

US EPA ARCHIVE DOCUMENT

GREENSBORO, N.C.

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|-----------------------------|----------------------|---|
| PAGE 1 of 16 | METHOD No. AG-334 | SUBJECT DETERMINATION OF RESIDUES OF METHIDATHION AND ITS METABOLITES GS-13007, GS-28369, AND GS-28370 IN <u>ANIMAL TISSUE</u> BY GAS CHROMATOGRAPHY EMPLOYING FLAME PHOTOMETRIC DETECTION |
| EDITION 1/9/79 | Update of AG-240 | |
| SUBMITTED BY: R.A. Kahrs | | |

APPROVED BY:

*R. T. Murphy*1.0 SCOPE

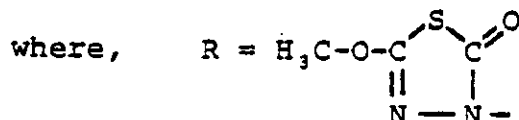
This method describes the analytical procedures for determining the residues of methidathion and three of its metabolites in turkey tissues.

Methidathion: $\{R\}-CH_2-S-\overset{S}{\underset{O}{P}}-(O-CH_3)_2$
 S-(2-methoxy-5-oxy- Δ^2 -1,3,4,-thiadiazolin-4-yl-methyl)
 -O,O-dimethyl-phosphorodithioate.

GS-13007: $\{R\}-CH_2-S-\overset{O}{\underset{O}{P}}-(O-CH_3)_2$
 S-(2-methoxy-5-oxo- Δ^2 -1,3,4,-thiadiazolin-4-yl-methyl)
 -O,O-dimethyl-phosphoro thioate.

GS-28369: $\{R\}-CH_2-SO_2-CH_3$ (Sulfone)
 2-methoxy-4-(methylsulfonylmethyl) Δ^2 -1,3,4,-thiadiazolin
 -5-one.

GS-28370: $\{R\}-CH_2-SO-CH_3$ (Sulfoxide)
 2-methoxy-4-(methylsulfinylmethyl) Δ^2 -1,3,4,-thiadiazolin
 -5-one.

2.0 PRINCIPLE OF METHOD

Methidathion and its metabolites are extracted from tissue by blending with an acetone/water mixture. The total acetone/water extract is combined with toluene to effect a phase separation. The organic phase contains methidathion, GS-13007, GS-28369 and 50% of the GS-28370, while the aqueous phase contains the remainder of the GS-28370. The aqueous phase is extracted with dichloromethane which is then combined with the organic phase, and evaporated to dryness. The residue is then partitioned between hexane and acetonitrile. The acetonitrile, containing the methidathion and its metabolites is evaporated to dryness, brought up in toluene. This solution is passed through a silica gel cleanup column to remove interfering material prior to gas chromatographic determination. The eluates containing the methidathion, GS-28368, GS-13007 and GS-28370 residues are evaporated to dryness, redissolved in acetone, and an aliquot taken for gas chromatographic analysis using a Flame Photometric Detector. Analysis of methidathion and GS-13007 residues can be made either phosphorus (526 nm) or sulfur (394 nm)

(over)

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2.0 PRINCIPLE OF METHOD, Cont'd.

specific filter (Phosphorus is the preferred mode of operation). GS-28369 and GS-28370 residues can only be determined using the sulfur specific filter. Quantitative measurements are made by comparison of peak height or areas with a standard curve.

Schematic Flow diagram of procedure shown in figure 1.

3.0 REAGENTS

- | | | |
|------|-------------------------|----------------------------------|
| 3.1 | <u>Acetone:</u> | Nanograde |
| 3.2 | <u>Toluene:</u> | Nanograde |
| 3.3 | <u>Dichloromethane:</u> | Spectroquality |
| 3.4 | <u>Distilled Water:</u> | |
| 3.5 | <u>Hexane:</u> | Nanograde |
| 3.6 | <u>Acetonitrile:</u> | Nanograde |
| 3.7 | <u>Sodium Sulfate:</u> | Anhydrous granular reagent |
| | | (Prewashed with dichloromethane) |
| 3.8 | <u>Silica Gel:</u> | Grade 923, 100-200 mesh |
| | | Davidson Chemical #923-08-08-226 |
| 3.9 | <u>GS-13005:</u> | Analytical Standard* |
| 3.10 | <u>GS-13007:</u> | Analytical Standard* |
| 3.11 | <u>GS-28369:</u> | Analytical Standard* |
| 3.12 | <u>GS-28370:</u> | Analytical Standard* |

*Available from Ciba-Geigy Corp.,
Greensboro, NC

4.0 EQUIPMENT

- | | | |
|-----|--------------------------|--|
| 4.1 | <u>Blender:</u> | Servall Omni-Mixer or equivalent #OM-115/XO |
| 4.2 | <u>Blender:</u> | Waring or equivalent |
| 4.3 | <u>Buchner Funnel:</u> | 9 cm |
| 4.4 | <u>Filter Paper:</u> | Whatman No. 1, 7 cm |
| 4.5 | <u>Vacuum adapter:</u> | For Erlenmeyer flasks; similar to No. K-20500 adapter, 24/40, Kontes Technical Glassware |
| 4.6 | <u>Filter vac:</u> | Rubber seal between Buchner funnel and vacuum adapter |
| 4.7 | <u>Flash Evaporator:</u> | "Rotavapor" Rotary Vacuum Evaporator Model VE 50 Rinco Instrument Co., or equivalent |

BIOCHEMISTRY DEPARTMENT

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4.0 EQUIPMENT, Cont'd.

- 4.8 Air Manifold: N-EVAP by Organomation or equivalent
- 4.9 Separatory funnels: 125, 250 and 500 ml
- 4.10 Chromatographic Column: 18 mm I.D., 200 mm long perforated plate bottom 100 ml reservoir joined at top Scient. Glass, Cat. No. 59.
- 4.11 Gas Chromatograph: Tracor-Microtek MT 220 equipped with a Flame Photometric Detector

5.0 PROCEDURE

Turkey tissues (breast skin, breast muscle, gizzard fat and liver) are worked up using the same procedure except where indicated.

5.1 Sample Preparation

- a) Cut tissue samples into 0.5-1.0g segments.
- b) Pulverize breast skin by blending sample with dry ice in a Waring blender. Allow the sample to come to room temperature before extraction.

