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Research Department

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Procedure No. 1047 Copy No. 1 Page No. 1 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed D. Furmanec RP

SUMMARY

A general analytical method for the determination of Resmethrin residues in diverse types of food products is described. The procedure has been successfully applied to the following substances; sliced american cheese, sliced ham, cream pie, white flour, polished rice, white bread, popcorn kernels, saltine crackers, sliced roast beef,chicken meat, skin, fat, hearts, liver, gizzard and eggs.

Whole milk and sugar require a different sample extraction, details are in notes 1 and 2. Recoveries of Resmethrin added to these substances at the 1 ppm level range from 85 to 101% with a detection limit of 0.1 ppm.

Sample preparation involves extraction in a blender with acetonitrile, partitioning between acetonitrile and petroleum ether and clean-up on Florisil[R] and Alumin AR columns.

Three chromatographic columns are mandated. The 3% OV-1 column is the primary column. If interfering peaks are encountered chromatography on 3% OV-210 or on "mixed column" (50:50 mixture of 3% OV-17 on 80-100 mesh Varaport 30 and 3% OV-1 on 100-120 mesh Gas Chrom Q) will separate the interferences or vice-versa.

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Procedure No. 1047 Copy No. 1 Page No. 2 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Signed D. Furmanec RP

Date 9/9/82

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EQUIPMENT

Hewlett Packard Gas Chromatograph 5840A
Automatic sampler 7671A Hewlett-Packard
GC Glass Column 6 ft. x 2 mm I.D. 1/4" O.D.
Blender (Waring)
Flash-evaporator (Searle)
Separatory Funnel 2 liter
Round Bottom Flask, 500 ml. 250 ml.
Pear-shaped Flask, 50 ml.
Clean-up column glass, 450 mm long x 15 mm I.D.
Pipet, 1.0 ml., 5.0 ml., 25.0 ml.
Buchner Funnel 70 mm Dia.
Suction Flask, 1000 ml.
Whatman #1 filter paper 70 mm.

MATERIALS:

Acetonitrile, HPLC grade
Hexane, HPLC grade
Petroleum Ether, Pesticide grade
Methylene Chloride, Pesticide grade
Ether Anhydrous, Reagent A.C.S.
Acetone, HPLC grade
Sodium Sulfate, Anhydrous - Reagent grade
Sodium Chloride, Reagent grade
3% OV-1 on 100-120 Mesh on Gas Chrom Q (Applied Science Lab., Inc.)
3% OV-210 on 100-120 Mesh on Gas Chrom Q (Applied Science Lab., Inc.)
3% OV-17 on 80-100 Mesh Varaport 30 (Varian Aerograph)
Florasil 60-100 PR Mesh (Floridin Company)
Alumin AR CC-10 100-200 Mesh Activity Grade 4 (Mallinckrodt Chemical Works)
Resmethrin Standard (Penick Corporation)

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Procedure No. 1047 Copy No. 1 Page No. 3 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed D. Fuhrmann RP

PROCEDURE:

Weigh accurately 25.0 gm. of a representative food sample into blender (If milk or sugar see notes 1 and 2). Add 100 ml. of acetonitrile and activate the blender for 5 minutes. Decant the solvent through Whatman #1 filter paper in a Buchner funnel using gentle suction. Repeat the extraction in the blender two additional times using 100 ml and 50 ml portions of acetonitrile and filter each time through the same Whatman #1 filter paper. Transfer combined extracts to a 2 liter separatory funnel.

Acetonitrile - Petroleum Ether Partition

Add 100 ml. petroleum ether to the acetonitrile filtrate and shake well. Then add 75 ml. saturated salt solution, 700 ml. of distilled water and shake again. Transfer the petroleum ether layer to 250 ml. round bottom flask. Extract two additional times with 100 ml. and 50 ml. portions of petroleum ether. Each time remove petroleum ether in the same round bottom flask on a flash evaporator under vacuum at 50°C to an oily residue. Quantitatively transfer the oily residue to 25 ml. volumetric flask with small portions of hexane and dilute to volume.

COLUMN CLEAN-UP:

Florisil^{R} Column

Pack a chromatographic column (450 mm l x 15 mm I.D.) with 10 gm. of Florisil^{R} (activated at 600°C). Add a 10 mm layer of anhydrous sodium sulfate, to the top of the column, pre-wet column with hexane and drain to the top of the bed. Transfer 5.0 ml. sample aliquot to the top of the column and drain into the bed of the column.

Elute the column with 100 ml. of solvent mixture 50% ether - 50% hexane, and collect the total eluate. Concentrate the eluate on flash-evaporator under vacuum at 50°C to an oily residue.

