PMSD/ISB 1587

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

JUN - 2 1987

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Alachlor Response to Registration Standard
Method Trial Request for 2 Alachlor metabolites on peanuts and peanut hay

FROM: Susan V. Hummel, Chemist
Special Review Section II
Residue Chemistry Branch
Hazard Evaluation Division (TS-769)

Susan V. Hummel

THRU: Charles L. Trichilo, Chief
Residue Chemistry Branch
Hazard Evaluation Division (TS-769)

TO: Donald A. Marlow, Chief
Chemical Operations Branch
Benefits and Use Division (TS-768)

Tolerances have been established for alachlor and its metabolites in or on peanuts and other commodities. Alachlor [2-chloro-2',6'-diethyl-N-(methoxymethyl) acetanilide] is the active ingredient in LASSO Herbicide. Alachlor metabolites include those containing the diethylaniline (DEA) moiety and those containing the hydroxyethylethylaniline (HEEA) moiety.

Monsanto Company has submitted analytical methodology for alachlor and its DEA and HEEA metabolites on Peanut hay, vines, hulls and nutmeats in response to the Alachlor Registration Standard.

A method trial is requested for 2 chemicals on 2 commodities. These two chemicals are representative metabolites of alachlor.

DEA metabolite: sodium salt of 2-[(2,6-diethylphenyl) (methoxy-methyl)amino]-2-oxo-ethane sulfonic acid (tertiary amide sulfonic acid metabolite, containing 2,6-DEA moiety)
Monsanto Code # CP 108065
EPA Code No. E 0033

HEEA metabolite: N-[2(1-hydroxyethyl)-6-ethylphenyl]-N-(methoxymethyl)-2-(methylsulfonyl) acetamide (hydroxyethyl tertiary amide sulfone metabolite containing 2,6-HEEA moiety)
Monsanto Code No. CP 101394
EPA Code No. E 0032

Standards of diethyl aniline (DEA, EPA Code No. E0346) and hydroxyethylethylaniline (HEEA, EPA Code NO. E0705) are needed as well

Samples should be run in duplicate at the requested fortification levels (See attached table). Two copies of the appropriate method(s) along with recoveries and sample chromatograms are attached.

Please return the requested information on the attached forms and any other information concerning the method trial that we should be aware of, including copies of chromatograms for representative controls and fortified samples, standard curves, and sample calculations.

The EPA Repository was contacted on 5/27/87 (FTS 629-3951). The standards for the tertiary amide sulfonic acid metabolite and the hydroxyethyl tertiary amide sulfone metabolite are available. The Repository code numbers are given above.

A short turnaround time is requested for this MTO. MTO results are needed to support the Alachlor Special Review.

Please forward results of this method trial directly to Edward Zager.

Attachment: Tables (2 pp): attached to all copies

Attachment: Analytical Method: attached to copies to addressee (2 copies), K. Kissler, W. Bontoyan, PMDS/ISB

cc: RF, circu, S. Hummel, M. Bradley, R. Thompson, FDA, Alachlor Reg. Std File (Boodee), PM#25, K. Kissler, W. Bontoyan, MTO F., PMSD/ISB
RDI:EZ:05/29/87:RDS:06/01/87
TS-769:RCB:SVH:svh:RM810:CM#2:x77324:06/01/87



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USE SEPARATE FORM FOR EACH METHOD

METHOD: (Report No. and/or/Title, date)

"Analytical Method for the Determination of 2,6-Diethylaniline (DEA) and 2-(1-Methoxyethyl)-6-Ethylaniline (MEEA)-Yielding Alachlor Metabolites in Peanut Hay, Vines, Hulls, and Nutmeats," Appendix D of MSL-5718 and MSL-4636 (Accession No. 263022). Author and date not given. Submission dated May, 1986.

This analytical method is different from previously submitted methods. It does not require the use of the custom made glassware used in the other methods. Methoxyethylethylaniline (MEEA) is produced from HEEA. The DEA and MEEA are determined by HPLC with oxidative coulometric electrochemical detection (OCED). No derivatization is required.

Do not use control values for recovery corrections.

Do not report control values as 0; for less than limit of detection, report as such.

| Commodity | Chemical Added | PPM added | PPM found | %Recovery |
|---|---|---|---|---|
| Peanuts | DEA metabolite | 0.010 | | |
| | DEA metabolite | 0.10 | | |
| | DEA metabolite | 0.50 | | |
| Peanuts | HEEA metabolite | 0.010 | | |
| | HEEA metabolite | 0.10 | | |
| | HEEA metabolite | 0.50 | | |
| Peanut Hay | DEA metabolite | 0.010 | | |
| | DEA metabolite | 0.20 | | |
| | DEA metabolite | 2.0 | | |
| Peanut Hay | HEEA metabolite | 0.010 | | |
| | HEEA metabolite | 0.20 | | |
| | HEEA metabolite | 2.0 | | |

Note:
DEA metabolite=sodium salt of 2-[(2,6-diethylphenyl)(methoxymethyl)amino]-2-oxo-ethane sulfonic acid (tertiary amide sulfonic acid metabolite, containing 2,6-DEA moiety)
HEEA metabolite=N-[2(1-hydroxyethyl)-6-ethylphenyl]-N-(methoxymethyl)-2-(methylsulfonyl) acetamide (hydroxyethyl tertiary amide sulfone metabolite containing 2,6-HEEA moiety)

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USE SEPARATE FORM FOR EACH METHOD

Modifications to method (major or minor)

Special Precautions to be taken:

Source of analytical reference:

If derivitized standard used, give source:

Instrumentation for quantitation:

Instrument for confirmation:

If instrument parameters differ from method given, list parameters used:

Commercial Source for anly special chemicals or apparatus:

Comments:

Chromatograms:

APPENDIX C: Analytical Method for the Determination of 2,6-Diethylaniline (DEA) and 2-(1-methoxyethyl)-6-ethylaniline (MEEA) Yielding Alachlor Metabolites in Peanut Hay, Vines, Hulls and Nutmeats

# Analytical Method for the Determination of 2,6-Diethylaniline (DEA) and 2-(1-methoxyethyl)-6-ethylaniline (MEEA) Yielding Alachlor Metabolites in Peanut Hay, Vines, Hulls and Nutmeats

## SCOPE

This method determines residues of alachlor [2-chloro-N-(2,6-diethylaniline)-N-(methoxymethyl)acetamide] and 2,6-diethylaniline (DEA) and 2-(1-methoxymethyl)-6-ethylaniline (MEEA) yielding alachlor metabolites in peanut hay, vines, hulls, and nutmeat.

## SUMMARY

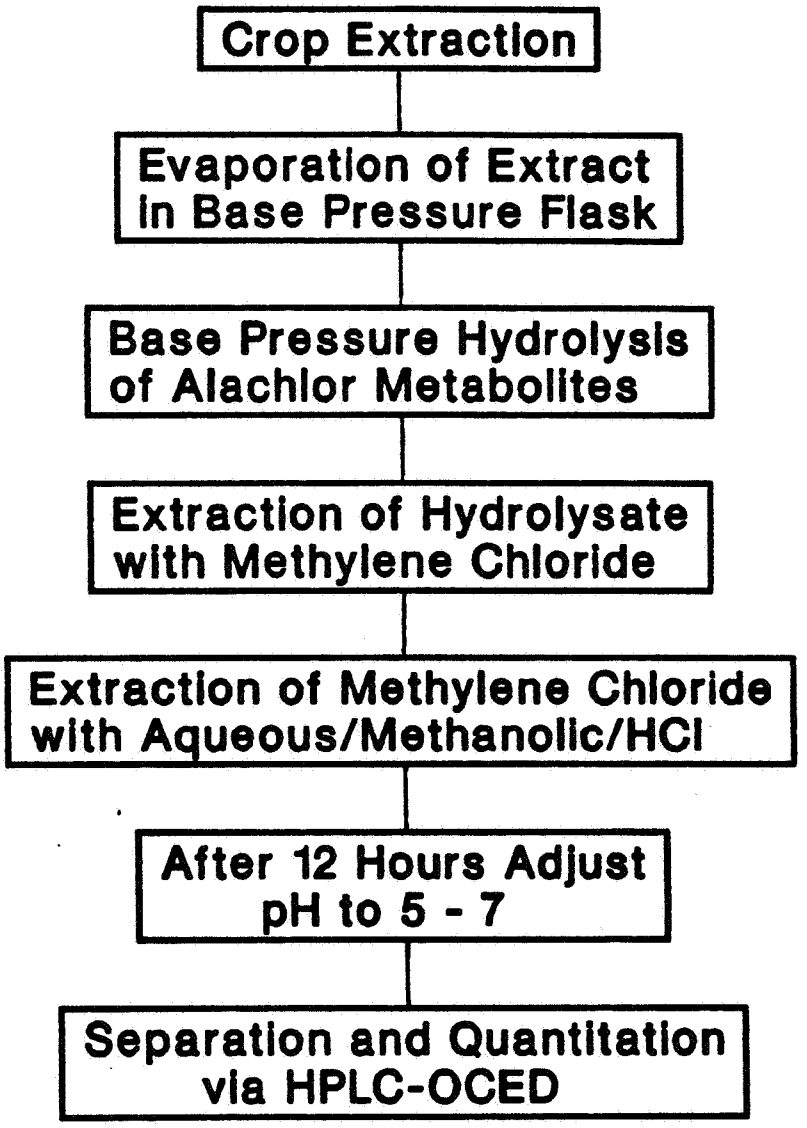
The methodology consists of extracting the sample with solvent in a blender, followed by filtration and evaporation. The extracted residue is hydrolyzed with base under pressure. Following hydrolysis, the hydrolysate is extracted with methylene chloride. The methylene chloride extract is extracted with an aqueous/methanolic/HCl solution. Following separation from the methylene chloride layer, additional methanol is added to the aqueous layer, and the solution is allowed to sit for approximately 12 hours, during which all HEEA is converted to MEEA. Prior to separation the pH of the aqueous/methanolic solution is adjusted to 5-7. Analytes are separated and quantitated by reverse phase High Performance Liquid Chromatography with Oxidative Coulometric Electrochemical Detection HPLC-OCED. Figure 1 summarizes the important steps in the methodology. Alachlor residue levels for each metabolite class are expressed as an equivalent amount of alachlor and corrected for the analytical recovery of each class. In order to estimate the analytical recovery of each alachlor metabolite class across the validation range, untreated samples of each peanut matrix are fortified with 2-[(2,6-diethylphenyl)(methoxymethyl)amino]-2-oxoethanesulfonic acid, sodium salt, and N-(methoxymethyl)-N-[2-(1-hydroxyethyl)-6-ethylphenyl]-2-(methylsulfonyl)acetamide. These two metabolites are representative of the two metabolite classes which produce DEA and MEEA, respectively.

