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Environmental Fate and Effects Department
Ciba Crop Protection
Ciba-Geigy Corporation
Greensboro, North Carolina

PHOTODEGRADATION OF ^{14}C-CGA-329351 ON SOIL UNDER ARTIFICIAL LIGHT

Environmental Fate Data Requirement
40 CFR 158
Subdivision N: Series 161-3

Report No.: ABR-95094

Ciba-Geigy Study: 151-95

Study Director: [redacted]

Title: Chemist III

Date: 12/8/95

Approved By: [redacted]

Title: Manager, Environmental Metabolism

Date: 12/8/95

Submitted by: [redacted]

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Ciba Crop Protection Division
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Study Initiation Date: March 9, 1995

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CERTIFICATE OF GOOD LABORATORY PRACTICES

This study (Ciba-Geigy study number 151-95) has been performed in compliance with EPA Good Laboratory Practice Standards (40 CFR Part 160) with one exception. Agvise laboratories sent a soil sample to North Dakota State University for x-ray diffraction analysis for the clay type determination. The university is not a GLP facility.

5.1.2.e Woo ____________ 12/8/95
Study Director Date

5.1.2.e Woo ____________ 12/8/95
Representative of Submitter/Sponsor Date

Submitter/Sponsor:

Ciba-Geigy Corporation
Ciba Crop Protection
Environmental Fate and Effects Department
410 Swing Road
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Ciba-Geigy Corporation
Ciba Crop Protection
Quality Assurance Unit

QUALITY ASSURANCE STATEMENT

Report Title: PHOTODEGRADATION OF ^{14}C-CGA-329351 ON SOIL UNDER ARTIFICIAL LIGHT

Study Director: [redacted] Ciba

Ciba Study No.: Protocol 151-95 with amendment(s)

Ciba Final Report No.: ABR-95094

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 DATES
Protocol Audit	3/9/95	3/9/95
In-Progress Inspections	5/4/95 10/3/95	5/4/95 10/3/95
Final Report Audit (ABR-95094)	10/2-6, 9-12/95	10/24/95

Prepared by: [redacted] Auditor

Date: 10/24/95

GENERAL INFORMATION

Ciba-Geigy Study Participants: [redacted]

Study Director: [redacted]

Test Substance:
Experiment 1
Ciba-Geigy Designation:
^{14}C-CGA-329351
Reference No.: MSR-II-90,
Specific Activity: 81.1 µCi/mg
Radiochemical Purity: 99.1% on 3/9/95
Chemical Purity: 99.9% on 3/9/95

Experiment 2
^{14}C-CGA-48988
Reference No.: WFH-IX-65,
Specific Activity: 73.2 µCi/mg
Radiochemical Purity: 98.4% on 3/21/95
Chemical Purity: 99.2% on 3/22/95

Testing Facility:

<u>Biological/Analytical Phase:</u>
Ciba-Geigy Corporation
Ciba Crop Protection
Environmental Fate and Effects Department
410 Swing Road
Post Office Box 18300
Greensboro, NC 27419
Kay Sparrow
(910) 632-7390

<u>Soil Characterization Phase:</u>
Agvise Laboratories
Post Office Box 510, Highway 15
Northwood, ND 58267-0510
Robert Deutsch
(701) 587-6010

Protocol Number: Protocol 151-95 and Amendment 1

Archives: The protocol, protocol amendments, raw data for method development, raw data for this report (ABR-95094), and this report (ABR-95094) will be archived at Ciba-Geigy Corporation, Ciba Crop Protection, Agricultural Group Archives, Greensboro, North Carolina. All extracts, residues and analysis subfractions will be retained as long as they warrant further analysis.

All non-study specific records, such as balance, sterilizer units, Fugix Imager, HPLC and LSC logbooks will be archived in the Agricultural Group Archives when the logbook is complete. A logbook accountability system is instituted in the Biochemistry Group and provides an audit trail of such records.

I. ABSTRACT

A soil photolysis study was conducted with radiolabeled CGA-329351 to satisfy the U.S. EPA Environmental Fate Data Requirement 40 CFR Section 158 (Subdivision N, Series 161-3). Two experiments were conducted simultaneously using ^{14}C-CGA-329351, the D isomer, (Experiment 1) and ^{14}C-CGA-48988, a racemic mixture of the D and L isomers, (Experiment 2). Figures 1 and 2 contain the structures for both compounds. In both experiments, the irradiated samples were irradiated with a xenon arc lamp for 12 hours per day. Both irradiated and non-irradiated (dark control) samples were collected for up to 30 days. After the volatiles were collected from these samples, two types of extractions were performed and subsequent fractions were analyzed. The second extraction usually released more of the same components seen in the first extraction, therefore the percent of total dose values quoted in this report for parent and each degradate generally reflect the combined values of Extractions 1 and 2. The results from Experiment 1 (CGA-329351) and Experiment 2 (CGA-48988), both irradiated and non-irradiated samples were qualitatively equivalent. There were a total of 8 degradates in Experiment 1 and 9 degradates in Experiment 2. Each of these degradates accounted for less than 3.90% of the total dose for the combined Extractions 1 and 2. In each case, parent appears to degrade either to CGA-62826 (Component F) or CGA-37734 (Component C) and then to CGA-42447 (Component B) (Figure 3). The half life value for CGA-329351, irradiated samples, was 248 days as compared to 303 days for CGA-48988. The half life value for CGA-329351, non-irradiated samples, was 358 days as compared to 755 days for CGA-48988. Volatiles accounted for up to 1.16% of the total dose for Experiments 1 and 2. The primary degradates identified for the irradiated and non-irradiated samples were CGA-42447 (maximum of 1.20% of total dose), CGA-37734 (maximum of 0.83% of total dose) and CGA-62826 (maximum of 3.90% of total dose) based on the combined total dose values of Extractions 1 and 2. Evidence from this study indicate that CGA-329351 and CGA-48988 on soil do not significantly degrade under photolytic conditions.

II. INTRODUCTION

There were two experiments conducted to study the photolytic effects of ^{14}C-CGA-329351 and ^{14}C-CGA-48988 on soil. Each experiment involved a topical treatment of ^{14}C-CGA-329351 or

^{14}C-CGA-48988 to individual soil samples. After dosing, the individual soil samples were mixed well to yield a homogeneous mixture. The results for Experiment 1 (CGA-329351) and Experiment 2 (CGA-48988) are reported in this document. In each case, the soil was incubated prior to dosing to insure the proper moisture content as well as microbial activity prior to dosing. The soils were dosed, and respective samples were either exposed to an artificial light source or were maintained as non-irradiated samples (dark controls) in a constant temperature room.

III. MATERIALS

Chemicals and materials utilized in this study were as follows:

1. SOLVENTS AND SUPPLIES

- Acetonitrile, chloroform, isopropanol, methanol, methylene chloride, toluene, all HPLC grade, (Fisher Scientific)
- Acrodisc, 0.45 micron, (Gelman Sciences)
- Actinomycete Agar, (Difco Bacto)
- Ammonium Hydroxide, Reagent Grade, (Fisher Scientific)
- Calcium Chloride, Certified, Anhydrous, (Fisher Scientific)
- Centrifuge, Optima L-60 Series Ultracentrifuge (Beckman)
- Cycłohexamide, (Sigma)
- Dichlorodimethylsilane, 99% [75-78-5] (Aldrich Chemical)
- Ethanol, 200 Proof, (Aaper)
- Formic Acid, 90% purified, (Fisher Scientific)
- Glucose (D-glucose, Dextrose), certified ACS, anhydrous, (Fisher Scientific)
- Glycerol (Difco Laboratories)
- Nalgene® Filterware Unit, 0.2 micron, (Nalgene)
- Nitrogen Gas, Departmental Supply, (Air Products, Inc.)
- Nystatin, (Sigma)
- Oxosol, (National Diagnostics)
- Plastic sterile disposable petri dishes, 100 x 15 mm (Fisher Scientific)
- Plate Count Agar, (Difco Bacto)
- Potassium Hydroxide, Certified ACS grade, pellets, (Fisher Scientific)
- Ready Safe and Ready Gel Liquid Scintillation Cocktails, (Beckman)

- Rose Bengal Agar, (Difco Bacto)
- Stainless Steel Canulas, 25 gauge, (Popper and Son)
- Sterile Needles, 18 or 25 gauge needles x 1.5 inches, (Becton Dickinson & Co.)
- Sterile Syringes, 1cc U-100 Insulin syringes, (Becton Dickinson & Co.)
- Sterile Pipettes, (Fisher)
- Syringes, 10 µl - 1.0 mL, Hamilton or Unimetrics
- Supplement C, Chloramphenicol, (Difco Bacto)
- Talcum, purified grade, Fisher
- Water, Reverse Osmosis, (Departmental Supply)
- Silica Gel TLC Plates, 0.25 mm Silica Gel, 60F-254, 20 x 20 cm, (Merck)
- Trifluoroacetic Acid, certified, (Fisher)
- Vials, type 1 borosilicate, 2.5 x 2.5 x 5.5 cm, (Ace Glass)

2. REFERENCE STANDARDS

The following reference standard was received from Production Technical Analytical Services, Ciba Geigy Corporation, Ciba Crop Protection, Greensboro, NC:

CGA-48988: Lot No. S87-1208, purity 95.8%

The following reference standards were received from Chemical Synthesis, Ciba-Geigy Corporation, Ciba Crop Protection, Greensboro, NC:

CGA-48988: Lot No. BPM-XIII-58, purity 98.0% on 1/26/94

CGA-42447: Lot No. MCO-I-2, purity 99.7% on 12/12/94

CGA-37734: Lot No. BPM-I-8, purity 96.9% on 2/2/94

CGA-62826: Lot No. BPM-I-4B, purity >99.9% on 12/8/94

CGA-119857: Lot No. GB-XLV-3, purity >99.9% on 12/8/94

Figure 3 contains the structures for all the reference standards listed above. The stability of the reference standards and solutions of reference standards were verified by consistent TLC Rf values throughout the study. Since CGA-48988 is a racemic mixture of D and L isomers and CGA-329351 contains only the D isomer, reference standard

CGA-48988 was used to cochromatograph with both CGA-48988 and CGA-329351.

3. TEST SUBSTANCE

Ciba-Geigy Designation:

^{14}C- CGA-329351
Reference No.: MSR-II-90,
Specific Activity: 81.1 µCi/mg
Radiochemical Purity: 99.1% on 3/9/95
Chemical Purity: 99.9% on 3/9/95

^{14}C-CGA-48988
Reference No.: WFH-IX-65,
Specific Activity: 73.2 µCi/mg
Radiochemical Purity: 98.4% on 3/21/95
Chemical Purity: 99.2% on 3/22/95

IV. METHODS

1. RADIOACTIVITY ASSESSMENT (LSC ANALYSIS)

Each sample was assayed in a Beckman Liquid Scintillation Counter (model 3801, 6000 or 6500). Most radioassays used a 5 minute dpm program. A 2 minute dpm program was used for some samples when multiple assays of the same sample were being analyzed. Most samples counted on the 6000 or 6500 used the 2 minute program that subtracted the background and calculated an average. Counting efficiencies were determined by external standardization. Limits of quantitation and detection were established by statistical methods of radioactivity counting. An equivalent volume of the appropriate cocktail was used as a background for all radioassays. Beckman Ready Gel (10 mL) scintillation cocktail along with an aliquot of silica gel was used for TLC quantitation. National Diagnostic Oxosol (15 mL) was used for combustion samples. All other radioassays used either 5 mL of Beckman Ready Safe or 10 mL of Ready Gel scintillation cocktail.

2. SOIL SHIPPING AND HANDLING

The soil was shipped by Federal Express from Ciba, Sanger, California to Ciba, Greensboro. The soil was received at

the Ciba Greensboro site on March 20, 1995. The soil was allowed to sit at room temperature overnight. The following day, the soil was sieved through a 2 mm USA Standard Testing Sieve. A subsample was shipped from Ciba, Greensboro to Agvise Laboratory for soil characterization and analysis. The remaining soil was placed in a plastic bag with small slits to allow for air circulation. The soil was stored in the constant temperature room (Environmental Specialties, Inc. 9-19 TC/GC Chamber) at 25±1°C. Section 7 contains details regarding the soil moisture and acclimation.

3. WEIGHT MEASUREMENTS

All weight measurements were made on either Mettler or Ohaus balances. The calibration of each balance was checked for accuracy by weighing a class "S" weight of a size similar to that of the sample to be weighed. Each balance deviated less than 1% from the calibration weight each time it was used.

At the time of dosing a Mettler BB240 balance was used which reports values to three decimal points. Most other weights were measured using a Mettler PM2000 balance which reports values to two decimal points. The weights recorded in the raw data and in Tables IV and V reflect the exact weight reported from the respective balance.

4. SILYLATION OF GLASSWARE

A 5% v/v solution of dichlorodimethylsilane (99%) in methylene choride was prepared. The glassware was allowed to soak in the silylation solution at least 15 minutes then rinsed with methanol followed by methylene chloride. The glassware was air dried completely then baked at 110°C for at least 2 hours, then rinsed with water.

5. STERILIZATION USING THE AMSCO EAGLE STERILIZER

An AMSCO EAGLE Series 2021 sterilizer was used to sterilize the agar media and the glassware used for dosing and the incubation of samples. The agar media was sterilized at 121°C for 15 minutes using a liquid cycle. The glassware and utensils were wrapped in aluminum foil and sterilized at 121°C for 30 minutes using a gravity cycle. The original paper printouts of the sterilization cycle, times, and

temperatures were retained in the study records. In a few cases, the printer on the sterilization unit jammed and a paper printout was not available.

6. PREPARATION OF AGAR PLATES

There were three types of agar prepared for the soil viability analysis. Rose bengal, actinomycete, and plate count agars were prepared with inhibitors for the analysis of fungi, actinomycete and total microbial populations respectively. In each case, the agar media was slowly dissolved in 2 L of almost boiling water. Each agar media was heated using a Nuova Thermolyne heat/stir plate until the solution was translucent in color. The agar was sterilized at 121°C for 15 minutes using a liquid cycle. The agar media was cooled approximately 15 minutes on a sterile Purified Clean Bench (Labconco). Any inhibitors were added while stirring the partially cooled agar. The agar media was poured into disposable petri dishes and allowed to cool further before replacing the lids. The petri dishes were placed in plastic sleeves and stored in a refrigerator. All procedures were conducted in a manner to insure aseptic conditions.

Rose Bengal Agar Preparation

Approximately 64 g of Difco Bacto Rose Bengal Agar were dissolved in water. Two milliliters of 200 proof ethanol were added to each of four bottles of Difco supplement C (0.05 g of chloramphenicol) and the contents were stirred to dissolve the supplement C. The supplement C solution, an inhibitor for bacteria, was added to the agar after it had cooled approximately 15 minutes.

Actinomycete Agar Preparation

Approximately 44 g of Difco Bacto Actinomycete Isolation Agar and 10 g of Difco Bacto Glycerol were dissolved in boiling water. Nystatin (0.10 g) and cyclohexamide (0.10 g) were weighed into a tared scintillation vial with 500 µl of methanol. The nystatin and cyclohexamide, fungi and yeast inhibitors, were added when the agar had cooled approximately 15 minutes.

Plate Count Agar Preparation

Difco plate count agar (47 g) were dissolved in 2 L of water.

7. SOIL MOISTURE ANALYSIS AND ACCLIMATION

The soil was sieved through a 2.0 mm USA Standard Testing Sieve #10 (Fisher Scientific). The moisture of the soil was measured using a Computrac Max 50 Moisture Analyzer. Three aliquots of soil, 4-5 g each, were measured. The average percent moisture was 9.06% on March 21, 1995. The average percent moisture was 6.83% when analyzed again on April 3, 1995 prior to dosing for Experiments 1 and 2. The Agvise soil characterization report[2] stated that the percent moisture at 1/3 bar was 8.3%, therefore 75% of FMC at 1/3 bar ± 12% equals 6.23%± 0.75%. Therefore the allowable moisture level ranged from 5.48 - 6.98%. The moisture was within the allowable range and therefore was not adjusted for this study.

On March 21, 1995 the soil had been transferred for acclimation to a 12 x 14 inch plastic bag and stapled closed. The soil was mixed in the plastic bag to insure a homogeneous mixture. The plastic bag was placed on its side in a foil lined metal tray and spread out to approximately a 1-2 inch soil depth. Several slits were cut in the top of the bag to allow for air circulation. The metal tray containing the soil was covered with foil and placed in the constant temperature room at 25±1°C for acclimation until the time of dosing.

8. BIOMASS ANALYSIS

GLUCOSE PREPARATION

Various amounts of glucose (0.2g, 0.5g, 1g and 2g) were weighed onto weighing paper then transferred to a mortar. A corresponding weight of talcum was weighed onto weighing paper to give a total weight of glucose plus talcum of 10.0g. The mixture was ground to insure a homogenous mixture. The mixture was transferred to a scintillation vial until needed for biomass measurement. One scintillation vial was filled with ground talcum only to serve as a background.

BIOMASS DETERMINATION

Approximately 25g of moist soil was added to each of 5 soil flasks. One gram of one of the five glucose amendment levels were added to each respective soil sample. The soil samples were immediately placed in a water bath at 21°C and connected to a Micro-Oxymax Biomass Analyzer. The CO_2 efflux from the soil microbes was monitored for approximately 20 hours. The graph of the maximal respiratory response indicated that the 0.10g glucose preparation produced the maximal initial respiration at 2 hours. A second biomass experiment was conducted on triplicate aliquots of soil, all amended with the 0.10g glucose preparation. The soil samples were connected to the Micro-Oxymax Biomass Analyzer and the CO_2 efflux was monitored for 44 hours. The maximal initial respiration occurred at the 3 hour interval. The corresponding CO_2 rate was used to calculate the biomass. The biomass measurement and calculation were done according to Anderson and Domsch[3].

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. Replicate 1: if the mL CO_2/hr = 0.0412 for 22.76 g dry weight òf soil

$$X = \left[\frac{(40.04)(0.0412\ \text{mL}\ CO_2\ /\ \text{hr}) + 0.37}{22.76\ \text{g soil}} \right] X\ 1000\ \frac{g}{kg}$$

X = 88.737 mg C/kg soil

9. LIGHT MEASUREMENTS

The total natural sunlight intensity measured hourly from 9 am-5 pm on July 11, 1991. The total intensity ranged from 150-543 W/m^2 with an average of 410 W/m^2. Measurements were made using a Heraeus Radialux equipped with a Radialux Global Sensor. On July 28,1992 at 1:14 PM, Eastern Daylight Savings Time, a natural sunlight spectral distribution was measured from 200-700 nm using an International Light IL1700, International Light IL760, and a Kratos double monochromator. All natural sunlight measurements were made

at Ciba, Greensboro, NC at 36°5.86'N latitude and 79°56.24'W longitude.

The Heraeus Radialux was used to set the intensity of the Suntest CPS Unit 3 and Unit 4 to an intensity of approximately 410 W/m^2 for the initiation of the study. On March 29,1995 an artificial light spectral distribution was measured for Suntest CPS+ Unit 3 and Suntest CPS+ Unit 4. A final intensity and spectral distribution were measured on April 18, 1995 for Suntest Unit 3 and May 5, 1995 for Suntest Unit 4.

10. TEMPERATURE MEASUREMENTS

Both irradiated and non-irradiated sample temperatures were monitored by the Environmental Monitoring System (EMS) composed of an Omega thermocouple, Omega data logger and computer support. In addition, the temperature and humidity were measured for the non-irradiated samples, using a Honeywell hydrothermograph.

11. PREPARATION OF TEST SUBSTANCES

The ^{14}C-CGA-329351 test substance and ^{14}C-CGA-48988 each were dissolved in 10 mL of acetonitrile. Each test substance was vortexed (Vortex Genie) and sonicated (Branson 2200 sonicator) for approximately 3 minutes to insure the test substance was properly dissolved.

12. PURITY OF TEST SUBSTANCE

An aliquot (20 µl) of each test substance was added to 1 mL of HPLC grade acetonitrile in a 7 mL scintillation vial. Each of the diluted test substance solutions were vortexed, sonicated, and radioassayed. An aliquot of the diluted test substances were applied to TLC plates and developed in two dimensions for TLC analysis (Method Section 26). The plates were scraped and quantitated to compare to the established radiochemical purity. In each case the radiochemical purity compared favorably with the purity established by the Chemical Synthesis Group of 99.1% on 3/9/95 for ^{14}C-CGA-329351 and 98.4% on 3/21/95 for ^{14}C-CGA-48988.

13. APPLICATION OF TEST SUBSTANCE

The soil was removed from the constant temperature room on April 4, 1995.

The lab bench was lined with aluminum foil and sprayed with a 70:30 ethanol:water solution to sterilize the area. All sample vials, glassware and any utensils needed for dosing were sterilized using an AMSCO Eagle Sterilizer (Method Section 5). Each sample vial was rectangular in shape (approximately 2.5 x 2.5 x 5.5 cm) and made of type 1 borosilicate glass with an open top cap and Teflon coated septum. Each sample vial was weighed and approximately 7 g of previously sieved (2.0 mm sieve) soil was added to each vial. Seven grams of soil was chosen to provide a 2-3 mm soil depth when the vial was laid on its side and to allow for maximum irradiation. When all vials were filled with soil, the vials were laid on their side. The vials were shaken slightly to produce an even layer of soil. The dose solution was radioassayed to determine the exact volume of dose solution needed to dose at 1.5 ppm.

Calculation:

^{14}C-CGA-329351
1.5 µg/g x 7g x 2.22x10^{6}dpm/µCi x 81.1 µCi/mg x mg/1000µg = 1,890,441 dpm needed to dose 7g of soil

^{14}C-CGA-48988
1.5 µg/g x 7g x 2.22x10^{6}dpm/µCi x 73.2 µCi/mg x mg/1000µg = 1,706,292 dpm needed to dose 7g of soil

Either ^{14}C-CGA-329351 or ^{14}C-CGA-48988 dose solution (71 µl = 1,890,441 dpm for ^{14}C-CGA-329351 or 67µl = 1,706,292 dpm for ^{14}C-CGA-48988) was dripped in a random fashion over the soil surface. Each sample vial was vortexed approximately 30 seconds, then lightly pressed with a spatula to form an even layer approximately 2 mm thick (Figure 6). The sample vials were tightly capped and the cap wrapped with parafilm. All sample vials were labeled for the incubation phase. The weight of the vial, and vial containing the moist soil were recorded to calculate any moisture lost during incubation and to calculate the dpm per gram of applied radiolabel. The samples were then placed in the appropriate incubation chamber.

PPM Determination for Soil Samples

Three aliquots of each dose solution (one at the beginning, middle and end of dosing) were analyzed by one dimensional TLC to verify if any degradation had occurred during the dosing procedure. Three aliquots of each dose solution (one at the beginning, middle and end of dosing) were radioassayed by LSC analysis using Talisman version 1.0 computer software. A known dpm of the dose solution was added to each soil sample. Based on the weight of the soil per vial and the known dpm applied, a ppm value was calculated for each sample.

LSC Counter Data for the Dose Solutions:

LSC COUNTER DATA FOR CGA-329351

SAMPLE	COUNTER DPM-BKG	ALIQUOT AMOUNT (mL)	SAMPLE DPM/mL	AVG. DPM
BKG	NA	NA		
329DOSE2	1904425	0.071	26822886	
329DOSE2	1934895	0.071	27252041	
329DOSE2	1929067	0.071	27169957	
				1,922,796

* = CALCULATED VALUES
NA = NOT APPLICABLE

Seventy one microliters (1,922,796 dpm)of the dose solution were applied to each sample vial for the CGA-329351 (Experiment 1).

LSC COUNTER DATA FOR CGA-48988

SAMPLE	COUNTER DPM-BKG	ALIQUOT AMOUNT (mL)	SAMPLE DPM/mL	AVG. DPM
BKG	NA	NA		
489DOSE2	1715882	0.067	25610178	
489DOSE2	1737196	0.067	25928298	
489DOSE2	1738873	0.067	25953328	
				1,730,650

* = CALCULATED VALUES
NA = NOT APPLICABLE

Sixty seven microliters (1,730,650 dpm) of the dose solution were applied to each sample vial for the CGA-48988 (Experiment 2).

Calculation of the PPM Dose per Vial (Table IV and V):

1. $\frac{\text{DPM Applied to Vial}}{\text{g of Soil per Vial}} = \text{DPM}/\text{g}$ of Soil per Sample

2. $\text{Avg DPM}/\text{g} \times \mu\text{Ci}/2.22 \times 10^{6}\,\text{dpm} \times \text{mg}/\mu\text{Ci} \times 1000\ \mu\text{g}/\text{mg} = \text{ppm or } \mu\text{g}/\text{g}$

ex.
DAY 0 R1 IRRADIATED, CGA-329351, EXPERIMENT 1

1. $\frac{1{,}922{,}796}{6.976\ \text{g}} = 275{,}630\ \text{DPM}/\text{g}$ of Soil per Sample

2. $275{,}630\ \text{DPM}/\text{g} \times \mu\text{Ci}/2.22 \times 10^{6}\,\text{dpm} \times \text{mg}/81.1\ \mu\text{Ci} \times 1000\ \mu\text{g}/\text{mg}$
$= 1.53\ \text{ppm or}\ \mu\text{g}/\text{g}$

The average ppm dose rate for 1.52 ppm for CGA-329351 and 1.51 ppm for CGA-48988.

14. NON-IRRADIATED INCUBATION

The dosed sample vials were tightly capped with an open top cap and a Teflon coated septum. The sample vials were labeled with the protocol number and a unique sample number during the incubation phase (Table 1). Additional label information was added at the time of harvest. These non-irradiated samples were wrapped in aluminum foil and placed in a aluminum foil lined box to insure no light reached the samples. The non-irradiated samples were incubated up to 30 days in the constant temperature room (Environmental Specialties, Inc. 9-19 TC/GC Chamber). The temperature was monitored by the Environmental Monitoring System (EMS). The temperature was maintained at 25±1°C. Figure 9 includes a copy of the EMS temperature plots.

15. IRRADIATED INCUBATION

The dosed sample vials were tightly capped with an open top cap and a Teflon coated septum. The vials were labeled with the protocol number and a unique sample number during the photolytic process (Table 1). Additional label information was added at the time of harvest. The lid of each vial was wrapped with parafilm and the vial placed on its side in a water bath to allow for maximum exposure to the artificial light (Figure 6). The water bath was designed to allow a maximum water flow to insure a minimum temperature fluctuation. The water bath was fed by two recirculating pumps in order to maintain the temperature at 25±1°C. One pump operated at a lower temperature (~ 20°C) during the light phase each day. Another pump operated at a higher temperature (~28°C) during the dark phase each day. The pumps were connected to a timing device. When the light came on each day, the timing device shut off the higher temperature pump and immediately began pumping water at the lower temperature. The temperature of the water bath was monitored by the Environmental Monitoring System (EMS). The EMS System consisted of an Omega thermocouple, an Omega data logger and computer support. The Omega thermocouple was inserted into a surrogate vial filled with 7 g of soil prior to dosing. The temperature was maintained at 25±1°C. Figure 9 includes a copy of the EMS temperature plots. The samples were covered with a Pyrex plate and placed directly under the Suntest Photolysis Unit (Suntest Accelerated Exposure Unit, W.C. Heraeus, Hanau, Germany). The light source was a xenon arc lamp (1.8 kW) equipped with a quartz glass dish with a selective reflecting coating and a UV glass filter (Figure 7). These filters and the Pyrex plate absorb wavelengths below 290 nm to simulate natural sunlight. The samples were irradiated for 12 hours per day, for up to 30 days. A diagram of the complete photolysis test system is displayed in Figure 8.

16. VIABILITY ANALYSIS

Viability analyses were performed 8 days prior to dosing and on Day 30. Day 30 analysis was conducted on a surrogate irradiated and non-irradiated sample for ^{14}C-CGA-48988.

Calcium chloride (5.5 g) were dissolved in 1 L of water. The calcium chloride solution was filtered, just prior to use, through a Nalgene Filterware Unit equipped with a 0.2 µm filter to cold sterilize the solution.

All glassware and utensils needed for the viability analyses was sterilized prior to use. The samples to be used for viability analyses were transferred to the sterile bench (Labconco, Purified Clean Bench). The sterile bench and the outside of the sample vials were previously rinsed with a 70:30 v/v ethanol:water solution. A known weight of soil from each sample was transferred to a 15 or 40 mL centrifuge tube. A sterile pipette was used to add 10 mL of a 0.05 M sterile calcium chloride solution to the centrifuge tube.

Each soil sample was extracted with calcium chloride by shaking vigorously for approximately 3 minutes, then centrifuging for 5 minutes. The supernatant was transferred to a sterile 15 mL centrifuge tube using a sterile B-D 1 cc, U-100 Insulin syringe. A series of dilutions were performed to give: A)10, B) 100, C) 1,000, D) 5,000, and E) 10,000 fold dilution factor. Approximately 100 µl of selected dilutions were applied to each of the three types of agar plates. Each assay was conducted in triplicate (replicates 1, 2 and 3). For example, dilutions A, B and C were streaked in triplicate on rose bengal agar plates. A sterile glass stir rod was used to streak the sample onto the plate. The plates were labeled with the time point, either replicate 1, 2 or 3, the type of agar plate and the dilution factor. The plates were stored in a constant temperature room (Environmental Specialties, Inc. 9-19 TC/GC Chamber) for incubation at 25±1°C. The temperature and humidity were monitored with a Honeywell hydrothermograph and the temperature was also monitored by the Environmental monitoring System (EMS). The plates were monitored for up to 14 days. Ideally, a set of dilution plates yielding a microbial population of approximately 30-100 were counted for each type of agar plate. For example, the three replicates of dilution C plates were counted for the plate count agar plates for the pre-dose soil. The total microbial population was then calculated based on the amount of soil extracted and the dilution factor for the plates counted. The results indicate that the soil samples remained viable throughout the study.

Calculation:

Plate Count for 3/27/95

A. $$\text{g of soil} \times \frac{\%\ \text{solid}}{100} = \text{g of dry soil}$$

$$4.38\ \text{g soil} \times \frac{90.94\ \%\ \text{solid}}{100} = 3.98\ \text{g of dry soil}$$

B. $\frac{\text{g of dry soil}}{\text{ml } CaCl_2}$ X aliquot (ml) applied to each plate

= g of soil extracted

$\frac{3.98 \text{ g dry soil}}{10\text{ml } CaCl_2}$ x 0.1 ml aliquot = 0.0398 g of soil extracted

C. $\frac{\text{\# of colonies}}{0.0361\text{g soil (B)}}$ X dilution factor = number of colony forming units (CFU)

$\frac{41 \text{ colonies}}{0.0398 \text{ g soil}}$ x 1000 = 1.03×10^6 CFU

17. SAMPLE HARVEST

Duplicate irradiated and non-irradiated samples (2 vials of each at each time point) were harvested on Days 0, 3, 7, 14, 21, and 30 for CGA-329351 and CGA-48988. The samples were weighed and the weight recorded on a spreadsheet to determine any moisture lost during the incubation phase. The duplicate irradiated and non-irradiated samples were purged for 30 minutes, then extracted. Later an Extraction 2 was performed. Each extract and the combined volatile traps were radioassayed and the final residue combusted to determine the radiochemical balance.

In addition, surrogate samples of the irradiated and non-irradiated incubations were harvested at Day 30 for viability analysis.

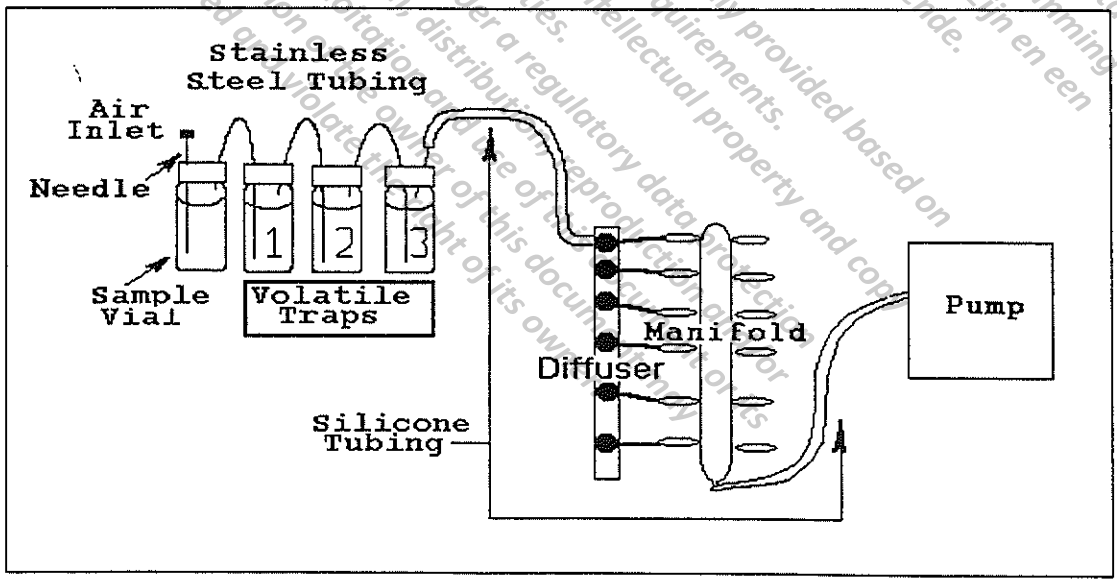
18. VOLATILE COLLECTION

Following Day 0, duplicate irradiated and non-irradiated samples were purged immediately after being harvested. The purge system consisted of a water aspirator pump (Cole Parmer Instrument Co.), a manifold, a diffuser for the manifold, three KOH (volatile) traps, a sample vial, and an

open top needle. The water aspirator pump was connected to the diffuser then to a glass manifold with multiple outlets. Most outlets were fitted with tygon tubing approximately 12 inches long and clamped to allow for optimization of the flow rate. Selected outlets were fitted with tygon tubing approximately 12 inches long with an 18 gauge needle attached to the end.

The volatiles were trapped by drawing air through the sample vial into a series of KOH traps at an approximate air flow of 19 mL/min. for 30 minutes. The KOH traps consisted of three 20 mL scintillation vials, each filled with 10 mL of a 10% w/v aqueous KOH solution and sealed with an open top cap and a Teflon coated septum. These volatile traps were connected by 25 gauge stainless steel canulas, inserted from the head space of the first trap into the solution of the next consecutive trap. The 18 gauge needle from the manifold was inserted through the septum of the third consecutive KOH trap.

A gentle negative pressure was established from the water aspirator pump, then the canula of the first trap was inserted into the head space of the sample. An open-top 25 gauge needle was immediately inserted into the solution of the sample to provide air flow.



Approximately 30 minutes later the canulas and needles were quickly removed from the sample vial and the KOH traps.

Assay of KOH Traps

Each KOH trap contained 10 mL of a 10% w/v aqueous KOH solution. After the samples were purged, duplicate aliquots of each of the three KOH traps were radioassayed in 10 mL of Ready Gel.

19. EXTRACTION 1

A solution of 8:2 acetonitrile:water was prepared by dispensing 800 mL of acetonitrile into a 1 liter graduated cylinder then adding 200 mL of water.

The moist soil was transferred to the Nalgene® bottle using a spatula. Approximately 10 mL of the 8:2 acetonitrile:water were added to the soil. The soil was placed in a Branson 2200 sonicator for thirty minutes. Ice was added to the water bath to prevent the samples from over-heating. The sample was centrifuged in the Beckman Optima L-60 Centrifuge for 10 minutes at 8000 rpm and 5°C. The supernatant was transferred to another 60 mL Nalgene® bottle. The procedure for extraction was repeated one additional time on the same sample and all supernatants were combined. The supernatant was centrifuged in the L-60 for 10 minutes at 8000 rpm and 5°C. The supernatant was transferred to a graduated cylinder. The volume was recorded and triplicate aliquots were radioassayed. Aliquots of the supernatants were assayed by two dimensional thin layer chromatography. Replicate 1 of all Extraction 1 supernatants were assayed by HPLC. The remaining centrifugation pellets were stored in the freezer, if necessary, for further analysis (Extraction 2).

20. EXTRACTION 2

A solution of 8:2 methanol:water was prepared by dispensing 800 mL of methanol into a 1 liter graduated cylinder then adding 200 mL of water.

The damp soil remaining from Extraction 1, including the pellet from centrifuging the Extraction 1 supernatant, was transferred to a glass centrifuge tube. Approximately 10 mL of 8:2 methanol:water was used to aid in transferring the soil and to rinse the Nalgene® bottle. The sample was vortexed and sonicated as needed to remove the soil from the Nalgene bottle.

The soil sample was placed in a pre-heated (~100°C) Pierce Reacti-therm heating module for approximately 2 hours. The samples were centrifuged at 1/2 of maximum speed for 30 minutes on a table top centrifuge.

The supernatant was transferred to a 15 mL graduated centrifuge tube. Triplicate 100 µl aliquots of the supernatant were radioassayed in 5 mL of Ready Safe. Aliquots of the supernatant were analyzed by two dimensional thin layer chromatography.

21. COMBUSTION OF RESIDUES

Aliquots of approximately 200 mg of homogenized soil samples were oxidized in a Harvey OX300 biological oxidizer for 4 minutes. The resulting radiolabeled carbon dioxide was trapped in 15 mL of Oxosol (National Diagnostics). Prior to combustion of the samples, a machine efficiency was determined by combustion of radiolabeled mannitol. The machine efficiency (calculated by Talisman version 1.0 computer software) needed to be greater than 90% to proceed with the sample combustions. The efficiency was determined both at the beginning and end of the day. The vials used for Talisman to calculate the efficiency were placed with the first sample group for the day. The remaining samples combusted during the day referenced the efficiency for the first sample group. Talisman version 1.0 computer software calculated the dpm and average dpm/g in a fraction based on the aliquot weight and sample weight of the fraction.

22. DETERMINATION OF RADIOCHEMICAL BALANCE

The total dpm of the dosed soil sample was based on the dpm applied at the time of dosing. Three aliquots of the dose solution were assayed by LSC analysis at various times during the application of the test substance to the soil surface. These assays were within ±0.08% of the mean.

The total radiochemical balance for each sample is equivalent to the sum of the percent radiochemical balance for each fraction (KOH or volatiles, Extraction 1, Extraction 2, and residue). The actual calculation for the radiochemical balance per fraction (or percent of original sample) was done in the Talisman Computer Program, version 1.0. Tables II and III contain the radioactivity

balance for each sample for Experiment 1 (CGA-329351) and Experiment 2 (CGA-48988), respectively.

Calculation:

Percent of Radiochemical Balance per fraction:

$$\frac{\text{DPM in the Fraction (volatiles, extract 1, 2, or residue)}}{\text{(Total dpm of dose)}} = \text{\% of Radiochemical Balance for the Fraction}$$

ex. Day 3, R1, Irradiated, CGA-329351 (extract 1, Table II)

$$\frac{\text{1,774,300 dpm in extract 1}}{\text{1,922,799 dpm}} \times 100 = \text{92.28\% of Radiochemical Balance for extract 1}$$

Percent of Radiochemical Balance:

% volatiles + % extract 1 + % extract 2 + % residue = % of Radiochemical Balance

ex. Day 3, R1, Irradiated, CGA-329351 (Table II)

0.08% (volatiles) + 92.28% (extract 1) + 3.98% (extract 2) + 1.83% (residue) =

98.17% Radiochemical Balance

23. SAMPLE STORAGE CONDITIONS

All samples were stored after harvest in Special Studies Unit I freezer which was maintained below -5°C. The freezer was monitored by the Environmental Monitoring System (EMS). A copy of all the temperature records is maintained in the freezer logbook. Some samples were stored temporarily in a freezer/refrigerator maintained at approximately -2°C.

24. PREPARATION OF SAMPLES FOR TLC

Extraction 1

Aliquots of approximately 100 µL of the Extraction 1 samples were applied to the TLC plates. Duplicate 100 µL aliquots of extraction samples were radioassayed in 5 mL of Ready Safe to determine the number of dpm of sample applied to each plate. Approximately 10,000 dpm of Extract 1 samples were applied to each TLC plate for initial two dimensional TLC characterization. The TLC plate recoveries ranged from 90.10% - 103.92%.

Calculation:

$$\frac{\text{dpm applied to TLC plate}}{\text{dpm recovered from TLC plate}} \times 100 = \text{TLC plate recovery}$$

Extraction 2

The extract 2 samples were light brown in color due to soil sediment. The samples were filtered using an Acrodisc to remove any remaining particulate. A 0.45 micron Acrodisc was attached to a 5 mL disposable leur-lok syringe and placed on a vac-elut system equipped with a water aspirator pump. The acrodisc was rinsed with methanol followed by water and the rinse discarded. The sample was eluted through the acrodisc followed by approximately a 1 mL methanol rinse. The clear filtrate was radioassayed and a recovery was calculated.

Aliquots of approximately 500 µL of the filtrate were applied to TLC plates for initial two dimensional TLC analysis. Duplicate 100 µl aliquots were radioassayed in 5 mL of Ready Safe to determine the number of dpm applied to each plate. The dpm value applied to the plate ranged from approximately 1455 to 7835 dpm with an average of approximately 4500 dpm. The TLC plate recoveries ranged from 89.53% - 102.47%.

25. PREPARATION AND USE OF REFERENCE STANDARDS

Receipt of Reference Standards

Reference standards were received from either Production Technical Analytical Services or the Chemical Synthesis Group of Ciba Crop Protection. Receipt documents contained but were not limited to: the compound name or number, the lot number, the quantity transferred, the purity, and the reassay date. The analyst assigned unique vial numbers to each reference standard.

Preparation of Reference Standards

Subsamples of the reference standards were prepared for use by dispensing the desired quantity into a tared vial and adding a measured volume of an appropriate solvent. All weight measurements were made on Mettler balances. All reference standards and their preparations were stored in a

freezer/refrigerator at approximately -2°C. Consistent TLC Rf values were used as a determination of reference standard stability. The TLC Rf values and the HPLC retention times remained consistent throughout the study. All information regarding reference standards and their preparations were recorded in a laboratory notebook.

Use of Reference Standards

Reference standards were used as a means of comparison between the UV visible reference standard and the radiolabel component. UV visible standards were used for TLC and HPLC. Since CGA-48988 is a racemic mixture of D and L isomers and CGA-329351 contains only the D isomer, reference standard CGA-48988 was used to cochromatograph with both CGA-48988 and CGA-329351.

26. THIN LAYER CHROMATOGRAPHY

Plate Preparation

One and two dimensional TLC plates were prepared and marked with a pencil:

Plates: 20 x 20 cm (one or two dimensional), 0.25 mm silica gel, fluorescence-visible, Merck

Origin: For one dimensional plates (preparatory plates), point or band approximately 1.5 cm from the bottom edge of the plate. For two dimensional plates, common point 1.5 cm from the bottom and left edge of the plate.

Solvent Line(s): marked across plate approximately 15 cm above the origin

Reference Standards: For two dimensional plates, non-radiolabeled standards were placed in the sample origin and in the margins that were created by the solvent lines. For one dimensional plates, the non-radiolabeled standards were place along side of the sample at the origin.

Plate Development

Solvent Systems were prepared for plate development and used within 2 days of preparation.

Solvent System I (SS I) chloroform:methanol:formic acid:water 75:20:4:2 v/v/v/v

Solvent System II (SS II) chloroform:methanol:ammonium hydroxide:water 80:30:4:2 v/v/v/v

Each prepared solvent system was transferred to a TLC chamber that contained a 20 x 20 cm saturation pad. One dimensional plates (preparatory plates) were developed in Solvent System II. Two dimensional plates were developed in Solvent System I, thoroughly dried, turned 90° counterclockwise, and developed in Solvent System II.

Application of Reference Standards and Radiolabeled Samples to TLC Plates

The solutions of nonradiolabeled reference standards were applied to TLC plates until the material was visible under a hand-held 254 nm UV lamp (Mineralight Lamp Model UVG-54 or UVGL-58). The locations of the reference standards were visualized under the same light after development and the locations of the reference standards were marked on the plates with a pencil.

Initially, all the standards were assayed by two dimensional TLC using both solvent systems. Rf values were calculated as a means of comparison between TLC plates.

$$Rf = \frac{\text{DISTANCE FROM THE ORIGIN TO THE CENTER OF THE UV SPOT (cm)}}{\text{THE DISTANCE FROM THE ORIGIN TO THE SOLVENT FRONT (cm)}}$$

Reference standards were applied in both margins of two dimensional plates. Direct comparison of the reference standards and samples was accomplished by applying appropriate reference standards and an aliquot of the sample being assayed to the origin. The plates were then developed and visualized as follows:

Visualization of Reference Standards and Radiolabeled Areas

Reference Standards: The non-radiolabeled reference standards were visualized using a hand held 254 nm UV lamp. The locations of the reference standards were visualized

under the same light after development and the locations were marked on the plates with a pencil.

A variety of methods were used to visualize the radiolabeled sample and non-radiolabeled reference standards on the plates:

Radioactive ink markers: Small spots of ^{14}C ink were placed arbitrarily in the plate margins to serve as positional guides.

Spark Chamber: Plates were placed in a spark chamber (Raytest Berta 7547) equipped with a Polaroid camera. The exposure time was adjusted according to the amount of radioactivity on the TLC plate.

Episcope: The spark chamber photograph was placed in the episcope which projected a 1:1 image of the photograph onto the TLC plate. The radiolabeled areas were marked with a pencil on the plate.

Fujix Scans: Plates were placed in a Fujix BAS 1000 Bioimaging Analysis System (Fuji), Tina software version 2.07a, 2.07d, 2.08 and BAS Read software version 2.6, for an amount of time relative to the total amount of radioactivity on the plate. Scans were computer enhanced. A 1:1 scale picture of the image was printed.

Light Box: A back-lighted stage was used to trace the radioactive areas from the 1:1 Fuji scans to the translucent plates. After UV visualization of the reference standards on the TLC plate, the locations of the reference standards and solvent fronts were traced onto the 1:1 Fuji scans.

Quantitation of TLC Plates

Two dimensional plates were used for quantitation. Each component was labeled with a letter. The remaining developed area of the plate was divided into four quadrants, labeled quadrant I-IV (quad I-IV). TLC plates were quantitated by scraping components and quadrants for radioassay. Approximately a 2 x 2 cm background was removed from a plate margin that contained no radiolabeled material. The individual components were placed in separate 20 mL scintillation vials with 5 mL water. Each vial was sonicated until the stationary phase was dispersed (Ultrasonic Processor, Branson 2200 or 3200 sonicator). The

fractions were vortexed to suspend the stationary phase in the water and 10 mL Ready Gel were immediately added and mixed to form a gel. The prepared fractions were equilibrated a minimum of 2 hours prior to radioassay.

Calculation of Percentages and Parts per Million of Plate Components

A. Percent Recovered of that Applied to the TLC Plate

The percent recovered of that applied to the TLC plate was calculated by the Talisman, version 1.0 computer program. Any hand calculations used the whole number dpm value rather than rounding.

$$\left[\frac{\text{Total DPM in fraction (Component)}}{\text{DPM Applied to TLC Plate}}\right] \times 100 = \%\ \text{Recovered of that Applied}$$

example: Day 0, R1, irradiated, extract 1, CGA - 329351 (component A, Table VI)

$$\left[\frac{10{,}530\ \text{dpm for component A}}{11{,}230\ \text{dpm applied to TLC plate}}\right] \times 100 = 93.77\ \%\ \text{Recovered of that Applied}$$

B. Total Dose per Component

$$\text{Total dose per component} = \frac{\%\ \text{Recovered of that Applied (calculation A)}}{100} \times \%\ \text{of total dose in fraction}$$

example: Day 0, R1, Irradiated, Extract 1, CGA - 329351 (Component A, Table VII)

$$\frac{93.77\ \%}{100} \times 96.48\ \%\ \text{of total dose in extract 1} = 90.47\%\ \text{Total Dose for Component A}$$

C. Distribution of Radiolabel Expressed as Parts Per Million (ppm)

The average dose rate was calculated for Experiment 1 incubation as 1.52 ppm for Experiment 1 and 1.51 ppm for Experiment 2(see Method Section 13). The ppm dose rate was calculated for each sample. The calculated ppm dose rate per sample was used in any calculations.

PPM represented by the Component or Quadrant =

$$\left[\frac{\text{\% Total Dose, [Extraction 1 (equation B) + Extraction 2]}}{100}\right] \times \text{ppX Dose Rate}$$

example: Day 0, R1, Irradiated, Extract 1 (Component A, Table XI)

$$\left[\frac{90.47\% + 3.24\%}{100}\right] \times 1.53 \text{ ppm Dose Rate} = 1.43 \text{ ppm}$$

27. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Analytical scale HPLC was used to profile Extraction 1, replicate 1 samples and to compare to TLC profiles. It was also used as a means of comparison of reference standards to radiolabeled components by comparison of retention times.

Each HPLC reservoir was filled with the appropriate solvent which had been filtered with a 0.2 µm filter. Reservoirs were degassed with a helium purge. The entire system was flushed each day of use. Internal reference standards were used to check the calibration of the instrument.

The instrument "lag time" (time in minutes between the UV detector and the fraction collector) was established. Aliquots of both non-labeled reference standard and radiolabeled Day 0, replicate 1, non-irradiated CGA-329351 were injected into the HPLC unit. The time between retention time of the reference standard by the UV monitor and elution of the major vial (representing 1 minute) in the histogram peak was 3.45 - 4.45 minutes.

The "lag time" (time in minutes between the UV detector and the ^{14}C beta ram detector) was also established. The difference between the retention time of the UV reference standard and the retention time for radiolabled Day 0 sample was 0.33 minutes. The instrument calculates the peak retention time from the middle of the peak, so the lag time between detectors may vary depending on the shape of the peak.

A. Stationary Phase

Injector: Rheodyne with 5 mL sample loop
Pump: Perkin Elmer LC Series 410
Flow Rate: 1 mL/minute

Spectrophotometer: Perkin Elmer LC-95, UV absorbance 235 ηm
Response Time: 2000 sec
^{14}C Detector: IN/US Beta Ram
Flow Cell: 600 µL glass cell

Fraction Collector: Foxy 200, 1 mL/minute per vial

Computer: Gateway 2000/4Sx-25
Monitor: Gateway 2000 Crystal Scan
Software: DOS Ver. 5.0; Microsoft Windows Ver. 3.1; β Ram (R) IN/US Systems, Inc. Ver. 3.10

Printer: Hewlett Packard Laser Jet 4SI

Column: Spherisorb ODS-1 , 4.6 mm ID x 250 mm, 5 µm
Guard Column: YMC Polymer C18

Pre column Filter: Upchurch Scientific, 0.5µm

<u>Preparation of 0.0125% Trifluoroacetic Acid (0.0125% TFA)</u>

One hundred twenty five microliters (125 µl) of Trifluoroacetic Acid (Fisher) was added to a 1 L volumetric flask filled almost to the graduation mark with water. Additional water was added bring the total volume to one liter.

B. <u>Mobile Phase</u>

Step	Time (Min.)	0.0125% TFA	% ACN	Gradient
1	0	90	10	
2	20	50	50	LINEAR
3	10	30	70	LINEAR
4	10	0	100	LINEAR
5	5	0	100	
6	5	90	10	LINEAR

28. <u>LIMITS OF DETECTION</u>

All balance and quantitation data was calculated in Talisman version 1.0 which used the counter dpm minus MQA (Minimal Quantifiable Amount). Hand calculations used the counter dpm minus background dpm so any value greater than background was considered a real value.

