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FMC CORPORATION
AGRICULTURAL CHEMICAL GROUP
Princeton, New Jersey

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P-2352M
Page 1 of 38

STUDY TITLE: Residue Analytical Method for the
Determination of Clomazone in/on the
Processed Parts of Field Corn Grain

TEST SUBSTANCE: Clomazone

DATA REQUIREMENT: Pesticide Assessment Guidelines
Subdivision O, 171-4: Residue
Analytical Method

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STUDY DATES:
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Completed: July 1990
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LABORATORY PROJECT ID: 164COF88R4-1

Non Propriety Information
FMC Corporation Authorizes the Release or Use
of This Method by Federal and State Agencies

FMC CORPORATION

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA §10(d)(1)(A), (B), OR (C).

Company: FMC Corporation
Agricultural Chemical Group



Ronald F. Cook
Manager, Residue Chemistry
COMPANY AGENT

20 July 90

Date

GOOD LABORATORY PRACTICES STATEMENT

To the best of my knowledge the study reported herein (Study ID: 164COF88R4-1, "Methodology for the Determination of Clomazone Residue in/on the Processed parts of Field Corn Grain," FMC Corporation, Agricultural Chemical Group, P-2352M) was conducted and reported in compliance with the Good Laboratory Practice Standards set forth in Title 40, Part 160 of the Code of Federal Regulations of the United States of America.

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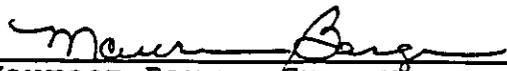
24 July 1990
Date

QUALITY ASSURANCE STATEMENT

It is the intent of FMC Corporation that all studies conducted by our facility shall be of the highest quality and meet or exceed the criteria promulgated by the EPA to assure the quality and integrity of the data generated. Study 164COF88R4-1 was inspected by the FMC ACG Research and Development Quality Assurance Unit and the findings submitted to the Study Director, the Manager of Residue Chemistry, and the Director of Developmental Chemistry on the following dates.

<u>INSPECTION DATE</u>	<u>SUBMITTED TO STUDY DIRECTOR</u>	<u>SUBMITTED TO REGIONAL MANAGER</u>	<u>SUBMITTED TO GROUP MANAGER</u>
09/25/89	09/27/89	10/02/89	10/03/89

FMC ACG Research and Development Quality Assurance Unit audited the raw data and all records on 11/21/89 and the report on 2/12/90 and 7/12/90. The report was found to be an accurate reflection of the study and the data generated. All raw data will be maintained by FMC Corporation, PO Box 8, Princeton, N.J. 08543 in the Quality Assurance Archives.



Maureen Barge, Supervisor
Quality Assurance Unit

11-8-90
Date

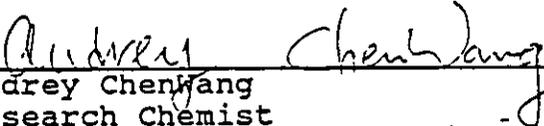
REVISIONS

Page 1 Date of revisions was added.

Page 4 Study number 182COF88R4-1 in line 5 was corrected to 164COF88R4-1.

Page 12 Name of chemical in line 17 was corrected to clomazone.

Page 22 Background residues of all the control matrices were added into Table 2.



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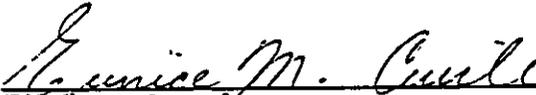
Date



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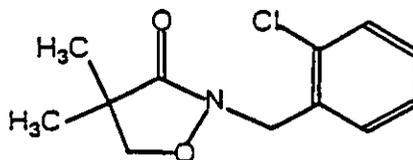
Date

TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	6
II. SUMMARY	6
III. SUMMARY TABLES AND GRAPHICS	8
A. Summary Table of Method Recovery	
B. Method Flow Schemes	
IV. MATERIALS AND STUDY DESIGN	11
A. Equipment	
B. Reagents	
C. Analytical Standards	
D. Study Design	
V. ANALYTICAL METHODS	12
A. Sample History	
B. Method for Non-Oil Sample	
C. Method for Oil Sample	
D. Instrumentation	
E. Interferences	
F. Confirmatory Techniques	
G. Time Required for Analysis	
H. Modifications or Potential Problems	
I. Method of Calculation	
VI. STORAGE STABILITY	19
VII. RESULTS AND DISCUSSION	19
A. Accuracy and Precision	
B. Limits of Detection and Quantification	
C. Ruggedness Testing	
D. Limitations	
VIII. CONCLUSION	20
IX. CERTIFICATION	21
X. TABLES AND FIGURES	22
XI. APPENDIX	24
Chromatograms	

I. INTRODUCTION

Clomazone, the common name of the active ingredient in Command herbicide, is currently being developed by FMC Corporation for use in the control of weeds on field corn. It has the chemical name 2-[(2'-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone, code number FMC 57020, and Chemical Abstract Service No. 81777-89-1. The structure follows:



CLOMAZONE

This report was generated as part of study 164COF88R4-1 to support the determination of clomazone in/on the processed parts of field corn grain. The objective of the cited study of the protocol were contained in report "Magnitude of the Residue of Clomazone in/on the Processed Parts of Field Corn Grain," FMC Report P-2352, July 1990.

The current existing analytical methods for clomazone in/on various crops are time- and labor-consuming and produce a considerable volume of solvent waste. It is for these reasons that the following solid phase extraction and clean up methods have been developed by FMC Corporation for the analysis of clomazone in/on the processed parts of field corn grain.

II. SUMMARY

The analytical method for the non-oil fractions of processed corn grain includes an acid hydrolyzing step, C₁₈ solid phase extraction, and clean-up by a Florisil® cartridge. Assay analysis for oil samples initiates with hexane dissolution, followed by acid-base wash, hexane/acetonitrile partition, acetonitrile/water/hexane partition and clean-up by a silica cartridge. Residue level of clomazone was

quantitated by a gas chromatograph equipped with a megabore capillary column and a nitrogen/phosphorous detector. Method validation was performed on control samples with no known residue of clomazone. Recovery calculation was based on an external standard calibration.

Both non-oil and oil methods had a sensitivity of 0.05 ppm and a detectability of 0.01 ppm. The average percent recovery was ranged from 74.2 to 87.3, and from 84.3 to 96.7 for non-oil and oil samples, respectively.

