Report No.: 66439

MOBAY CHEMICAL CORPORATION
AGRICULTURAL DIVISION

Research and Development Department

TITLE: A Method for the Determination of ®GUTHION and GUTHION Oxygen Analog in Bovine Tissues and Milk Utilizing Gas Chromatography and High Pressure Liquid Chromatography

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ABSTRACT: A method is described for the analysis of GUTHION and GUTHION oxygen analog in bovine tissues and milk. The method for tissues and milk involves an initial extraction using acetone and dichloromethane (DCM) followed by an acetonitrile:hexane partition clean-up. The initial extraction for fat utilized hexane and acetonitrile followed by a partition clean-up. An additional solvent partition clean-up followed by a silica gel column clean-up and another acetonitrile:hexane partition clean-up was performed prior to gas liquid chromatographic analysis for GUTHION and high pressure liquid chromatographic analysis for GUTHION oxygen analog. The sensitivity of the method is 0.01 ppm for tissues and 0.001 ppm for milk.

DATE: April 10, 1978

NOTEBOOK REFERENCE: 78-R-183

APPROVED BY T. Bill Waggoner

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GUTHION is a Reg. TM of the Parent Company of Farbenfabriken Bayer GmbH, Leverkusen.

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RESEARCH REPORT

To: Chemagro Agricultural Division
Mobay Chemical Corporation
P. O. Box 4913 - Hawthorn Road
Kansas City, Missouri 64120

ADC Project: #378

Date: April 10, 1978

ANALYTICAL METHOD FOR THE DETERMINATION OF RESIDUES OF GUTHION AND GUTHION OXYGEN ANALOG IN BOVINE TISSUES (LIVER, MUSCLE, FAT, KIDNEY) AND MILK

I. INTRODUCTION

This report contains an analytical method to determine residues of Guthion and Guthion oxygen analog in bovine tissues and milk. The sensitivity of detection is 0.01 ppm in tissue and 0.001 ppm in milk. The parent Guthion is quantitated on a gas chromatograph equipped with a flame photometric detector, while the oxygen analog is quantitated on an HPLC at a wavelength of 280 nm.

Recovery data and chromatograms obtained from the validation analyses of each tissue and milk are contained in this report.* The data were obtained by analyzing the samples according to the method contained in this report.

II. MATERIALS AND METHOD

A. Apparatus Required

1. Assorted laboratory glassware.
2. Waring (explosion proof) Laboratory Blendor.
3. Gas Chromatograph, Tracor 222, equipped with a flame photometric detector in the phosphorous mode.
4. High Pressure Liquid Chromatograph.
5. Rotary vacuum evaporator.

B. Reagents Required

1. Acetone - Nanograde® - Registered trademark of Mallinckrodt Chemical Works, St. Louis, Missouri.
2. Acetonitrile - Nanograde®.
3. Benzene - Nanograde®.

*See Chemagro Report No. 66440.

II. MATERIALS AND METHOD

B. Reagents Required (cont.)

4. Dichloromethane - Nanograde®.
5. Ethyl Acetate - Nanograde®.
6. Filter paper, Whatman No. 31.
7. Filter paper, Whatman 2V fluted.
8. Glass wool.
9. Hexane - Nanograde®.
10. Methanol - Nanograde®.
11. Silica Gel - 10% water; heat the silica gel (80/100 mesh) 24 hours at 120°C; deactivate 10% with water (90g silica gel and 10 ml distilled water); mix well and allow the deactivated silica gel to stand 24 hours before use.
12. Sodium Chloride (10% w/v in distilled water).
13. Sodium Sulfate, - granular anhydrous - benzene wash before use.
14. Super Cel, Hyflow.

C. Special Reagents

1. PS Guthion Spiking Solutions

a. Solution "A" - Dissolve 100.0 mg of recrystallized PS Guthion in benzene and dilute to volume in a 50 ml volumetric flask. This solution contains 2 mg PS Guthion per ml.

b. Solution "B" - Pipet 5.0 ml of Solution "A" into a 250 ml volumetric flask and dilute to volume with benzene. This solution contains 40 µg of PS Guthion per ml.

c. Solution "C" - Pipet 1.0 ml of Solution "A" into a 200 ml volumetric flask and dilute to volume with benzene. This solution contains 10 µg of PS Guthion per ml.

II. MATERIALS AND METHOD

C. Special Reagents

1. PS Guthion Spiking Solutions (cont.)

d. Solution "D" - Pipet 5.0 ml of Solution "B" into a 100 ml volumetric flask and dilute to volume with benzene. This solution contains 2 µg of PS Guthion per ml.

2. PO Guthion Spiking Solutions

a. Solution "A" - Dissolve 95.0 mg of recrystallized PO Guthion in benzene and dilute to volume in a 50 ml volumetric flask. This solution contains 2 mg PO Guthion equivalents per ml.

b. Solution "B" - Pipet 5.0 ml of Solution "A" into a 250 ml volumetric flask and dilute to volume with benzene. This solution contains 40 µg of PO Guthion per ml.

c. Solution "C" - Pipet 1.0 ml of Solution "A" into a 200 ml volumetric flask and dilute to volume with benzene. This solution contains 10 µg of PO Guthion per ml.

d. Solution "D" - Pipet 5.0 ml of Solution "B" into a 100 ml volumetric flask and dilute to volume with benzene. This solution contains 2 µg of PO Guthion per ml.

3. PS Guthion Standard Solutions (For GC Analysis)

a. Solution #1 - Dissolve 10.0 mg of recrystallized Guthion in benzene and dilute to volume in a 500 ml volumetric flask. This solution contains 20 µg of PS Guthion per ml. Store in a freezer.

b. Solution #2 - Pipet 5.0 ml of Solution #1 into a 50 ml volumetric flask and dilute to volume with ethyl acetate. This solution contains 2.0 µg of PS Guthion per ml. (The stability of Guthion of ethyl acetate for long duration is not known.)

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II. MATERIALS AND METHOD

C. Special Reagents (cont.)

4. PO Guthion (Equivalent) Standard Solution (For LC Analysis)

a. Solution #1 - Dissolve 9.50 mg of recrystallized PO Guthion in 50% acetonitrile/dichloromethane and dilute to volume in a 500 ml volumetric flask. This solution contains 20 µg PO Guthion equivalents per ml. Store in a freezer.

b. Solution #2 - Pipet 5.0 ml of Solution #1 into a 50 ml volumetric flask and dilute to volume with 50% acetonitrile/dichloromethane. This solution contains 2.0 µg of PO Guthion per ml.

D. Detailed Procedure

1. Initial Extraction (except for fat; see Procedure Notes, pages 9 & 10)

a. Weigh 200 grams of milk or 100 grams of tissue into a blender jar and add 5 grams Hyflow Super Cel (10 grams for milk).

b. Add 200 ml of acetone and blend 2 minutes (400 ml for milk).

c. Filter under vacuum through Whatman No. 31 filter paper.

d. Return the filter cake and paper to the blender jar, add 200 ml of dichloromethane (DCM) and blend for 5 minutes.

e. Filter under vacuum through Whatman No. 31 filter paper and wash the filter cake with 100 ml DCM.

f. Transfer the combined filtrates to a 1,000 ml separatory funnel. Rinse the suction flask with 100 ml DCM and add to the combined filtrates.

g. Shake the separatory funnel vigorously for 30 seconds. Allow the layers to separate and drain lower organic phase through Whatman 2V fluted filter paper into a 1,000 ml flat bottom (FB) flask.

II. MATERIALS AND METHOD

D. Detailed Procedure

1. Initial Extraction (cont.)

h. Evaporate the extract just to dryness (oil).