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Procedure No. 1047 Copy No. \ Page No. 4 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed D. Fuhrmann RP

Alumin AR Column:

Pack a chromatographic column (450 mm l x 15 mm I.D.) with 20 gm of Alumin AR. Add a 10 mm. layer of anhydrous sodium sulfate to the top of the column. Pre-wet column with hexane and drain to the top of the bed. Transfer quantitatively the residue with three 5 ml. portions of hexane to the top of the column and drain each time into the bed of the column. Elute the column with 100 ml. hexane and discard the eluate. Continue eluation with 100 ml. of solvent mixture 25 ml. methylene chloride and 75 ml. hexane and collect the eluate in 250 ml. round bottom flask. Evaporate the eluate on a flash-evaporator under vacuum at 50°C to about 5 ml. Transfer quantitatively the residue with 2 x 5 ml. hexane to 50 ml. pear-shaped flask and evaporate the hexane on a flash evaporator again.

Sample Preparation:

Add 1.0 ml. hexane to the pear shaped flask and dissolve the residue.

Standard Preparation:

Dissolve 25 mg. of standard SBP-1382 accurately weighed in a 250 ml. volumetric flask and dilute to volume with hexane. Dilute 5.0 ml. aliquot to 100 ml. with hexane. Final concentration 0.005 mg/ml.

GLC Analysis:

Equipment and Conditions:

Instrument:	Hewlett Packard Gas Chromatograph 5840A
Column:	a) 6 ft. x 2 mm. I.D. 1/4" O.D. glass packed with 3% OV-1 on 100-120 mesh Gas Chrom Q
	b) 6 ft. x 2 mm. I.D. 1/4" O.D. glass packed with 3% OV-210 on 100-120 Mesh Gas Chrom Q
	c) "Mixed Column" - 50:50 mixture of 3% OV-17 on 80-100 mesh Varaport 30 and 3% OV-1 on 80-120 mesh Gas Chrom Q.
Temperature:	Injector: 250°C
	Detector (F.I.D.) 250°C

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Procedure No. 1047 Copy No. 1 Page No. 5 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed D. Fuhrmann RP

Column Oven Temperature:

I. Primary Column: 3% OV-1 180-250°C at the rate of 5°C per minute and held at 250°C for 15 min.

II. First Alternate Column: 3% OV-210 100-250°C at the rate of 10.°C per minute and held at 250°C for 15 min

III. Second Alternate Column: "Mixed Column" - 50:50 mixture of 3% OV-17 on 80-100 mesh Varaport 30 and 3% OV-1 on 100-120 mesh Gas Chrom Q.
(A) 180-250°C at the rate of 5°C per minute and held at 250°C for 15 min.
(B) Isothermally at 230°C.

Carrier Gas: Helium at 30 ml per minute
Hydrogen pressure - 20 p.s.i.g.
Air pressure - 40 p.s.i.g.

Attenuation: 2 (4)

Determination:

Condition the column before use by injecting the standard several times. Inject the sample and check for interfering peaks. If interfering peaks are encountered use the alternate column (3% OV-210). Inject 5.0 ul of the standard and sample preparation until sufficient chromatograms of each are obtained.

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed L. F. Wiemann RP

Calculation:

Calculation are based on equal volume injection using peak areas. From the chromatograms of the standard and sample preparation calculate ppm of SBP-1382 in the sample by the following formula:

$$\frac{A_{(s)}}{A_{(std)}} \times \frac{W_{(std)}}{W_{(s)}} \times 10^6 = \text{ppm of SBP-1382}$$

Where:

$A_{(s)}$ = peak area of SBP-1382 in the sample

$A_{(std)}$ = peak area of SBP-1382 in the standard

$W_{(s)}$ = peak of the sample in mgs.

$W_{(std)}$ = weight of the standard in mgs./ml. in final dilution.

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Procedure No. 1047 Copy No. 1 Page No. 7 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS - REVISED

Date 9/9/82 CONFIDENTIAL Signed D. Fuhrmann RF

NOTE #1 (MILK)

Weigh 25 gm. of milk sample in 125 ml. Erlenmeyer flask. Add 50 ml. 5% acetone in acetonitrile and shake the mixture vigorously for 2 min. Decant the organic phase through Whatman #1 filter paper into a 250 ml. round bottom flask. Repeat three additional extractions with 50 ml. 5% acetone in acetonitrile each time shaking for 2 min.and filtering through the same Whatman #1 filter paper. Strip about 25 ml. of the organic phase to remove most of the acetone on flash-evaporator transfer quantitatively to 2 liter separatory funnel and proceed with acetonitrile petroleum ether partitioning as described on page 3.