III. SUMMARY TABLES AND GRAPHICS

A. Method Recovery Summary

TABLE 1
CLOMAZONE RECOVERIES
IN/ON CORN GRAIN AND PROCESSED PARTS

Sample I.D.: 88-CRS-16C
Fortification Level: 0.05 ppm

MATRIX	NUMBER OF ANALYSIS	RECOVERY RANGE (%)	AVERAGE RECOVERY (%)
Corn Grain	3	80.4 - 96.5	87.3
<u>DRY MILLING</u>			
Medium Grits	3	72.0 - 77.2	74.2
Meal	3	71.8 - 101.0	82.7
Flour	3	75.7 - 85.2	82.0
Crude Oil	3	85.7 - 112.4	96.7
Refined Deodorized Oil	3	72.2 - 98.2	85.2
<u>WET MILLING</u>			
Starch	3	69.1 - 84.0	79.0
Crude Oil	3	65.2 - 97.8	85.1
Refined Deodorized Oil	3	75.0 - 99.1	84.3

B. Method Flow Schemes

FIGURE 1

METHOD FLOW SCHEME FOR CLOMAZONE
ON CORN GRAIN, GRITS, MEAL, FLOUR, STARCH

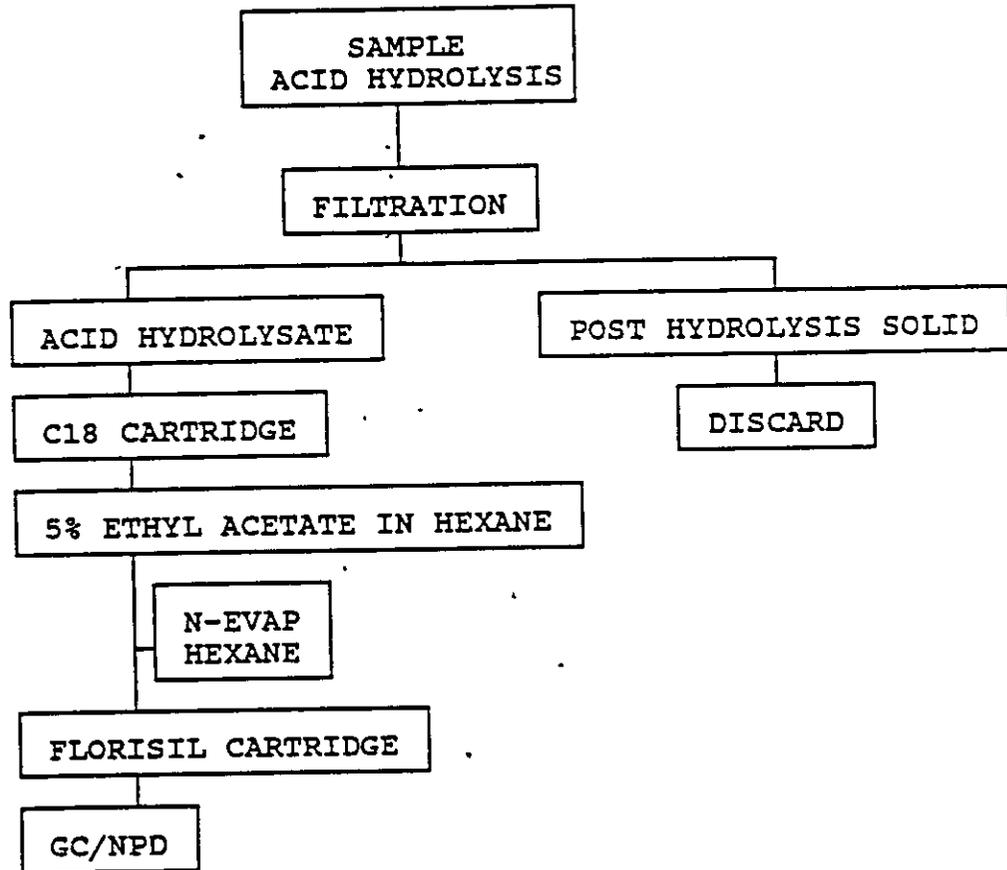
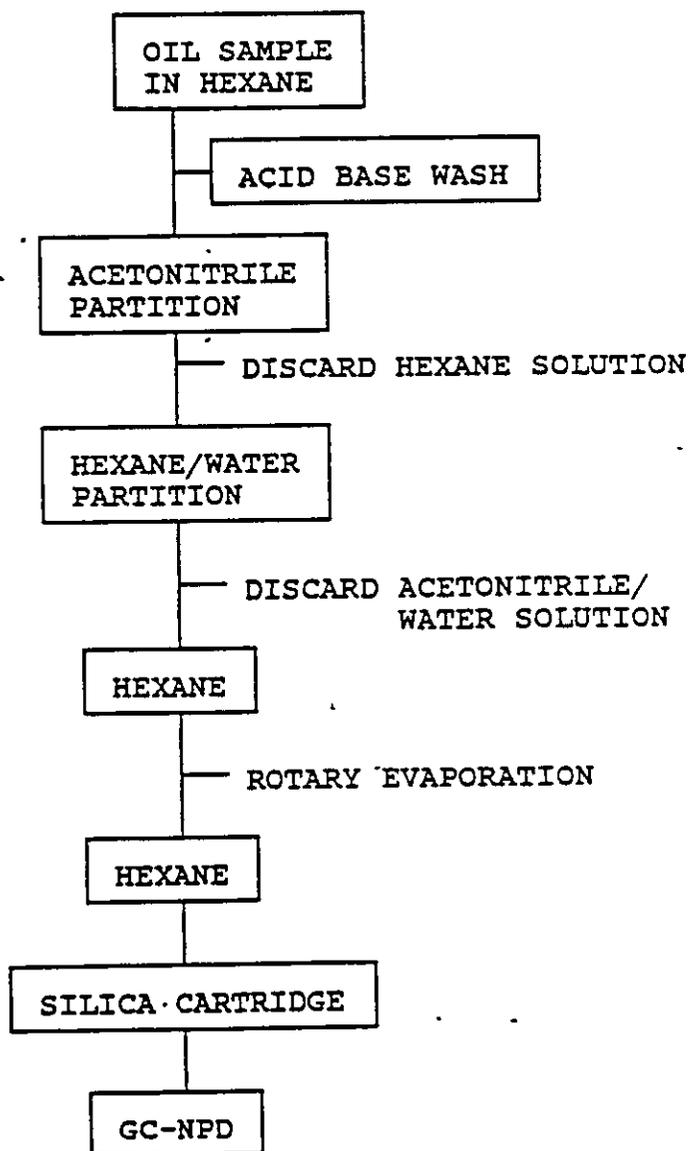