Specific Activity for CGA-329351 = 81.1 µCi/mg
Specific Activity for CGA-48944 = 73.2 µCi/mg

$$\text{Specific Activity} \times \left(2.22 \times 10^{6}\ \text{dpm}/\mu\text{Ci}\right) \times \text{mg}/1000\mu\text{g} = \text{dpm}/\mu\text{g}\ {}^{14}\text{C-CGA-329351 or } {}^{14}\text{C-CGA-48988}$$

$$81.1\ \mu\text{Ci}/\text{mg} \times \left(2.22 \times 10^{6}\ \text{dpm}/\mu\text{Ci}\right) \times \text{mg}/1000\mu\text{g} = 180{,}042\ \text{dpm}/\mu\text{g}\ {}^{14}\text{C-CGA-329351}$$

$$73.2\ \mu\text{Ci}/\text{mg} \times \left(2.22 \times 10^{6}\ \text{dpm}/\mu\text{Ci}\right) \times \text{mg}/1000\mu\text{g} = 162{,}504\ \text{dpm}/\mu\text{g}\ {}^{14}\text{C-CGA-48988}$$

Background values for TLC quantitations varied from 24-47 dpm for CGA-329351.

$$24\ \text{dpm} \div 180{,}042\ \text{dpm}/\mu\text{g} = 0.00013\ \mu\text{g}$$

$$47\ \text{dpm} \div 180{,}042\ \text{dpm}/\mu\text{g} = 0.00026\ \mu\text{g}$$

Background values for TLC quantitations varied from 24-48 dpm for CGA-48988.

$$24\ \text{dpm} \div 162{,}504\ \text{dpm}/\mu\text{g} = 0.00015\ \mu\text{g}$$

$$48\ \text{dpm} \div 162{,}504\ \text{dpm}/\mu\text{g} = 0.0003\mu\text{g}$$

Background values for radioassays and HPLC radioassays ranged from approximately 16 to 32 for CGA-329351.

$$16\ \text{dpm} \div 180{,}042\ \text{dpm}/\mu\text{g} = 0.000089\ \mu\text{g}$$

$$32\ \text{dpm} \div 180{,}042\ \text{dpm}/\mu\text{g} = 0.00018\ \mu\text{g}$$

Background values for radioassays and HPLC radioassays ranged from approximately 16 to 32 for CGA-48988.

$$16\ \text{dpm} \div 162{,}504\ \text{dpm}/\mu\text{g} = 0.000098\ \mu\text{g}$$

$$32\ \text{dpm} \div 162{,}504\ \text{dpm}/\mu\text{g} = 0.0002\mu\text{g}$$

Background values for radioassay of KOH fractions ranged from approximately 23 to 41 for CGA-329351.

$$23 \text{ dpm} \div 180{,}042 \ {}^{\text{dpm}}/_{\mu\text{g}} = 0.00013 \ \mu\text{g}$$

$$41 \text{ dpm} \div 180{,}042 \ {}^{\text{dpm}}/_{\mu\text{g}} = 0.00023 \ \mu\text{g}$$

Background values for radioassay of KOH fractions ranged from approximately 23 to 37 for CGA-48988.

$$23 \text{ dpm} \div 162{,}504 \ {}^{\text{dpm}}/_{\mu\text{g}} = 0.00014 \ \mu\text{g}$$

$$37 \text{ dpm} \div 162{,}504 \ {}^{\text{dpm}}/_{\mu\text{g}} = 0.00023 \mu\text{g}$$

Therefore, the limit of detection ranged from 0.000089 µg to 0.00026 µg for CGA-329351 and 0.000098 µg to 0.0003 µg for CGA-48988 for all assay types. In Tables XI and XVII, any values calculated to be less than 0.01 ppm were reported as <0.01 ppm.

29. SAMPLE ISOLATION AND PURIFICATION PROCEDURE

Selected Extraction 1 samples from CGA-329351, CGA-48988 and a combined bulk sample of both CGA-329351 and CGA-48988 each were combined in a graduated cylinder and radioassayed. Each subsequent combined fraction was transferred to a silylated round bottom flask and concentrated to approximately 1-2 mL on a Buchi RE111 Rotovapor equipped with a dry ice/acetone condenser. Each sample was transferred to a graduated tube. Each round bottom flask was rinsed three times with methanol and sonicated in a Branson 2200 table top sonicator. The methanol rinses were added to the respective graduated tube. Each sample was further concentrated under a stream of N_2 and analyzed by preparatory one dimensional TLC for total of three preparatory plates. Figure 74 is a representative preparatory TLC plate.

Each preparatory TLC plate was visualized as in Method Section 26 for radioactivity and UV. Each radiolabeled lane was marked on the TLC plate according the 1:1 Fujix scan. Each lane was scraped from the plate and placed in a centrifuge tube. Approximately 2 mL of isopropanol was added to the silica gel for each lane. Each sample was

vortexed approximately 30 seconds and sonicated (Branson 2200) for approximately 15 minutes. The water in the sonicator was changed several times to prevent the sample from over-heating. The samples were centrifuged for 10 minutes at 1/2 speed in a IEC-HN-SII centrifuge (International Equipment Co.). The supernatant was transferred to a clean centrifuge tube and concentrated to dryness under N_2. Only component A, Experiment 1, CGA-329351 and component A, Experiment 2, CGA-48988 contained enough radiolabel for further identification.

These samples were resolubilized in chloroform then were vortexed and sonicated approximately 5 minutes for further purification. Most of the residue still appeared on the glass. The chloroform fraction was transferred to a clean reaction vial and concentrated to dryness. The samples still contained a small amount of impurity. The samples were resolubilized in toluene then vortexed and sonicated. A greenish-yellow precipitant formed. The sample was centrifuged and the toluene fraction was transferred to a clean reaction vial and concentrated to dryness for mass spectrometry.

30. GAS CHROMATOGRAPHY/MASS SELECTIVE DETECTION (GC/MSD)

INSTRUMENT
Hewlett Packard 5890A GC
5971A MS Interface

COMPUTER AND SOFTWARE
Gateway 2000 4DX2-66W CPU
Crystal Scan 1572FX Monitor
MS DOS version 6.21
Microsoft Windows version 3.11
HP GC/MS program, version B.00.01
Data Analysis, version B.00.01

METHOD
Mode: Electron Ionization (EI)
Column: J&W DB1701
Scan Time: 0.5 sec
Mass Range: 50-500 mass units
Injector temperature: 225°C
Detector temperature (MS): 280°C
Initial temperature: 35°C
Initial time: 1 min.
Rate: 20°C /min

Final temperature: 280°C
Final time: 15 min.

All samples were dissolved in acetone.

31. RATE CONSTANT AND HALF LIFE DETERMINATION

The rate constant and half life determination were calculated using the Microsoft Excel computer software, version 5.0. The half life is displayed by two methods in Figures 83-86. The percent of the total dose for component A, CGA-329351 for Experiment 1 or CGA-48988 for Experiment 2 (Tables X and XVI) was entered into an Excel worksheet. The average percent parent was calculated for each time point. In method one, the actual percent of total dose for parent was plotted against time. In method two, the natural log of the average percent of total dose of parent was plotted against time. The TREND function was used to generate the best fit line based on the actual data points. The SLOPE function was used to generate the slope or rate constant of the best fit line generated from the graph of actual data points for the percent of total dose represented by component A. Figures 83-86 show a graphic representation (generated by Excel) of the half life for CGA-329351 and CGA-48988 irradiated and non-irradiated incubations.

Calculations:

$$\text{HALF LIFE} = -\frac{\ln 2}{\text{slope}}$$

V. RESULTS AND DISCUSSION

1. CIRCUMSTANCES AFFECTING THE OUTCOME OF THE STUDY

Agvise Laboratories conducted the soil analysis. Agvise Laboratories is an organization in GLP compliance and a signed compliance statement was included as a part of their report.[2] The clay mineralogy analysis by x-ray diffraction was contracted out from Agvise Laboratories to North Dakota State University. The university is not a GLP facility. The clay mineralogy is not a part of the Subdivision N Series 161-3 guidelines but instead is an additional analysis and should not affect this study.

Therefore there were no circumstances that adversely affected the outcome of the study.

2. SOIL HANDLING

The soil was harvested on March 17, 1995 from Ciba Crop Protection's Western Research Station, Sanger, California.[1] The soil was shipped from Sanger, California on March 17, 1995 and received at Ciba, Greensboro on March 20, 1995. The soil was sieved through a 2 mm sieve. An aliquot of the soil was double bagged in plastic for shipment to Agvise Laboratories for soil analysis. The soil moisture was determined prior to incubation. The initial moisture was 9.06%. The remaining soil was weighed and transferred to a plastic bag. The plastic bag was laid on its side in a metal tray to allow for approximately a 1-2 inch soil depth. Small slits were cut in the top of the bag to allow for air circulation. The metal tray containing the soil was loosely covered with foil to prevent the soil from drying out. The soil was stored in a constant temperature room at 25±1°C and approximately 50% humidity.

The results of the microbial analysis performed on March 27, 1995 indicated the soil contained an ample supply of bacteria and fungi (Table XVIII). The biomass determination conducted on March 28, 1995, indicated an average biomass of 82.230 mg C/kg soil (Table XVIII).

When a draft copy of the Agvise report was received on April 3, 1995, the soil moisture was determined again. The moisture was analyzed using a Computrac Max 50 Moisture Analyzer. The soil moisture was within 75% of FMC at 1/3 bar ± 12% and therefore was not adjusted for this study. The soil was returned to the constant temperature room until time for dosing. The final draft of the Agvise report was issued on April 7, 1995 with the same documented percent moisture.[2] Experiments 1 (CGA-329351) and Experiment 2 (CGA-48988) were dosed on April 4, 1995.

3. ARTIFICIAL AND NATURAL SUNLIGHT DISTRIBUTION

The average total natural sunlight intensity measured 410 Wm^2 on July 11, 1991 using a Radiolux Global Sensor. On July 28, 1992 at 1:14 PM the natural spectral distribution was measured from 200-700 nm. The intensity of the artificial light Suntest Unit was adjusted accordingly. The

artificial light spectral distribution was measured on March 29, 1995 for Suntest Unit 3 and Suntest Unit 4. Two Suntest Units were used for Experiment 1 and 2 due to the number of samples needed. Experiments 1 and 2 were run simultaneously therefore a beginning measurement was made for Suntest Units 3 and 4. Final measurements were made for Suntest Units 3 and 4 after the last samples were removed from the individual instrument. A final light measurement was made for Suntest Unit 3 on April 18, 1995 and for Suntest Unit 4 on May 5, 1995. A plot of the natural and artificial spectral distributions are displayed on Figure 5.

Even though the natural sunlight intensity was an average intensity, the spectral distribution used for the comparison graph of the natural to artificial light was measured during the most intense period of the day. For this reason the artificial light measurements are lower than the natural sunlight measurements but still approximate a daily average intensity. In all cases there was a minimal variation between the wavelengths of primary interest (200-400 nm) (Figure 5).

4. RADIOCHEMICAL PURITY OF THE TEST SUBSTANCE

The test substance provided by Ciba Crop Protection, Chemical Synthesis group was fully characterized under protocol and in accordance with experimental procedures approved by Ciba as Standard Operating Procedures. The radiochemical purity for CGA-329351 was 99.1% on March 9, 1995. The radiochemical purity for CGA-48988 was 98.4% on March 21, 1995. Prior to dosing, each test substance was analyzed by two dimensional TLC assays and quantitated to confirm the radiochemical purity stated by the Chemical Synthesis group. Quantitation results indicated a radiochemical purity of 99.87% for CGA-329351 and 99.43% for CGA-48988 (Figures 10 and 11 respectively).

Three aliquots of the dose solution (one at the beginning, middle and end of the dosing procedure) were analyzed by one dimensional TLC (Figure 12). The TLC results indicate there was no degradation of the dose solution during dosing.

5. VIABILITY ANALYSES

The viability analyses were conducted 8 days prior to dosing and on Day 30. The Day 30 analysis was performed on

surrogate samples of the irradiated and non-irradiated (i.e., dark control) CGA-48988 samples. The overall results indicate the samples remained viable throughout the Experiment 1 and 2 incubation time (Table XVIII).

6. TEMPERATURE MEASUREMENTS

The temperature of the irradiated and of the non-irradiated (dark control) samples was monitored by the Environmental Monitoring System (EMS). In addition, the temperature of the non-irradiated (dark control) samples was monitored by a Honeywell hydrothermograph. The temperature remained well within the 25±1°C range for the irradiated and non-irradiated incubations for CGA-329351 and CGA-48988. The results of the EMS data are shown in Figure 9.

7. HARVEST OF SAMPLES

Table I contains a sample list for Experiment 1 and Experiment 2. Duplicate irradiated and non-irradiated samples were pulled from their respective incubation chambers on Days 0,3,7,14,21, and 30 for Experiment 1 (CGA-329351) and Experiment 2 (CGA-48988).

At the time of dosing and time of harvest each sample was weighed and the weights recorded in a spreadsheet. Table IV and V contain the sample vial number, timepoint, description, identification number, weights at various time throughout the study and calculation values from the spreadsheet.

8. REFERENCE STANDARDS

All of the reference standards received from either Production Technical Analytical Services or from Chemical Synthesis, Ciba Geigy Corporation, Greensboro, NC, were fully characterized under protocol guidelines and in accordance with Standard Operating Procedures approved by Ciba. Figure 4 summarize assays of reference standards by thin layer chromatography using Solvent System I and II on silica gel plates. The stability of reference standards was determined by consistent Rf values by TLC analysis. Since CGA-48988 is a racemic mixture of D and L isomers and CGA-329351 contains only the D isomer, reference standard CGA-48988 was used to cochromatograph with both CGA-48988

and CGA-329351. Figure 10 indicates that CGA-329351 radiolabel test substance cochromatographs with CGA-48988 reference standard by two dimensional TLC. Figures 38 and 39 verify that CGA-48988 reference standard cochromatographs by HPLC with the CGA-329351 radiolabel sample. CGA-329351 and CGA-48988 were observed as a single component by both by TLC and HPLC.

9. COLLECTION OF VOLATILES

Volatiles were collected at each time point following Day 0 sampling by purging through three successive traps containing 10% w/v aqueous KOH. The total percent volatiles for each sample is the sum of the percent total radiolabel for all three KOH traps. The percent volatiles slightly increased over time to 1.16% of total dose for Experiments 1 and 2 (Tables II and III). The irradiated samples for both experiments contained a slightly higher percent volatiles than the non-irradiated samples.

10. EXTRACTION 1 and 2

The soil samples were extracted at the time of harvest. The percent of total dose present in Extraction 1 for Experiments 1 and 2 ranged from 90.35% - 97.16%, while the percent of total dose present in Extraction 2 ranged from 1.63% - 6.80%. The bound residues for Experiments 1 and 2 ranged from 1.34% - 6.96% of the total dose. The total radiochemical balance ranged from 98.17% - 104.67% of the total dose. The radiochemical balance for Experiments 1 and 2 is reported in Tables II and III and graphically displayed in Figures 81 and 82.

According to the TLC assays there is an overlap of degradates between the Extraction 1 and Extraction 2 fractions. Extraction 2 seems to release more of the same components seen in Extraction 1. Based on the two dimensional TLC assays, Experiments 1 and 2 contain 8 and 9 degradates, respectively. Each degradate accounted for less than 3.90% of the total dose. In both Extraction 1 and Extraction 2 fractions all the degradates with the exception of component F accounted for less than 2.21% of the total dose. The TLC results for Extraction 1 and Extraction 2 indicate that the irradiated and non-irradiated samples were qualitatively and quantitatively equivalent.

11. COMBUSTION OF RESIDUES

Multiple aliquots of the pellet remaining after Extractions 1 and 2, were oxidized to determine the concentration of unextractable radiolabel. The sample was combusted, the radiolabel trapped in an Oxosol fluor, then analyzed by LSC analysis. The residues for Experiment 1 and 2 generally increased over time to approximately 5% of the total dose. The percent of radiochemical balance was calculated and reported in Tables II and III as residue.

12. RADIOCHEMICAL BALANCE AND PPM DETERMINATION

The ppm dose rates were calculated based on the dpm applied at the time of dosing and the grams of soil per sample vial (Table IV and V). The average dose rate was 1.52 ppm for CGA-329351 and 1.51 ppm for CGA-48988 based on the moist soil weight. The radiochemical balance (recovery after harvest) was based on the radioassay of the dose solution at/near the time of dosing versus the radioassay of the volatiles, extracted sample and residues following harvest.

Some KOH fractions were assayed resulting in a less than the Minimum Quantifiable Amount (<MQA) generated by Talisman. These values were not included in the radiochemical balance calculation. The radiochemical balance for Experiment 1 ranged from 98.17% - 104.67% (Table II) and 99.86% - 104.24% for Experiment 2 (Table III).

13. TLC QUANTITATION OF DEGRADATES

Two dimensional TLC assays of the Extraction 1 and Extraction 2 fractions were used to qualitatively and quantitatively assay the samples from each time point. The amount of material assayed for Extraction 1 samples by two dimensional TLC was approximately 10,000 dpm. Extraction 2 samples represent a low percent of total dose (<6.80%). In order to avoid sample loss during concentration, a lesser number of total dpm was applied to TLC plates for Extraction 2 samples. There was still enough radiolabel, (usually 3,000 - 6,000 dpm) applied to the TLC plates to adequately quantitate these samples.

The results of the two dimensional TLC assays are shown in Figures 13-36 for CGA-329351 and 43-66 for 48988. Some samples were repeated for TLC analysis due to poor TLC recovery (<90%).

When some of the TLC plates were scraped and quantitated, Talisman generated "less than" MQA (Minimal Quantifiable Amount) values for several minor components. In these cases, less than MQA "(<MQA)" was listed in the Tables VI-XVII rather than the MQA value.

The distribution of radioactivity for each degradate from the TLC plate is summarized in Tables VI-XVII. These tables reflect the TLC recovery values as a part of the calculations. In Tables VI, VIII, XII and XIV the distribution of radiolabel is expressed as the percent of total applied radioactivity to the TLC plate. Tables VII, IX, XIII and XV contain the distribution of radiolabel expressed as the percent of total dose. In Tables X and XVI the distribution of radioactivity is expressed as percent of total dose for Extractions 1 and 2 combined since both extractions yielded the same components. Tables XI and XVII contain the distribution of radiolabel expressed in parts per million (ppm) for Extraction 1 and 2 combined.

14. RATE CONSTANT AND HALF LIFE DETERMINATION

The rate constant and half life were calculated using the Microsoft Excel computer software, version 5.0. A plot of the actual data points and best fit lines based on percent of parent is displayed on the top of Figures 83-86. A plot of the actual data points and the best fit line based on the natural log of the percent parent is displayed on the bottom of Figures 83-86. For the irradiated and non-irradiated incubations of both CGA-329351 and CGA-48988, approximately 13% of parent degraded by Day 30. This relatively minor degradation of parent was extrapolated to calculate half lives of 248 days for irradiated CGA-329351 samples as compared to 303 days for CGA-48988 samples. The half live value for the non-irradiated CGA-329351 was 358 days as compared to 755 days for CGA-48988. For both CGA-329351 and CGA-48988 the half lives were shorter for the irradiated versus the non-irradiated samples. The difference between the irradiated and non-irradiated half life values were exaggerated by the extrapolation of the 30 day results. The evidence indicates that photolysis on soil surfaces plays a minor role in the soil degradation of both CGA-329351 and

CGA-48988. The long half life values indicate that photolysis on soil was not a significant contributor to the degradation of either CGA-329351 or CGA-48988.

15. SAMPLE STORAGE CONDITIONS AND STABILITY

All extract and residue samples were stored following harvest in a freezer maintained below -5°C. The freezer was monitored by the Environmental Monitoring System (EMS). A copy of all the temperature records are maintained in the freezer logbook. Some samples were stored temporarily in a freezer/refrigerator maintained at approximately -2°C.

All samples were initially characterized within one week of the time they were harvested. Some extracts, such as Day 0 replicate 2 non-irradiated, however were stored in the freezer for up to four months before full characterization was completed. The Day 0, replicate 2, non-irradiated, CGA-48988 sample was analyzed by TLC at the initiation of the study. Near the completion of the study, the same Day 0 sample was analyzed again by TLC to verify if any degradation was observed. There did not appear to be any significant degradation due to storage conditions. Figure 73 shows the TLC both before storage (April 5, 1995) and after storage (July 26, 1995) for the Extraction 1 sample. This verifies that the extract was stable for this period of storage.

16. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Extraction 1, replicate 1 samples were analyzed by HPLC to confirm the results of two dimensional TLC. In each case, at least one reference standard was co-injected with the sample for comparison. The eluent was collected in 1 mL/min. vials. The vials were analyzed by LSC analysis and a histogram was generated by Talisman computer software version 1.0. Selected sample chromatographs and histograms are shown in Figures 37-42 and 67-72. Results indicate that CGA-329351 or CGA-48988 was the major component at all time points. The HPLC quantitation for parent was generally slightly higher than the TLC quantitation but compared favorably.

17. APPEARANCE AND DECLINE OF MAJOR DEGRADATES

The distribution of total dose between the volatiles, Extraction 1, Extraction 2 and residue fractions reported in Tables II and III are graphically displayed in Figure 81 and 82. In each case, the percent of total dose present in Extraction 1 decreased to approximately 90% by Day 30, while the Extraction 2 remained relatively constant at approximately 3-6%. Residue fractions increased then plateaued at approximately 3-5%. Volatiles were negligible ($\leq$1.16%) in every case. In both irradiated and non-irradiated samples from both experiments, parent appears to degrade either to CGA-62826 (component F) or to CGA-37734 (component C) and then to CGA-42447 (component B). CGA-62826 accounts for a maximum of 3.90% of total dose by Day 30. CGA-37734 accounts for a maximum 0.87% of total dose by Day 7. CGA-42447 accounts for a maximum 1.20% of total dose by Day 30.

18. CHARACTERIZATION AND IDENTIFICATION OF COMPONENTS

A. Component A (CGA-329351, Experiment 1)

Component A was isolated from one replicate each of the Day 14 and Day 30 irradiated, Experiment 1, Extraction 1 samples. The combined Day 14 and Day 30 sample was concentrated, spotted and developed for preparatory TLC. The lane corresponding to component A was scraped and extracted. Additional sample clean up was accomplished with chloroform followed by toluene. The sample was concentrated to dryness under N_2 and resolubilized in acetone. The isolated sample was analyzed by two dimensional TLC. The TLC results indicated that Experiment 1, component A was successfully isolated and did cochromatograph with reference standard CGA-48988 (Figure 74). An aliquot of the sample was also analyzed by GC/MS analysis (Figure 77). The fragment pattern matched CGA-48988 reference standard (Figure 75). In each experiment component A slowly degrades to approximately 87% of total dose by Day 30.

B. Component A, (CGA-48988, Experiment 2)

Component A was isolated from one replicate of Day 14, 21 and 30 irradiated, Experiment 2, Extraction 1 samples. The samples were spotted for preparatory TLC. TLC plates were developed, visualized and scraped. The silica gel was extracted and the radiolabeled material was isolated as

outlined for CGA-329351. The two dimensional TLC indicated that the isolation process was successful (Figure 74). An aliquot was analyzed by GC/MS (Figure 76). The fragment pattern matched the pattern produced by CGA-48988 reference standard (Figure 75).

C. Components B(CGA-42447), C(CGA-37734) and F(CGA-62826)

Components B, C and F were minor components usually representing less than 1.21% of total dose. Figure 78 indicates that CGA-42447 cochromatographed with Component B by two dimensional TLC. Figure 79 indicates that CGA-62826 cochromatographed with Component F by two dimensional TLC and by HPLC.

Component C is such a minor component (<0.83% of total dose) that even two dimensional TLC chromatography was difficult. A very light spot could be seen on some of the 1:1 Fujix scans which could match CGA-37734. The area where the reference standard CGA-37734 was visualized was scraped and quantitated to verify the presence of radiolabeled CGA-37734 (Figure 80). This component was also too minor to be visualized by HPLC.

19. General

Volatiles generally ranged from 0.04%-1.16% of total dose and therefore were considered negligible and were not further characterized. The volatiles were consistently slightly higher for the irradiated samples. For both experiments, CGA-42447 (component B), CGA-37734 (component C) and CGA-62826 (component F) accounted for a maximum of 1.20%, 0.83% and 3.90% of total dose respectively in the Extraction 1 and 2 samples. With the exception of component F (3.90% of total dose) in one of the irradiated Extraction 1 replicates, all components from both experiments each accounted for less than 2.21% of the total dose and therefore did not require further identification. The pattern of degradates in the irradiated and non-irradiated extracts from both experiments were qualitatively equivalent. There were some slight qualitative differences between the irradiated and non-irradiated samples however the levels were too low to make a valid distinction. For example, component J (Table X, CGA-329351, Experiment 1) accounted for 0.04% and 0.35% of total dose for Days 21 and 30 in the irradiated samples, respectively while these were not detected in the non-irradiated samples. The minor quantitative differences in

the irradiated versus the non-irradiated samples indicates that photolysis is a very minor factor in the degradation of these compounds.

VI. CONCLUSIONS

The soil photolysis results indicate that CGA-329351 has the same metabolic pathway and rate of degradation as seen in CGA-48988. CGA-329351 and CGA-48988, both irradiated and non-irradiated, are qualitatively equivalent. In all cases, parent seems to degrade either to CGA-62826 (Component F) or to CGA-37734 (Component C) and then to CGA-42447 (Component B). The relatively minor degradation of the parent compounds observed during the 30 day study was extrapolated to calculate approximate half life values. The half life for the CGA-329351 irradiated samples was 248 days as compared to 303 days for CGA-48988. The half life for non-irradiated CGA-329351 was 358 days as compared to 755 days for CGA-48988. Quantitative differences between irradiated and non-irradiated samples indicate that photolysis on soil is a very minor route of degradation for these compounds.

TABLE I: SAMPLE LIST FOR CGA-329351, EXPERIMENT 1 AND CGA-48988, EXPERIMENT 2

CGA-329351, EXPERIMENT 1			
VIAL #	TIMEPOINT	DESCRIPTION	ID NUMBER
1	DAY 0	IRRAD SOIL REP 1	129386
2	DAY 0	IRRAD SOIL REP 2	129392
3	DAY 0	NIR SOIL REP 1	129398
4	DAY 0	NIR SOIL REP 2	129404
5	DAY 3	IRRAD SOIL REP 1	129387
6	DAY 3	IRRAD SOIL REP 2	129393
15	DAY 3	NIR SOIL REP 1	129399
16	DAY 3	NIR SOIL REP 2	129405
9	DAY 7	IRRAD SOIL REP 1	129388
10	DAY 7	IRRAD SOIL REP 2	129394
11	DAY 7	NIR SOIL REP 1	129400
12	DAY 7	NIR SOIL REP 2	129406
13	DAY 14	IRRAD SOIL REP 1	129389
14	DAY 14	IRRAD SOIL REP 2	129395
7	DAY 14	NIR SOIL REP 1	129401
8	DAY 14	NIR SOIL REP 2	129407
18	DAY 21	IRRAD SOIL REP 1	129390
21	DAY 21	IRRAD SOIL REP 2	129396
27	DAY 21	NIR SOIL REP 1	129402
28	DAY 21	NIR SOIL REP 2	129408
17	DAY 30	IRRAD SOIL REP 1	129391
26	DAY 30	IRRAD SOIL REP 2	129397
23	DAY 30	NIR SOIL REP 1	129403
24	DAY 30	NIR SOIL REP 2	129409

CGA-48988, EXPERIMENT 2			
VIAL #	TIMEPOINT	DESCRIPTION	ID NUMBER
50	DAY 0	IRRAD SOIL REP 1	129410
51	DAY 0	IRRAD SOIL REP 2	129416
52	DAY 0	NIR SOIL REP 1	129422
53	DAY 0	NIR SOIL REP 2	129428
54	DAY 3	IRRAD SOIL REP 1	129411
55	DAY 3	IRRAD SOIL REP 2	129417
56	DAY 3	NIR SOIL REP 1	129423
57	DAY 3	NIR SOIL REP 2	129429
62	DAY 7	IRRAD SOIL REP 1	129412
63	DAY 7	IRRAD SOIL REP 2	129418
60	DAY 7	NIR SOIL REP 1	129424
61	DAY 7	NIR SOIL REP 2	129430
58	DAY 14	IRRAD SOIL REP 1	129413
66	DAY 14	IRRAD SOIL REP 2	129419
76	DAY 14	NIR SOIL REP 1	129425
77	DAY 14	NIR SOIL REP 2	129431
74	DAY 21	IRRAD SOIL REP 1	129414
75	DAY 21	IRRAD SOIL REP 2	129420
64	DAY 21	NIR SOIL REP 1	129426
65	DAY 21	NIR SOIL REP 2	129432
67	DAY 30	IRRAD SOIL REP 1	129415
78	DAY 30	IRRAD SOIL REP 2	129421
68	DAY 30	NIR SOIL REP 1	129427
69	DAY 30	NIR SOIL REP 2	129433

IRRAD = IRRADIATED
NIR = NON-IRRADIATED

VIAL NUMBERS ARE NOT IN NUMERICAL ORDER BUT REFLECT THE VIAL NUMBER FOR THE SAMPLE HARVESTED AT A PARTICULAR TIME POINT

TABLE II: DISTRIBUTION OF RADIOACTIVITY IN FRACTIONS FROM CGA-329351, EXPERIMENT 1, EXPRESSED AS PERCENT OF TOTAL DOSE

SAMPLE	VOLATILES	EXTRACTION 1	EXTRACTION 2	RESIDUE	BALANCE
DAY 0 R1 IRRAD	NA	96.48	3.48	1.34	101.30
DAY 0 R2 IRRAD	NA	95.09	3.14	1.61	99.84
DAY 0 R1 NIR	NA	94.93	4.49	1.37	100.79
DAY 0 R2 NIR	NA	96.52	4.18	1.46	102.16
DAY 3 R1 IRRAD	0.08	92.28	3.98	1.83	98.17
DAY 3 R2 IRRAD	0.09	96.06	4.36	1.42	101.93
DAY 3 R1 NIR	0.06	97.16	4.27	1.55	103.04
DAY 3 R2 NIR	0.05	91.31	6.76	2.34	100.46
DAY 7 R1 IRRAD	0.13	93.90	4.81	3.92	102.76
DAY 7 R2 IRRAD	0.17	93.35	3.55	4.41	101.48
DAY 7 R1 NIR	0.05	94.64	3.28	4.42	102.39
DAY 7 R2 NIR	0.05	96.08	3.02	5.52	104.67
DAY 14 R1 IRRAD	0.41	91.84	4.20	4.00	100.45
DAY 14 R2 IRRAD	0.35	93.18	2.59	5.32	101.44
DAY 14 R1 NIR	0.10	92.66	4.19	3.22	100.17
DAY 14 R2 NIR	0.10	92.99	3.93	3.00	100.02
DAY 21 R1 IRRAD	0.35	90.35	3.42	5.21	99.33
DAY 21 R2 IRRAD	0.37	93.76	2.71	5.34	102.18
DAY 21 R1 NIR	0.06	96.06	2.79	5.73	104.64
DAY 21 R2 NIR	0.06	95.43	3.25	4.82	103.56
DAY 30 R1 IRRAD	0.86	93.78	5.46	4.55	104.65
DAY 30 R2 IRRAD	0.14	95.23	3.70	3.68	102.75
DAY 30 R1 NIR	0.07	93.86	6.05	2.66	102.64
DAY 30 R2 NIR	0.04	92.88	6.32	2.62	101.86

0 HOUR SAMPLES WERE NOT PURGED FOR VOLATILES AND THEREFORE A PERCENT VOLATILE IS NOT APPLICABLE (NA)

TABLE III: DISTRIBUTION OF RADIOACTIVITY IN FRACTIONS FROM CGA-48988, EXPERIMENT 2, EXPRESSED AS PERCENT OF TOTAL DOSE

SAMPLE	VOLATILES	EXTRACTION 1	EXTRACTION 2	RESIDUE	BALANCE
DAY 0 R1 IRRAD	NA	96.55	2.65	3.17	102.37
DAY 0 R2 IRRAD	NA	94.13	2.98	3.90	101.01
DAY 0 R1 NIR	NA	94.41	4.74	1.45	100.60
DAY 0 R2 NIR	NA	95.17	2.04	3.44	100.65
DAY 3 R1 IRRAD	0.11	93.15	3.13	4.32	100.71
DAY 3 R2 IRRAD	0.11	92.26	3.08	4.41	99.86
DAY 3 R1 NIR	0.07	96.33	3.76	3.12	103.28
DAY 3 R2 NIR	0.07	95.80	3.68	4.32	103.87
DAY 7 R1 IRRAD	0.26	92.24	6.80	4.43	103.73
DAY 7 R2 IRRAD	0.27	93.52	5.16	2.91	101.86
DAY 7 R1 NIR	0.11	93.19	5.40	3.64	102.34
DAY 7 R2 NIR	0.12	92.28	5.95	3.48	101.83
DAY 14 R1 IRRAD	0.51	92.17	3.85	6.96	103.49
DAY 14 R2 IRRAD	0.39	93.67	5.33	3.99	103.38
DAY 14 R1 NIR	0.07	93.71	5.69	4.77	104.24
DAY 14 R2 NIR	0.06	94.12	5.03	3.87	103.08
DAY 21 R1 IRRAD	0.24	92.24	4.82	4.92	102.22
DAY 21 R2 IRRAD	0.43	93.84	4.68	4.52	103.47
DAY 21 R1 NIR	0.09	92.85	5.18	4.29	102.41
DAY 21 R2 NIR	0.06	93.48	5.52	3.81	102.87
DAY 30 R1 IRRAD	1.16	91.36	1.63	6.64	100.79
DAY 30 R2 IRRAD	0.47	92.04	4.49	4.94	101.94
DAY 30 R1 NIR	0.09	92.82	5.69	4.92	103.52
DAY 30 R2 NIR	0.08	96.02	3.85	3.14	103.09

0 HOUR SAMPLES WERE NOT PURGED FOR VOLATILES AND THEREFORE A PERCENT VOLATILE IS NOT APPLICABLE (NA)

TABLE IV: WEIGHTS, DPM/G AND PPM CALCULATIONS FOR CGA-329351 INCUBATIONS

				INITIAL (DOSE TIME)			POST HARVEST						
VIAL #	TIMEPOINT	DESCRIPTION	ID NUMBER	VIAL TARE W/ CAP (G)	VIAL + MOIST SOIL (G)	MOIST SOIL (G)	FINAL WEIGHT (G)	POST PURGE (G)	FINAL SOIL WEIGHT (G)	MOIST LOST (G)	% MOIST LOST	DPM/G	PPM
1	DAY 00	IRRAD SOIL REP 1	129386	36.443	43.419	6.976	NA	NA	6.976	NA	NA	275,630	1.53
2	DAY 00	IRRAD SOIL REP 2	129392	36.847	43.849	7.002	NA	NA	7.002	NA	NA	274,607	1.53
3	DAY 00	NIR SOIL REP 1	129398	38.728	45.768	7.040	NA	NA	7.040	NA	NA	273,124	1.52
4	DAY 00	NIR SOIL REP 2	129404	35.779	42.748	6.969	NA	NA	6.969	NA	NA	275,907	1.53
5	DAY 03	IRRAD SOIL REP 1	129387	33.217	40.240	7.023	40.30	40.25	7.08	-0.060	-0.85	273,786	1.52
6	DAY 03	IRRAD SOIL REP 2	129393	36.896	43.734	6.838	43.76	43.71	6.86	-0.026	-0.38	281,193	1.56
15	DAY 03	NIR SOIL REP 1	129399	32.945	39.872	6.927	39.92	39.88	6.98	-0.048	-0.69	277,580	1.54
16	DAY 03	NIR SOIL REP 2	129405	37.666	44.727	7.061	44.77	44.73	7.10	-0.043	-0.61	272,312	1.51
9	DAY 07	IRRAD SOIL REP 1	129388	36.352	43.358	7.006	43.70	43.68	7.35	-0.342	-4.88	274,450	1.52
10	DAY 07	IRRAD SOIL REP 2	129394	37.537	44.513	6.976	44.55	44.45	7.01	-0.037	-0.53	275,630	1.53
11	DAY 07	NIR SOIL REP 1	129400	37.427	44.447	7.020	44.48	44.45	7.05	-0.033	-0.47	273,903	1.52
12	DAY 07	NIR SOIL REP 2	129406	36.977	44.089	7.112	44.14	43.98	7.16	-0.051	-0.72	270,359	1.50
13	DAY 14	IRRAD SOIL REP 1	129389	36.268	43.340	7.072	43.36	43.32	7.09	-0.020	-0.28	271,889	1.51
14	DAY 14	IRRAD SOIL REP 2	129395	37.788	44.805	7.017	45.14	45.07	7.35	-0.335	-4.77	274,020	1.52
7	DAY 14	NIR SOIL REP 1	129401	39.157	46.127	6.970	46.15	46.12	6.99	-0.023	-0.33	275,867	1.53
8	DAY 14	NIR SOIL REP 2	129407	33.431	40.510	7.079	40.53	40.52	7.10	-0.020	-0.28	271,620	1.51
18	DAY 21	IRRAD SOIL REP 1	129390	31.418	38.563	7.145	38.94	38.86	7.52	-0.377	-5.28	269,111	1.49
21	DAY 21	IRRAD SOIL REP 2	129396	34.846	41.868	7.022	41.94	41.92	7.09	-0.072	-1.03	273,825	1.52
27	DAY 21	NIR SOIL REP 1	129402	32.111	39.169	7.058	39.22	39.21	7.11	-0.051	-0.72	272,428	1.51
28	DAY 21	NIR SOIL REP 2	129408	33.889	40.787	6.898	40.84	40.84	6.95	-0.053	-0.77	278,747	1.55
17	DAY 30	IRRAD SOIL REP 1	129391	32.967	40.054	7.087	40.09	40.04	7.12	-0.036	-0.51	271,313	1.51
26	DAY 30	IRRAD SOIL REP 2	129397	31.021	38.079	7.058	38.17	38.13	7.15	-0.091	-1.29	272,428	1.51
23	DAY 30	NIR SOIL REP 1	129403	33.147	40.189	7.042	40.25	40.24	7.10	-0.061	-0.87	273,047	1.52
24	DAY 30	NIR SOIL REP 2	129409	31.896	38.828	6.932	38.81	38.81	6.91	0.018	0.26	277,380	1.54

DEFINITIONS

VIAL TARE WITH CAP = WEIGHT OF EMPTY VIAL WITH CAP

VIAL + MOIST SOIL = WEIGHT OF VIAL AND ~7 g OF MOIST SOIL

FINAL WEIGHT = WEIGHT OF VIAL AND MOIST SOIL AT HARVEST

POST PURGE= WEIGHT OF VIAL AND MOIST SOIL AFTER PURGING

CALCULATIONS

MOIST SOIL =(VIAL TARE WITH CAP) - (VIAL + MOIST SOIL)

FINAL SOIL WEIGHT= (FINAL WEIGHT - VIAL TARE)

MOISTURE LOST = (FINAL SOIL WEIGHT) - (MOIST SOIL WEIGHT)

% MOISTURE LOST =[(MOIST LOST)/ (MOIST SOIL)] X 100

DPM/G = DPM APPLIED / WEIGHT OF MOIST SOIL

TABLE V: WEIGHTS, DPM/G AND PPM CALCULATIONS FOR CGA-48988 INCUBATIONS

				INITIAL (DOSE TIME)			POST HARVEST						
VIAL #	TIMEPOINT	DESCRIPTION	ID NUMBER	VIAL TARE W/ CAP (G)	VIAL + MOIST SOIL (G)	MOIST SOIL (G)	FINAL WEIGHT (G)	POST PURGE (G)	FINAL SOIL WEIGHT (G)	MOIST LOST (G)	% MOIST LOST	DPM/G	PPM
50	DAY 00	IRRAD SOIL REP 1	129410	36.543	43.521	6.978	NA	NA	6.978	NA	NA	248,015	1.53
51	DAY 00	IRRAD SOIL REP 2	129416	37.523	44.523	7.000	NA	NA	7.000	NA	NA	247,236	1.52
52	DAY 00	NIR SOIL REP 1	129422	32.814	39.763	6.949	NA	NA	6.949	NA	NA	249,050	1.53
53	DAY 00	NIR SOIL REP 2	129428	36.925	43.933	7.008	NA	NA	7.008	NA	NA	246,953	1.52
54	DAY 03	IRRAD SOIL REP 1	129411	36.767	43.801	7.034	43.94	43.92	7.15	-0.119	-1.69	246,041	1.51
55	DAY 03	IRRAD SOIL REP 2	129417	36.430	43.493	7.063	43.54	43.52	7.09	-0.027	-0.38	245,030	1.51
56	DAY 03	NIR SOIL REP 1	129423	34.265	41.298	7.033	41.34	41.31	7.05	-0.012	-0.17	246,076	1.51
57	DAY 03	NIR SOIL REP 2	129429	36.852	43.924	7.072	43.97	43.92	7.07	0.004	0.06	244,719	1.51
62	DAY 07	IRRAD SOIL REP 1	129412	36.646	43.717	7.071	43.88	43.86	7.21	-0.143	-2.02	244,753	1.51
63	DAY 07	IRRAD SOIL REP 2	129418	33.018	40.061	7.043	40.40	40.39	7.37	-0.329	-4.67	245,726	1.51
60	DAY 07	NIR SOIL REP 1	129424	37.319	44.395	7.076	44.44	44.42	7.10	-0.025	-0.35	244,580	1.51
61	DAY 07	NIR SOIL REP 2	129430	32.971	40.021	7.050	40.05	40.00	7.03	0.021	0.30	245,482	1.51
58	DAY 14	IRRAD SOIL REP 1	129413	36.895	43.895	7.000	44.16	44.09	7.20	-0.195	-2.79	247,236	1.52
66	DAY 14	IRRAD SOIL REP 2	129419	38.041	45.071	7.030	45.21	45.15	7.11	-0.079	-1.12	246,181	1.51
76	DAY 14	NIR SOIL REP 1	129425	32.547	39.437	6.890	39.49	39.48	6.93	-0.043	-0.62	251,183	1.55
77	DAY 14	NIR SOIL REP 2	129431	32.066	39.008	6.942	39.03	39.02	6.95	-0.012	-0.17	249,301	1.53
74	DAY 21	IRRAD SOIL REP 1	129414	32.845	39.943	7.098	40.01	39.98	7.14	-0.037	-0.52	243,822	1.50
75	DAY 21	IRRAD SOIL REP 2	129420	33.417	40.405	6.988	40.48	40.47	7.05	-0.065	-0.93	247,660	1.52
64	DAY 21	NIR SOIL REP 1	129426	37.154	44.219	7.065	44.24	44.22	7.07	-0.001	-0.01	244,961	1.51
65	DAY 21	NIR SOIL REP 2	129432	36.379	43.377	6.998	43.40	43.38	7.00	-0.003	-0.04	247,306	1.52
67	DAY 30	IRRAD SOIL REP 1	129415	36.736	43.753	7.017	44.11	43.90	7.16	-0.147	-2.09	246,637	1.52
78	DAY 30	IRRAD SOIL REP 2	129421	33.599	40.595	6.996	40.71	40.68	7.08	-0.085	-1.21	247,377	1.52
68	DAY 30	NIR SOIL REP 1	129427	33.867	41.479	7.612	41.51	41.47	7.60	0.009	0.12	227,358	1.40
69	DAY 30	NIR SOIL REP 2	129433	34.506	41.618	7.112	41.68	41.65	7.14	-0.032	-0.45	243,342	1.50

DEFINITIONS
VIAL TARE WITH CAP = WEIGHT OF EMPTY VIAL WITH CAP
VIAL + MOIST SOIL = WEIGHT OF VIAL AND ~7 g OF MOIST SOIL
FINAL WEIGHT = WEIGHT OF VIAL AND MOIST SOIL AT HARVEST
POST PURGE= WEIGHT OF VIAL AND MOIST SOIL AFTER PURGING

CALCULATIONS
MOIST SOIL =(VIAL TARE WITH CAP) - (VIAL + MOIST SOIL)
FINAL SOIL WEIGHT= (FINAL WEIGHT - VIAL TARE)
MOISTURE LOST = (FINAL SOIL WEIGHT) - (MOIST SOIL WEIGHT)
% MOISTURE LOST =[(MOIST LOST)/ (MOIST SOIL)] X 100
DPM/G = DPM APPLIED / MOIST SOIL WEIGHT

TABLE VI: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 1, EXPRESSED AS PERCENT OF TOTAL APPLIED RADIOACTIVITY RECOVERED FROM THE TLC PLATE

CGA-329351, EXTRACTION 1

CGA#		329351	42447	37734		62826								
IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	94.18	93.77	ND	ND	ND	ND	ND	ND	ND	ND	0.10	<MQA	0.07	0.24
DAY 0 R2	93.70	93.42	ND	ND	ND	ND	ND	ND	ND	ND	0.14	<MQA	0.10	0.05
DAY 3 R1	94.87	93.67	ND	ND	ND	ND	ND	ND	ND	ND	0.22	<MQA	0.56	0.42
DAY 3 R2	93.18	90.77	ND	ND	ND	ND	ND	ND	ND	ND	1.12	0.26	0.48	0.55
DAY 7 R1	99.94	96.65	0.24	ND	0.18	0.33	0.20	ND	ND	ND	0.50	0.42	0.57	0.65
DAY 7 R2	97.35	96.73	ND	ND	ND	0.01	0.25	ND	ND	ND	0.12	0.07	0.02	0.14
DAY 14 R1	91.45	89.24	0.44	ND	ND	ND	0.36	ND	ND	ND	0.38	0.03	0.36	0.64
DAY 14 R2	94.04	91.40	ND	ND	ND	0.71	ND	ND	ND	ND	0.39	0.17	0.41	0.97
DAY 21 R1	96.71	92.98	0.69	0.27	ND	0.63	0.32	ND	ND	0.34	0.44	0.09	0.45	0.51
DAY 21 R2	91.48	88.75	0.71	0.17	ND	0.35	0.24	ND	ND	ND	0.29	0.08	0.41	0.47
DAY 30 R1	92.25	86.17	1.15	ND	ND	0.82	0.42	0.64	0.37	0.62	0.64	0.12	0.36	0.95
DAY 30 R2	90.39	88.20	0.51	ND	ND	0.48	ND	ND	ND	ND	0.11	0.08	0.46	0.54

NON-IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	94.32	92.11	ND	ND	ND	ND	ND	ND	ND	ND	1.87	0.07	0.09	0.18
DAY 0 R2	94.75	94.17	ND	ND	ND	ND	ND	ND	ND	ND	0.05	0.18	0.14	0.21
DAY 3 R1	93.77	92.68	ND	ND	ND	ND	ND	ND	ND	ND	0.38	0.12	0.33	0.27
DAY 3 R2	93.11	91.51	ND	ND	ND	ND	ND	ND	ND	ND	0.78	0.19	0.35	0.29
DAY 7 R1	92.84	91.91	ND	ND	ND	0.32	ND	ND	ND	ND	0.08	0.04	0.18	0.31
DAY 7 R2	96.45	94.54	0.32	ND	0.23	0.29	0.20	ND	ND	ND	0.14	0.04	0.22	0.48
DAY 14 R1	94.85	92.87	ND	ND	ND	0.70	ND	ND	ND	ND	0.35	0.24	0.23	0.46
DAY 14 R2	94.63	92.98	ND	ND	ND	0.48	ND	ND	ND	ND	0.28	0.18	0.28	0.43
DAY 21 R1	96.35	95.24	ND	ND	ND	0.56	ND	ND	ND	ND	0.21	0.07	0.08	0.19
DAY 21 R2	97.94	97.12	ND	ND	ND	0.46	ND	ND	ND	ND	0.11	0.05	0.06	0.14
DAY 30 R1	91.16	90.11	ND	ND	ND	0.60	ND	ND	ND	ND	0.06	0.03	0.09	0.28
DAY 30 R2	92.44	88.24	ND	ND	ND	3.73	ND	ND	ND	ND	0.04	0.02	0.23	0.17

CALCULATION: $\frac{\text{DPM per Component}}{\text{DPM applied to TLC plate}} \times 100 =$ Percent of total Applied Radioactivity per Component

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE VII: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 1, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-329351, EXTRACTION 1

CGA#		329351	42447	37734		62826								
IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	96.48	90.47	ND	ND	ND	ND	ND	ND	ND	ND	0.10	< MQA	0.07	0.23
DAY 0 R2	95.09	88.83	ND	ND	ND	ND	ND	ND	ND	ND	0.13	< MQA	0.10	0.05
DAY 3 R1	92.28	86.44	ND	ND	ND	ND	ND	ND	ND	ND	0.20	< MQA	0.52	0.39
DAY 3 R2	96.06	87.19	ND	ND	ND	ND	ND	ND	ND	ND	1.08	0.25	0.46	0.53
DAY 7 R1	93.90	90.94	0.23	ND	0.17	0.31	0.19	ND	ND	ND	0.47	0.39	0.54	0.61
DAY 7 R2	93.35	90.30	ND	ND	ND	0.01	0.23	ND	ND	ND	0.11	0.07	0.02	0.13
DAY 14 R1	91.84	81.96	0.40	ND	ND	ND	0.33	ND	ND	ND	0.35	0.03	0.33	0.59
DAY 14 R2	93.18	85.17	ND	ND	ND	0.66	ND	ND	ND	ND	0.36	0.16	0.38	0.90
DAY 21 R1	90.35	84.01	0.62	0.24	ND	0.57	0.29	ND	ND	0.31	0.40	0.08	0.41	0.46
DAY 21 R2	93.76	83.21	0.67	0.16	ND	0.33	0.23	ND	ND	ND	0.27	0.08	0.38	0.44
DAY 30 R1	93.78	80.81	1.08	ND	ND	0.77	0.39	0.60	0.35	0.58	0.60	0.11	0.34	0.89
DAY 30 R2	95.23	83.99	0.49	ND	ND	0.46	ND	ND	ND	ND	0.10	0.08	0.44	0.51

NON-IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	94.93	87.44	ND	ND	ND	ND	ND	ND	ND	ND	1.78	0.07	0.09	0.17
DAY 0 R2	96.52	90.89	ND	ND	ND	ND	ND	ND	ND	ND	0.05	0.17	0.14	0.20
DAY 3 R1	97.16	90.05	ND	ND	ND	ND	ND	ND	ND	ND	0.37	0.12	0.32	0.26
DAY 3 R2	91.31	83.56	ND	ND	ND	ND	ND	ND	ND	ND	0.71	0.17	0.32	0.26
DAY 7 R1	94.64	86.98	ND	ND	ND	0.30	ND	ND	ND	ND	0.08	0.04	0.17	0.29
DAY 7 R2	96.08	90.83	0.31	ND	0.22	0.28	0.19	ND	ND	ND	0.13	0.04	0.21	0.46
DAY 14 R1	92.66	86.05	ND	ND	ND	0.65	ND	ND	ND	ND	0.32	0.22	0.21	0.43
DAY 14 R2	92.99	86.46	ND	ND	ND	0.45	ND	ND	ND	ND	0.26	0.17	0.26	0.40
DAY 21 R1	96.06	91.49	ND	ND	ND	0.54	ND	ND	ND	ND	0.20	0.07	0.08	0.18
DAY 21 R2	95.43	92.68	ND	ND	ND	0.44	ND	ND	ND	ND	0.10	0.05	0.06	0.13
DAY 30 R1	93.86	84.58	ND	ND	ND	0.56	ND	ND	ND	ND	0.06	0.03	0.08	0.26
DAY 30 R2	92.88	81.96	ND	ND	ND	3.46	ND	ND	ND	ND	0.04	0.02	0.21	0.16

CALCULATION: $\frac{\text{\% of Total Recovered Radioactivity (Table VI)}}{100}$ x % of Total Dose in Fraction = % Total dose per Component, Extraction 1

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE VIII: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 2, EXPRESSED AS PERCENT OF TOTAL APPLIED RADIOACTIVITY RECOVERED FROM THE TLC PLATE

CGA-329351, EXTRACTION 2

CGA#		329351	42447	37734		62826								
IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	97.29	93.19	ND	ND	ND	ND	ND	ND	ND	ND	1.34	0.88	0.96	0.92
DAY 0 R2	95.41	91.32	ND	ND	ND	ND	ND	ND	ND	ND	1.96	0.36	0.78	1.00
DAY 3 R1	89.95	88.77	ND	ND	ND	ND	ND	ND	ND	ND	0.33	<MQA	0.20	0.65
DAY 3 R2	95.21	92.78	ND	ND	ND	ND	ND	ND	ND	ND	0.61	0.34	0.15	1.33
DAY 7 R1	93.30	90.22	ND	ND	ND	ND	ND	ND	ND	ND	0.95	0.07	1.09	0.97
DAY 7 R2	93.87	88.58	ND	ND	ND	ND	ND	ND	ND	ND	1.37	0.56	1.45	1.91
DAY 14 R1	90.13	84.69	ND	ND	ND	0.91	1.27	ND	ND	ND	1.02	0.13	0.47	1.64
DAY 14 R2	93.99	85.39	0.83	0.55	ND	0.79	1.25	ND	ND	ND	1.32	1.25	1.29	1.32
DAY 21 R1	91.11	80.11	0.79	0.26	ND	1.29	1.49	2.82	1.16	ND	1.17	0.15	0.86	1.00
DAY 21 R2	95.88	88.71	ND	ND	ND	0.81	1.37	ND	ND	ND	1.54	0.09	1.49	1.86
DAY 30 R1	98.12	86.43	1.30	0.56	ND	1.33	1.29	ND	ND	ND	3.95	0.93	1.16	1.00
DAY 30 R2	94.19	89.38	0.79	ND	ND	1.02	0.79	ND	ND	ND	0.93	<MQA	0.45	0.82

NON-IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	92.51	89.56	ND	ND	ND	ND	ND	ND	ND	ND	1.00	0.56	0.61	0.78
DAY 0 R2	100.24	97.60	ND	ND	ND	ND	ND	ND	ND	ND	0.98	0.48	0.50	0.68
DAY 3 R1	95.48	94.32	ND	ND	ND	ND	ND	ND	ND	ND	0.19	0.34	0.22	0.41
DAY 3 R2	96.53	95.31	ND	ND	ND	ND	ND	ND	ND	ND	0.28	0.23	0.15	0.55
DAY 7 R1	95.01	91.70	ND	ND	ND	ND	ND	ND	ND	ND	1.07	0.42	1.00	0.81
DAY 7 R2	93.12	90.88	ND	ND	ND	0.42	ND	ND	ND	ND	0.56	0.25	0.54	0.46
DAY 14 R1	89.53	86.43	ND	ND	ND	0.78	0.75	ND	ND	ND	0.44	<MQA	0.50	0.63
DAY 14 R2	93.57	91.25	ND	ND	ND	0.70	ND	ND	ND	ND	0.60	0.32	0.29	0.41
DAY 21 R1	92.63	90.38	ND	ND	ND	0.93	ND	ND	ND	ND	0.30	0.25	0.23	0.54
DAY 21 R2	93.13	91.42	ND	ND	ND	0.83	ND	ND	ND	ND	0.36	<MQA	0.24	0.28
DAY 30 R1	89.99	87.37	ND	ND	ND	ND	ND	ND	ND	ND	0.36	0.32	1.09	0.84
DAY 30 R2	93.12	83.03	ND	ND	ND	7.00	ND	ND	ND	ND	0.61	0.30	1.43	0.75

