Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Maleic Hydrazide

MRID #: 447125-01

Matrix: Soil

Analysis: HPLC/ELCD

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STUDY TITLE

Analytical Method for Maleic Hydrazide in soil

Data Requirement

Not Applicable

Author

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Study Completion Date

October 19, 1998

Performing Laboratory

Uniroyal Chemical Co.
Middlebury, CT 06749

Laboratory Project Identification

Analytical Method AC 6001
Uniroyal Project No. 98241

Related Reports

Uniroyal Research Project	MIRD
9366	42736901
9367	42744801
9354	42693301

Key Words: Analytical method, MH, Maleic Hydrazide, soil

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

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Sponsor: Uniroyal Chemical Company, Inc.

Company Agent: _____ 11-17-98
Signature Date

Willard F. Cummings

U.S. Registration Manager

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STATEMENT OF ADHERENCE TO GLP's

This submission is not considered a "study" as defined by 40CFR 160 and as such falls outside the scope of GLP requirements. It consists of an analytical method which has been compiled and reformatted to conform more closely with data reporting guideline # 850.7100 (draft) and EU guidelines under commission directive 96/46/EC of 16 July 1996. Information for this report was taken from previously submitted GLP studies as indicated on the title page.

Certification

This analytical method was compiled from information in the following reports:

1) Uniroyal project 9366
2) Uniroyal project 9367
3) Uniroyal project 9354

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Date: October 19, 1998

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SUMMARY

Maleic Hydrazide (MH) is extracted from soil using methanol:water (50:50 v/v) as extractant. The extract is analyzed by HPLC using a reverse phase column and electrochemical detection. The analytical method compiled here is taken mainly from Uniroyal Chemical report 9366.

A) MATERIALS

A.1 Equipment

HPLC vials with teflon lined caps.

Centrifuge

Solvent filtration apparatus with 0.2 µm filters to filter and degass mobile phase.

Screw capped glass jars (ca 100 ml capacity).

Sieve for soil preparation (2-3 mm)

Analytical balances, both top pan and 5-figure precision.

Solvent filters (0.45 µm) for extract filtration prior to chromatography.

Orbital shaker capable of shaking the screw capped glass jars.

A.2 Reagents/Supplies

1. Maleic Hydrazide (3,6-Dihydroxypyridazine; 1,2-Dihydro-3,6-Pyridazinedione).

2. Deionised water
3. Methanol ('HPLC' grade)
4. Potassium Hydroxide (AnalaR grade)
5. Formic Acid (AnalaR grade; 90%)
6. Ammonia Solution (AnalaR grade; 35%. 0.88 $g.ml^{-1}$)

Reagents (5) and (6) are used to prepare buffer:

Mobile Phase Buffer (0.05 M Ammonium Formate)

2.2 g formic acid (90%) is dissolved in ca 900 ml of deionised water. The pH is adjusted to 3.2 with ammonium hydroxide solution and the volume diluted to 1 litre.

A.3 Analytical Standards

Maleic hydrazide standard can be obtained from Uniroyal Chemical Inc. division of Crompton & Knowles. A typical COA for maleic hydrazide and its MSDS sheet is found in appendix 1.

B. SAFETY AND HEALTH

This method should be performed by trained chemical personnel. Hazards associated with the use of maleic hydrazide are shown in the MSDS sheet in Appendix 1. Analysts should refer to the MSDS sheets for the other reagents listed in section A.2.

C. ANALYTICAL METHOD

C.1 Principle of the Method

Soil samples are sieved and then extracted with methanol/water (50:50 v/v) for at least 15 hours. The solvent phase is analyzed by HPLC using an electrochemical detector.

C.2 Types of Soils

This method is predicted to be applicable to most soil types. In Uniroyal Chemical Inc. project 9366 the soil was from a potato growing area in Washington state USA. Samples of this soil obtained from various depths 0-30 cm, 30-60 cm, 60-90 cm and 90-120 cm were all classified as sandy loam. In Uniroyal Chemical Inc. project 9354 the soil was from a tobacco growing area in North Carolina USA. Samples of the soil obtained from depths of 0-30 cm, 30-60 cm and 60-90 cm were all classified as loam. Soil from the 90-120 cm depth was classified as sandy clay loam. In Uniroyal Chemical Inc project 9367 the soil was from a turf growing area in California USA. Samples of soil obtained from 0-30 cm and 30-60 cm depths were classified as sandy loam while those obtained from 60-90 cm and 90-120 cm depths were classified as sand. The analytical method described here worked equally well on all these soil types.

C.3 Sample Processing

Soil samples obtained from a typical study (like Uniroyal study 9366) were preprepared before being sent to the analytical laboratory. The cores were normally frozen in dry ice chests immediately after being taken. Upon being

received at the preparation facility the samples were stored in a freezer at -50°C to -25°C. The samples were prepared by allowing the soil to warm only until it was warm enough to break up the frozen soil. They were then sieved through a No. 3 ½ sieve (5.6 mm ± 0.2 mm opening) to remove pebbles and organic debris such as twigs and leaves. Following this, the sieved soil was mixed homogeneously in a Hobart brand food chopper. The sample was then frozen and sent to the analytical laboratory. At the analytical laboratory the frozen sample was thawed and sieved once more before being analyzed.

C.4 Extraction Method

Soil samples (25g) are extracted by adding 25 ml of methanol:water (50:50 v/v) and shaking for a minimum of 15 h (ie overnight). Following extraction, the samples are allowed to settle, a portion of the supernatant is centrifuged at an appropriate speed and time to separate the extraction solvent from the soil particles, and filtered (0.45 µm filter) prior to injection onto the HPLC.