FIGURE 2
METHOD FLOW SCHEME FOR CLOMAZONE
IN CRUDE AND DEODORIZED OILS



IV. MATERIALS AND STUDY DESIGN

A. Equipment

Balance (Mettler PM600)
Boiling chip, granules, plain (Hengar Co.)
Cartridge, C₁₈, 1 g (Analytichem International)
Cartridge, Florisil, 1 g (Analytichem International)
Cartridge, Silica gel, 2 g (Analytichem International)
Centrifuge tube, disposable polypropylene, 50 ml (Corning, VWR Scientific)
Centrifuge tube, pyrex, 13 ml, 0.1 ml graduation
Filter paper, No. 1 (Whatman)
Filtering flask, pyrex
Flask, Erlenmeyer, 250 ml
Flask, flat-bottom, 500 ml with 24/40 \overline{S} joint
Flask, volumetric, 100 ml
Gas chromatograph (Hewlett-Packard 5890 with NPD)
Graduate cylinder, 25 ml, 100 ml
Heating mantles
N₂-Evap evaporator (Organomation Associates 111)
Porcelain buchner filter funnel, 10.5 cm i.d., (Coors)
Rotavapor evaporator (Buchi/Brinkman R-110)
Separatory funnel, 250 ml
Solid Phase Extraction Manifold (Supelco)
Syringe, (Hamilton)
Test tube mixer (Bernstead/Theymolyne M16715)
Thomas-Wiley Mill, ED-5 (Thomas Co.)

B. Reagents

Acetone, ethyl acetate, hexane, acetonitrile, cyclohexane, all Resi-analyzed^R grade solvent (J.T. Baker Chemical)

Hydrochloric acid, reagent grade (J.T. Baker Chemical)

0.25N HCl solution, prepared from reagent grade HCl

Sodium Bicarbonate, powder, reagent grade (J.T. Baker Chemical)

Saturated NaHCO₃ solution, prepared from reagent grade NaHCO₃

Distilled deionized water (house still)

C. Analytical Standards

The structure and purity of the chemical used as analytical standard can be found in Table 3, Section X. Stock solution (1000 ng/uL) was prepared annually in hexane solution. Fresh dilute solutions were prepared at suitable concentrations (0.05-2.0 ng/uL) in hexane on a monthly basis. These dilute solutions were used for sample fortification and instrument linearity calibration.

D. Study Design

A method validation set consisted of one control sample and three fortified control samples for each matrix. Fortification was accomplished by adding a known amount of clomazone standard in hexane solution directly onto the control sample matrix using a syringe. The concentration of clomazone standard solution used to fortify the control sample was 1.0 ng/uL. The volume of clomazone in hexane added was 250 uL. After spiking, the sub-sample containers were left open at room temperature to allow the hexane to evaporate. The level of fortification was set at method sensitivity (0.05 ppm). The average method recovery was obtained by averaging three recoveries.

V. ANALYTICAL METHODS

A. Sample History

The field corn grain samples after harvest were collected and shipped to Texas A&M University for processing by dry milling and wet milling techniques. Corn grain and processed products were then shipped to FMC residue laboratory at Princeton, NJ (Receiving Numbers PRF 15 and 16, sample I.D. 88-CRS-16C). Corn grain was ground by using a Thomas-Wiley Mill after the addition of liquid nitrogen. These samples were stored under frozen (ca. -18°C) until analysis.

B. Method for Non-Oil Corn Fractions (Corn Grain, Medium Grits, Meal, Flour, and Starch)

1. Acid Hydrolysis

Five grams of sample was weighed and placed into a 500 ml flat-bottom boiling flask. The fortification was done at this time. One hundred ml of 0.25N HCl solution and two or three boiling chips were added after hexane solvent evaporation. The heating mantle variac power supply was set at 60 volts and the sample solution was refluxed for one hour.

The hydrolysate was cooled down to room temperature after the reflux and was filtered through a Whatman filter paper (No. 1) with a buchner funnel by vacuum. About 100 ml of deionized water was used for the rinse and wash of flask and sample matrix.

2. C₁₈ Solid Phase Extraction

The filtrates (~ 200 ml) were pump^{ed} through a C₁₈ cartridge (1 g, 6 cc), which was pre-conditioned first with 12 ml (two times bed volume) of ethyl acetate/hexane (5/95, v/v) solution, the cartridge was vacuum dry^{ed} completely, and then with 12 ml of deionized water. Under vacuum, the sample solution flow rate was maintained at five ml per minute. The vacuum was kept on for about 30 minutes to let the cartridge become completely dry after the solution was run through. The clomazone residue was eluted with 12 ml of ethyl acetate/hexane (5/95, v/v) (collection rate ~2 ml/min) into a graduated test tube and was N₂-evaporated to 1 ml.

3. Florisil Cartridge Clean-up

The solution was further cleaned up by using a Florisil cartridge (1 g, 6 cc), which was pre-conditioned by gravity with two times bed volume (12 ml) of ethyl acetate/hexane (20/80, v/v) and hexane solvent (or 5:95 ethyl acetate/hexane). One ml of cyclohexane was added to rinse the test tube and was then transferred onto the cartridge. Twelve ml of ethyl acetate/hexane (20/80, v/v) was used to elute clomazone residue

into a clean graduated test tube (by gravity) and was N₂-evaporated down to exactly 1 ml. The final solution was quantitated by a gas chromatograph equipped with a nitrogen/phosphorous detector. Figure 1 in Section III presents the method flow scheme for non-oil samples.

C. Method for Corn Oil Fractions

1. Acid-Base Wash

Five grams of oil sample was weighed into a 250 ml separatory funnel. The fortification was done at this time. One hundred ml of hexane was added and the sample/hexane solution was washed with 50 ml of 0.25N HCl and saturated sodium bicarbonate solutions, respectively. The acid and base solutions were discarded.

2. Solvents Partitions

The oil/hexane solution after wash was collected in a flat-bottom boiling flask and rotary evaporated to ~ 5 ml. Hexane was added to rinse and transfer the oil sample from the boiling flask to a 50 ml centrifuge tube (polypropylene). The volume of the oil/hexane solution was made to 20 ml and was partitioned twice, each with 10 ml of acetonitrile solvent. The hexane solution was discarded. The combined acetonitrile solutions were collected into another clean separatory funnel. One hundred ml of deionized water was added and the acetonitrile/water solution was again partitioned twice, each with 100 ml of hexane solvent. The hexane solutions were collected in a clean boiling flask and rotary evaporated to ~ 2 ml. A total of 8-9 ml of hexane was added to rinse and transfer the clomazone residue into a centrifuge test tube (three times, each with 2-3 ml).

3. Silica Cartridge Clean-up

The sample/hexane solution was N₂-evaporated to ~ 2 ml and was further cleaned up by a silica cartridge (2 g, 12 cc), which was pre-conditioned first with two times bed volume (24

ml) of ethyl acetate/hexane (20/80, v/v) solution, and then with hexane solvent. Ten ml of 10% ethyl acetate in hexane was added to rinse the test tube and was then transferred onto the cartridge. Solution flow rate was maintained at ~ 2 ml/min under vacuum. Twelve ml of ethyl acetate/hexane (20/80, v/v) was used to elute clomazone residue into a test tube and was N₂-evaporated down to exactly 1 ml. The final solution was quantitated by a gas chromatograph equipped with a nitrogen/phosphorous detector. Figure 2 in Section III shows the method flow diagram for oil samples.