5.2 Sample Extraction

- a) Add 25g of sodium sulfate to a 5-10 gram tissue sample and blend using the omni-mixer for one minute. (This will insure complete cell rupture).
- b) Add 125 ml of 8:2 Acetone:Water to the tissue sample and blend for 15 minutes at 7000-8000 rpm. The extraction ratio is as follows:

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5.2 Sample Extraction, Cont'd.Extraction Ratio

| | |
|----------------|------------|
| Breast Skin : | 10g/125 ml |
| Breast Muscle: | 10g/125 ml |
| Gizzard Fat : | 5g/125 ml |
| Liver : | 10g/125 ml |

- c) Filter the extract through one piece of filter paper in a Büchner funnel under vacuum.
- d) Reblend the filter pad for an additional 5 minutes with 50 ml of 8:2 Acetone: Water.
- e) Filter the extract as in step 5.2-c combining the two extracts for a total extraction. Wash the filtrate with 25 ml of 8:2 Acetone:Water for a total extract of 200 ml.

5.3 Wash-Out

- a) Transfer the total extract from step 5.2 to a 500 ml separatory funnel with 200 ml of toluene, and shake for 3 minutes to effect a good phase separation.
- b) Transfer the aqueous (lower phase) fraction to a 250 separatory funnel and extract with 3x50 ml of dichloromethane. Combine the extracts and dry through a 2 inch pad of sodium sulfate.
- c) Dry the organic (toluene-acetone upper phase) fraction through the sodium sulfate pad used in step 5.3-b combining the two fractions. Rinse the pad with 25 ml of toluene.
- d) Evaporate the combined fractions to an oil residue using a flash evaporator.

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5.4 Partition

- a) Dissolve the residue from 5.3 in 50 ml of hexane and transfer quantitatively to a 125 ml separatory funnel.
- b) Extract the hexane with three successive portions of acetonitrile (25,10,10 ml) by shaking vigorously for 30 seconds.
- c) Draw off the acetonitrile extracts and combine in a second 125 ml separatory funnel containing 50 ml of fresh hexane.
- d) Shake this mixture for 30 seconds and allow the layers to separate.
- e) Draw off the acetonitrile and evaporate to dryness using a flash-evaporator.

5.5 Column Cleanup

A silica gel column is used for removing interfering materials.

The column is prepared as follows:

- a) A glass wool plug is tamped in place at the bottom of the column.
- b) Add 2g of sodium sulfate, 4g of silica gel, and 2g sodium sulfate to the column and tap the column to eliminate channeling and insure uniform packing.
- c) Place another glass wool plug on top.
- d) Prewash the column with 20 ml of toluene. Discard this eluant.
- e) Dissolve the sample from 5.4 in 5 ml of toluene and transfer to the column.
- f) Transfer the remainder of the residue to the column with two successive 5 ml portions of toluene.
- g) When the last of the toluene fraction has just run into the column surface add 50 ml of 2% acetone in toluene to the column. This eluant will contain the methidathion.

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5.5 Column Cleanup, Cont'd.

- h) When the 2% fraction has run into the column, place a clean receiver flask under the column, and add 50 ml of 20% acetone in toluene to the column. This eluant will contain the GS-28369 and GS-13007.
- i) When the 20% fraction has run into the column, place a clean receiver flask under the column and add 50 ml of acetone to the column. This eluant will contain the GS-28370.
- j) Concentrate each of the three eluants to 2 ml on a flash-evaporator, transfer to 3 dram vials with acetone and evaporate to dryness with a gentle stream of nitrogen on an N-EVAP.

5.6 Determination of methidathion, GS-13007, GS-28369 and GS-28370

The final determination is made using a gas chromatograph (MT-220) equipped with a Flame Photometric Detector. A phosphorus filter (526 nm) is used for methidathion analysis and a sulfur filter (394nm) for GS-13007, GS-28369 and GS-28370. If instrument is not sensitive enough, a phosphorus filter is used for GS-13007.

Conditions used for gas chromatography are given in Table I.

Analysis of each compound follows the same procedure described below.

- a) Dissolve the residue from 5.5 in an appropriate amount of acetone.
- b) Prepare a stock solution of each compound by dissolving 100 mg of analytical standard in 100 ml of acetone. Make dilutions from this using acetone to obtain concentrations of 1, 2, 3 and 4 ng/ μ l.
- c) Standardize the gas chromatographic method by injecting known amounts of each compound.
- d) Inject samples for methidathion residues by using a phosphorus specific filter and a 3% SE-30 +0.3% Epon 1001 column (conditions are described in Table I) or the DC-200 column described in e).

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- e) Inject samples for GS-28369, GS-13007, and GS-28370 analysis using a sulfur filter and a 10% DC-200 column. (Conditions described in Table I).
- f) If sulfur sensitivity is inadequate for GS-13007, then inject sample again using the phosphorus specific filter.

5.6 Determination of methidathion, GS-13007, GS-28369 and GS-28370, Cont'd.

Typical chromatograms of injected standards are shown in Figures 2 thru 7. Peak areas or heights of unknown samples are compared directly with this graph to obtain the amount of compound present. Calculate results in terms of ppm by dividing the nanograms of the compound found by the milligrams of crop equivalent injected.

6.0 RECOVERY STUDIES

The following recoveries have been obtained from samples analyzed in our laboratory using this method. Samples were fortified with known amounts of methidathion, GS-13007, GS-28369 and GS-28370 prior to extraction at levels between 0.01-5.0 ppm.

Tissue samples of breast skin, breast muscle, gizzard fat, and liver were analyzed from AG-A 2534 and 2400.

Recoveries through cleanup column were:

Average Recovery (%)

| Compound | Breast Skin | Breast Muscle | Gizzard Fat | Liver |
|---------------|-------------|---------------|-------------|---------|
| Methidathion: | 110 (5) | 97 (5) | 98 (5) | 91 (4) |
| *GS-13007: | 100 (5) | 100 (5) | 100 (5) | 95 (5) |
| GS-28369: | 90 (4) | 112 (5) | 84 (5) | 110 (4) |
| GS-28370: | 96 (4) | 96 (5) | 81 (5) | 99 (5) |

Value in parenthesis indicates number of recoveries averaged.

*The recovery data for GS-13007 were obtained by TLC determination and not by GLC. They are included however to show that the GS-13007 does quantitatively pass through the procedure.