During Method Establishment 25 ml extraction solvent was added to each standard, blank or Q.C. sample. Subsequent analysis during method validation and routine analysis should ensure that the final volume in each blank, standard, QC and test sample during extraction is identical.

C.5 Chromatography Method

C.5.1 HPLC Method

The chromatographic system used in this method consisted of a Waters Model 712 W1SP autosampler, Waters Model 600E quaternary solvent

delivery system and an ESA Coulochem 5100A Electrochemical Detector. The chromatographic conditions employed were as detailed below. These conditions may be altered when using alternative instrumentation or columns to provide adequate resolution and sensitivity. In particular voltammograms must be produced for each new analytical cell employed and also the voltammograms currently being employed with the analytical cell should be confirmed periodically. Instrumentation employed must be shown to the precise with respect to injection of sample onto the column since no internal standard is employed in the assay. An ESA Coulochem II electrochemical detector is also satisfactory for the analysis. If this instrument is employed then appropriate range settings must be established to relate to the quantity of test material being injected on column.

Analytical column:	Partisil ODS Cartridge, 5 µm particle size, 250 x 4.6 mm i.d.
Guard column:	Partisil ODS Cartridge, 5 or 10 µm particle size, 10 x 4.6 mm i.d.
Filter:	A 0.45 µm inlet filter should be employed between the injector and guard column.
Mobile phase:	0.05M ammonium formate, pH 3.2 (2.2 g formic acid (90%) prepared in deionised water, pH adjusted to 3.2 with ammonium hydroxide and volume diluted to 1 litre)
Flow rate:	1.3 $ml.min^{-1}$

Injection volume: 20 µl

Column temperature: Ambient

Detection: Coulochem 5100A Electrochemical Detector (see note above) equipped with a Model 5020 guard cell and a Model 5011 analytical cell. (Model 5010 also suitable)

Detector Potentials: Guard Cell: +1.0 V
Screen Cell: +0.60 V
Analytical Cell: +0.85 V

Note that potentials employed during this study were for a Model 5011 analytical cell (Serial No. 3253HL). Potentials must be established for each analytical cell.

Data handling: Trivector Trilab 3000 or a Trivector Trio data station coupled to a thermal printer.

Run time: ca 15-20 minutes for retained peak to be eluted during subsequent sample

C.5.2 Selection of Conditions for Electrochemical Detection

The oxidation potential applied to the electrochemical analytical cell must be selected by reference to a voltammogram relating to the conditions of the analytical cell at the time of analysis and using the

chromatographic conditions described in section C.5.1.

A representative voltammogram for Maleic Hydrazide is shown in Figure 1. The cell conditions producing this voltammogram would suggest that an oxidation potential of +0.85 V would be appropriate to quantify Maleic Hydrazide and the screening electrochemical cell would be set at +0.6 V. The guard cell being set at a potential slightly above the voltage applied to the analytical cell, say +1.0 V.

C.5.3 Chromatographic Conditions

The chromatographic conditions are as described under C.5.1. Representative chromatograms are shown in Figures 2, 3 and 4. It should be noted that the upper limit of linearity of detector response should be known prior to chromatographing the extracted samples. An appropriate detection range and injection volume should be selected to chromatograph the samples in order to inject a suitable volume of extract for each sample to elicit a response within the linear working range of the detector (under the detector range setting used). This linear range may also vary from day to day due to inherent properties of the electrochemical detector.

A representative linear range of Detection can be seen in Figure 5. Under these conditions the limit of detection was ca 70 picograms and the upper standard injected was ca 110,000 picograms (cell Model 5011, Serial No. 3253HL).

C.6 Preparation of Spiking and Standard Solutions

C.6.1 Preparation of Standard Solutions and Soil Samples

Procedural standardization methodology is employed. That is, a series of soil samples are spiked with standard amounts of MH and are then extracted as per the method shown in C.4.

Accurately weigh by difference ca 20-50 mg Maleic Hydrazide into a 100.0 ml flask, add one pellet of potassium hydroxide and ca 80 ml deionised water. Sonicate for 5-10 min, if necessary, allow to cool and make to volume with deionised water. A series of dilutions of this stock standard are prepared in methanol:water (50:50 v/v) in order to produce appropriate concentrations for spiking soil samples with ca 1-2 ml of standard solution. The spiked soil standard curve should have a limit of reliable determination ie lowest standard of 10 p.p.b. (10 $ng.g.^{-1}$ soil) up to the highest standard concentration appropriate for the assay (ca 1000-5000 p.p.b).

C.6.2 Preparation of Quality Control Solutions and Soil Samples

Accurately weigh by difference ca 20-50 mg Maleic Hydrazide into a 100.0 ml flask, add one pellet of potassium hydroxide and ca 80 ml deionised water. Sonicate for 5-10 minutes, allow to cool and make to volume with deionised water. A series of dilutions of this stock quality control solution are prepared in methanol:water (50:50, v/v) in order to produce appropriate concentrations for spiking soil samples with ca 1-2 ml of quality control solution. Quality control soil spike concentrations should be selected with reference to the standard curve range.

C.7 Extraction Efficiency

C.7.1 Determination of Optimum Period of Extraction

A series of 6 sieved soil samples weighing 25.0 g were weighed into screw-capped glass jars. The soil which was a bulk sample from the turf site of IRI Project No. 352866 (Uniroyal Chemical Project 9367) had previously been sterilised by autoclaving for 2 h. Each soil sample was spiked with 1 ml of a solution of [^{14}C]-Maleic Hydrazide containing ca 2.0×10^8 d.p.m. and 179.5 µg Maleic Hydrazide resulting in a soil concentration of 7.18 p.p.m. Each soil sample was thoroughly shaken to disperse the sample as evenly as possible.