NOTE #2 (SUGAR)

Dissolve 25 gm. of sugar in 500 ml. distilled water in 2 l. separatory funnel and proceed with acetonitrile petroleum ether partitioning as described on page 3.

PENICK
CORPORATION

1050 Wall Street West, Lyndhurst, New Jersey 07071 • Telephone: (201) 935-6600 • Telex: 133525

December 21, 1982

Mr. Franklin D. R. Gee(PM#17)
Insecticide and Rodenticide Branch
Registration Division(TS-767)
Environmental Protection Agency
401 M Street S.W.
Washington, D.C. 20460

Subject: Analytical Method
Penick's Procedure No.
1047, Revised

Dear Mr. Gee:

Authorization is hereby given to US EPA for trial of the subject method and for publication in the Pesticide Analytical Method Book.

Please inform Ms. Martha Bradley, Chemical Branch(703-557-7377).

Should you need further assistance, call us.

Sincerely,

Emily F. Hedal

Emily F. Hedal
Supervisor Regulatory Affairs
Pesticides Technical Support Group

EFH:FH

cc: M. deVries
S. Kutch

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Procedure No. 1047 Copy No. 1 Page No. 1 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed [signature] RP

SUMMARY:

A general analytical method for determination of Resmethrin residues in diverse types of food products is described. The procedure has been successfully applied to the following substances; sliced american cheese, sliced ham, cream pie, white flour, polished rice, white bread, popcorn kernels, saltine crackers, sliced roast beef, chicken meat, skin, fat, hearts, liver, gizzard and eggs.

Whole milk and sugar require a different sample extraction, details are in notes 1 and 2. Recoveries of Resmethrin added to these substances at the 1 ppm level range from 85 to 101% with a detection limit of 0.1 ppm.

Sample preparation involves extraction in a blender with acetonitrile, partitioning between acetonitrile and petroleum ether and clean-up on Florisil® and Alumin AR columns.

Two gas chromatographic columns are mandated. The 3% OV-1 column is the primary column, if interfering peaks are encountered chromatography on 3% OV-210 will separate the interferences or vice-versa.

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Procedure No. 1047 Copy No. \ Page No. 2 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382)

RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed L.T. Furmanec RP

Equipment:

Hewlett Packard Gas Chromatograph 5840A
Automatic sampler 7671A Hewlett-Packard
GC Glass Column 6 ft. x 2 mm I.D. 1/4" O.D.
Blender (Waring)
Flash-evaporator (Searle)
Separatory Funnel 2 liter
Round Bottom Flask, 500 ml. 250 ml
Pear-shaped Flask, 50 ml
Clean-up column glass, 450 mm long x 15 mm I.D.
Pipet, 1.0 ml., 5.0 ml., 25.0 ml.
Buchner Funnel 70 mm Dia.
Suction Flask, 1000 ml.
Whatman #1 filter paper 70 mm.

Materials:

Acetonitrile, HPLC grade
Hexane, HPLC grade
Petroleum Ether, Pesticide grade
Methylene Chloride, Pesticide grade
Ether Anhydrous, Reagent A.C.S.
Acetone, HPLC grade
Sodium Sulfate, Anhydrous, Reagent grade
Sodium Chloride, Reagent grade
3% OV-1 on 100-120 Mesh on Gas Chrom Q (Applied Science Lab., Inc
3% OV-210 on 100-120 Mesh on Gas Chrom Q (Applied Science Lab., I
Florasil 60-100 PR Mesh (Floridin Company)
Alumin AR CC-10 100-200 Mesh Activity Grade 4 (Mallinckrodt Chemical Works)
Resmethrin Standard (Penick Corporation)

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Procedure No. 1047 Copy No. \ Page No. 3 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (S2P-1322) RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed [signature]

Procedure:

Weigh accurately 25.0 gm. of a representative food sample into blender (If milk or sugar see notes 1 and 2). Add 100 ml of acetonitrile and activate the blender for 5 minutes. Decant the solvent through Whatman #1 filter paper in a Buchner funnel using gentle suction. Repeat the extraction in the blender two additional times using 100 ml and 50 ml portions of acetonitrile and filter each time through the same Whatman #1 filter paper. Transfer combined extracts to a 2 liter seapratory funnel.

Acetonitrile - Petroleum Ether Partition

Add 100 ml petroleum ether to the acetonitrile filtrate and shake well. Then add 75 ml saturated salt solution, 700 ml of distilled water and shake again. Transfer the petroleum ether layer to 250 ml round bottom flask. Extract two additional times with 100 ml and 50 ml portions of petroleum ether. Each time remove petroleum ether in the same round bottom flask on a flash evaporator under vacuum at 50°C to an oily residue. Quantitatively transfer the oily residue to 25 ml volumetric flask with small portions of hexane and dilute to volume.