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7.0 ANALYSIS OF TREATED SAMPLES

Raw data obtained in the analysis of tissue samples is given in the following tables and figures.

| <u>AGA</u> | <u>Tissue</u> | <u>Figure</u> |
|------------|---------------|---------------|
| 2534 | Breast Muscle | 3,5,7,8 |

8.0 NOTES

- 8.1 The Flame Photometric Detector is very sensitive and specific in the phosphorus mode (526nm filter) of operation. Analysis of samples using this mode can be tried after the washout (step 5.3) or the partitioning (step 5.4) since partitioning and column cleanup are not always necessary.
- 8.2 In our laboratory, the Flame Photometric Detector in the sulfur (394 nm filter) mode is injection volume dependent. Therefore only 5 µl injections of standards and samples are used.
- 8.3 Fractions II (20% acetone/toluene) and III (acetone) may be combined after Step 5.5 and the residue injected to determine GS-28369 and GS-28370 simultaneously.
- 8.4 Determination of methidathion, GS-13007, GS-28369 and GS-28370 residues can be made using either of the gas chromatographic columns listed in Table I. All four compounds will elute from each of the columns.
- 8.5 Although written specifically for turkey tissues, this method has been successfully employed on chicken and beef tissue samples.

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TABLE I GAS CHROMATOGRAPHIC CONDITIONS

| | | |
|-------------------------------|---|---|
| Instrument: | Microtek MT-220 equipped with a Flame Photometric Detector | |
| Column: | a) 3% SE-30+0.3% Epon 1001 on Gas Chrom Q(60/80) in Pyrex tubing (4'x 1/4"). | b) 10% DC-200 on Gas Chrom.Q (60/80) in pyrex tubing (4'x1/4") |
| Injection port Temp: | 225°C | 225°C |
| Column Temp: | 220°C | a) 160°C b) 175°C |
| Detector Temp: | 190°C | 160°C |
| N ₂ Carrier Flow: | 80 cc/min | 80 cc/min |
| O ₂ Flow: | 20 cc/min | 20 cc/min |
| Air Flow: | 10 cc/min | 10 cc/min |
| H ₂ Flow: | 100 cc/min | 75 cc/min |
| Attenuation: | 10 ³ x 16 | 10 ⁴ x 8 |
| Detector Filter: | Phosphorus (526 nm) | Sulfur (394 nm) |
| Minimum Detectable amount: | 5 ng | 5 ng |
| Column Injected | 5 µl | 5 µl |
| Chart Speed: | 1/4"/min. | 1/4"/min. |
| Retention Time: | GS-13005-4.4 min. | a) GS-28369-4.8 min. b) GS-28370-4.0 min. |

Figure 1 SCHEMATIC FLOW DIAGRAM FOR ANALYSIS OF METHIDATHION
GS-13007, GS-28369 and GS-28370 RESIDUES IN
TURKEY TISSUES

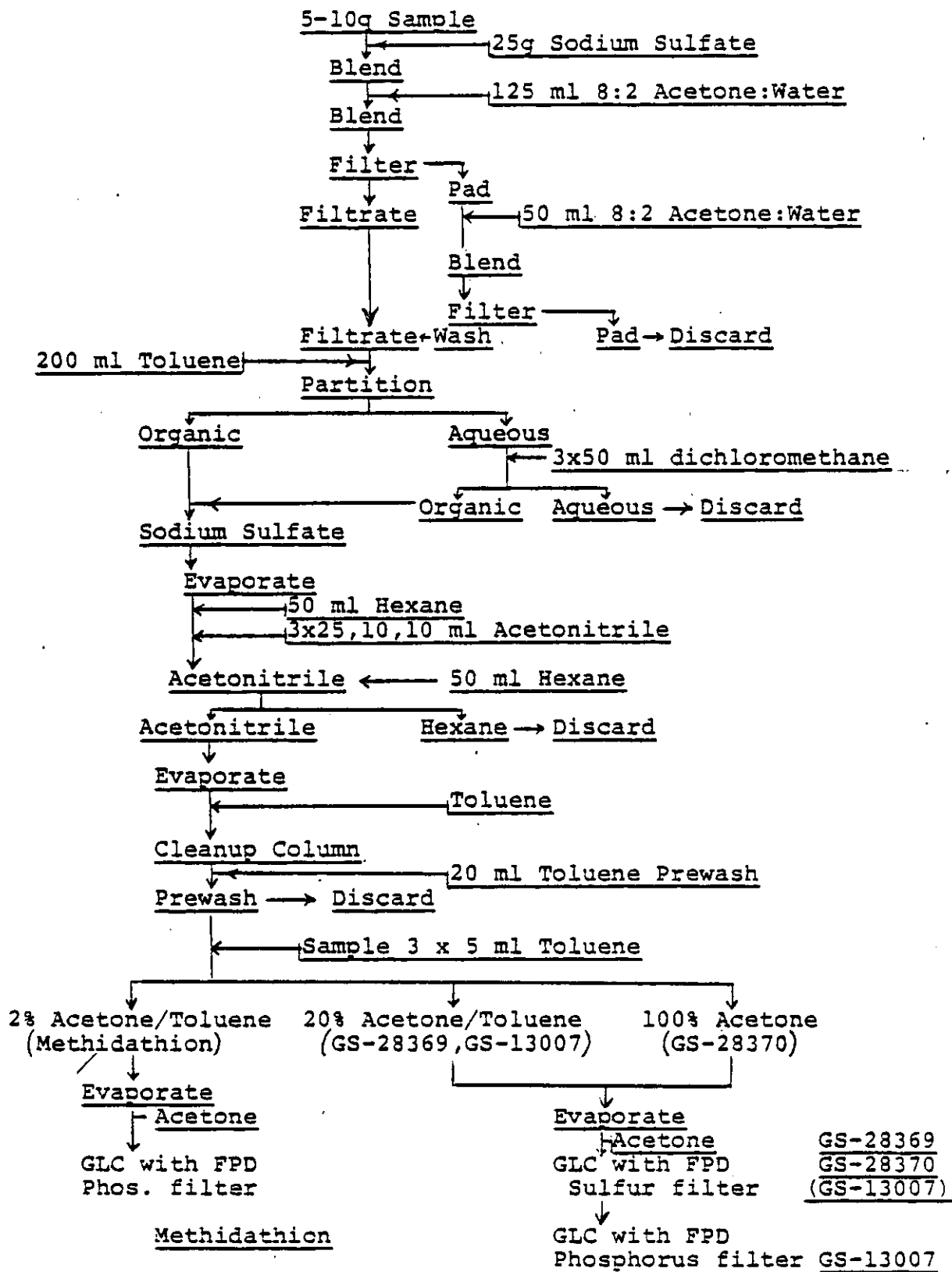
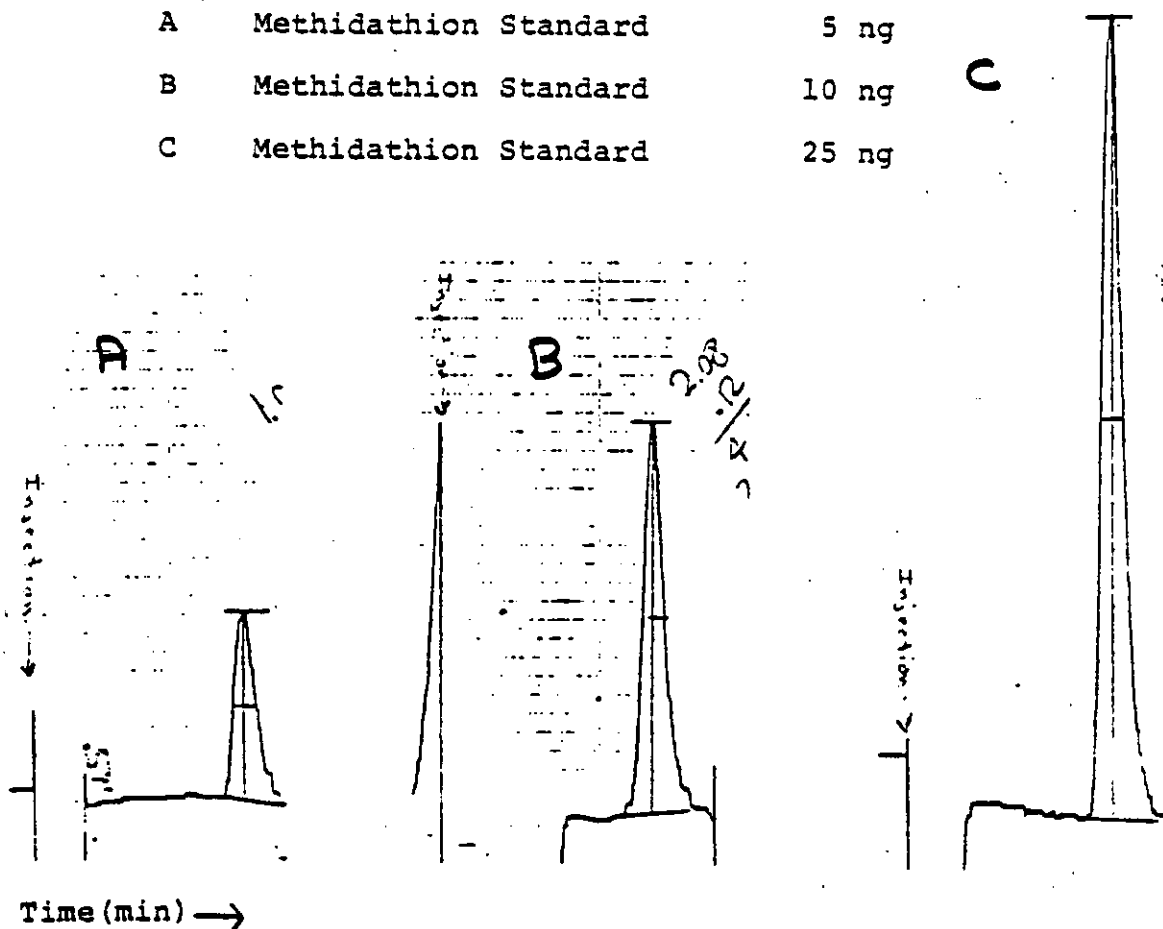