The soil samples were then stored for 2 days at ambient room temperature in the dark. After 2 days storage the samples were extracted by orbital shaking for variable periods of time using 25 ml methanol: water (50:50, v/v) as solvent. Two samples were shaken for 1 h, 2 for 3 h and the last 2 for 6 h. Following extraction each sample was centrifuged at 3000 r.p.m. for 10 min and 1 ml of the supernatant was counted in a liquid scintillation counter preset to count [^{14}C] using 10 ml of Quickszint 1 (Zinsser Analytic, Maidenhead) as scintillant. 2 ml of the spiking solution was diluted to 50 ml and a 1 ml portion of the resultant dilution was counted for radioactivity to represent 100% recovery of test material.

Table 1 tabulates the data obtained after extracting spiked soil extracts for Maleic Hydrazide during a varying period of time. The results indicate that the extraction must be performed for a period of between 3 and 6 h in order to obtain satisfactory recovery.

The method described here recommends 15 hrs of extraction because it is convenient to prepare samples during the day and let them extract overnight

C.7.2 Recovery from Soil After Storage

The recovery of Maleic Hydrazide from soil after storage was assessed by spiking a series of seived soil samples, with [^{14}C] labelled test compound. Four soil samples weighing 25.0 g were weighed into screw capped glass jars. The soil which was a bulk sample from the turf site of IRI Project No. 352866 (Uniroyal Project 9367) had previously been sterilised by autoclaving for 2 h.

Each soil sample was spiked with 1 ml of a solution of [^{14}C] Maleic Hydrazide containing ca 1.7 x 10^8 d.p.m. and 154 µg Maleic Hydrazide, resulting in a soil concentration of 6.16 p.p.m. Each soil sample was thoroughly shaken to disperse the sample as evenly as possible. Two spiked soil samples were sealed and stored at ambient room temperature in the dark to be analysed after 8 days storage. The remaining 2 jars were extracted immediately as follows:

Following the addition of 25 ml water:methanol (50:50, v/v) each jar was shaken for 90 minutes at room temperature on an orbital shaker. The samples were then centrifuged at 300 r.p.m. for 10 min and 1 ml of the supernatant counted in a liquid scintillation counter preset to count [^{14}C] using 10 ml of Quickszint 1 (Zinsser Analytic, Maidenhead) as scintillant. 2 ml of the spiking solution was diluted to 50 ml and a 1 ml portion of

the resultant dilution was counted for radioactivity to represent 100% recovery of test material. The 2 samples stored at room temperature in the dark were extracted and analysed for Maleic Hydrazide after 8 days storage in an identical manner to that described above, except that the extraction period was lengthened to 6 h.

The recovery of Maleic Hydrazide from soil which was spiked with test compound 8 days prior is shown in Table 2. Approximately 89% of the Maleic Hydrazide is recovered after an extraction period of 6 h.

C.8 Fortifications

Soil samples from the untreated control plot, spiked in the field, accompanied each set of field samples analyzed in Uniroyal Chemical report 9366. These spiked samples were transported and stored along with (and hence under the same conditions as) the field samples. These QC spikes were then analyzed along with the field samples to ensure that the methodology provided reliable results during the course of the study. The QC spikes recovery data at various spike levels is summarized in Table X of Unroyal Chemical report 9366 and this table has also been included in this report as Table 3. Recoveries at the spiking levels of 2.0 µg/g, 0.2 µg/g and 0.05 µg/g were all in the range of 89.3% to 120.1%.

D. INSTRUMENTATION

The instrumentation used is described below:

Liquid Chromatograph with a passivated pump (passivated with nitric acid)

ESA Coulochem Model 5100A or Coulochem II Electrochemical Detector fitted with a Model 5020 guard cell and a Model 5010 or Model 5011 Analytical cell.

Analytical Column: Partisil ODS cartridge, 5 μm particle size 250 x 4.6 mm i.d.

Guard column: Partisil ODS cartridge, 5 or 10 μm particle size, 10 x 4.6 mm i.d.

HPLC Inlet Filter: A 0.45 μm inlet filter to be placed between the injector and guard column.

E. SAMPLE BRACKETING

The calibration was done for each set of samples by standard bracketing. A typical run involved running the standard curve, a control containing no MH, 6 spikes to check recovery (two each at 2040 ppb, 204 ppb and 51.0 ppb), and finally the actual soil samples. Data from a typical run done for Uniroyal Report 9366 is shown in appendix 2 of this report (Appendix 7 of Uniroyal report 9366). Typical chromatograms generated from a standard curve and for some soil samples are also shown in Appendix 3 (appendix 9 of Uniroyal report 9366).

F. POTENTIAL INTERFERENCES

This method could have interferences from other oxidizable compounds which chromatograph with similar retention times. The potentials applied to the guard column, the screening cell, and the analytical cell were choosen to give good detector response for MH. These potentials were determined as follows:

Solutions containing ca 8, 16 and 79 ng Maleic Hydrazide per 20 µl to be injected onto the HPLC were prepared in deionised water which had been made alkaline with potassium hydroxide (see Appendix 1, Section VI). These solutions were injected onto the HPLC repeatedly with the potential applied across the working electrode surface of the electrochemical cell being varied between injections. The Maleic Hydrazide response at the electrode was recorded during each injection. The following potentials were applied to the ESA guard and analytical cells:

Guard cell:	+1.0 V
Cell 2:	0 V
Cell 1:	0.55 V → 0.90 V altering by 0.05 V increments; the output from this cell was recorded.

The background current at each applied potential was monitored.

Table 4 tabulates the data showing the relationship between potential applied to the electrochemical analytical cell and the Maleic Hydrazide response. The responses, shown graphically in Figure 6, demonstrate that a potential of +0.85 V is appropriate for the analysis of Maleic Hydrazide using the analytical cell under investigation and employing the mobile phase described in Section C.5.1. An appropriate potential at which to set the screen cell of the electrochemical detector is +0.6 V. The guard cell should then be set at a potential slightly greater than that applied at the analytical cell, say +1.0 V.