## METHOD PERFORMANCE

The method validation limit of this procedure is 0.010 ppm for each metabolite class in peanut hay, vines, hulls and nutmeat.

FIGURE 1

# FLOW CHART FOR BASE PRESSURE/HPLC-OCED ALACHLOR RESIDUE METHOD



APPARATUS AND EQUIPMENT

Explosion proof blender: Fisher No. 14-509-33

Blender jars: Fisher No. 14-509-11A

Mettler balance, Model PC-440 or equivalent

85 mm Buchner funnel: Fisher No. 10-356C

7 cm Filter paper: Fisher No. 9-805C

Caleb rotary evaporator

Wheaton pressure bottle: Wheaton No. 223077

500 mL Round bottom flask: Fisher No. 10-067G

Rotary evaporator adapter for 24/40 joint and to fit a Wheaton pressure bottle (made in Monsanto Glass Shop)

125 mL separatory funnel: Fisher No. 10-437-10B

25 mL graduated cylinder: Fisher No. 8-565B

10 mL graduated centrifuge tubes: Fisher No. K45201-10

Pasteur pipette 9" length: Fisher No. 13-678-6B

Serological pipettes from 0.1 through 5 mL: Fisher No. 13-675-28A,B,C,D,E,F

Perkin-Elmer ISS-100 automatic sampler equipped with a 50 µL injection loop

Zorbax® C-8 analytical column (4.6 mm x 15 cm): Fisher No. 6-642-2G

2 In-line high pressure prefilter assemblies with ESA carbon filter element: SSI No. 05-0149

Model 5100A Coulochem Detector with the Model 5010 analytical cell and the Model 5020 guard cell: ESA, Inc.

Pulse dampener: Scientific Systems, Inc. Model No. 20-0218

Varian 2010 HPLC Pump

Fisher Recordall Series 5000 strip chart recorder

Vials for HPLC automatic sampler: Varian No. 66-000104-00

Teflon septa for HPLC automatic sampler vials: IBM No. 8635463

Oil Baths: Fisher No. 11-481

Automatic Oil Bath Lift: Built in the Monsanto Shop

SOLVENTS, REAGENTS, AND SOLUTIONS

Acetonitrile: Fisher No. A-998

Methylene Chloride: Fisher No. D-143

Methanol: Fisher No. A-452

50% Sodium Hydroxide Solution (w/w): Fisher No. SO-S-254

Hydrochloric Acid: Fisher No. A144-500

Acetic Acid: Fisher No. A38-500

Sodium Acetate: Fisher No. S209-500

pH Paper: American Scientific Products P1119-1

Deionized water from a Milli-Q water purification system (Millipore Co.)

Dow Corning antifoam B emulsion: Fisher No. CS-283-4

Mineral Oil

25% Sodium Hydroxide in deionized water

4N Hydrochloric Acid

20% Water in Acetonitrile

2,6-diethylaniline (99.5%): Aldrich No. 23,759-0

2-[2,6-diethylphenyl)(methoxymethyl)amino]-2-oxoethane sulfonic acid, sodium salt: synthesized in house, referred to as the tertiary amide sulfonic acid metabolite

2-(1-hydroxyethyl)-6-ethylaniline: synthesized by Stark Laboratories

2-(1-methoxyethyl)-6-ethylaniline: synthesized in-house

N-[2-(1-hydroxyethyl)-6-ethylphenyl]-N-(methoxymethyl)-2-(methylsulfonyl)acetamide: synthesized in-house, referred to as the hyroxyethyltertiary amide sulfone metabolite

STANDARD SOLUTIONS

FORTIFICATION STANDARDS

Weigh 0.165 grams of the tertiary amide sulfonic acid metabolite of alachlor and 0.153 grams of the hydroxyethyltertiary amide sulfone metabolite of alachlor into a 100 mL volumetric flask and dilute to volume with methanol, mix well to insure complete dissolution. This solution contains 1250 µg/mL alachlor equivalent of each representative metabolite.

Pipette 10 mL of the 1250 µg/mL solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard contains 125 µg/mL equivalent of each metabolite.

Pipette 10 mL of the 125 µg/mL solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard contains 12.5 µg/mL alachlor equivalent of each metabolite.

Pipette 10 mL of the 12.5 µg/mL solution into a 100 mL volumetric, dilute with methanol and mix well. This standard contains 1.25 µg/mL alachlor equivalent of each metabolite.

HPLC-OCED STANDARDS

At the initiation of the study detector calibration standards were made up from solutions of DEA and MEEA for each set as described below.

Weigh 0.010 gram of 2,6-diethylaniline (DEA) into a 100 mL volumetric flask and dilute to volume with methanol and mix well. This solution contains 100 µg/mL of DEA.

Weigh 0.0125 gram of 2-(1-methoxyethyl)-6-ethylaniline (MEEA) into a 100 mL volumetric flask and dilute to volume with methanol and mix well. This solution contains 125 µg/mL of MEEA.

Pipette 10 mL of each of the above solutions into a 100 mL volumetric, dilute to volume and mix well. These two solutions contain 10 µg/mL of DEA and 12.5 µg/mL of MEEA.

Pipette 1.0 mL of the 10 µg/mL DEA solution and 1.0 mL of the 12.5 µg/mL MEEA solution into a 100 mL volumetric, dilute to volume with deionized water and mix well. This standard solution is standard 5 and contains 0.100 µg/mL DEA and 0.125 µg/mL MEEA.

Pipette 0.50 mL of the 10 µg/mL DEA solution and 0.50 mL of the 12.5 µg/mL MEEA solution into a 100 mL volumetric, dilute to volume with deionized water and mix well. This standard solution is standard 4 and contains 0.050 µg/mL DEA and 0.0625 µg/mL MEEA.

Pipette 25 mL of standard 4 into a 50 mL volumetric, dilute to volume with deionized water and mix well. This standard solution is standard 3 and contains 0.0250 µg/mL DEA and 0.03125 µg/mL MEEA.

Pipette 5 mL of standard 4 into a 50 mL volumetric, dilute to volume with deionized water and mix well. This standard solution is standard 2 and contains 0.005 µg/mL DEA and 0.0065 µg/mL MEEA.

Pipette 25 mL of standard 2 into a 5 mL volumetric, dilute to volume with deionized water and mix well. This standard solution is standard 1 and contains 0.0025 µg/mL DEA and 0.003125 µg/mL MEEA.

Detector calibration standards prepared from solid MEEA were used for all the peanut hay jobs and for two of the peanut vine jobs. Following exhaustion of the supply of solid MEEA a new standard preparation procedure (described below) was implemented and used for the rest of the analyses.

Weigh 0.100 gram of 2,6-diethylaniline (DEA) into a 100 mL volumetric flask and dilute to volume with methanol and mix well. This solution contains 1000 µg/mL of DEA.

Weigh 0.100 gram of 2-(1-hydroxyethyl)-6-ethylaniline (HEEA) into a 100 mL volumetric flask and dilute to volume with methanol and mix well. This solution contains 1000 µg/mL of HEEA.

For each of the above solutions, pipette 10 mL of the 1000 µg/mL solution into a 100 mL volumetric, dilute to volume and mix well. These two solutions contain 100 µg/mL of DEA and 100 µg/mL of HEEA.