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Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed [signature]

Column Clean-up

Florisil[R] Column:

Pack a chromatographic column (450 mm l x 15 mm I.D.) with 10 gm. of Florisil[R] (activated at 600°C). Add a 10 mm layer of anhydrous sodium sulfate, to the top of the column, prewet column with hexane and drain to the top of the bed. Transfer 5.0 ml sample aliquot to the top of the column and drain into the bed of the column.

Elute the column with 100 ml of solvent mixture 50% ether - 50% hexane, and collect the total eluate. Concentrate the eluate on flash-evaporator under vacuum at 50°C to an oily residue.

Alumin AR Column:

Pack a chromatographic column (450 mm l x 15 mm I.D.) with 20 gm of Alumin AR.

Add a 10 mm layer of anhydrous sodium sulfate to the top of the column. Prewet column with hexane and drain to the top of the bed. Transfer quantitatively the residue with three 5 ml portions of hexane to the top of the column and drain each time into the bed of the column. Elute the column with 100 ml hexane and discard the eluate. Continue elution with 100 ml of solvent mixture 25 ml methylene chloride and 75 ml hexane and collect the eluate in 250 ml round bottom flask. Evaporate the eluate on a flash-evaporator under vacuum at 50°C to about 5 ml. Transfer quantitatively the residue with 2 x 5 ml hexane to 50 ml pear-shaped flask and evaporate the hexane on a flash evaporator again.

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Procedure No. 1047 Copy No. 1 Page No. 5 of 7 Pages

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382)

RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed G. Fujimoto RP

Sample Preparation:

Add 1.0 ml hexane to the pear shaped flask and dissolve the residue.

Standard Preparation:

Dissolve 25 mg of standard SBP-1382, accurately weighed in a 250 ml volumetric flask and dilute to volume with hexane. Dilute 5.0 ml aliquot to 100 ml with hexane. Final concentration 0.005 mg/ml

GLC Analysis:

Equipment and Conditions:

Instrument: Hewlett Packard Gas Chromatograph 5840A

Column:
a) 6 ft. x 2 mm I.D. 1/4" O.D. glass packed with 3% OV-1 on 100-120 mesh Gas Chrom Q
b) 6 ft. x 2 mm. I.D. 1/4" O.D. glass packed with 3% OV-210 on 100-120 Mesh Gas Chrom Q

Temperature:
Injector 250°C
Detector (F.I.D.) 250°C

Column Oven Temperature:

Primary Column: 3% OV-1 180-250°C at the rate of 5°C per minute and held at 250°C for 15 min.

Alternate Column: 3% OV-210 100-250°C at the rate of 10°C per minute and held at 250°C for 15 min.

Carrier Gas: Helium at 30 ml per min.
Hydrogen pressure - 20 p.s.i.g.
Air pressure - 40 p.s.i.g.

Attenuation: 2 (3)

EA

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382)

RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed R. F. Fleissner RP

Determination:

Conditon the column before use by injecting the standard several times. Inject the sample and check for interfering peaks. If interfering peaks are encountered use the alternate column (3% OV-210). Inject 5.0 ul of the standard and sample preparation until sufficient chromatograms of each are obtained.

Calculation:

Calculation are based on equal volume injection using peak areas. From the chromatograms of the standard and sample preparation calculat ppm of SBP-1382 in the sample by the following formula:

$$\frac{A_{(s)}}{A_{(std)}} \times \frac{W_{(std)}}{W_{(s)}} \times 10^6 = \text{ppm of SBP-1382}$$

Where:

$A_{(s)}$ = peak area of SBP-1382 in the sample

$A_{(std)}$ = peak area of SBP-1382 in the standard

$W_{(s)}$ = weight of the sample in mgs

$W_{(std)}$ = weight of standard in mgs/ml in final dilution

Title GLC PROCEDURE FOR THE DETERMINATION OF RESMETHRIN (SBP-1382) RESIDUES IN FOOD PRODUCTS

Date 11/14/80 Signed R. F. Furmanec RP

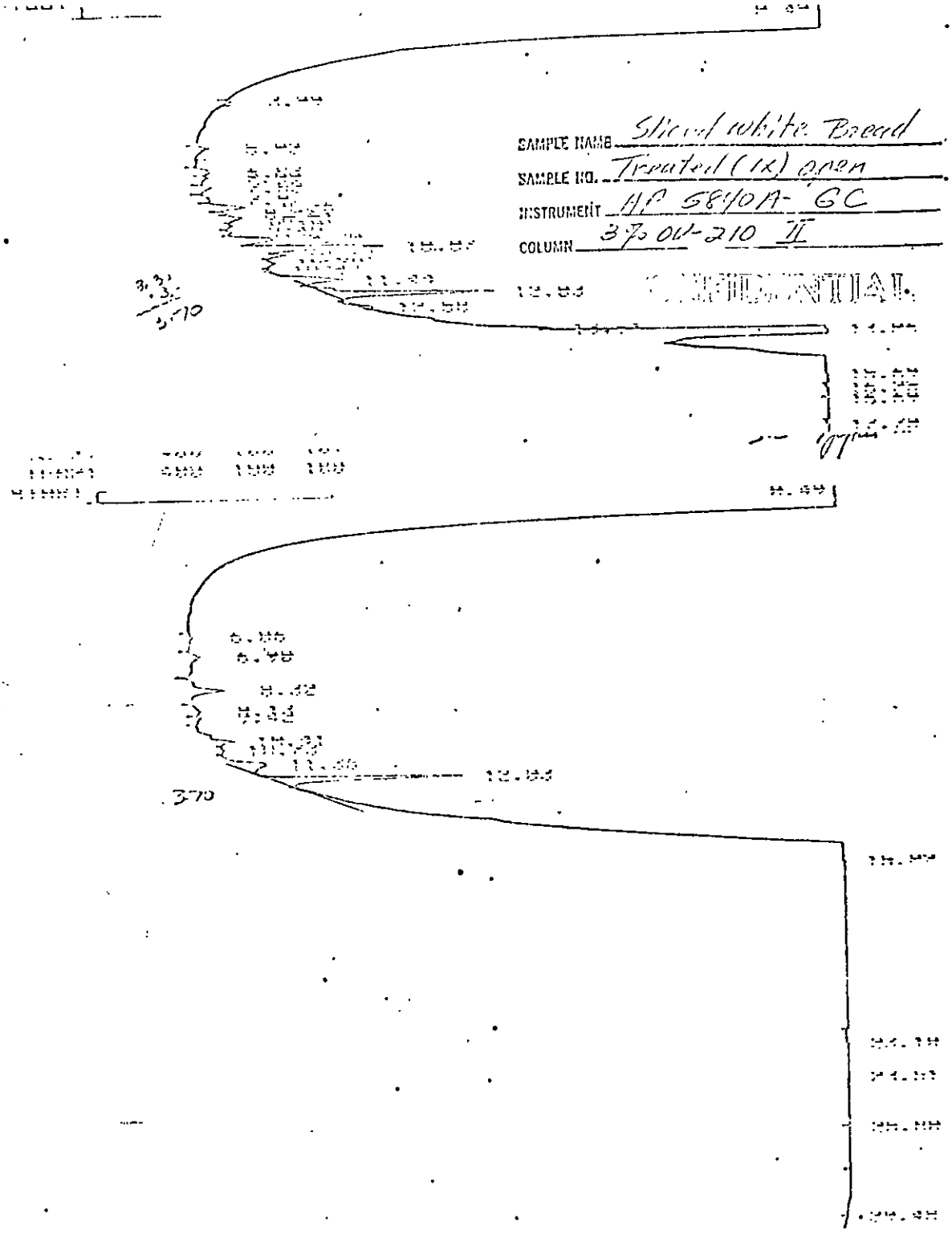
NOTE #1 (MILK)