It should be noted that these selected potentials relate to this specific cell under the described conditions at that point in time. The conditions of the analytical

cell will change and may result in alterations being observed in the voltammogram.

Blank soil types should be run under the conditions for maximum detection of MH to ensure that no interferences are present.

G. CONFIRMATORY TECHNIQUES

No confirmatory techniques were used in this study.

H. TIME REQUIRED FOR ANALYSIS

In Uniroyal report 9366 one run involved preparing 8 standards for linearity, 6 spikes and 35 soil samples. This total of 49 samples could be prepared in one eight hour day and extracted overnight (15 hours). In the second 8 hour day the extracts could be worked up to prepare for HPLC analysis which could be run overnight. Hence the total time for analysis would be two days.

I. MODIFICATION OR POTENTIAL PROBLEMS

None.

J. CALCULATIONS

Weighted linear regression analysis is performed on a plot of peak height versus Maleic Hydrazide concentration in each standard. The concentration of Maleic Hydrazide in each quality control sample and test sample is then computed by linear interpolation from this line. A representative calibration line can be seen in Appendix 2.

K. COPIES OF CHROMATOGRAMS

Copies of chromatograms for a control sample, and spiked samples at 10.6, 21.1, 52.8, 106, 211, 528, 1055, and 2532 ppb of MH in soil are shown in Appendix 3. Also shown are several chromatograms of soil samples from the field dissipation study in Uniroyal Report 9366. In these samples MH occurs at levels of <10 ppb, 19 ppb and 148 ppb.

L. METHOD VALIDATION

L.1 Accuracy (USA) / Recovery (EU)

A study of the accuracy of the MH determination using this method was done as part of Uniroyal study 9366 and is reported in attachment 5 table 9 of that report. This table is also included as Table 3 of this report. The numbers in Table 3 have been reformatted for the purpose of this report to clearly show the average, standard deviation, relative standard deviation, range and 95% confidence levels at each spiking level as required by EPA guidance. These results are shown in Table 5 of this report. Table 5 summarizes recoveries, standard deviations (SD), relative standard deviations (RSD), the range of recoveries, and the ± confidence limits for 95% confidence for the four spiking levels of 9.9 to 10.5; 19.8 to 20.9; 41.8 to 49.6; and 836 to 992 ng of MH per gram of soil. The data in Table 5 show that average recoveries at each level are all between 70 and 110% as required by the EU (70 – 120% as required by the USA).

In Table 5 the RSD was calculated as:

$$RSD = \frac{SD}{Average} \times 100\%$$

The 95% confidence limits (CL) were calculated as:

$$CL = \frac{t \times SD}{\sqrt{n}}$$

Where SD = standard deviation

n = the number of observations

t = the value t for n-1 degrees of freedom at 95% confidence as taken from table C.3 page 267 of Quality Assurance of Chemical Measurements, John K. Taylor, Lewis Publishers Inc. 1987.

L.2 Precision

The USA requires a calculation of the relative standard deviation of recoveries (RSD's) at various concentration levels. These RSD's are shown in Table 5 and are less or equal to 20% as required by EPA.

The EU requires a repeatability study where the same sample is used at least 5 times on the same instrument with the same operator within a short time interval. Unfortunately one set of analyses was done on March 11, 1992 approximately 4 months after most of the analyses were performed (November 6, 1991). Table 6 shows only the analyses done on November 6, 1991. Although only 3 or 4 rather than the required 5 repeats were done the RSD's are well below the 20% criteria. Additionally the 95% confidence levels including all the data (November 6, 1991 and March 11, 1992) at each level indicate that

the method has good repeatability.

The systems or instruments precision was also evaluated in report 9366 by replicate injections of a solution of Maleic Hydrazide prepared in methanol:water (50:50, v/v). The solution contained 13775 pg in a volume of 20 µl which was injected onto the HPLC column. System Precision was assessed by calculation of the coefficient of variation of the peak heights for all the injections.

The results are shown below. The coefficient of variation for the peak height on repeated injection of Maleic Hydrazide was 0.4% at 13775 pg on column.

Establishment of System Precision

Quantity of Maleic Hydrazide Injected onto Column (Picograms)	Peak Height Maleic Hydrazide
13775	65970.54
	66115.31
	66231.31
	66330.34
	66465.64
	66647.39
	66644.02
	66502.05
	66691.35
	66459.17
Mean Peak Height	66406
Coefficient of Variation	0.4%

L.3 Limit of Quantitation (USA) / Limit of Determination (EU)

The lowest concentration tested as shown in Table 5 was approximately 10 ng/g of soil. At this level the mean recovery for MH was between 70 and 110% and the relative standard deviation was equal or less than 20%. Thus the limit of quantitation (LOQ) is 10 ng/g.

L.4 Limit of Detection

No statistical estimate of the limit of detection (LOD) was made from the data in Table 5. However if we assume that the LOD is roughly one-third of the LOQ the LOD would be about 3 ng/g (3 ppb). In this connection the chromatographic traces for a blank soil extract and the spiked extract at 10.6 ppb and 21.1 ppb can be considered (Appendix 3).

L.5 Specificity

This is an HPLC method with electrochemical detection and as demonstrated by typical chromatograms of the spikes (see Appendix 3) there is excellent separation of the MH from other interfering peaks. In the soils tested there were no interfering compounds. However it is recommended that confirmatory identification of the MH peaks be occasionally carried out if one suspects an interfering peak may be present.