Figure 2 TYPICAL CHROMATOGRAMS OF METHIDATHION STANDARDS
ON FLAME PHOTOMETRIC DETECTOR IN THE PHOSPHORUS
MODE (526 nm)

A Methidathion Standard 5 ng
 B Methidathion Standard 10 ng
 C Methidathion Standard 25 ng



| | TRIANGLE | | | ng. |
|---|-----------|-------------|-------------------------|-----|
| | Base (in) | Height (in) | Area (in ²) | |
| A | 0.12 | 1.00 | .120 | 5 |
| B | 0.12 | 2.08 | .250 | 10 |
| C | 0.12 | 4.26 | .511 | 25 |

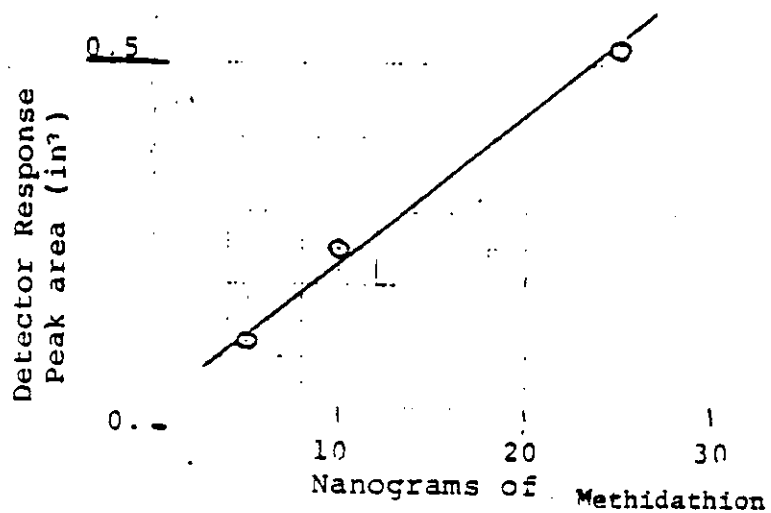
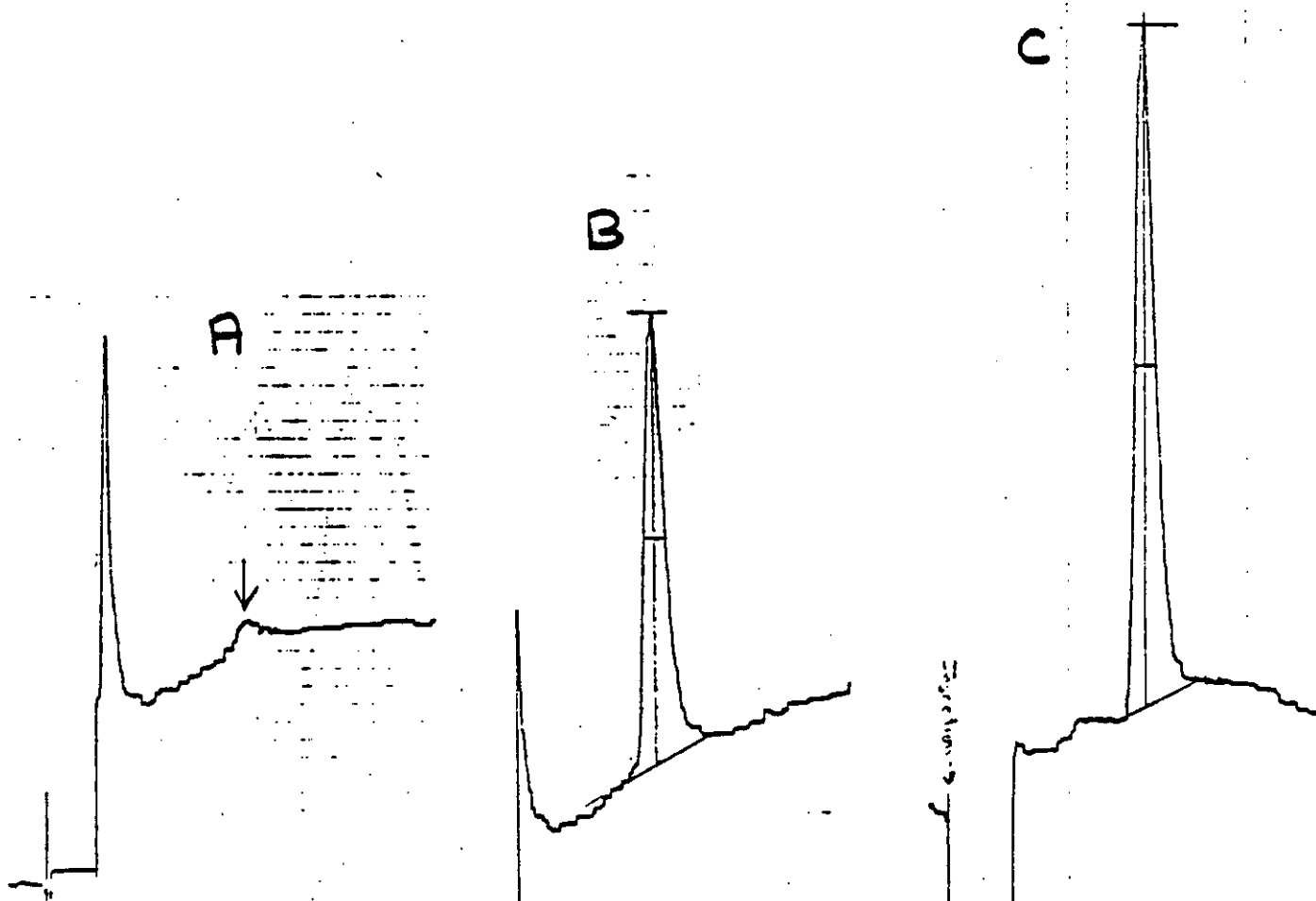


Figure 3 TYPICAL CHROMATOGRAMS OF METHIDATHION RECOVERY
FROM BREAST MUSCLE (AG-A 2534) FORTIFIED
AND TREATED SAMPLES

- A Untreated Sample
 B Fortified Sample at 0.05 ppm
 C Treated Sample 40+40 lb/A 2 day

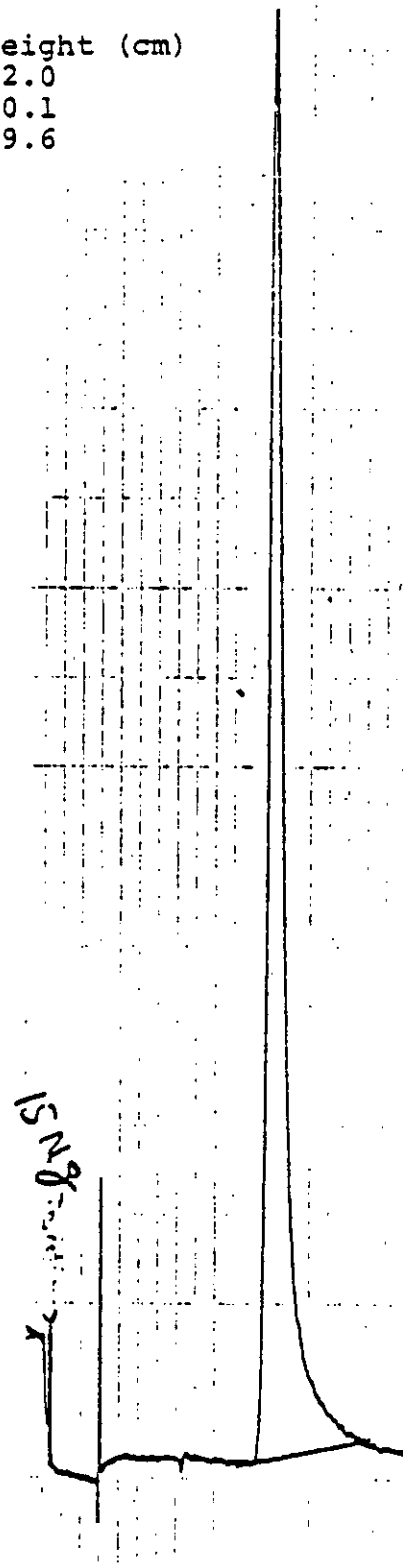
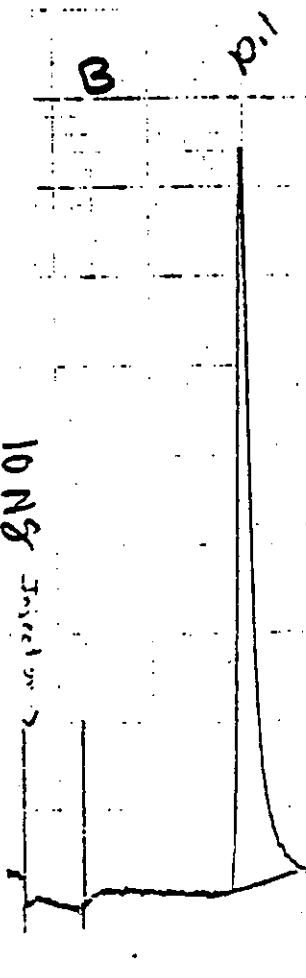
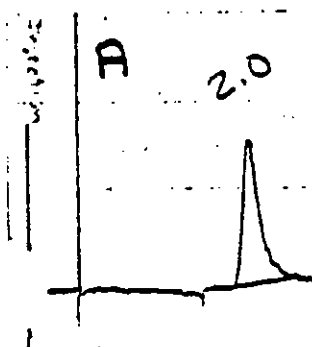
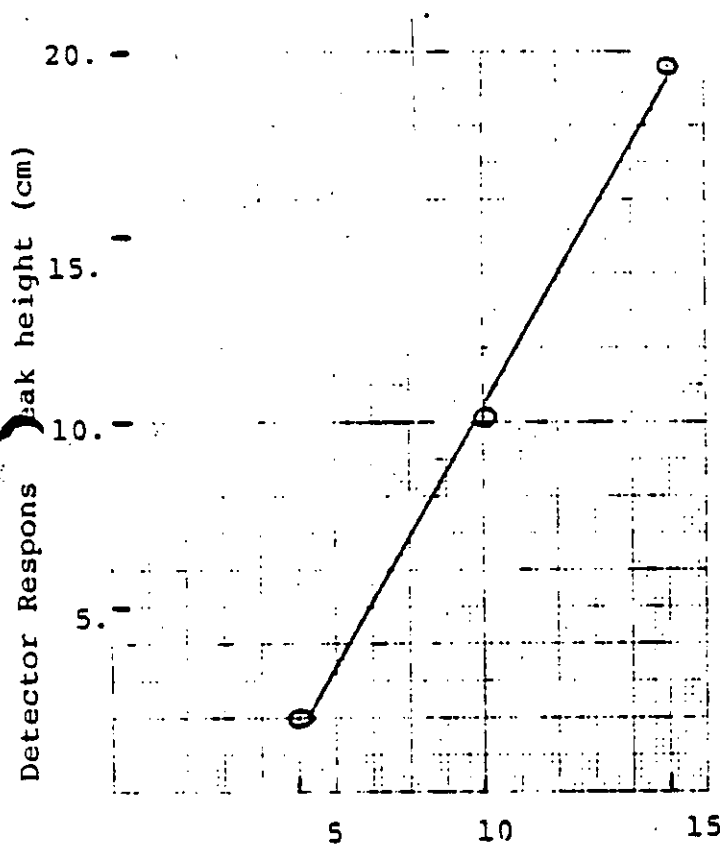