CALCULATION: $\frac{\text{DPM per Component}}{\text{DPM applied to TLC plate}} \times 100 =$ Percent of total Applied Radioaclivity per Component

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE IX: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 2, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-329351, EXTRACTION 2

CGA#		329351	42447	37734		62826								
IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	3.48	3.24	ND	ND	ND	ND	ND	ND	ND	ND	0.05	0.03	0.03	0.03
DAY 0 R2	3.14	2.87	ND	ND	ND	ND	ND	ND	ND	ND	0.06	0.01	0.02	0.03
DAY 3 R1	3.98	3.53	ND	ND	ND	ND	ND	ND	ND	ND	0.01	<MQA	0.01	0.03
DAY 3 R2	4.36	4.05	ND	ND	ND	ND	ND	ND	ND	ND	0.03	0.01	0.01	0.06
DAY 7 R1	4.81	4.34	ND	ND	ND	ND	ND	ND	ND	ND	0.05	< 0.01	0.05	0.05
DAY 7 R2	3.55	3.14	ND	ND	ND	ND	ND	ND	ND	ND	0.05	0.02	0.05	0.07
DAY 14 R1	4.20	3.56	ND	ND	ND	0.04	0.05	ND	ND	ND	0.04	0.01	0.02	0.07
DAY 14 R2	2.59	2.21	0.02	0.01	ND	0.02	0.03	ND	ND	ND	0.03	0.03	0.03	0.03
DAY 21 R1	3.42	2.74	0.03	0.01	ND	0.04	0.05	0.10	0.04	ND	0.04	0.01	0.03	0.03
DAY 21 R2	2.71	2.40	ND	ND	ND	0.02	0.04	ND	ND	ND	0.04	< 0.01	0.04	0.05
DAY 30 R1	5.46	4.72	0.07	0.03	ND	0.07	0.07	ND	ND	ND	0.22	0.05	0.06	0.05
DAY 30 R2	3.70	3.31	0.03	ND	ND	0.04	0.03	ND	ND	ND	0.03	<MQA	0.02	0.03

NON-IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	4.49	4.02	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.03	0.03	0.04
DAY 0 R2	4.18	4.08	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.02	0.02	0.03
DAY 3 R1	4.27	4.03	ND	ND	ND	ND	ND	ND	ND	ND	0.01	0.01	0.01	0.02
DAY 3 R2	6.76	6.44	ND	ND	ND	ND	ND	ND	ND	ND	0.02	0.02	0.01	0.04
DAY 7 R1	3.28	3.01	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.01	0.03	0.03
DAY 7 R2	3.02	2.74	ND	ND	ND	0.01	ND	ND	ND	ND	0.02	0.01	0.02	0.01
DAY 14 R1	4.19	3.62	ND	ND	ND	0.03	0.03	ND	ND	ND	0.02	<MQA	0.02	0.03
DAY 14 R2	3.93	3.59	ND	ND	ND	0.03	ND	ND	ND	ND	0.02	0.01	0.01	0.02
DAY 21 R1	2.79	2.52	ND	ND	ND	0.03	ND	ND	ND	ND	0.01	0.01	0.01	0.02
DAY 21 R2	3.25	2.97	ND	ND	ND	0.03	ND	ND	ND	ND	0.01	<MQA	0.01	0.01
DAY 30 R1	6.05	5.29	ND	ND	ND	ND	ND	ND	ND	ND	0.02	0.02	0.07	0.05
DAY 30 R2	6.32	5.25	ND	ND	ND	0.44	ND	ND	ND	ND	0.04	0.02	0.09	0.05

CALCULATION: $\frac{\text{\% of Total Recovered Radioactivity (Table VIII)}}{100}$ x % of Total Dose in Fraction = % Total dose per Component, Extraction 2

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE X: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 1 AND 2, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-329351, SUM OF EXTRACTION 1 AND 2

CGA#	329351	42447	37734		62826								
IRRADIATED	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	93.71	ND	ND	ND	ND	ND	ND	ND	ND	0.15	0.03	0.10	0.26
DAY 0 R2	91.70	ND	ND	ND	ND	ND	ND	ND	ND	0.19	0.01	0.12	0.08
DAY 3 R1	89.97	ND	ND	ND	ND	ND	ND	ND	ND	0.21	<MQA	0.53	0.42
DAY 3 R2	91.24	ND	ND	ND	ND	ND	ND	ND	ND	1.11	0.26	0.47	0.59
DAY 7 R1	95.28	0.23	ND	0.17	0.31	0.19	ND	ND	ND	0.52	0.39	0.59	0.66
DAY 7 R2	93.44	ND	ND	ND	0.01	0.23	ND	ND	ND	0.16	0.09	0.07	0.20
DAY 14 R1	85.52	0.40	ND	ND	0.04	0.38	ND	ND	ND	0.39	0.04	0.35	0.66
DAY 14 R2	87.38	0.02	0.01	ND	0.68	0.03	ND	ND	ND	0.39	0.19	0.41	0.93
DAY 21 R1	86.75	0.65	0.25	ND	0.61	0.34	0.10	0.04	0.31	0.44	0.09	0.44	0.49
DAY 21 R2	85.61	0.67	0.16	ND	0.35	0.27	ND	ND	ND	0.31	0.08	0.42	0.49
DAY 30 R1	85.53	1.15	0.03	ND	0.84	0.46	0.60	0.35	0.58	0.82	0.16	0.40	0.94
DAY 30 R2	87.30	0.52	ND	ND	0.50	0.03	ND	ND	ND	0.13	0.08	0.46	0.54

NON-IRRADIATED	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	91.46	ND	ND	ND	ND	ND	ND	ND	ND	1.82	0.10	0.12	0.21
DAY 0 R2	94.97	ND	ND	ND	ND	ND	ND	ND	ND	0.09	0.19	0.16	0.23
DAY 3 R1	94.08	ND	ND	ND	ND	ND	ND	ND	ND	0.38	0.13	0.33	0.28
DAY 3 R2	90.00	ND	ND	ND	ND	ND	ND	ND	ND	0.73	0.19	0.33	0.30
DAY 7 R1	89.99	ND	ND	ND	0.30	ND	ND	ND	ND	0.12	0.05	0.20	0.32
DAY 7 R2	93.57	0.31	ND	0.22	0.29	0.19	ND	ND	ND	0.15	0.05	0.23	0.47
DAY 14 R1	89.67	ND	ND	ND	0.68	0.03	ND	ND	ND	0.34	0.22	0.23	0.46
DAY 14 R2	90.05	ND	ND	ND	0.48	ND	ND	ND	ND	0.28	0.18	0.27	0.42
DAY 21 R1	94.01	ND	ND	ND	0.57	ND	ND	ND	ND	0.21	0.08	0.09	0.20
DAY 21 R2	95.65	ND	ND	ND	0.47	ND	ND	ND	ND	0.11	0.05	0.07	0.14
DAY 30 R1	89.87	ND	ND	ND	0.56	ND	ND	ND	ND	0.08	0.05	0.15	0.31
DAY 30 R2	87.21	ND	ND	ND	3.90	ND	ND	ND	ND	0.08	0.04	0.30	0.21

CALCULATION: % of Total Dose for Extraction 1(Table VII) + % of Total Dose for Extraction 2(Table IX) = % of Total Dose per Component for Extractions 1 and 2

ND = NOT DETECTED

<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XI: DISTRIBUTION OF RADIOACTIVITY IN CGA-329351, EXPERIMENT 1, EXTRACTION 1 AND 2, EXPRESSED AS PPM

CGA-329351, SUM OF EXTRACTION 1 AND 2

CGA#		329351	42447	37734		62826								
IRRADIATED	PPM	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	1.53	1.43	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 0 R2	1.53	1.40	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 3 R1	1.52	1.37	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<MQA	0.01	0.01
DAY 3 R2	1.56	1.42	ND	ND	ND	ND	ND	ND	ND	ND	0.02	<0.01	0.01	0.01
DAY 7 R1	1.52	1.45	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	0.01	0.01	0.01	0.01
DAY 7 R2	1.53	1.43	ND	ND	ND	<0.01	<0.01	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 14 R1	1.51	1.29	0.01	ND	ND	<0.01	0.01	ND	ND	ND	0.01	<0.01	0.01	0.01
DAY 14 R2	1.52	1.33	<0.01	<0.01	ND	0.01	<0.01	ND	ND	ND	0.01	0.00	0.01	0.01
DAY 21 R1	1.49	1.29	0.01	<0.01	ND	0.01	0.01	<0.01	<0.01	<0.01	0.01	<0.01	0.01	0.01
DAY 21 R2	1.52	1.30	0.01	<0.01	ND	0.01	<0.01	ND	ND	ND	<0.01	<0.01	0.01	0.01
DAY 30 R1	1.51	1.29	0.02	<0.01	ND	0.01	0.01	0.01	0.01	0.01	0.01	<0.01	0.01	0.01
DAY 30 R2	1.51	1.32	0.01	ND	ND	0.01	<0.01	ND	ND	ND	<0.01	<0.01	0.01	0.01

NON-IRRADIATED	PPM	A	B	C	D	F	G	I	J	K	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	1.52	1.39	ND	ND	ND	ND	ND	ND	ND	ND	0.03	<0.01	<0.01	<0.01
DAY 0 R2	1.53	1.45	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 3 R1	1.54	1.45	ND	ND	ND	ND	ND	ND	ND	ND	0.01	<0.01	0.01	<0.01
DAY 3 R2	1.51	1.36	ND	ND	ND	ND	ND	ND	ND	ND	0.01	<0.01	<0.01	<0.01
DAY 7 R1	1.52	1.37	ND	ND	ND	<0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 7 R2	1.50	1.40	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 14 R1	1.53	1.37	ND	ND	ND	0.01	<0.01	ND	ND	ND	0.01	<0.01	<0.01	0.01
DAY 14 R2	1.51	1.36	ND	ND	ND	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 21 R1	1.51	1.42	ND	ND	ND	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 21 R2	1.55	1.48	ND	ND	ND	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 30 R1	1.52	1.37	ND	ND	ND	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 30 R2	1.54	1.34	ND	ND	ND	0.06	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01

CALCULATION: $\frac{\text{\% of Total Dose per Component (Table X)}}{100} \times \text{PPM} = \text{PPM per Component for Extraction 1 and 2}$

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMAL QUANTIFIABLE AMOUNT

TABLE XII: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 1, EXPRESSED AS PERCENT OF TOTAL APPLIED RADIOACTIVITY RECOVERED FROM THE TLC PLATE

CGA-48988, EXTRACTION 1

CGA#		48988	42447	37734		62826									
IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	91.45	90.85	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.08	0.05	0.01	0.46
DAY 0 R2	92.73	91.31	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.18	0.17	0.30	0.77
DAY 3 R1	90.39	87.99	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.70	0.14	0.08	1.48
DAY 3 R2	93.36	91.78	0.58	0.48	ND	ND	ND	ND	ND	ND	ND	0.17	0.06	0.01	0.29
DAY 7 R1	97.03	94.16	0.94	0.72	ND	ND	ND	ND	ND	ND	ND	0.42	0.32	<MQA	0.47
DAY 7 R2	94.54	92.37	0.40	0.22	ND	0.42	0.39	ND	ND	ND	ND	0.18	0.03	0.27	0.25
DAY 14 R1	93.69	88.41	1.08	ND	ND	0.78	0.73	ND	ND	ND	ND	0.61	0.27	0.70	1.12
DAY 14 R2	90.17	85.52	0.85	0.48	ND	0.78	0.58	ND	ND	ND	ND	0.50	0.18	0.57	0.71
DAY 21 R1	91.20	87.23	0.93	0.32	ND	0.74	0.48	ND	ND	ND	ND	0.43	0.15	0.39	0.54
DAY 21 R2	90.10	85.73	1.13	0.46	ND	1.01	0.55	ND	ND	ND	ND	0.34	0.10	0.35	0.43
DAY 30 R1	99.15	92.64	ND	ND	ND	0.65	ND	2.42	0.38	1.17	ND	0.54	0.02	0.75	0.58
DAY 30 R2	90.56	84.64	1.17	0.51	ND	0.69	0.51	ND	ND	0.70	ND	0.56	0.19	0.69	0.89

NON-IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	91.24	90.27	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.04	<MQA	0.07	0.86
DAY 0 R2	98.88	97.64	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.11	0.07	0.42	0.64
DAY 3 R1	95.29	92.85	0.49	0.80	ND	ND	ND	ND	ND	ND	ND	0.35	0.29	0.15	0.35
DAY 3 R2	93.96	92.95	ND	ND	ND	0.21	0.25	0.04	ND	ND	ND	0.08	<MQA	0.09	0.34
DAY 7 R1	97.24	95.23	0.79	0.43	ND	ND	ND	ND	ND	ND	ND	0.26	0.06	0.12	0.35
DAY 7 R2	103.92	96.63	0.80	0.50	ND	ND	ND	ND	ND	ND	ND	1.56	0.92	2.19	1.32
DAY 14 R1	93.49	90.52	ND	ND	ND	0.99	0.50	ND	ND	ND	ND	0.34	0.19	0.30	0.64
DAY 14 R2	93.46	91.36	0.10	0.03	ND	0.61	0.41	ND	ND	ND	ND	0.33	0.12	0.29	0.21
DAY 21 R1	93.46	91.05	ND	ND	ND	1.09	0.42	ND	ND	ND	ND	0.20	0.08	0.22	0.39
DAY 21 R2	98.96	97.20	ND	0.10	ND	0.61	0.36	ND	ND	ND	ND	0.26	0.04	0.15	0.23
DAY 30 R1	93.01	86.26	1.29	0.73	ND	0.67	0.83	ND	ND	0.72	ND	0.67	0.26	0.57	1.01
DAY 30 R2	90.52	88.09	0.11	ND	ND	0.53	0.38	ND	ND	0.34	ND	0.30	0.09	0.21	0.47

CALCULATION: $\frac{\text{DPM per Component}}{\text{DPM applied to TLC plate}} \times 100 =$ Percent of total Applied Radioactivity per Component

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XIII: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 1, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-48988, EXTRACTION 1

CGA#		48988	42447	37734		62826									
IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	96.55	87.72	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.08	0.05	0.01	0.44
DAY 0 R2	94.13	85.95	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.17	0.16	0.28	0.72
DAY 3 R1	93.15	81.96	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.65	0.13	0.07	1.38
DAY 3 R2	92.26	84.68	0.54	0.44	ND	ND	ND	ND	ND	ND	ND	0.16	0.06	0.01	0.27
DAY 7 R1	92.24	86.85	0.87	0.66	ND	ND	ND	ND	ND	ND	ND	0.39	0.30	<MQA	0.43
DAY 7 R2	93.52	86.38	0.37	0.21	ND	0.39	0.36	ND	ND	ND	ND	0.17	0.03	0.25	0.23
DAY 14 R1	92.17	81.49	1.00	ND	ND	0.72	0.67	ND	ND	ND	ND	0.56	0.25	0.65	1.03
DAY 14 R2	93.67	80.11	0.80	0.45	ND	0.73	0.54	ND	ND	ND	ND	0.47	0.17	0.53	0.67
DAY 21 R1	92.24	80.46	0.86	0.30	ND	0.68	0.44	ND	ND	ND	ND	0.40	0.14	0.36	0.50
DAY 21 R2	93.84	80.45	1.06	0.43	ND	0.95	0.52	ND	ND	ND	ND	0.32	0.09	0.33	0.40
DAY 30 R1	91.36	84.64	ND	ND	ND	0.59	ND	2.21	0.35	1.07	ND	0.49	0.02	0.69	0.53
DAY 30 R2	92.04	77.90	1.08	0.47	ND	0.64	0.47	ND	ND	0.64	ND	0.52	0.17	0.64	0.82

NON-IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	94.41	85.22	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.04	<MQA	0.07	0.81
DAY 0 R2	95.17	92.92	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.10	0.07	0.40	0.61
DAY 3 R1	96.33	89.44	0.47	0.77	ND	ND	ND	ND	ND	ND	ND	0.34	0.28	0.14	0.34
DAY 3 R2	95.80	89.05	ND	ND	ND	0.20	0.24	0.04	ND	ND	ND	0.08	<MQA	0.09	0.33
DAY 7 R1	93.19	88.74	0.74	0.40	ND	ND	ND	ND	ND	ND	ND	0.24	0.06	0.11	0.33
DAY 7 R2	92.28	89.17	0.74	0.46	ND	ND	ND	ND	ND	ND	ND	1.44	0.85	2.02	1.22
DAY 14 R1	93.71	84.83	ND	ND	ND	0.93	0.47	ND	ND	ND	ND	0.32	0.18	0.28	0.60
DAY 14 R2	94.12	85.99	0.09	0.03	ND	0.57	0.39	ND	ND	ND	ND	0.31	0.11	0.27	0.20
DAY 21 R1	92.85	84.54	ND	ND	ND	1.01	0.39	ND	ND	ND	ND	0.19	0.07	0.20	0.36
DAY 21 R2	93.48	90.86	ND	0.09	ND	0.57	0.34	ND	ND	ND	ND	0.24	0.04	0.14	0.22
DAY 30 R1	92.82	80.07	1.20	0.68	ND	0.62	0.77	ND	ND	0.67	ND	0.62	0.24	0.53	0.94
DAY 30 R2	96.02	84.58	0.11	ND	ND	0.51	0.36	ND	ND	0.33	ND	0.29	0.09	0.20	0.45

CALCULATION: $\frac{\text{\% of Total Recovered Radioactivity (Table XII)}}{100}$ x % of Total Dose in Fraction = % Total dose per Component, Extraction 1

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XIV: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 2, EXPRESSED AS PERCENT OF TOTAL APPLIED RADIOACTIVITY RECOVERED FROM THE TLC PLATE

CGA-48988, EXTRACTION 2

CGA#		48988	42447	37734		62826									
IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	92.25	87.57	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.39	0.46	0.62	2.21
DAY 0 R2	97.28	93.31	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.17	1.07	0.34	1.39
DAY 3 R1	97.37	90.77	0.99	2.80	ND	ND	ND	ND	ND	ND	ND	1.85	0.34	0.27	0.34
DAY 3 R2	96.77	91.70	0.82	2.14	ND	ND	ND	ND	ND	ND	ND	1.09	0.27	0.18	0.57
DAY 7 R1	92.71	88.42	0.49	ND	0.45	1.12	ND	ND	ND	ND	ND	0.62	0.26	0.32	1.02
DAY 7 R2	93.79	86.95	ND	ND	ND	1.76	ND	ND	ND	ND	ND	1.32	0.56	0.76	2.44
DAY 14 R1	90.76	86.90	0.54	ND	ND	0.51	0.86	ND	ND	ND	ND	1.14	0.06	0.20	0.55
DAY 14 R2	95.25	90.51	ND	ND	ND	ND	0.95	ND	ND	ND	ND	1.22	0.06	0.91	1.61
DAY 21 R1	97.18	84.56	1.28	0.68	ND	1.93	1.23	0.65	ND	ND	ND	3.74	0.65	1.38	1.09
DAY 21 R2	95.46	86.85	ND	ND	ND	0.90	0.87	ND	ND	ND	ND	2.07	0.73	1.47	2.58
DAY 30 R1	90.98	69.57	2.15	0.98	ND	ND	5.69	ND	ND	ND	ND	6.28	0.49	4.09	1.72
DAY 30 R2	96.63	82.51	1.97	0.80	ND	ND	3.29	ND	ND	ND	ND	3.12	0.34	3.27	1.34

NON-IRRADIATED	% RECOVERY	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	93.77	90.02	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.89	0.66	0.66	1.53
DAY 0 R2	102.47	94.98	ND	ND	ND	ND	ND	ND	ND	ND	ND	2.06	1.58	1.31	2.54
DAY 3 R1	95.77	88.50	0.75	1.60	ND	ND	ND	ND	ND	ND	ND	1.06	0.89	1.06	1.91
DAY 3 R2	89.67	85.70	0.66	1.27	ND	ND	ND	ND	ND	ND	ND	0.48	0.20	0.52	0.84
DAY 7 R1	94.07	89.81	ND	ND	ND	1.23	ND	ND	ND	ND	ND	0.81	0.21	0.85	1.17
DAY 7 R2	96.27	91.20	ND	ND	ND	1.31	ND	ND	ND	ND	ND	0.77	0.54	0.89	1.56
DAY 14 R1	94.78	93.31	ND	ND	ND	0.53	ND	ND	ND	ND	ND	0.31	0.09	0.26	0.28
DAY 14 R2	94.59	88.95	ND	ND	ND	0.94	1.11	0.33	ND	ND	ND	1.22	0.44	1.09	0.50
DAY 21 R1	94.97	90.47	0.72	ND	ND	0.46	0.48	0.47	ND	ND	0.69	0.73	0.15	0.33	0.46
DAY 21 R2	93.31	90.20	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.56	<MQA	1.75	0.80
DAY 30 R1	92.40	87.42	ND	ND	ND	1.26	1.75	ND	ND	ND	ND	0.75	<MQA	0.31	0.92
DAY 30 R2	92.98	86.52	ND	ND	ND	1.16	2.04	ND	ND	ND	ND	0.91	0.30	0.91	1.16

CALCULATION: $\frac{\text{DPM per Component}}{\text{DPM applied to TLC plate}} \times 100 =$ Percent of total Applied Radioactivity per Component

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XV: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 2, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-48988, EXTRACTION 2

CGA#		48988	42447	37734		62826									
IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	2.65	2.32	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.01	0.02	0.06
DAY 0 R2	2.98	2.78	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.03	0.03	0.01	0.04
DAY 3 R1	3.13	2.84	0.03	0.09	ND	ND	ND	ND	ND	ND	ND	0.06	0.01	0.01	0.01
DAY 3 R2	3.08	2.82	0.03	0.07	ND	ND	ND	ND	ND	ND	ND	0.03	0.01	0.01	0.02
DAY 7 R1	6.80	6.01	0.03	ND	0.03	0.08	ND	ND	ND	ND	ND	0.04	0.02	0.02	0.07
DAY 7 R2	5.16	4.49	ND	ND	ND	0.09	ND	ND	ND	ND	ND	0.07	0.03	0.04	0.13
DAY 14 R1	3.85	3.35	0.02	ND	ND	0.02	0.03	ND	ND	ND	ND	0.04	< 0.01	0.01	0.02
DAY 14 R2	5.33	4.82	ND	ND	ND	ND	0.05	ND	ND	ND	ND	0.07	< 0.01	0.05	0.09
DAY 21 R1	4.82	4.08	0.06	0.03	ND	0.09	0.06	0.03	ND	ND	ND	< 0.01	0.18	0.03	0.07
DAY 21 R2	4.68	4.06	ND	ND	ND	0.04	0.04	ND	ND	ND	ND	0.10	0.03	0.07	0.12
DAY 30 R1	1.63	1.13	0.04	0.02	ND	ND	0.09	ND	ND	ND	ND	0.10	0.01	0.07	0.03
DAY 30 R2	4.49	3.70	0.09	0.04	ND	ND	0.15	ND	ND	ND	ND	0.14	0.02	0.15	0.06

NON-IRRADIATED	% FRACTION	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	4.74	4.27	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.03	0.03	0.07
DAY 0 R2	2.04	1.94	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.04	0.03	0.03	0.05
DAY 3 R1	3.76	3.33	0.03	0.06	ND	ND	ND	ND	ND	ND	ND	0.04	0.03	0.04	0.07
DAY 3 R2	3.68	3.15	0.02	0.05	ND	ND	ND	ND	ND	ND	ND	0.02	0.01	0.02	0.03
DAY 7 R1	5.40	4.85	ND	ND	ND	0.07	ND	ND	ND	ND	ND	0.04	0.01	0.05	0.06
DAY 7 R2	5.95	5.43	ND	ND	ND	0.08	ND	ND	ND	ND	ND	0.05	0.03	0.05	0.09
DAY 14 R1	5.69	5.31	ND	ND	ND	0.03	ND	ND	ND	ND	ND	0.02	0.01	0.01	0.02
DAY 14 R2	5.03	4.47	ND	ND	ND	0.05	0.06	0.02	ND	ND	ND	0.06	0.02	0.05	0.03
DAY 21 R1	5.18	4.38	0.07	ND	ND	0.10	0.06	0.03	ND	ND	0.04	0.19	0.03	0.07	0.06
DAY 21 R2	5.52	4.98	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.03	<MQA	0.10	0.04
DAY 30 R1	5.69	4.97	ND	ND	ND	0.07	0.10	ND	ND	ND	ND	0.04	<MQA	0.02	0.05
DAY 30 R2	3.85	3.33	ND	ND	ND	0.04	0.08	ND	ND	ND	ND	0.04	0.01	0.04	0.04

CALCULATION: $\frac{\text{\% of Total Recovered Radioactivity (Table XIV)}}{100}$ x % of Total Dose in Fraction = % Total dose per Component, Extraction 2

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XVI: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 1 AND 2, EXPRESSED AS PERCENT OF TOTAL DOSE

CGA-48988, SUM OF EXTRACTION 1 AND 2

CGA#	48988	42447	37734		62826									
IRRADIATED	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	90.04	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.12	0.06	0.03	0.50
DAY 0 R2	88.73	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.20	0.19	0.29	0.76
DAY 3 R1	84.80	0.03	0.09	ND	ND	ND	ND	ND	ND	ND	0.71	0.14	0.08	1.39
DAY 3 R2	87.50	0.57	0.51	ND	ND	ND	ND	ND	ND	ND	0.19	0.07	0.02	0.29
DAY 7 R1	92.86	0.90	0.66	0.03	0.08	ND	ND	ND	ND	ND	0.43	0.32	0.02	0.50
DAY 7 R2	90.87	0.37	0.21	ND	0.48	0.36	ND	ND	ND	ND	0.24	0.06	0.29	0.36
DAY 14 R1	84.84	1.02	ND	ND	0.74	0.70	ND	ND	ND	ND	0.60	0.25	0.66	1.05
DAY 14 R2	84.93	0.80	0.45	ND	0.73	0.59	ND	ND	ND	ND	0.54	0.17	0.58	0.76
DAY 21 R1	84.54	0.92	0.33	ND	0.77	0.50	0.03	ND	ND	ND	0.40	0.32	0.39	0.57
DAY 21 R2	84.51	1.06	0.43	ND	0.99	0.56	ND	ND	ND	ND	0.42	0.12	0.40	0.52
DAY 30 R1	85.77	0.04	0.02	ND	0.59	0.09	2.21	0.35	1.07	ND	0.59	0.03	0.76	0.56
DAY 30 R2	81.60	1.17	0.51	ND	0.64	0.62	ND	ND	0.64	ND	0.66	0.19	0.79	0.88

NON-IRRADIATED	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	89.49	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.08	0.03	0.10	0.88
DAY 0 R2	94.86	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.14	0.10	0.43	0.66
DAY 3 R1	92.77	0.50	0.83	ND	ND	ND	ND	ND	ND	ND	0.38	0.31	0.18	0.41
DAY 3 R2	92.20	0.02	0.05	ND	0.20	0.24	0.04	ND	ND	ND	0.10	0.01	0.11	0.36
DAY 7 R1	93.59	0.74	0.40	ND	0.07	ND	ND	ND	ND	ND	0.28	0.07	0.16	0.39
DAY 7 R2	94.60	0.74	0.46	ND	0.08	ND	ND	ND	ND	ND	1.49	0.88	2.07	1.31
DAY 14 R1	90.14	ND	ND	ND	0.96	0.47	ND	ND	ND	ND	0.34	0.19	0.29	0.62
DAY 14 R2	90.46	0.09	0.03	ND	0.62	0.45	0.02	ND	ND	ND	0.37	0.13	0.32	0.23
DAY 21 R1	88.92	0.07	ND	ND	1.11	0.45	0.03	ND	ND	0.04	0.38	0.10	0.27	0.42
DAY 21 R2	95.84	ND	0.09	ND	0.57	0.34	ND	ND	ND	ND	0.27	0.04	0.24	0.26
DAY 30 R1	85.04	1.20	0.68	ND	0.69	0.87	ND	ND	0.67	ND	0.66	0.24	0.55	0.99
DAY 30 R2	87.91	0.11	ND	ND	0.55	0.44	ND	ND	0.33	ND	0.33	0.10	0.24	0.49

CALCULATION: % of Total Dose for Extraction 1(Table XIII) + % of Total Dose for Extraction 2(Table XV) = % of Total Dose per Component for Extractions 1 and 2 (calculation does correct for % TLC recovery)

ND = NOT DETECTED
<MQA = LESS THAN THE MINIMUM QUANTIFIABLE AMOUNT

TABLE XVII: DISTRIBUTION OF RADIOACTIVITY IN CGA-48988, EXPERIMENT 2, EXTRACTION 1 AND 2, EXPRESSED AS PPM

CGA-48988, SUM OF EXTRACTION 1 AND EXTRACTION 2

CGA#		48988	42447	37734		62826									
IRRADIATED	PPM	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	1.53	1.38	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 0 R2	1.52	1.35	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 3 R1	1.51	1.28	<0.01	<0.01	ND	ND	ND	ND	ND	ND	ND	0.01	<0.01	<0.01	0.02
DAY 3 R2	1.51	1.32	0.01	0.01	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 7 R1	1.51	1.40	0.01	0.01	<0.01	<0.01	ND	ND	ND	ND	ND	0.01	<0.01	<0.01	0.01
DAY 7 R2	1.51	1.37	0.01	<0.01	ND	0.01	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 14 R1	1.52	1.29	0.02	ND	ND	0.01	0.01	ND	ND	ND	ND	0.01	<0.01	0.01	0.02
DAY 14 R2	1.51	1.28	0.01	0.01	ND	0.01	0.01	ND	ND	ND	ND	0.01	<0.01	0.01	0.01
DAY 21 R1	1.50	1.27	0.01	<0.01	ND	0.01	0.01	<0.01	ND	ND	ND	0.01	<0.01	0.01	0.01
DAY 21 R2	1.52	1.28	0.02	0.01	ND	0.02	0.01	ND	ND	ND	ND	0.01	<0.01	0.01	0.01
DAY 30 R1	1.52	1.30	<0.01	<0.01	ND	0.01	<0.01	0.03	0.01	0.02	ND	0.01	<0.01	0.01	0.01
DAY 30 R2	1.52	1.24	0.02	0.01	ND	0.01	0.01	ND	ND	0.01	ND	0.01	<0.01	0.01	0.01

NON-IRRADIATED	PPM	A	B	C	D	F	G	I	J	K	L	QUAD I	QUAD II	QUAD III	QUAD IV
DAY 0 R1	1.53	1.37	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 0 R2	1.52	1.44	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.01	<0.01	0.01	0.01
DAY 3 R1	1.51	1.40	0.01	0.01	ND	ND	ND	ND	ND	ND	ND	0.01	<0.01	<0.01	0.01
DAY 3 R2	1.51	1.39	<0.01	<0.01	ND	<0.01	<0.01	<0.01	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 7 R1	1.51	1.41	0.01	0.01	ND	<0.01	ND	ND	ND	ND	ND	<0.01	<0.01	<0.01	0.01
DAY 7 R2	1.51	1.43	0.01	0.01	ND	<0.01	ND	ND	ND	ND	ND	0.02	0.01	0.03	0.02
DAY 14 R1	1.55	1.40	ND	ND	ND	0.01	0.01	ND	ND	ND	ND	0.01	<0.01	<0.01	0.01
DAY 14 R2	1.53	1.38	<0.01	<0.01	ND	0.01	0.01	<0.01	ND	ND	ND	0.01	<0.01	<0.01	<0.01
DAY 21 R1	1.51	1.34	<0.01	ND	ND	0.02	0.01	<0.01	ND	ND	<0.01	0.01	<0.01	<0.01	0.01
DAY 21 R2	1.52	1.46	ND	<0.01	ND	0.01	0.01	ND	ND	ND	ND	<0.01	<0.01	<0.01	<0.01
DAY 30 R1	1.40	1.19	0.02	<0.01	ND	0.01	0.01	ND	ND	0.01	ND	0.01	<0.01	0.01	0.01
DAY 30 R2	1.50	1.32	<0.01	ND	ND	0.01	0.01	ND	ND	<0.01	ND	<0.01	<0.01	<0.01	0.01

CALCULATION: $\frac{\text{\% of Total Dose per Component (Table XVI)}}{100} \times \text{PPM} = \text{PPM per Component for Extraction 1 and 2}$

ND = NOT DETECTED

TABLE XVIII: RESULTS OF THE BIOMASS AND VIABILITY ASSAYS FOR CGA-329351 AND CGA-48988

RESULTS OF THE BIOMASS ANALYSIS

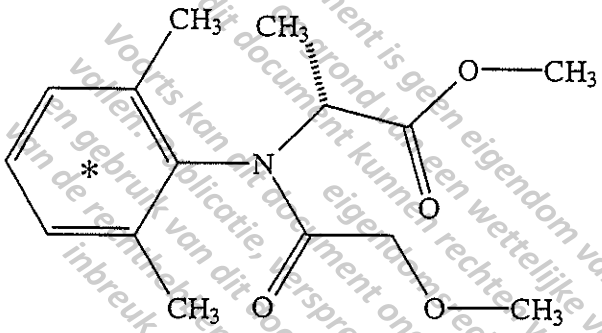
Replicate #	mL CO2 Efflux	g Dry Soil	Biomass (mg C/kg Soil)
1	0.0412	22.76	88.737
2	0.0343	22.79	76.497
3	0.0370	22.73	81.455

Average Biomass	82.230

RESULTS OF THE VIABILITY ANALYSIS

	PRE-DOSE	IRRADIATED DAY 30, CGA-48988	NON-IRRADIATED DAY 30, CGA-48988
DATE STREAKED	3/27/95	5/5/95	5/5/95
ROSE BENGAL CFU/g	6.03E+03	0	0
DAYS OF INCUBATION	14	14	14
ACTINOMYCETE CFU/g	8.79E+05	4.98E+06	1.17E+06
DAYS OF INCUBATION	14	14	14
PLATE COUNT CFU/g	1.03E+06	3.11E+06	4.63E+06
DAYS OF INCUBATION	3	3	3

ACTINOMYCETE PLATES REFLECT ACTINOMYCETES AND BACTERIA IN MOST CASES.



* denotes radiolabel

Ciba Name: ^{14}C-CGA-329351

CAS Name: D-Alanine, N-(2,6-Dimethylphenyl)-N-(methoxyacetyl)-, methyl ester

CAS No.: 70630-17-0

Molecular Formula/Weight: $C_{15}H_{21}NO_4$/279.34

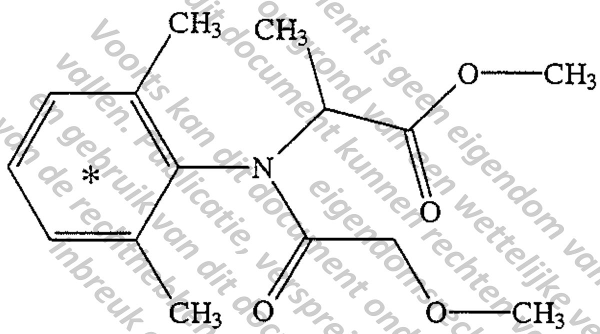
Physical State: Viscous Liquid at 20°C

Water Solubility: 26 g/L at 20°C

Vapor Pressure: 5.2×10^{-3} Pa at 25°C

Octanol/Water Partition Coefficient: Log P = 1.53
(flask method)

FIGURE 1. STRUCTURE AND PHYSICAL CHARACTERISTICS OF CGA-329351



* denotes radiolabel

Ciba Name: ^{14}C-CGA-48988

CAS Name: DL-Alanine, N-(2,6-Dimethylphenyl)-N-(methoxyacetyl)-, methyl ester

CAS No.: 57837-19-1

Molecular Formula/Weight: $C_{15}H_{21}NO_4$/279.34

Physical State: Crystalline Solid

Water Solubility: 0.7% at 20°C

Vapor Pressure: 2.2 x 10^{-6} Torr at 20°C

FIGURE 2. STRUCTURE AND PHYSICAL CHARACTERISTICS OF CGA-48988

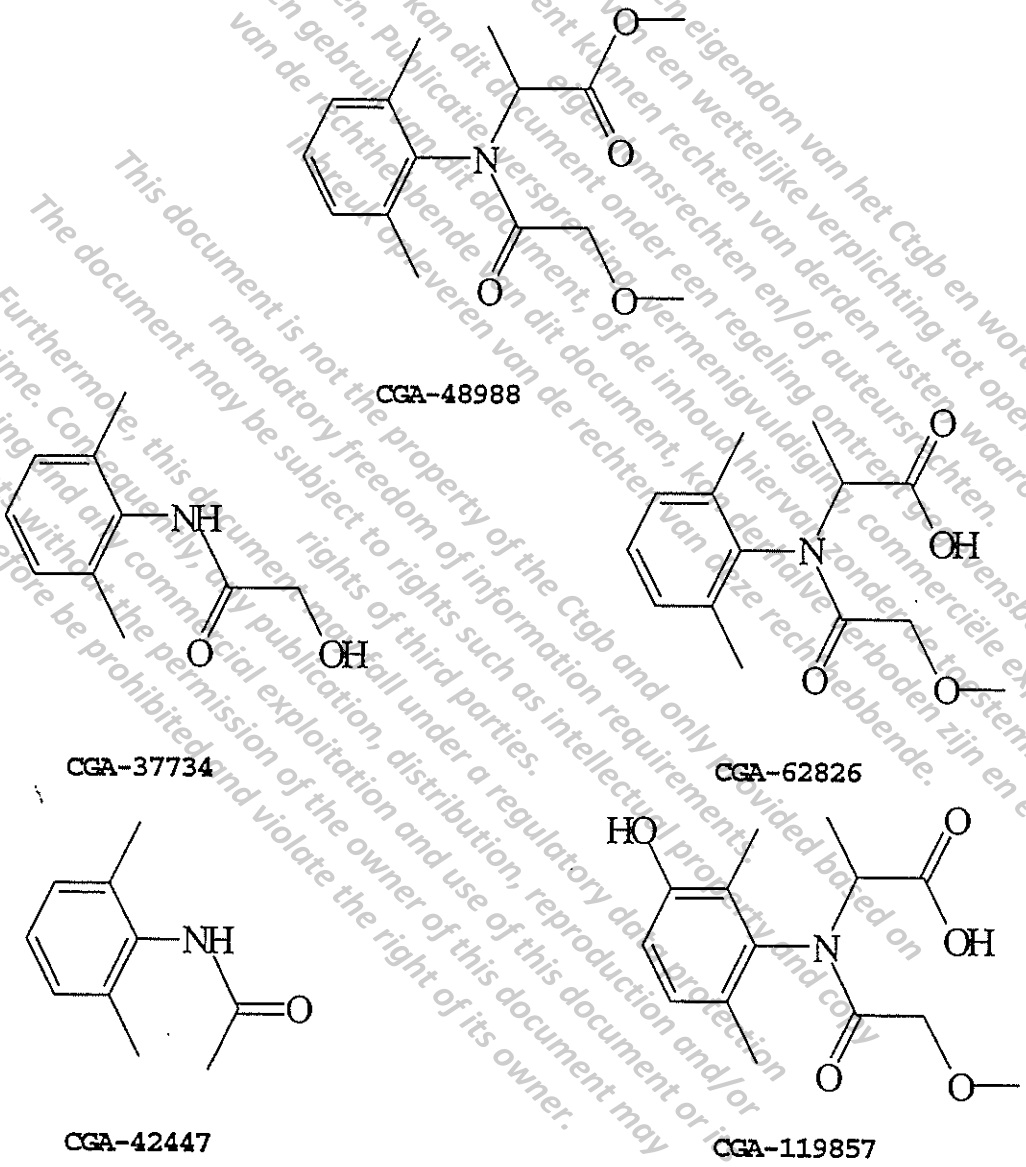
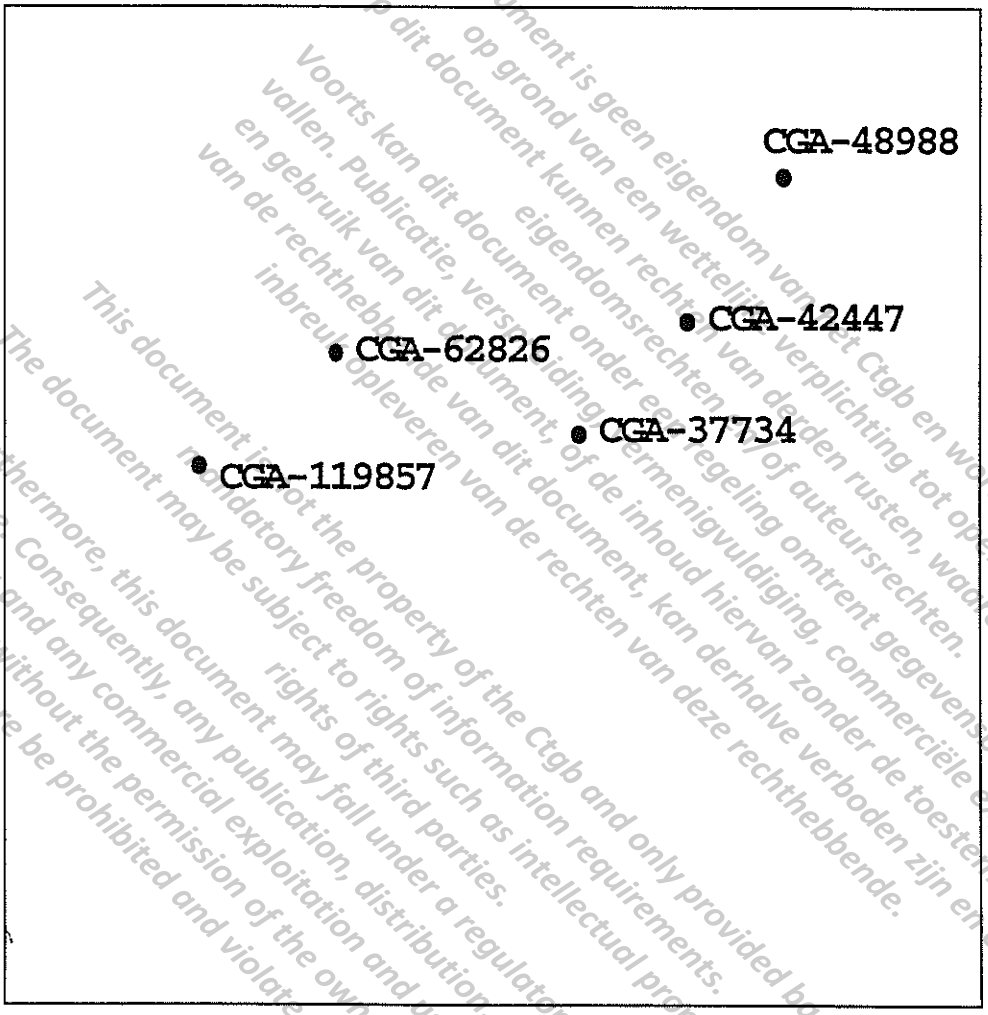


FIGURE 3: STRUCTURES AND NOMENCLATURE FOR REFERENCE STANDARDS



SOLVENT SYSTEM I (↑): CHLOROFORM:METHANOL:FORMIC ACID:WATER (75:20:4:2 V/V/V/V)

SOLVENT SYSTEM II (→): CHLOROFORM:METHANOL: AMMONIUM HYDROXIDE:WATER (80:30:4:2 V/V/V/V)

FIGURE 4: REPRESENTATIVE TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF THE REFERENCE STANDARDS IN SOLVENT SYSTEMS I AND II

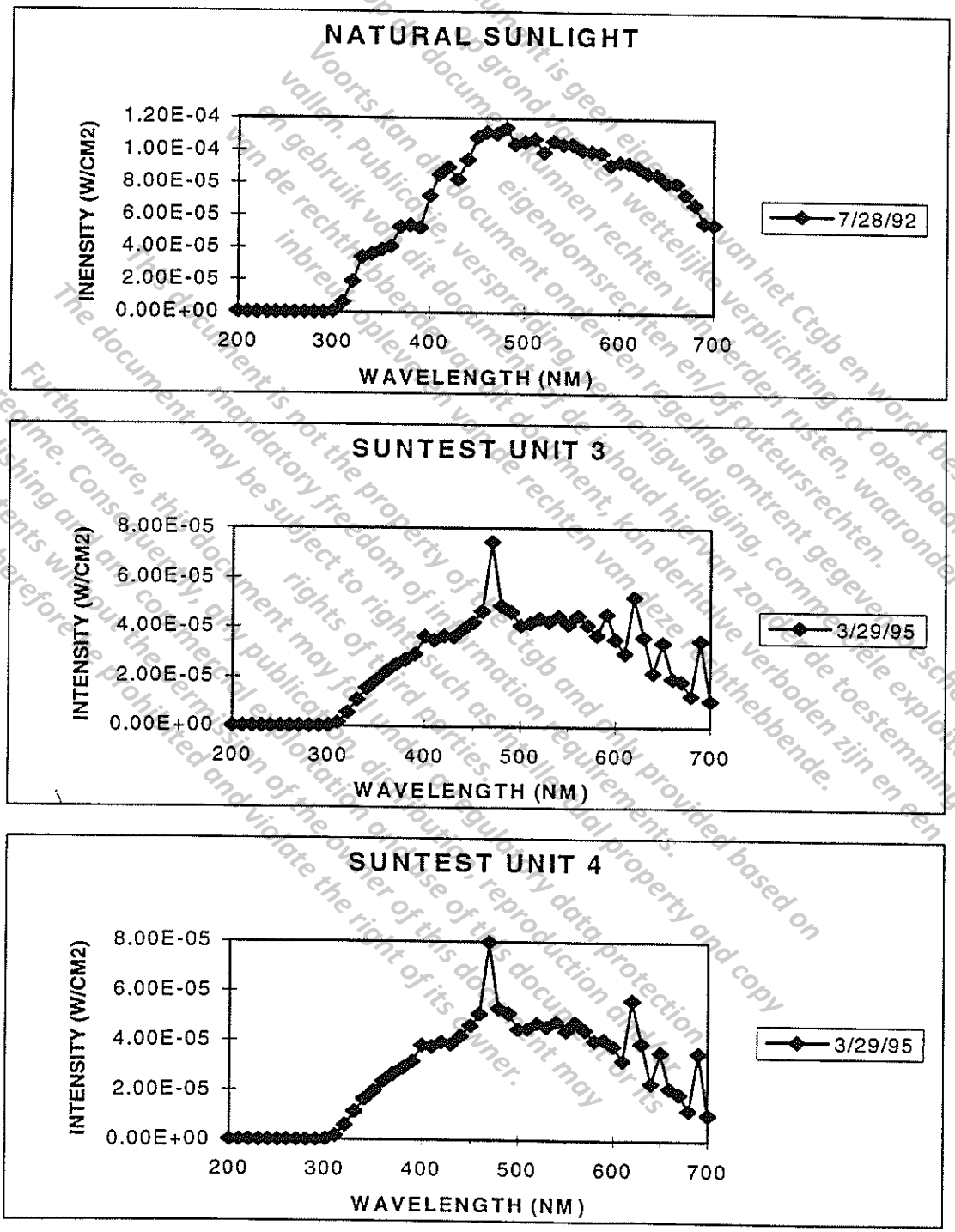


FIGURE 5: NATURAL AND ARTIFICIAL LIGHT SPECTRAL DISTRIBUTIONS

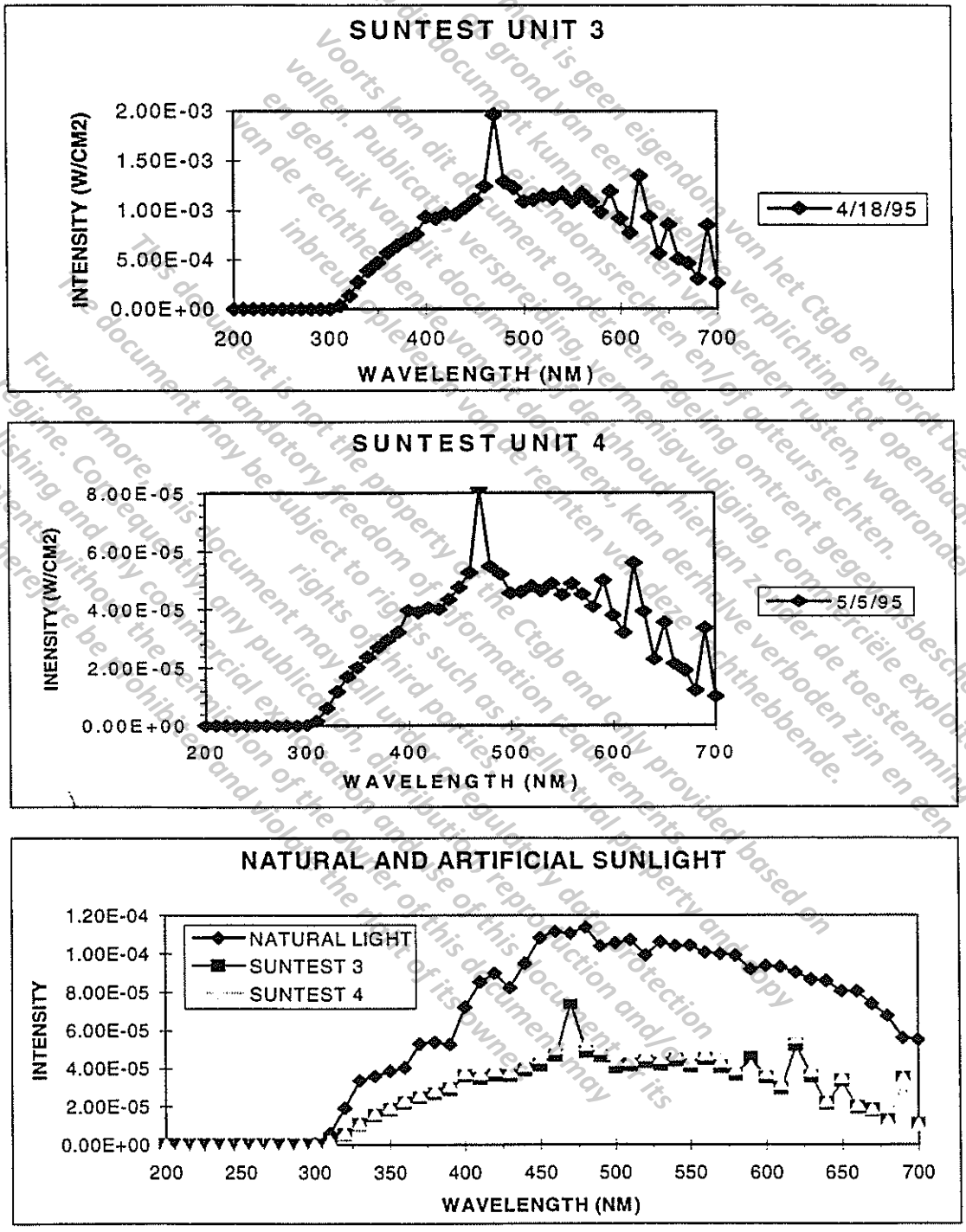
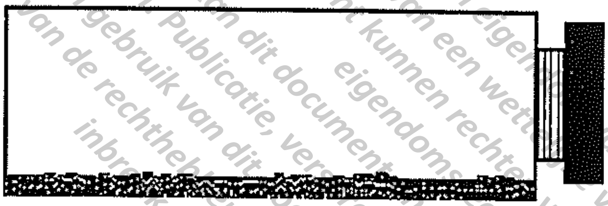
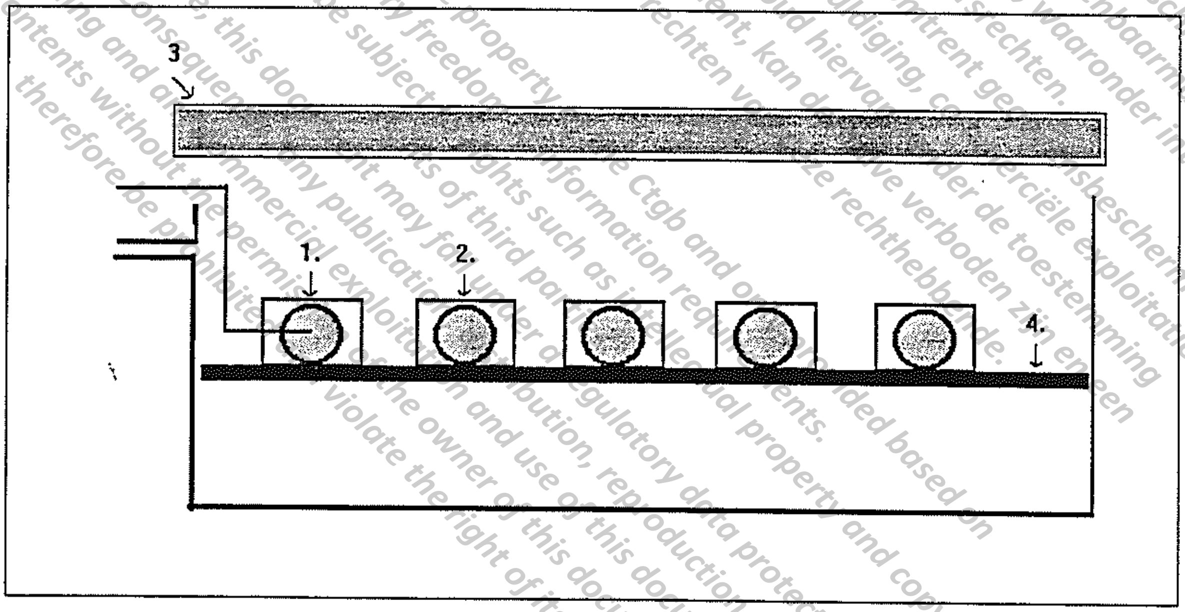