2. Acetonitrile Hexane Partition

a. Dissolve the residue in 200 ml of hexane presaturated with acetonitrile (ACN), and transfer to a 500 ml separatory funnel.

b. Extract the hexane solution with 100 ml of ACN presaturated with hexane.

c. Drain lower ACN phase into a second 500 ml separatory funnel containing 200 ml hexane presaturated with ACN. Shake for one minute.

d. Draw off the lower ACN phase from the second separatory funnel into a 500 ml ~~RB~~ RB flask.

e. Repeat Steps b through d twice, with 50 ml portions of ACN presaturated with hexane.

f. Evaporate the combined ACN fractions just to dryness.

3. Dichloromethane/Aqueous Partition

a. Dissolve the residue in 25 ml methanol and add 100 ml of 10% NaCl in distilled water. Transfer to a 250 ml separatory funnel.

b. Extract with 3 x 50 ml portions of DCM.

c. Drain lower layers into a 250 ml ~~RB~~ RB flask.

d. Evaporate the combined DCM fractions just to dryness.

e. Dissolve residue in 10 ml benzene.

II. MATERIALS AND METHOD

D. Detailed Procedure (cont.)

4. Chromatographic Column Cleanup

a. Tamp a plug of glass wool into the bottom of a 46 cm x 2 cm bell column having a 300 ml reservoir.

b. Fill the column to the bell with benzene and begin a slow drip. Slowly pour 15 grams of 10% deactivated silica gel into the benzene. Add 10 grams of Na_2SO_4 to the top of the silica gel and allow the benzene to drain to the top of the Na_2SO_4.

c. Transfer the residue dissolved in benzene from above (II.D.3.e.) to the column. Start column drip rate at one per second into a beaker.

d. Just as the top of the solvent reaches the silica gel bed, add ~~a 15~~ ml benzene rinse of sample flask to column.

e. Just as the top of the solvent reaches the silica gel bed, add 25 ml benzene.

f. Just as the top of the solvent reaches the silica gel bed, change receivers to a 250 ml FB flask and add 150 ml DCM and collect. This fraction contains PS Guthion. (Label the flasks "A" Fraction.)

g. Evaporate the DCM just to dryness and proceed with the ACN Hexane Partition #2 in Section D.5. below.

h. Change receivers to a second 500 ml FB flask and add 250 ml 25% ACN in DCM and collect. This fraction contains the oxygen analog, PO Guthion. (Label the flasks "B" Fraction.)

i. Evaporate the ACN/DCM just to dryness and proceed with ACN Hexane Partition #2 in Section D.5. below.

II. MATERIALS AND METHOD

D. Detailed Procedure (cont.)

5. Acetonitrile Hexane Partition #2

a. Dissolve the residue in 100 ml of hexane presaturated with ACN and transfer to a 250 ml separatory funnel.

b. Extract the hexane solution with 50 ml of ACN presaturated with hexane.

c. Drain lower ACN phase into a second 250 ml separatory funnel containing 100 ml hexane presaturated with ACN. Shake.

d. Draw off the lower ACN phase from the second separatory funnel into a 250 ml FB flask.

e. Repeat Steps b through d twice, with 25 ml portions of ACN presaturated with hexane.

f. Evaporate the combined ACN fractions just to dryness.

g. Proceed to Section D.6. with the "A" Fractions (PS Guthion Fraction).

h. Proceed to Section D.7. with the "B" Fractions (PO Guthion Fraction).

6. Gas Liquid Chromatography

Column: 24" x 2 mm i.d. borosilicate glass column packed with 5% OV-210 on 80/100 mesh Chromosorb W (HP).

Column Conditioning: Attach column to injection port fitting. Purge the column for 15 minutes, 40 cc/min helium. Cap end of column and heat to 250°C for 1 hour. Cool, remove cap, purge 2 hours, 40 cc/min helium at 240°C. Attach column to detector and proceed with analysis.

II. MATERIALS AND METHOD

D. Detailed Procedure

6. Gas Liquid Chromatography (cont.)

Gases: Rotometer Settings

N_2 = 4.0
O_2 = 12.0
Air = 100
H_2 = 150

Temperature °C: Column: 225°C
Injection Port: 250°C

Sensitivity: Instrumental response for the standard (2 ng/µl equivalent to 0.1 ppm in tissues or 0.01 ppm in milk) must be sufficient to obtain greater than 50% full scale response.

a. Dissolve the sample from Section D.5.g. above in ethyl acetate, 5 ml volume for tissue samples and 1 ml volume for milk. [If the sample contains a sizeable residue (volume), transfer quantitatively to a graduated tube and dilute to volume]. The sample or standard are generally injected in a volume of 5 µl. A greater volume may be used to obtain the desired sensitivity.

b. Identify Guthion by its retention time and measure the peak produced.

Calculations:

$$ppm = \frac{\text{sample peak}}{\text{standard peak}} \times \text{Std}^1 \times \text{sample dilution}$$

[1]Use 0.1 for tissues and 0.01 for milk calculations.

7. High Pressure Liquid Chromatography

Column: Partisil PXS 10/25 Silica Gel Whatman, 25 cm x 4.6 mm i.d.

Mobile Phase: 50% ACN/50% DCM

II. MATERIALS AND METHOD

D. Detailed Procedure

7. High Pressure Liquid Chromatography (cont.)

Pressure: approximately 500 psi

Sensitivity Setting: 0.50 AUFS for milk
0.25 AUFS for tissue

Wavelength: 280 nm

Pre-Column: HC Pellosil, 6 cm x 4 mm i.d.

Instrument: Glenco HPLC System I, Glenco Scientific, Inc., Houston, Texas

a. Dissolve the sample from Section D.5.h. above in 50% ACN/50% DCM, 5 ml volume for tissue samples and 1 ml volume for milk. Filter through a 0.5 micron Milli-Pore filter using a syringe and Swinny adaptor. Inject 100 µl samples interspersed with 100 µl samples of a 2 ng/µl standard.

b. Identify PO Guthion (oxygen analog) by its retention time and measure the peak produced.

Calculations:

$$ppm = \frac{\text{sample peak}}{\text{standard peak}} \times \text{Std}^1 \times \text{sample dilution}$$

[1]Use 0.1 for tissues and 0.01 for milk calculations.

8. Procedure Notes

a. Initial Extraction for Fat (Only)

(1) Weigh 100 grams of fat into a blender jar and add 10 grams of Hyflow Super Cel.

(2) Add 250 ml hexane and blend for 2 minutes.

(3) Filter under vacuum through Whatman No. 31 filter paper.

II. MATERIALS AND METHOD

D. Detailed Procedure

8. Procedure Notes

a. Initial Extraction for Fat (Only) (cont.)

(4) Return the filter cake to the blender jar, add 250 ml hexane and blend for 5 mintues.

(5) Filter under vacuum through Whatman No. 31 filter paper and wash the filter cake with 150 ml of acetonitrile.

(6) Transfer the combined filtrates to a 1,000 ml separatory funnel. Rinse the suction flask with 50 ml acetonitrile and add to the combined filtrate.

(7) Shake the separatory funnel vigorously for one minute and allow the layers to separate.

(8) Proceed with Section D.2.c. and follow the procedure to the end (D.7.b.).

9. Recoveries Through Method

The following tables (I through V) (Appendix I) and enclosed chromatograms (Appendix II)* show recovery values and chromatograms obtained for the analysis of each matrix for PS Guthion and PO Guthion. Typical control, recovery and standard chromatograms are included in this report.

