Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Daminozide

MRID #: 447838-01

Matrix: Soil

Analysis: GC/NPD

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Uniroyal Chemical Company,Inc.
Uniroyal Project No.:99001
Analytical Method No.: AC-6004

STUDY TITLE

Analytical Method for Daminozide and its Metabolites as UDMH in Soil

Data Requirement

Not Applicable

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Uniroyal Project No.'s: 85124 and 8647

Compiled by:

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Key Words: *Analytical method, Daminozide, Cyclic Daminozide, UDMH, Soil*

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COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

This submission is not considered a "study" as defined by 40 CFR 160 and as such falls outside the scope of GLP requirements. It consists of an analytical method that has been compiled and reformatted to conform more closely to data reporting guidelines # 850.7100 (draft) and EU guidelines under commission directive 94/46/EC of 16 July 1996. Information for this report was taken from previously submitted studies (Project No.: 85124, 8647) conducted prior to the implementation of GLPs'.

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CERTIFICATE OF AUTHENTICITY

This analytical method (AC-6004) report was compiled from information in the following reports:

1. Uniroyal Project 85124
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SUMMARY

The analytical method AC-6004 described in this report is applicable to the analysis of daminozide and UDMH in a variety of soil types. For daminozide, the LOD is ~0.3 ppm and the LOQ is 1 ppm. For UDMH, the LOD is ~0.1 ppm and the LOQ is 0.4 ppm.. Recovery of daminozide from the 0-12 inch (0-6 and 6-12 inch) soil depths, averaged 54.03% with a RSD of 24.78%. Although the recovery and RSD are outside the limits required by the EU and the EPA, these results are considered to be about the best that can be done because of the inherent instability of daminozide in soil. Recovery of UDMH averaged 120.65% at 0.4 ppm fortification level and 110.73% at the 2.0 ppm fortification level. The recovery values at both the fortification levels for UDMH are between the 70-120% as required by the EPA. However, at the 0.4 ppm fortification level, the recovery was slightly higher than that allowed (70-110%) by the EU. The RSD for UDMH on the other hand is <20% and satisfies both EU and EPA requirements at both the fortification levels.

Since daminozide degrades very rapidly in soil (half-life <1 day), the low and variable recovery of daminozide from soil samples could be partly due to this rapid degradation. The recovery of UDMH, the major degradate of daminozide from field dissipation studies was however good and therefore the method should be acceptable.

1.0 INTRODUCTION

Two analytical methods were used for analysis of daminozide and its residues in soil; one for determining daminozide following derivatization to cyclic daminozide and the other for determining UDMH (unsymmetrical dimethyl hydrazine) residues. A GC equipped with a nitrogen-phosphorus detector was used for determining cyclic daminozide following derivatization of daminozide with acetic anhydride to form cyclic daminozide. The analytical method for UDMH involves distilling UDMH from soil followed by derivatization and analysis by GC/MS. These methods were developed in 1985-1986, prior to the implementation of the GLPs. The analytical method was taken from the soil field dissipation study and therefore does not strictly follow the EU or EPA requirements for analytical methods.

The objective of this report is to provide analytical method(s) for determining daminozide and its residues in soil and address the method specificity, linearity, precision (repeatability) and accuracy/recovery

2.0 MATERIALS AND METHODS

2.1 CHEMICALS

Structure of Daminozide, cyclic Daminozide and UDMH are provided in Figure 1.

2.1.1 *Test Substance*

The test substance is Daminozide (succinic acid-2,2-dimethylhydrazide).

2.1.2 Solvents and Reagents

- Daminozide analytical standard
- Acetic anhydride
- Sodium bicarbonate
- Hydrochloric acid
- Dichloromethane
- Anhydrous sodium sulfate

2.2 SAFETY AND HEALTH

This method should be performed by trained chemical personnel. Hazards associated with the chemicals used in this analytical method are shown in the MSDS sheets in Appendix 1.