L.6 Ruggedness

No ruggedness testing was done but the HPLC with electrochemical detection is generally considered a reliable method.

L.7 Limitations

None are known.

L.8 Independent Laboratory Validation (ILV) (USA)/Reproducability (EU)

Reproducability (EU) is defined as an independent lab validation. Reproducability is not required for soil samples according to EU directive 91/414/EEC, July 16, 1996. An ILV is suggested by the USA EPA. This has not been done in a formal sense. However several field dissipation studies for MH in different USA locations have been done at various times (see reports 9366, 9367 and 9354). Although the same laboratory analyzed the samples, the fact that these analyses were done successfully over a number of years suggests that the method in this report can be considered as having been independently lab validated.

M. CONCLUSIONS

The analytical method AC-6001 described in this report is applicable to the analysis of MH in a variety of soil types. The LOD is about 3 ng/g of soil the LOQ is 10 ng/g of soil. Recoveries and relative standard deviations are excellent and well within the regulatory guidelines of both the EPA and EU.

FIGURES

Voltammogram Relating to the Electrochemical Oxidation of Maleic Hydrazide

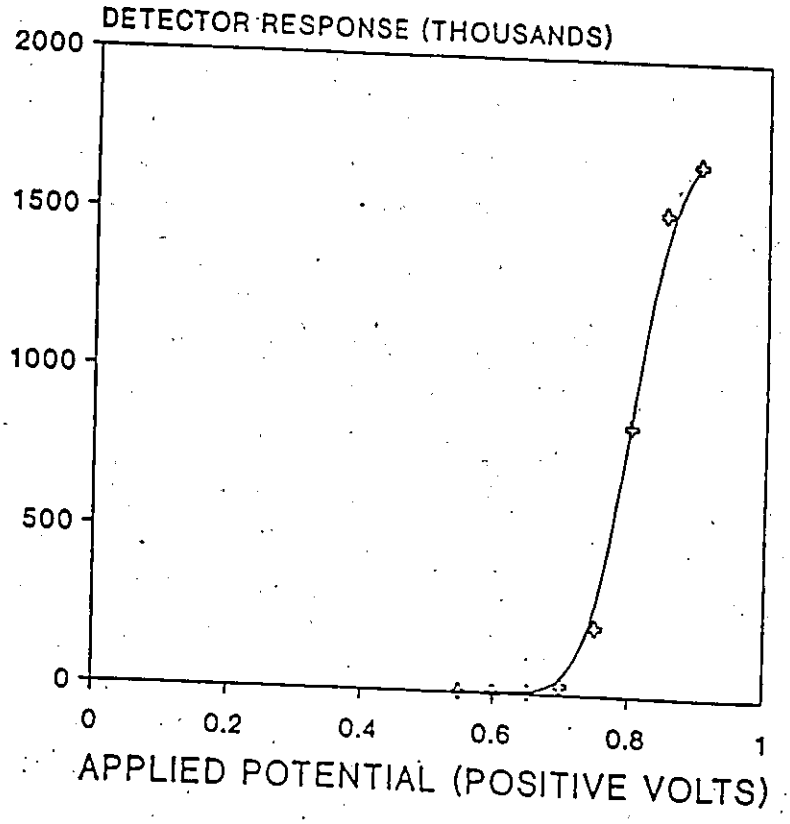
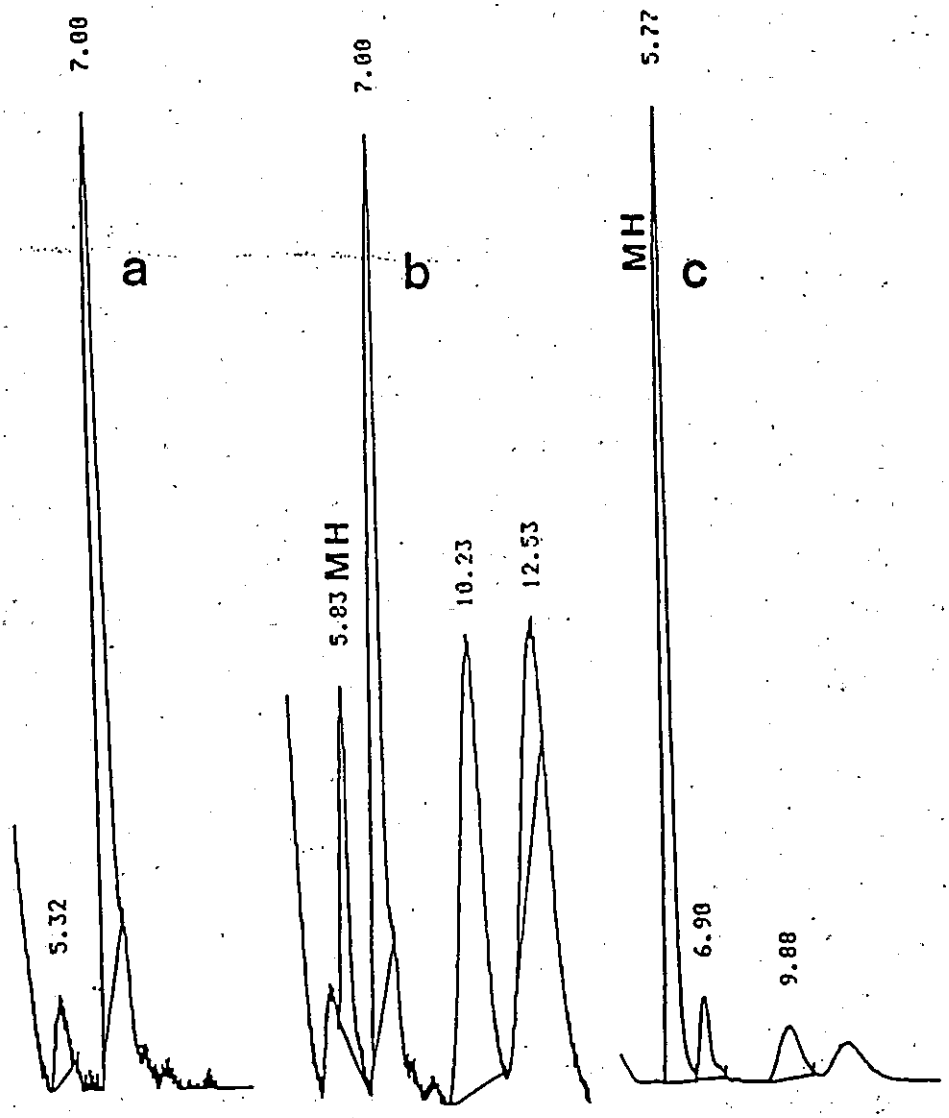