Pipette 10 mL each of the 100 µg/mL DEA solution and the 100 µg/mL DEA solution and the 100 µg/mL HEEA solution into a 100 mL volumetric, dilute to volume and mix well. These two solutions contain 10 µg/mL of DEA and 10 µg/mL of HEEA.

Pipette 1.25 mL of the 10 µg/mL DEA solution and 1.25 mL of the 10 µg/mL HEEA solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard solution contains 0.125 µg/mL of both DEA and HEEA.

Pipette 10 mL of the 10 µg/mL DEA solution and 10 mL of the 10 µg/mL HEEA solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard solution contains 1.00 µg/mL of both DEA and HEEA.

Pipette 2 mL of the 100 µg/mL DEA solution and 2 mL of the 100 µg/mL HEEA solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard solution contains 2.00 µg/mL of both DEA and HEEA.

Pipette 3 mL of the 100 µg/mL DEA solution and 4 mL of the 100 µg/mL HEEA solution into a 100 mL volumetric, dilute to volume with methanol and mix well. This standard solution contains 3 µg/mL of DEA and 4 µg/mL of HEEA.

All standards are stored in amber glass bottles at 4°C.

Five standards are prepared from the above standard solutions with every sample set. In this way the preparation of the detector calibration standards also accounts for the completness of the HEEA conversion to MEEA. The appropriate amounts of DEA and HEEA are pipetted into a 25 mL graduated cylinder to which is added 10 mL 4N HCl and 10 mL MeOH. The graduated cylinder is capped, mixed, and the contents are allowed to sit overnight together with the rest of the samples in the set. The procedure for the standards from this point on is the same as that for the samples (see procedure following this section). Ultimately, the standards are diluted to 25 mL. For the resulting DEA and MEEA concentrations, see the table below.

| Std. No. | mL of Standard Solution | Final DEA Conc. µg/mL | Final MEEA Conc. µg/mL |
|---|---|---|---|
| 1 | 1 mL of the 0.125 µg/mL | 0.005 | 0.00542 |
| 2 | 2 mL of the 0.125 µg/mL | 0.010 | 0.01084 |
| 3 | 1 mL of the 1.00 µg/mL | 0.040 | 0.04336 |
| 4 | 1 mL of the 2.00 µg/mL | 0.080 | 0.08672 |
| 5 | 1 mL of the 3 µg/mL DEA and 4 µg/mL HEEA | 0.120 | 0.17344 |

PROCEDURE

A. SAMPLE PREPARATION

Peanut Hay and Vines

Chop the frozen peanut commodities in a Hobart chopper with dry ice added. Place the samples in a cold room to allow evaporation of the dry ice. Sub-sample the prepared samples for analysis.

Peanut Hulls and Nutmeat

Shell the peanuts with a peanut sheller. Mix the nuts for 10 minutes in a mixer, sub-sample, and grind in a Waring blender with dry ice. Store the nutmeat samples in a cold room. Mix the shells, sub-sample, and grind in a Waring blender with dry ice. Store the hulls in a cold room.

B. CROP EXTRACTION

Weigh 25.0 grams of the previously prepared crop matrix into a blender jar. (Make fortifications at this stage.) Add 150 mL of 20% water/acetonitrile to the jar and blend at medium speed for 4 minutes. Vacuum filter the extract through a Buchner funnel into a 500 mL round bottom flask. Rinse the blender jar and filter cake twice with 25-50 mL portions of 20% water/acetonitrile and collect each rinse in the 500 mL round bottom flask. Add about 5 drops of Dow Corning DB 31 Antifoam to the extract and evaporate down to about 75 mL using a rotary evaporator equipped with a dry ice condensor. During evaporation the flasks are immersed in a 40-50°C water bath. Transfer the solution from the 500 mL round bottom flask to a 200 mL Wheaton pressure bottle. Wash the round bottom flask with an additional 10 mL of 20% water/acetonitrile and transfer this to the Wheaton pressure flask. Attach the Wheaton flasks to the rotary evaporators by means of a modified adaptor fitted with a rubber cork. Concentrate the remainder of the solution to near dryness by rotary evaporation in a 50°C oil bath.

C. HYDROLYSIS

Add 45 mL 25% sodium hydroxide in water and 5 mL methanol to each Wheaton pressure flask. Stir gently. Replace the black septum on the top of the pressure flask with a high temperature blue septum (Alltech Cat. No. 7832). Line the septum with two pieces of teflon tape and seal the flask. Place the flasks on an oil bath rack or lift and lower them into the 155°C oil baths. The temperature of the baths will drop slightly when the flasks are in the oil. Do not let the temperature fall below 150°C. After 45 minutes, raise the flasks out of the oil and allow them to cool to room temperature within the hood over one hour. (They can be left cooling for several hours, if desired.)

D. CLEANUP

Transfer the hydrolysis solution to a 125 mL separatory funnel. Rinse the hydrolysis flask out with 20 mL water and 20 mL of methylene chloride and transfer both of these rinses to the separatory funnel. Extract the hydrolysis solution with this portion of methylene chloride and then extract two more times with 20 mL portions of methylene chloride. Shake the funnels gently (for one minute) to avoid emulsions. After each extraction, allow at least 15 minutes for the layers to separate. Collect the three portions of methylene chloride directly into another 125 mL separatory funnel. Extract the combined methylene chloride portions with a mixture of 10 mL 4N HCl and 3 mL methanol. Allow a few minutes for phase separation. Collect the acidic aqueous/methanolic layer in a 25 mL graduated cylinder and add to this an additional 6-7 mL of MeOH. (This additional MeOH can also be used to rinse out the separatory funnel, before it is added to the graduated cylinder.) Cap the graduated cylinder, shake gently, and allow it to stand overnight at room temperature. The next morning, add 25% sodium hydroxide in water (~3.5-4 mL) dropwise, while cooling the graduated cylinder in ice. Then adjust the pH to 5-7 with dilute acid and base. Dilute to an appropriate volume with the mobile phase or a solution of 50% methanol/water. The sample is now ready for separation and quantitation.

E. SEPARATION AND QUANTITATION

The analytes, DEA and MEEA, are separated and quantitated by reverse phase high performance liquid chromatography with oxidative coulometric electrochemical detection, HPLC-OCED. A block diagram of the system is shown in Figure 2. Details of the operating conditions are as follows:

HPLC-OCED OPERATING CONDITIONS

| | |
|---|---|
| Flow Rate: | 1.0 mL/min |
| Mobile Phase: | 45% pH 4.8 acetate buffer, (dissolve 6.8 gram sodium acetate and 3 mL glacial acetic acid in 1000 mL deionized water) 55% methanol (v/v) |
| Column: | DuPont Zorbax® C-8 (150 mm x 4.6 mm i.d.) |
| Temperature: | Ambient |
| Detection: | Operate in the screen mode, guard cell potential =+0.80V, detector 1=+0.35V, detector 2=+0.70V |
| Gain: | 200 |
| Response Time: | 0.4 sec |
| Sample Size: | 50 µL |

The amounts of DEA and MEEA are determined based upon external standard calibration. A non-weighed linear least squares estimate of the calibration curve is used to calculate the amount of DEA and MEEA in the unknowns.