FIGURE 5: NATURAL AND ARTIFICIAL SPECTRAL DISTRIBUTIONS
(Continued)

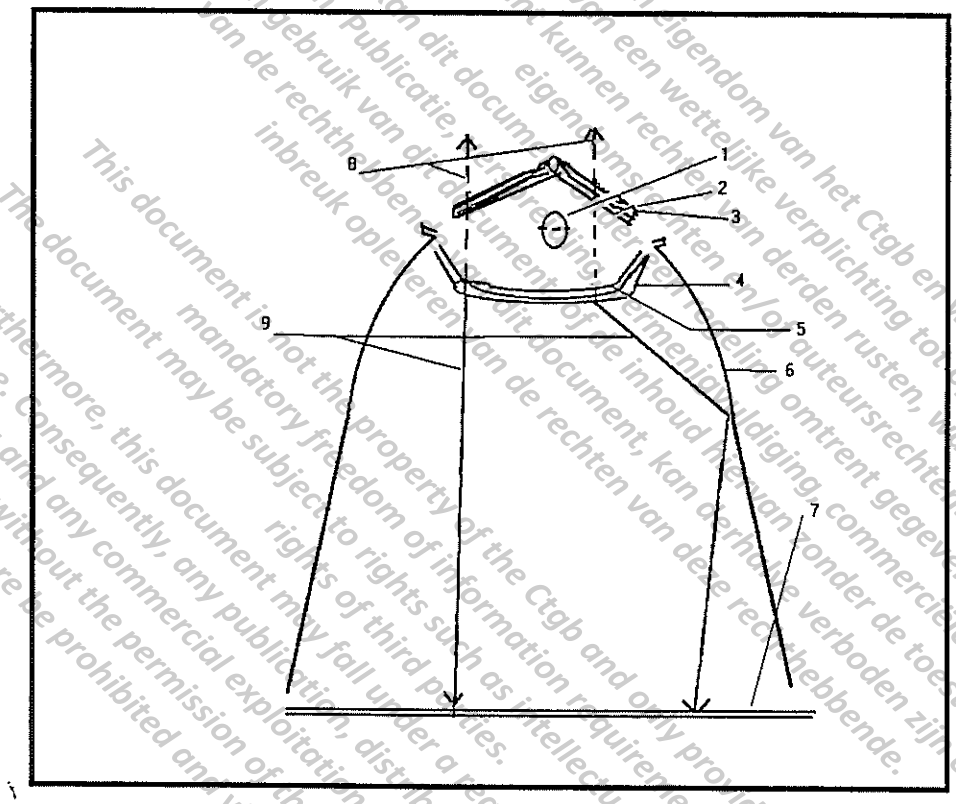


SAMPLE VIAL WITH SOIL



1. SURROGATE VIAL WITH THERMOCOUPLE PROBE
2. EXAMPLE OF SAMPLE VIAL WITH LEAD WEIGHT
3. PYREX GLASS PLATE
4. SUPPORT RACK

FIGURE 6: DIAGRAM OF VIAL WITH SOIL AND THE WATER BATH FOR THE IRRADIATED INCUBATIONS (SIDE VIEW)



1. Xenon Arc lamp
2. Ultra-violet mirror
3. Light mirror
4. Quartz glass
5. UV special glass filter (290 nm cutoff)
6. Reflector
7. Test material location level
8. Infra-red radiation
9. UV and visible light

FIGURE 7. DIAGRAM OF XENON ARC LAMP AND EXPOSURE CHAMBER

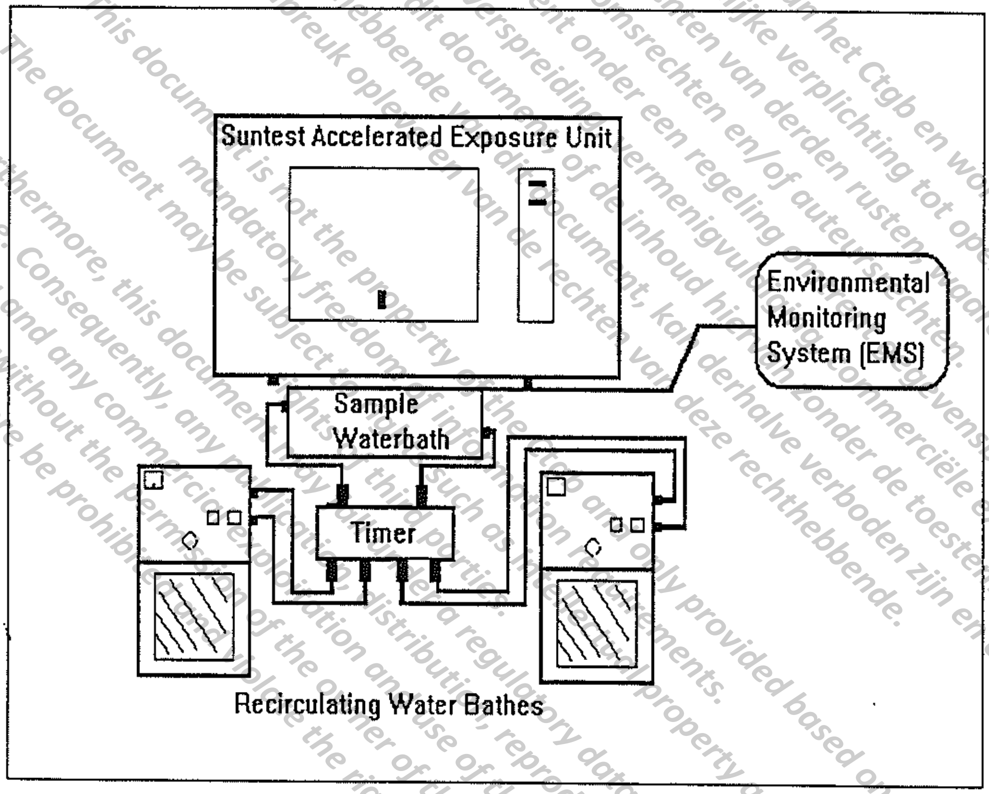
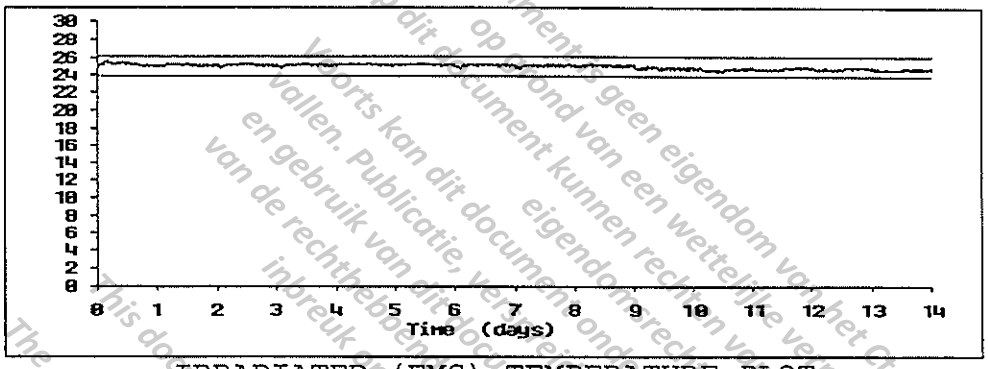
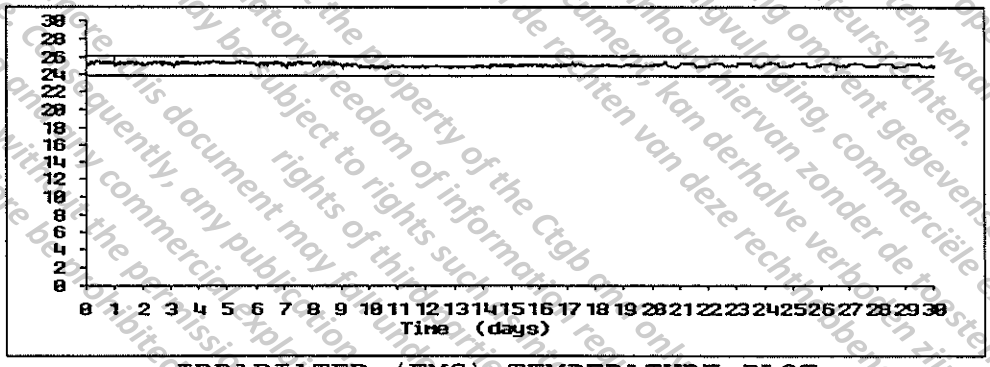


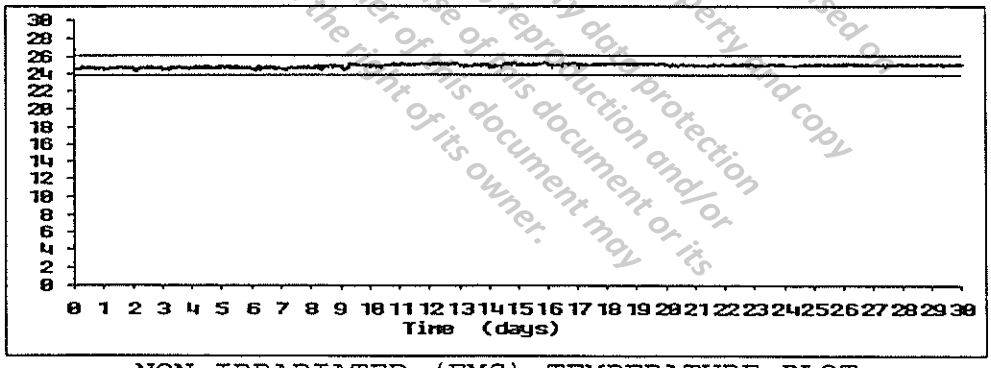
FIGURE 8: DIAGRAM OF THE COMPLETE PHOTOLYSIS TEST SYSTEM



IRRADIATED (EMS) TEMPERATURE PLOT
SUNTEST UNIT 3 TEMPERATURE PLOT
APRIL 4, 1995 THROUGH APRIL 18, 1995



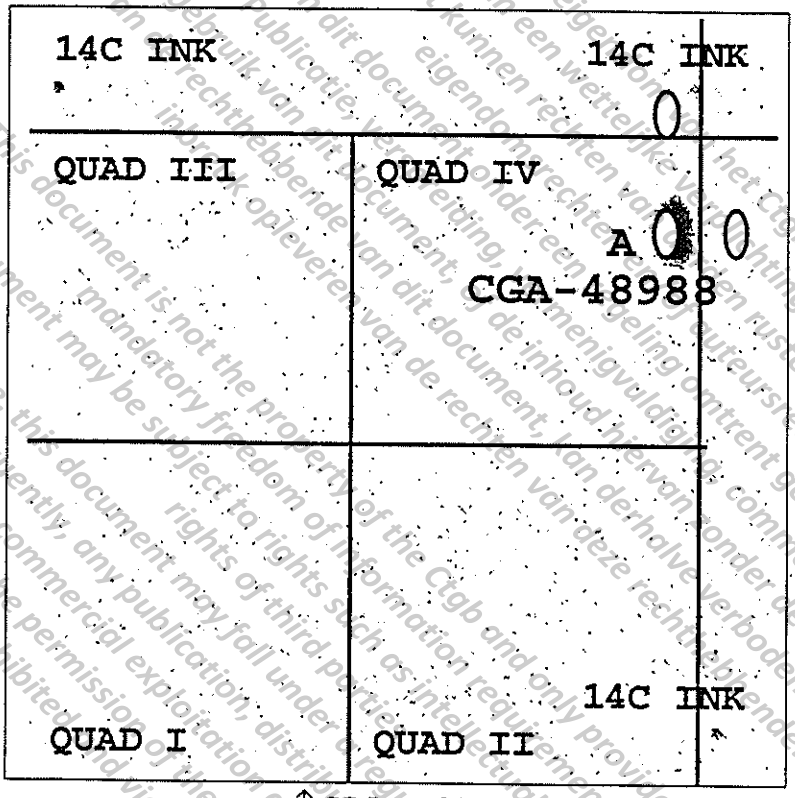
IRRADIATED (EMS) TEMPERATURE PLOT
SUNTEST UNIT 4 TEMPERATURE PLOT
APRIL 4, 1995 THROUGH MAY 4, 1995



NON-IRRADIATED (EMS) TEMPERATURE PLOT
APRIL 4, 1995 THROUGH MAY 4, 1995

FIGURE 9: TEMPERATURE PLOTS FOR THE IRRADIATED AND NON-IRRADIATED CGA-329351 AND CGA-48988 INCUBATIONS

CGA-329351 TLC PURITY

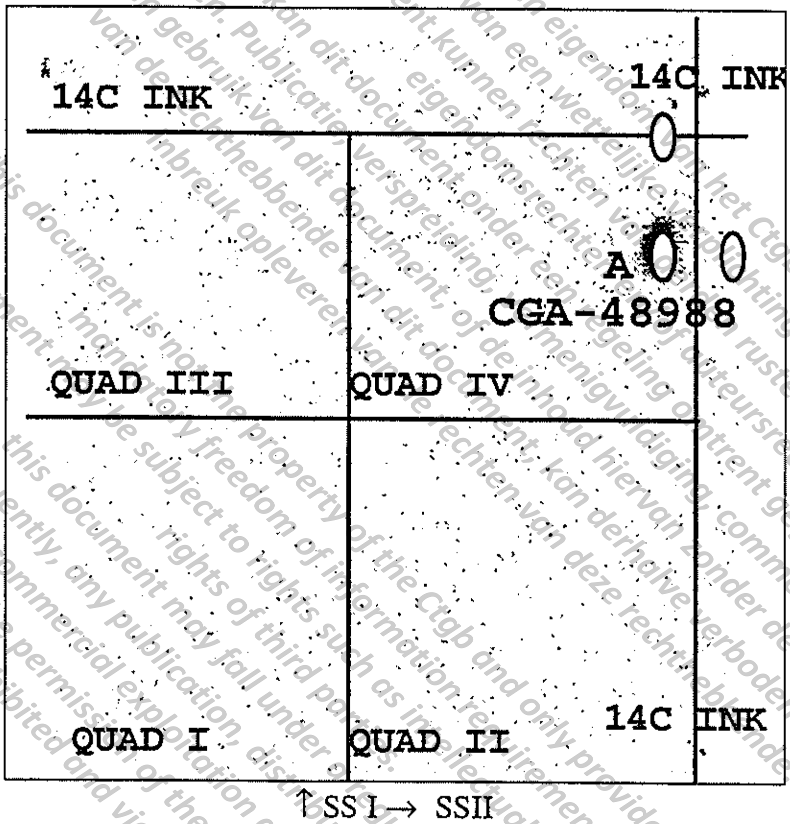


AREA	PERCENT
A	99.87
QUAD I	0.01
QUAD II	0.01
QUAD III	0.04
QUAD IV	0.07

PERCENT EQUALS THE PERCENT RECOVERED FROM THE TLC PLATE

FIGURE 10: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF CGA-329351 FOR PURITY ANALYSIS

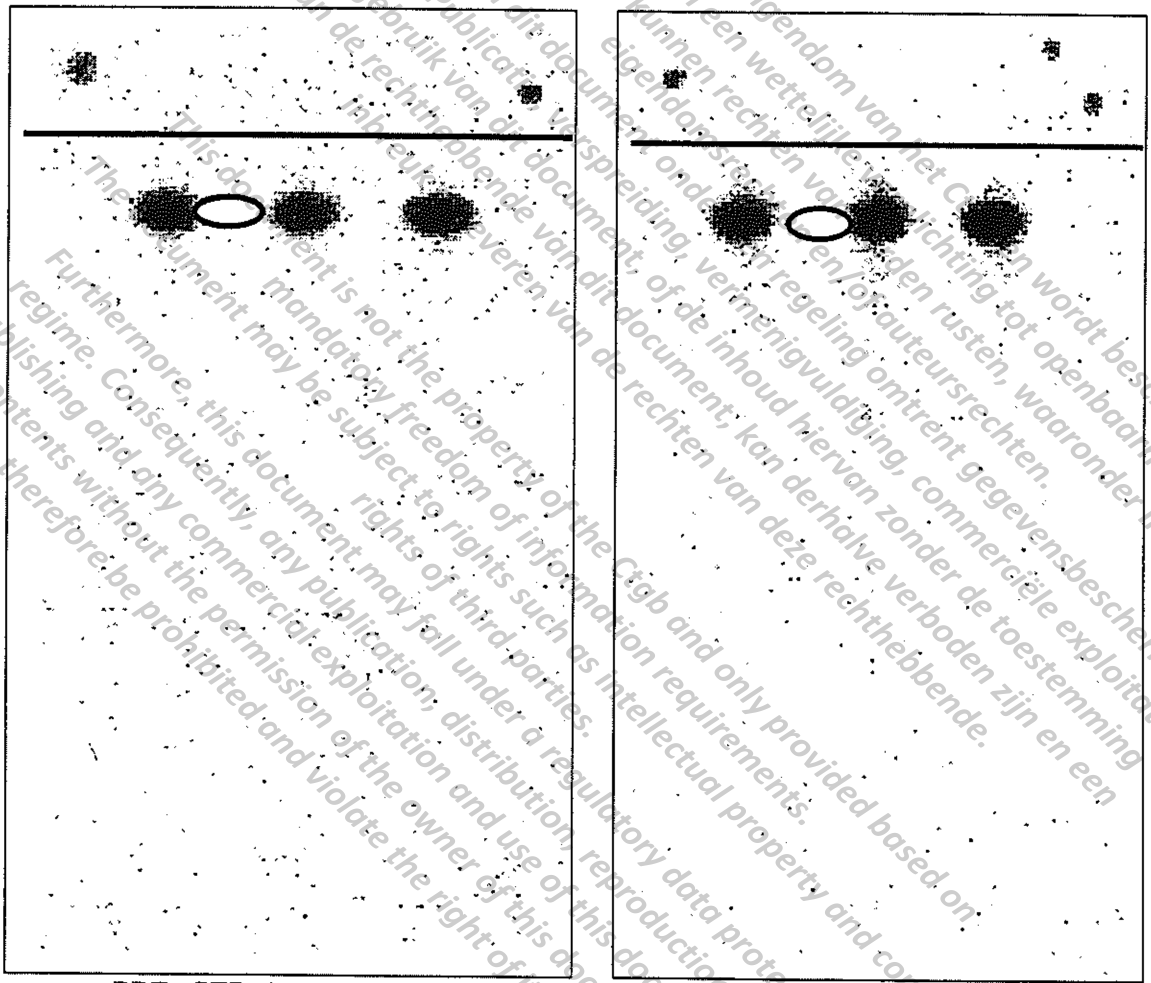
CGA-48988 TLC PURITY



AREA	PERCENT
A	99.43
QUAD I	0.03
QUAD II	0.02
QUAD III	0.20
QUAD IV	0.32

PERCENT EQUALS THE PERCENT RECOVERED FROM THE TLC PLATE

FIGURE 11: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF CGA-48988 FOR PURITY ANALYSIS



PRE STD MID POST
CGA-329351

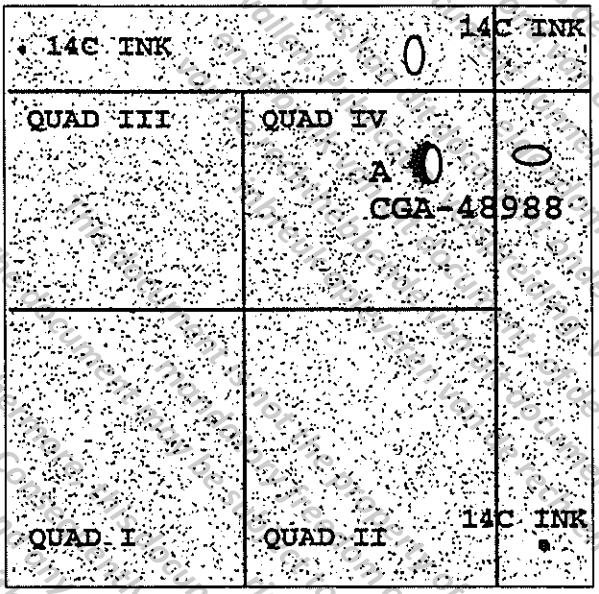
PRE STD MID POST
CGA-48988

PRE = PRE DOSE
STD = STANDARD CGA-48988
MID = MIDDLE OF DOSE
POST = POST DOSE

FIGURE 12: STABILITY OF THE DOSE SOLUTIONS BY TLC

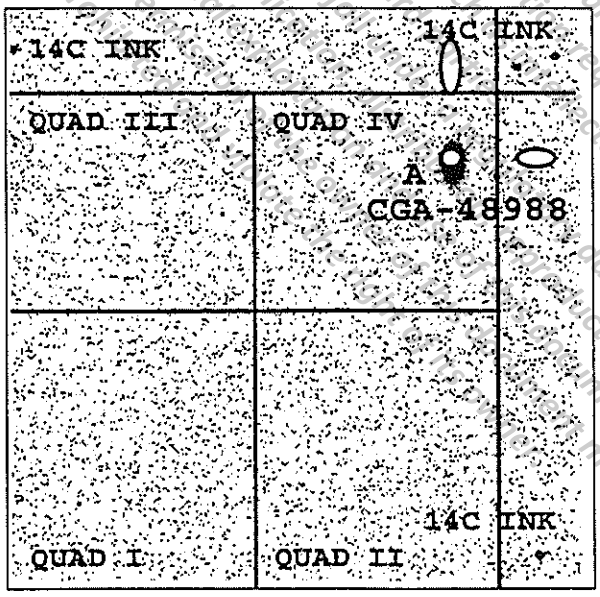
CGA-329351 EXTRACTION 1

DAY 0 R1 IRRADIATED



AREA	PERCENT
A	90.47
QUAD I	0.10
QUAD II	<MQA
QUAD III	0.07
QUAD IV	0.23

DAY 0 R2 IRRADIATED



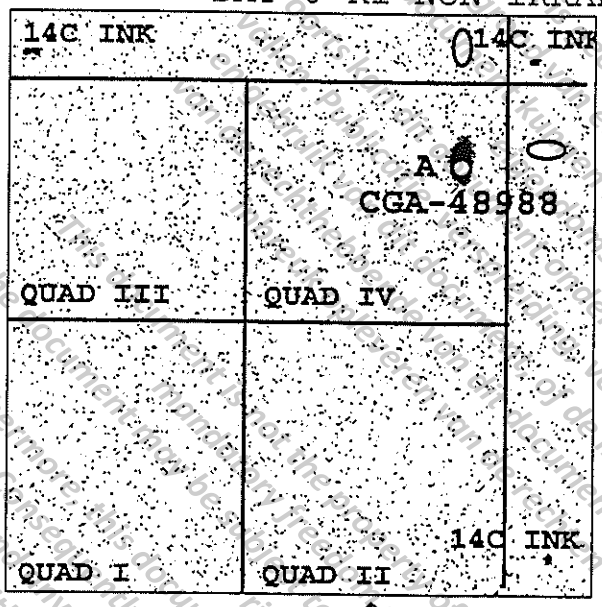
AREA	PERCENT
A	88.83
QUAD I	0.13
QUAD II	< MQA
QUAD III	0.10
QUAD IV	0.05

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 13: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

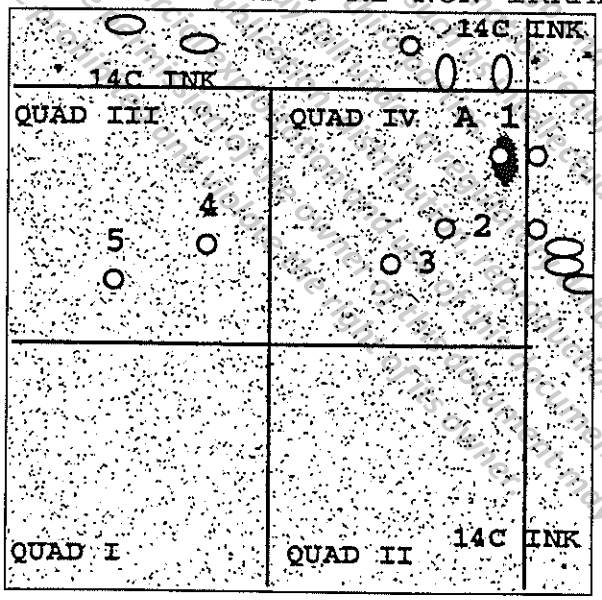
CGA-329351 EXTRACTION 1

DAY 0 R1 NON-IRRADIATED



AREA	PERCENT
A	87.44
QUAD I	1.78
QUAD II	0.07
QUAD III	0.09
QUAD IV	0.17

DAY 0 R2 NON-IRRADIATED



AREA	PERCENT
A	90.89
QUAD I	0.05
QUAD II	0.17
QUAD III	0.14
QUAD IV	0.20

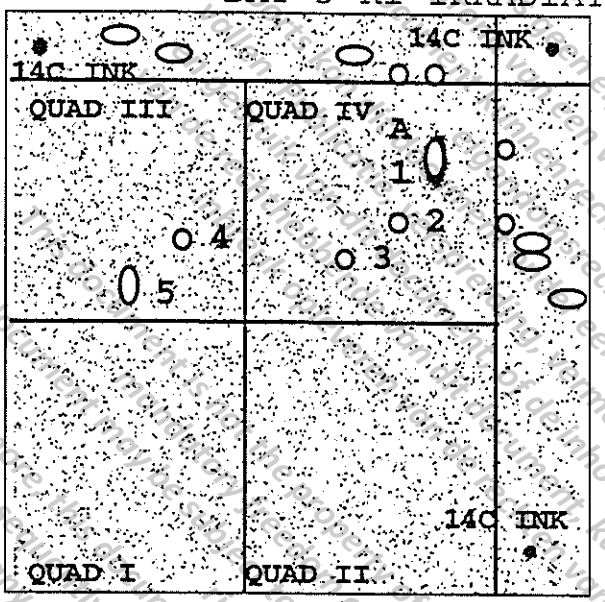
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 14: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 1

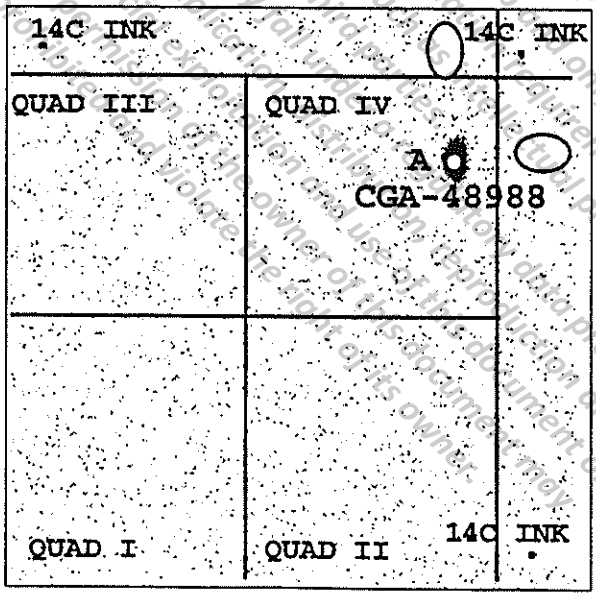
DAY 3 R1 IRRADIATED



AREA	PERCENT
A	86.44
QUAD I	0.20
QUAD II	< MQA
QUAD III	0.52
QUAD IV	0.39

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 3 R2 IRRADIATED



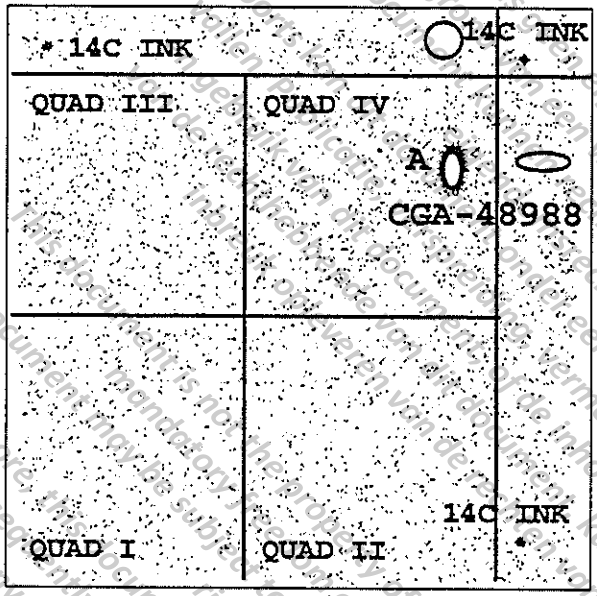
AREA	PERCENT
A	87.19
QUAD I	1.08
QUAD II	0.25
QUAD III	0.46
QUAD IV	0.53

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 15: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

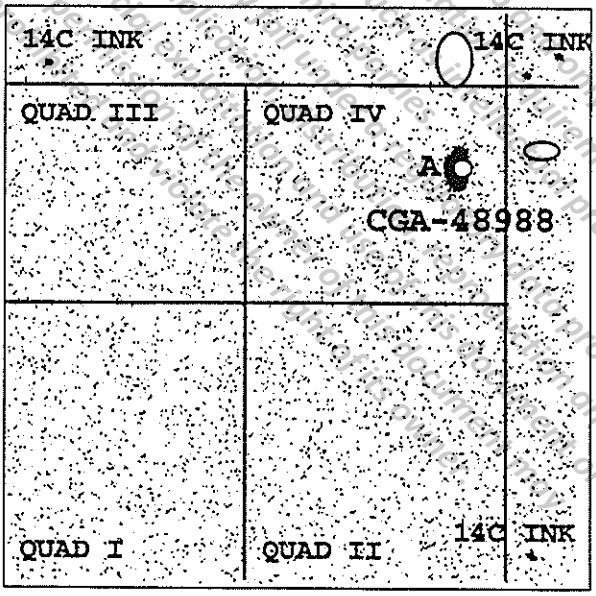
CGA-329351 EXTRACTION 1

DAY 3 R1 NON-IRRADIATED



AREA	PERCENT
A	90.05
QUAD I	0.37
QUAD II	0.12
QUAD III	0.32
QUAD IV	0.26

DAY 3 R2 NON-IRRADIATED



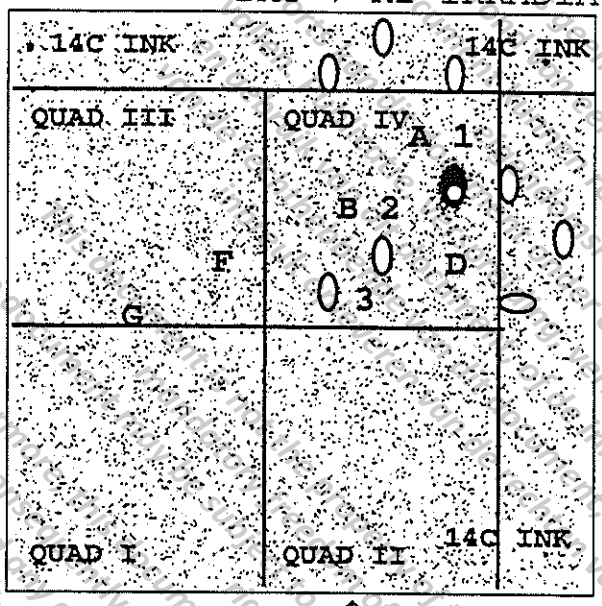
AREA	PERCENT
A	83.56
QUAD I	0.71
QUAD II	0.17
QUAD III	0.32
QUAD IV	0.26

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 16: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 1

DAY 7 R1 IRRADIATED

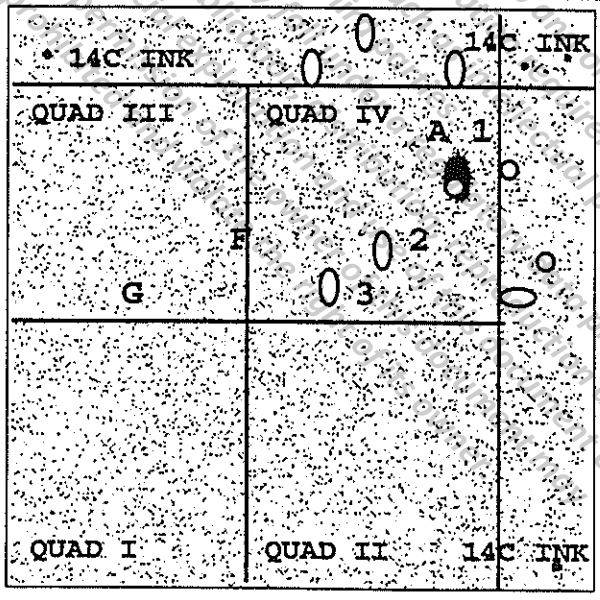


↑SS I → SSII

AREA	PERCENT
A	90.94
B	0.23
D	0.17
F	0.31
G	0.19
QUAD I	0.47
QUAD II	0.39
QUAD III	0.54
QUAD IV	0.61

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 7 R2 IRRADIATED



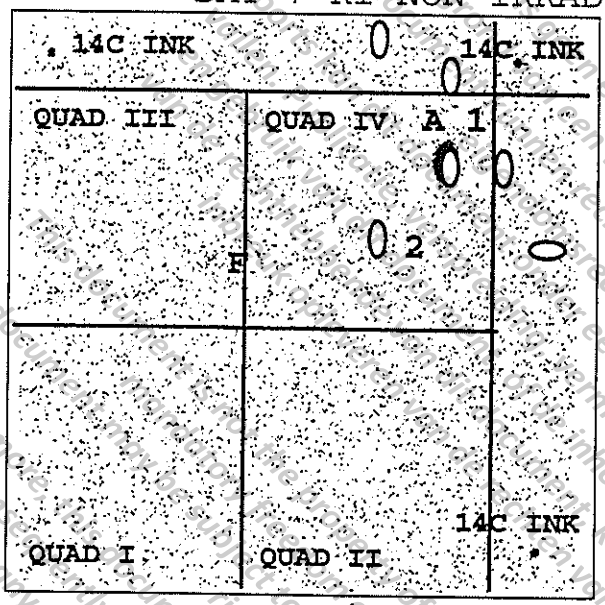
AREA	PERCENT
A	90.30
F	0.01
G	0.23
QUAD I	0.11
QUAD II	0.07
QUAD III	0.02
QUAD IV	0.13

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 17: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 1

DAY 7 R1 NON-IRRADIATED

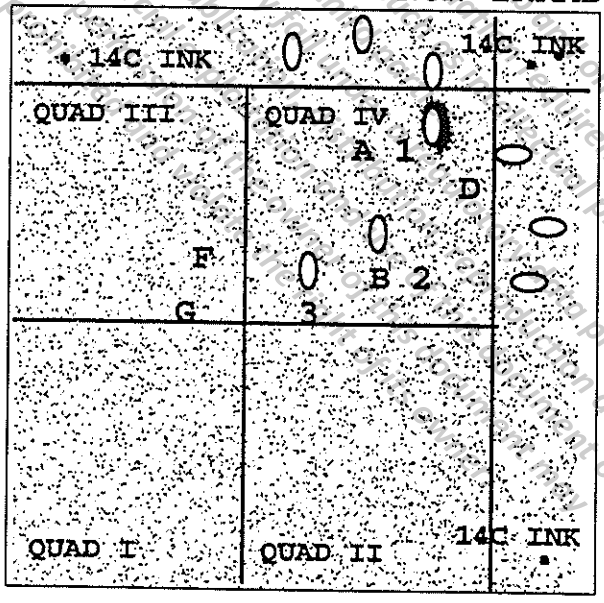


AREA	PERCENT
A	86.98
F	0.30
QUAD I	0.08
QUAD II	0.04
QUAD III	0.17
QUAD IV	0.29

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑SS I → SSII

DAY 7 R2 NON-IRRADIATED



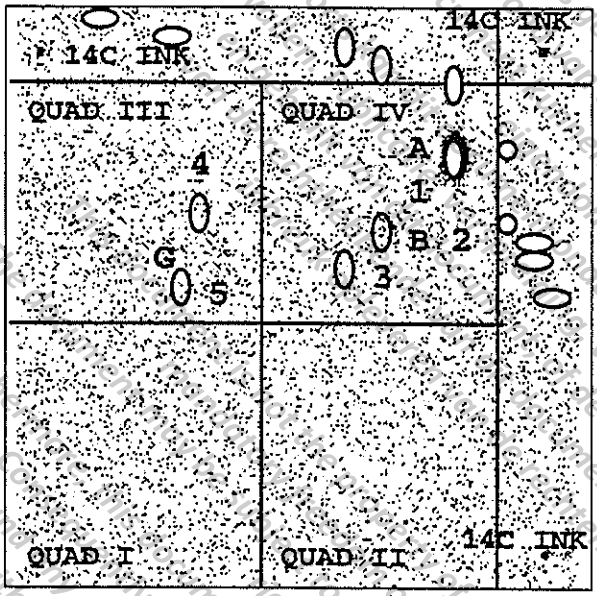
AREA	PERCENT
A	90.83
B	0.31
D	0.22
F	0.28
G	0.19
QUAD I	0.13
QUAD II	0.04
QUAD III	0.21
QUAD IV	0.46

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 18: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

329351 EXTRACTION 1

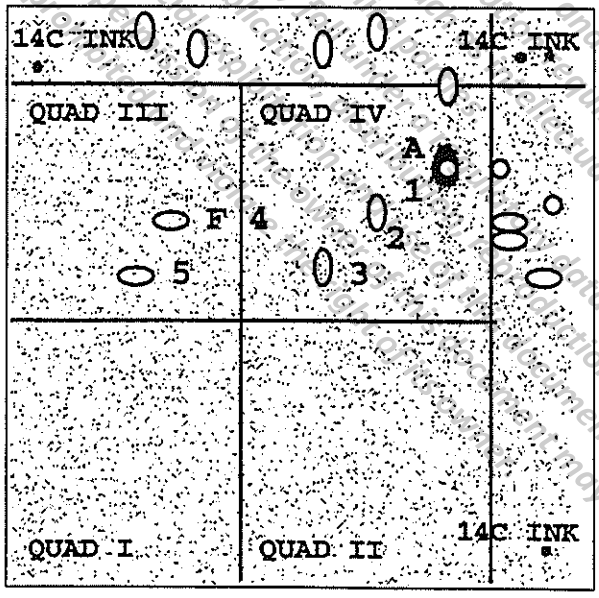
DAY 14 R1 IRRADIATED



AREA	PERCENT
A	81.96
B	0.40
G	0.33
QUAD I	0.35
QUAD II	0.03
QUAD III	0.33
QUAD IV	0.59

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 IRRADIATED



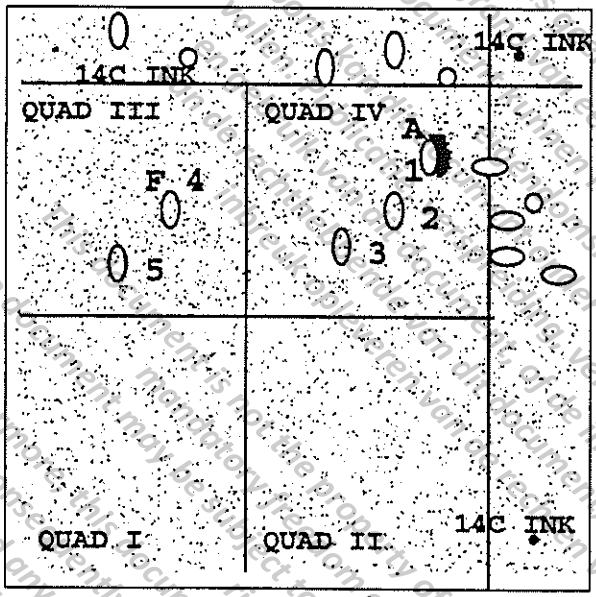
AREA	PERCENT
A	85.17
F	0.66
QUAD I	0.36
QUAD II	0.16
QUAD III	0.38
QUAD IV	0.90

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 19: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 1

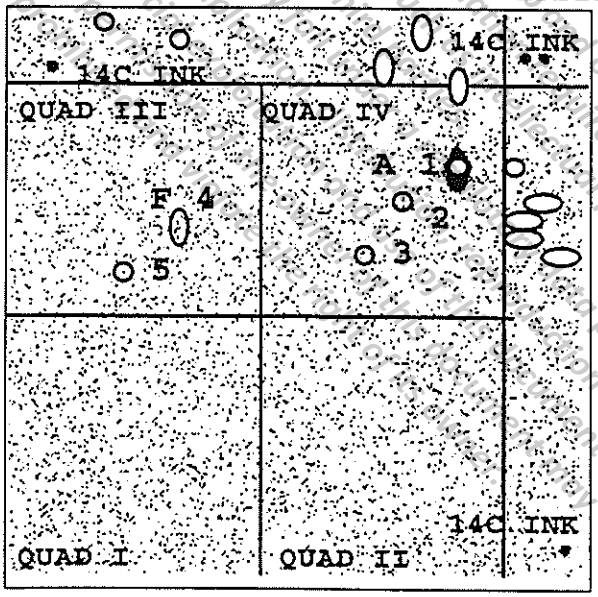
DAY 14 R1 NON-IRRADIATED



AREA	PERCENT
A	86.05
F	0.65
QUAD I	0.32
QUAD II	0.22
QUAD III	0.21
QUAD IV	0.43

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 NON-IRRADIATED



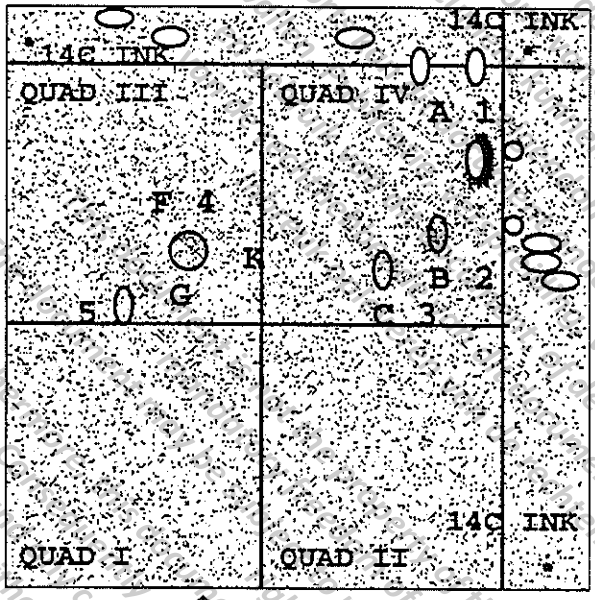
AREA	PERCENT
A	86.46
F	0.45
QUAD I	0.26
QUAD II	0.17
QUAD III	0.26
QUAD IV	0.40

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 20: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

329351 EXTRACTION 1

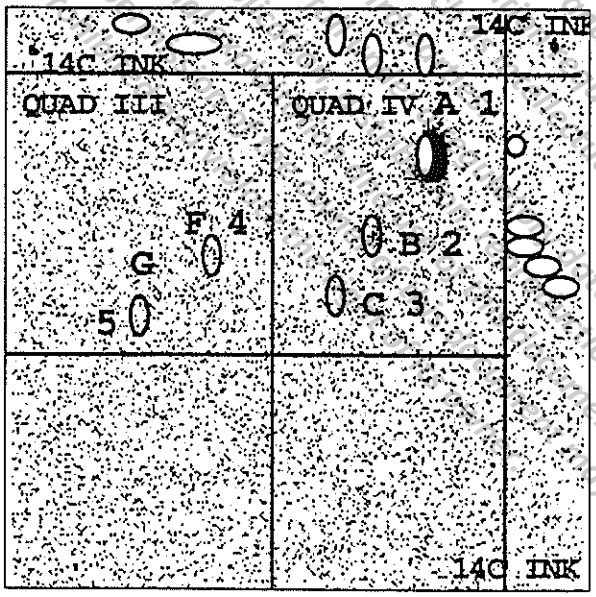
DAY 21 R1 IRRADIATED



AREA	PERCENT
A	84.01
B	0.62
C	0.24
F	0.57
G	0.29
K	0.31
QUAD I	0.40
QUAD II	0.08
QUAD III	0.41
QUAD IV	0.46

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 21 R2 IRRADIATED



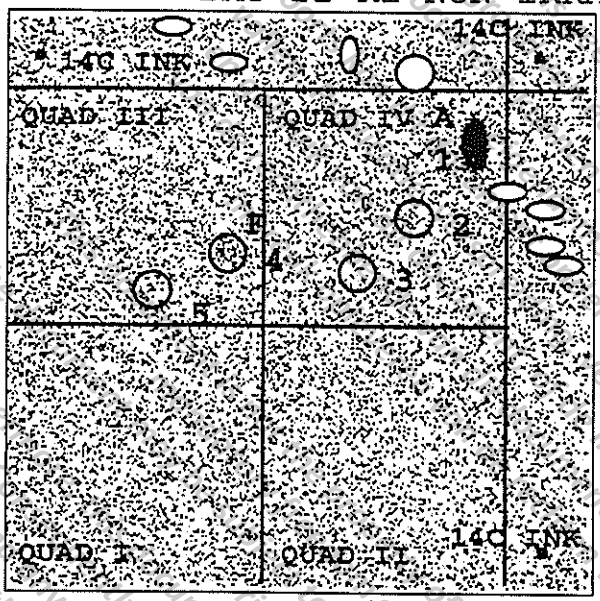
AREA	PERCENT
A	83.21
B	0.67
C	0.16
F	0.33
G	0.23
QUAD I	0.27
QUAD II	0.08
QUAD III	0.38
QUAD IV	0.44

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 21: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

329351 EXTRACTION 1

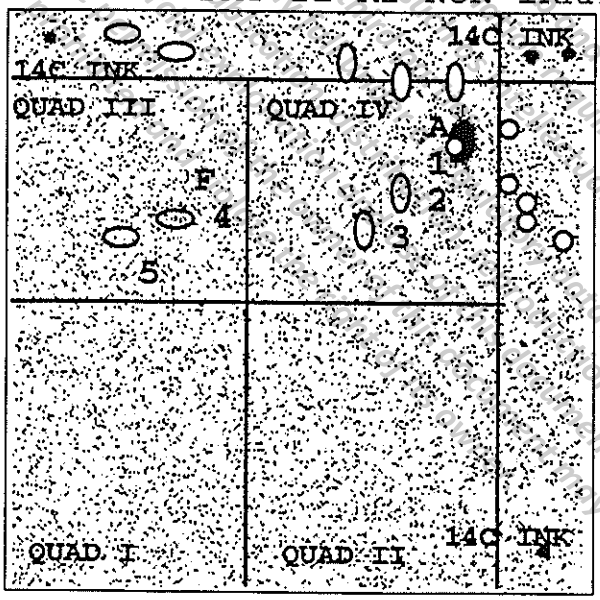
DAY 21 R1 NON-IRRADIATED



AREA	PERCENT
A	91.49
F	0.54
QUAD I	0.20
QUAD II	0.07
QUAD III	0.08
QUAD IV	0.18

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 21 R2 NON-IRRADIATED



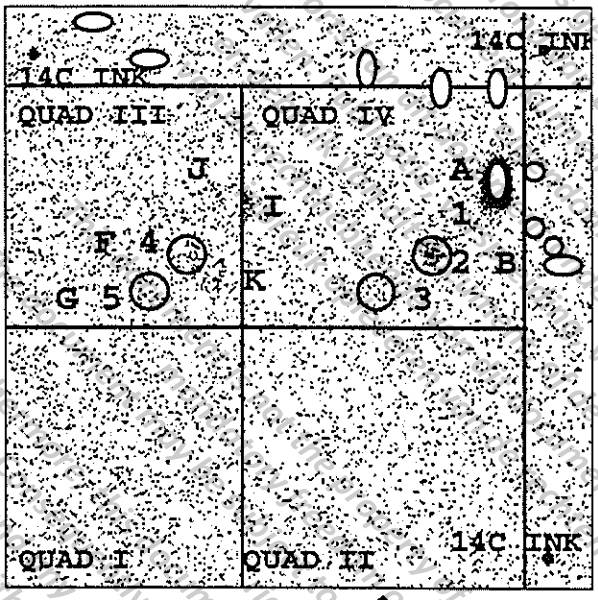
AREA	PERCENT
A	92.68
F	0.44
QUAD I	0.10
QUAD II	0.05
QUAD III	0.06
QUAD IV	0.13

PERCENT EQUALS THE PERCENT OF TOTAL DOSE.

FIGURE 22: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351.