Figure 1

Representative Chromatograms Obtained from Extracts of Potato Soil Spiked with
(a) 0 p.p.b. Maleic Hydrazide
(b) 10.25 ng Maleic Hydrazide g^{-1} soil (p.p.b.) and
(c) 410 ng Maleic Hydrazide g^{-1} soil (p.p.b.)

Chromatographic Conditions as Described in Text



IRI 352007

Figure 2

Representative Chromatograms Obtained from Extracts of Turf Soil Spiked with
(a) 0 p.p.b. Maleic Hydrazide
(b) 9.94 ng Maleic Hydrazide g^{-1} soil (p.p.b.) and
(c) 497 ng Maleic Hydrazide g^{-1} soil (p.p.b.)

Chromatographic Conditions as Described in Text

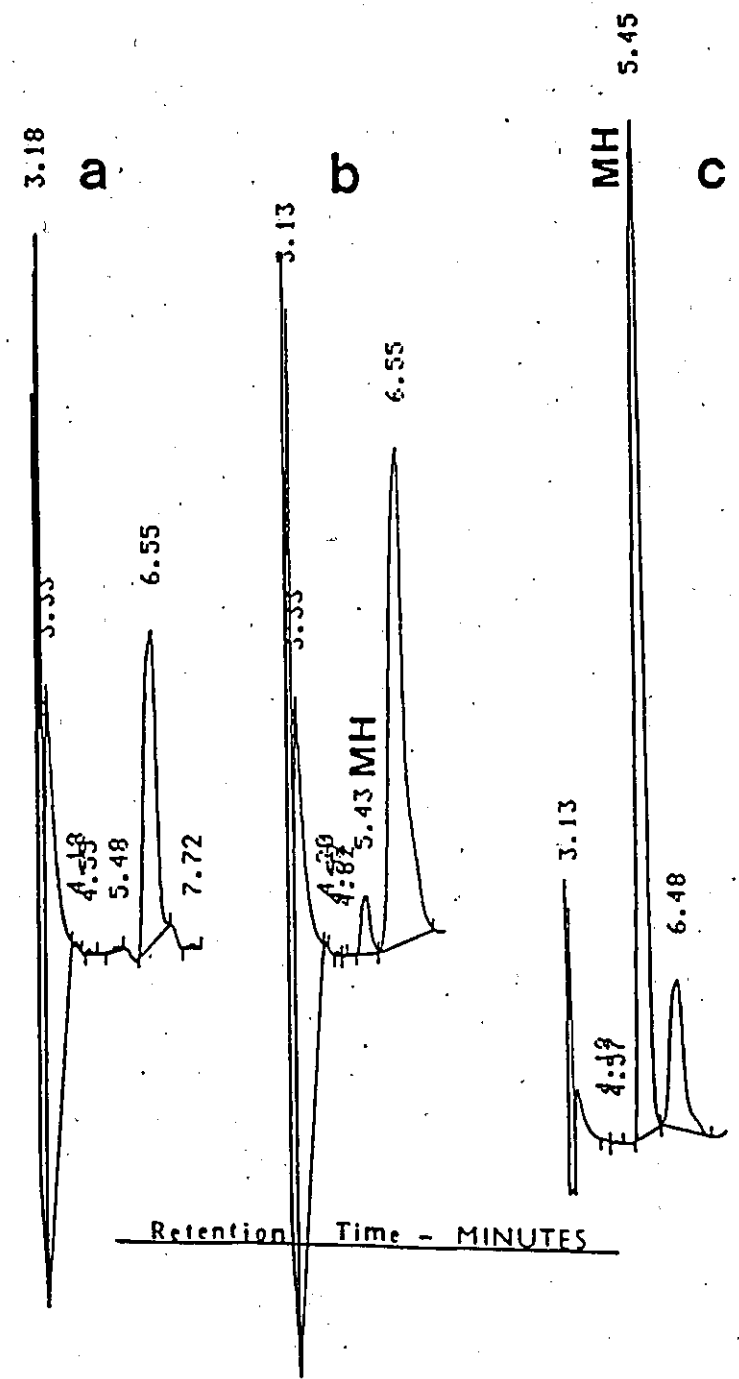


Figure 3

IRI 352007

Representative Chromatograms Obtained from Extracts of Tobacco Soil Spiked with
(a) 0 p.p.b. Maleic Hydrazide
(b) 9.94 ng Maleic Hydrazide g^{-1} soil (p.p.b.) and
(c) 557 ng Maleic Hydrazide g^{-1} soil (p.p.b.)