D. Instrumentation

1. Gas Chromatography

Clomazone was determined by a Hewlett-Packard 5890 gas chromatograph equipped with a nitrogen/phosphorous detector, a HP 3392A integrator, and a HP 7672A autosampler. Operating conditions are described as follows:

COLUMN:	J&W DB-1 dimethyl silicone 15 m X 0.53 mm X 1.5 um
INLET:	Direct injection mode (250°C)
OVEN TEMP:	170°C
Run Time:	10 min
DETECTOR TEMP:	300°C
GAS FLOW RATE:	He, carrier, -5 ml/min He, make-up, -15 ml/min H ₂ , -3.5 ml/min Air, -85 ml/min
RETENTION TIME:	- 5 min

The chromatographic system was calibrated after every two sample injections using the external standard method. A linearity curve, consisting of at least three points, was generated to insure the linear response of the instrument during sample analysis for every set assay.

E. Interferences

Sample Matrices - No visible interference was noted in any check fractions.

Other Pesticides - No interference due to other pesticides was expected.

Solvents and Labware - No interference was observed from solvents and labware.

F. Confirmatory Techniques

No other analytical instrument or technique was used to confirm clomazone residue in/on corn grain or processed parts.

G. Time Required for Analysis

The analytical procedures required approximately seven hours for non-oil fractions and 12 hours for oil samples, during which time one person completed a set of eight samples from initial weighing until gas chromatographic measurement.

H. Modification or Potential Problems

1. When the variac power supply of the heating mantle was set higher than 60 Volts, some corn grain sample would float up and stay inside the condenser or around the neck of the boiling flask during the strong acid reflux, and slightly lower recovery (- 5-10% less) has been observed.
2. Plenty of deionized water to rinse and wash the sample matrix during the filtration step was necessary, because - 10% loss of recovery was also observed when the amount of deionized water was decreased from 100 mL to 50 mL.
3. C₁₈ cartridge had to be completely dry before clomazone was eluted with 5% of ethyl acetate in hexane. Blowing the cartridge dry with nitrogen gas worked as well as air sucking dry through pump.

4. The crude oil/hexane solution had a tendency to form an emulsion when washed with base solution after acid wash. Such a problem could be prevented if vigorous shaking was avoided and the pressure was rapidly released.
5. When cartridges from other manufacturers were used, a different elution pattern has been observed. Therefore when the method is being performed, it is better either to use the cartridges from the same company or test the elution solvents with cartridges from different companies for the method recovery.
6. Oven temperature of gas chromatograph could be programmed to higher final temperatures after initial isothermal condition to prevent any possible matrix interference from late elutions.

I. Methods of Calculation

The amount of clomazone was quantitated from the detector electrical response transmitted to the instrument integrator. For most of the fractions (except wet milling crude oil) the responses as peak area were calculated as ng of clomazone based on a 0.25 ng/ul injection of run standard. As for the wet milling crude oil, the noisy matrix background seemed to interfere with the integration accuracy; peak heights (mm) were used to calculate the ng of clomazone based on a 0.25 ng/ul injection of run standard. A run standard was injected at the beginning of every set and subsequently after every two sample solutions. Clomazone magnitude of each sample was determined by an external standard calibration method based on an average of all run standards.

The nanogram values reported were calculated by comparing the area (or height) units of unknown sample to that of the average run standard using the following formulae:

$$\text{ng of clomazone in sample} = \frac{\text{Area or height unit (sample)}}{\text{Average area or height unit (standard)}} \times \text{ng (standard)}$$

Results of each analysis were reported on a ppm (ug/g) basis by using the following formulas:

$$\text{clomazone content (ug/g)} = \frac{\text{ng of clomazone in sample}}{\text{mg of sample injected}}$$

Method recovery was then obtained by comparing the clomazone content in sample to the initial fortification level.

$$\text{Method recovery (\%)} = \frac{\text{clomazone content (ppm)}}{\text{fortification level (ppm)}} \times 100$$

An example of how to calculate the method recovery in a fortified corn grain sample (Figure 5) is given below:

ng (Standard)	$2 \text{ ul} \times 0.25 \text{ ng/ul} = 0.5 \text{ ug}$
Average Area Units of Standards	$= 1825.6$
Area Units of Fortified Grain Sample	$= 1552$
ng of Clomazone in Sample	$= \frac{1552 \times 0.5 \text{ ng}}{1825.6} = 0.4251 \text{ ng}$
Clomazone Content (ppm)	$= \frac{0.4251 \text{ ug}}{10 \text{ mg}} = 0.0425 \text{ ppm}$
Method Recovery (%)	$= \frac{0.0425 \text{ ppm} \times 100}{0.05 \text{ ppm}} = 85.0\%$

VI. STORAGE STABILITY

Clomazone analytical standard was assayed on a regular basis for percent purity. The standard had a proven pattern of stability. Stock solution (1000 ug/mL) was prepared annually in hexane solution. Fresh dilute solutions were prepared at suitable concentrations on a monthly basis. All solutions were stored in volumetric containers in refrigerator/freezer units and have proven stability for their respective storage periods.

VII. RESULTS AND DISCUSSION

A. Accuracy and Precision

The control samples were analyzed and no detectable clomazone was found in/on all control corn grain and processed parts. The control samples were then fortified with clomazone at 0.05 ppm. These samples were carried through the same analytical procedures as for the treated samples. The results were used to calculate method recoveries. The accuracy and the precision of the analytical method were determined by the average recovery and standard deviation of the results from the fortified control samples. Table 2 in Section X presents the individual method recovery data with average method recovery and standard deviation of three recovery values for each matrix. The average percent recovery in this study ranged from 74.2 ± 2.7 to 96.7 ± 14.0 , depending on the fraction analyzed.

B. Limits of Detection and Quantification

Method sensitivity was validated as 0.05 ppm based on acceptable fortified recovery values. Method detectability or recognition of detector response was set at 0.01 ppm (signal/noise ratio = 3). Any response below this limit was considered not detectable (ND).

C. Ruggedness Testing

The non-oil method was initially developed and validated for corn grain and was then applied on medium grits, meal, flour, and starch. This method has been practiced on corn silage and stover samples with satisfactory results (for stover 2.5 g of initial sample weight and 200 mL of 0.25N HCl were used, and the final sample solution was 0.5 mL).