ANALYTICAL DEVELOPMENT CORPORATION

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DVG:as

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A P P E N D I X I

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TABLE I

MUSCLE TISSUE (BOVINE) RECOVERIES

A. PS Guthion - Analyzed by GC/FPD

Sample No.	Sample Fortification	ppm Found	Corrected ppm	% Recovery
1a	Muscle Control (100 grams)	0.0101)		
2a	Muscle Control	0.0057)	0.0079 (Avg)	
3a	Muscle Control + 0.05 ppm PS Guthion	0.0344	0.0265	53.0
4a	Muscle Control + 0.05 ppm PS Guthion	0.0476	0.0397	79.4
5a	Muscle Control + 0.10 ppm PS Guthion	0.1117	0.1038	103.8
6a	Muscle Control + 0.10 ppm PS Guthion	0.0987	0.0908	90.8

B. PO Guthion - Analyzed by HPLC

Sample No.	Sample Fortification	ppm Found	Corrected ppm	% Recovery
1b	Muscle Control (100 grams)	0.0000	0.0000	---
2b	Muscle Control	0.0000	0.0000	---
3b	Muscle Control + 0.05 ppm PO Guthion	0.0422	0.0422	84.4
4b	Muscle Control + 0.05 ppm PO Guthion	0.0430	0.0430	86.0
5b	Muscle Control + 0.10 ppm PO Guthion	0.0734	0.0734	73.4
6b	Muscle Control + 0.10 ppm PO Guthion	0.0796	0.0796	79.6

REFERENCE: NBR #A-31

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TABLE II

FAT TISSUE (BOVINE) RECOVERIES

A. PS Guthion - Analyzed by GC/FPD

Sample No.	Sample Fortification	ppm Found	% Recovery
1a	Fat Control (100 grams)	0.0000	---
2a	Fat Control	0.0000	---
3a	Fat Control + 0.05 ppm PS Guthion	0.0457	87.4
4a	Fat Control + 0.05 ppm PS Guthion	0.0459	91.8
5a	Fat Control + 0.1 ppm PS Guthion	0.0974	97.4
6a	Fat Control + 0.1 ppm PS Guthion	0.0980	98.0

B. PO Guthion - Analyzed by HPLC

Sample No.	Sample Fortification	ppm Found	% Recovery
1b	Fat Control (100 grams)	0.0000	---
2b	Fat Control	0.0000	---
3b	Fat Control + 0.05 ppm PO Guthion	0.0474	94.8
4b	Fat Control + 0.05 ppm PO Guthion	0.0520	104.0
5b	Fat Control + 0.1 ppm PO Guthion	0.0820	82.0
6b	Fat Control + 0.1 ppm PO Guthion	0.0890	89.0

REFERENCE: NBR #A-31

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TABLE III

MILK (BOVINE) RECOVERIES

A. PS Guthion - Analyzed by GC/FPD

Sample No.	Sample Fortification	ppm Found	% Recovery
1a	Milk Control (200 grams)	0.0000	---
2a	Milk Control	0.0000	---
3a	Milk Control + 0.005 ppm PS Guthion	0.0039	78.0
4a	Milk Control + 0.005 ppm PS Guthion	0.0041	82.0
5a	Milk Control + 0.01 ppm PS Guthion	0.0083	83.0
6a	Milk Control + 0.01 ppm PS Guthion	0.0074	74.0
7a	Milk Control + 0.02 ppm PS Guthion	0.0189	95.0
8a	Milk Control + 0.02 ppm PS Guthion	0.0162	81.0

B. PO Guthion - Analyzed by HPLC

Sample No.	Sample Fortification	ppm Found	Corrected ppm	% Recovery
1b	Milk Control (200 grams)	0.0024)	0.0022 (Avg)	
2b	Milk Control	0.0020)		
3b	Milk Control + 0.005 ppm PO Guthion	0.0053	0.0031	62.0
4b	Milk Control + 0.005 ppm PO Guthion	0.0057	0.0035	70.0
5b	Milk Control + 0.01 ppm PO Guthion	0.0100	0.0078	78.0
6b	Milk Control + 0.01 ppm PO Guthion	0.0106	0.0084	84.0
7b	Milk Control + 0.02 ppm PO Guthion	0.0192	0.0170	85.0
8b	Milk Control + 0.02 ppm PO Guthion	0.0182	0.0160	80.0

REFERENCE: NBR #A-31

TABLE IV

KIDNEY TISSUE (BOVINE) RECOVERIES

A. PS Guthion - Analyzed by GC/FPD

Sample No.	Sample Fortification	ppm Found	% Recovery
1a	Kidney Control (100 grams)	0.0000	---
2a	Kidney Control	0.0000	---
3a	Kidney Control + 0.05 ppm PS Guthion	0.0557	111.4
4a	Kidney Control + 0.05 ppm PS Guthion	0.0559	111.8
5a	Kidney Control + 0.1 ppm PS Guthion	0.1049	104.9
6a	Kidney Control + 0.1 ppm PS Guthion	0.0980	98.0

B. PO Guthion - Analyzed by HPLC

Sample No.	Sample Fortification	ppm Found	% Recovery
1b	Kidney Control (100 grams)	0.0000	---
2b	Kidney Control	0.0000	---
3b	Kidney Control + 0.05 ppm PO Guthion	0.0411	82.2
4b	Kidney Control + 0.05 ppm PO Guthion	0.0453	90.6
5b	Kidney Control + 0.1 ppm PO Guthion	0.0818	81.8
6b	Kidney Control + 0.1 ppm PO Guthion	0.0901	90.1

REFERENCE: NBR #A-31

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TABLE V

LIVER TISSUE (BOVINE) RECOVERIES

A. PS Guthion - Analyzed by GC/FPD

Sample No.	Sample Fortification	ppm Found	% Recovery
1a	Liver Control (100 grams)	0.0000	---
2a	Liver Control	0.0000	---
3a	Liver Control + 0.05 ppm PS Guthion	0.0421	84.2
4a	Liver Control + 0.05 ppm PS Guthion	0.0447	89.2
5a	Liver Control + 0.1 ppm PS Guthion	0.0944	94.4
6a	Liver Control + 0.1 ppm PS Guthion	0.0806	80.6

B. PO Guthion - Analyzed by HPLC

Sample No.	Sample Fortification	ppm Found	% Recovery
1b	Liver Control (100 grams)	0.0000	---
2b	Liver Control	0.0000	---
3b	Liver Control + 0.05 ppm PO Guthion	0.0450	90.0
4b	Liver Control + 0.05 ppm PO Guthion	0.0449	89.8
5b	Liver Control + 0.1 ppm PO Guthion	0.0844	84.4
6b	Liver Control + 0.1 ppm PO Guthion	0.0866	86.6

REFERENCE: NBR #A-31

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Sample # No _____ Date 3-24-98

PS GUTHION LIVER

CONTROL

Run A-31 Inj. No. #19

PEAK HEIGHT IN mm (Sample/Std.)