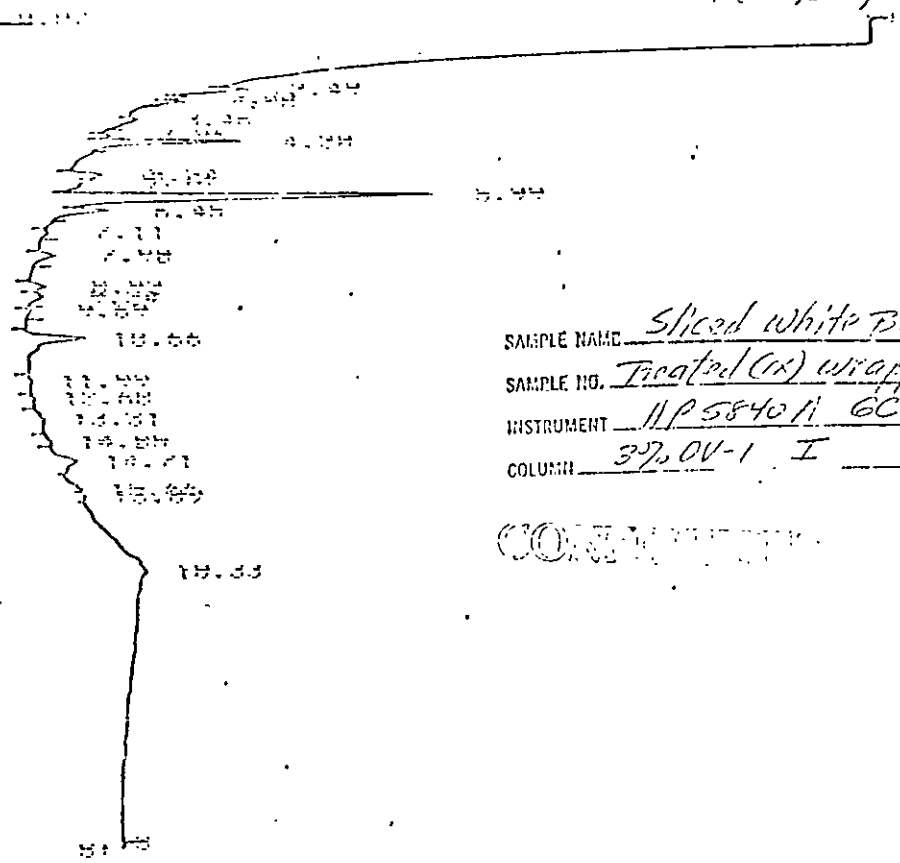
Weigh 25 gm of milk sample in 125 ml Erlenmeyer flask. Add 50 ml 5% acetone in acetonitrile and shake the mixture vigorously for 2 min. Decant the organic phase through Whatman #1 filter paper into a 250 ml round bottom flask. Repeat three additional extractions with 50 ml 5% acetone in acetonitrile each time shaking for 2 min. and filtering through the same Whatman #1 filter paper. Strip about 25 ml of the organic phase to remove most of the acetone on flash-evaporator, transfer quantitatively to 2 liter separatory funnel and procede with acetonitrile petroleum ether partitioning as described on page 3.

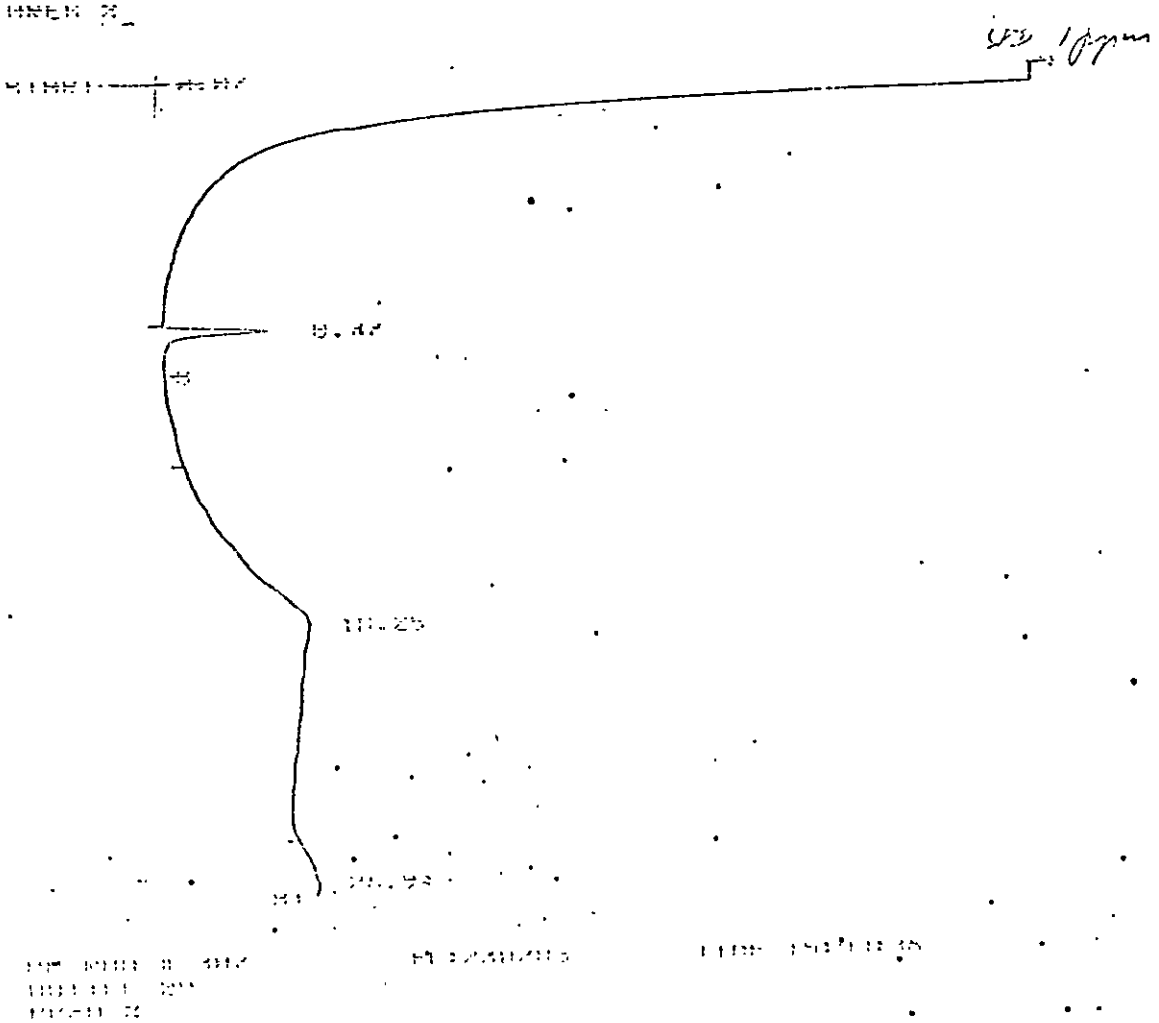
NOTE #2 (SUGAR)