The oil method was first validated on crude oil and then refined deodorized oil. No other oil was tested.

D. Limitations

No potential limitations were experienced during the assay analyses.

VIII. CONCLUSION

The analytical methods were developed for the residue determination of clomazone in/on corn grain and its non-oil and oil processed parts. All the equipment needed to perform the analysis, e.g., gas chromatograph with nitrogen/phosphorous detector, is readily available in most residue analytical laboratories. An experienced residue analyst following the procedure exactly as written, and being aware of the possible potential problems, should not experience interference problems and should obtain adequate recoveries.

IX. CERTIFICATION

We, the undersigned, hereby declare that this study was performed under our supervision according to the procedures herein described, and that this report provides a true and accurate record of the results obtained.

Audrey Chen Wang
Audrey Chen Wang
Research Chemist
AUTHOR/STUDY DIRECTOR

7/20/90
Date

R. F. Cook
Ronald F. Cook
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SUPERVISOR

2/24/90
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ADDITIONAL STUDY PERSONNEL
Deborah A. Eng, Chemist

X. TABLES AND FIGURES

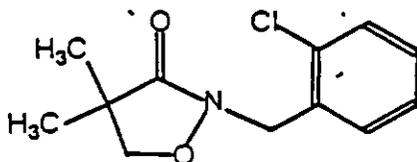
TABLE 2
 METHOD RECOVERIES FOR CLOMAZONE

MATRIX	SAMPLE IDENTIFICATION	BACKGROUND RESIDUE	FORTIFICATION LEVEL (PPM)	RECOVERY (%)	AVERAGE RECOVERY (%)
Corn Grain	88-CRS-16C	ND*	0.05	85.0	87.3 ± 8.3
			0.05	80.4	
			0.05	96.5	
<u>Dry milling</u> Medium Grits	88-CRS-16C	ND	0.05	73.3	74.2 ± 2.7
			0.05	77.2	
			0.05	72.0	
Meal	88-CRS-16C	ND	0.05	101.0	82.7 ± 16.0
			0.05	71.8	
			0.05	75.3	
Flour	88-CRS-16C	ND	0.05	85.2	82.0 ± 5.5
			0.05	85.1	
			0.05	75.7	
Crude Oil	88-CRS-16C	ND	0.05	112.4	96.7 ± 14.0
			0.05	92.0	
			0.05	85.7	
Refined Deodorized Oil	88-CRS-16C	ND	0.05	85.3	85.2 ± 13.0
			0.05	72.2	
			0.05	98.2	
<u>Wet milling</u> Starch	88-CRS-16C	ND	0.05	69.1	79.0 ± 8.6
			0.05	83.9	
			0.05	84.0	
Crude Oil	88-CRS-16C	ND	0.05	65.2	85.1 ± 17.5
			0.05	97.8	
			0.05	92.4	
Refined Deodorized Oil	88-CRS-16C	ND	0.05	78.8	84.3 ± 12.9
			0.05	99.1	
			0.05	75.0	

* Non-Detectable (< 0.01 ppm)

TABLE 3
ANALYTICAL STANDARD

COMPOUND	CHEMICAL NAME AND STRUCTURE	INVENTORY NUMBER	PERCENT PURITY
Clomazone	2-[(2'-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone	219	99.7



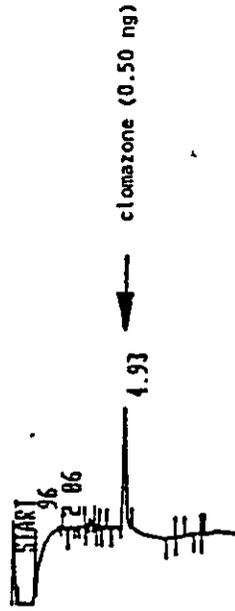
XII. APPENDIX

Chromatograms

FIGURE NUMBER	DESCRIPTION
3	Standard, Clomazone
4	IL corn grain, Control
5	IL corn grain, Fortified
6	Standard, Clomazone
7	Medium grits, Control
8	Medium grits, Fortified
9	Standard, Clomazone
10	Meal, Control
11	Meal, Fortified
12	Standard, Clomazone
13	Flour, Control
14	Flour, Fortified
15	Standard, Clomazone
16	Dry Mill Crude Oil, Control
17	Dry Mill Crude Oil, Fortified
18	Standard, Clomazone
19	Dry Mill Refined Deodorized Oil, Control
20	Dry Mill Refined Deodorized Oil, Fortified
21	Standard, Clomazone
22	Starch, Control
23	Starch, Fortified
24	Standard, Clomazone
25	Wet Mill Crude Oil, Control
26	Wet Mill Crude Oil, Fortified
27	Standard, Clomazone
28	Wet Mill Refined Deodorized Oil, Control
29	Wet Mill Refined Deodorized Oil, Fortified

FIGURE 3

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



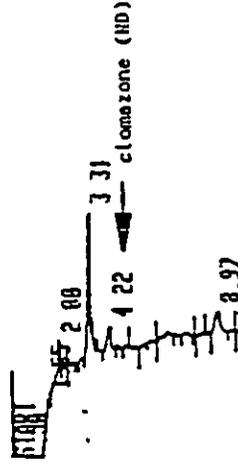
RUN # 711
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1
NO ISTD PK

OCT/06/09 11:17:06

AREA%	RT	AREA TYPE	AR/HIT	AREA%
0.96	2.96	PV	0.364	38.398
2.06	4.215	VV	4.215	59.002
4.93	4.93	PD	0.136	2.600

FIGURE 4

CONTROL CORN GRAIN
(88-CRB-16C, 11-1)
10 mg INJECTED

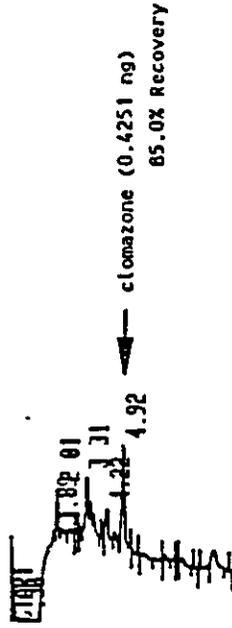


RUN # 712
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO ISTD PK

OCT/06/09 11:28:29

FIGURE 5

0.05 PPM FORTIFIED
CORN GRAIN
(88-CRB-16C, 11-2)
10 mg INJECTED



RUN # 713
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 3
NO 1STD PK

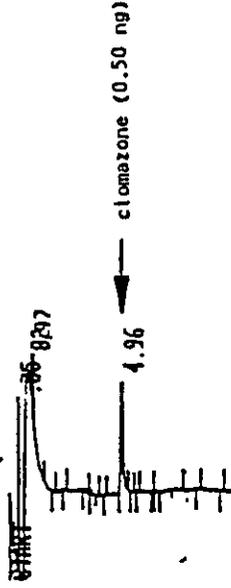
OCT/06/89 11:39:54

AREA%	RT	AREA TYPE	AR/HT	AREA%
0.50	4.16	PB	0.154	0.608
1.09	58226	PV	1.227	85.133
2.01	6811	VV	0.155	9.959
3.31	873	PV	0.131	1.276
4.22	516	BY	0.151	0.755
4.92	1552	PB	0.135	2.269