0.0/197.0

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Sample & No. Date 3-24-78

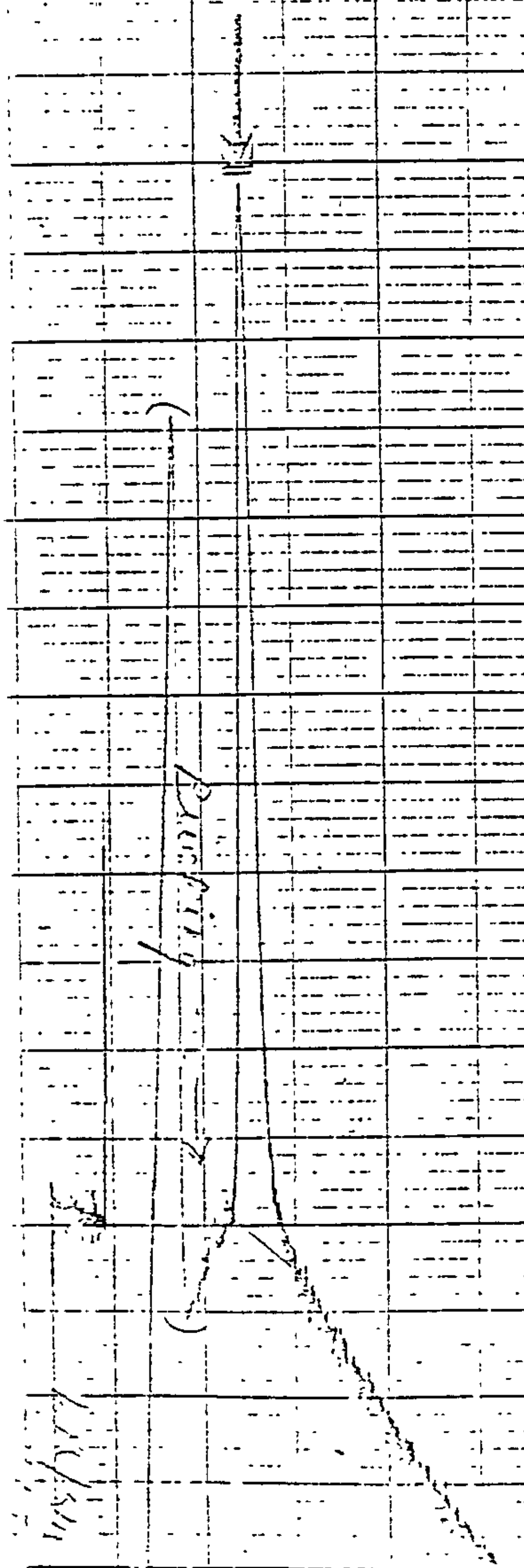
PS Guthion - Liver

Fortified At 0.1ppm PS

A-31 No. 11 #19

Peak Height 14 mm ( )- Sample/Std.

150.8/187.0



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0.0806 ppm

80.6% Rec.

150.8mm

20

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Sample & No. Date 4-4-78

PO GUTHION - LIVER

CONTROL

NBR A-3.1 C~~ON~~ #23

PEAK HEIGHT IN MM Sample/Std.

0.0/169.0

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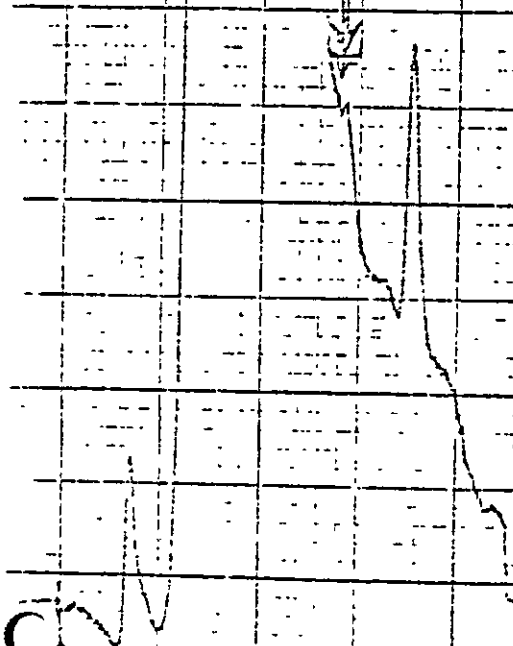

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Date 4-4-78

PO EMULSION: LIVER FORTIFIED

AT 0.1 gm PO

Number A-31 PO #23

PEAK HEIGHT IN mm

136.0/157.0

131

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Sample & No. ________ Date 3-24-78

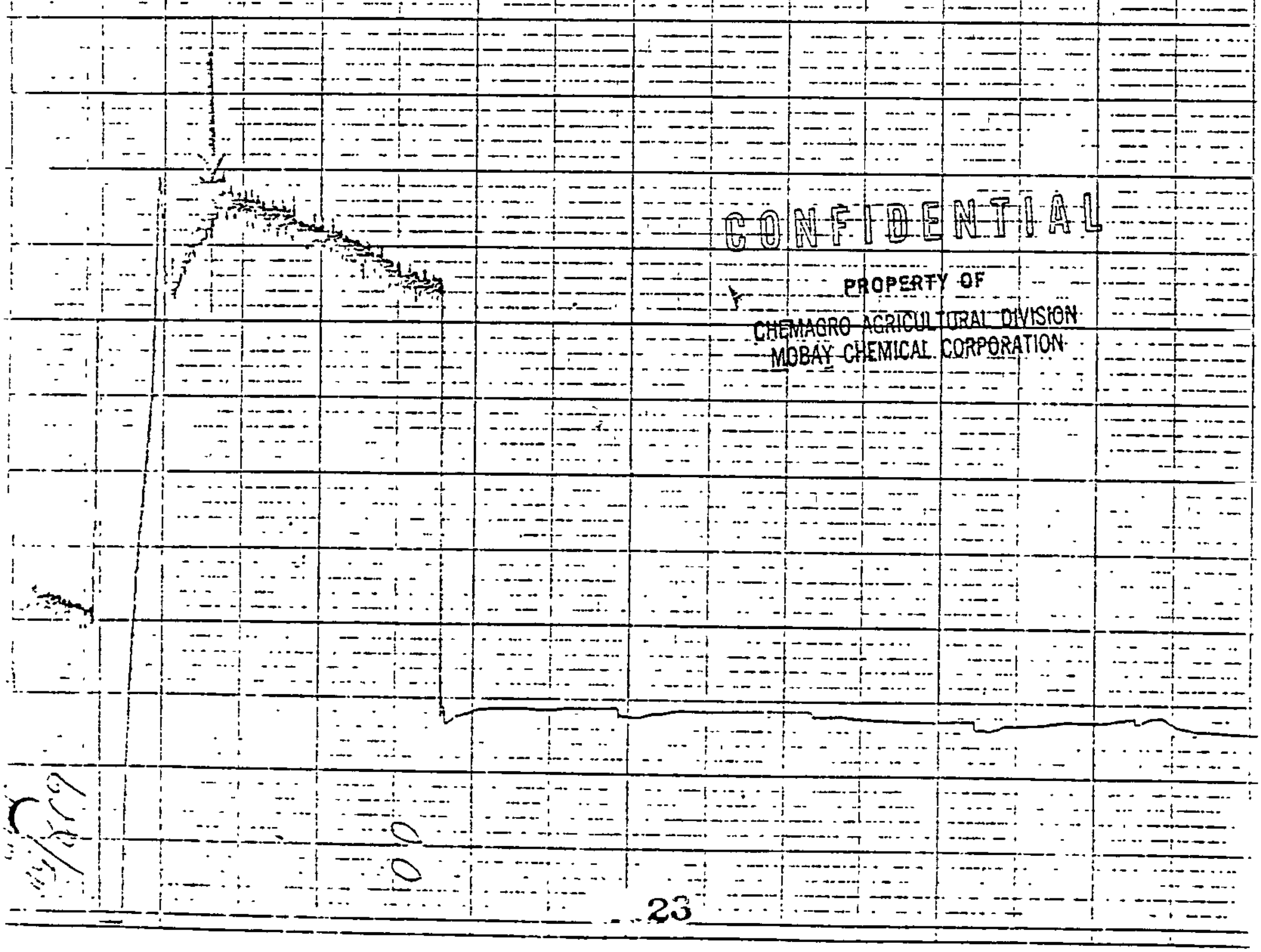
P.S. GUTHION KIDNEY

CONTROL

MBR A-31 cP9 #18

PEAK HEIGHT IN MM in Sample/Std.