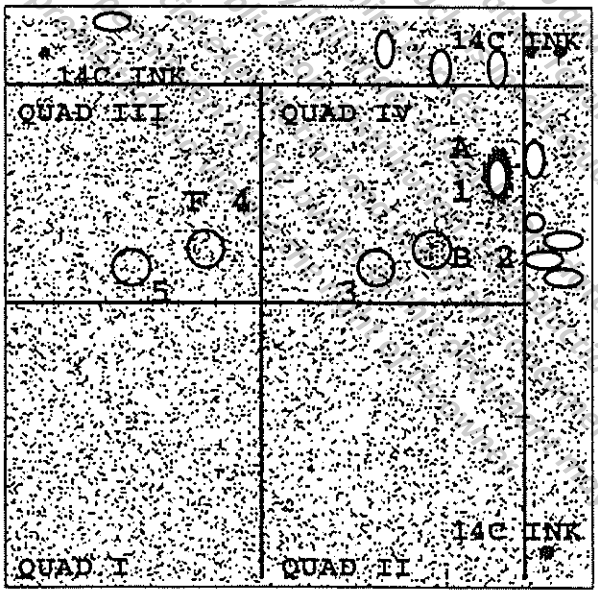
CGA-329351 EXTRACTION 1

DAY 30 R1 IRRADIATED



AREA	PERCENT
A	80.81
B	1.08
F	0.77
G	0.39
I	0.60
J	0.35
K	0.58
QUAD I	0.60
QUAD II	0.11
QUAD III	0.34
QUAD IV	0.89

DAY 30 R2 IRRADIATED



AREA	PERCENT
A	83.99
B	0.49
F	0.46
QUAD I	0.10
QUAD II	0.08
QUAD III	0.44
QUAD IV	0.51

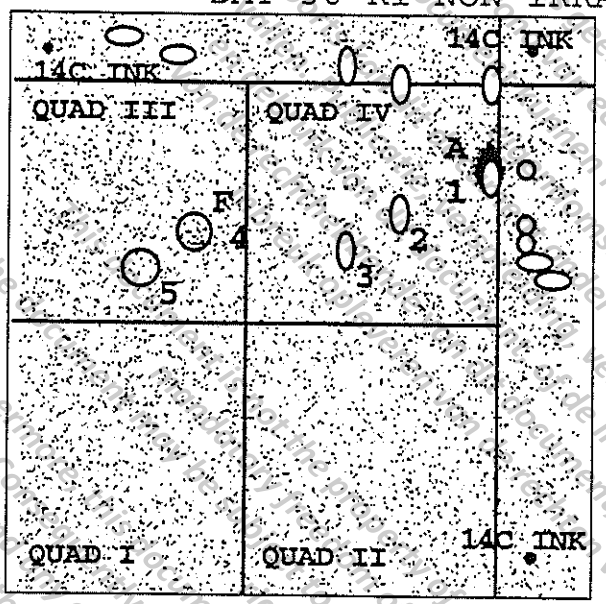
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 23: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30, EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 1

DAY 30 R1 NON-IRRADIATED

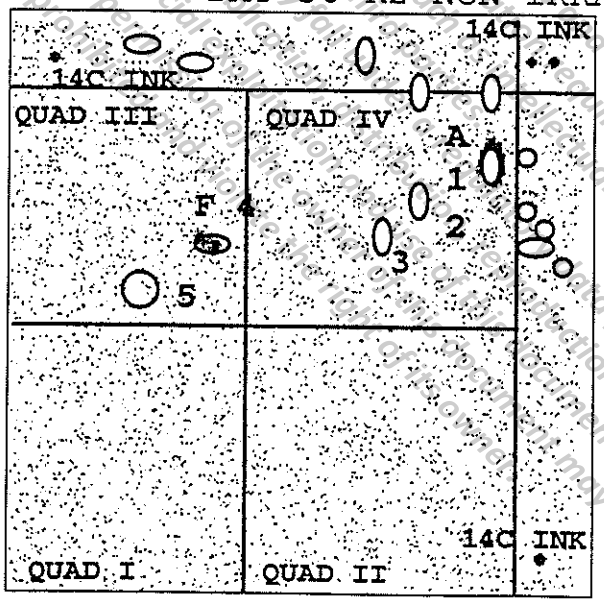


↑SS I → SSII

AREA	PERCENT
A	84.58
F	0.56
QUAD I	0.06
QUAD II	0.03
QUAD III	0.08
QUAD IV	0.26

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 30 R2 NON-IRRADIATED



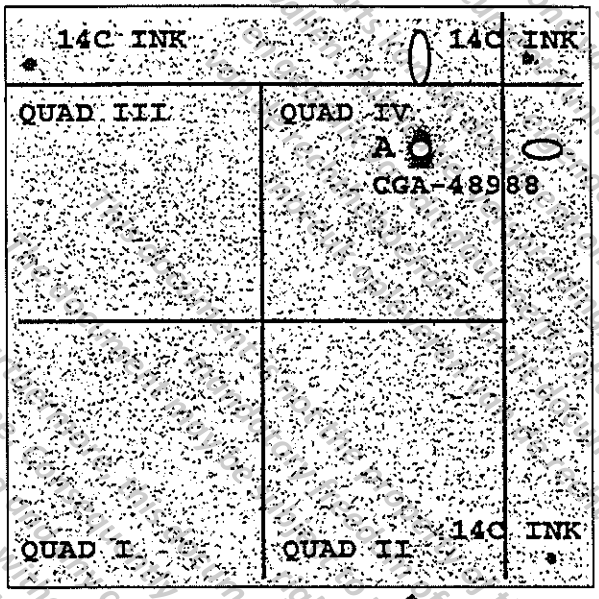
AREA	PERCENT
A	81.96
F	3.46
QUAD I	0.04
QUAD II	0.02
QUAD III	0.21
QUAD IV	0.16

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 24: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30, EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

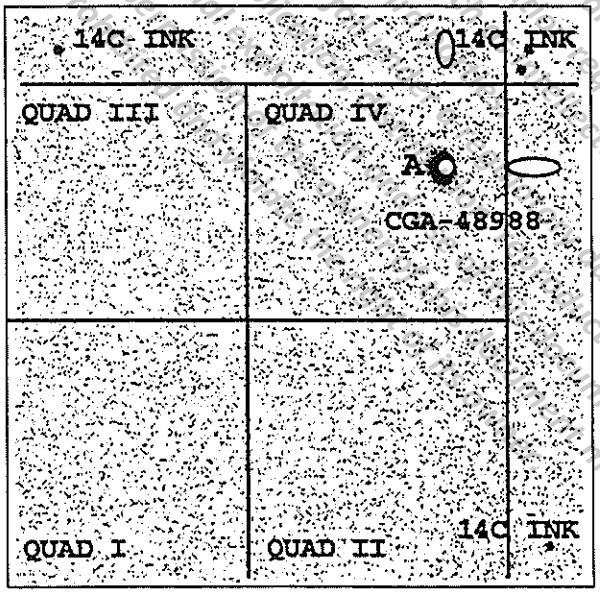
CGA-329351 EXTRACTION 2

DAY 0 R1 IRRADIATED



AREA	PERCENT
A	3.24
QUAD I	0.05
QUAD II	0.03
QUAD III	0.03
QUAD IV	0.03

DAY 0 R2 IRRADIATED



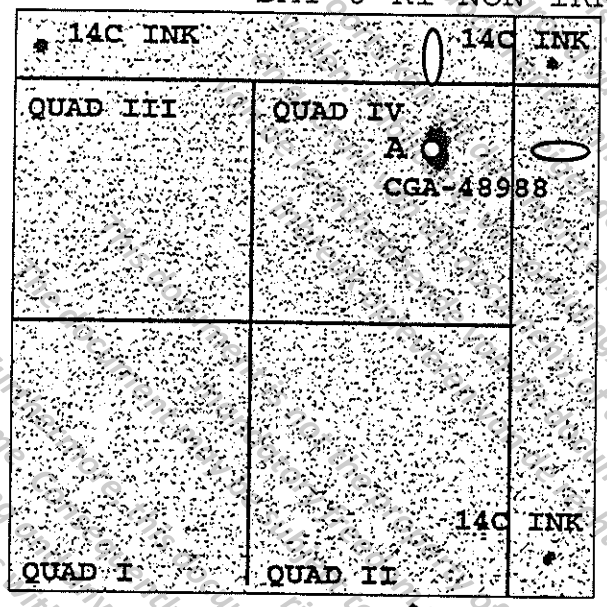
AREA	PERCENT
A	2.87
QUAD I	0.06
QUAD II	0.01
QUAD III	0.02
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE.

FIGURE 25: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

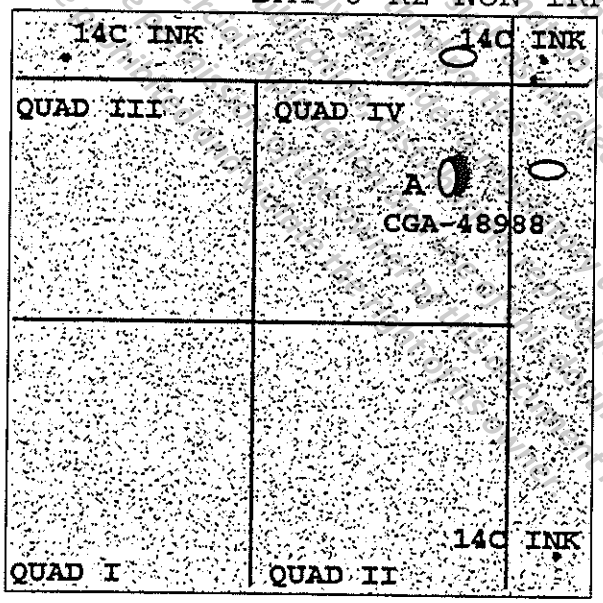
CGA-329351 EXTRACTION 2

DAY 0 R1 NON-IRRADIATED



AREA	PERCENT
A	4.02
QUAD I	0.04
QUAD II	0.03
QUAD III	0.03
QUAD IV	0.04

DAY 0 R2 NON-IRRADIATED



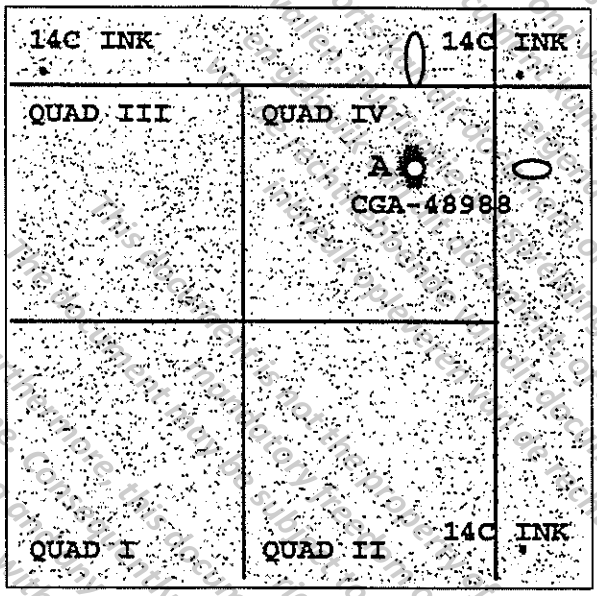
AREA	PERCENT
A	4.08
QUAD I	0.04
QUAD II	0.02
QUAD III	0.02
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 26: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

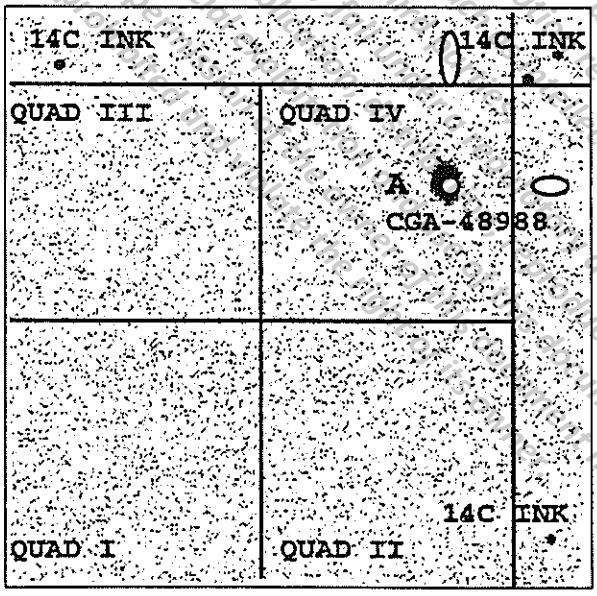
CGA-329351 EXTRACTION 2

DAY 3 R1 IRRADIATED



AREA	PERCENT
A	3.53
QUAD I	0.01
QUAD II	<MQA
QUAD III	0.01
QUAD IV	0.03

DAY 3 R2 IRRADIATED



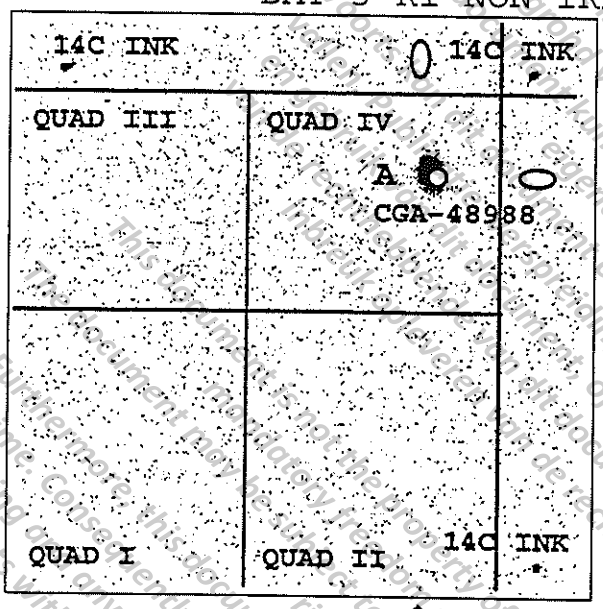
AREA	PERCENT
A	4.05
QUAD I	0.03
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.06

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 27: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

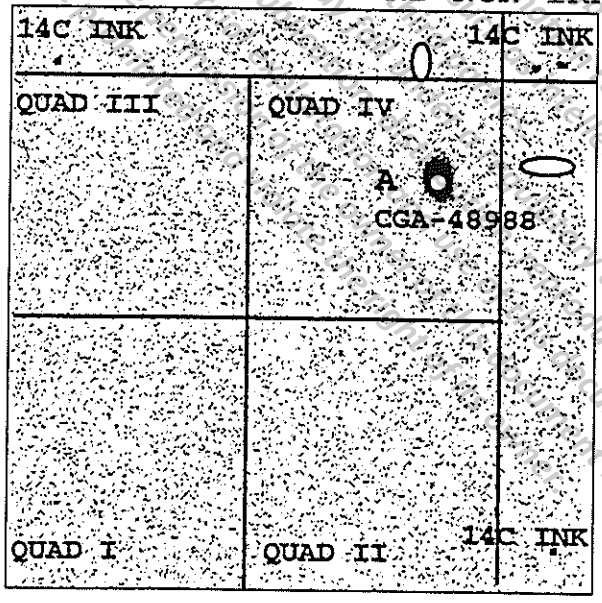
CGA-329351 EXTRACTION 2

DAY 3 R1 NON-IRRADIATED



AREA	PERCENT
A	4.03
QUAD I	0.01
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.02

DAY 3 R2 NON-IRRADIATED



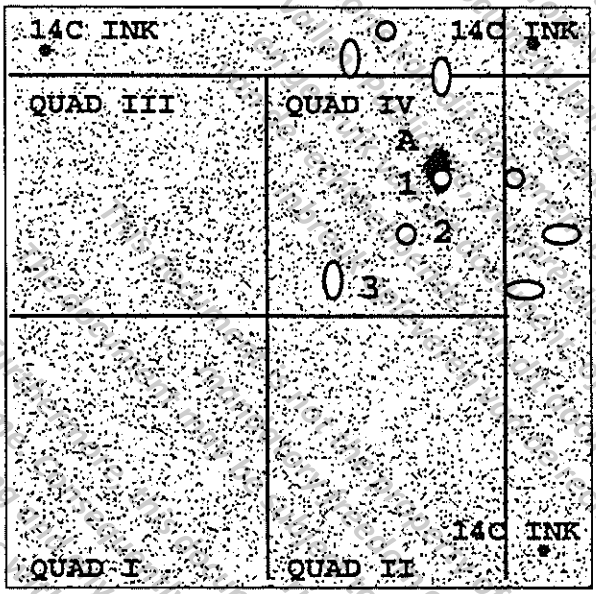
AREA	PERCENT
A	6.44
QUAD I	0.02
QUAD II	0.02
QUAD III	0.01
QUAD IV	0.04

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 28: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

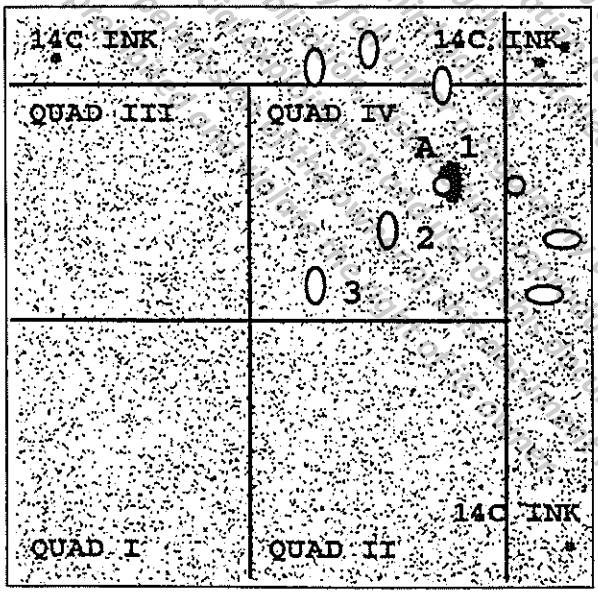
DAY 7 R1 IRRADIATED



AREA	PERCENT
A	4.34
QUAD I	0.05
QUAD II	< 0.01
QUAD III	0.05
QUAD IV	0.05

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 7 R2 IRRADIATED



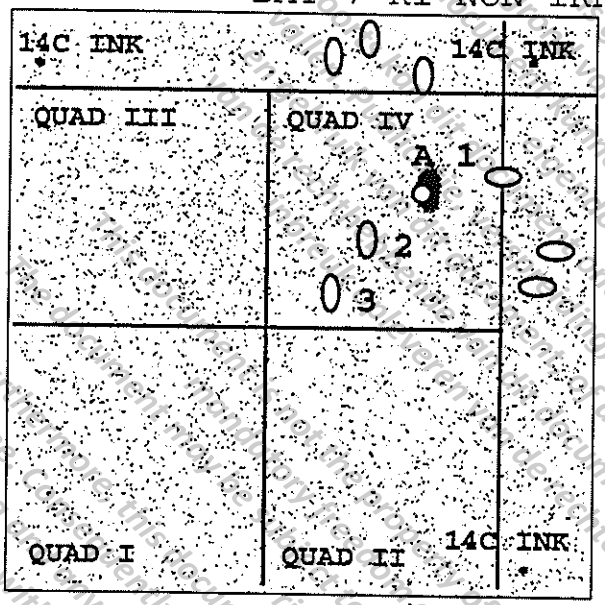
AREA	PERCENT
A	3.14
QUAD I	0.05
QUAD II	0.02
QUAD III	0.05
QUAD IV	0.07

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 29: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

DAY 7 R1 NON-IRRADIATED

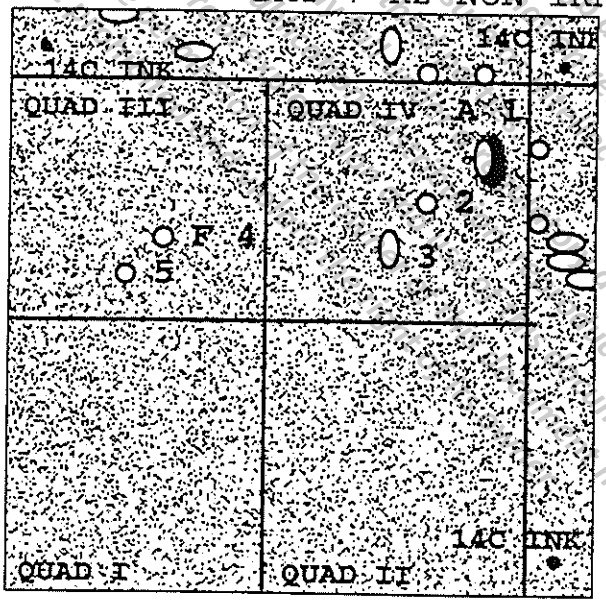


AREA	PERCENT
A	3.01
QUAD I	0.04
QUAD II	0.01
QUAD III	0.03
QUAD IV	0.03

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

DAY 7 R2 NON-IRRADIATED



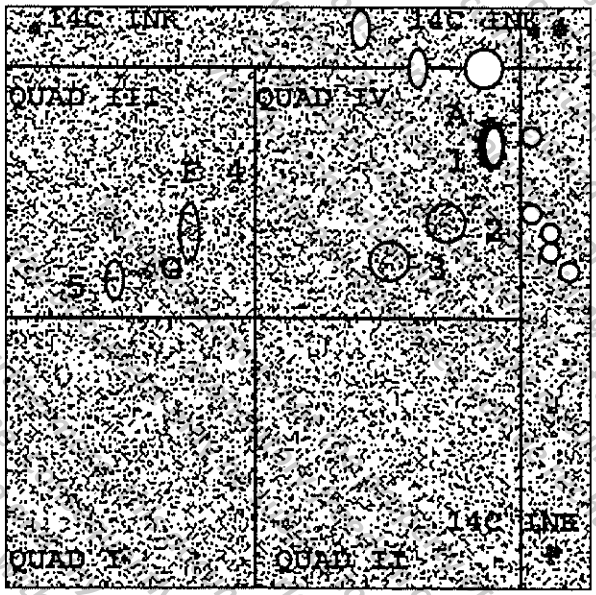
AREA	PERCENT
A	2.74
F	0.01
QUAD I	0.02
QUAD II	0.01
QUAD III	0.02
QUAD IV	0.01

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 30: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

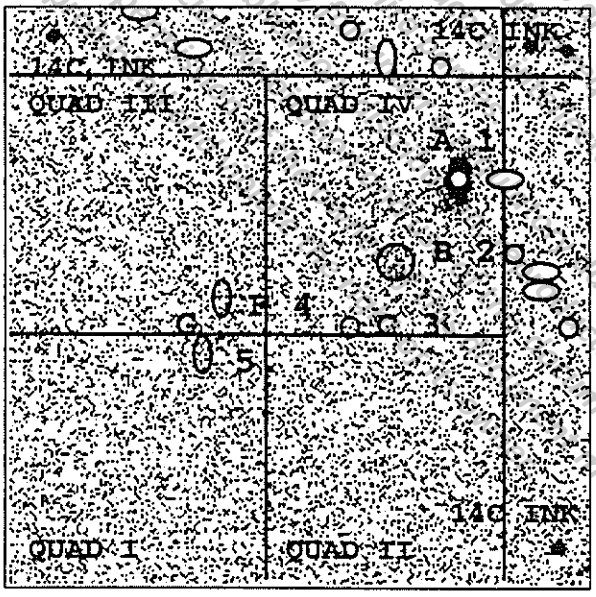
DAY 14 R1 IRRADIATED



AREA	PERCENT
A	3.56
F	0.04
G	0.05
QUAD I	0.04
QUAD II	0.01
QUAD III	0.02
QUAD IV	0.07

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 IRRADIATED



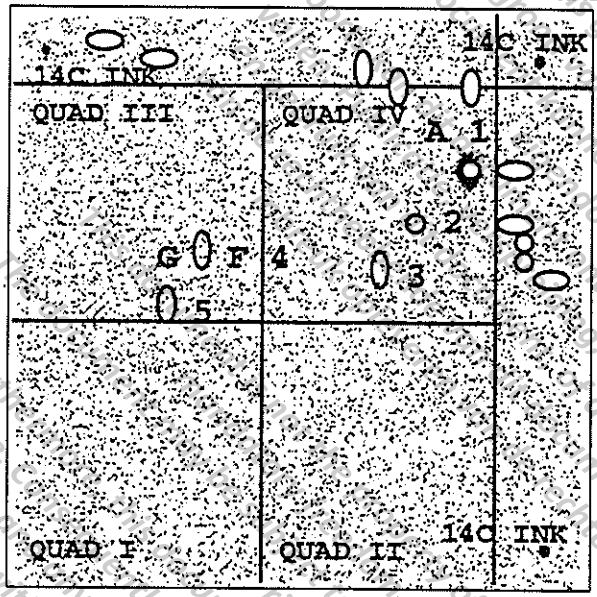
AREA	PERCENT
A	2.21
B	0.02
C	0.01
F	0.02
G	0.03
QUAD I	0.03
QUAD II	0.03
QUAD III	0.03
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 31: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

DAY 14 R1 NON-IRRADIATED

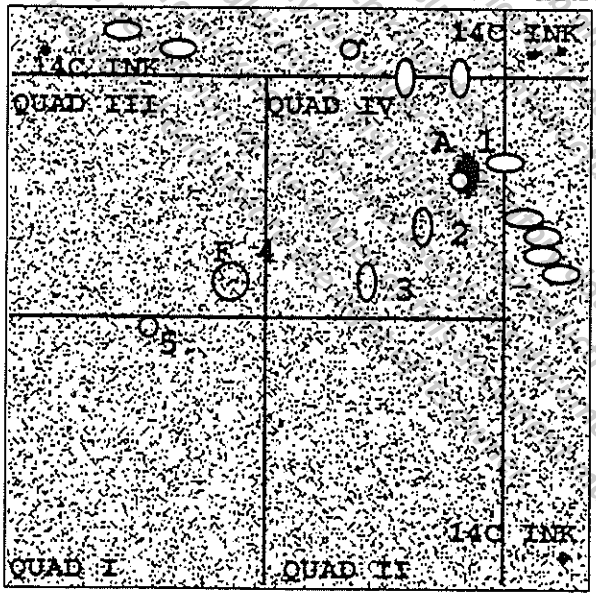


AREA	PERCENT
A	3.62
F	0.03
G	0.03
QUAD I	0.02
QUAD II	<MQA
QUAD III	0.02
QUAD IV	0.03

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

DAY 14 R2 NON-IRRADIATED



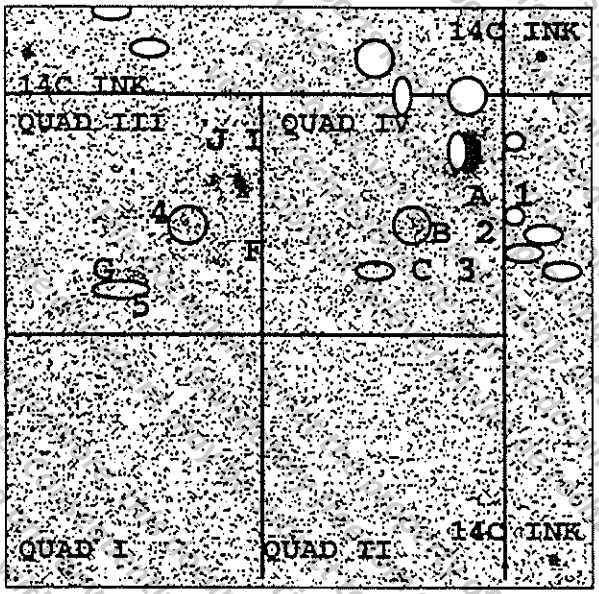
AREA	PERCENT
A	3.59
F	0.03
QUAD I	0.02
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.02

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 32: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

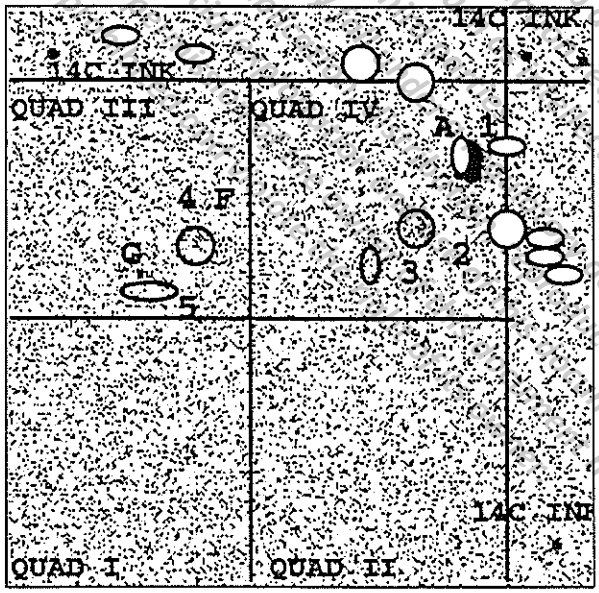
DAY 21 R1 IRRADIATED



↑ SS I → SSII

AREA	PERCENT
A	2.74
B	0.03
C	0.01
F	0.04
G	0.05
I	0.10
J	0.04
QUAD I	0.04
QUAD II	0.01
QUAD III	0.03
QUAD IV	0.03

DAY 21 R2 IRRADIATED



AREA	PERCENT
A	2.40
F	0.02
G	0.04
QUAD I	0.04
QUAD II	<0.01
QUAD III	0.04
QUAD IV	0.05

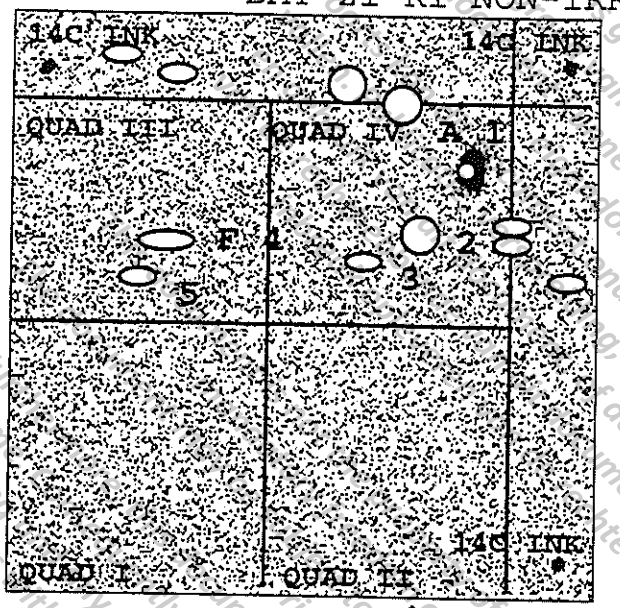
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 33: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

DAY 21 R1 NON-IRRADIATED

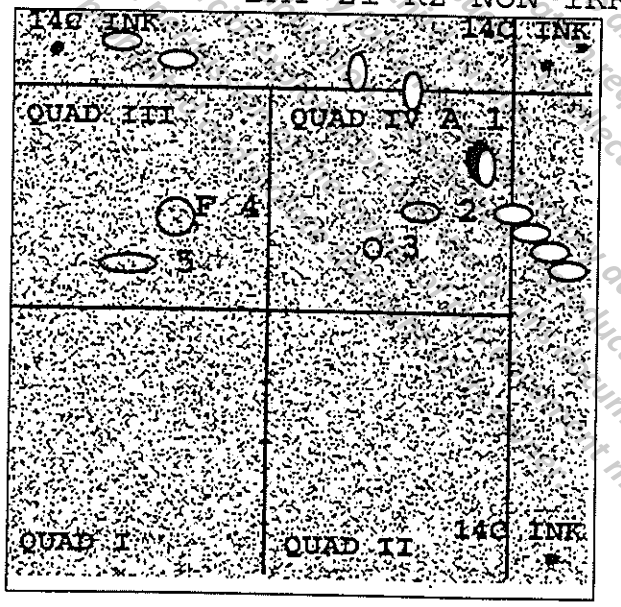


AREA	PERCENT
A	2.52
F	0.03
QUAD I	0.01
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.02

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

DAY 21 R2 NON-IRRADIATED



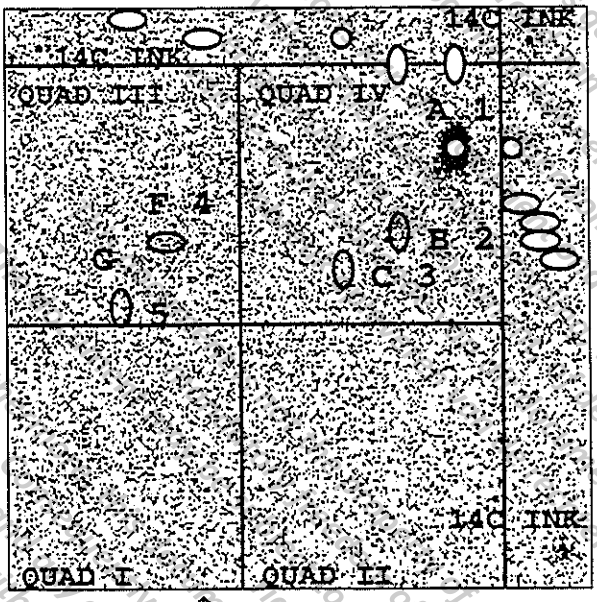
AREA	PERCENT
A	2.97
F	0.03
QUAD I	0.01
QUAD II	<MQA
QUAD III	0.01
QUAD IV	0.01

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 34: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

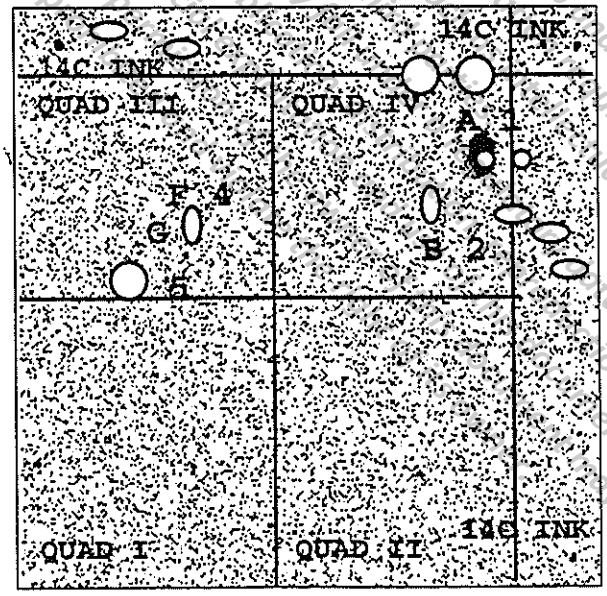
DAY 30 R1 IRRADIATED



AREA	PERCENT
A	4.72
B	0.07
C	0.03
F	0.07
G	0.07
QUAD I	0.22
QUAD II	0.05
QUAD III	0.06
QUAD IV	0.05

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 30 R2 IRRADIATED



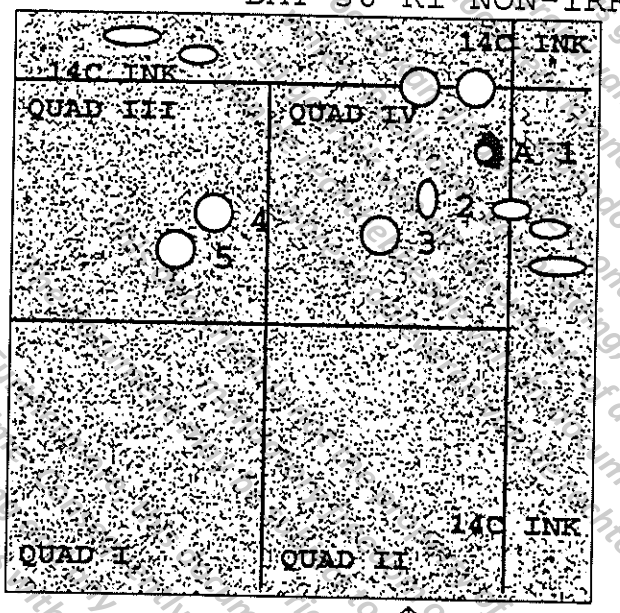
AREA	PERCENT
A	3.31
B	0.03
F	0.04
G	0.03
QUAD I	0.03
QUAD II	< MQA
QUAD III	0.02
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 35: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30, EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351

CGA-329351 EXTRACTION 2

DAY 30 R1 NON-IRRADIATED

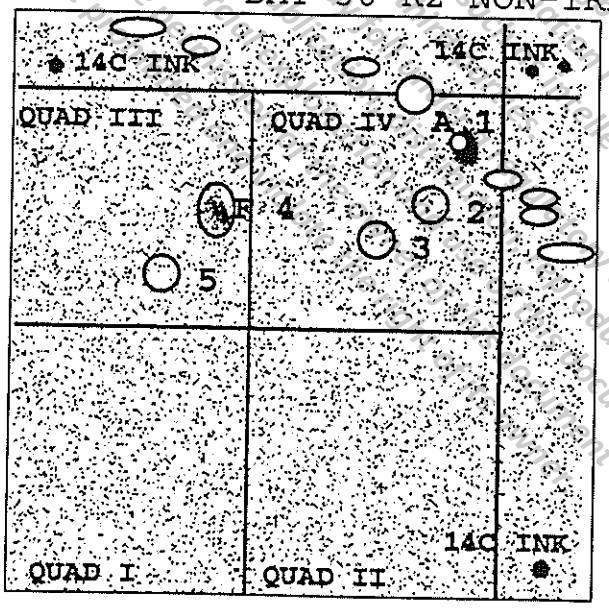


AREA	PERCENT
A	5.29
QUAD I	0.02
QUAD II	0.02
QUAD III	0.07
QUAD IV	0.05

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

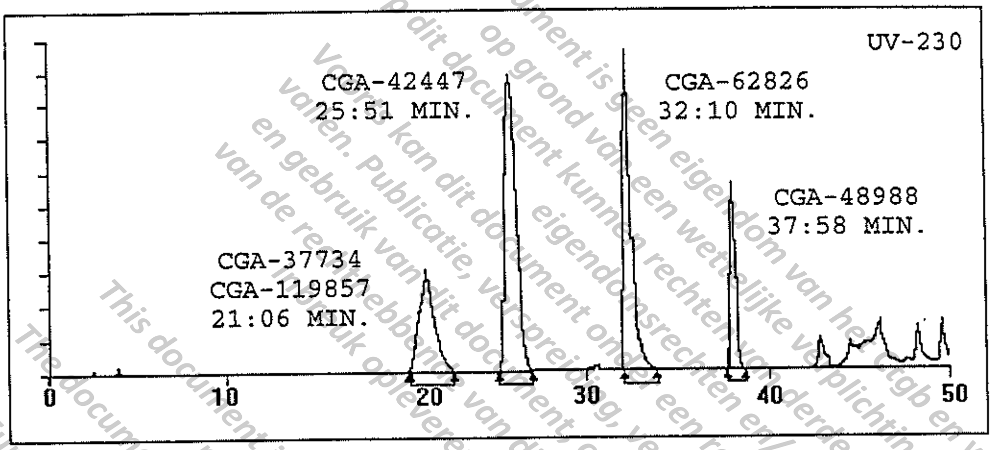
DAY 30 R2 NON-IRRADIATED



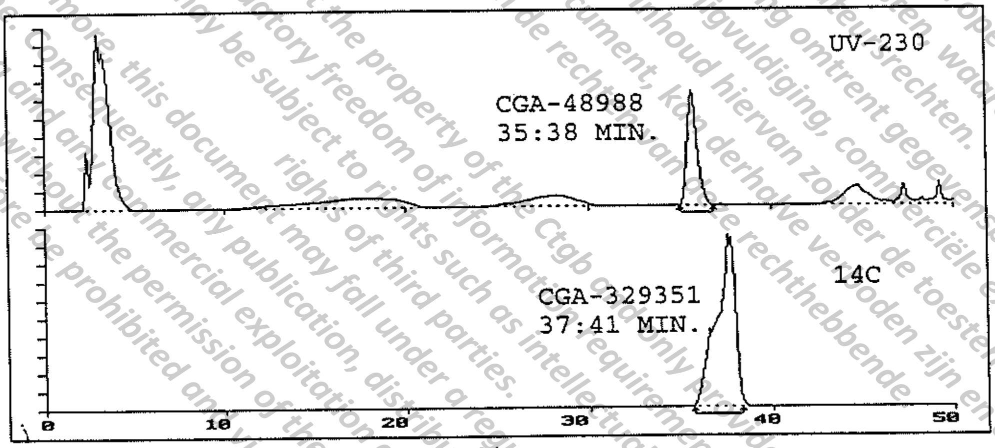
AREA	PERCENT
A	5.25
F	0.44
QUAD I	0.04
QUAD II	0.02
QUAD III	0.09
QUAD IV	0.05

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

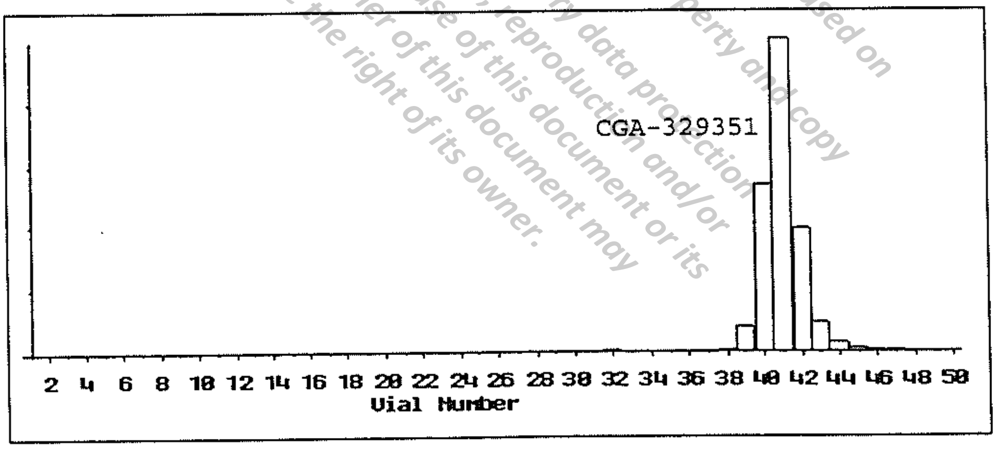
FIGURE 36: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30, EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-329351



UV TRACE OF THE STANDARD MIXTURE

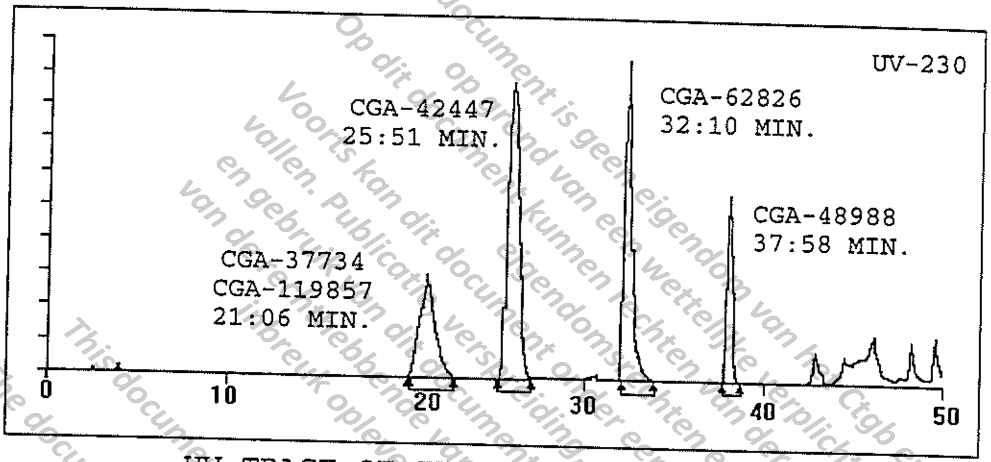


UV AND ^{14}C TRACES

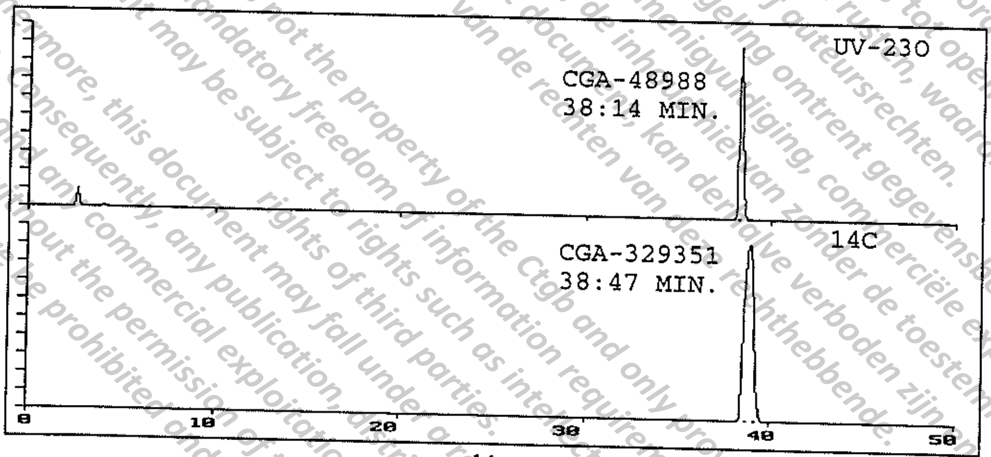


HISTOGRAM

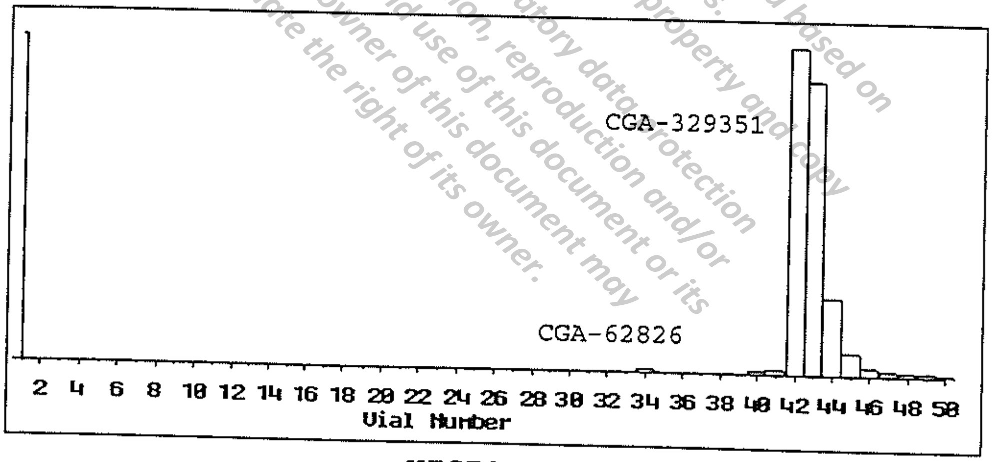
FIGURE 37: <u>HPLC OF DAY 0, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-329351</u>



UV TRACE OF THE STANDARD MIXTURE

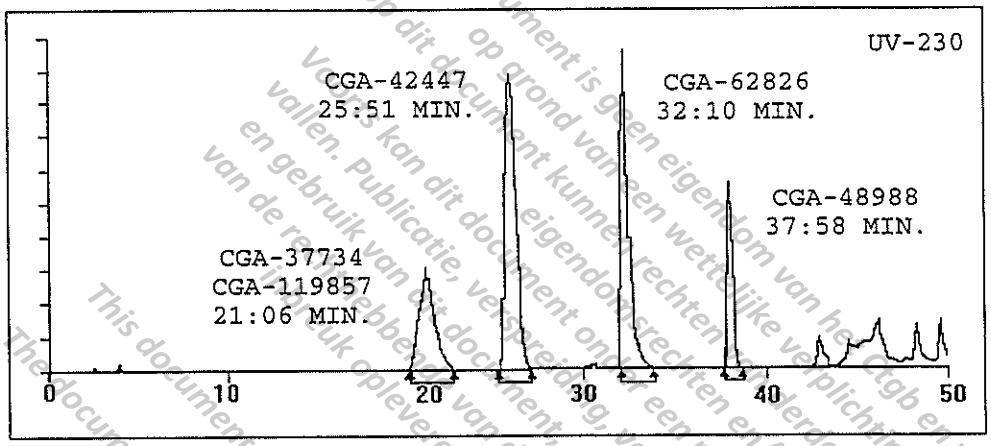


UV AND ^{14}C TRACES

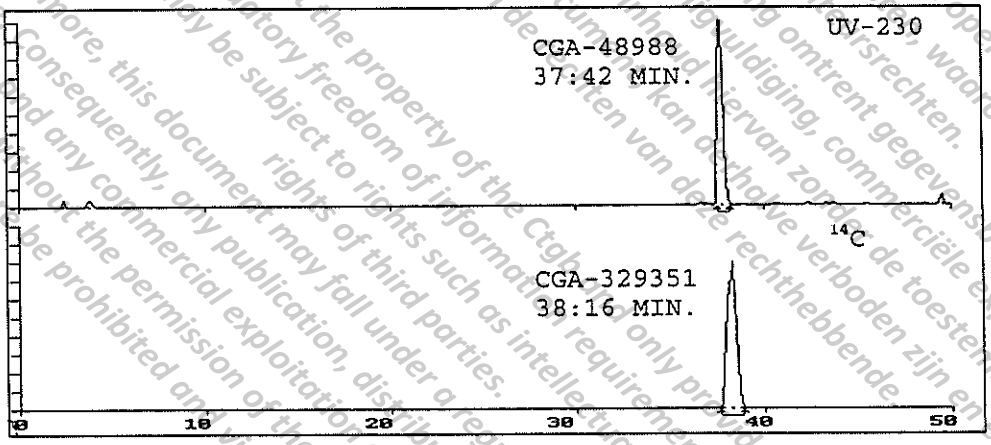


HISTOGRAM

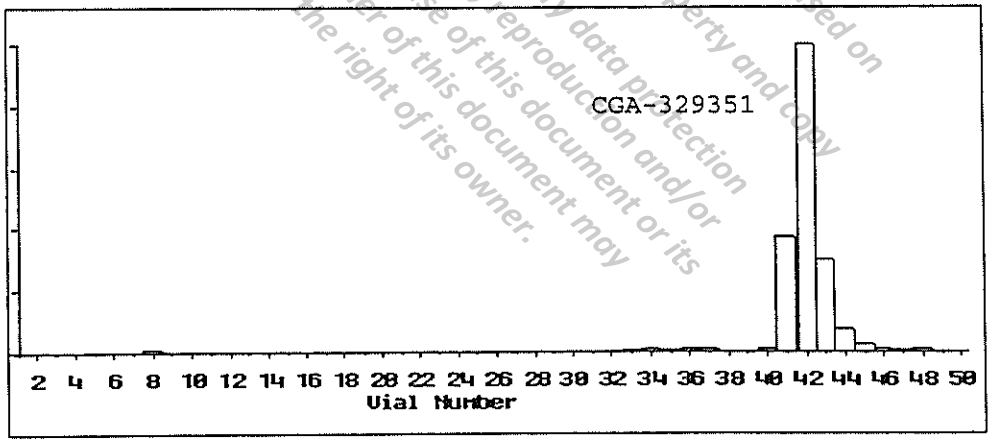
FIGURE 38: HPLC OF DAY 0, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-329351



UV TRACE OF THE STANDARD MIXTURE

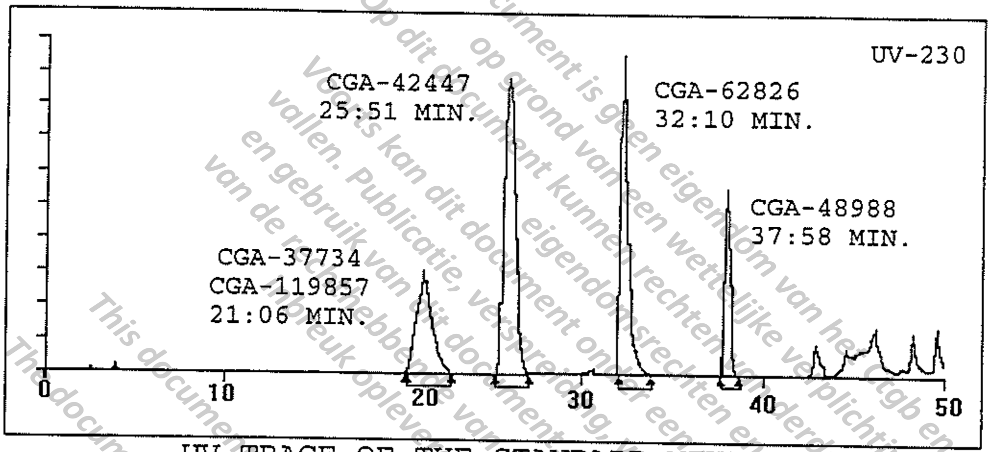


UV AND ^{14}C TRACES

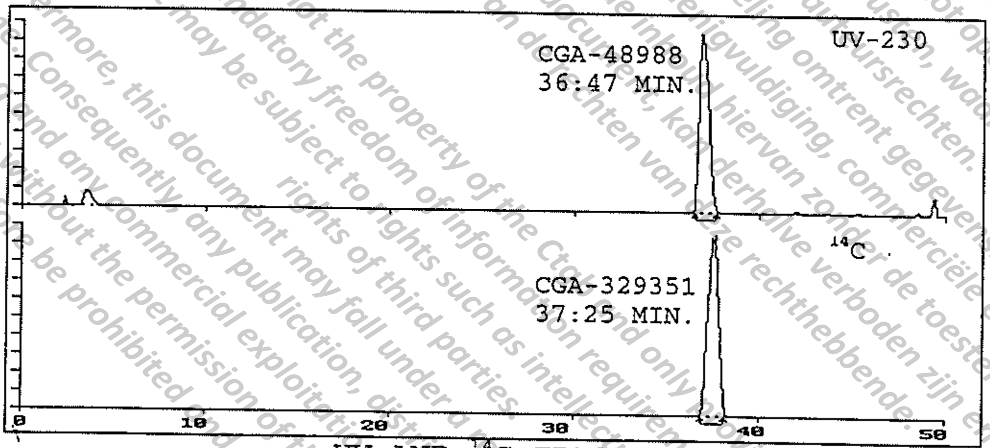


HISTOGRAM

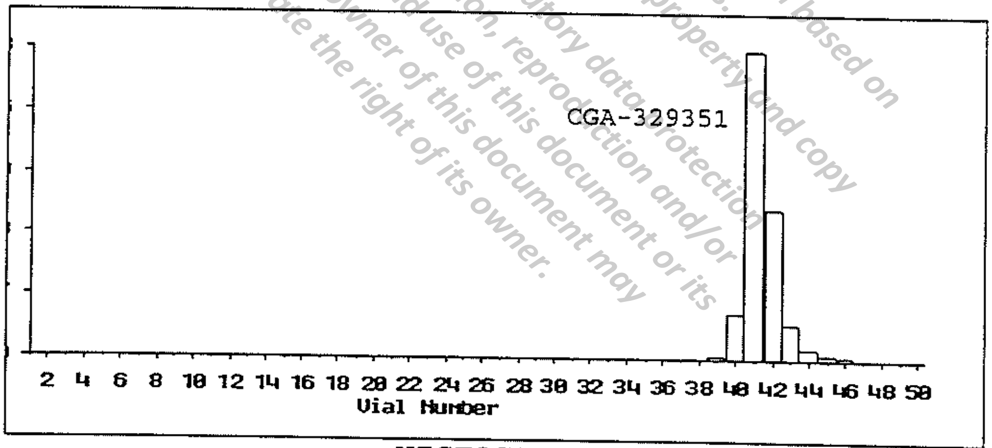
FIGURE 39: HPLC OF DAY 14, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-329351