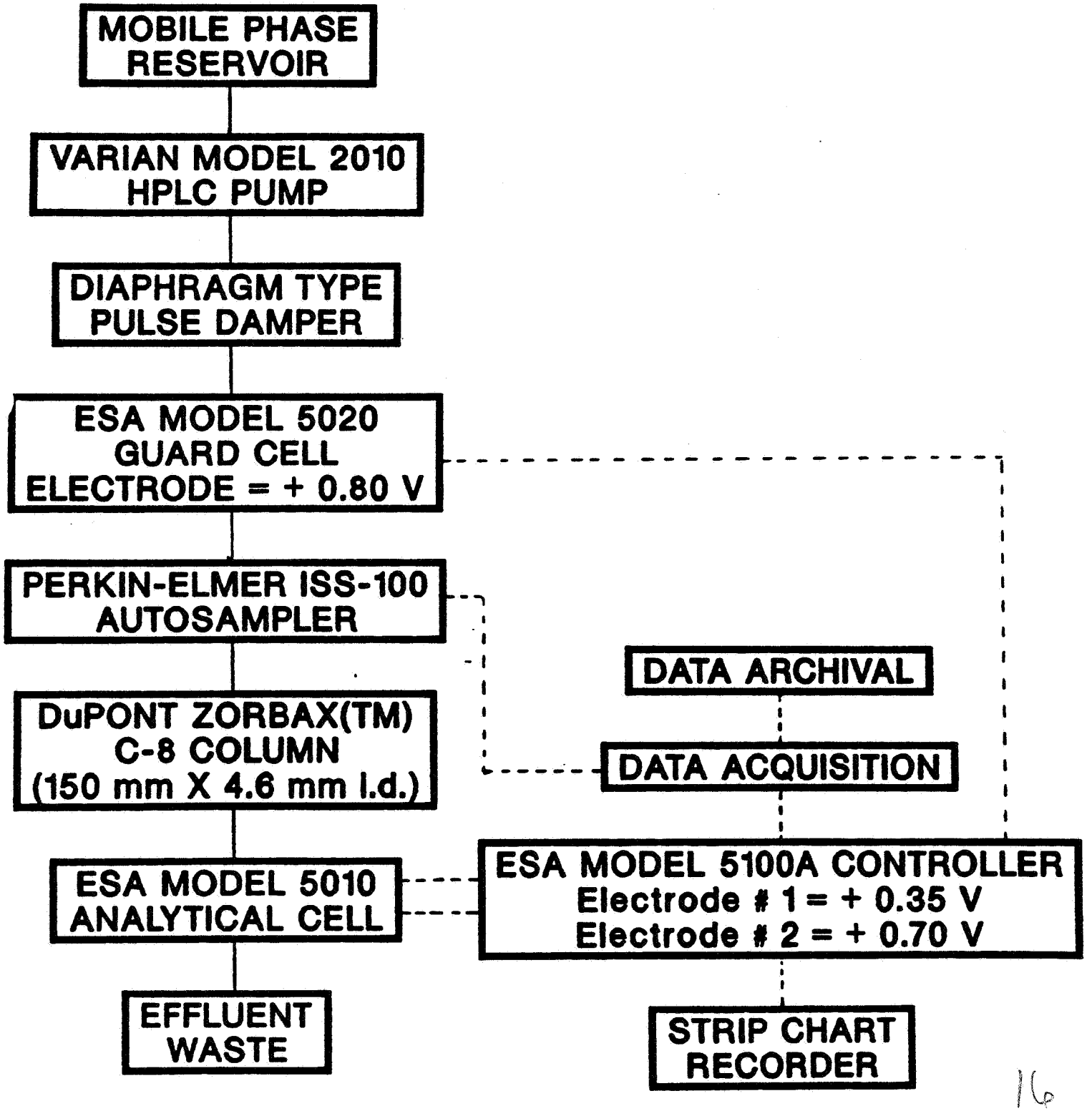
F. SAMPLE CHROMATOGRAMS

Included in this section are sample chromatograms which are considered typical for this procedure.

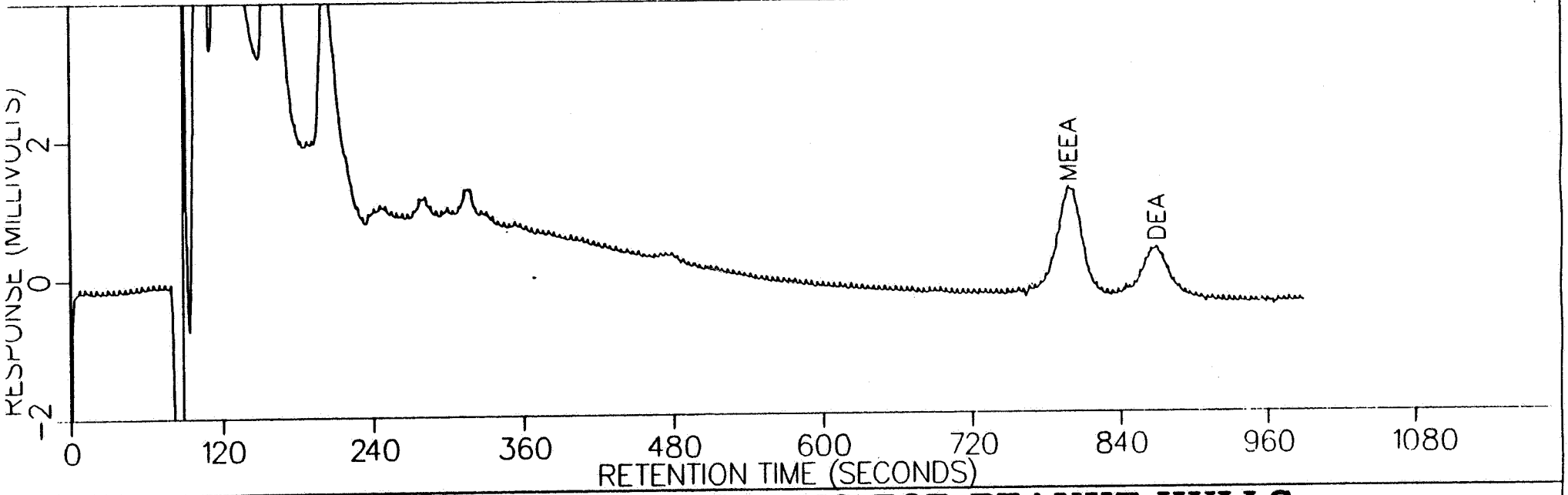
FIGURE 2

# BLOCK DIAGRAM OF HPLC-OCED SYSTEM

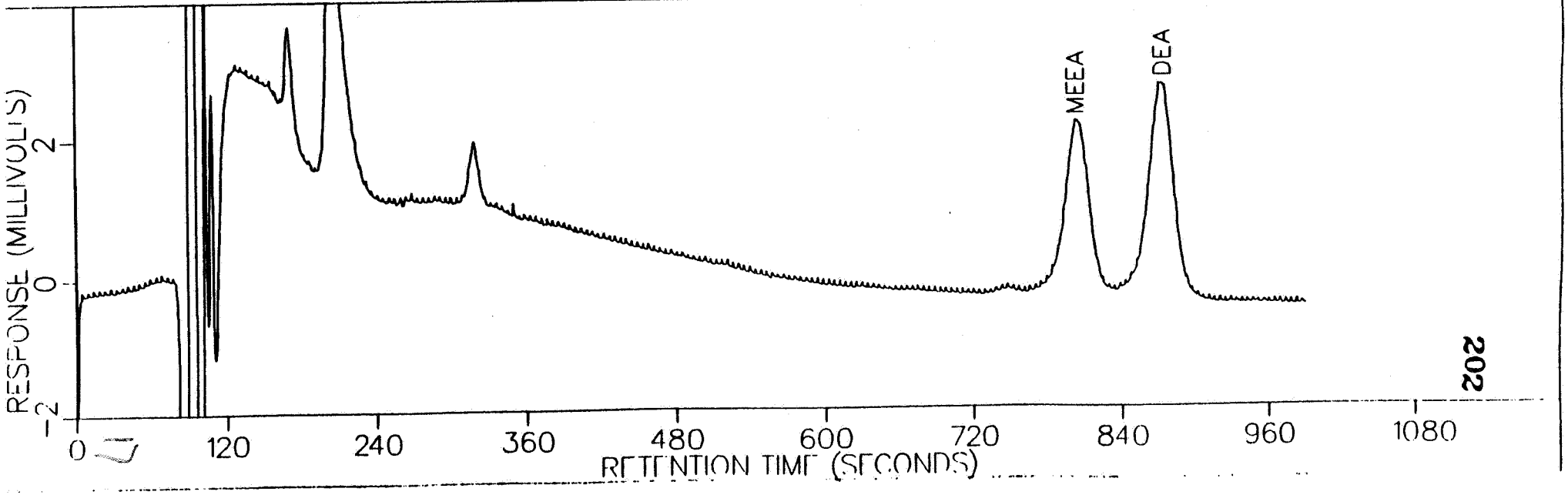
**Solid Line: Hydraulic Connections**
**Dotted Line: Electronic Connections**