0.0/152.3



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0.0

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Sample & No. PS GUTHION FORTIFIED AT 0.1ppm PS
Date 3-24-78 KIDNEY
pg #18
NBR A31
PEAK HEIGHT IN MM Sample/Std
155.2 / 148.0

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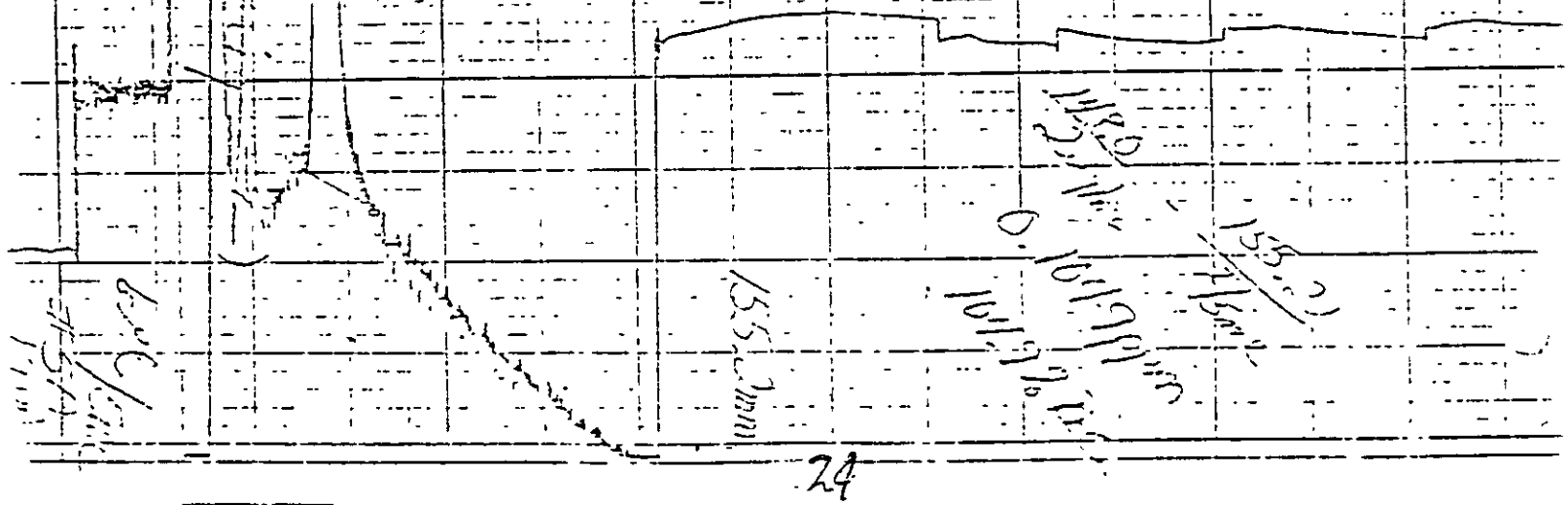