UV TRACE OF THE STANDARD MIXTURE

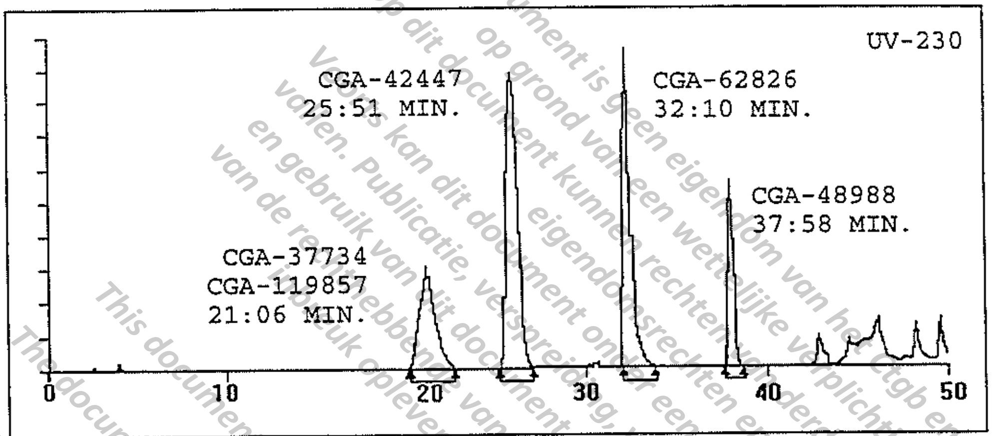


UV AND ^{14}C TRACES

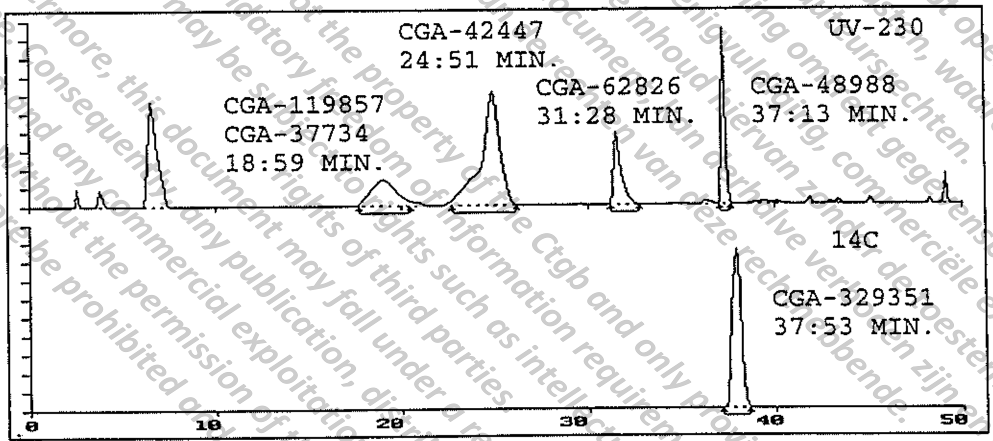


HISTOGRAM

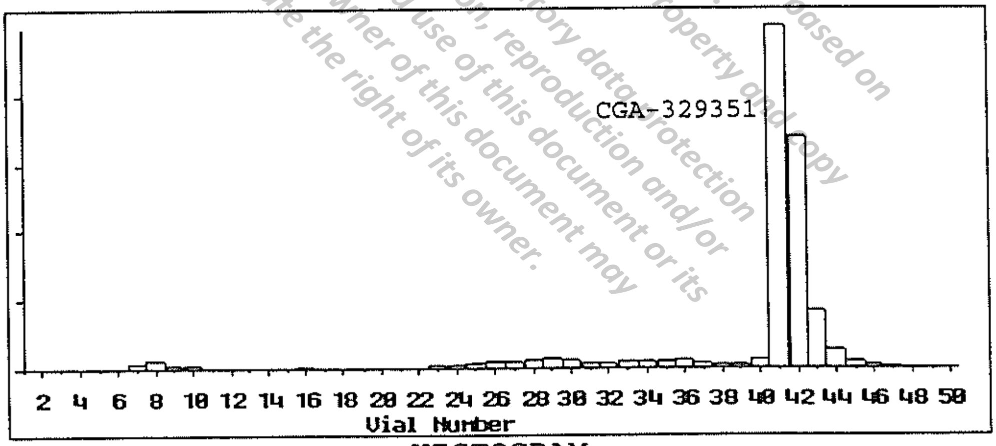
FIGURE 40: HPLC OF DAY 14, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-329351



UV TRACE OF THE STANDARD MIXTURE

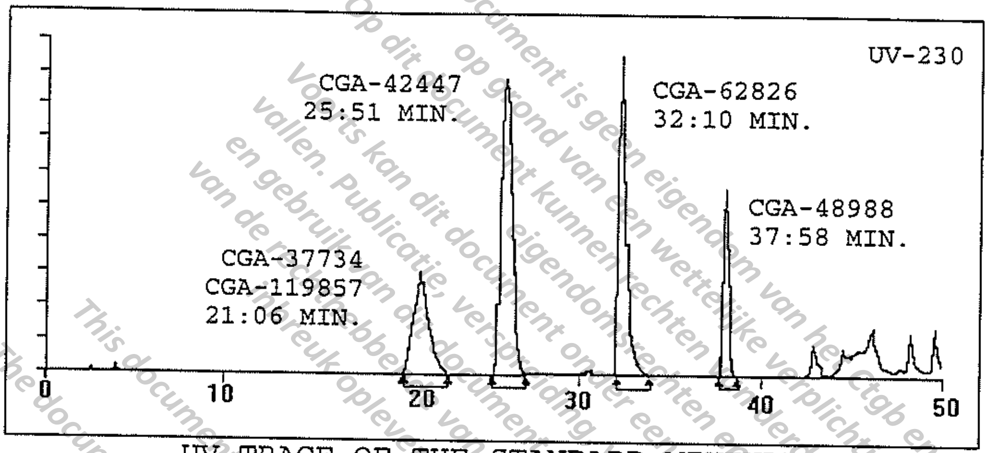


UV AND 14C TRACES

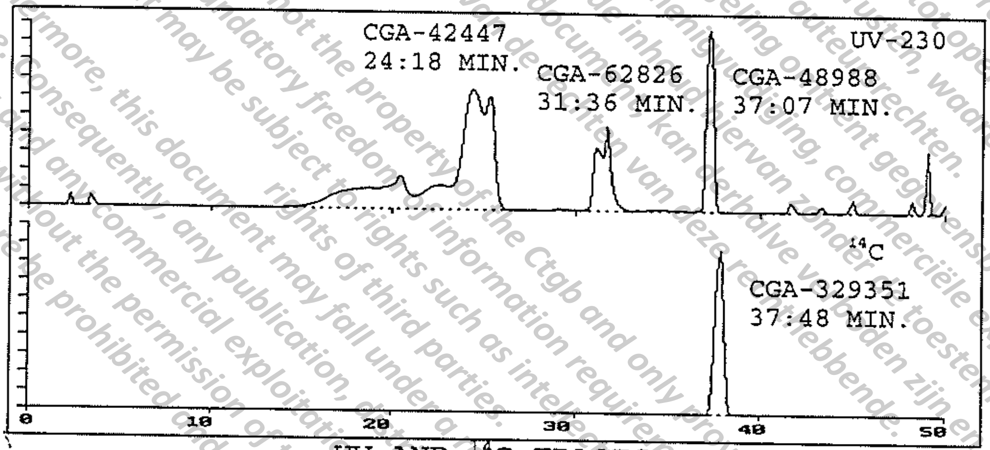


HISTOGRAM

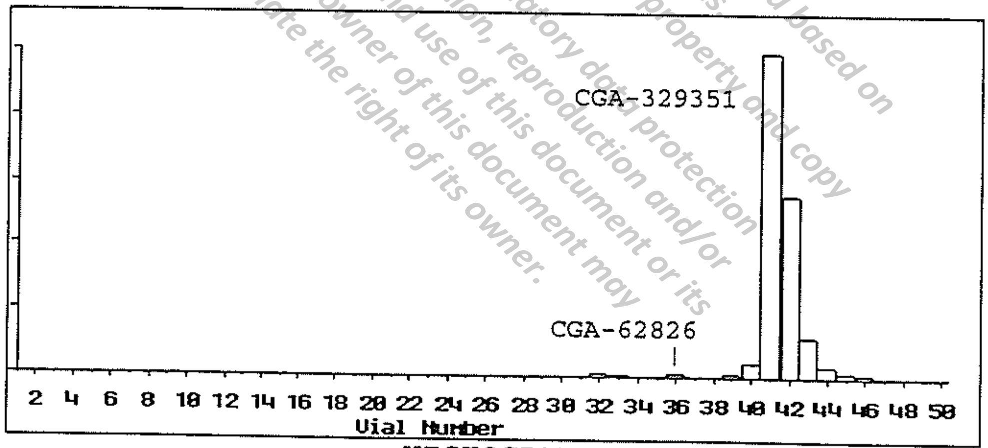
FIGURE 41: <u>HPLC OF DAY 30, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-329351</u>



UV TRACE OF THE STANDARD MIXTURE



UV AND ^{14}C TRACES

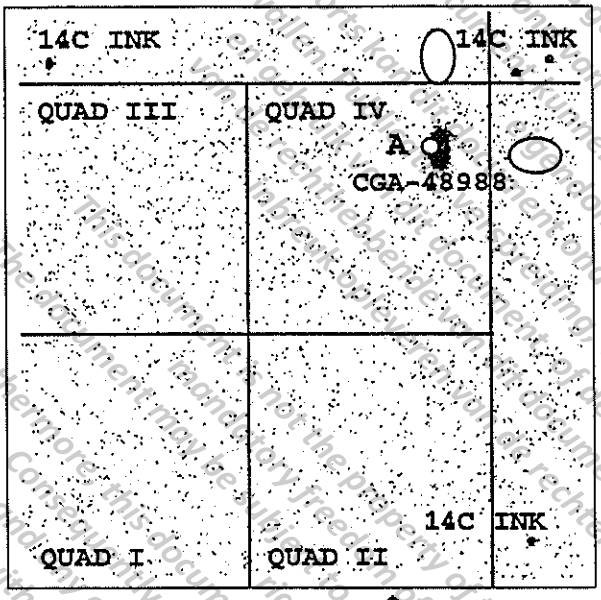


HISTOGRAM

FIGURE 42: HPLC OF DAY 30, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-329351

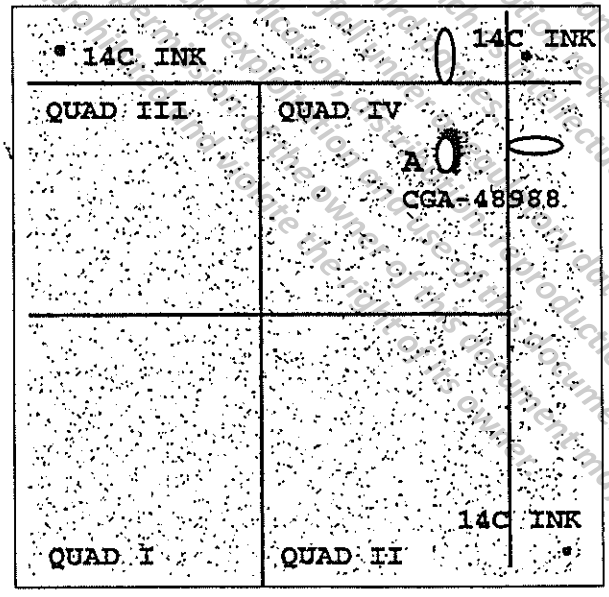
CGA-48988 EXTRACTION 1

DAY 0 R1 IRRADIATED



AREA	PERCENT
A	87.72
QUAD I	0.08
QUAD II	0.05
QUAD III	0.01
QUAD IV	0.44

DAY 0 R2 IRRADIATED



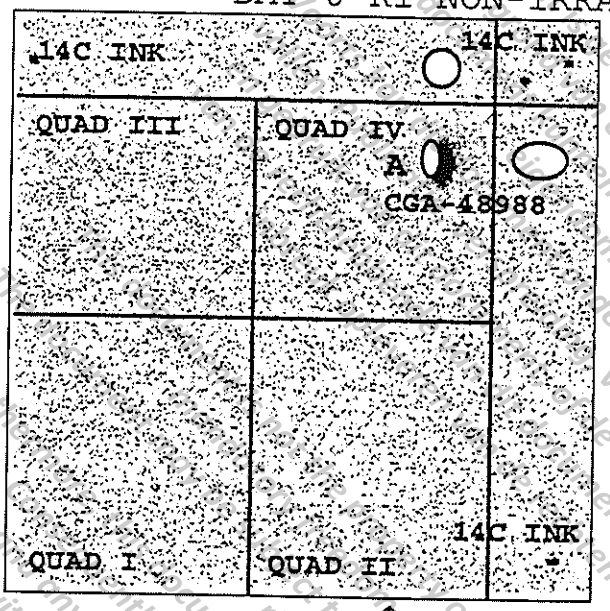
AREA	PERCENT
A	85.95
QUAD I	0.17
QUAD II	0.16
QUAD III	0.28
QUAD IV	0.72

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 43: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

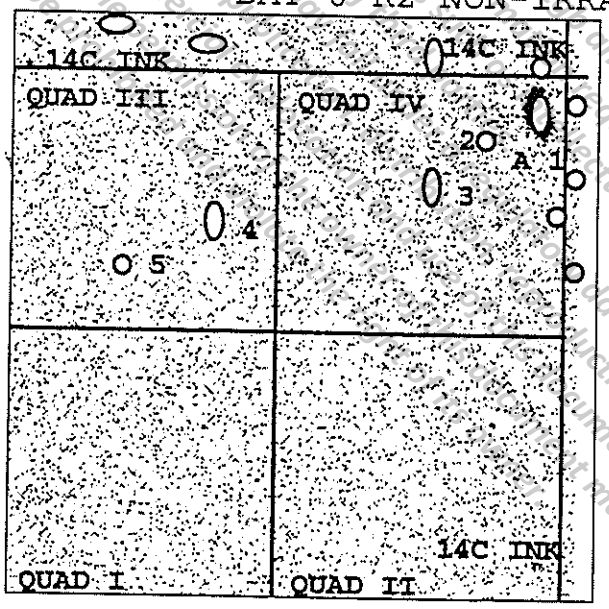
CGA-48988 EXTRACTION 1

DAY 0 R1 NON-IRRADIATED



AREA	PERCENT
A	85.22
QUAD I	0.04
QUAD II	< MQA
QUAD III	0.07
QUAD IV	0.81

DAY 0 R2 NON-IRRADIATED



AREA	PERCENT
A	92.92
QUAD I	0.10
QUAD II	0.07
QUAD III	0.40
QUAD IV	0.61

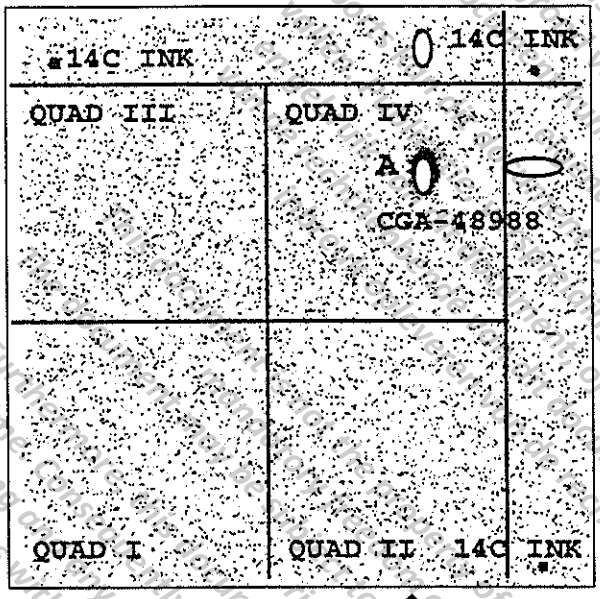
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 44: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

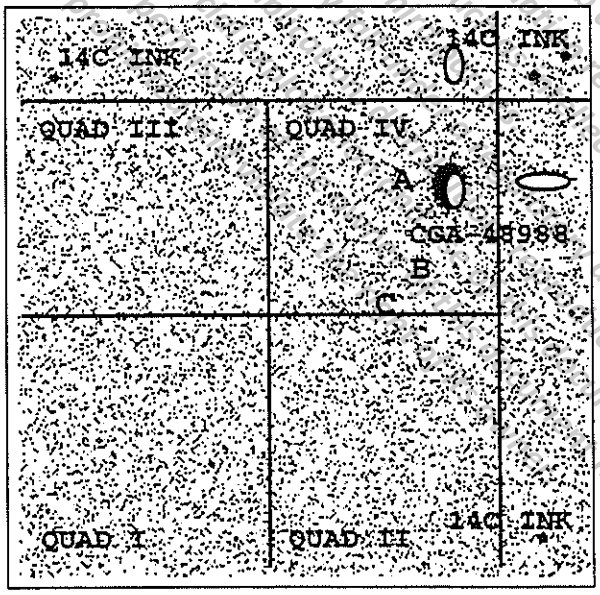
CGA-48988 EXTRACTION 1

DAY 3 R1 IRRADIATED



AREA	PERCENT
A	81.96
QUAD I	0.65
QUAD II	0.13
QUAD III	0.07
QUAD IV	1.38

DAY 3 R2 IRRADIATED



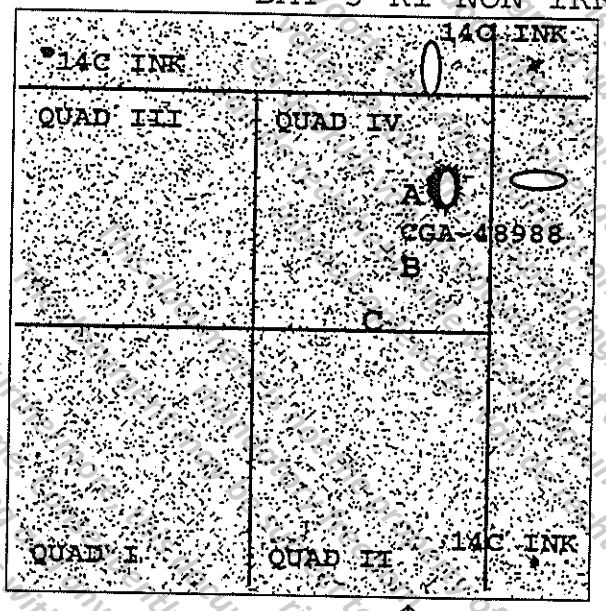
AREA	PERCENT
A	84.68
B	0.54
C	0.44
QUAD I	0.16
QUAD II	0.06
QUAD III	0.01
QUAD IV	0.27

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 45: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

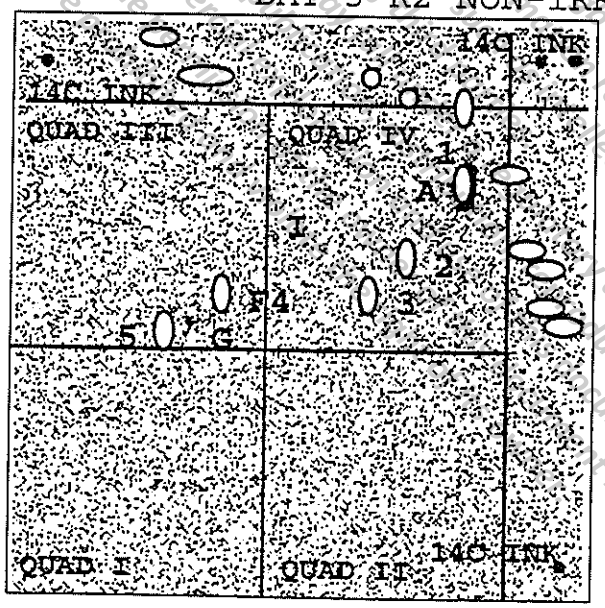
CGA-48988 EXTRACTION 1

DAY 3 R1 NON-IRRADIATED



AREA	PERCENT
A	89.44
B	0.47
C	0.77
QUAD I	0.34
QUAD II	0.28
QUAD III	0.14
QUAD IV	0.34

DAY 3 R2 NON-IRRADIATED



AREA	PERCENT
A	89.05
F	0.20
G	0.24
I	0.04
QUAD I	0.08
QUAD II	<MQA
QUAD III	0.09
QUAD IV	0.33

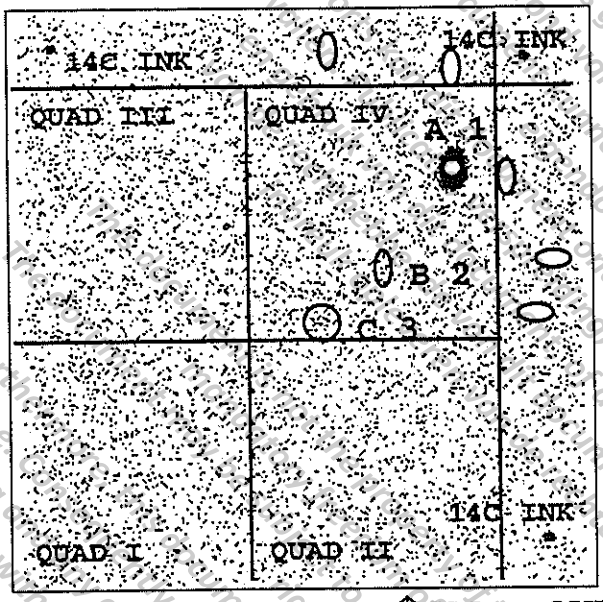
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 46: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

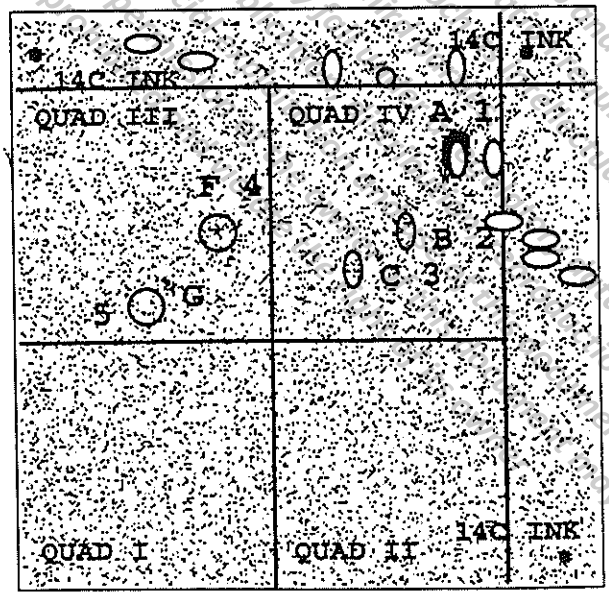
DAY 7 R1 IRRADIATED



AREA	PERCENT
A	86.85
B	0.87
C	0.66
QUAD I	0.39
QUAD II	0.30
QUAD III	<MQA
QUAD IV	0.43

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 7 R2 IRRADIATED



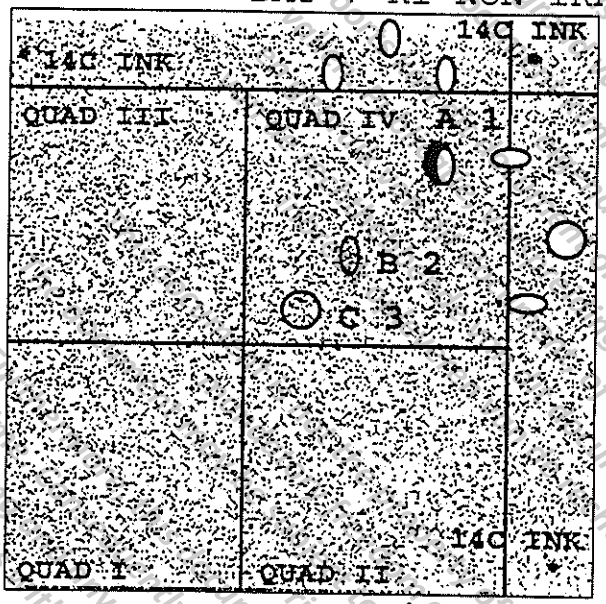
AREA	PERCENT
A	86.38
B	0.37
C	0.21
F	0.39
G	0.36
QUAD I	0.17
QUAD II	0.03
QUAD III	0.25
QUAD IV	0.23

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 47: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

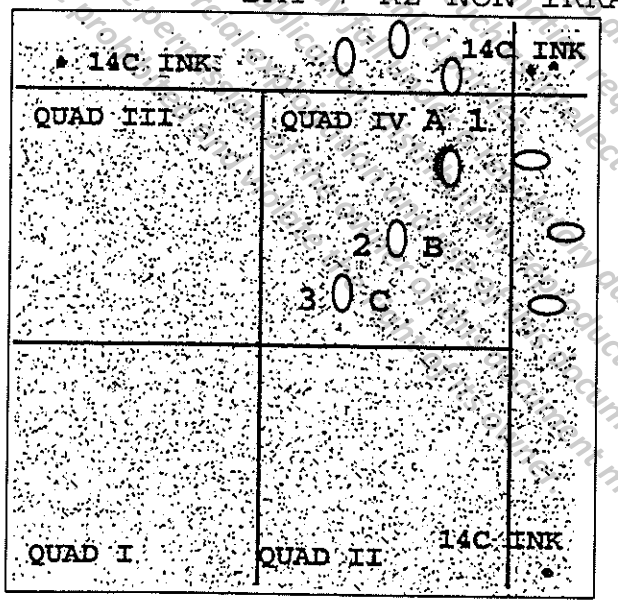
DAY 7 R1 NON-IRRADIATED



AREA	PERCENT
A	88.74
B	0.74
C	0.40
QUAD I	0.24
QUAD II	0.06
QUAD III	0.11
QUAD IV	0.33

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 7 R2 NON-IRRADIATED



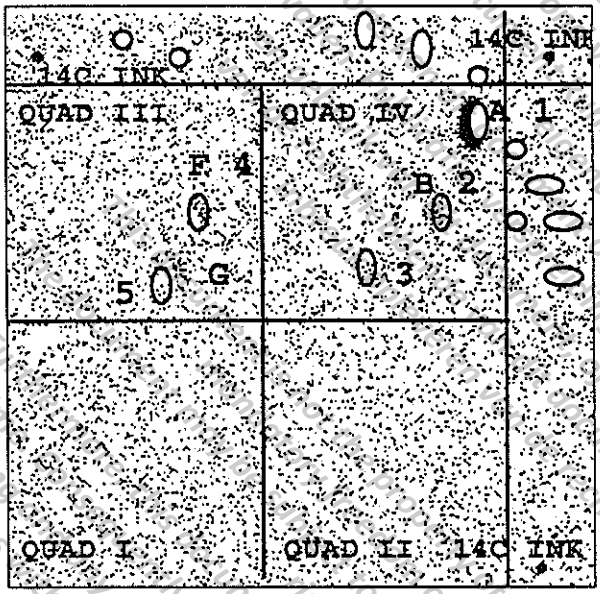
AREA	PERCENT
A	89.17
B	0.74
C	0.46
QUAD I	1.44
QUAD II	0.85
QUAD III	2.02
QUAD IV	1.22

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 48: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

DAY 14 R1 IRRADIATED

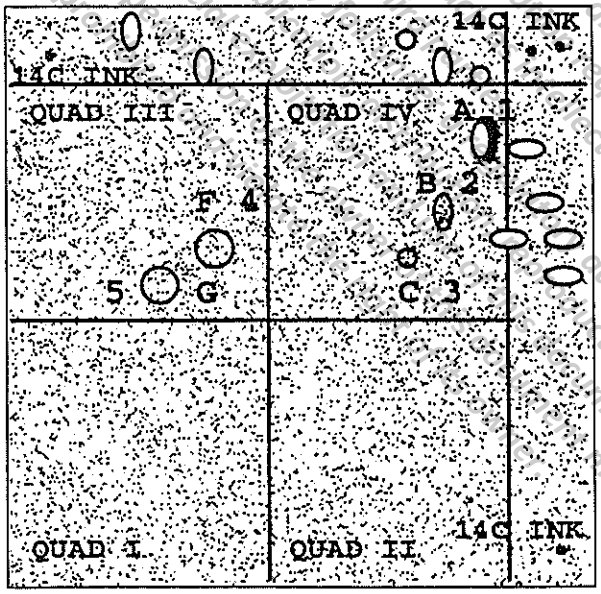


AREA	PERCENT
A	81.49
B	1.00
F	0.72
G	0.67
QUAD I	0.56
QUAD II	0.25
QUAD III	0.65
QUAD IV	1.03

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

DAY 14 R2 IRRADIATED



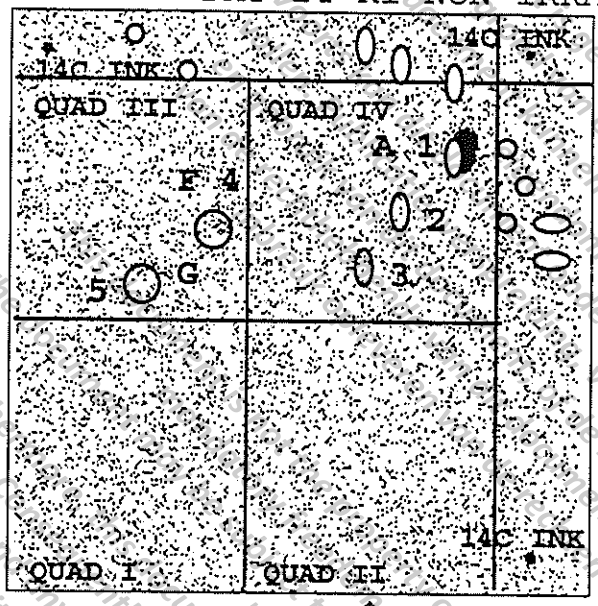
AREA	PERCENT
A	80.11
B	0.80
C	0.45
F	0.73
G	0.54
QUAD I	0.47
QUAD II	0.17
QUAD III	0.53
QUAD IV	0.67

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 49: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

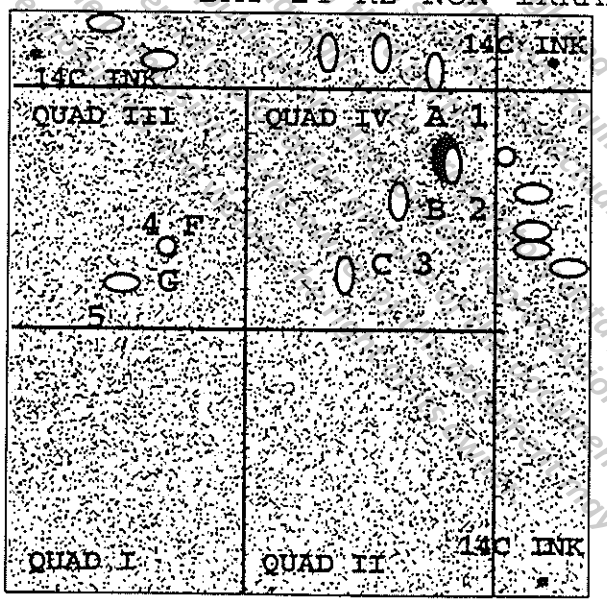
DAY 14 R1 NON-IRRADIATED



AREA	PERCENT
A	84.83
F	0.93
G	0.47
QUAD I	0.32
QUAD II	0.18
QUAD III	0.28
QUAD IV	0.60

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 NON-IRRADIATED



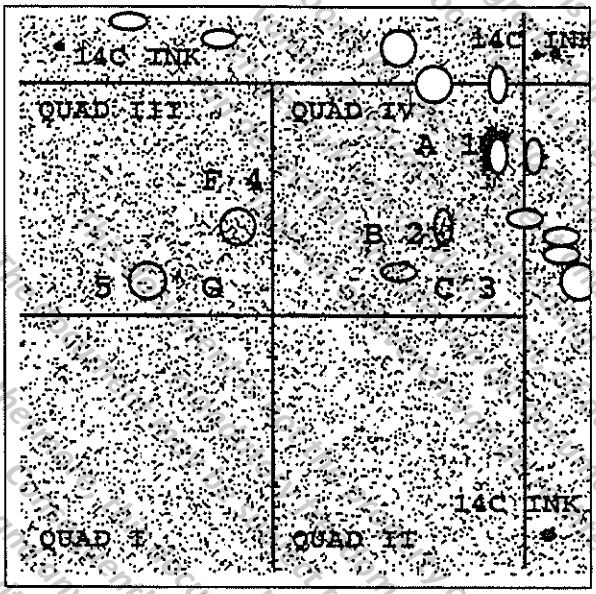
AREA	PERCENT
A	85.99
B	0.09
C	0.03
F	0.57
G	0.39
QUAD I	0.31
QUAD II	0.11
QUAD III	0.27
QUAD IV	0.20

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 50: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

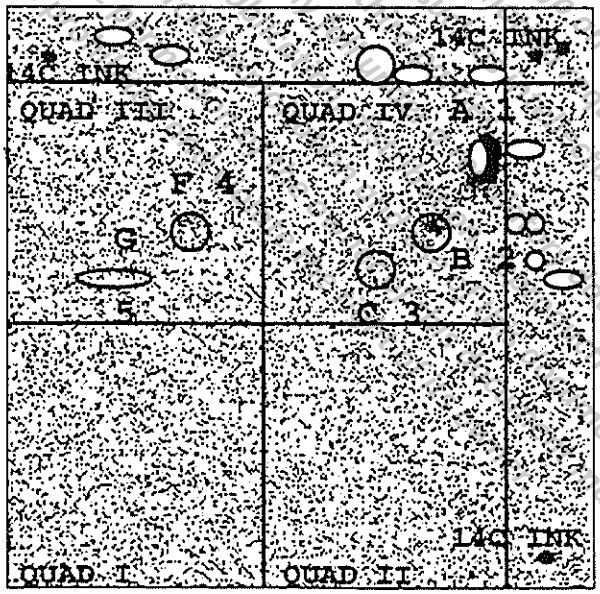
DAY 21 R1 IRRADIATED



AREA	PERCENT
A	80.46
B	0.86
C	0.30
F	0.68
G	0.44
QUAD I	0.40
QUAD II	0.14
QUAD III	0.36
QUAD IV	0.50

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 21 R2 IRRADIATED



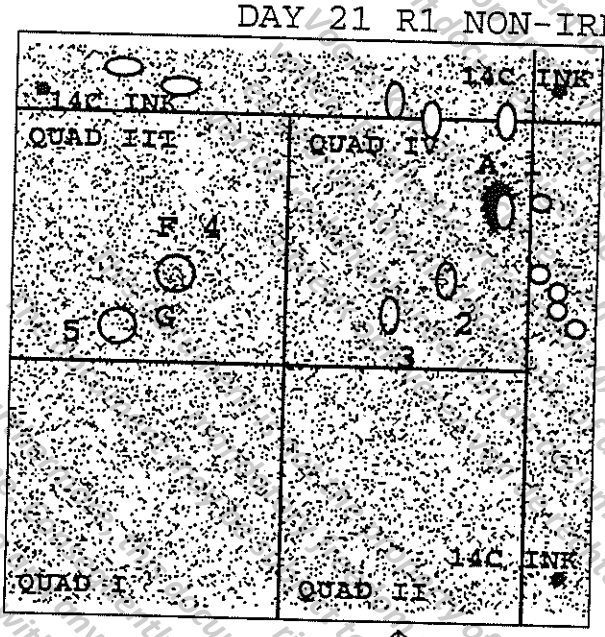
AREA	PERCENT
A	80.45
B	1.06
C	0.43
F	0.95
G	0.52
QUAD I	0.32
QUAD II	0.09
QUAD III	0.33
QUAD IV	0.40

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 51: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

DAY 21 R1 NON-IRRADIATED

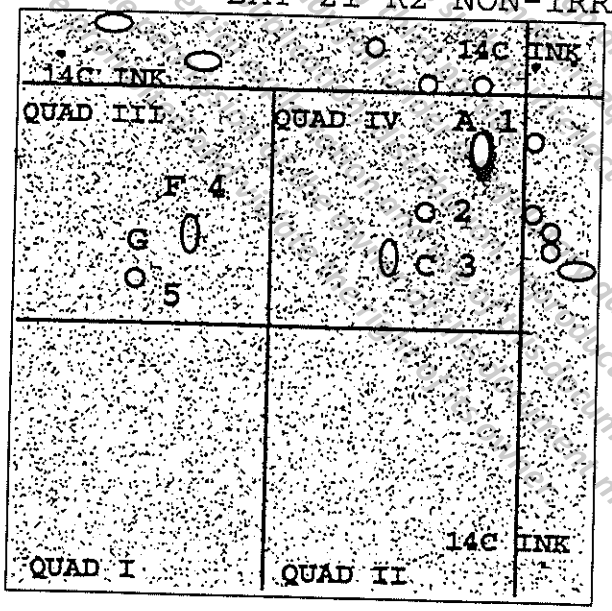


AREA	PERCENT
A	84.54
F	1.01
G	0.39
QUAD I	0.19
QUAD II	0.07
QUAD III	0.20
QUAD IV	0.36

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑ SS I → SSII

DAY 21 R2 NON-IRRADIATED



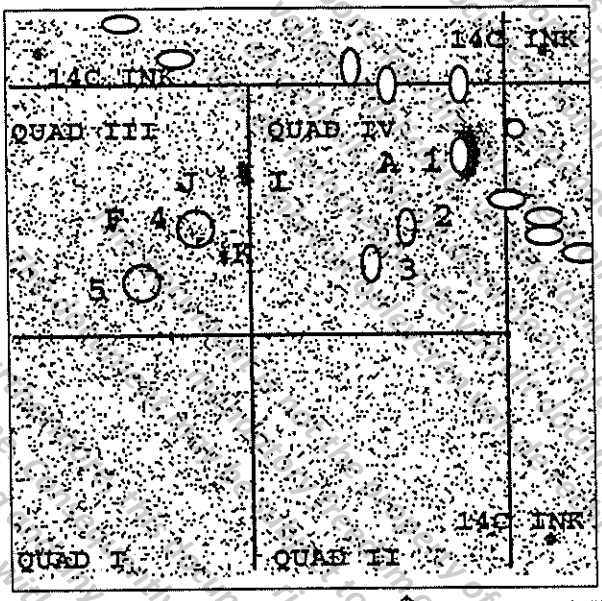
AREA	PERCENT
A	90.86
C	0.09
F	0.57
G	0.34
QUAD I	0.24
QUAD II	0.04
QUAD III	0.14
QUAD IV	0.22

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 52: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

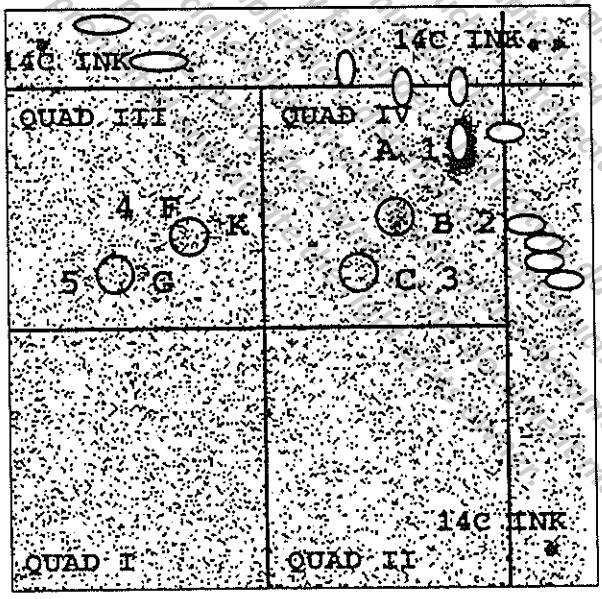
DAY 30 R1 IRRADIATED



AREA	PERCENT
A	84.64
F	0.59
I	2.21
J	0.35
K	1.07
QUAD I	0.49
QUAD II	0.02
QUAD III	0.69
QUAD IV	0.53

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 30 R2 IRRADIATED



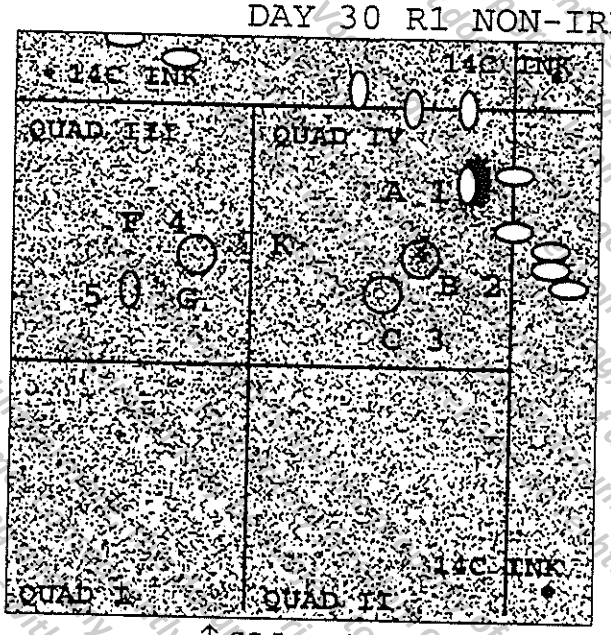
AREA	PERCENT
A	77.90
B	1.08
C	0.47
F	0.64
G	0.47
K	0.64
QUAD I	0.52
QUAD II	0.17
QUAD III	0.64
QUAD IV	0.82

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 53: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30 EXTRACTION 1, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 1

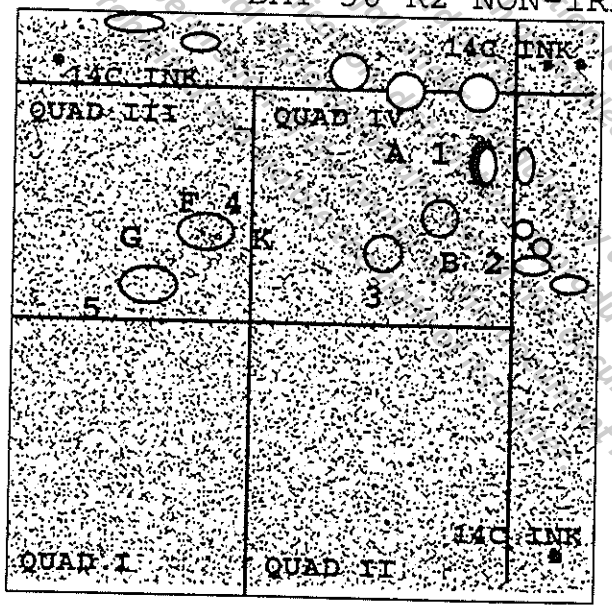
DAY 30 R1 NON-IRRADIATED



AREA	PERCENT
A	80.07
B	1.20
C	0.68
F	0.62
G	0.77
K	0.67
QUAD I	0.62
QUAD II	0.24
QUAD III	0.53
QUAD IV	0.94

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 30 R2 NON-IRRADIATED



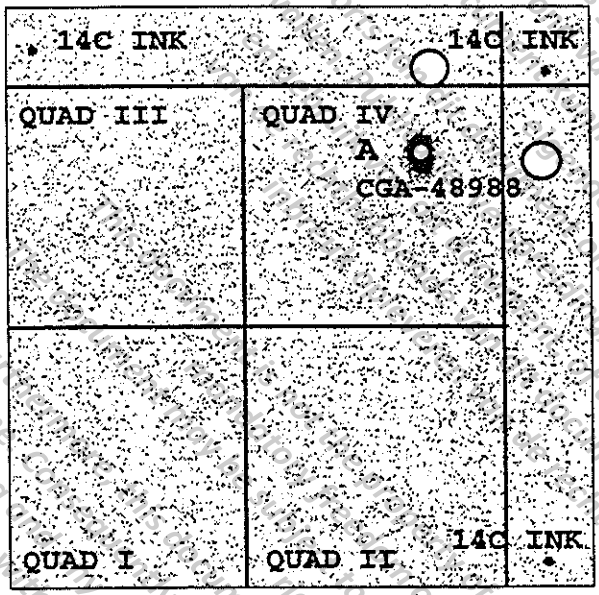
AREA	PERCENT
A	84.58
B	0.11
F	0.51
G	0.36
K	0.33
QUAD I	0.29
QUAD II	0.09
QUAD III	0.20
QUAD IV	0.45

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 54: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30 EXTRACTION 1, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

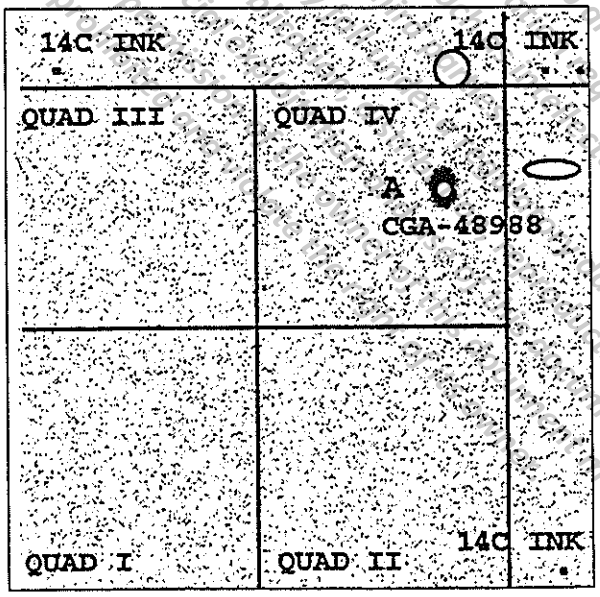
CGA-48988 EXTRACTION 2

DAY 0 R1 IRRADIATED



AREA	PERCENT
A	2.32
QUAD I	0.04
QUAD II	0.01
QUAD III	0.02
QUAD IV	0.06

DAY 0 R2 IRRADIATED



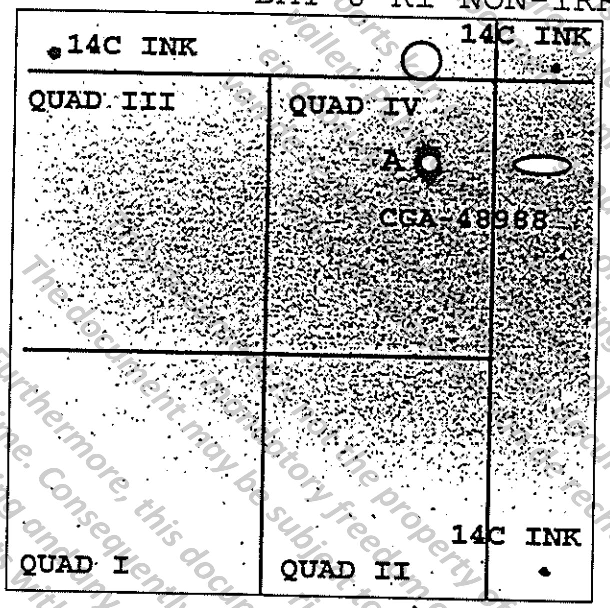
AREA	PERCENT
A	2.78
QUAD I	0.03
QUAD II	0.03
QUAD III	0.01
QUAD IV	0.04

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 55: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

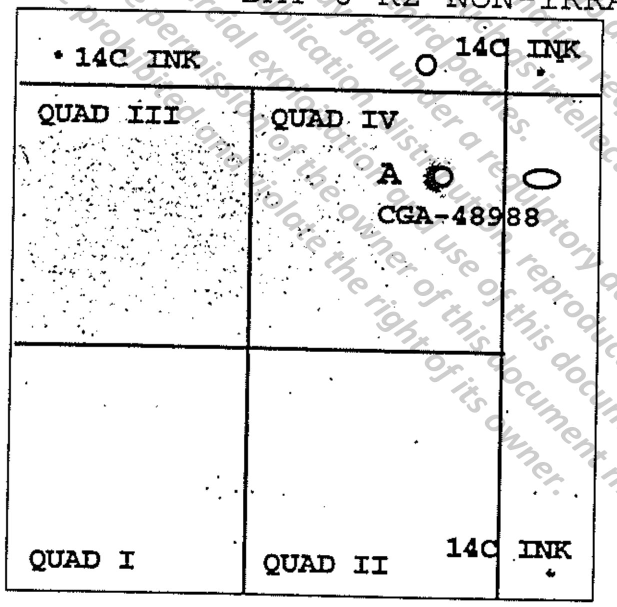
DAY 0 R1 NON-IRRADIATED



↑ SS I → SSII

AREA	PERCENT
A	4.27
QUAD I	0.04
QUAD II	0.03
QUAD III	0.03
QUAD IV	0.07

DAY 0 R2 NON-IRRADIATED



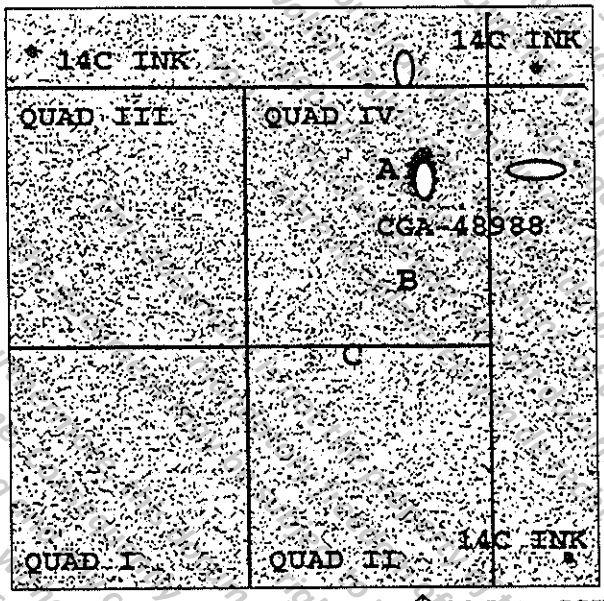
AREA	PERCENT
A	1.94
QUAD I	0.04
QUAD II	0.03
QUAD III	0.03
QUAD IV	0.05

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 56: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 0 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

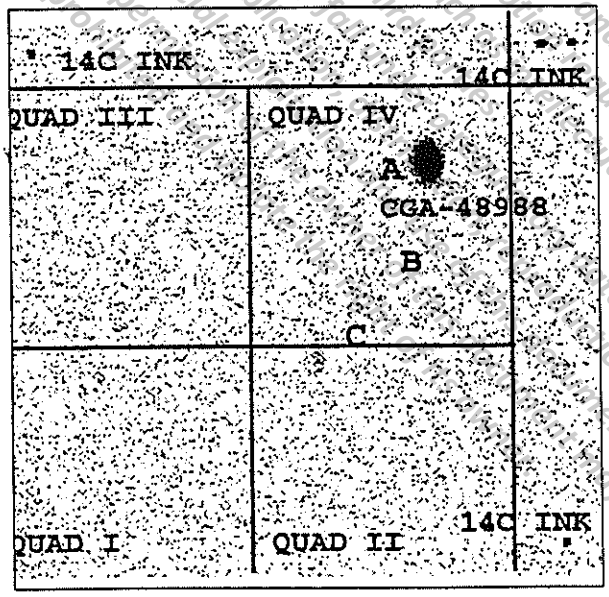
CGA-48988 EXTRACTION 2

DAY 3 R1 IRRADIATED



AREA	PERCENT
A	2.84
B	0.03
C	0.09
QUAD I	0.06
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.01

DAY 3 R2 IRRADIATED



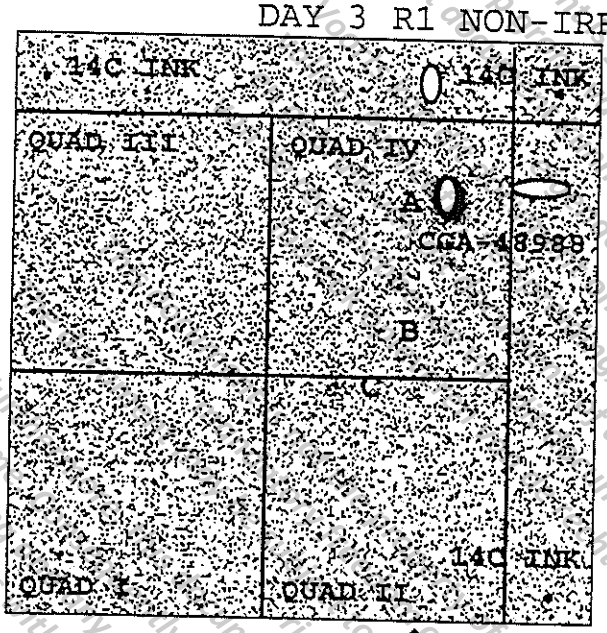
AREA	PERCENT
A	2.82
B	0.03
C	0.07
QUAD I	0.03
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.02

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 57: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

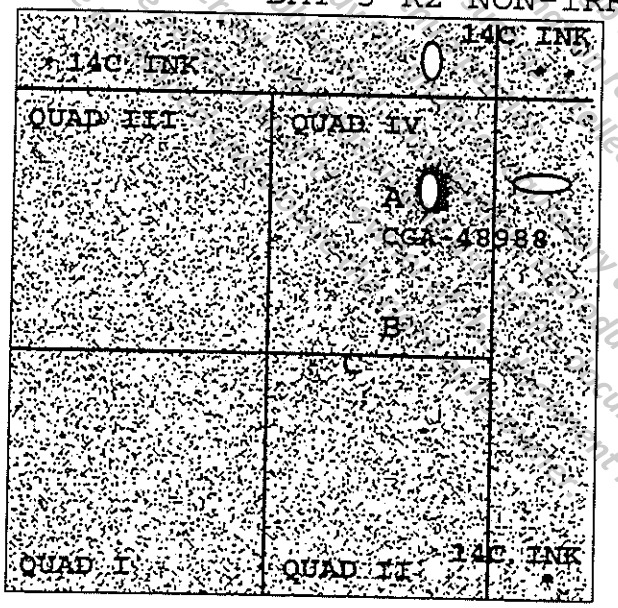
CGA-48988 EXTRACTION 2

DAY 3 R1 NON-IRRADIATED



AREA	PERCENT
A	3.33
B	0.03
C	0.06
QUAD I	0.04
QUAD II	0.03
QUAD III	0.04
QUAD IV	0.07

DAY 3 R2 NON-IRRADIATED



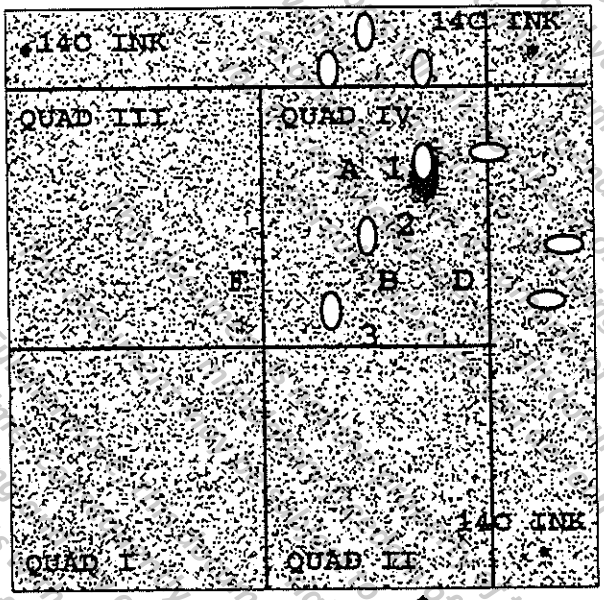
AREA	PERCENT
A	3.15
B	0.02
C	0.05
QUAD I	0.02
QUAD II	0.01
QUAD III	0.02
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 58: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 3 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