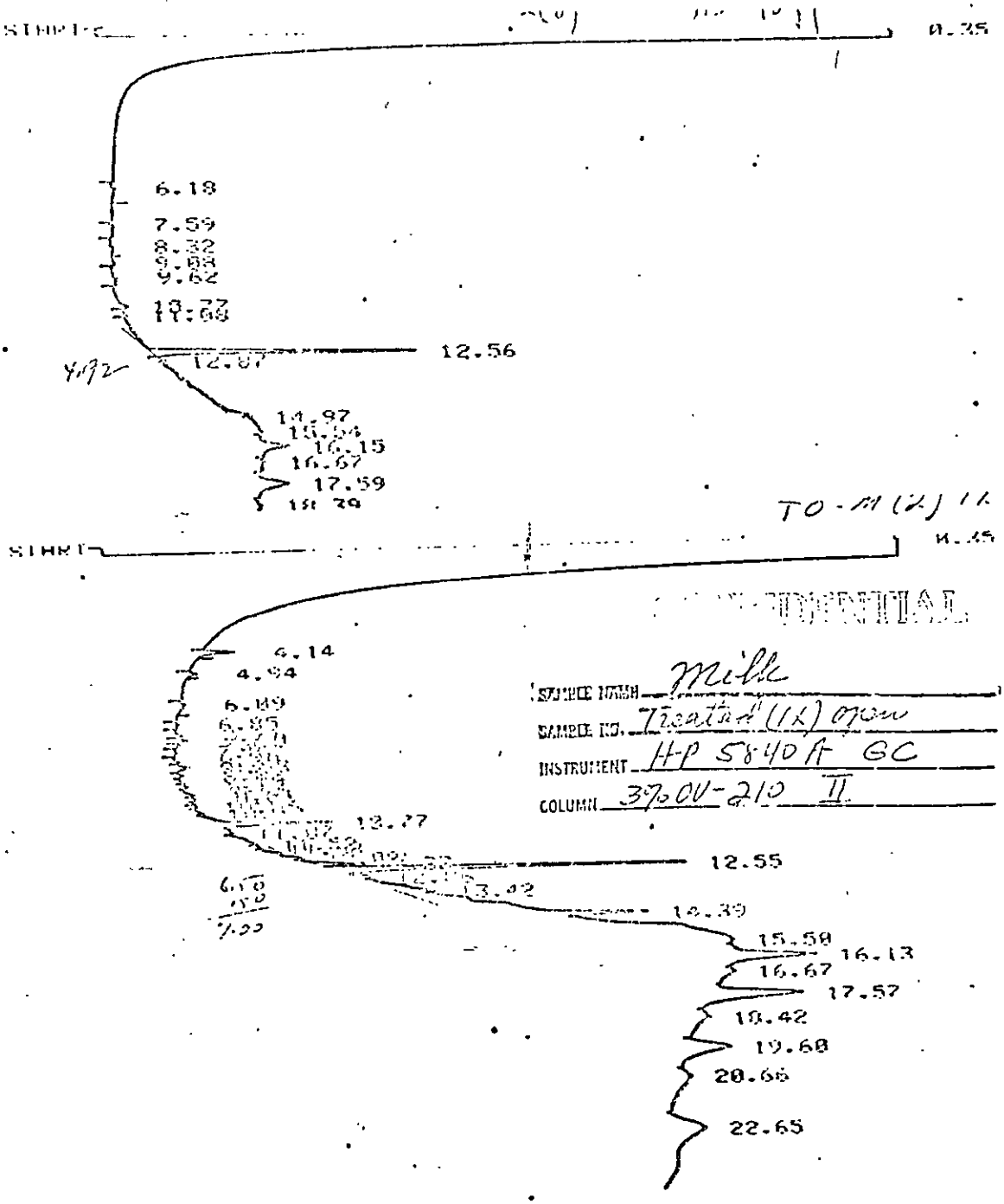
Dissolve 25 gm of sugar in 500 ml distilled water in 2 l. separatory funnel and procede with acetonitrile petroleum ether partitioning as described on page 3.