2.3 SUPPLIES

- Gas Chromatograph with Mass Selective Detector
- Module heater
- Circulating bath, Refrigerated
- Grinder
- Vortex junior mixer
- Magnetic stirrer and Hot plate with ceramic Top
- Lab-Jack
- Condenser, Liebig 14/20 JTS
- Distillation flasks
- Adapter, Enlarging
- Adapter, 75 ° Bend
- Adapter, 105 ° Bend
- Receiving Tube
- Syringe
- Vials
- Volumetric pipets
- Volumetric flasks
- Graduated Cylinders
- Stoppers-Glass
- Magnetic stirring bars
- Rubber tubing
- Sodium hydroxide, 50% Aqueous
- Titanium Trichloride, 20% Aqueous
- Calcium Chloride
- Daminozide (Succinic acid 2,2-dimethylhydrazide)
- Salicylaldehyde
- Sodium chloride
- Methylene Chloride

2.4 TEST MATRIX

2.4.1 *Soil*

This method should be applicable to most soil types. In Uniroyal Chemical Inc., Project 8647, soil obtained from Uniroyal Crop Protection field station, Bethany, CT was used. The soil was characterized as Fine sandy loam soil (Spodsol to Alfisol/gray-brown Podzolic soil). The soil characterization is as follows:

pH	Sand	Silt	Clay	Organic Matter
6.1	54%	34%	10%	3.3%

2.4.2 *Sampling*

Samples were taken from three random locations in the plot. Soil around the immediate sampling site was pushed away using a shovel and an 8x8x6 inch sample was taken for the 0-6 inch level. A 6-12 inch sample was then taken using a shovel from the same hole. Each soil sample was mixed thoroughly on a plastic sheet.

2.4.3 *Soil Fortification*

The following soil samples were spiked with daminozide:

Soil Depth	Amount of Soil	Fortification level (ppm)	Residue Analyzed	Analytical Method
0-6 inch	20 g	1 ppm	Cyclic daminozide	GC-NPD
6-12 inch	20 g	1 ppm	Cyclic daminozide	GC-NPD
0-6 inch	50 g	0.4 ppm	UDMH Residues	GC/Mass Spec.
6-12 inch	50 g	0.4 ppm	UDMH Residues	GC/Mass Spec.
0-6 inch	10 g	2 ppm	UDMH Residues	GC/Mass Spec.
6-12 inch	10 g	2 ppm	UDMH Residues	GC/Mass Spec.

2.4.4 *Soil Extraction and Analysis*

The laboratory spiked soil samples along with the field treated soil samples were extracted as described in section 2.6 and analyzed as described in section 2.5.

2.5 EQUIPMENT

2.5.1 *Gas Chromatograph-Nitrogen Phosphorous Detector*

The following GC conditions were used for analyzing Cyclic Daminozide

Instrument:	Gas Chromatograph
Detector:	Nitogen-Phosphorus Detector (NPD)
Column:	1.8 m x 2 mm (i.d.) coiled glass column packed with 5% SP-100 (Supelco Inc.), or equivalent on Chromosorb W 100/120.
Gas Flow rate:	
Hydrogen:	3 mL/min
Air:	100 mL/min
Helium (Carrier gas):	40 mL/min
Injector Temperature:	230°C
Detector Temperature	300°C
Oven Temperature:	
Initial Temperature:	160°C for 8 min.
Final Temperature:	180°C for 7 min.

2.5.2 *Gas Chromatograph – Mass Spectrometry*

The following GC-MS conditions were used for analyzing UDMH Residues

Instrument:	Gas Chromatograph
Column:	30 m x 0.32 mm DB-5
Carrier gas:	Helium @ 4 psi head pressure
Injector Temperature:	225°C
Detector Temperature	300°C

Oven Temperature:

Initial Temperature:	145°C (no initial hold time)
Final Temperature:	190°C (no final hold time)
Rate:	4.0°Cmin.

Injection: Split mode, split ratio = 7.5:1

Injection Volume: 2μL

<u>*Mass Spectrometer Conditions:*</u>

Scan:	Multiple Interval Detection (MID)
Mode:	EI (GC)
Manifold Temp:	92°C
Ion Source Temp:	150°C
High Vacuum:	3.2 x 10^{-6} Torr
Separator Oven Temp.:	208°C
Transfer line Temp.:	213°C
Filament Current: -	0.3 mA
Electron energy:	70.0 eV.
Electron Multiplier Voltage:	-1025 V

Calibrated in EI mode using FC-43 reference standard

<u>Instrument Operation</u>

- The instrument must be tuned daily.
- Prepare data acquisition parameters to perform selected ion monitoring of m/e 44 (NC_2H_6) and m/e 164 (molecular ion of salicylaldehyde dimethylhydrazone). Quantification is performed on the m/e 164 ion chromatogram and the m/e 44 ion is used as a "confirmation" ion (to make sure the ratio of the m/e 44 to 164 is the same for the standards and the samples).
- Rapidly inject 2.0 μL of the methylene chloride extract into the chromatographic column and immediately activate the data system.
- After the 10 minute analysis time, measure the area under the peak for the hydrazone (RT ~6 min). Compare that peak area with the standard curve generated that day.