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3-22-78

PO GOMCO - KIDNEY

CONTROL

A-31 PP #18

PEAK HEIGHT

0.0 / 155.0

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Sample # No. Date 3-22-78

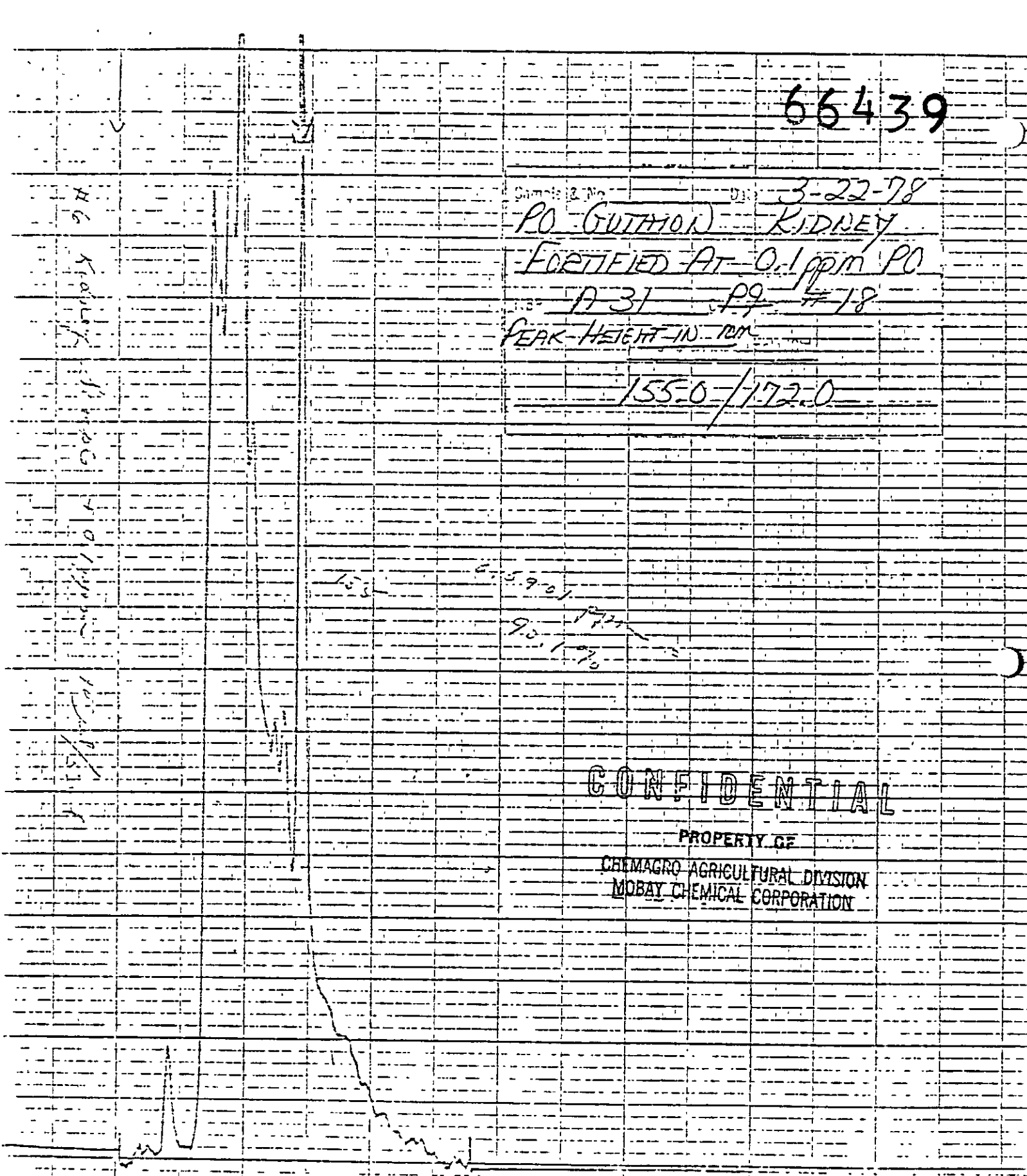
PO (GUTHION) KIDNEY

FORTIFIED AT 0.1 ppm PO

A 31 P9 # 18

PEAK HEIGHT IN mm

155.0 / 172.0



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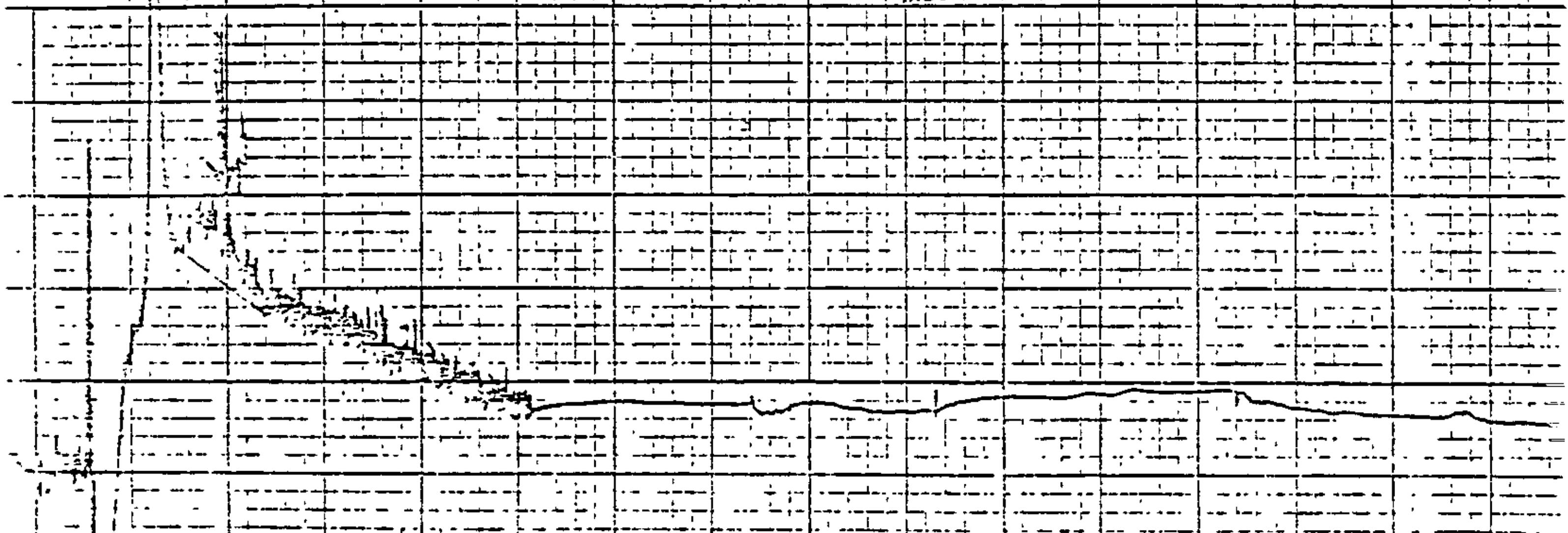
Sample: PS Guthion Dig. Muscle
Control

Date: 3-7-78

NSR: A-31

Peak Height in mm

9.0 / 158.0



152.0 / 2 = 2.0 / 2/3 = 0.0054 ppm

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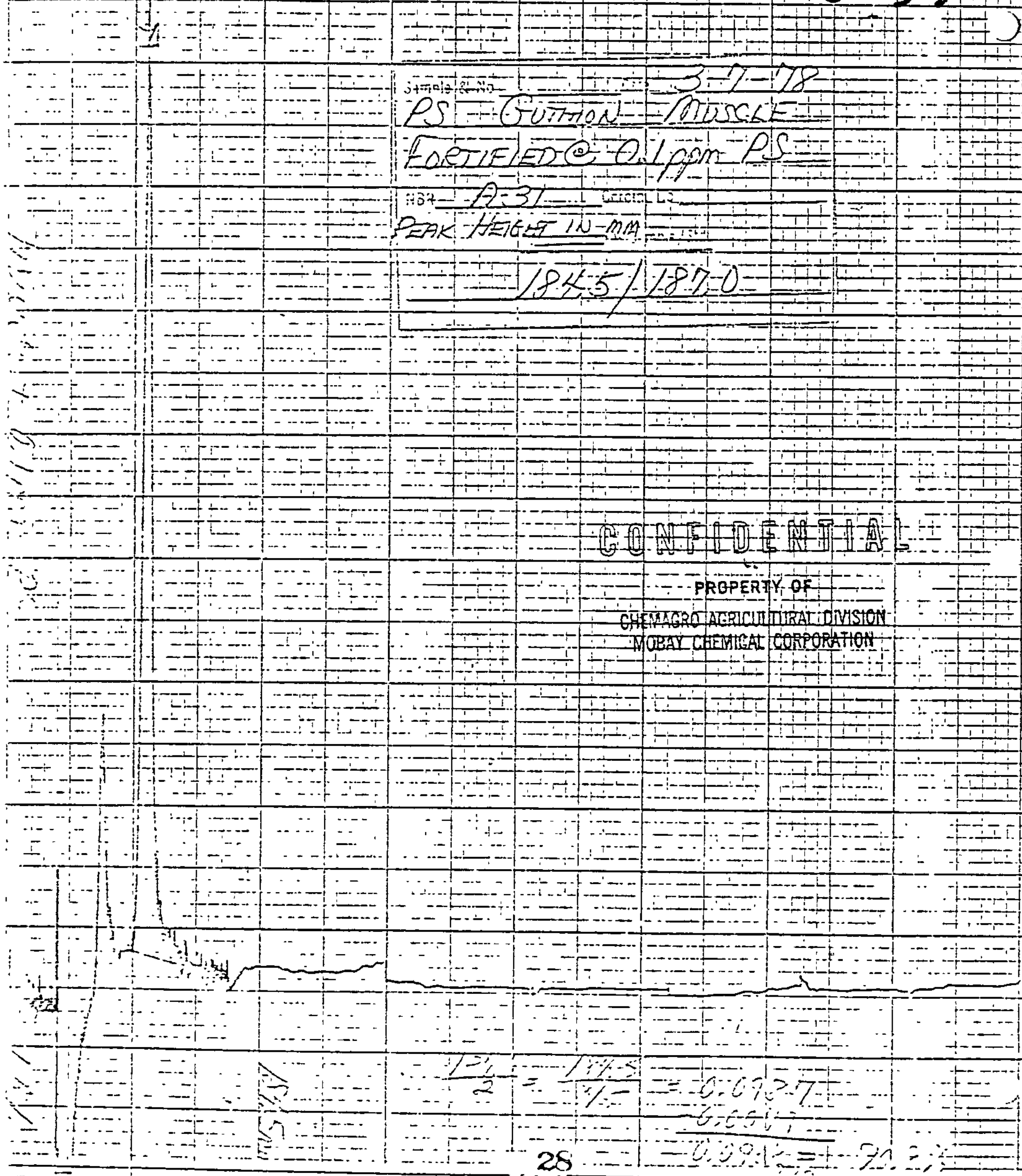

Sample & No. 3-7-78

PS Guthion Muscle

Fortified @ 0.1 ppm PS

NB# A-31

Peak Height in mm

184.5 / 187.0

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$\frac{[illegible]}{2} = \frac{184.5}{[illegible]} = 0.0987$

0.00[illegible]

0.09[illegible] = [illegible]

