a subsequent temporary decline in efficiency due to photomultiplier fatigue (19). Alpha and beta particles would not normally penetrate to the sensitive crystal, but even so would be very poorly detected. In an ion chamber, alpha and low-energy beta particles (^{14}C, ^{35}S) would be nearly 100 per cent detected, if the chamber was sufficiently large. Detection efficiencies would decrease with increasing beta energy, and gamma rays would pass through virtually undetected. The value of f_r in liquid scintillation detectors is strongly dependent on beta particle energy, degree of quenching, nature of the solvent and fluor, concentration of fluor, and the like (see Chapter 6).

Not only do detectors show a different intrinsic efficiency for different nuclides, but the sensitivity of a specific detector to a given nuclide will vary over a period of time. This problem particularly plagues scintillation and proportional counters in which the detection efficiency is a function of detector potential. Even slight fluctuations in this potential can offset detection efficiency noticeably. For this reason, stability of the high-voltage supply for these counters is essential. In addition, G-M detectors may show a variation in radial response within the sensitive volume of the tube (21).

In organically quenched G-M detectors, failure of the quenching mechanism may occur with increasing age, resulting in multiple pulses for a single ionizing event. The factor f_m (for multiple-pulsing) must then be incorporated into factor *E*. Generally speaking, however, multiple pulses are not a significant factor in counting with G-M detectors. Similarly, electrons may be created in a photomultiplier tube without a photon interaction. Such a phenomenon may create a serious problem in γ-ray spectrometry.

In order to determine the absolute value of *E*, it would be necessary to calibrate the detector with a standard source of the same nuclide to be later assayed, making the measurements under physical conditions identical to those later to be used (29). In most radiotracer assays, however, only relative values of *E* need be known for the purpose of correcting counting rates for variation in efficiency with time. This is readily accomplished by making routine measurements of the activity of a standard source, such as $Ba^{14}CO_3$, $Na_2{}^{14}CO_3$, toluene-^3H, or U_3O_8. These and many other radionuclide standards can be obtained from the National Bureau of Standards or commercial sources. Any variation in the counting rate of this standardized source over a few days or weeks can be attributed to a change in detector efficiency and all sample counts correspondingly corrected.

4. Coincidence Loss

A radiation counting assembly requires a finite time to clear the pulse created by each radiation particle. During this period, other incident radiation is either not detected or not differentiated. The result of this failure to resolve two incident radiation particles that are closely spaced in time is coincidence loss. Note that disintegrations are not evenly spaced in time. The minimum time interval between which two events can be registered is termed the *resolving time* of the counter.

Coincidence loss has several sources in a counting assembly. Loss can occur due to the amplifier circuitry. Normally this is negligible, since the better-quality amplifiers have resolving times of 1-5μsec. In the economical "training" scalers, however, this may be as high as 250μsec. A more common source of coincidence loss is the mechanical register used to record counting events. These registers, being very slow devices, must be used with scaling circuits to avoid coincidence loss. It is desirable to use a high scaling factor to group a number of pulses prior to the mechanical registers (100, or 1000 with decade scalers; 128 or 256 with binary scalers), if fast counting is anticipated. Davidon (28) has prepared a nomogram for computing such register losses. Recently, it has become common to eliminate the mechanical register altogether and to substitute a series of scaling units employing cold cathode decade tubes or similar devices. To accommodate the very high counting rates attainable with scintillation and proportional detectors, it is necessary to employ an initial "hard valve" scaling unit, which is obtainable with a 1-5μsec resolving time.

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29

Total Carbon, Organic Carbon, and Organic Matter[1]

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29-1 GENERAL INTRODUCTION

This chapter is an updated, revised version of the material contained in Chapters, 89, 90, and 92 of *Methods of Soil Analysis* (Black et al., 1965). Much of the material presented in the original versions is contained in this revised chapter, because methodology for C and organic matter has changed little in the past 15 years and because of the excellence of the original chapters. However, some of the methods described in the original chapters have been modified or replaced by more modern procedures, and recent literature pertaining to the methods has been included. In addition, the total C section has been modified to include a subsection dealing with automatic or semiautomatic instruments currently available.

Total C in soils is the sum of both organic and inorganic C. Most organic C is present in the soil organic matter fraction, whereas inorganic C is largely found in carbonate minerals. Not all soils contain inorganic C because of dissolution during soil formation of carbonate minerals originally present in parent material. However, organic C is present in all agricultural soils. In soils formed from calcareous parent material under arid conditions, it is not unusual for the inorganic C concentration to exceed the amount of organic C present.