Time (min) →

| Analytical Number | Total (g/ml) | ALIQOT | | INJECTED | | TRIANGLE | | | ng. | PPM | | | Rec. % |
|-------------------|--------------|--------|-----|---------------------------|------|-----------|-------------|------|------|-------|------|------|--------|
| | | (ml) | (g) | ($\frac{\mu l}{\mu l}$) | mg. | Base (In) | Height (In) | A | | Find | Corr | Add | |
| A | | | 10 | 5/200 | 250. | - | - | - | <5. | <0.05 | | | |
| B | | | 10 | 5/200 | 250. | 0.12 | 2.55 | .306 | 14.6 | 0.06 | 0.06 | 0.05 | 117 |
| C | | | 10 | 5/500 | 100. | 0.12 | 3.83 | .457 | 22.6 | 0.23 | | | |

Figure 4 TYPICAL CHROMATOGRAMS OF GS-28369 STANDARD
ON FLAME PHOTOMETRIC DETECTOR IN THE SULFUR
MODE (394 nm)

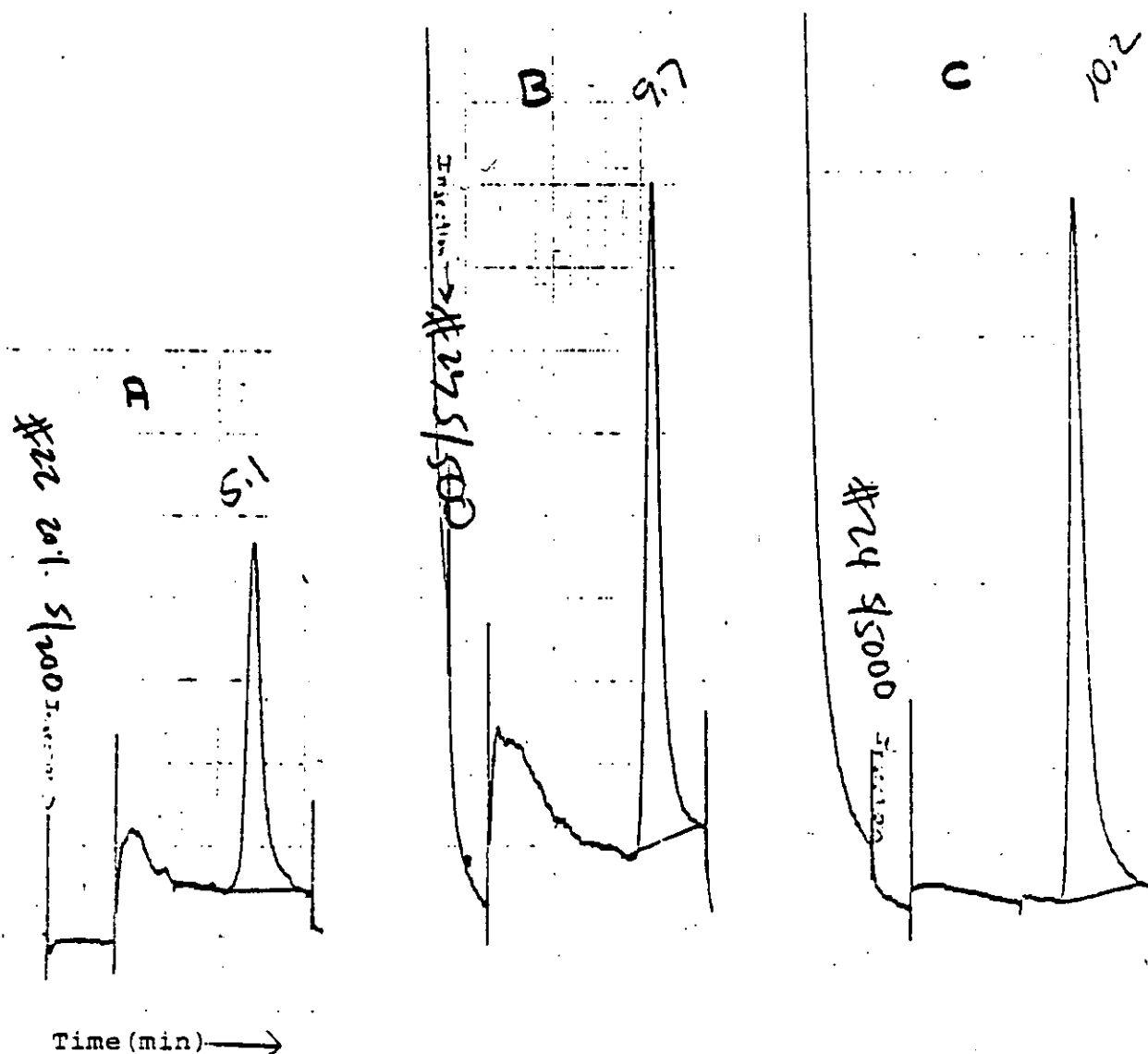
| Standard | Ng | Peak height (cm) |
|----------|----|------------------|
| A | 5 | 2.0 |
| B | 10 | 10.1 |
| C | 15 | 19.6 |