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3-2-78

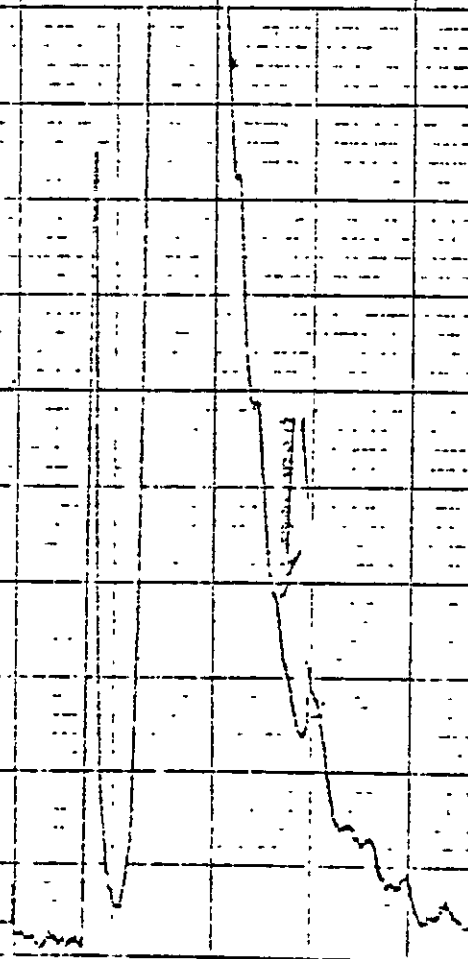
PO - GUTHION MUSCLE

CONTROL

A-31

PEAK HEIGHT IN MM

0.0/204.0



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Sample # No. _____ Date 3-2-78

PO GUTHION MUSCLE

FORTIFIED @ 0.1ppm PO

TLBR A-31 Chrom. No. _____

PEAK HEIGHT IN MM Sample/Std.

164.0/206.0

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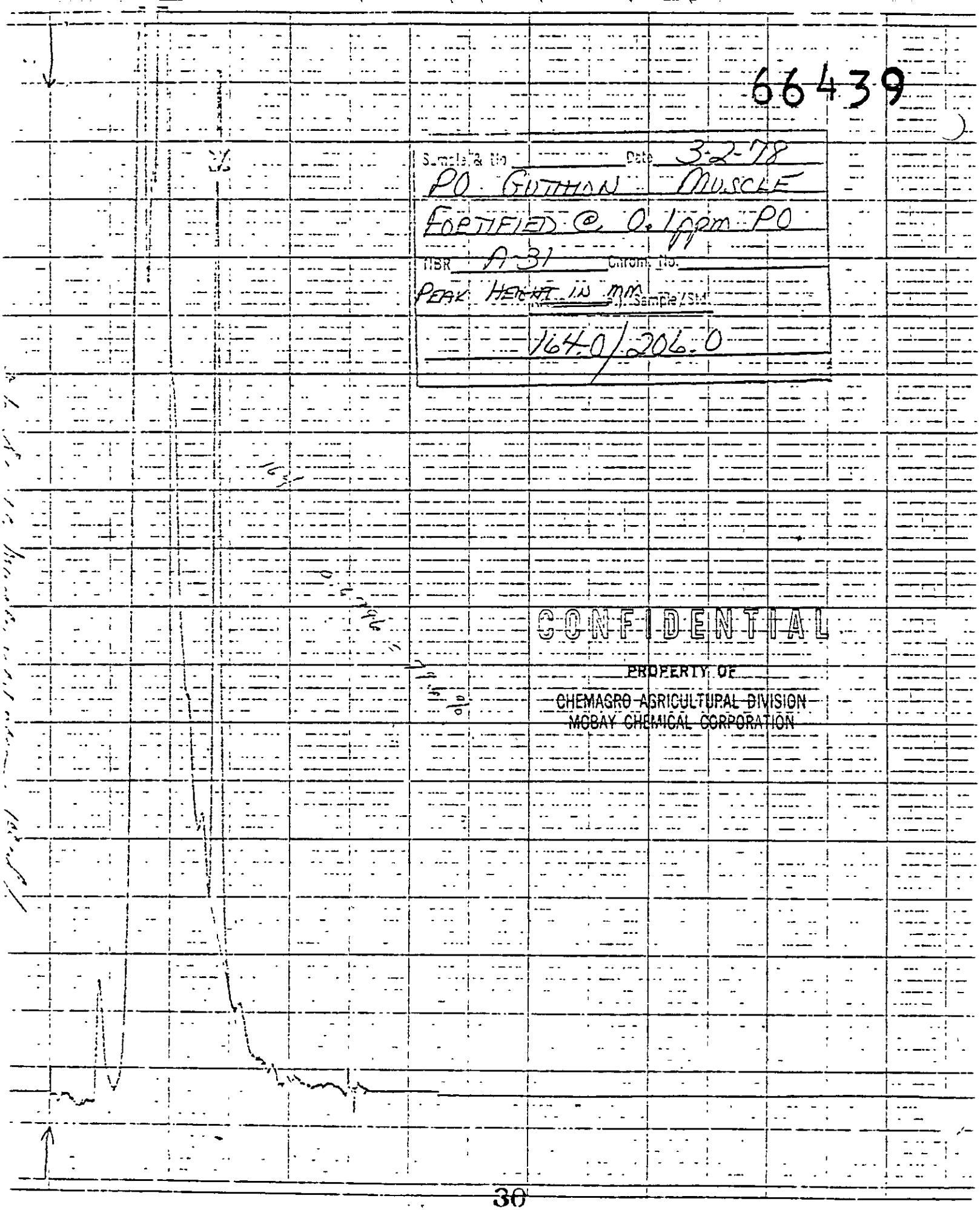
MOBAY CHEMICAL CORPORATION



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PS — Guthion — Fat

Control

A-31

0.0/1490

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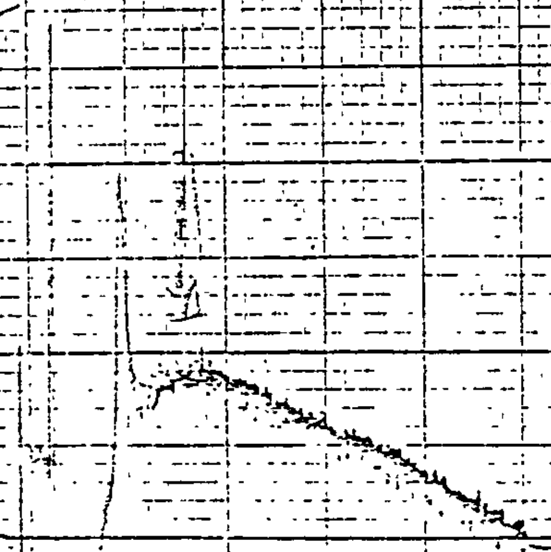

0.000 ppm

06439

Date 3-6-78

p-CUMIC FAT

FORTIFIED FAT 0.1ppm-PS

NBR P-51 Chrom No

PEAK HEIGHT in mm Sample/Std

120.2/132.7

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$\frac{132.7}{x} = \frac{120.2}{2K}$

32 0.0214 → 97.47

66439

Sample & No. _____ Date 3-6-72

PD-GUTHION FF

COUNTY

NBR. F-31 Chrom. No. _____

PEAK-HEIGHT-IN-MM Sample/Std.

0.0/158.0

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66439

Sample & No. L Date 3-6-72

PO - GUTHION — FAT

FORTIFIED @ 0.1 ppm PO

NBR A-31 Chrom. No.

PEAK HEIGHT - IN MM (Area) - Sample / Std.