Organic C is contained in the soil organic fraction, which consists of the cells of microorganisms, plant and animal residues at various stages of decomposition, stable "humus" synthesized from residues, and highly carbonized compounds such as charcoal, graphite, and coal (elemental forms of C). Organic C in soil may be estimated as the difference between total C and inorganic C. Organic C can be determined directly by total C procedures after removal of inorganic C or by rapid dichromate oxidation-titra-

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tion techniques. Procedures involving a total C analysis generally recover all forms of organic C in soils, whereas dichromate oxidation procedures recover variable proportions of elemental C (e.g., charcoal) and, in some procedures, variable amounts of organic C contained in "humus."

Calcite and dolomite are the principal carbonate minerals present in soil, and most inorganic C is associated with these compounds. However, in some alkaline soils, significant amounts of inorganic C may be present as soluble carbonate-CO_3^{2-} and HCO_3^- salts. The amounts of soluble carbonates present in soil may be determined by procedures outlined in section 20-3.2, and a variety of methods for the estimation of total inorganic C in soils are presented in section 11-2.

Total C determinations for soil involve conversion of all forms of C in soils to CO_2 by wet or dry combustion and subsequent quantitation of evolved CO_2 by gravimetric, titrimetric, volumetric, spectrophotometric, or gas chromatographic techniques. Dry combustion is carried out by heating (~1,000°C) a soil-catalyst mixture in a resistance furnace or induction furnace in a stream of O_2 or CO_2-free air, followed by quantitation of evolved CO_2. Wet combustion is normally carried out by boiling a soil sample with a mixture of $K_2Cr_2O_7$, H_2SO_4, and H_3PO_4 in a closed system flushed with a stream of CO_2-free air and absorption of evolved CO_2 in a tared weighing bulb filled with Ascarite (Allison, 1960). Alternatively, wet combustion may be carried out in a Van Slyke-Neil apparatus and evolved CO_2 estimated by manometric procedures (Bremner, 1949). Two dry combustion and one wet combustion procedures for total C analysis are described in this chapter.

Soil organic matter has been defined as the organic fraction of soil, including plant, animal, and microbial residues, fresh and at all stages of decomposition, and the relatively resistant soil humus (SSSA, 1979). However, soil organic matter includes only those organic materials that accompany soil particles through a 2-mm sieve. It is difficult to quantitatively estimate the amount of organic matter present in a soil. Procedures used in the past involve determination of the change in weight of a soil sample resulting from destruction of organic compounds by H_2O_2 treatment or by ignition at high temperature. Both techniques are subject to error. The H_2O_2 method does not quantitatively remove organic matter, and the ignition method gives an overestimate because both inorganic and organic constituents lose weight during heating. Alternatively, the organic matter content of a soil may be estimated by multiplying the organic C concentration by a constant factor based on the percentage of C in organic matter. Published organic C-organic matter conversion factors for surface soils have varied from 1.724 to 2.0. The appropriate factor must be determined experimentally for each soil. Although neither the direct determination of organic matter nor the calculation of organic matter content is completely accurate, the best procedures currently available are described in this chapter. Because of the problem associated with determining the organic matter content of a soil, it is strongly suggested that investigators *determine* and *report* the organic C concentration as a measure of the organic matter in a soil.

NINTH

The Nature and Properties of SOILS

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United States Agency for International Development

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5.10 Georgraphic Distribution of Clays

The clay of any particular soil is generally made up of a mixture of different colloidal minerals. In a given soil the mixture may vary from horizon to horizon. This occurs because the kind of clay that develops depends not only upon climatic influences and profile conditions but also upon the nature of the parent material. The situation may be further complicated by the presence in the parent material itself of clays that were formed under a preceding and perhaps an entirely different type of climatic regime. Nevertheless, some very general deductions seem possible, taking advantage of the relationships shown in Figure 5.12.

Regional Differences. The well-drained and well-weathered soils of humid and subhumid tropics tend to be dominated by the oxides of iron and aluminum. These clays are also prominent in the warmer humid regions of the temperate zone such as are found in the southeastern part of the United States. Kaolinite is commonly the dominant silicate mineral in these soils (Table 5.5) and is also found along with the hydrous oxide clays in more tropical areas.