Time (min) →

Figure 5 TYPICAL CHROMATOGRAMS OF RECOVERY OF GS-28369
IN BREAST MUSCLE (AG-A 2534) FORTIFIED AND
TREATED SAMPLES

- A Untreated Sample
 B Fortified Sample at 0.05 ppm
 C Treated Sample 40+40 lb/A 2 days

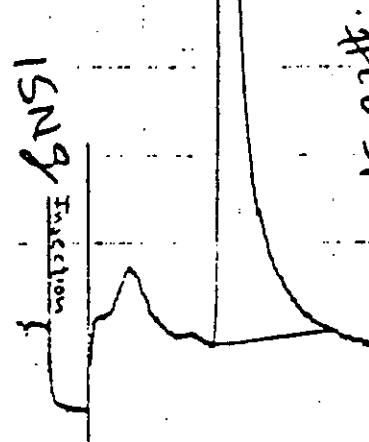
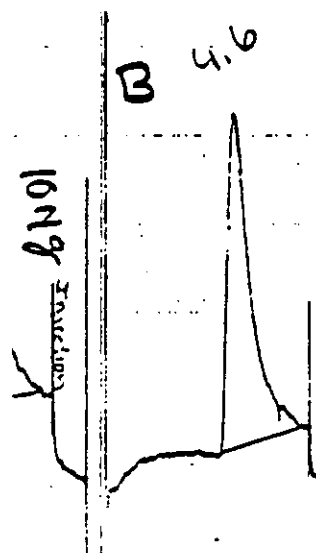
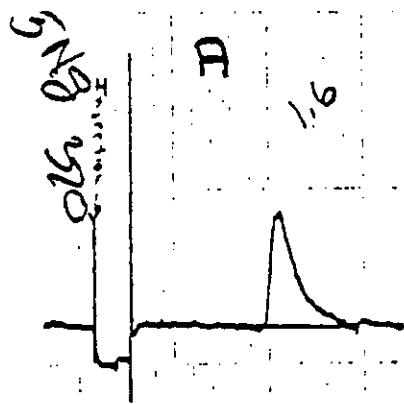
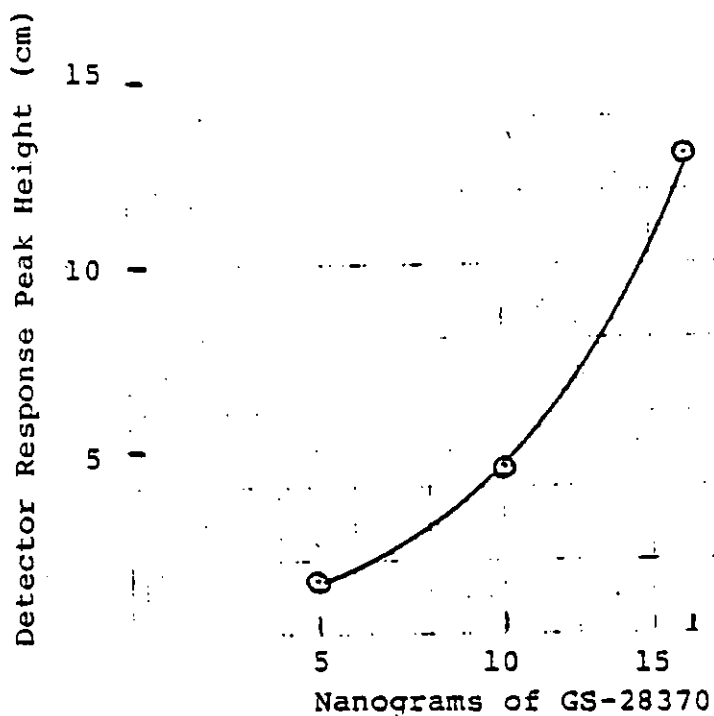


| Analytical Number | Total (g/ml) | ALIQOT | | INJECTED | | TRIANGLE | | | ng. | PPM | | | Rec. % |
|-------------------|--------------|--------|-------|---------------------------|-----|-----------|-------------|---|-----|-------|------|------|--------|
| | | (ml) | (g) | ($\frac{\mu l}{\mu l}$) | mg. | Base (in) | (cm) Height | A | | End | Corr | Add | |
| A | | 10 | 5/200 | 250. | | | 5.1 | | 6.6 | <0.05 | | | |
| B | | 10 | 5/200 | 100. | | | 9.7 | | 9.1 | 0.09 | 0.06 | 0.05 | 120 |
| C | | 10 | 5/200 | 10.0 | | | 10.2 | | 9.4 | 0.94 | | | |

Figure 6

TYPICAL CHROMATOGRAMS OF GS-28370 STANDARD
ON FLAME PHOTOMETRIC DETECTOR IN THE SULFUR
MODE (394 nm)

| Standard | Ng | Peak Height (cm) |
|----------|----|------------------|
| A | 5 | 1.6 |
| B | 10 | 4.6 |
| C | 15 | 13.0 |