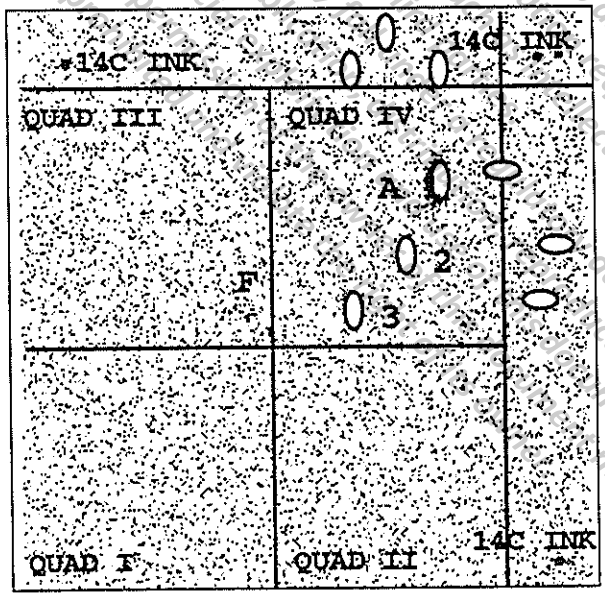
CGA-48988 EXTRACTION 2

DAY 7 R1 IRRADIATED



AREA	PERCENT
A	6.01
B	0.03
D	0.03
F	0.08
QUAD I	0.04
QUAD II	0.02
QUAD III	0.02
QUAD IV	0.07

DAY 7 R2 IRRADIATED



AREA	PERCENT
A	4.49
F	0.09
QUAD I	0.07
QUAD II	0.03
QUAD III	0.04
QUAD IV	0.13

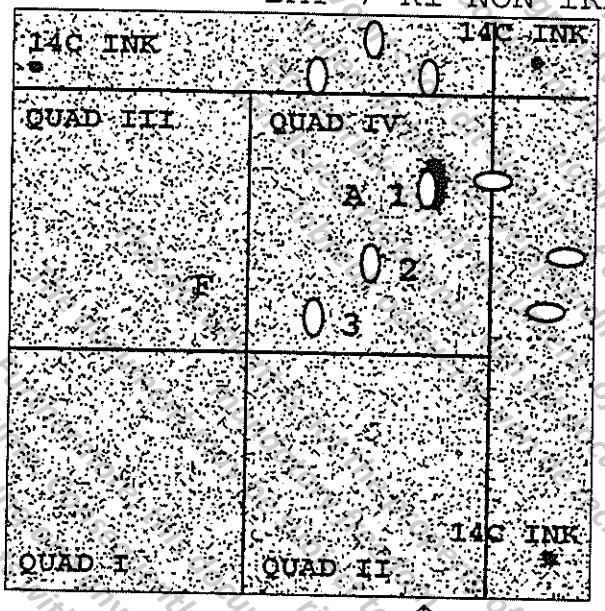
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 59: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

DAY 7 R1 NON-IRRADIATED

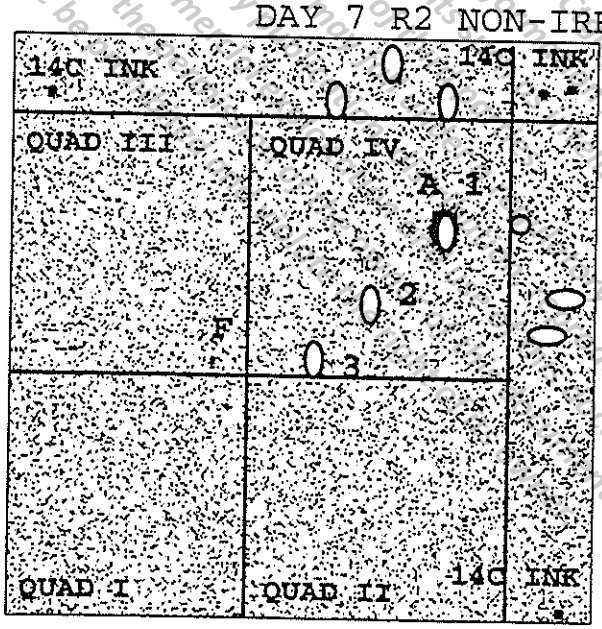


AREA	PERCENT
A	4.85
F	0.07
QUAD I	0.04
QUAD II	0.01
QUAD III	0.05
QUAD IV	0.06

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

↑SS I→ SSII

DAY 7 R2 NON-IRRADIATED



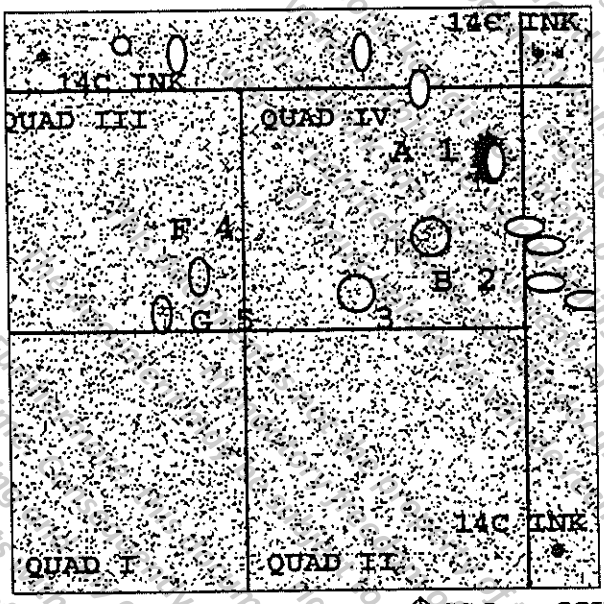
AREA	PERCENT
A	5.43
F	0.08
QUAD I	0.05
QUAD II	0.03
QUAD III	0.05
QUAD IV	0.09

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 60: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 7 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

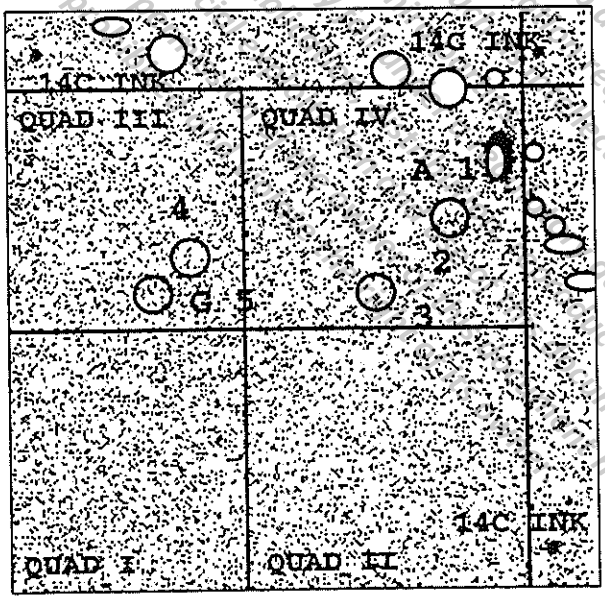
DAY 14 R1 IRRADIATED



AREA	PERCENT
A	3.35
B	0.02
F	0.02
G	0.03
QUAD I	0.04
QUAD II	< 0.01
QUAD III	0.01
QUAD IV	0.02

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 IRRADIATED



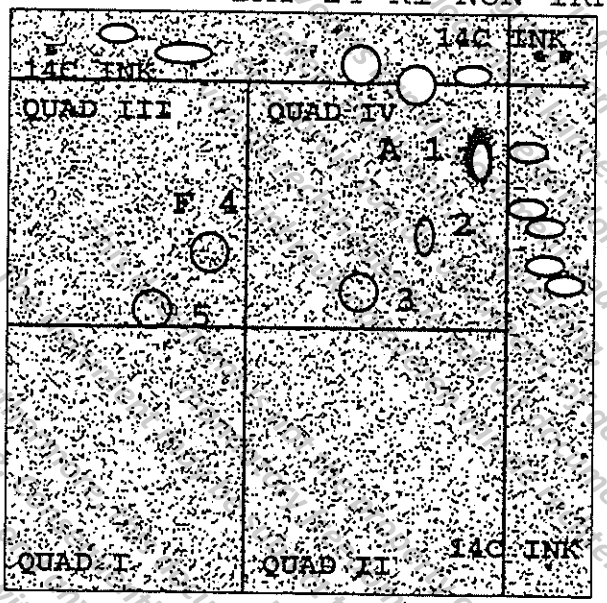
AREA	PERCENT
A	4.82
G	0.05
QUAD I	0.07
QUAD II	< 0.01
QUAD III	0.05
QUAD IV	0.09

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 61: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

DAY 14 R1 NON-IRRADIATED

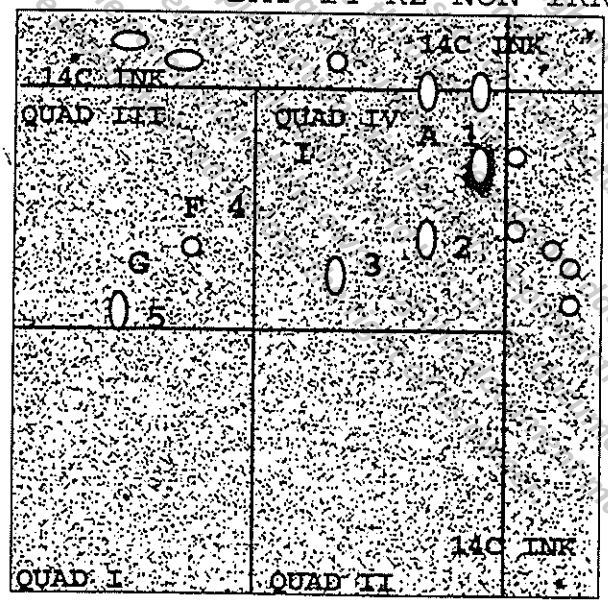


↑SS I→ SSII

AREA	PERCENT
A	5.31
F	0.03
QUAD I	0.02
QUAD II	0.01
QUAD III	0.01
QUAD IV	0.02

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

DAY 14 R2 NON-IRRADIATED



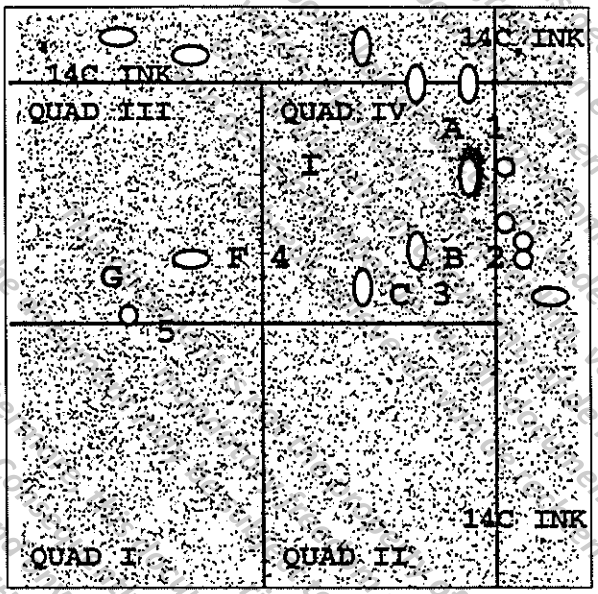
AREA	PERCENT
A	4.47
F	0.05
G	0.06
I	0.02
QUAD I	0.06
QUAD II	0.02
QUAD III	0.05
QUAD IV	0.03

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 62: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 14 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

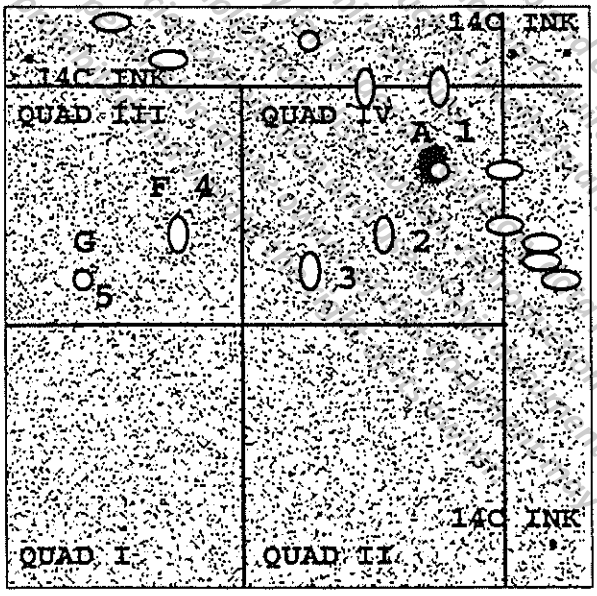
CGA-48988 EXTRACTION 2

DAY 21 R1 IRRADIATED



AREA	PERCENT
A	4.08
B	0.06
C	0.03
F	0.09
G	0.06
I	0.03
QUAD I	< 0.01
QUAD II	0.18
QUAD III	0.03
QUAD IV	0.07

DAY 21 R2 IRRADIATED



AREA	PERCENT
A	4.06
F	0.04
G	0.04
QUAD I	0.10
QUAD II	0.03
QUAD III	0.07
QUAD IV	0.12

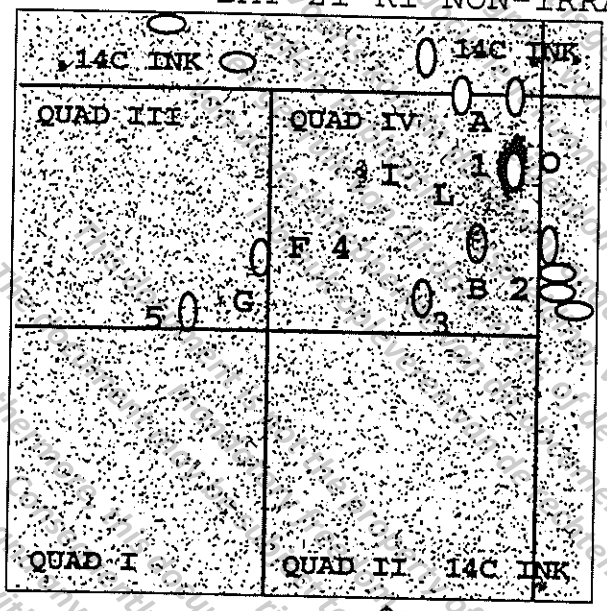
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 63: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

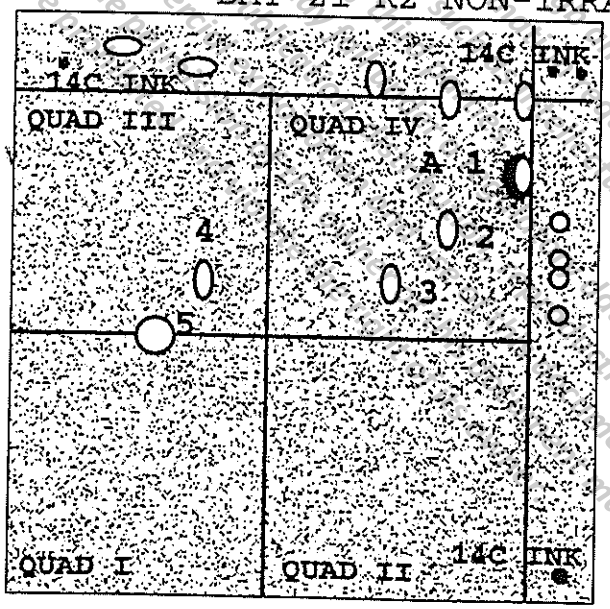
CGA-48988 EXTRACTION 2

DAY 21 R1 NON-IRRADIATED



AREA	PERCENT
A	4.38
B	0.07
F	0.10
G	0.06
I	0.03
L	0.04
QUAD I	0.19
QUAD II	0.03
QUAD III	0.07
QUAD IV	0.06

DAY 21 R2 NON-IRRADIATED



AREA	PERCENT
A	4.98
QUAD I	0.03
QUAD II	< MQA
QUAD III	0.10
QUAD IV	0.04

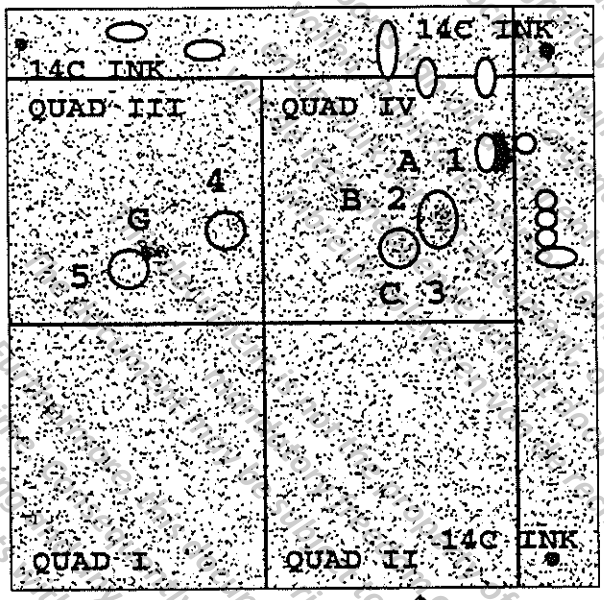
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 64: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 21 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

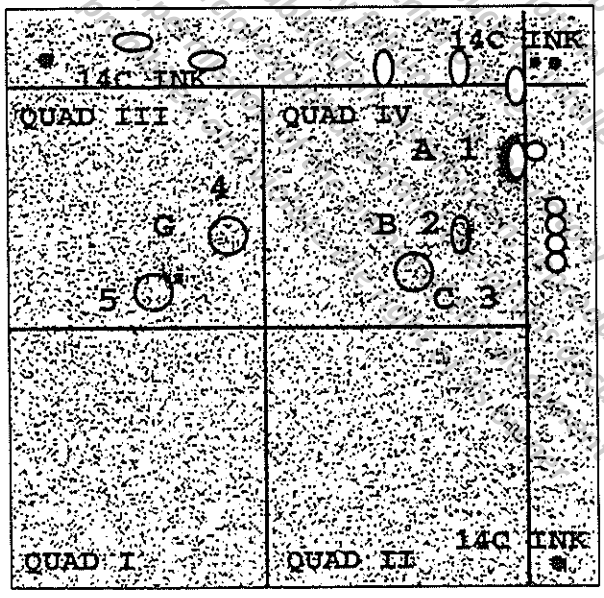
DAY 30 R1 IRRADIATED



↑SS I → SSII

AREA	PERCENT
A	1.13
B	0.04
C	0.02
G	0.09
QUAD I	0.10
QUAD II	0.01
QUAD III	0.07
QUAD IV	0.03

DAY 30 R2 IRRADIATED



AREA	PERCENT
A	3.70
B	0.09
C	0.04
G	0.15
QUAD I	0.14
QUAD II	0.02
QUAD III	0.15
QUAD IV	0.06

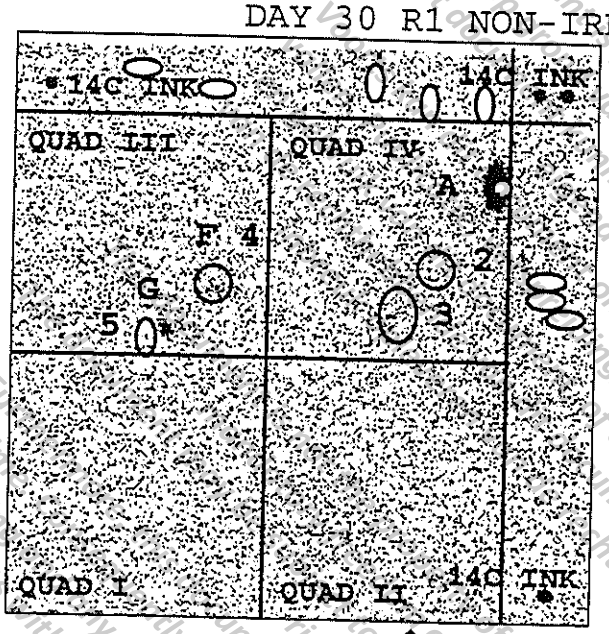
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

FIGURE 65: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30 EXTRACTION 2, IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988

CGA-48988 EXTRACTION 2

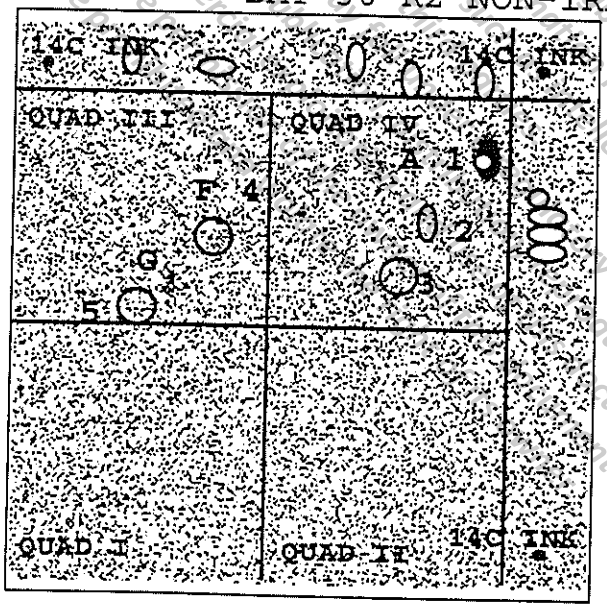
DAY 30 R1 NON-IRRADIATED



↑ SS I → SSII

AREA	PERCENT
A	4.97
F	0.07
G	0.10
QUAD I	0.04
QUAD II	< MQA
QUAD III	0.02
QUAD IV	0.05

DAY 30 R2 NON-IRRADIATED

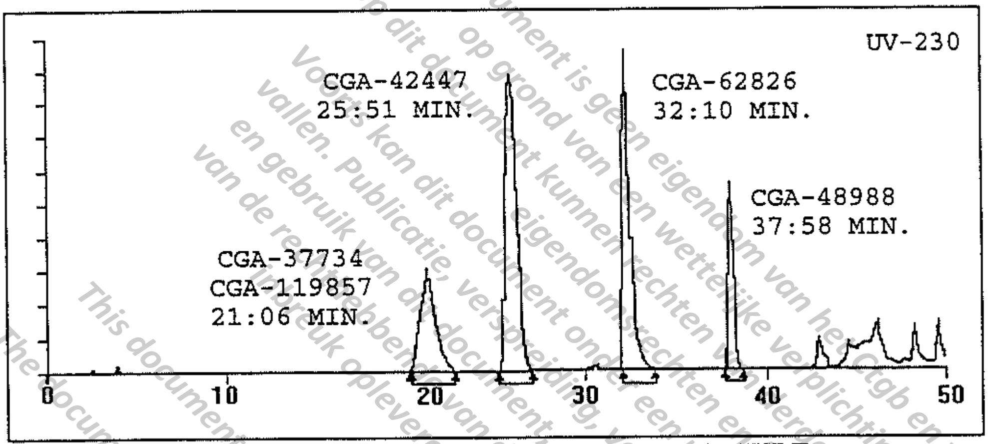


AREA	PERCENT
A	3.33
F	0.04
G	0.08
QUAD I	0.04
QUAD II	0.01
QUAD III	0.04
QUAD IV	0.04

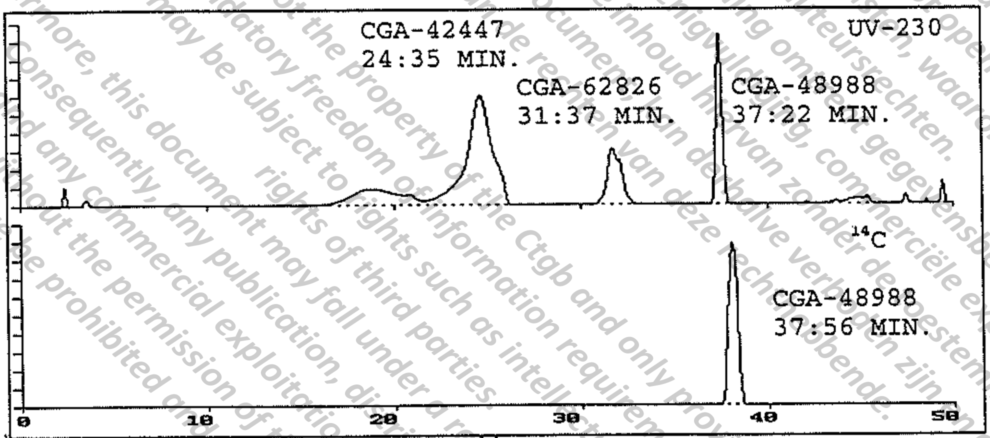
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT EQUALS THE PERCENT OF TOTAL DOSE

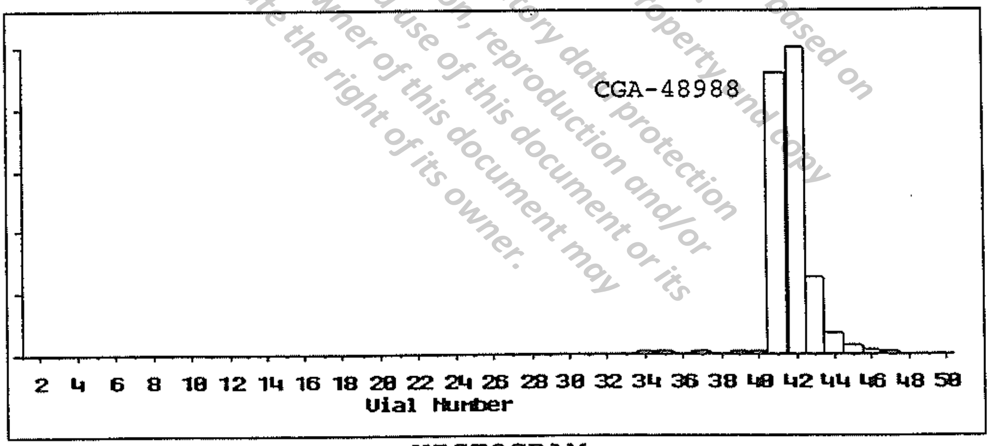
FIGURE 66: TWO DIMENSIONAL THIN LAYER CHROMATOGRAPHY OF DAY 30 EXTRACTION 2, NON-IRRADIATED, REPLICATES 1 AND 2 OF CGA-48988



UV TRACE OF THE STANDARD MIXTURE



UV AND ^{14}C TRACES



HISTOGRAM

FIGURE 67: <u>HPLC OF DAY 0, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-48988</u>

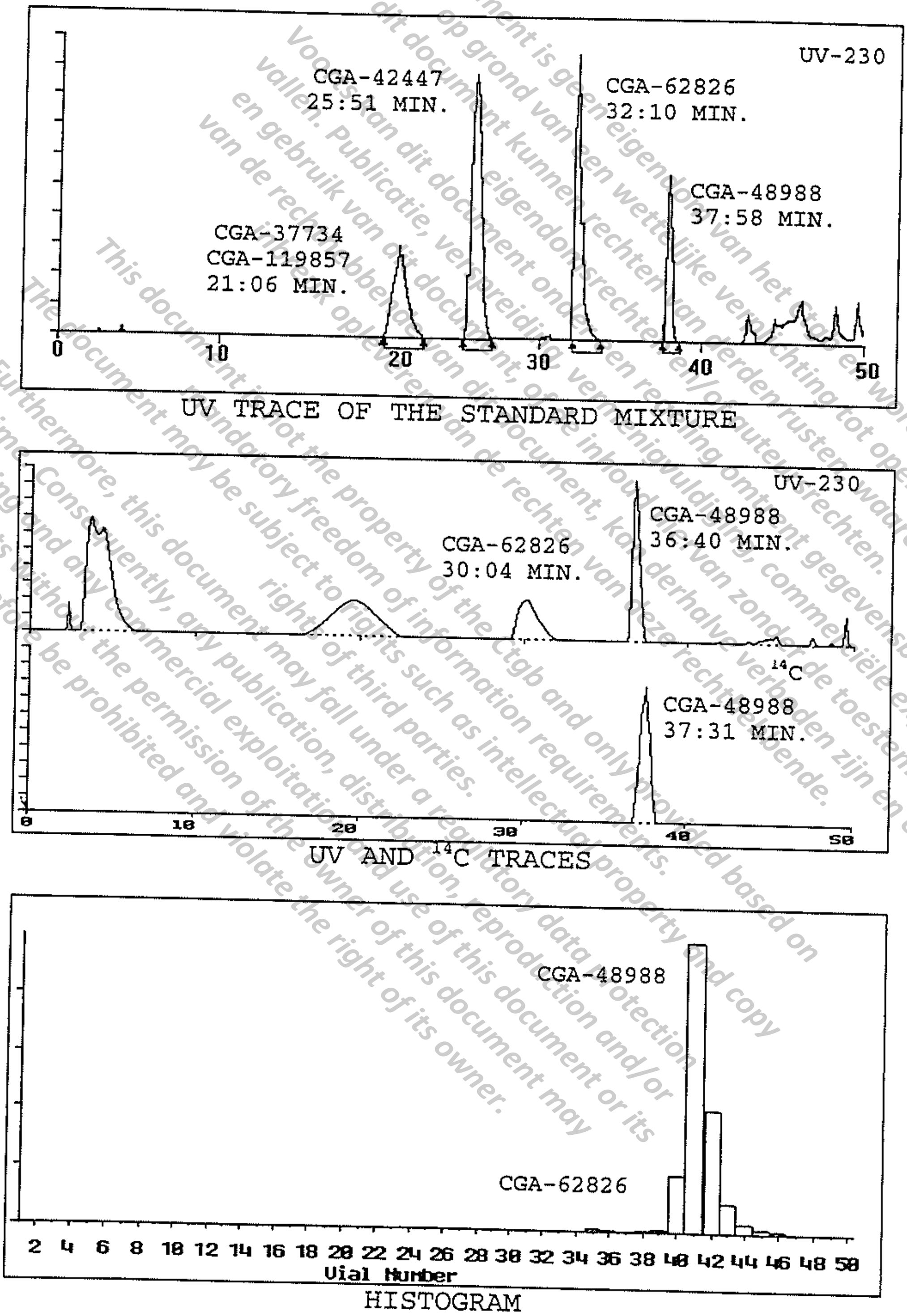
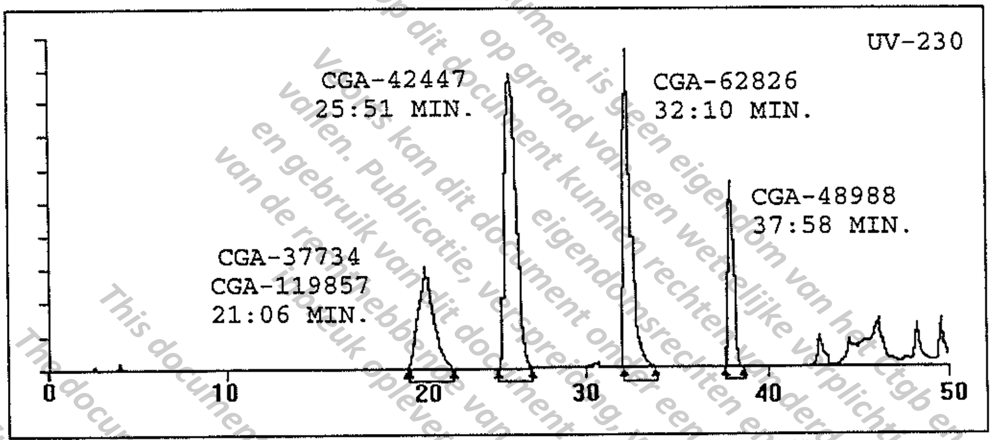
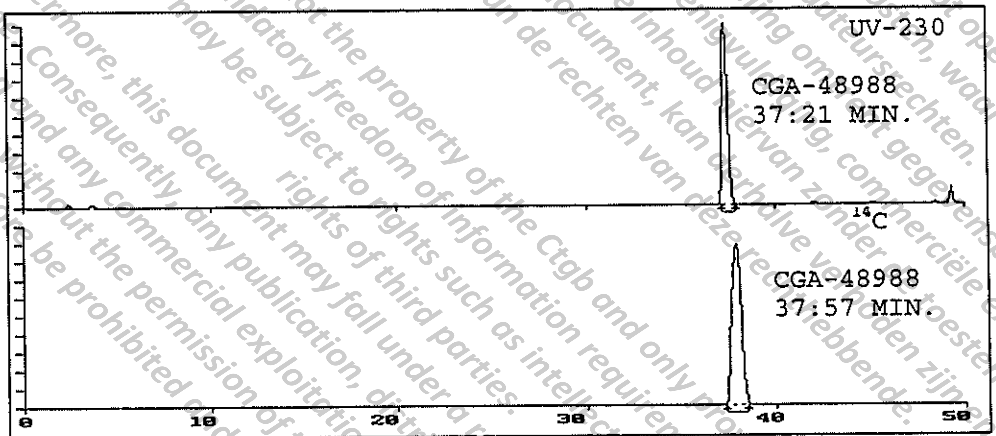


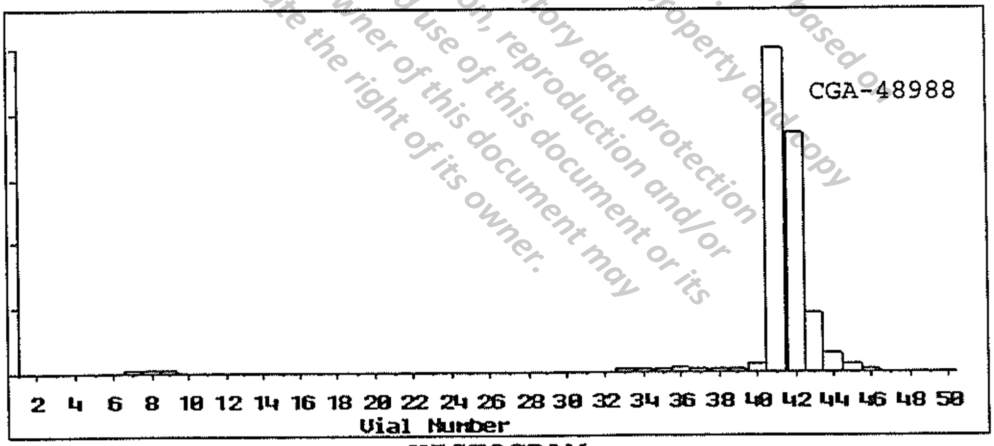
FIGURE 68: HPLC OF DAY 0, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-48988



UV TRACE OF THE STANDARD MIXTURE

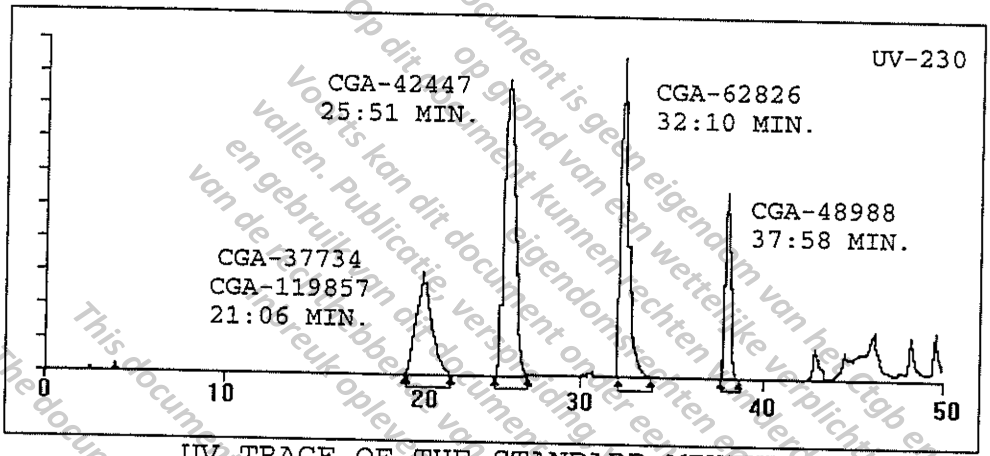


UV AND ^{14}C TRACES

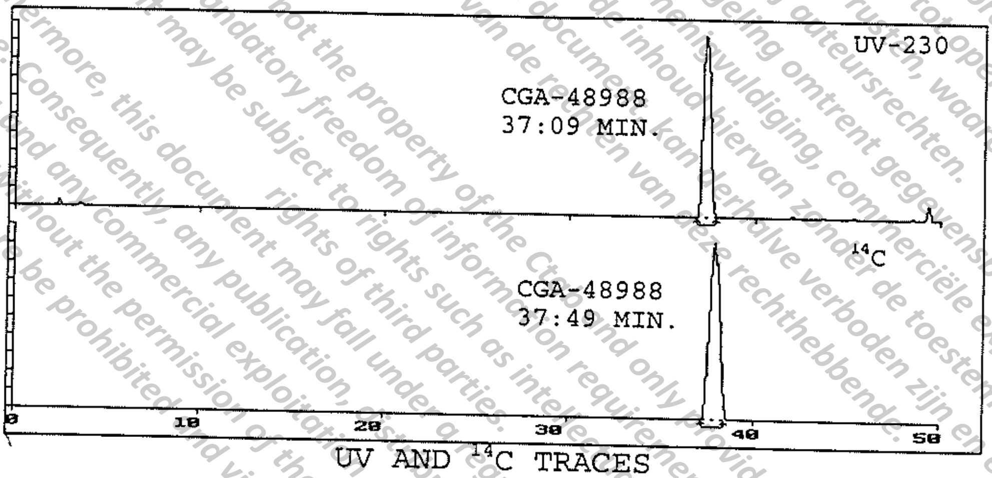


HISTOGRAM

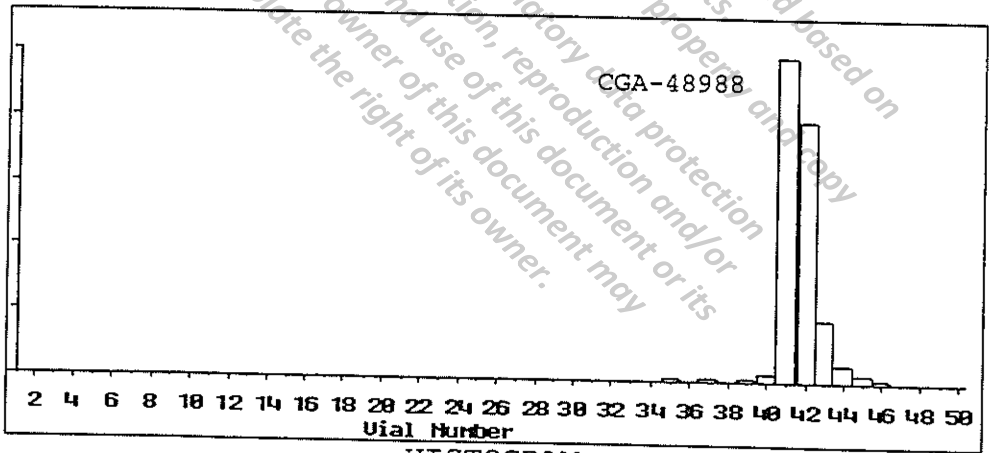
FIGURE 69: HPLC OF DAY 14, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-48988



UV TRACE OF THE STANDARD MIXTURE

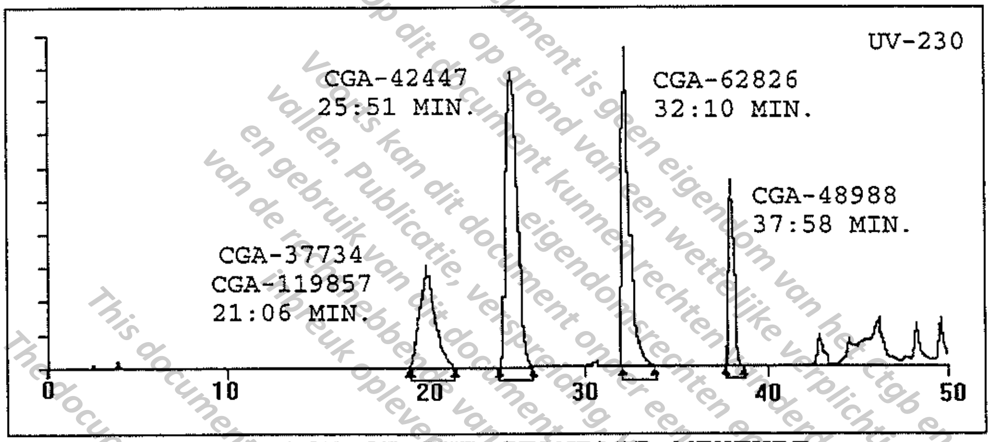


UV AND ^{14}C TRACES

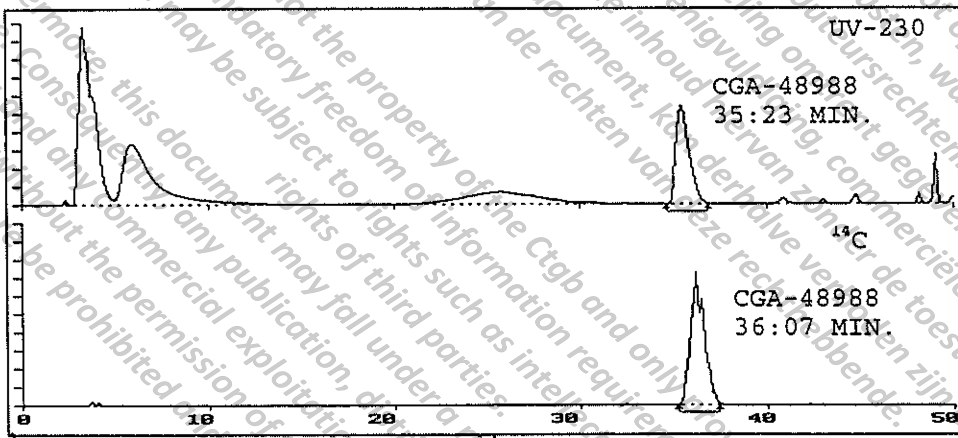


HISTOGRAM

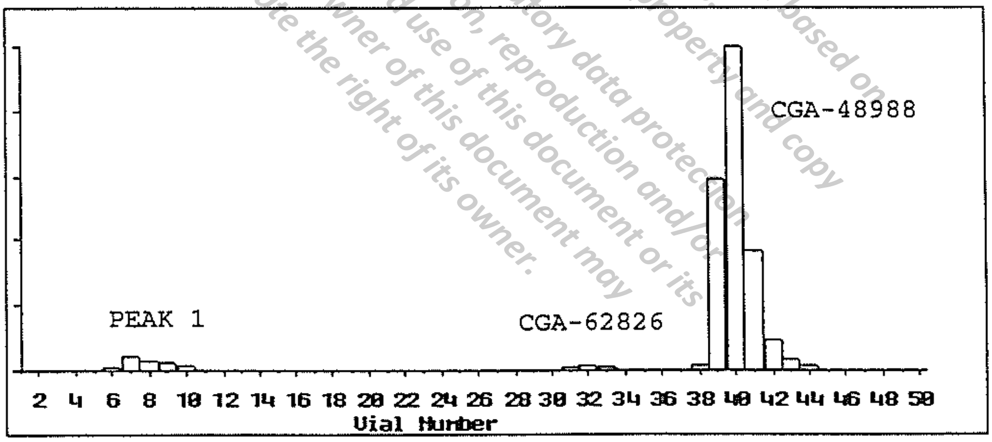
FIGURE 70: HPLC OF DAY 14, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-48988



UV TRACE OF THE STANDARD MIXTURE

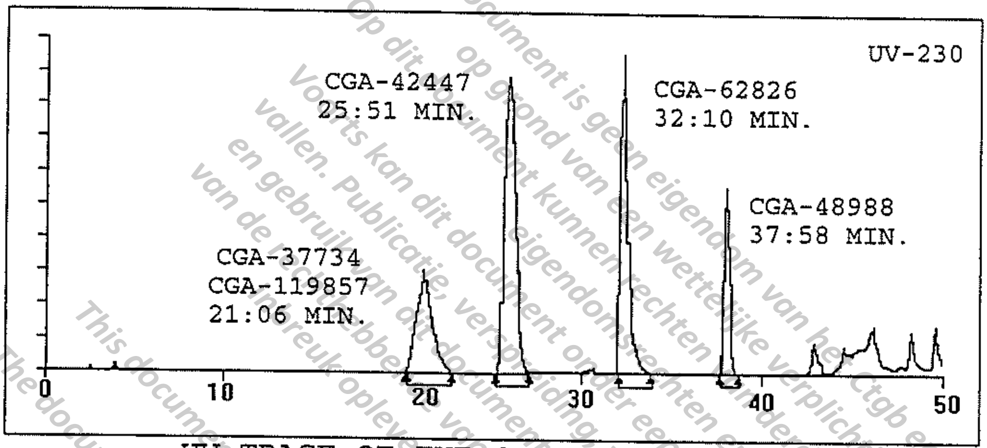


UV AND ^{14}C TRACES

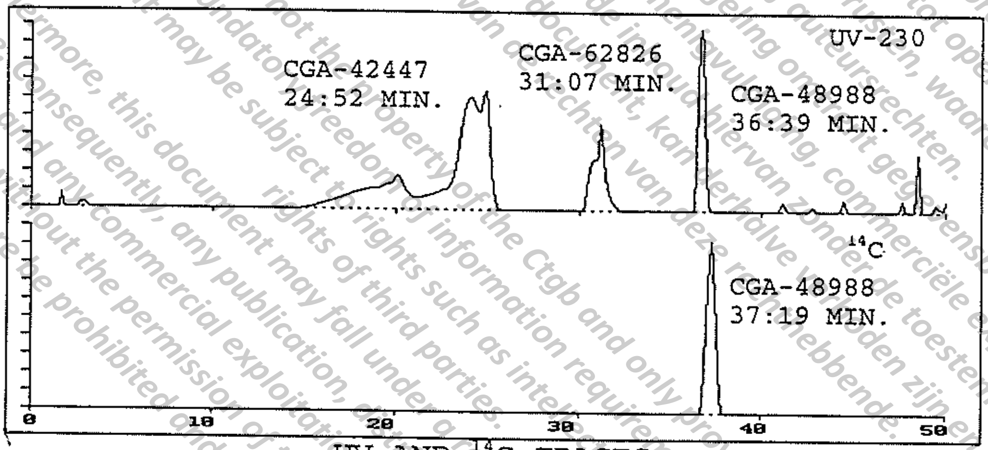


HISTOGRAM

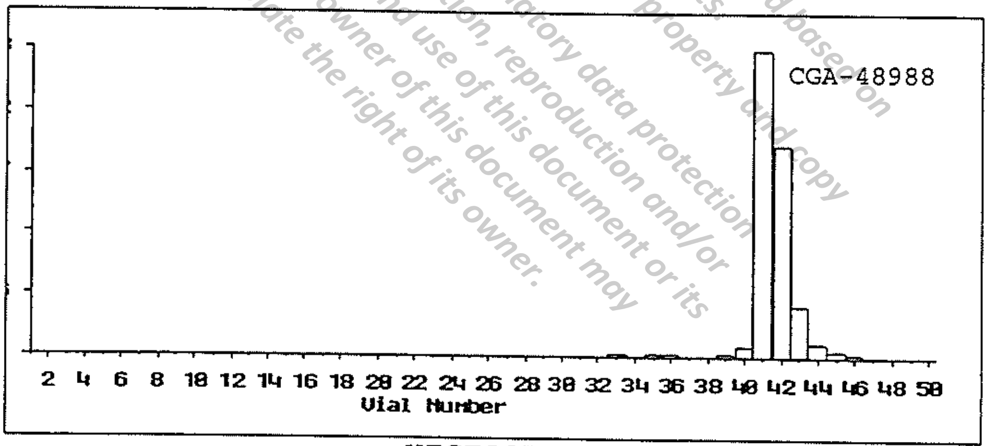
FIGURE 71: HPLC OF DAY 30, REPLICATE 1, IRRADIATED, EXTRACT 1 OF CGA-48988



UV TRACE OF THE STANDARD MIXTURE



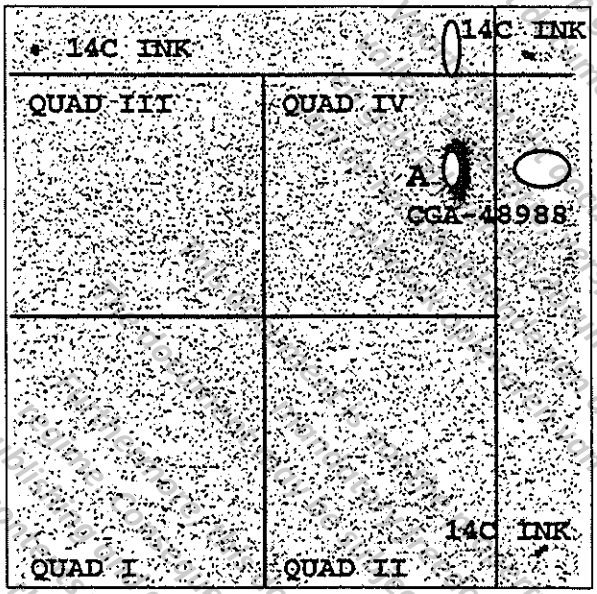
UV AND ^{14}C TRACES



HISTOGRAM

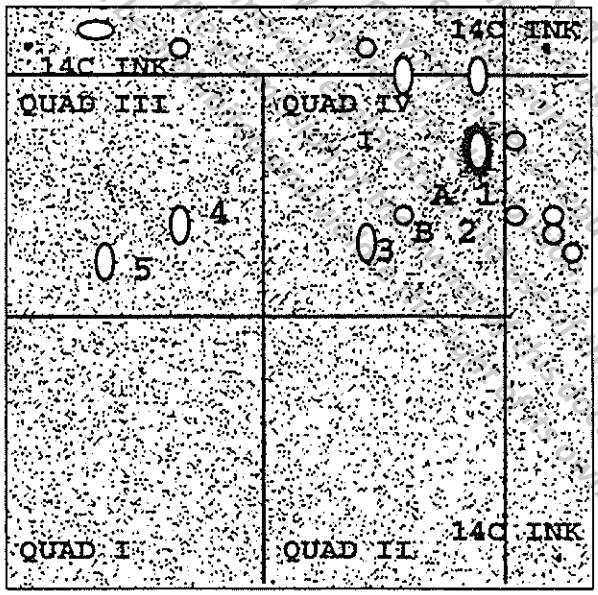
FIGURE 72: HPLC OF DAY 30, REPLICATE 1, NON-IRRADIATED, EXTRACT 1 OF CGA-48988

APRIL 5, 1995



AREA	PERCENT
A	91.85
QUAD I	0.08
QUAD II	0.06
QUAD III	0.10
QUAD IV	0.60

JULY 26, 1995



AREA	PERCENT
A	90.28
B	0.33
I	0.16
QUAD I	0.13
QUAD II	0.08
QUAD III	0.50
QUAD IV	0.26

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

PERCENT = PERCENT RECOVERED OF THAT APPLIED

FIGURE 73: TLC ANALYSIS OF DAY 0 REPLICATE 2 NON-IRRADIATED, EXTRACTION 1 OF CGA-48988 AT THE BEGINNING AND THE END OF THE STUDY TO SHOW SAMPLE STORAGE STABILITY UNDER FREEZER CONDITIONS