TOTAL AREA= 60394
MUL FACTOR= 1.0000E+00

FIGURE 6

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



RUN # 781
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 4
NO 1STD PK

OCT/11/89 13:22:28

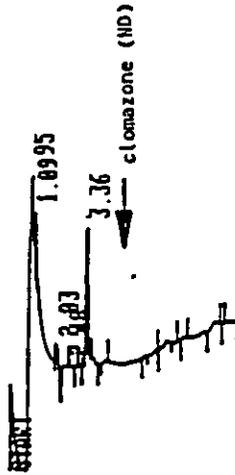
AREA%	RT	AREA TYPE	AR/HT	AREA%
0.35	0	D BB	0.000	0.000
0.02	31459	PV	0.356	57.841
0.97	21406	D VB	0.291	39.357
4.96	1524	BB	0.123	2.802

TOTAL AREA= 54309
MUL FACTOR= 1.0000E+00

RECALIBRATION
NO 1STD PK MATCH

FIGURE 7

CONTROL MEDIUM GRITS
(88-CR8-16C, 13-1)
.10 mg INJECTED



RUN # 779
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO 1STD PK

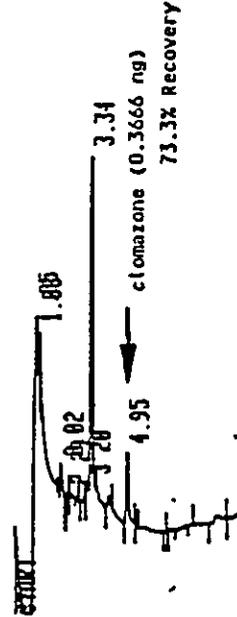
OCT/11/09 12:59:37

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.95	1.09	33955	PY	0.338	60.771
1.09	2.03	10449	VV	0.114	18.701
2.03	2.48	6943	VV	0.235	12.426
2.48	3.36	3051	VB	0.636	5.461
3.36	4.95	1476	VB	0.101	2.642

TOTAL AREA= 55074
MUL FACTOR= 1.0000E+00

FIGURE 8

0.05 PPM FORTIFIED
MEDIUM GRITS
(88-CR8-16C, 13-2)
10 mg INJECTED



RUN # 780
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 3
NO 1STD PK

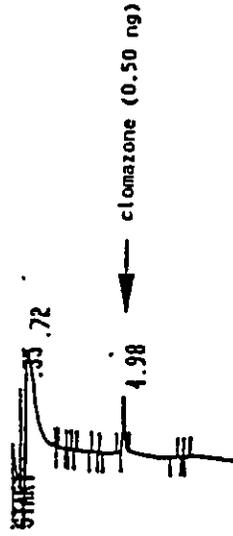
OCT/11/09 13:11:02

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.95	1.08	32165	PV	0.295	60.135
1.08	2.02	10709	VV	0.108	20.021
2.02	2.29	5003	VV	0.199	9.354
2.29	3.20	686	VB	0.130	1.283
3.20	3.34	139	VB	0.075	0.260
3.34	4.95	3794	VB	0.101	7.093
4.95		992	DB	0.124	1.855

TOTAL AREA= 53400

FIGURE 9

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



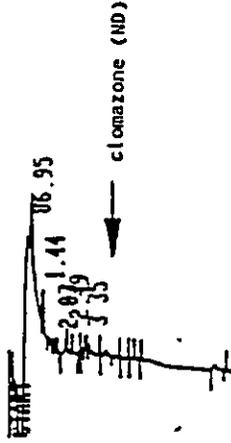
RUN # 855
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1
NO 1STD PK

OCT/13/89 09:52:44

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.35	0.35	35	D	DB	0.030
0.72	0.72	90275	F0	0.752	98.285
4.98	4.98	1540	DB	0.119	1.677

FIGURE 10

CONTROL MEAL
(88-CR8-16C, 14-1)
10 mg INJECTED

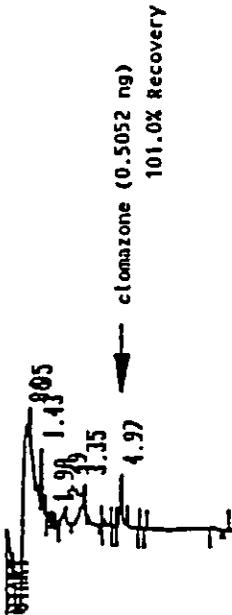


RUN # 856
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO 1STD PK

OCT/13/89 10:04:00

FIGURE 11

0.05 PPM FORTIFIED MEAL
(88-CRS-16C, 14-2)
10 mg INJECTED



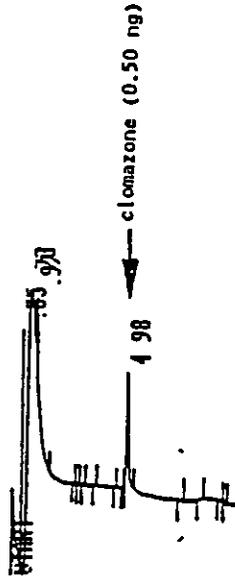
RUN # 857
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # .3
NO 1STD PK

OCT/13/89 10:15:32

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.86	0.86	28684	PV	0.301	49.566
0.95	0.95	18939	VV	0.210	32.727
1.43	1.43	4380	VB	0.129	7.569
1.90	1.90	257	VV	0.084	0.444
2.49	2.49	1602	PV	0.264	2.768
3.35	3.35	2502	VV	0.258	4.462
4.97	4.97	1426	BB	0.123	2.464

FIGURE 12

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



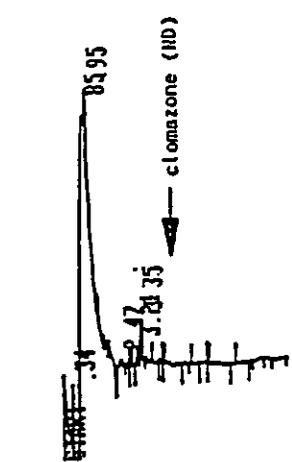
RUN # 820
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1
NO 1STD PK