The GC/MS with a switchable EI/CI source was operated under the INCOS (ver: 4.07) operating system in the EI mode using capillary GC conditions. The instrument was tuned daily. The mass spectra were obtained using MID (Multiple Interval Detection) scanning program for mass 164 (molecular ion of salicylaldehyde dimethylhydrazone).

The raw ion chromatogram (MID RIC) for each sample was printed for the scan range of 800 to 1100. Typical scan number for the apex of desired peak was 980. The data was then enhanced using the Finnigan MSDS program "NHA" from scan 800 to 1100, covering masses 162-166 m/z, using no smoothing. Background was subtracted using the programs default parameters. The mass list for the desired peak was printed. The intensity of the 164 m/z ion from the mass list was used for quantitation purposes (corresponds to peak height).

2.6 ANALYTICAL METHODOLOGY

2.6.1 *Preparation of Calibration Standards for Standard Curve*

1. Weigh 0.1015 g of daminozide into a 100 mL volumetric flask. Dilute to the mark with distilled water. Stir for 30 minutes using a magnetic stir bar to ensure complete dissolution. This 1015 µg/mL stock solution may be kept for two days in a 4°C refrigerator.

2. Pipet 1.0 mL of the 1015 µg/mL stock solution into a 100 mL volumetric flask and dilute to the mark with distilled water. This 10.15µg/mL solution must be prepared daily.

3. Pipet 5.0 mL of the 1015 µg/mL solution into a 100 mL volumetric flask and dilute to the mark with distilled water. This 50.45µg/mL solution must be prepared daily.

4. Distill four daminozide standards ranging from 0 to 100 µg from 160 mL of 50% NaOH containing 5 mL of 20% $TiCl_3$ solution and 20 g of $CaCl_3$. Follow steps 2 to 7 of the distillation section.2.6.2.

   Note: When $TiCl_3$ is added to the flask, an exothermic reaction occurs. Wait for the bubbling to subside before spiking the daminozide standard into the flask.
   Note: The amounts selected for the standard curve should bracket the amount of daminozide that you expect in 10 to 50 g of sample.

   e.g.: $\frac{10\ \mu g\ \text{daminozide}}{10\ g\ \text{sample}} = 1\ \text{ppm}$

5. Prepare a standard curve with "peak area" on the *Y-axis* and µg of daminozide" spiked into the flask on the *X-axis*. The standard curve should be linear.

6. The standard should be distilled every day and the daily slopes recorded. Once the analyst feels comfortable that the data analysis and the distillation techniques can be reproduced in that laboratory, then single standards can be distilled daily to check the response.

2.6.2 *Distillation Procedure*

1. Add the following reagents to a 100 mL distillation flask containing a teflon stir bar:

   10 to 50 ± 0.01 g Homogeneous sample
   20 g Calcium chloride
   160 mL NaOH (50% aqueous solution)
   5 mL* Titanium Trichloride (20% solution) – reducing agent

   *Add $TiCl_3$ just before assembling distillation apparatus.

2. Place the flask on a "cold" hot plate.

   Note: A "cold" hot plate is used to prevent a loss of UDMH that may be hydrolyzed upon the first application of heat. If repetitive distillations are being performed, a second "cold" hot plate should be used in order to give the "hot" hot plate a chance to cool off. Also, ceramic hot plates cool off faster than metal hot plates.

3. Add 20µL of neat salicylaldehyde to the empty receiving tube prior to distillation.

4. Assemble the distillation apparatus using the following items:
   - Flask
   - Reducing adapter
   - Elbow – 75° bend
   - Liebig condenser – cooled to 0°C with a circulating 50/50 mixture of methanol/H_2O.
   - Elbow with vent port - 105° bend.
   - Receiving tube – 25 mL graduated tube with 14/20 joint.

   Use liberal amount of stopcock grease on all of the joints except the joint to the receiving vessel.

Recommended using clips at the ground glass joints to prevent separation and leakage during distillation.

5. Carefully heat the contents of the flask while stirring rapidly (low heat and stirring will help control the foaming).

6. After 10 mL of distillate is collected, turn off the hot plate. Remove the receiving tube, stopper and place in the 50°C module heater for 90 minutes to allow the derivatization to go to completion.