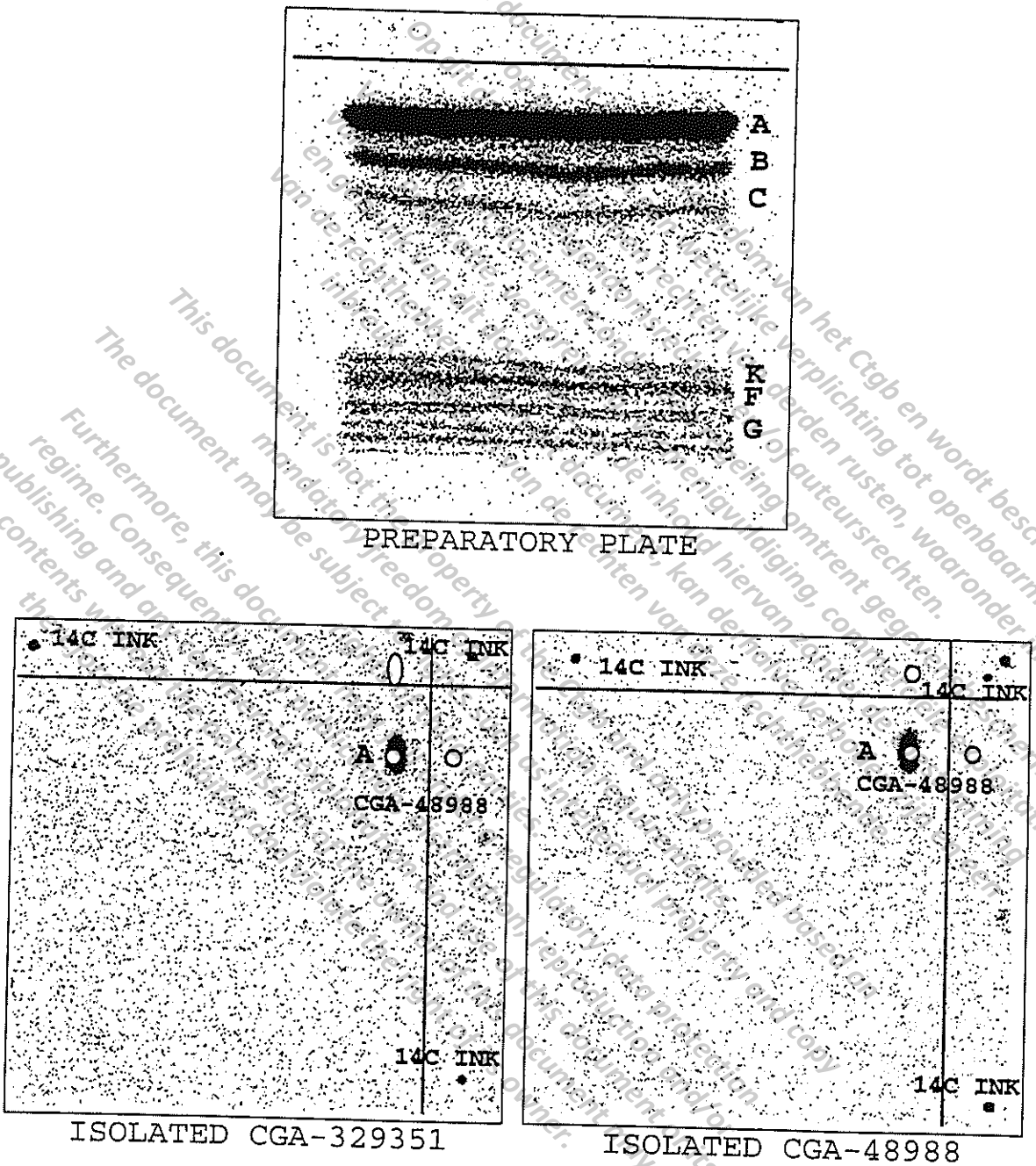
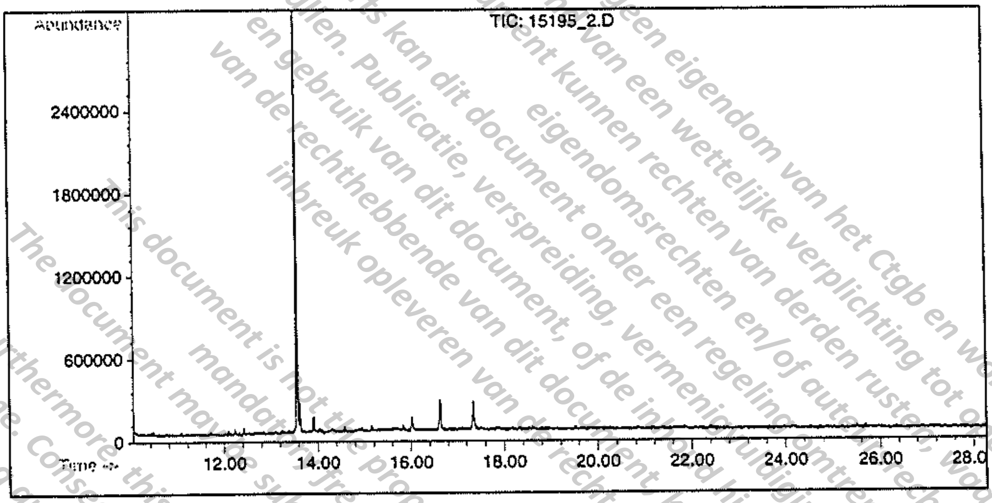
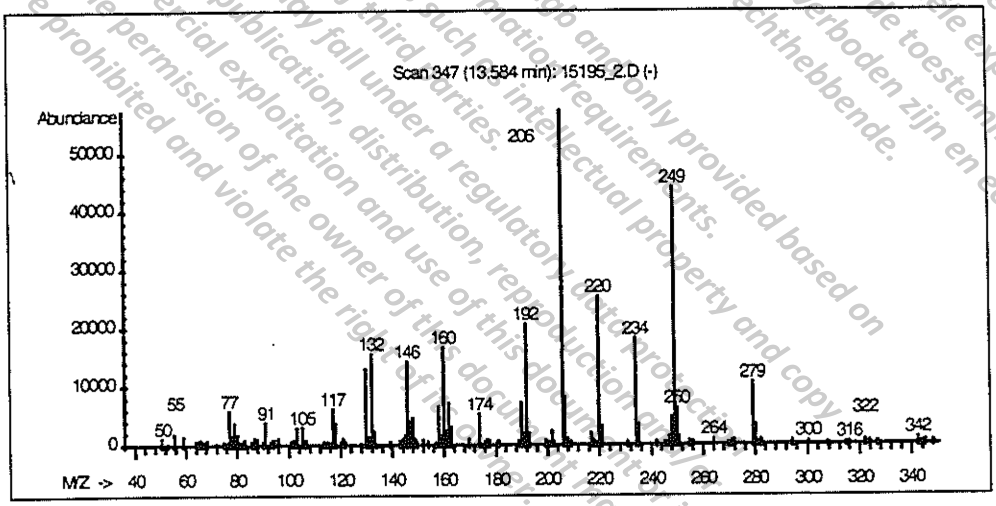


FIGURE 74: REPRESENTATIVE PREPARATORY TLC PLATE AND THE TWO DIMENSIONAL TLC OF ISOLATED CGA-329351 AND CGA-48988 (COMPONENT A)

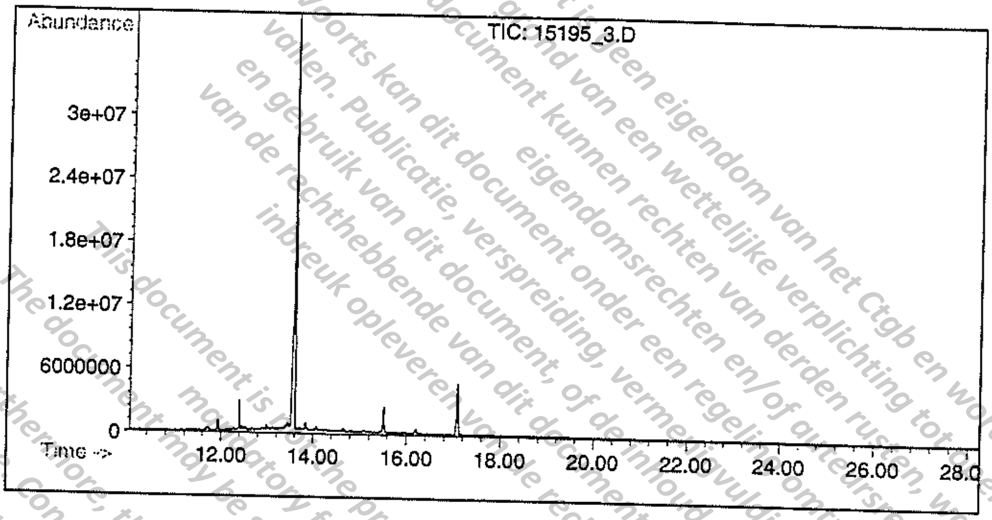


GC CHROMATOGRAM

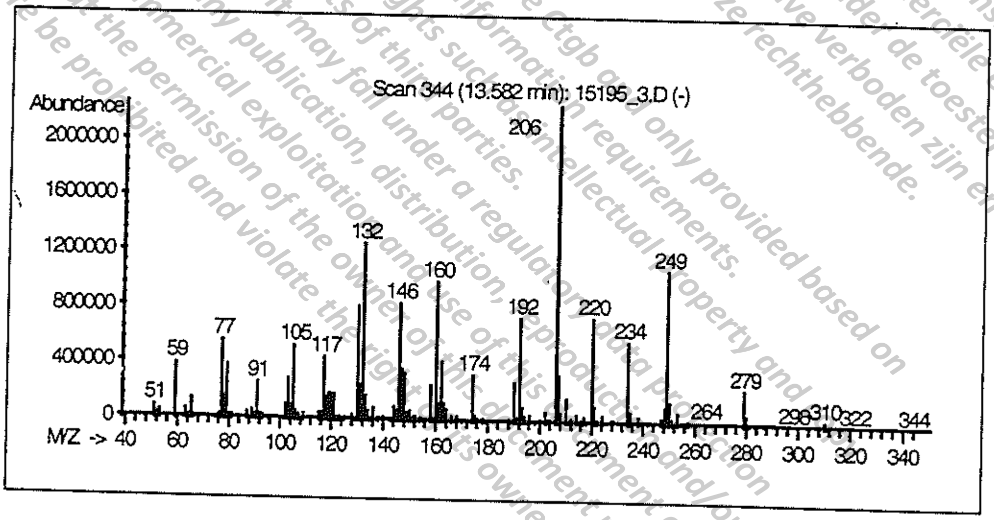


MSD SCAN AT 13.584 MINUTES

FIGURE 75: <u>GC/MSD OF CGA-48988 REFERENCE STANDARD</u>

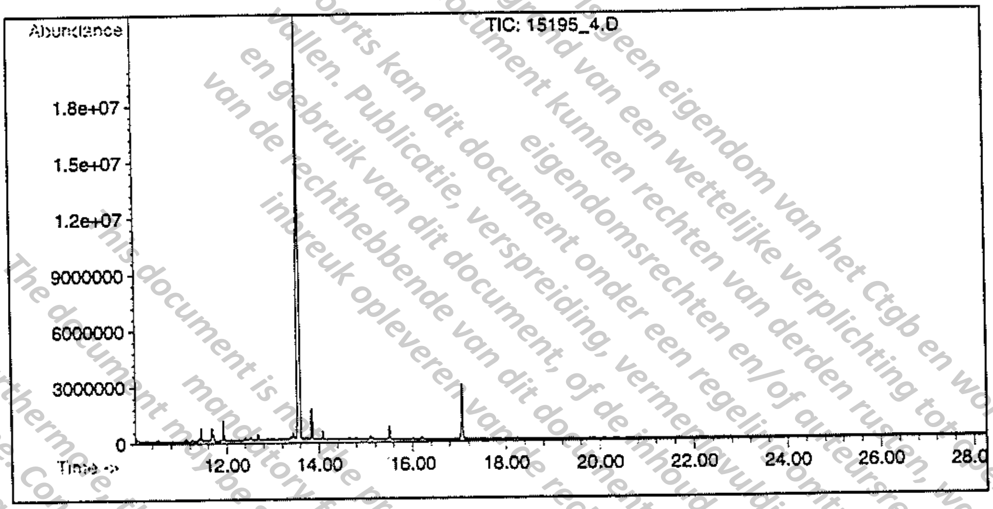


GC CHROMATOGRAM

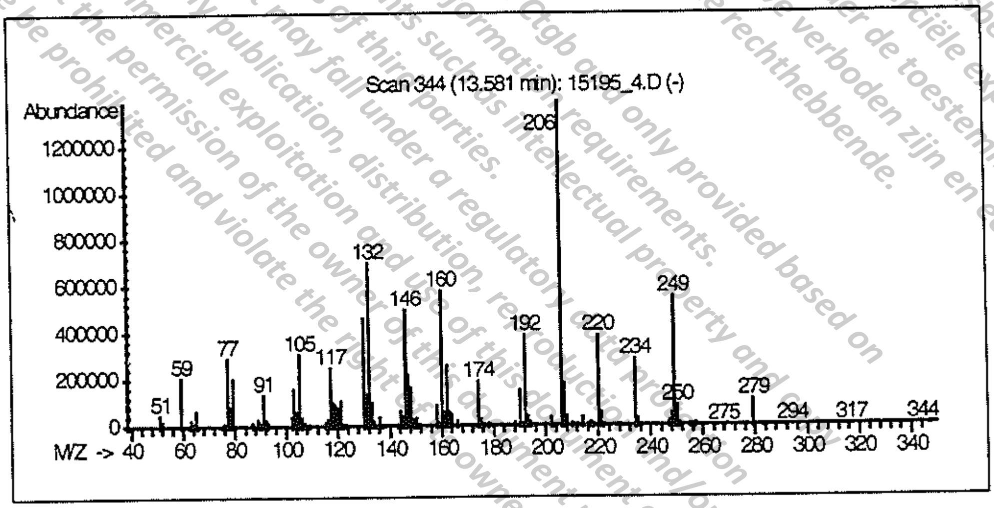


MSD SCAN AT 13.582 MINUTES

FIGURE 76: GC/MSD OF CGA-48988, EXPERIMENT 2, ISOLATED COMPONENT A

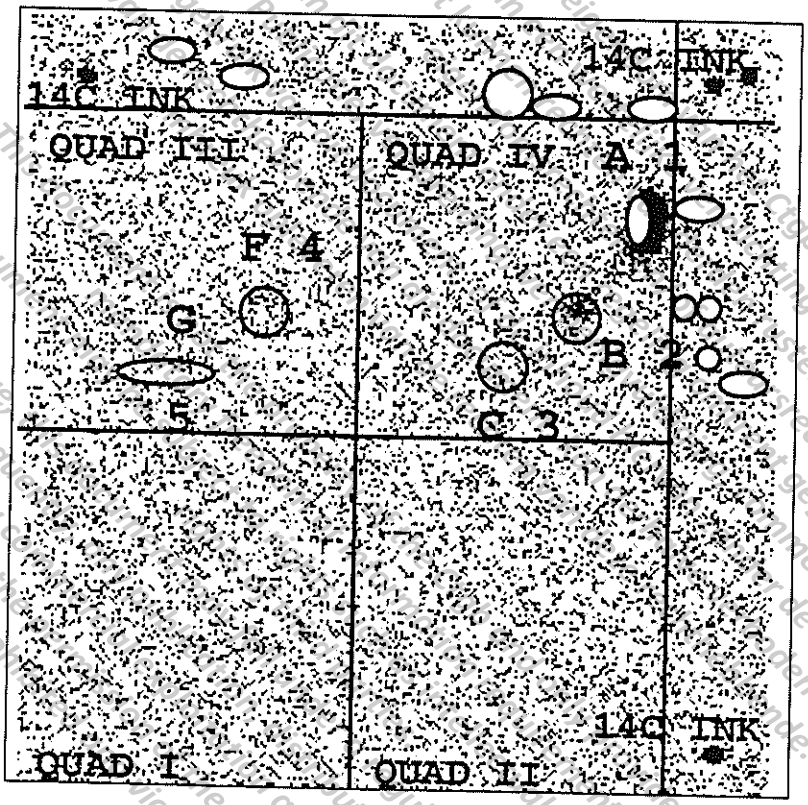


GC CHROMATOGRAM



MSD SCAN AT 15.581 MINUTES

FIGURE 77: GC/MSD OF CGA-329351, EXPERIMENT 1, ISOLATED COMPONENT A

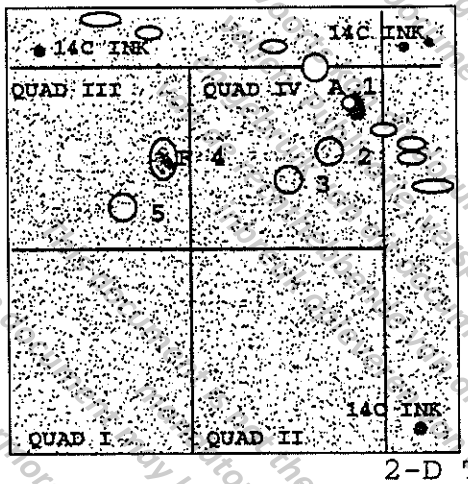


CGA-329351
DAY 21 R2, IRRADIATED, EXTRACTION 1

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

FIGURE 78: TWO DIMENSIONAL TLC OF COMPONENT B COCHROMATOGRAPHED WITH REFERENCE STANDARD CGA-42447

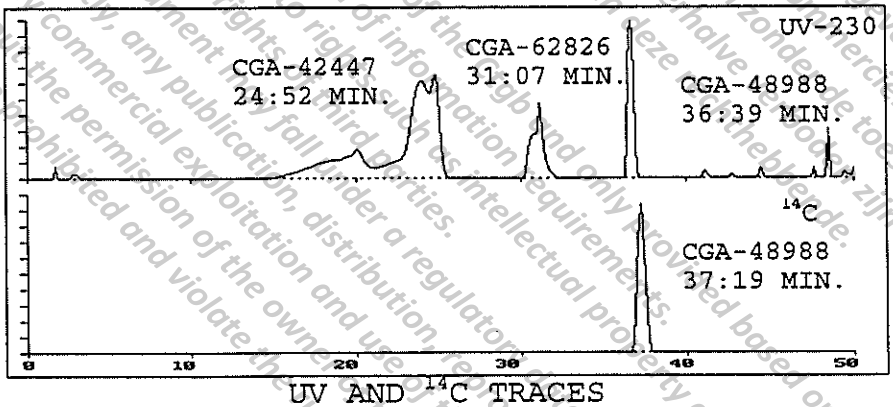
CGA-329351
DAY 30 R2, NON-IRRADIATED, EXTRACTION 2



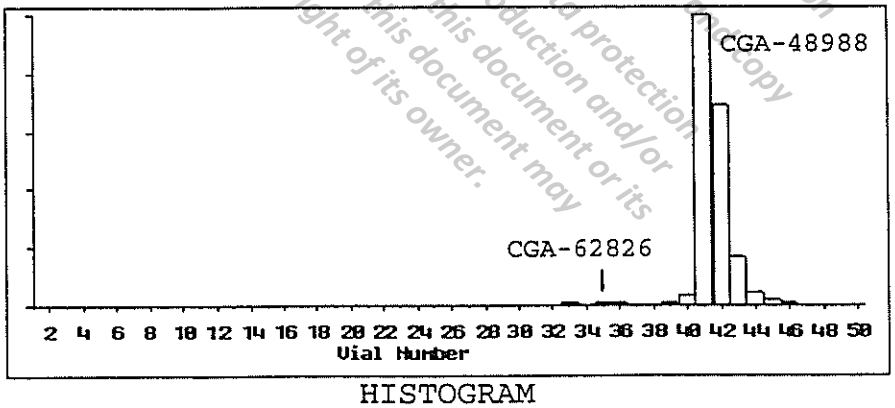
STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

2-D TLC

CGA-48988
DAY 30 R1, NON-IRRADIATED, EXTRACTION 1

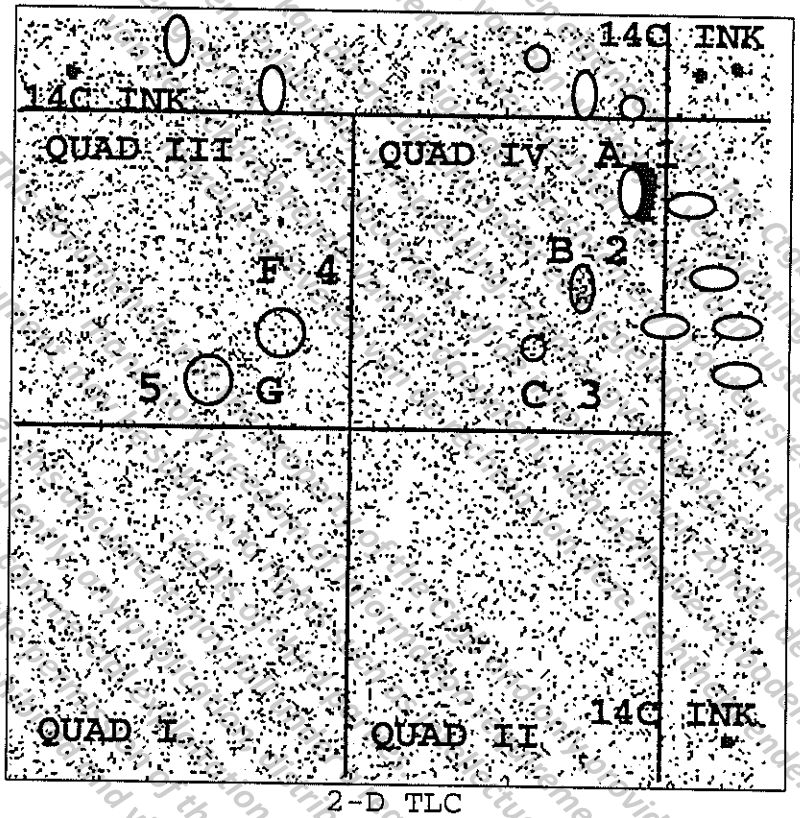


UV AND ^{14}C TRACES



HISTOGRAM

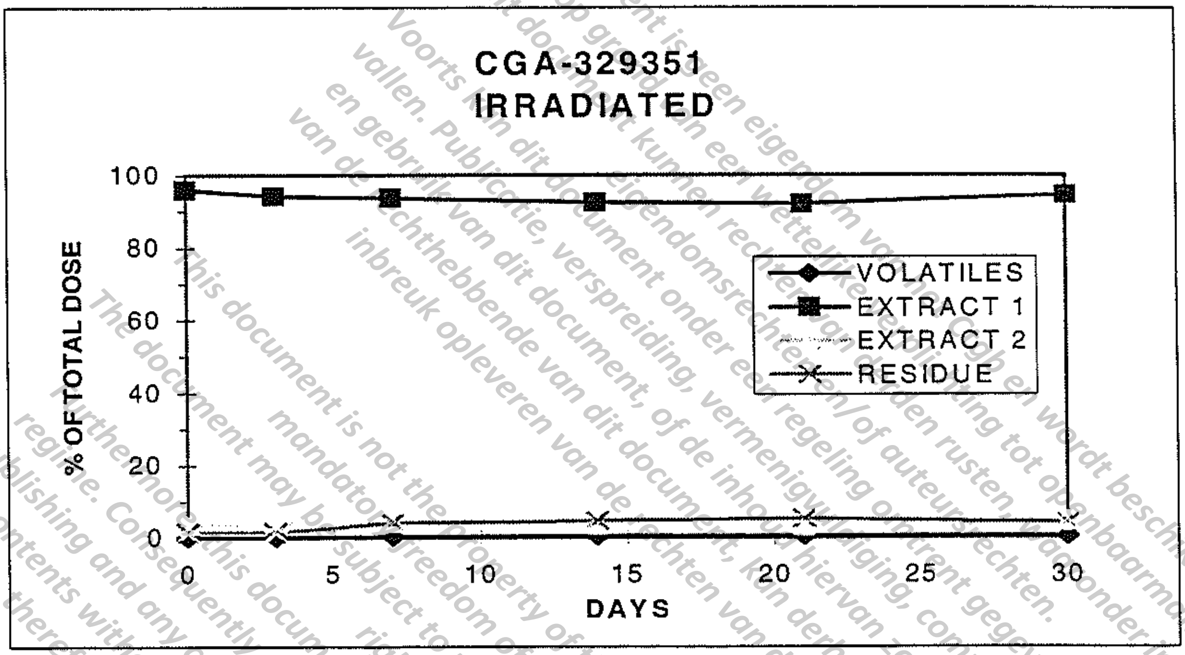
FIGURE 79: TLC AND HPLC COCHROMATOGRAPHY OF COMPONENT F WITH CGA-62826 REFERENCE STANDARD



2-D TLC
CGA-48988
DAY 14 R2, IRRADIATED, EXTRACTION 1

STANDARDS	
1	CGA-48988
2	CGA-42447
3	CGA-37734
4	CGA-62826
5	CGA-119857

FIGURE 80: TWO DIMENSIONAL TLC OF COMPONENT C CHROMATOGRAPHED WITH REFERENCE STANDARD CGA-37734



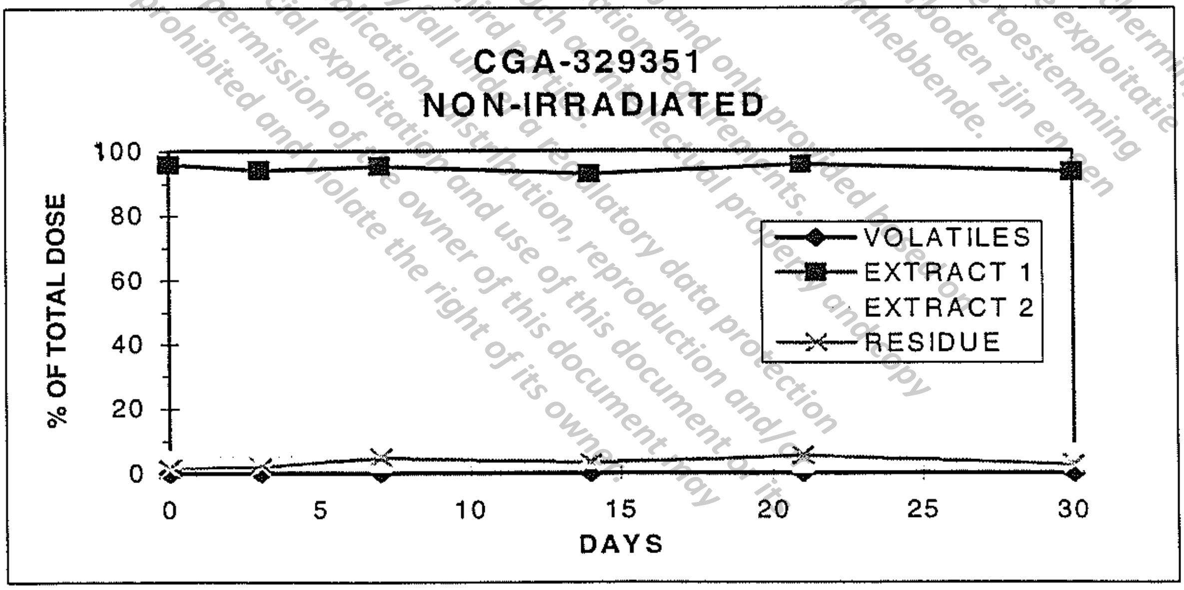
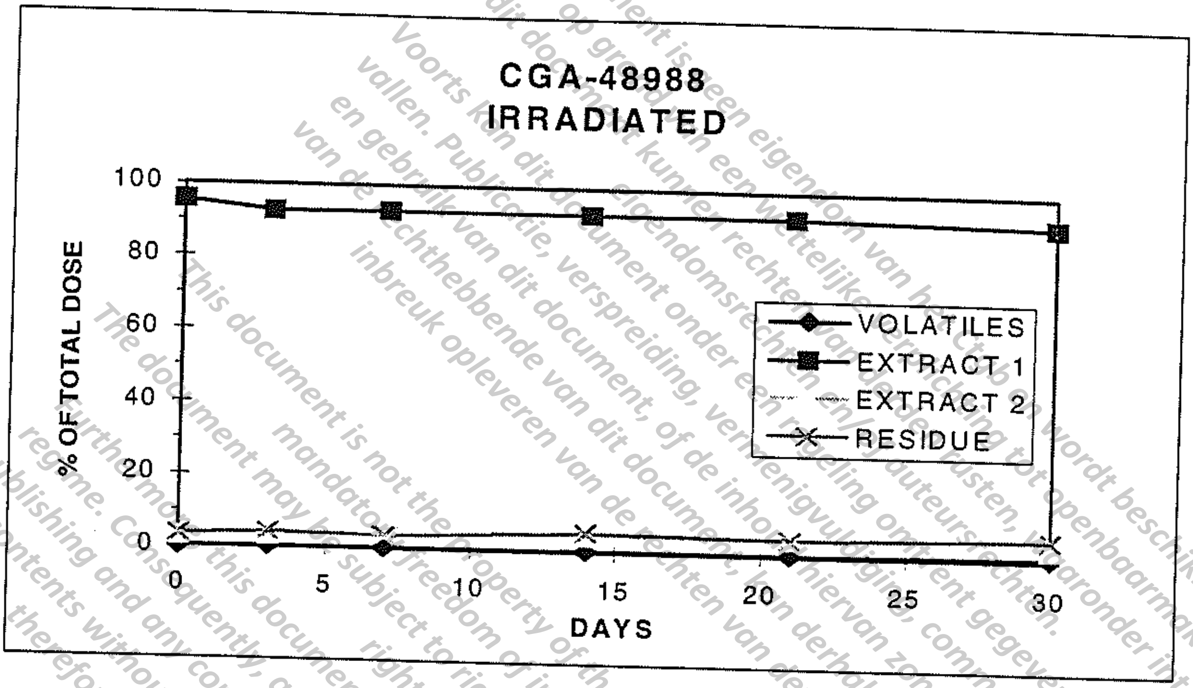


FIGURE 81: PLOT OF THE AVERAGE PERCENT OF TOTAL DOSE FOR EACH FRACTION FROM CGA-329351, EXPERIMENT 1



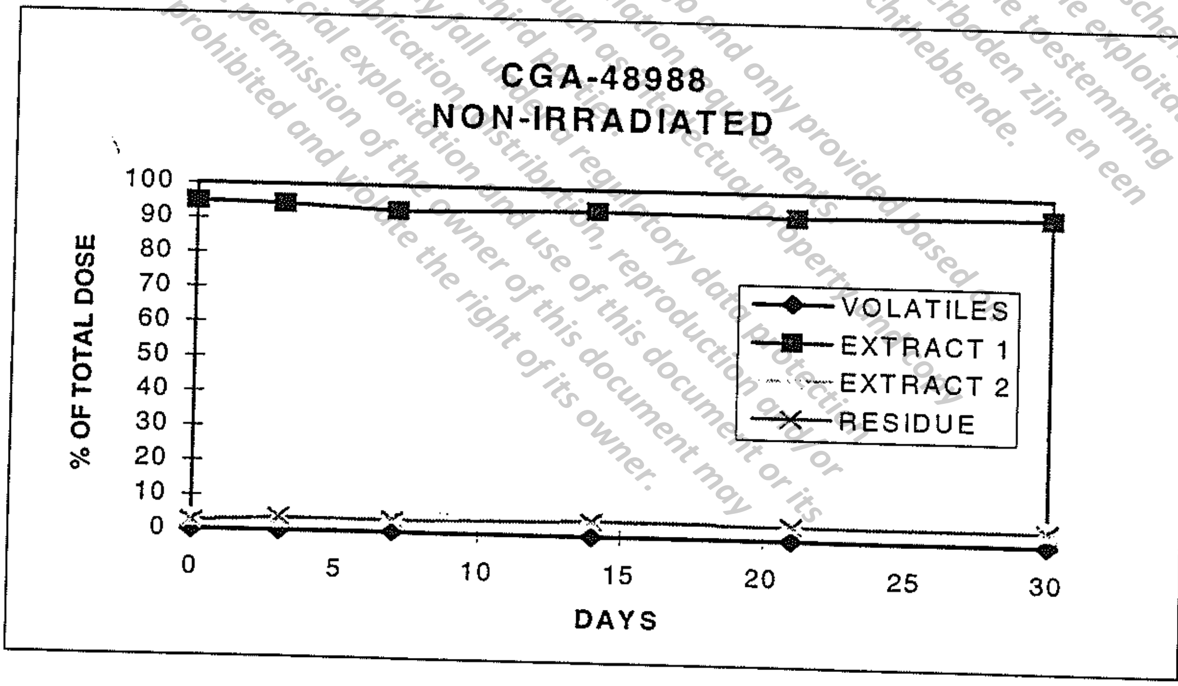
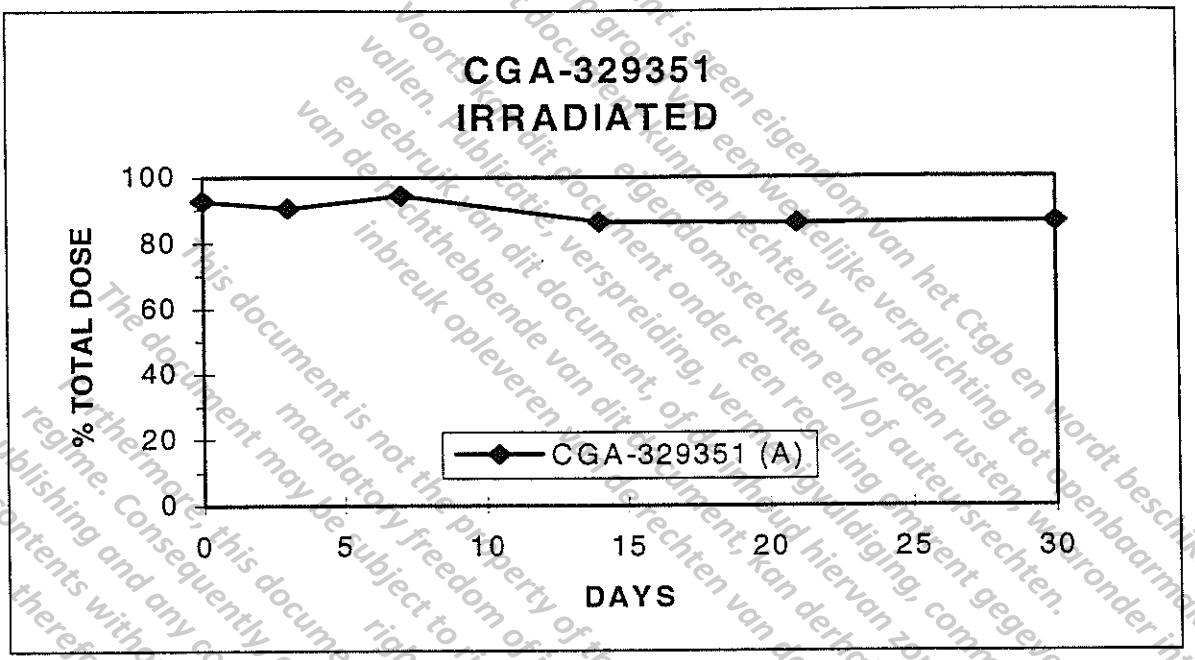


FIGURE 82: PLOT OF THE AVERAGE PERCENT OF TOTAL DOSE FOR EACH FRACTION FROM CGA-48988, EXPERIMENT 2



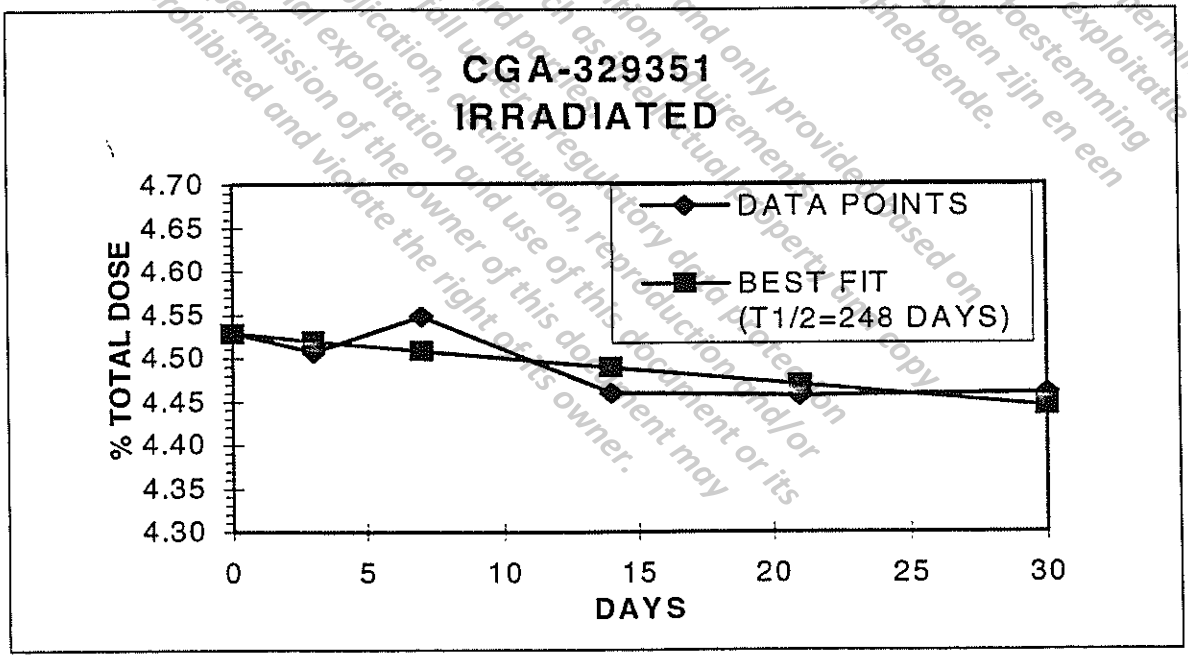
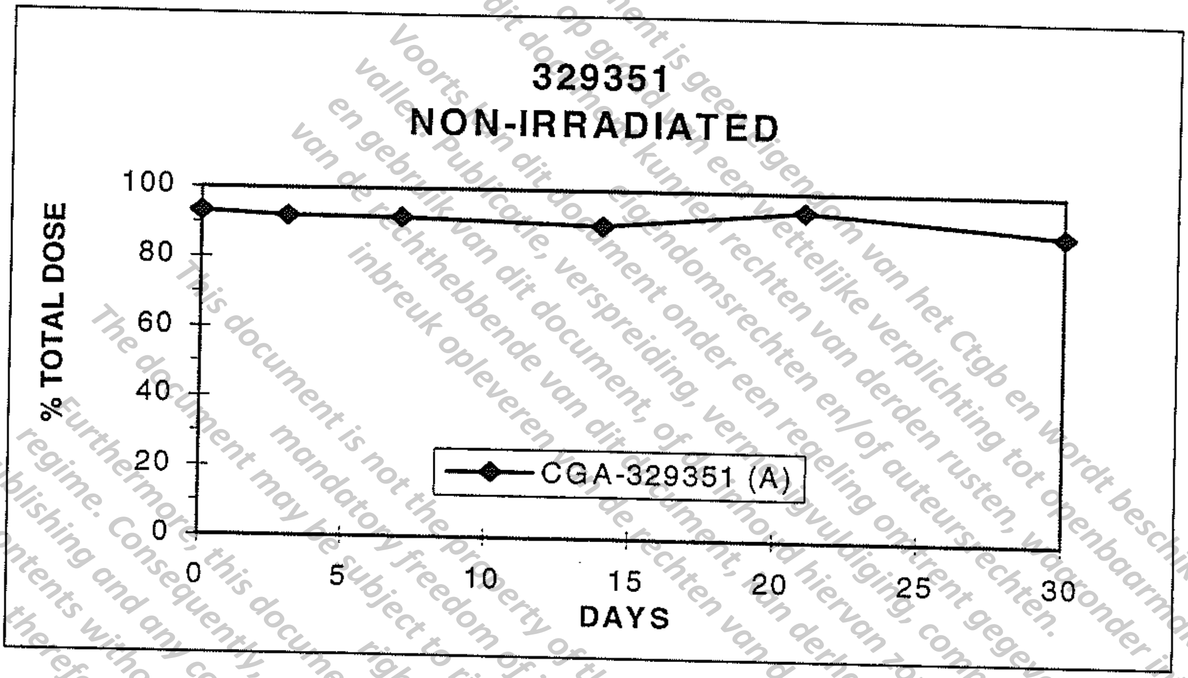


FIGURE 83: THE AVERAGE PERCENT OF TOTAL DOSE AND HALF LIFE PLOTS FOR CGA-329351, EXPERIMENT 1, IRRADIATED INCUBATION



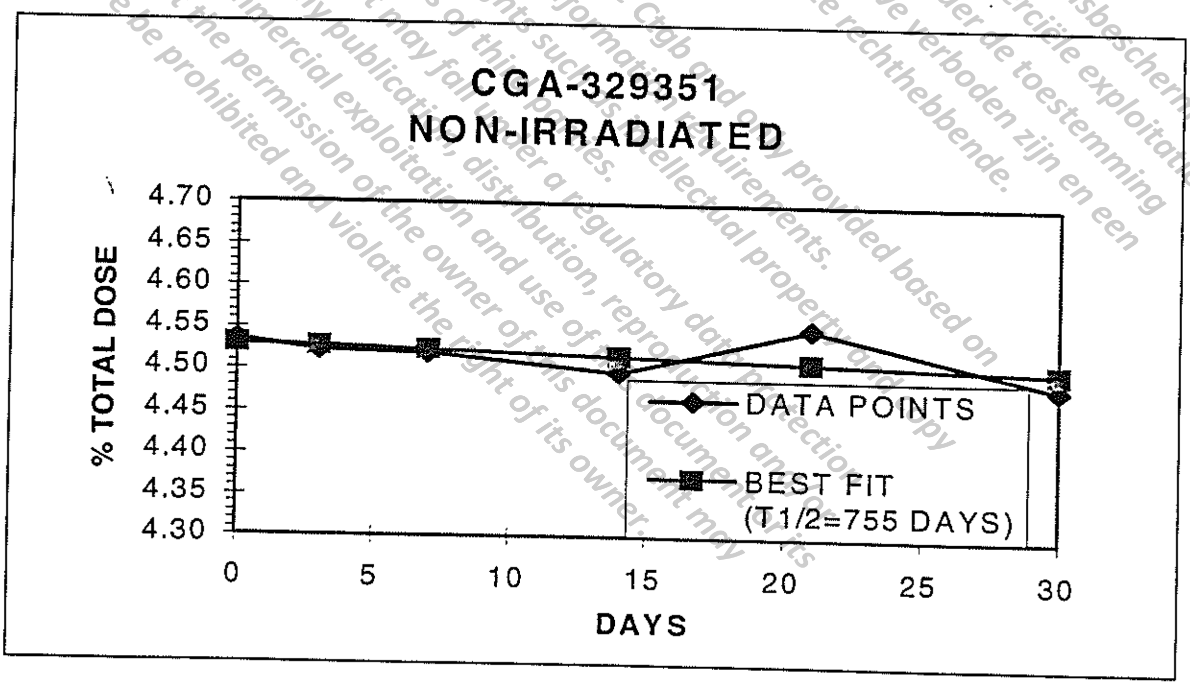
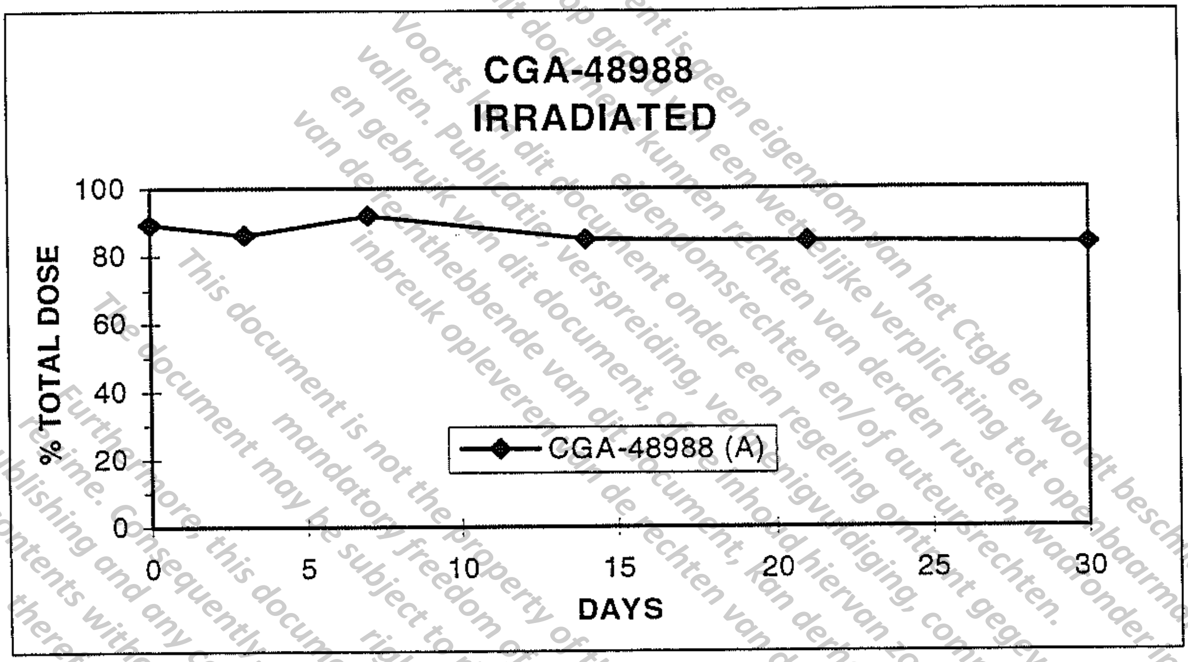


FIGURE 84: THE AVERAGE PERCENT OF TOTAL DOSE AND HALF LIFE PLOTS FOR CGA-329351, EXPERIMENT 1, NON-IRRADIATED INCUBATION



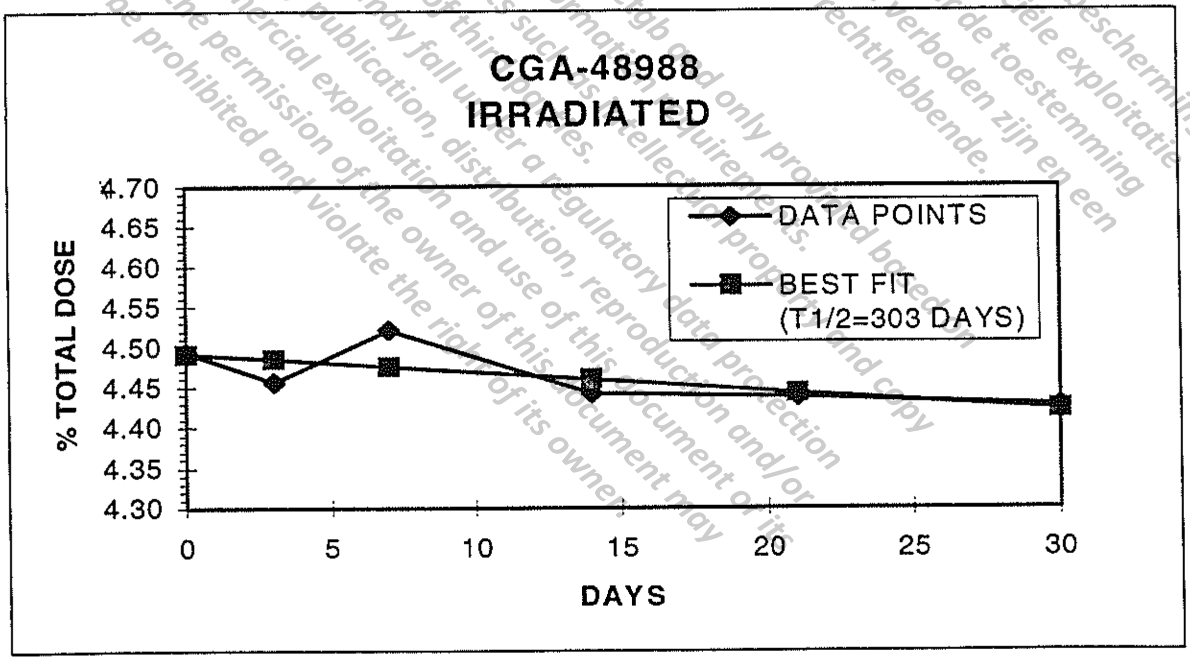
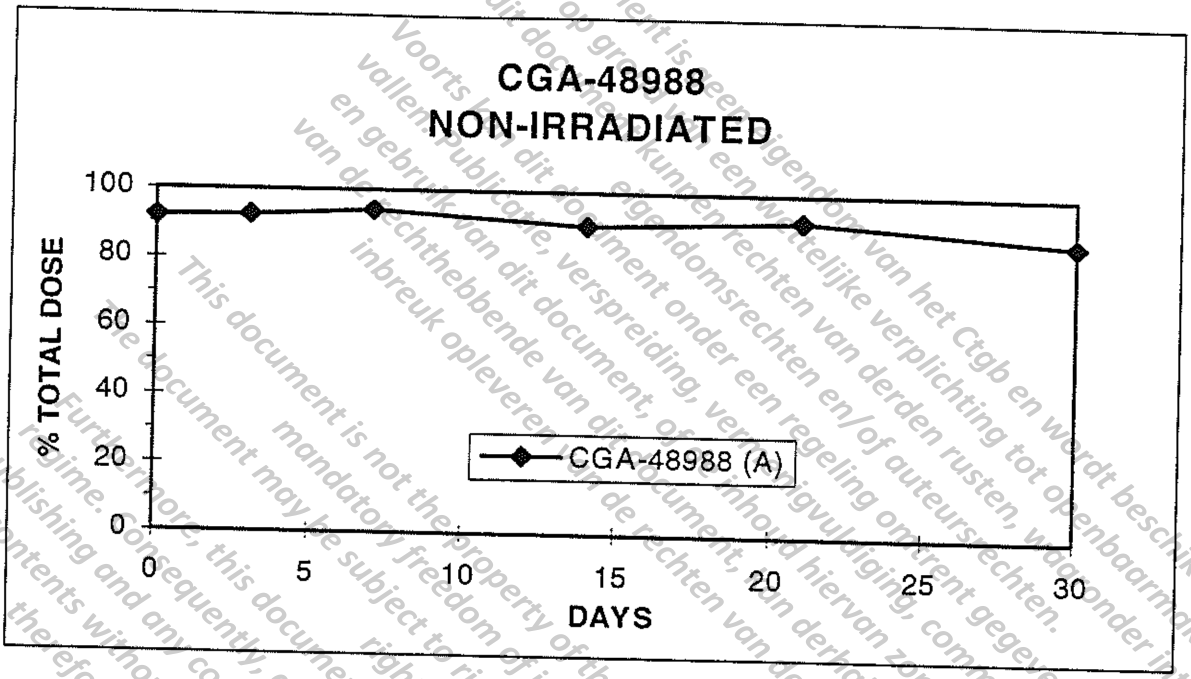


FIGURE 85: THE AVERAGE PERCENT OF TOTAL DOSE AND HALF LIFE PLOTS FOR THE CGA-48988, EXPERIMENT 2, IRRADIATED INCUBATION



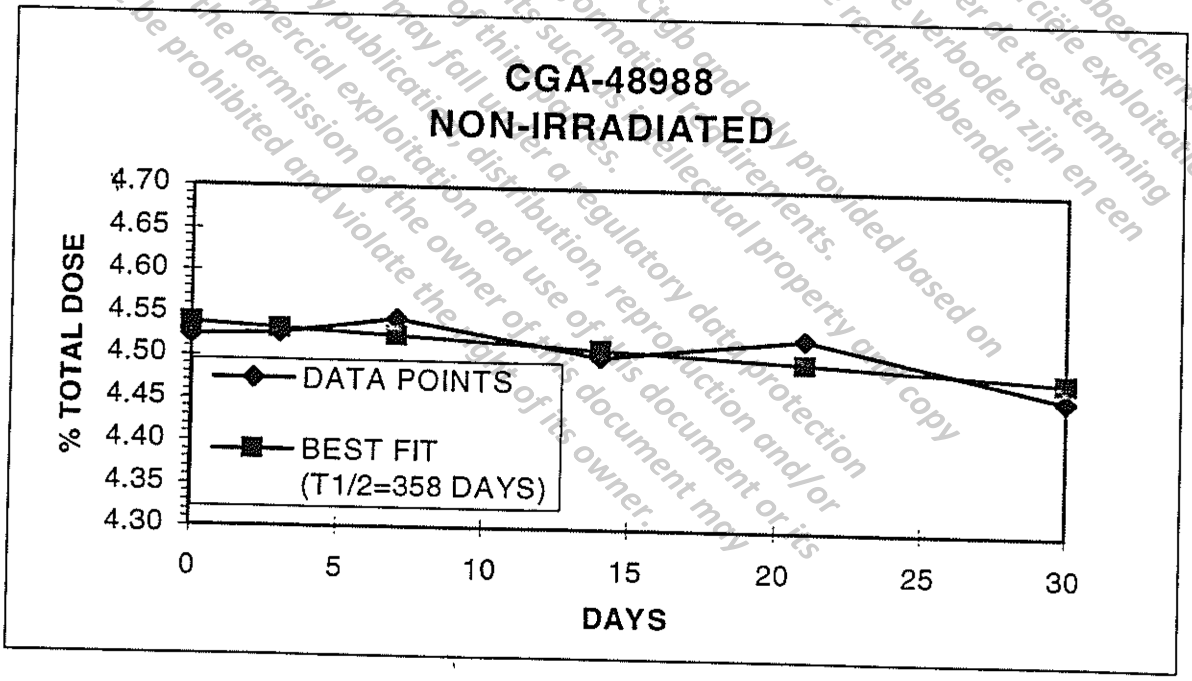


FIGURE 86: THE AVERAGE PERCENT OF TOTAL DOSE AND HALF LIFE PLOTS FOR CGA-48988, EXPERIMENT 2, NON-IRRADIATED INCUBATION

VIII. REFERENCES

1. Agrisearch Map of Soil Collection

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3. Anderson, J.P.E. and Domsch, K.H., Soil Biol. Biochem. Vol. 10., 215-221, 1978.