TO-B(1)K


SAMPLE NAME Sliced white Bread
SAMPLE NO. Treated (1x) open
INSTRUMENT HP 5890A- GC
COLUMN 30 OV-210 II
CONFIDENTIAL
3.33
5.10
370


SAMPLE NAME Sliced White Bread
SAMPLE NO. Treated (1X) wrapped
INSTRUMENT HP 5840A GC
COLUMN 3% OV-1 I





TO-11 (2) 11

CONFIDENTIAL

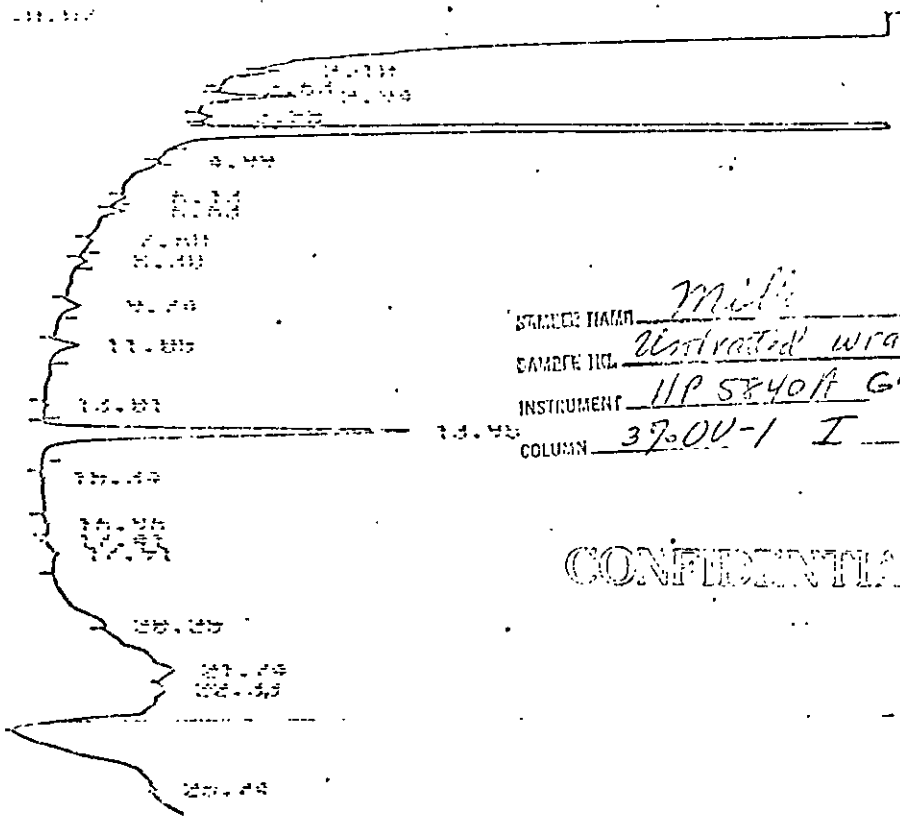
SAMPLE NO. Treated (12) open

INSTRUMENT HP 5840A GC

COLUMN 3% OV-210 II

S HP RUN # 4
SHTTLE 24
AREA %

RT	AREA	AREA %
2.35	8306	99.692
4.14	22	0.000
4.94	5	0.000
6.89	2	0.000
6.85	1	0.000
7.31	6	0.000
7.50	3	0.000
7.66	4	0.000
7.87	1	0.000
8.30	3	0.000
8.54	11	0.000
8.82	3	0.000
9.10	2	0.000
9.27	1	0.000
9.61	2	0.000
9.79	1	0.000
9.92	1	0.000
11.06	1	0.000
11.42	1	0.000



SAMPLE NAME Milk
SAMPLE NO. Undiluted wrapped
INSTRUMENT HP 5840A GC
COLUMN 37.0V-1 I

CONFIDENTIAL

[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]

SD Upm