7. After the 90-minute derivatization step, the receiving tube is allowed to cool. The contents of the receiving tube are quantitatively transferred, using 2 mL of water, to a 20-mL glass scintillation vial containing 3.5 g of NaCl. The salt saturated aqueous phase is then extracted with 4.0 mL methylene chloride by placing the vial on a vortex mixer for 1 minute. The contents were mixed on a vortex mixer for 30 seconds. The phases were allowed to separate and 2.0 µL aliquots of the of the methylene chloride phase (lower layer) were injected into the GC.

   The data was acquired using the scan program noted above. The raw ion chromatogram (MID RIC) for each sample was printed for the scan range of 800 to 1100 (typical scan number for the apex of desired peak was 980). The data was then enhanced using the Finigan MSDS program "ENHA" from 800 to 1100, covering masses 162-166 m/z, using no smoothing. Background was subtracted using the program default parameters. The mass list for the desired peak was printed. The intensity of 164 m/z ion from the mass list was used for quantitation purposes (corresponds to peak height).

2.6.3 *Preparation of Derivatized Standard (Cyclic Daminozide) for Fortification*

A stock solution was prepared by weighing 0.1015 g daminozide and dissolving it in 100 mL water. Aliquots of the stock solution were diluted 1:100 and 5:100 in water to obtain 10.15 µg/mL and 50.75 µg/mL, respectively.

Two milliliters of aliquots of each of the above two dilutions were used to spike 20 g of an aqueous blank (standard curve) or control soil samples, 0-6" and 6-12" depths for fortified sample recovery.

For the standard curve, 15 mL of the 0.01N HCl was added to the standard. All of the sample was derivatized with acetic anhydride. For the soil samples, treated and spiked, 30 mL of 0.01 N HCl was added to the soil. Following a 30 minute extraction, 15 ml (one-half of the extractant volume) was derivatized and assayed. A factor of 2 was used to calculate the recovery and test sample ppm levels measured against the standard curve.

2.7 SAMPLE BRACKETING

Calibration curves using daminozide and UDMH standards were generated using a standard bracketing technique. Four concentration (0, 10.15, 20.30 and 101.5 µg) of daminozide and five different concentrations (0, 5, 10, 30 and 50 µg) of UDMH were used for generating the standard curves. A linear regression analysis was done and the correlation coefficient 'R^2' was determined. A second calibration curve was generated at day 7 (168 hours) following the first one and was used for quantification of the 7 day treated samples. The results are provided in Table II/ and Figure 5.

2.8 POTENTIAL INTERFERENCES

This method could have interferences from some soils and was observed with soil samples from different locations. In such cases one should modify the method. In the field dissipation study where the treated soil samples were extracted and analyzed concurrently with the spiked soil samples, the capillary column used was changed from the OV-1701 liquid phase to an equivalent SE-52 phase.

2.9 TIME REQUIRED FOR ANALYSIS

Following the above procedure, in one day, a reference sample (to check reproducibility) along with test samples and analytical standards for generating a calibration curve can be done.

2.10 DATA CALCULATIONS

The peak area for the sample was compared to the standard curve. The amount of daminozide (measured as cyclic daminozide) or UDMH residues in a sample was calculated using regression analysis as follows:

$$y = mx + b$$

where:

y = peak area
m = slope from standard curve
b = y-intercept
x = μg of analyte

The *concentration of the analyte* in a given sample is calculated as follows:

$$\frac{\mu g\ \text{Daminozide}}{g\ \text{Sample}} \times \frac{100}{\%\ \text{Recovery}} = \text{ppm Daminozide}$$

Based on duplicate (in most cases) injections of standards and samples, the peak area (or peak height) of the analyte of interest, was determined.

Percent Recovery (% R) is calculated as follows:

$\% R = [(C_F - C_U)/C_A] \times 100$

where:

C_F = Concentration of analyte measured in fortified sample.
C_U = Concentration of analyte measured in unfortified sample.
C_A = Concentration of analyte added to fortified sample.

2.11 METHOD TESTING

2.11.1 *Specificity*

Two different analytical methods were used for the separation of daminozide and UDMH. The GC-NPD method used for analysis of daminozide is specific in separating it from any interfering peaks. The GC-MS method used for UDMH is specific in not only separating it from interfering peaks but the molecular ion of UDMH has been confirmed by Mass Selective detection.

2.11.2 *Linearity*

The linearity of the test procedure is the ability to obtain test results proportional to the concentration of analyte within the sample. Linearity data were obtained for daminozide using a four-point calibration curve to get a correlation coefficient. For UDMH, linearity data were obtained using a five-point calibration curve to get a correlation coefficient. Each concentration containing the analyte was injected in duplicate and the average value was used to determine the linearity of the method. Out of the four to five concentrations used, the concentration of the analyte of interest was in the middle with one to two concentrations above and below the fortification level selected to ensure the linearity of the method.