OCT/12/89 14:13:30

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.35	0.35	197	D BB	0.003	0.297
0.73	0.73	30192	PV	0.353	57.581
0.97	0.97	26364	D VB	0.319	39.748
4.98	4.98	1575	BB	0.121	2.375

FIGURE 13

CONTROL FLOUR
(88-CR8-16C, 15-1)
10 mg INJECTED

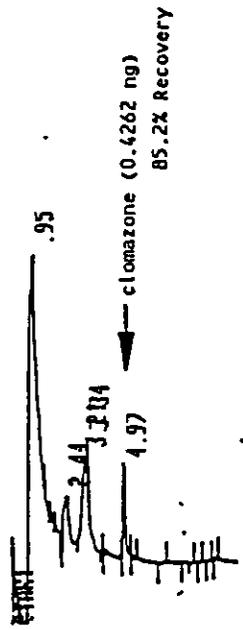


OCT/12/89 14:24:54

RUN # 821
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO ISTD PK

FIGURE 14

0.05 PPM FORTIFIED FLOUR
(88-CR8-16C, 15-2)
10 mg INJECTED



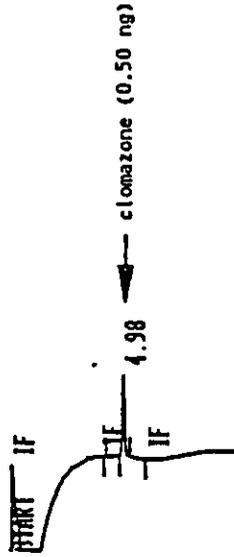
OCT/12/89 14:36:19

RUN # 822
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 3
NO ISTD PK

AREA%	RT	AREA	TYPE	AR/HIT	AREA%
0.95	0.95	84099	PV	0.649	59.307
2.44	2.44	20877	VV	0.513	20.172
3.21	3.21	17704	VV	0.494	12.367
3.34	3.34	10307	VV	0.306	7.256
4.97	4.97	1205	DB	0.122	0.098

FIGURE 15

CLOMAZONE STANDARD (307-23)
2 ul X 0.25 ng/ul



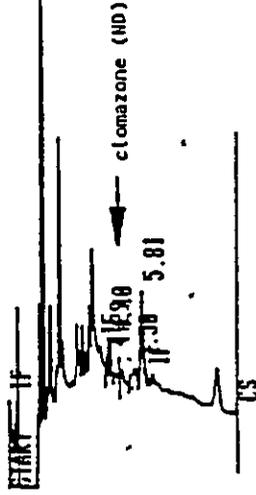
RUN # 1050
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1

OCT/31/89 09:00:33

ESTD	RT	AREA	TYPE	CAL #	AMOUNT
	4.98	2177	BB	1R	0.275

FIGURE 16

CONTROL DRY MILL CRUDE OIL
(88-CR8-16C, 8-1)
10 mg INJECTED

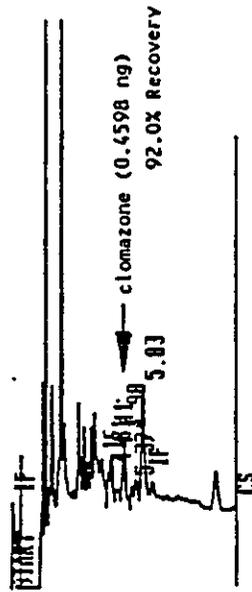


RUN # 1051
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND

OCT/31/89 09:11:57

FIGURE 17

0.05 PPM FORTIFIED
DRY HILL CRUDE OIL
(88-CR8-16C, 8-3)
1.10 mg INJECTED



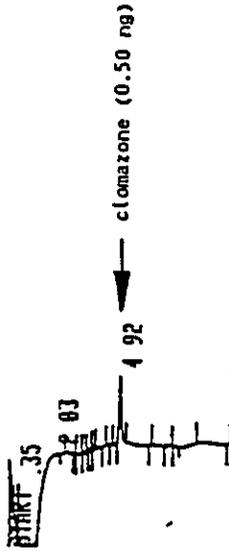
RUN # 1854
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 5

ESTD
RT 4.98
AREA TYPE CAL # AMOUNT
1920 VP IR 0.417

OCT/31/89 10:36:37

FIGURE 18

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



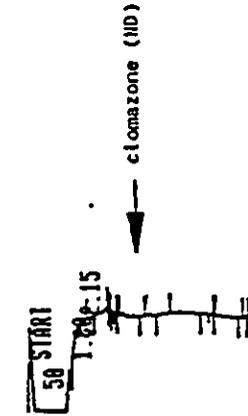
RUN # 626
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 4
HO 1STD PK

AREA%
RT 0.35
2.03
4.92
AREA TYPE
0 D BB
38995 PV
993 BB
AR/HT 0.000
2.838
0.129
AREA%
0.000
97.517
2.483

OCT/02/89 17:43:26

FIGURE 19

CONTROL
DRY MILL DEODORIZED OIL
(88-CRS-16C, 7-1)
10 mg INJECTED

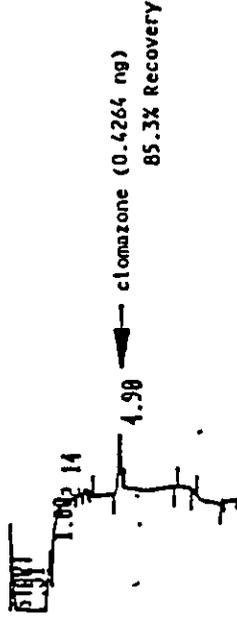


RUN # 624
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO ISTD PK

OCT/02/89 17:20:40

FIGURE 20

0.05 PPM FORTIFIED
DRY MILL DEODORIZED OIL
(88-CRS-16C, 7-2)
10 mg INJECTED



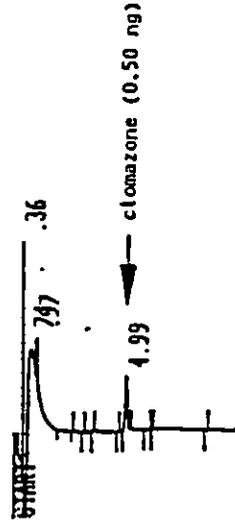
RUN # 625
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 3
NO ISTD PK

OCT/02/89 17:32:03

AREA%	RT	AREA	TYPE	AR/INT	AREA%
0.51	257	806	PB	0.138	0.652
1.80	26929	806	PV	0.796	68.304
2.14	11433	806	VV	0.361	28.999
4.90	806	806	PB	0.129	2.044

FIGURE 21

FHC 54800 STANDARD (307-15)
2 ul X 0.25 ng/ul.