As one might expect, the smectite, vermiculite, and illite (fine mica) groups are more prominent in cooler than in warm climates. Weathering is less intense there than in tropical and subtropical regions. In the northern part of the United States, in Canada, and in similar temperature regions throughout the world,

TABLE 5.5 Dominant Clay Minerals Found in Different Areas of the United States[a]

			Dominant clay minerals						
Soil order[b]	General weathering intensity	Typical location in U.S.	Hydrous oxides	Kaolinite	Montmorillonite	Illite	Vermiculite	Chlorite	Intergrades
Entisol	Low	Variable			x	x			
Inceptisol		Variable				x			x
Aridisols		Desert				x	x	x	x
Vertisols		Ala., Tex.			x				
Mollisols		Central U.S.			x	x	x	x	
Alfisols		Ohio, Pa., N.Y.		x	x	x		x	x
Spodosols		New England	x			x			x
Ultisols		Southeast U.S.	x	x			x		x
Oxisols	High	Tropical zones	x	x					x

[a] Adapted from Jackson (1955).

[b] See Chapter 13 for soil descriptions.

these clay minerals are common. The particular minerals that form depend largely on the parent materials and on the soil-water regime. Where either the parent material or the soil solution surrounding the weathering minerals is high in potassium, illite and related minerals are apt to be formed. Parent materials high in bases, particularly magnesium, or a soil drainage situation that discourages the leaching of these bases, encourages smectite formation. For these reasons, illite and montmorillonite are more likely to be prominent in soils of semiarid and arid regions than in the more humid areas.

The strong influence of parent material on the geographic distribution of clays can be seen in the "black belt" Vertisols of Alabama, Mississippi, and Texas. These soils, which are dark in color, have developed from base-rich marine parent materials and are dominated by montmorillonitic clays. They are surrounded by soils high in kaolinite and hydrous oxides that are more representative of this warm, humid region. Similar situations exist in central India and Sudan.

Data in Table 5.5 show the dominant clay minerals in different soil orders, the descriptions of which are given in Chapter 13. These data tend to substantiate the generalization just discussed. For example, Oxisols and Ultisols are characteristic of areas of intense weathering, and Aridisols are found in desert areas. The dominant clay minerals for these areas are as expected on the basis of Figure 5.12.

Although a few broad generalizations relating to the geographic distribution of clays are possible, these examples suggest that local parent materials and weathering conditions tend to dictate the kinds of clay minerals found in soils.

5.11 The Effect of pH on Surface Charges of Soil Colloids[5]

With the principles affecting the nature and formation of silicate clays in mind, we are now prepared to consider the practical implications of surface charges associated with not only silicate clays but with all soil colloids. As previous sections have suggested, both positive and negative charges are present and at least some of them are influenced by soil pH. These influences will now be discussed.

Negative Charges. There are two sources of negative charges on soil colloids. First are the *constant* (permanent) charges resulting from the isomorphous substitution with crystals of 2:1-type silicate clays of lower-valent cations for higher valent ones. This process accounts for nearly all of the high negativity associated with these 2:1-type clays.

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[5] For a discussion of the theory of surface charge see Uehara and Gillman (1980).

Family names in general identify groups of soil series similar in texture, mineral composition, and in soil temperature at a depth of 50 cm. Thus the name *Typic Argiaquoll, fine, mixed, mesic,* applies to a family in the *Typic Argiaquoll* subgroup with a fine texture, mixed clay mineral content, and mesic (8–15°C) soil temperature.

Soil series names have local significance since they normally identify the particular locale in which the soil is found. Thus, names such as Fort Collins, Cecil, Miami, Norfolk, and Ontario are used to identify the soil series. When the textural class name of the surface horizon is added to that of the series, a soil phase name has been identified. Fort Collins loam and Cecil clay are examples. Likewise, Cecil, eroded phase, is a Cecil series that has been eroded.

With this brief explanation of the nomenclature of the new system, the order category of the system will now be considered.

13.7 Soil Orders

Ten orders are recognized. With the exception of one order (Entisols), which roughly corresponds to the azonal soils of the 1949 system, the new orders bear little resemblance to those formerly used. The names of these orders and their major characteristics are shown in Table 13.3. Note that all order names have a common end, *sol* (from the Latin *solum,* soil).