2.11.3 *Precision*

Precision or Repeatability is the closeness of agreement between independent test results obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

The precision of the method was determined by making duplicate injections of the 0-6 inch and 6-12 inch control soil spiked with daminozide at the 1ppb level on two different days.

2.11.4 Accuracy/Recovery

The accuracy/recovery of the method is the degree to which the observed results correspond to the true value of the analyte in the sample.

Two control untreated soil (0-6 inch and 6-12 inch) samples were each fortified with 1 ppm daminozide .

Each of the fortified soil samples were extracted and analyzed in duplicate as described in Sections 2.5 and 2.6. The recovery of the method was determined based on the fortified soil samples.

The samples were analyzed in the following order:

- Calibration standards for standard curve
- Control soil (0-6 inch).
- Control soil (0-6 inch) spiked with 1 ppm daminozide.
- 0 Time point treated soil (0-6 inch)
- Control soil (6-12 inch)
- Control soil (0-6 inch) spiked with 1 ppm daminozide.
- 0 Time point treated soil (6-12 inch)
- 3-Hour Time point treated soil (0-6 inch)
- 6-Hour Time point treated soil (0-6 inch), etc.

2.12 QUALITY CONTROL PROCEDURES

Quality control measures were taken to ensure that the data were accurate. Analytical sample sets consisted of control soil fortified with daminozide plus calibration standards consisting of 4 to 5 different concentrations of the analyte (daminozide or UDMH) bracketing the analyte concentration. The fortified soil samples were analyzed on a different day to establish recovery.

2.13 DATA HANDLING

Copies of chromatograms are included in Appendix 3. Standard calibration curves and control soil samples fortified with the analyte of interest were analyzed concurrently with the treated soil samples (data are provided in Appendix 2).

2.13.1 *Linearity*

From the four or five-point calibration curve, a regression analysis was done and the following were determined: 1) slope, 2) intercept and 3) correlation coefficient.

2.13.2 *Precision*

The precision of the method was determined from the duplicate injections of the control untreated soil samples fortified with the analyte of interest. From the above analysis, the recovery, standard deviation (SD) and relative standard deviation (RSD) were calculated.

2.13.3 *Accuracy/Recovery*

Recovery of daminozide from the 0-6 inch and 6-12 inch control soil fortified at 1 ppm and recovery of UDMH from the 0-6 inch and 6-12 inch control soil fortified at 0.4 ppm and 2 ppm was determined following extraction and analysis as described in Sections 2.5 and 2.6. The % recovery was determined from regression analysis. Standard deviation was performed on the mean % recovery data and the relative standard deviation was calculated from this.

2.13.4 *Limit of Detection (LOD) and Limit of Quantitation (LOQ)*

The limit of detection (LOD) was calculated as approximately 1/3 the value of the limit of quantitation. The limit of determination (often referred to as the limit of quantitation) is defined as the lowest concentration tested, at which an acceptable mean recovery is obtained (normally 70 to 110% with a relative standard deviation of ≤20%; in certain justified cases lower or higher mean recovery rates as well as higher relative standard deviations may be acceptable).

3.0 RESULTS AND DISCUSSION

3.1 SPECIFICITY

Two different analytical methods were used for the separation of daminozide and UDMH. The GC-NPD method used for analysis of daminozide is specific in separating it from any interfering peaks. The GC-MS method used for UDMH is specific in not only separating it from interfering peaks but the molecular ion of UDMH has been confirmed by Mass Selective detection.

3.2 LINEARITY

Based on regression analysis (see Table II), using a four-point (0, 10.15, 20.30 and 101.50 µg) standard calibration curve, the R^2 value was 0.9993 for daminozide. For UDMH (see Table III), the R^2 value was 0.9980 using a five point (0, 5, 10, 30 and 50 µg) standard calibration curve (Figure 5).

3.3 PRECISION

The Precision of the method (Table IV) was determined from different samples of the 0-6 inch and 6-12 inch control soil fortified with daminozide or UDMH and analyzed on two consecutive days. From the above data, the standard deviation and relative standard deviation (RSD) were calculated.

Based on the 0-6 and 6-12 inch soil data for *daminozide* at 1 ppm fortification level, the standard deviation was 13.39% with a relative standard deviation of 24.78%.