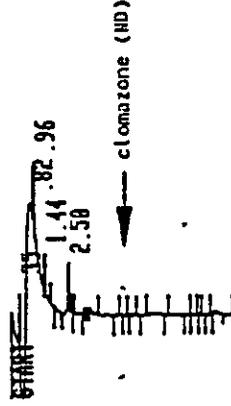
RUN # 868
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1
NO ISID PK

OCT/13/89 13:18:13

AREA%	RT	AREA	TYPE	AR/HIT	AREA%
0.36	0.36	889	D	0.010	0.028
0.74	4.99	42349	PV	0.374	39.440
0.97	4.99	62702	VV	0.603	50.395
4.99	4.99	1436	BB	0.116	1.337

FIGURE 22

CONTROL STARCH
(88-CR8-16C, 16-1)
10 mg INJECTED



RUN # 869
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO CALIB PEAKS FOUND
NO ISID PK

OCT/13/89 13:29:37

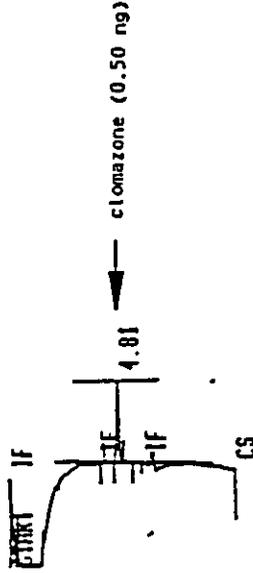
FIGURE 23

0.05 PPM FORTIFIED
STARCH
(88-CR8-16C, 16-4)
10 mg INJECTED



FIGURE 24

CLOMAZONE STANDARD (307-23)
2 ul X 0.25 ng/ul



RUN # 873
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 6
NO ISID PK

OCT/13/89 14:15:18

AREA%	RT	AREA	TYPE	AR/HT	AREA%
0.35	0.01	0.000	0 D BD	0.000	0.000
0.01	0.96	34612	PV	0.354	53.774
0.96	1.44	24560	VV	0.200	30.157
2.50	1.96	2231	VB	0.095	3.466
3.22	3.08	1242	BB	0.113	1.930
3.36	4.98	229	BY	0.087	0.356
4.90		270	VV	0.091	0.432
		1214	BD	0.120	1.886

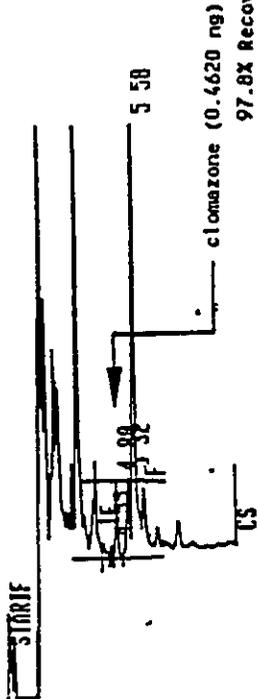
RUN # 1133
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 1

NOV/03/89 09:46:88

ESTD	RT	AREA	TYPE	CAL #	AMOUNT
	4.81	2097	PB	1R	0.622

FIGURE 25

0.05 PPM FORTIFIED
WET MILL CRUDE OIL
(88-CRB-16C, 18-3)
10 mg INJECTED



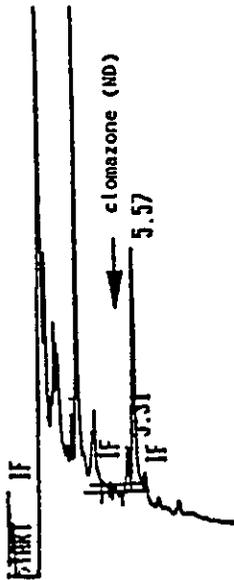
RUN # 1134 NOV/03/89 10:07:37
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2

NO CALIB PEAKS FOUND

ESTD	RT	AREA	TYPE	CAL #	AMOUNT
	4.80	2363	PP	1R	0.686

FIGURE 26

CONTROL
WET MILL CRUDE OIL
(88-CRB-16C, 18-1)
10 mg INJECTED



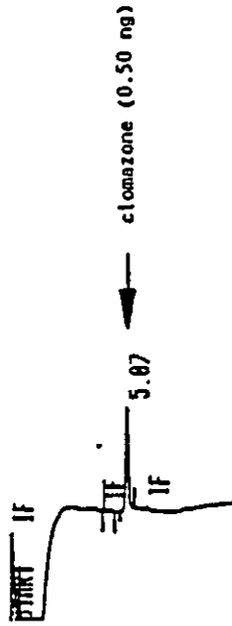
RUN # 1138 NOV/03/89 11:20:39
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 5

97.8% Recovery

ESTD	RT	AREA	TYPE	CAL #	AMOUNT
	4.80	2363	PP	1R	0.686

FIGURE 27

CLOMAZONE STANDARD (307-15)
2 ul X 0.25 ng/ul



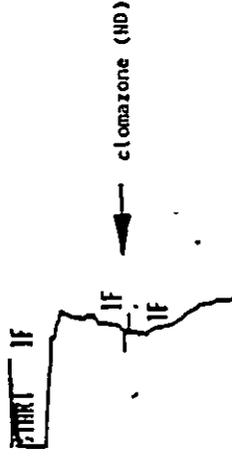
RUN # 930
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020.
SAMPLE # 1

OCT/25/89 14:22:59

ESTD	RT	AREA	TYPE	CAL #	AMOUNT
	5.07	1542	PB	1R	1.283

FIGURE 28

CONTROL
WET MILL DEODORIZED OIL
(88-CR8-16C, 17-1)
10 mg INJECTED

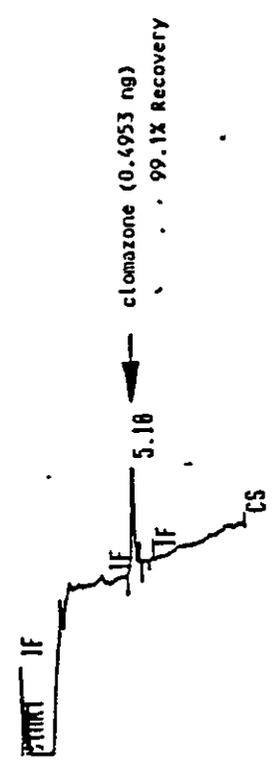


RUN # 931
WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 2
NO RUN PEAKS STORED

OCT/25/89 14:34:17

FIGURE 29

0.05 PPM FORTIFIED
WET MILL DEODORIZED OIL
(88-CR8-F16C, 17-3)
10 MG INJECTED



RUN # 934
OCT/25/89 15:17:02

WORKFILE ID: C8
WORKFILE NAME:
ID: 57020
SAMPLE # 5

ESTD	RT	AREA	TYPE	CAL	AMOUNT
	5.10	1540	PB	1R	1.281