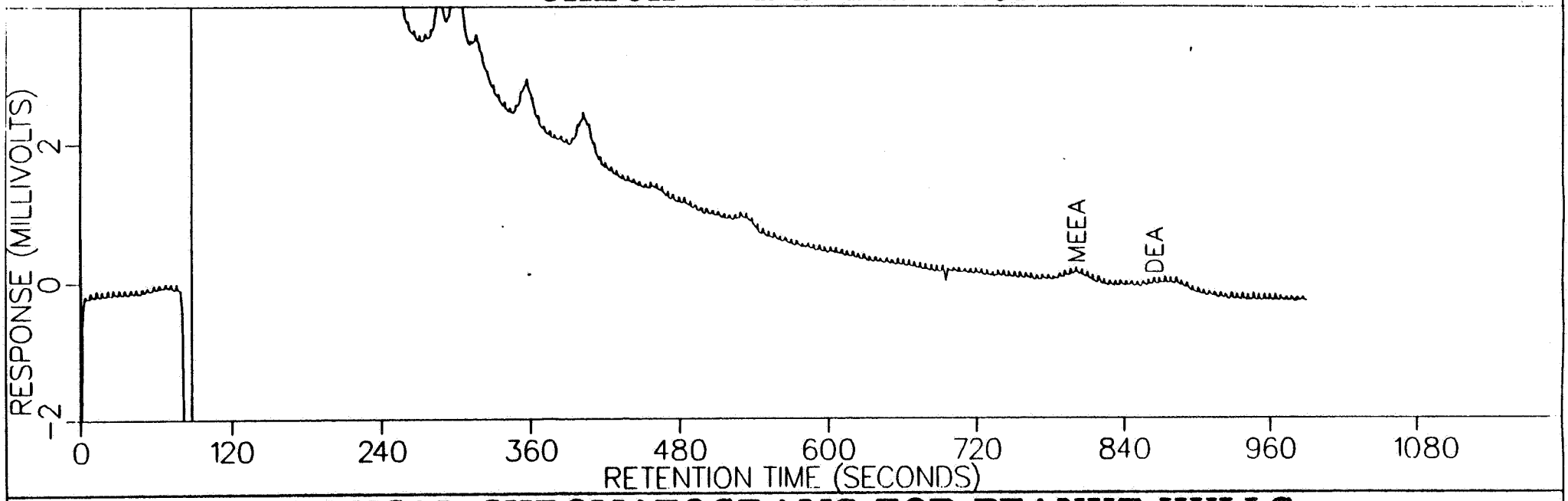
# TYPICAL CHROMATOGRAMS FOR PEANUT HULLS
# TREATED -- 8 LB/A -- 1/250 DILUTION



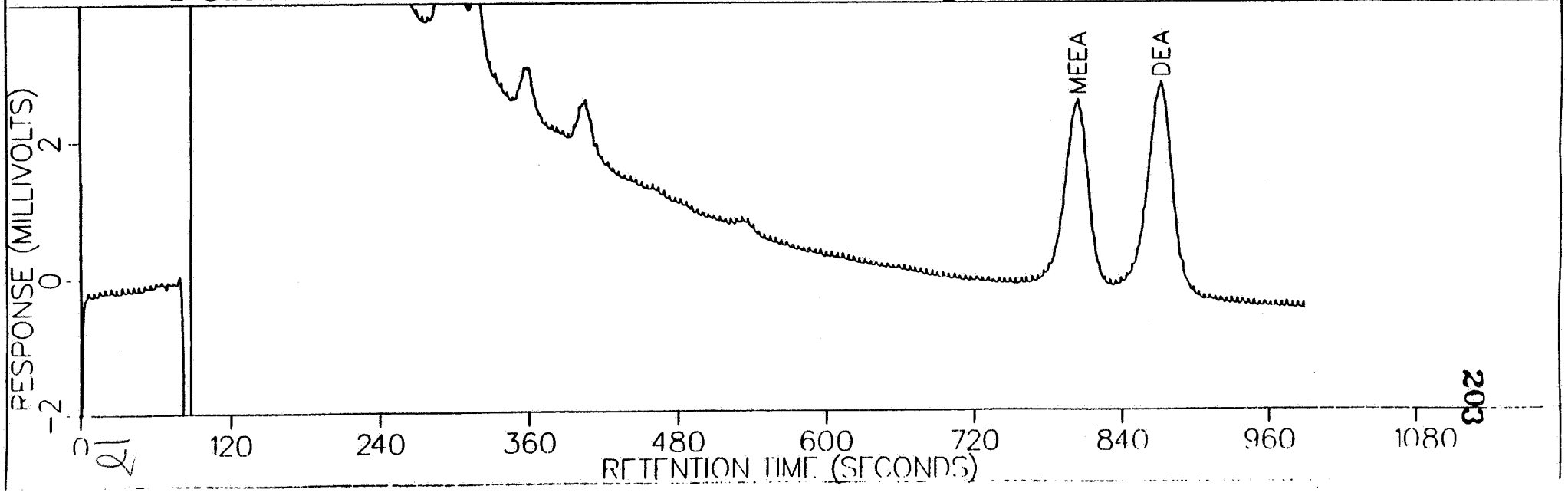

# TYPICAL CHROMATOGRAMS FOR PEANUT HULLS
# STANDARD 3



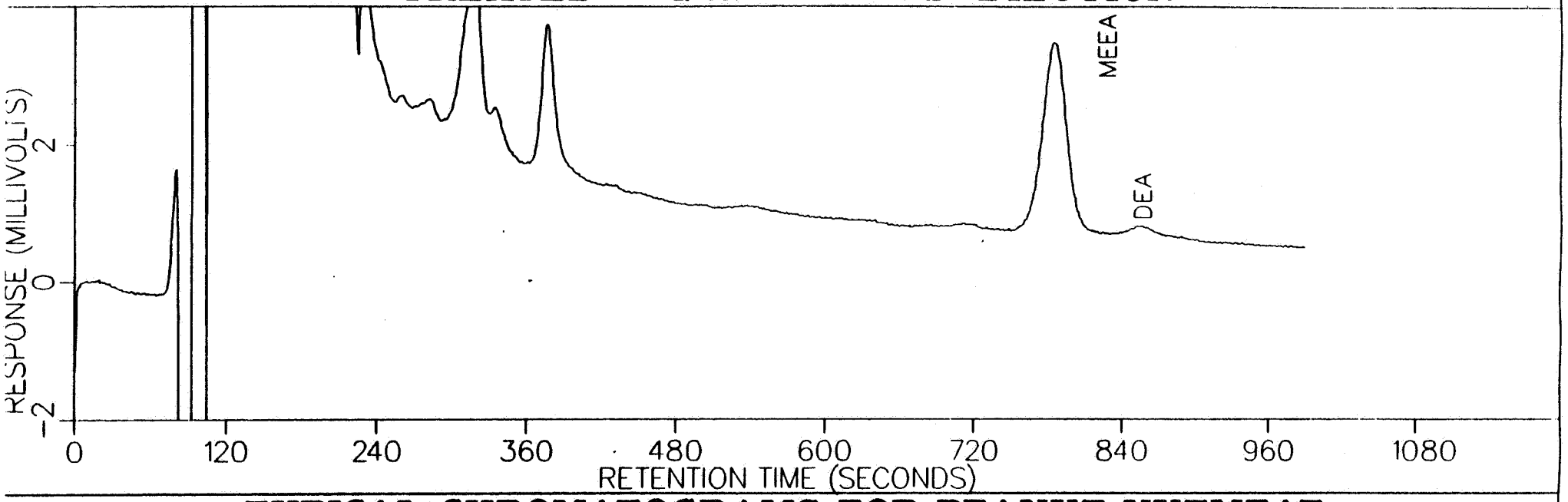
## TYPICAL CHROMATOGRAMS FOR PEANUT HULLS
## CHECK -- 1/25 DILUTION



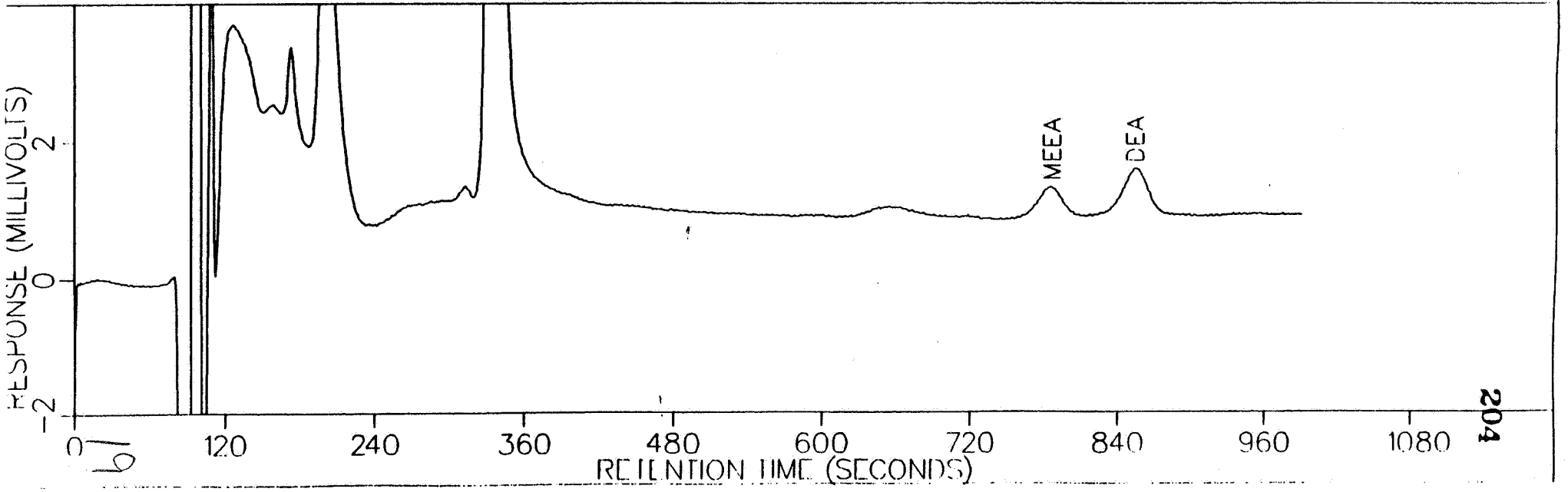

## TYPICAL CHROMATOGRAMS FOR PEANUT HULLS
## FORTIFIED -- 0.10 PPM ALACHLOR