Based on the 0-6 and 6-12 inch soil data for *UDMH* at 0.4 ppm fortification level, the standard deviation was 13.14% with a relative standard deviation of 10.89%. At the 2.0 ppm *UDMH* fortification level, the standard deviation was 13.35% with a relative standard deviation of 12.05%.

Although the RSD for daminozide is slightly higher than that required by EU and EPA, the RSD for UDMH is <20% and satisfies both the regulatory requirements.

3.4 ACCURACY/RECOVERY

Control 0-6 inch and 6-12 inch soil samples fortified with daminozide (Table V) or UDMH (Table VI) were extracted and analyzed. Appendix 3 contains representative chromatograms. Detailed information on all the samples is available in study reports 85124 and 8647. The results were compared to the area response for the standards.

Recovery of *daminozide* (Table V) from both the 0-6 inch and 6-12 inch soil depths fortified at 1 ppm , averaged 54.03% with a RSD of 24.78%. Although the recovery and RSD for daminozide are outside the limits required by the guidelines, these results are considered to be about the best that can be done because of the inherent instability of daminozide in soil.

Recovery of *UDMH* (Table VI) fortified at 0.4 ppm averaged 120.65% from both the 0-6 inch and 6-12 inch soil depths. At 2.0 ppm fortification level, recovery of UDMH averaged 110.73%. Both the recovery values are between the 70-120% as required by the EPA. However, at the 0.4 ppm fortification level, the recovery was slightly higher than that allowed (70-110%) by the EU. The RSD for UDMH however is adequate at both the fortification levels.

3.5 LIMIT OF QUANTITATION (LOQ) AND LIMIT OF DETECTION (LOD)

The limit of determination (often referred to as the limit of quantitation) is defined as the lowest concentration tested, at which an acceptable mean recovery is obtained (normally 70 to 110% with a relative standard deviation of ≤20%; in certain justified cases lower or higher mean recovery rates as well as higher relative standard deviations may be acceptable). The limit of quantitation (LOQ) is 1 ppm for daminozide and 0.4 ppm for UDMH. The LOQ for daminozide is based on the lower recovery value (54.03%) and the LOQ for UDMH is based on a slightly higher recovery value than that normally allowed by the EU. The RSD for UDMH however is ≤20% as required by both the guidelines.

The limit of detection (LOD) was calculated as approximately 1/3 the value of the limit of quantitation and is ~0.3 ppm for daminozide and ~0.1 ppm for UDMH.

3.6 RUGGEDNESS

No ruggedness testing was done but the GC-NPD for determining daminozide and GC-MS method for UDMH used are reliable analytical methods. Copies of representative chromatograms are provided in Appendix 3.

3.7 LIMITATIONS

Extraction method may be improved to get better recovery of daminozide. Recovery of UDMH, however was good.

3.8 INDEPENDENT LABORATORY VALIDATION (ILV) (USA)/REPRODUCIBILITY (EU)

Reproducibility (EU) is defined as an independent lab validation. Reproducibility 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. The field dissipation study for daminozide has been done as part of the analytical method development in the same laboratory (see Appendix 3 for results). This indicates that the method has not been independently validated.

4.0 CONCLUSIONS

The analytical method AC-6004 described in this report is applicable to the analysis of daminozide and UDMH in a variety of soil types. For daminozide, the LOD is ~0.3 ppm and the LOQ is 1 ppm. For UDMH, the LOD is ~0.1 ppm and the LOQ is 0.4 ppm.. Recovery of daminozide from the 0-6 and 6-12 inch soil depths, averaged 54.03% with a RSD of 24.78%. Although the recovery and RSD are outside the limits required by the EU and the EPA, these results are considered to be about the best that can be done because of the inherent instability of daminozide in soil. Inspite of the low recovery from either the 0-6 or 6-12 inch soil depth, the RSD however was <20% as favored by the guidelines. Recovery of UDMH averaged 120.65% at 0.4 ppm fortification level and 110.73% at the 2.0 ppm fortification level. The recovery values at both the fortification levels for UDMH are between the 70-120% as required by the EPA. However, at the 0.4 ppm fortification level, the recovery was slightly higher than that allowed (70-110%) by the EU. The RSD for UDMH on the other hand satisfies both EU and EPA requirements at both the fortification levels.

Since daminozide degrades very rapidly in soil (half-life <1 day), the low and variable recovery of daminozide from soil samples could be partly due to this rapid degradation. The recovery of UDMH, the major degradate of daminozide from field dissipation studies was however good and therefore the method should be acceptable.