Figure 13.8 is a general soil map showing the distribution of soil orders of the United States. Similarly, Figure 13.9 is a general soil map of the world. Profiles of each soil order are shown in color on Plate I (following page 430).

Entisols (little if any profile development). These are mineral soils without natural genetic horizons or with only the beginnings of such horizons. Some have an ochric epipedon and a few man-made anthropic or agric epipedons. The extremes of highly productive soils on recent alluvium and infertile soils on barren sands, as well as shallow soils on bedrock, are included. The common characteristic of all Entisols is lack of significant profile development.

Soils of this order are found under a wide variety of climatic conditions in the United States (Figure 13.8). For example, in the Rocky Mountain region and in southwest Texas, shallow, medium-textured Entisols (Orthents) over hard rock are common. They are used mostly as rangeland. Sandy Entisols (Psamments) are found in Florida, Alabama, and Georgia and typify the sand hill section of Nebraska. Psamments are used mostly for grazing in the drier climates. They may be forested or used for cropland in humid areas. Some of the citrus-, vegetable-, and peanut-producing areas of the South are typified by Psamments.

Entisols are probably found under even more widely varied climatic conditions outside the United States (Figure 13.9). Psamments are typical of the shifting sands of the Sahara Desert and Saudi Arabia. Large areas of Psamments dominate parts of southern Africa and of central and north central Australia.