EQUIV. -- 1/25 DILUTION



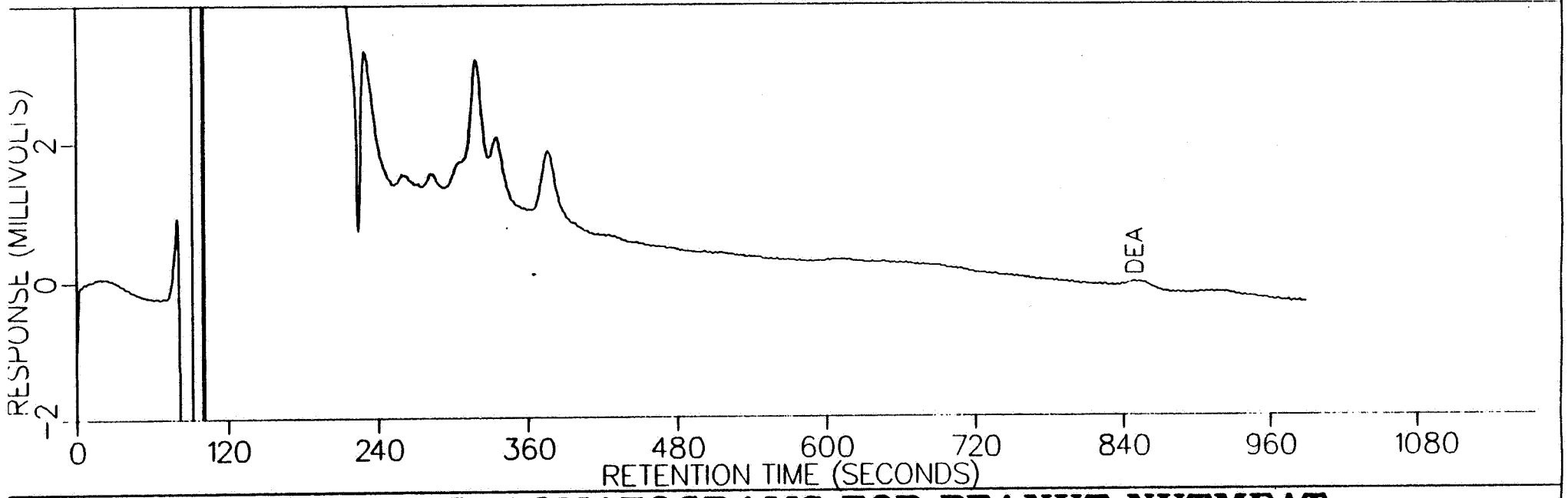
## TYPICAL CHROMATOGRAMS FOR PEANUT NUTMEAT TREATED -- 2 LB/A -- 1/25 DILUTION



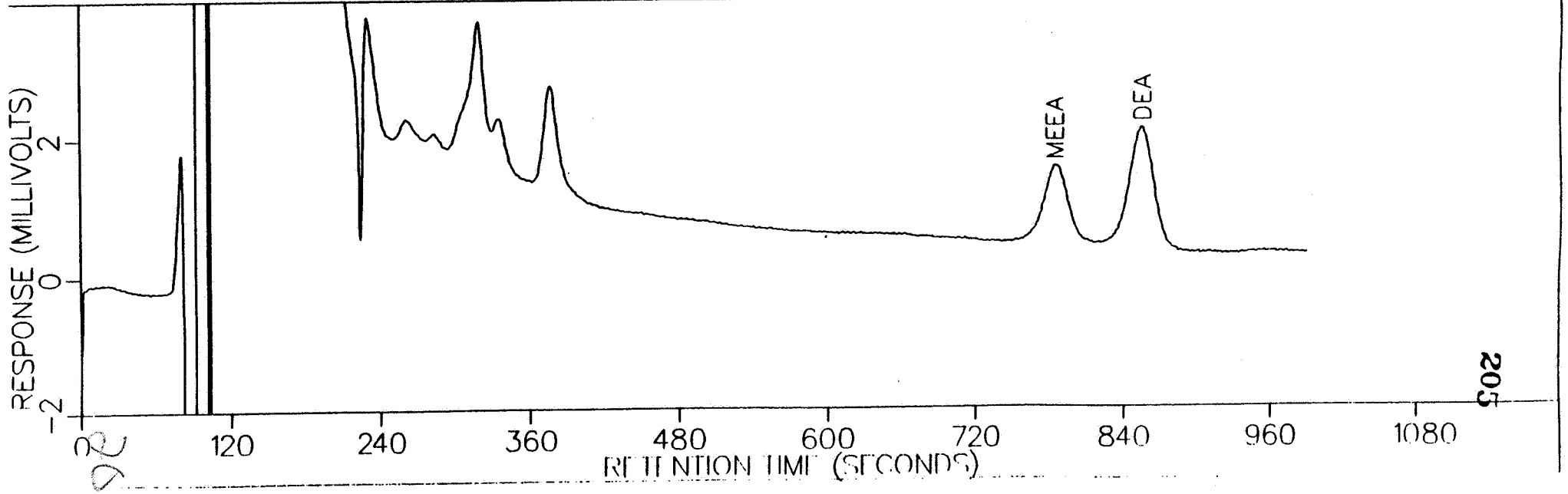

## TYPICAL CHROMATOGRAMS FOR PEANUT NUTMEAT STANDARD 2



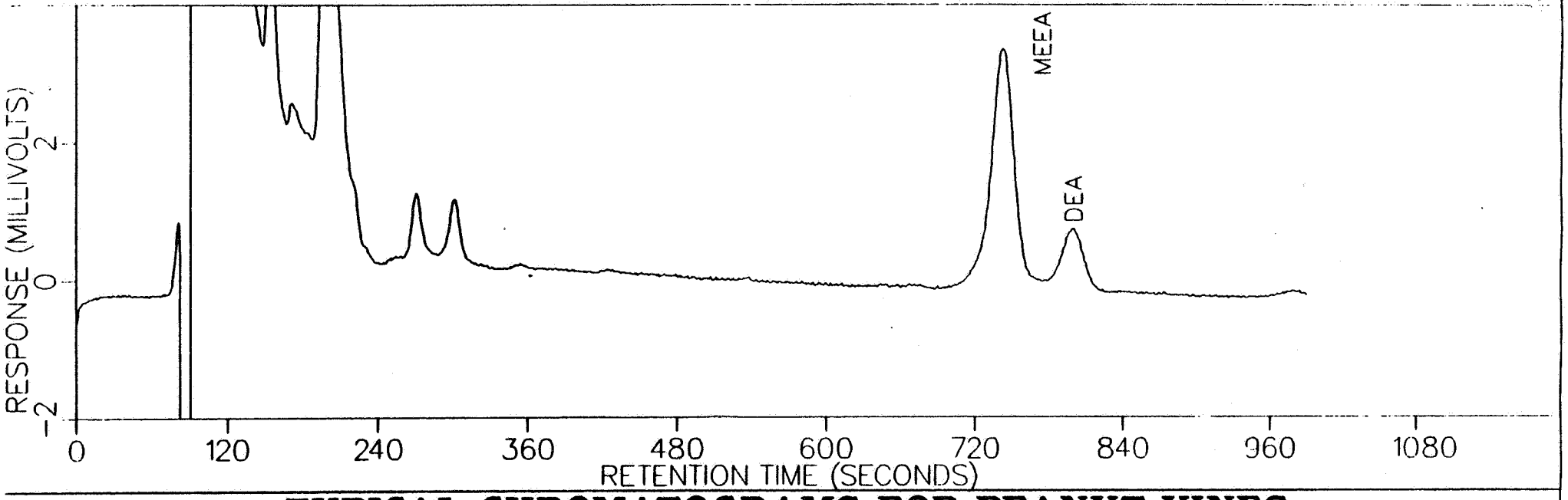
## TYPICAL CHROMATOGRAMS FOR PEANUT NUTMEAT
## CHECK -- 1/25 DILUTION



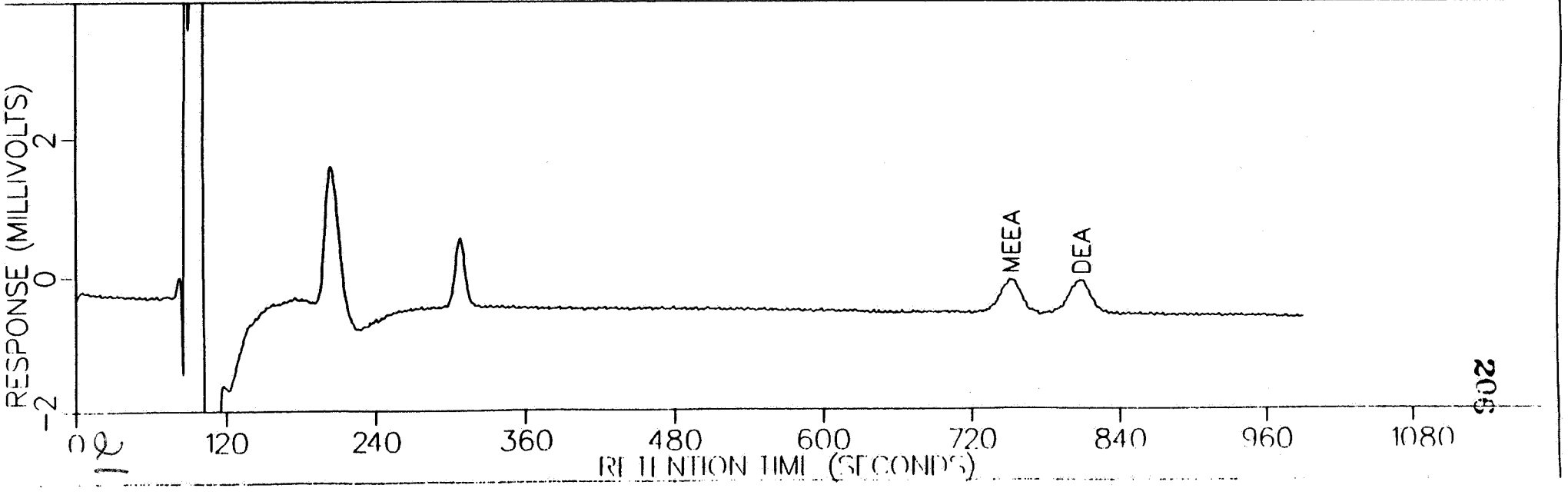