Time (min) →

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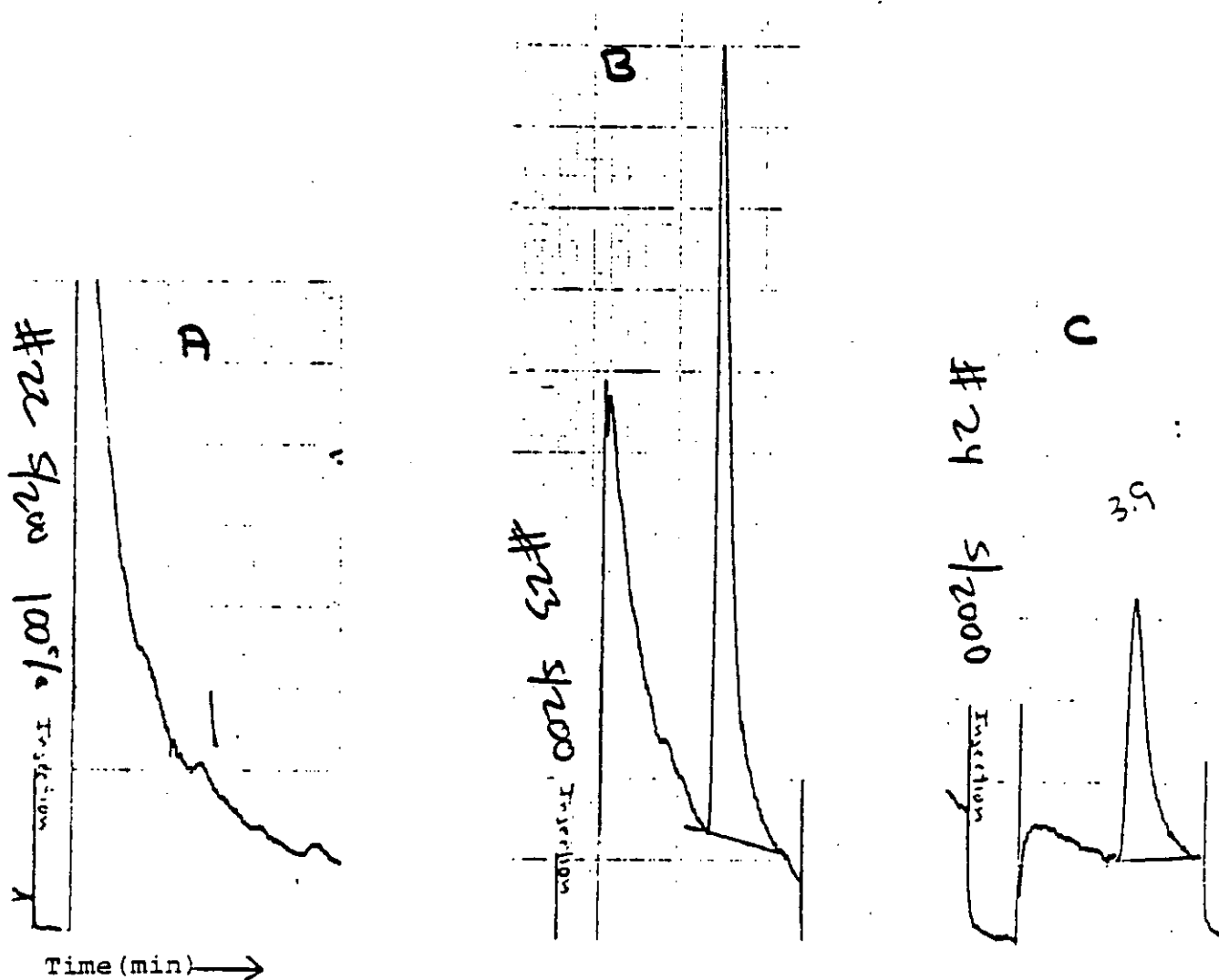
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Figure 7

TYPICAL CHROMATOGRAMS OF GS-28370 RECOVERY
IN BREAST MUSCLE (AG-A 2534) FORTIFIED AND
TREATED SAMPLES

- A Untreated Sample
 B Fortified Sample at 0.05 ppm
 C Treated Sample 40+40 lb/A 2 day



| Analytical Number | Total (g/ml) | ALIQOT | | INJECTED | | TRIANGLE | | | ng. | PPM | | | Rec. % |
|-------------------|--------------|--------|-----|---------------------------|------|-----------|-------------|---|------|-------|------|------|--------|
| | | (ml) | (g) | ($\frac{\mu l}{\mu l}$) | mg. | Base (In) | (cm) Height | A | | End | Corr | Add | |
| A | | | 10 | 5/200 | 250. | | - | | <5.0 | <0.05 | | | |
| B | | | 10 | 5/200 | 250. | | 11.7 | | 14.2 | 0.06 | 0.06 | 0.05 | 114 |
| C | | | 10 | 5/200 | 25.0 | | 3.9 | | 9.2 | 0.31 | | | |

ADDENDUM TO AG-334

GC CONDITIONS USED BY ADC LABS

7/15/82

All samples were analyzed according to methodology supplied by Ciba-Geigy Corporation. Milk samples were analyzed as described in Method No. AG-335 "Determination of Methidathion and Some of Its Metabolites in Milk by Gas Chromatography". Tissue samples were analyzed according to Method No. AG-334 "Determination of Residues of Methidathion and Its Metabolites, GS-13007, GS-38269, and GS-28370 in Animal Tissue by Gas Chromatography Employing Flame Photometric Detection". These methods were used without modification of any steps, except for filtration of liver samples. Liver extracts were filtered through two pieces of filter paper instead of one.

All gas chromatography was done on a Tracor Model 222 gas chromatograph operated as follows:

Column: 4' x 1/4" i.d. glass packed with 10% DC-200 on Gas Chrom Q, 80/100 mesh

Injection Port Temperature: 239°C
Column Temperature: 200°C
Detector Temperature: 219°C

N₂ Carrier Flow (Rotameter): 10
O₂ Flow (Rotameter): 8
Air Flow (Rotameter): 100+
H₂ Flow (Rotameter): 100

Detector Filter: Sulfur for milk; Phosphorous for tissues

Attenuation: 10³ x 32 for milk; 10³ x 128 & 10³ x 64 for tissues

Injection Volume: 5 µl

Chart Speed: 0.25 in/min

Retention Time: GS-13007 - 4.25 min
GS-13005 - 5.83 min

meat

ADDENDUM TO AG-334

GC CONDITIONS USED BY ADC LABS

7/15/82

All samples were analyzed according to methodology supplied by Ciba-Geigy Corporation. Milk samples were analyzed as described in Method No. AG-335 "Determination of Methidathion and Some of Its Metabolites in Milk by Gas Chromatography". Tissue samples were analyzed according to Method No. AG-334 "Determination of Residues of Methidathion and Its Metabolites, GS-15007, GS-28369, and GS-28370 in Animal Tissue by Gas Chromatography Employing Flame Photometric Detection". These methods were used without modification of any steps.

All gas chromatography was done on a Tracor Model 222 gas chromatograph operated as follows:

Column: 4' x 1/4" i.d. glass packed with 10% DC-200 on Gas Chrom Q, 80/100 mesh

Injection Port Temperature: 239°C
Column Temperature: 163°C
Detector Temperature: 219°C

N₂ Carrier Flow (Rotameter): 10
O₂ Flow (Rotameter): 8
Air Flow (Rotameter): 100+
H₂ Flow (Rotameter): 100

Detector Filter: Sulfur

Attenuation: 10³ x 32 and 10³ x 64

Injection Volume: 5 µl

Chart Speed: 0.25 in/min

Retention Time: GS-28369 - 5.24 min
GS-28370 - 6.03 min