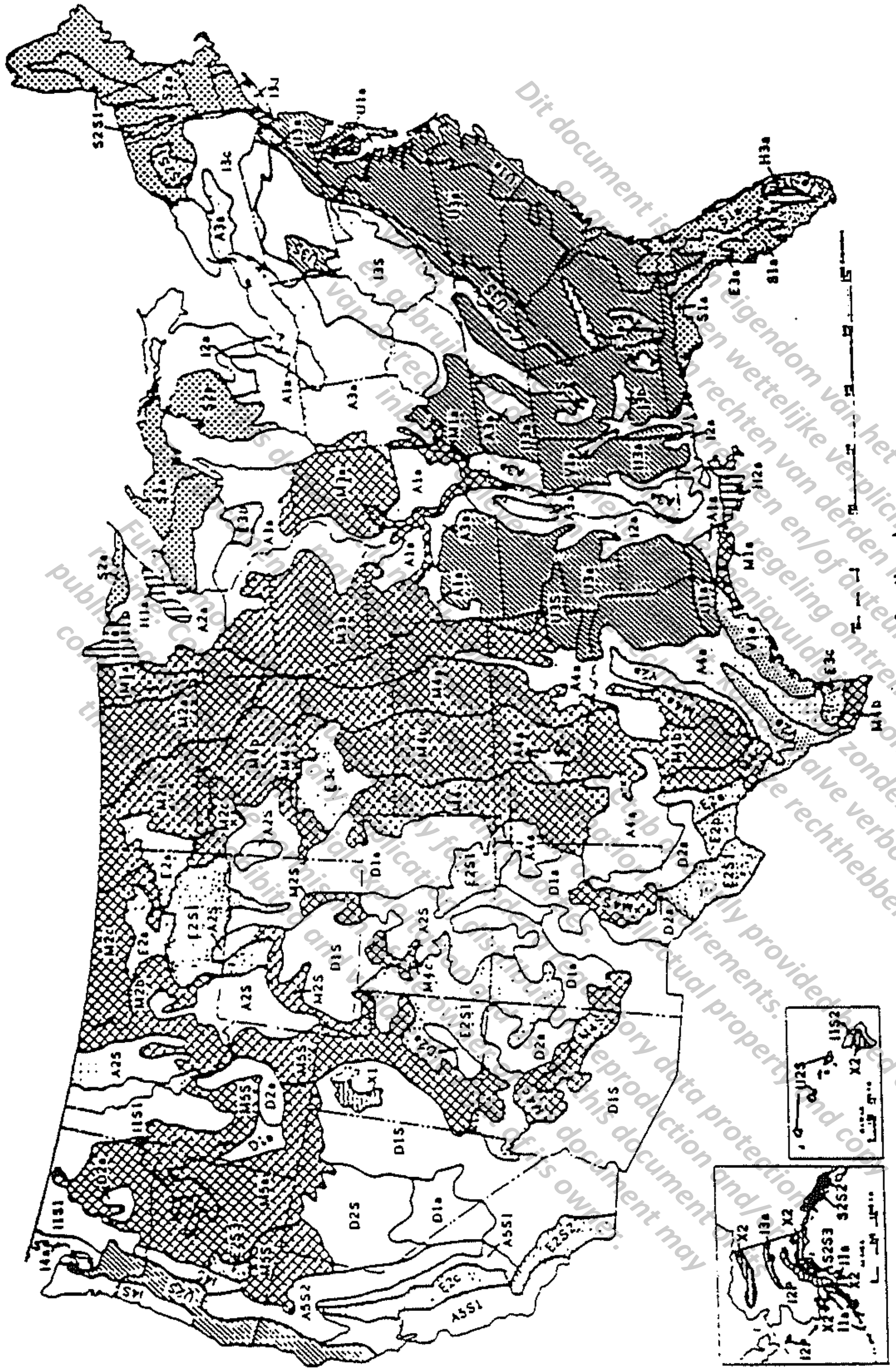

FIGURE 13.8 General soil map of the United States showing patterns of soil orders and suborders based on *Soil Taxonomy*. Explanations of symbols follow on pages 438–39. [*Courtesy USDA Soil Conservation Service, Soil Survey Staff.*]

Figure 13.8 (*Cont.*)

ALFISOLS

AQUALFS

A1a Aqualfs with Udalfs, Haplaquepts, Udolls; gently sloping.

BORALFS

A2a Boralfs with Udipsamments and Histosols; gently and moderately sloping.

A2a Cryoboralfs with Borolls, Cryochrepts, Cryorthods, and Rock outcrops; steep.

UDALFS

A3a Udalfs with Aqualfs, Aquolls, Rendolls, Udolls, and Udults; gently or moderately sloping.

USTALFS

A4a Ustalfs with Ustochrepts, Ustolls, Usterts, Ustipsamments, and Ustorthents; gently or moderately sloping.

XERALFS

A5S1 Xeralfs with Xerolls, Xerorthents, and Xererts; moderately sloping to steep.

A5S2 Ultic and lithic subgroups of Haploxeralfs with Andepts, Xerults, Xerolls, and Xerochrepts; steep.

ARIDISOLS

ARGIDS

D1a Argids with Orthids, Orthents, Psamments, and Ustolls; gently and moderately sloping.

D1S Argids with Orthids, gently sloping; and torriorthents, gently sloping to steep.

ORTHIDS

D2a Orthids with Argids, Orthents, and Xerolls; gently or moderately sloping.

D2S Orthids, gently sloping to steep, with Argids, gently sloping; lithic subgroups of Torriorthents and Xerorthents, both steep.

ENTISOLS

AQUENTS

E1a Aquents with Quartzipsamments, Aquepts, Aquolls, and Aquods; gently sloping.

ORTHENTS

E2a Torriorthents, steep, with borollic subgroups of Aridisols; Usterts and aridic and vertic subgroups of Borolls; gently or moderately sloping.

E2b Torriorthents with torrerts; gently or moderately sloping.

E2c Xerorthents with Xeralfs, Orthids, and Argids; gently sloping.

E2S1 Torriorthents; steep, and Argids, Torrifluvents, Ustolls, and Borolls; gently sloping.

E2S2 Xerorthents with Xeralfs and Xerolls; steep.

E2S3 Cryorthents with Cryopsamments and Cryandepts; gently sloping to steep.

PSAMMENTS

E3a Quartzipsamments with Aquults and Udults; gently or moderately sloping.

E3b Udipsamments with Aquolls and Udalfs; gently or moderately sloping.

E3c Ustipsamments with Ustalfs and Aquolls; gently or moderately sloping.

HISTOSOLS

HISTOSOLS

H1a Hemists with Psammaquents and Udipsamments; gently sloping.

H2a Hemists and Saprists with Fluvaquents and Haplaquepts; gently sloping.

H3a Fibrists, Hemists, and Saprists with Psammaquents; gently sloping.

INCEPTISOLS

ANDEPTS

I1a Cryandepts with Cryaquepts, Histosols, and Rock land; gently or moderately sloping.

I1S1 Cryandepts with Cryochrepts, Cryumbrepts, and Cryorthods; steep.

I1S2 Andepts with Tropepts, Ustolls, and Tropofolists; moderately sloping to steep.

AQUEPTS

I2a Haplaquepts with Aqualfs, Aquolls, Udalfs, and Fluvaquents; gently sloping.