## TYPICAL CHROMATOGRAMS FOR PEANUT NUTMEAT
## FORTIFIED -- 0.05 PPM ALACHLOR EQUIV. -- 1/25 DILUTION



# TYPICAL CHROMATOGRAMS FOR PEANUT VINES
# TREATED -- 8 LB/A -- 1/250 DILUTION




# TYPICAL CHROMATOGRAMS FOR PEANUT VINES
# STANDARD 2

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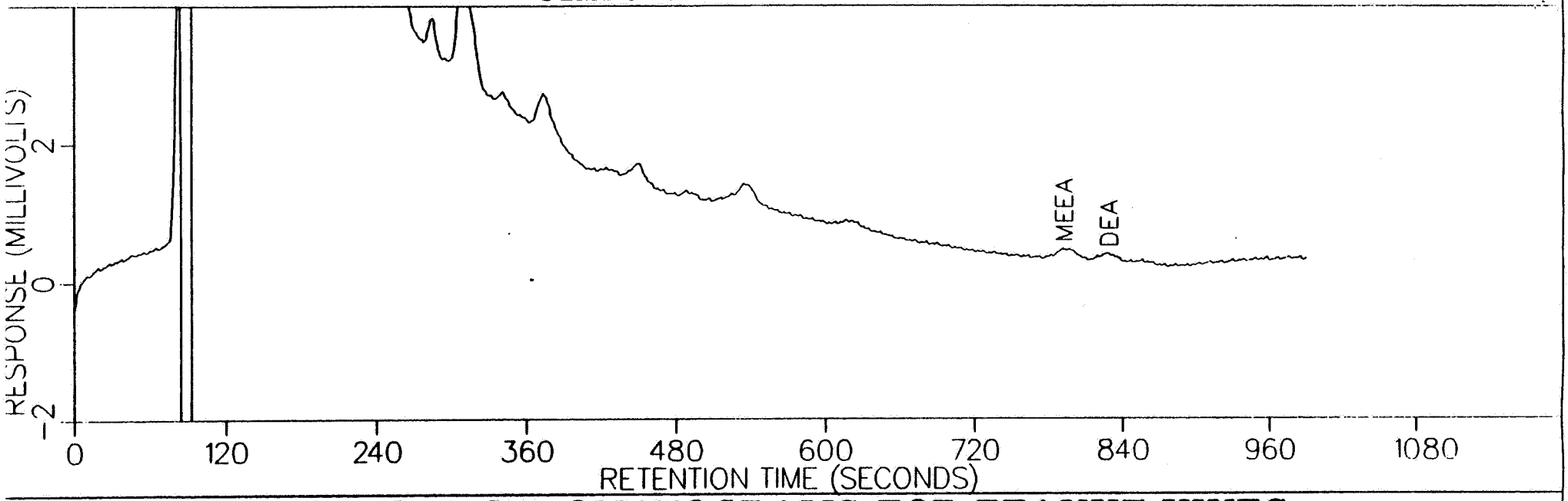
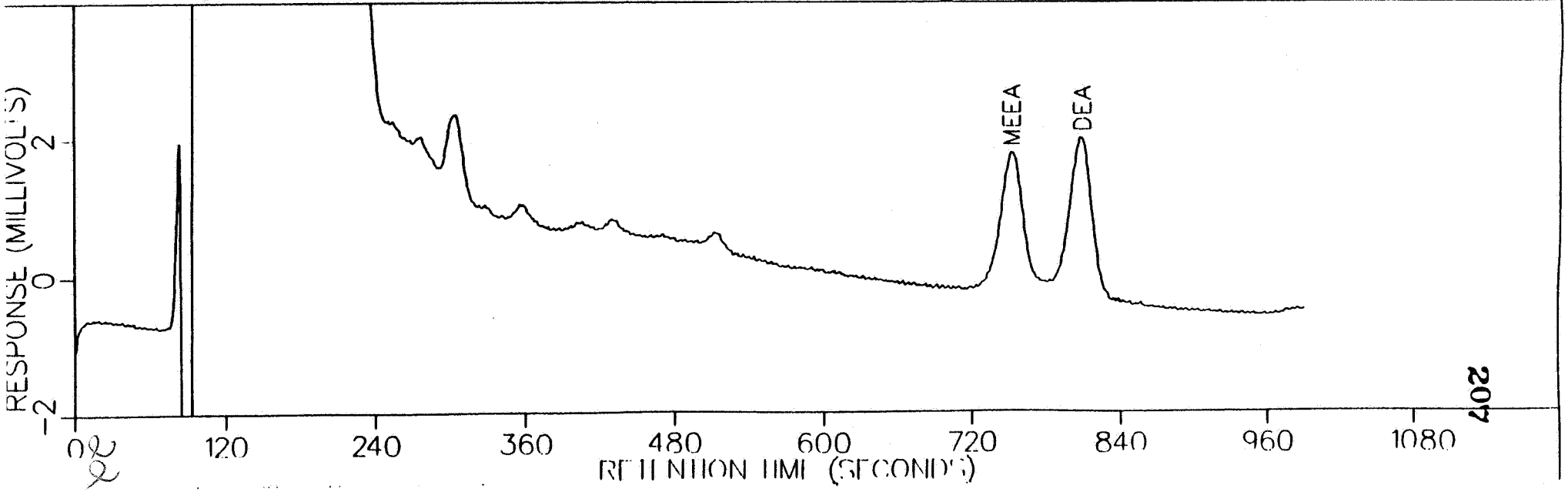
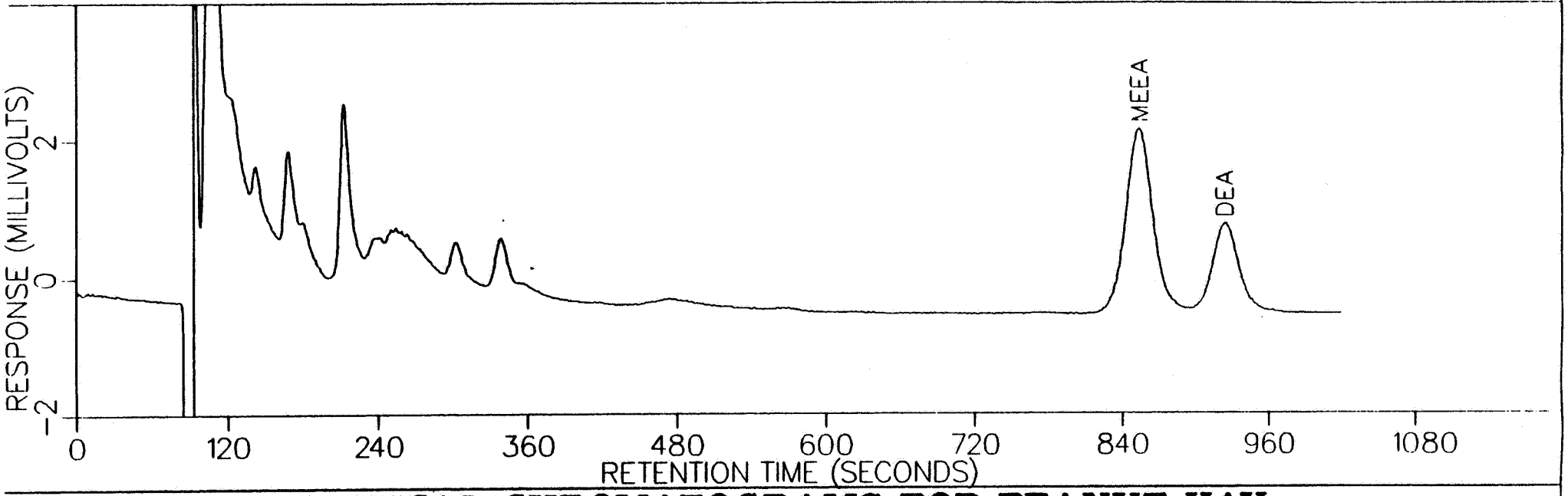
## TYPICAL CHROMATOGRAMS FOR PEANUT VINES
## CHECK -- 1/25 DILUTION