Chromatographic Conditions as Described in Text

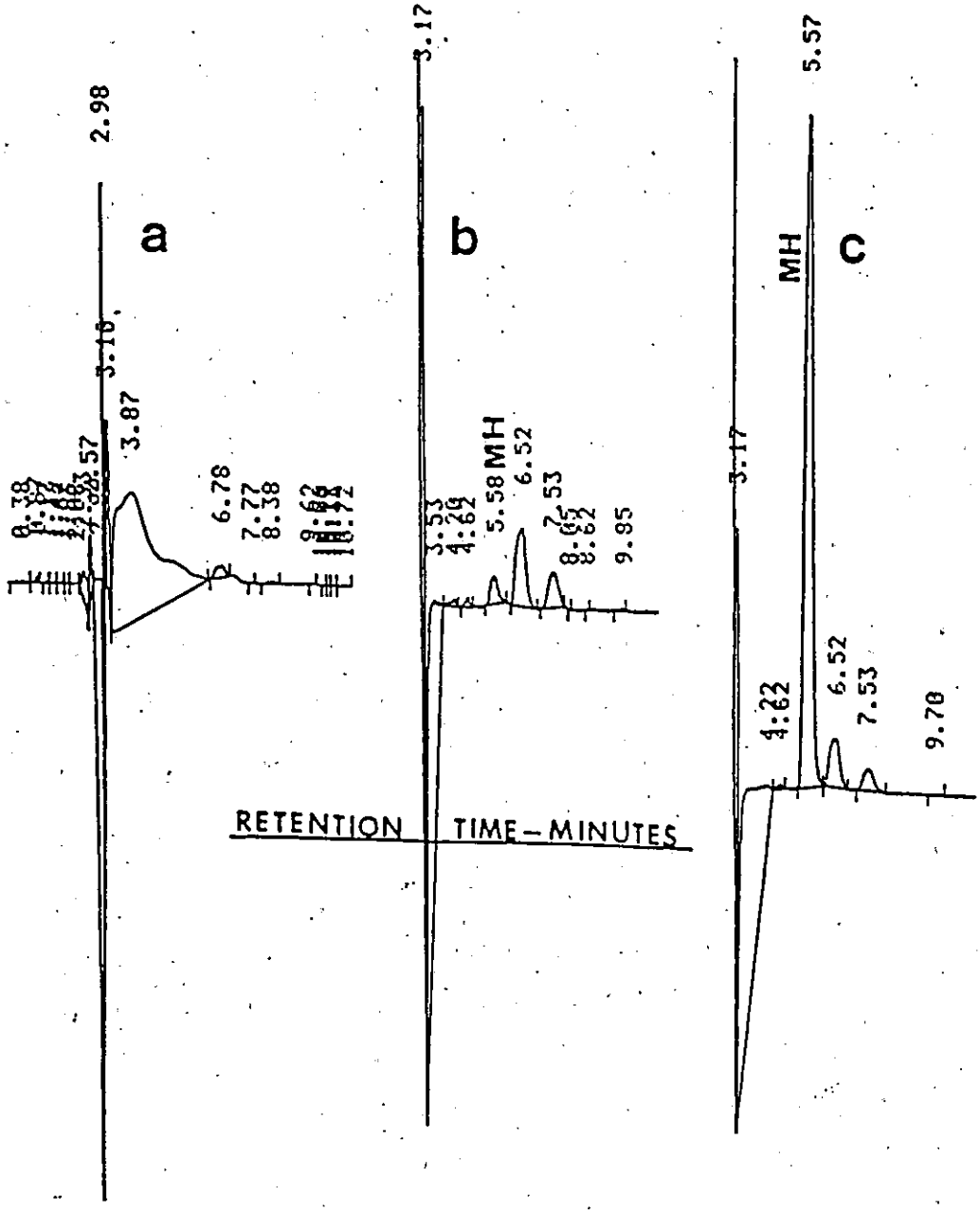


Figure 4

APPENDIX 1 (continued)

Linearity of Detector Response to Maleic Hydrazide Over the Range ca 70-110200 Picograms