I2P Cryaquepts with cryic great groups of Orthents, Histosols, and Ochrepts; gently sloping to steep.

I3a Cryochrepts with cryic great groups of Aquepts, Histosols, and Orthods; gently or moderately sloping.

I3b Eutrochrepts with Uderts; gently sloping.

I3c Fragiochrepts with Fragiaquepts, gently or moderately sloping; and Dystrochrepts, steep.

I3d Dystrochrepts with Udipsamments and Haplorthods; gently sloping.

I3S Dystrochrepts, steep, with Udalfs and Udults; gently or moderately sloping.

UMBREPTS

I4a Haplumbrepts with Aquepts and Orthods; gently or moderately sloping.

I4S Haplumbrepts and Orthods; steep, with Xerolls and Andepts; gently sloping.

MOLLISOLS

AQUOLLS

M1a Aquolls with Udalfs, Fluvents, Udipsamments, Ustipsamments, Aquepts, Eutrochrepts, and Borolls; gently sloping.

BOROLLS

M2a Udic subgroups of Borolls with Aquolls and Ustorthents; gently sloping.

M2b Typic subgroups of Borolls with Ustipsamments, Ustorthents, and Boralfs; gently sloping.

M2c Aridic subgroups of Borolls with Borollic subgroups of Argids and Orthids, and Torriorthents; gently sloping.

M2S Borolls with Boralfs, Argids, Torriorthents, and Ustolls; moderately sloping or steep.

UDOLLS

M3a Udolls, with Aquolls, Udalfs, Aqualfs, Fluvents, Psamments, Ustorthents, Aquepts, and Albolls; gently or moderately sloping.

USTOLLS

M4a Udic subgroups of Ustolls with Orthents, Ustochrepts, Usterts, Aquents, Fluvents, and Udolls; gently or moderately sloping.

M4b Typic subgroups of Ustolls with Ustalfs, Ustipsamments, Ustorthents, Ustochrepts, Aquolls, and Usterts; gently or moderately sloping.

M4c Aridic subgroups of Ustolls with Ustalfs, Orthids, Ustipsamments, Ustorthents, Ustochrepts, Torriorthents, Borolls, Ustolls, and Usterts, gently or moderately sloping.

M4S Ustolls with Argids and Torriorthents; moderately sloping or steep.

XEROLLS

M5a Xerolls with Argids, Orthids, Fluvents, Cryoboralfs, Cryoborolls, and Xerorthents; gently or moderately sloping.

M5S Xerolls with Cryoboralfs, Xeralfs, Xerorthents, and Xererts; moderately sloping or steep.

SPODOSOLS

AQUODS

S1a Aquods with Psammaquents, Aquolls, Humods, and Aquults; gently sloping.

ORTHODS

S2a Orthods with Boralfs, Aquents, Orthents, Psamments, Histosols, Aquepts, Fragiochrepts, and Dystrochrepts; gently or moderately sloping.

S2S1 Orthods with Histosols, Aquents, and Aquepts; moderately sloping or steep.

S2S2 Cryorthods with Histosols; moderately sloping or steep.

S2S3 Cryorthods with Histosols, Andepts and Aquepts; gently sloping to steep.

ULTISOLS

AQUULTS

U1a Aquults with Aquents, Histosols, Quartzipsamments, and Udults; gently sloping.

HUMULTS

U2S Humults with Andepts, Tropepts, Xerolls, Ustolls, Orthox, Torrox, and Rock land; gently sloping to steep.

UDULTS

U3a Udults with Udalfs, Fluvents, Aquents, Quartzipsamments, Aquepts, Dystrochrepts, and Aquults; gently or moderately sloping.

U3S Udults with Dystrochrepts; moderately sloping or steep.

VERTISOLS

UDERTS

V1a Uderts with Aqualfs, Eutrochrepts, Aquolls, and Ustolls, gently sloping.

USTERTS

V2a Usterts with Aqualfs, Orthids, Udifluvents, Aquolls, Ustolls, and Torrerts; gently sloping.

Areas with Little Soil

X1 Salt flats.

X2 Rock land (plus permanent snow fields and glaciers).

Slope Classes

Gently sloping—Slopes mainly less than 10%, including nearly level.

Moderately sloping—Slopes mainly between 10 and 25%.

Steep—Slopes mainly steeper than 25%.