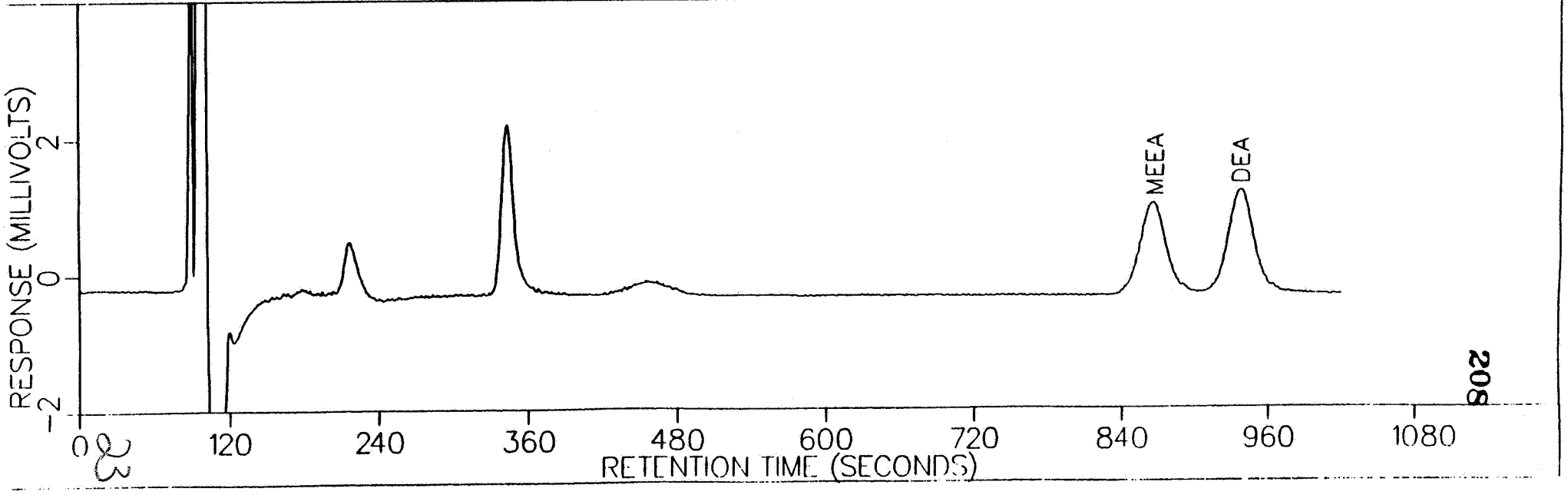

## TYPICAL CHROMATOGRAMS FOR PEANUT VINES
## FORTIFIED -- 0.10 PPM ALACHLOR EQUIV. -- 1/25 DILUTION



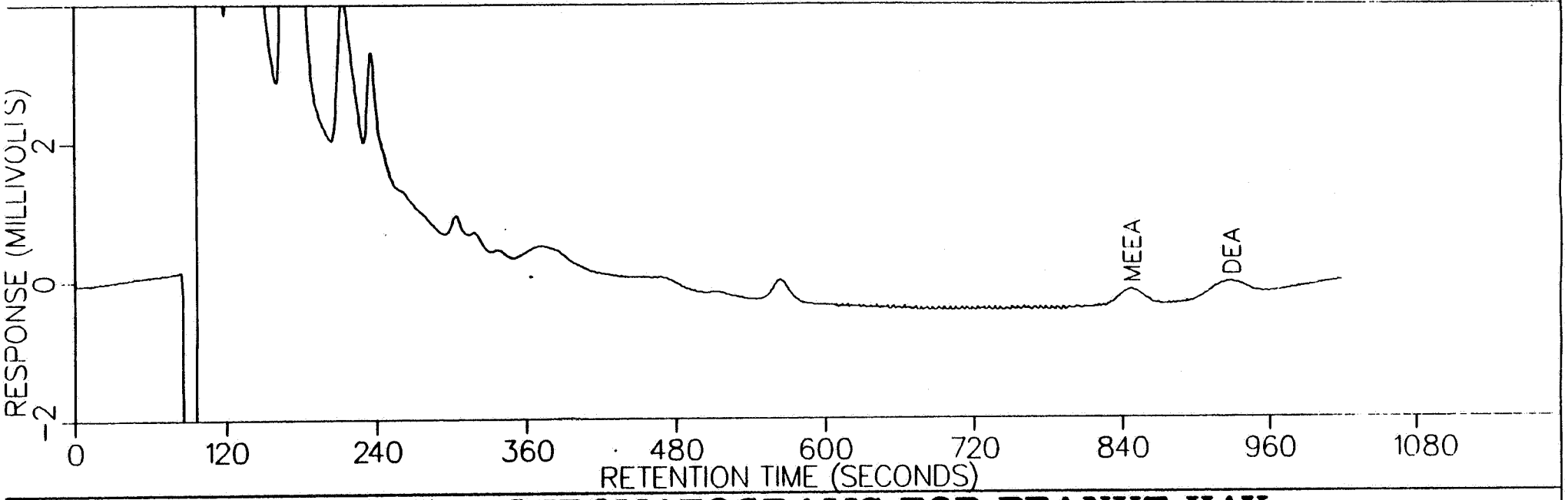
# TYPICAL CHROMATOGRAMS FOR PEANUT HAY TREATED -- 3 LB/A -- 1/250 DILUTION



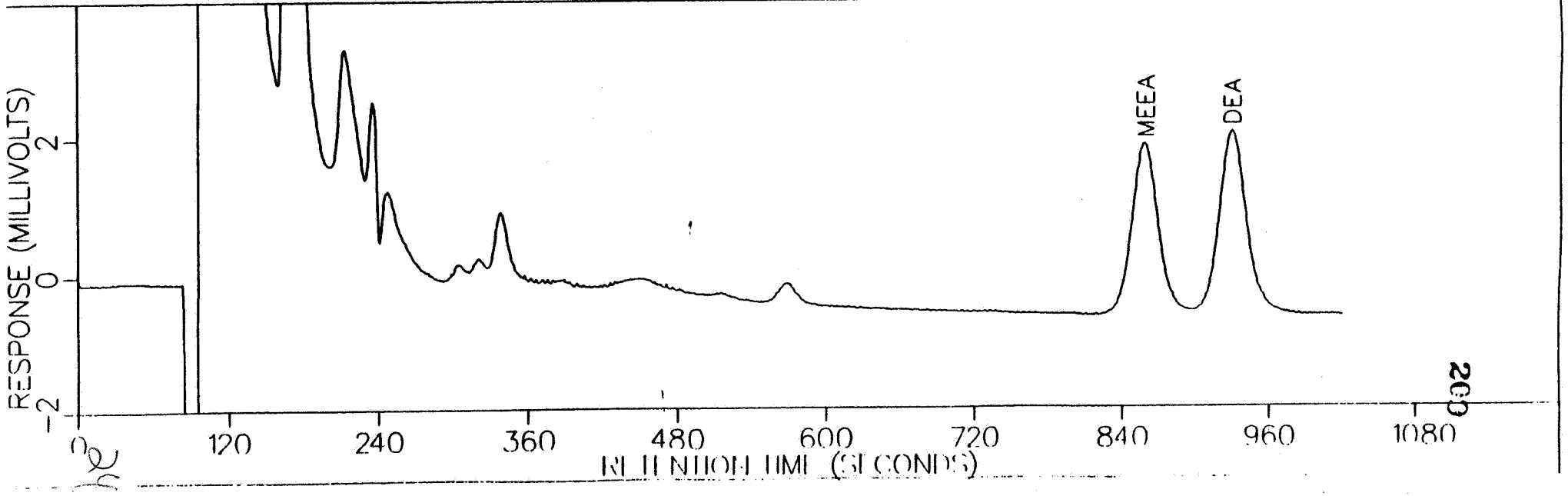

# TYPICAL CHROMATOGRAMS FOR PEANUT HAY STANDARD 3



# TYPICAL CHROMATOGRAMS FOR PEANUT HAY
## CHECK -- 1/25 DILUTION




# TYPICAL CHROMATOGRAMS FOR PEANUT HAY
## FORTIFIED -- 0.10 PPM ALACHLOR EQUIV. -- 1/25 DILUTION



Alachlor residue chemistry review

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Page 25 is not included in this copy.

Pages _____ through _____ are not included in this copy.

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The material not included contains the following type of information:

___ Identity of product inert ingredients

___ Identity of product impurities

___ Description of the product manufacturing process

___ Description of product quality control procedures

___ Identity of the source of product ingredients

X Sales or other commercial/financial information

___ A draft product label

___ The product confidential statement of formula

___ Information about a pending registration action

___ FIFRA registration data

___ The document is a duplicate of page(s) _____

___ The document is not responsive to the request

---

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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APPENDIX E: Storage Location and Raw Data

All raw data and associated documents are stored in the Residue Section archives. Crop samples are stored, frozen by the sample preparation group.

The raw data and documentation of procedures and methods used in this study can be found in the following Monsanto Company notebook pages:

3150238 - 3150300
3360801 - 3360809