Chromatographic Conditions as Described in Text

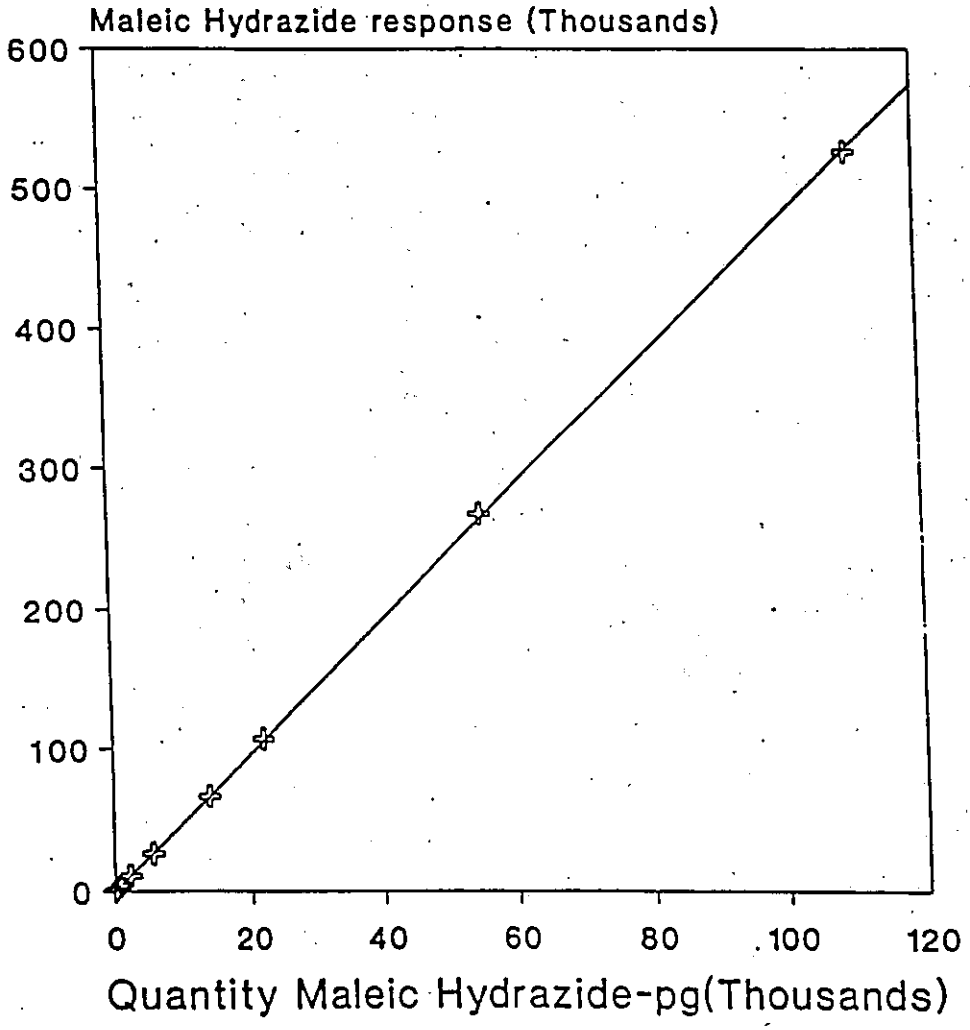
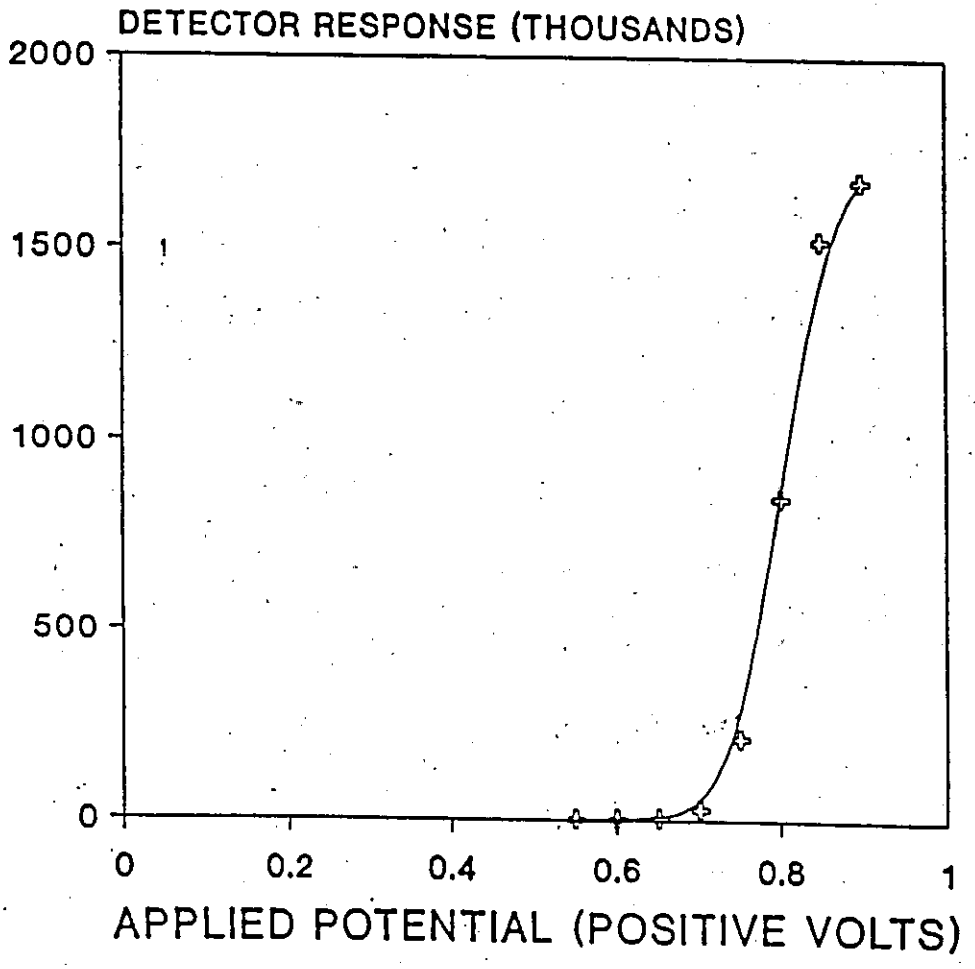


Figure 5

Voltammogram Relating to the Electrochemical Oxidation of Maleic Hydrazide



Note: Analytical Cell Model No. 5011 (Serial No: 3253HL)
HPLC conditions as Appendix 1, Section IV
Date of Voltammogram: 10 July 1991

Figure 6

TABLES

Recovery of Maleic Hydrazide from Soil After Extraction for 1-6 h

Sample Description	d.p.m. of 1 ml Sample	Percentage Recovery
100% Recovery	6711221	-
Soil 1 - Extracted 1 h	5892781	87.8
Soil 2 - Extracted 1 h	5815770	86.7
Soil 3 - Extracted 3 h	5696847	84.9
Soil 4 - Extracted 3 h	6227734	92.8
Soil 5 - Extracted 6 h	6679382	99.5
Soil 6 - Extracted 6 h	6721977	100.2

Table 1

Recovery of Maleic Hydrazide from Soil After Storage

Sample Description	d.p.m. of 1 ml Sample	Percentage Recovery
Day 0		
100% Recovery	6218566	-
Soil 1 - Day 0	6175378	99.3
Soil 2 - Day 0	6275664	100.9
Day 8		
100% Recovery	6205483	-
Soil 1 - Day 8	5467304	88.1
Soil 2 - Day 8	5601871	90.3

Table 2