123.0 / 150.0

66439

3-16-78

PS GUTHION - MILK

CONTROL

A 31 pg. #13

PEAK HEIGHT 10 mm

0.0/1620

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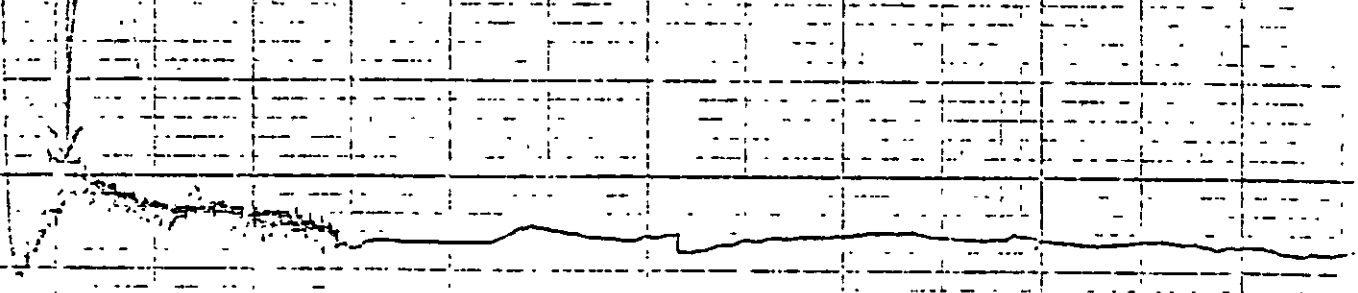

66439

3-16-78

PS Guthion Milk

fortified @ 0.01ppm PS

A-31 pg. # 13

156.0/187.0

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Sample & No. Date 3-10-78

PO-GUTHION MILK

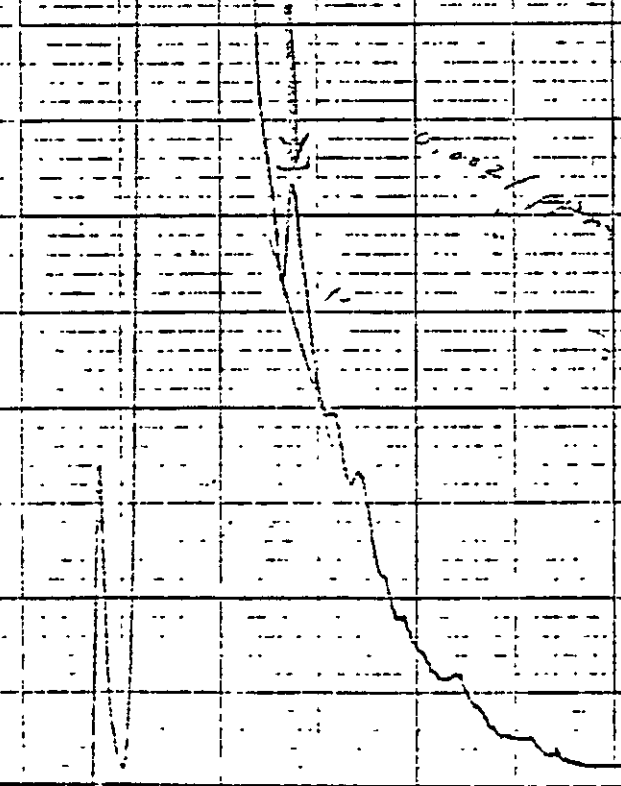
CONTROL

Vol. A31 PAGE 13

Areas (sq. in.) - Sample/Std.

18.0/75.0

#1 Milk Control 100µl/1ml



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Sample # No _____ Date 3-10-78

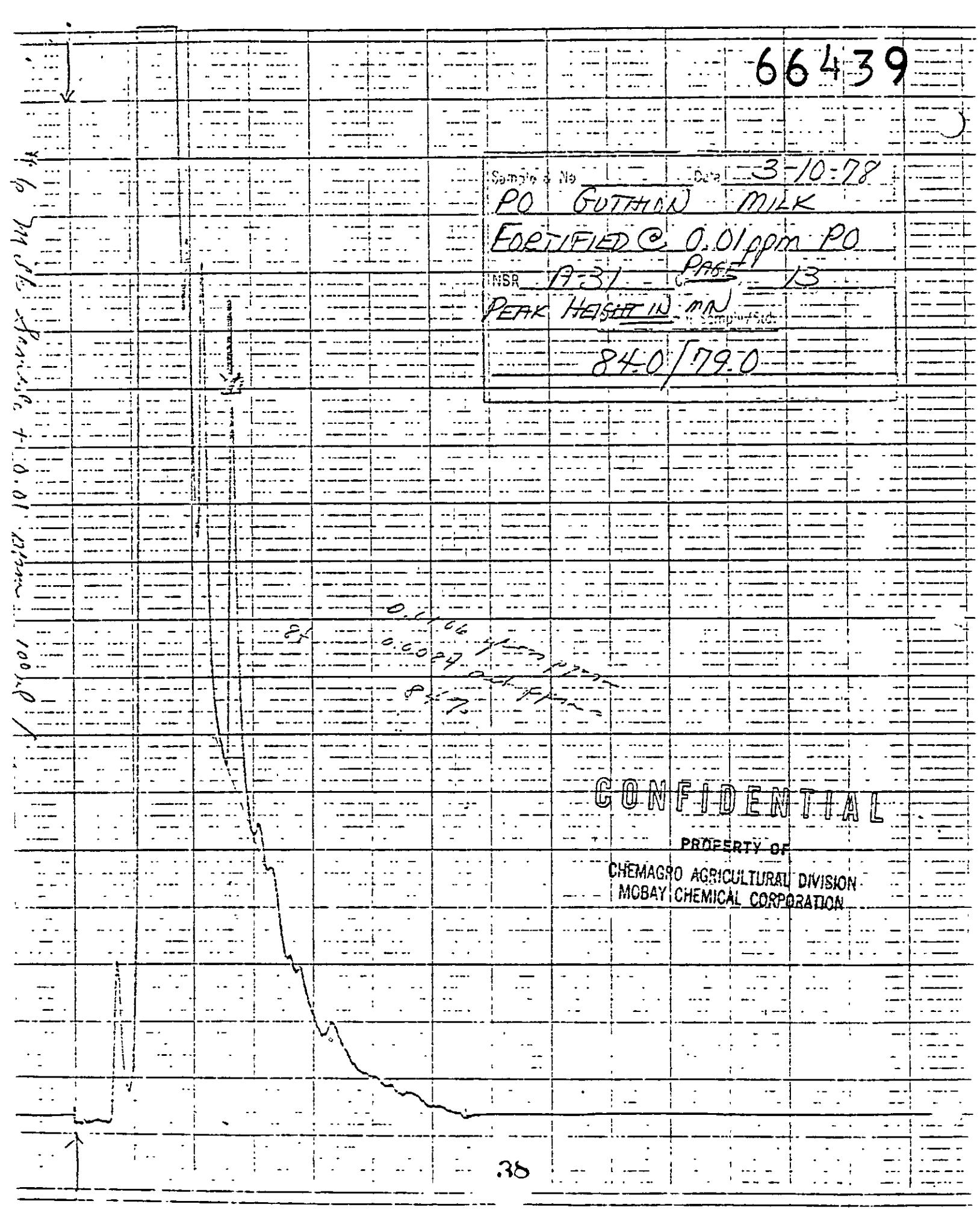
PO GUTHION MILK

FORTIFIED @ 0.01 ppm PO

NSR 931 Page 13

PEAK HEIGHT IN mm Sample/Std.

840/790



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3-7-78

PS Guthion Standard

2 oz/1 mile

A-31

Peak Height 10 mm

187.0

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MOBAY CHEMICAL CORPORATION

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Sample ID(s) _____ Date 3-2-78

PO GUTHION STANDARD

2 ug/ml

Inj. Vol. 0.3 µl Chrom. No.

PEAK HEIGHT IN mm

Inj. Sample/S.d.

206.0

PO Std Guthion 2ug/ml