METHOD FOR DETERMINATION OF RESIDUES OF OXADIXYL (SAN 371F) AND ITS M-3 METABOLITE IN VARIOUS CROPS

METHOD # AM-0827-0289-0

Sandoz Crop Protection Corporation
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Des Plaines, Illinois 60018
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ANALYTICAL METHOD

SANDOZ CROP PROTECTION CORPORATION

Location: 1300 E. Touhy Ave.
Des Plaines, IL 60018

DEVELOPMENT QUALITY CONTROL

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METHOD FOR DETERMINATION OF RESIDUES OF OXADIXYL (SAN 371F)
AND ITS M-3 METABOLITE IN VARIOUS CROPS

1. SUMMARY/INTRODUCTION

1.1 Scope

Oxadixyl and its M-3 metabolite have been analyzed in many crop and soil matrices since 1981. Previous methods used were CBK 3079/82031\textsuperscript{1}, CBK 3092/83014\textsuperscript{2}, and AM-0808\textsuperscript{3}. The present method expands on this previous work with the inclusion of capillary chromatography and Solid Phase Extraction (SPE) techniques. This method is being presented as the Residue Enforcement Method. Crop matrices analyzed to date are; alfalfa, carrot (tuber), carrot (foliage), corn, cottonseed, cucumber, green beans, lettuce, pea, potato (tuber), radish, sorghum grain, soybean grain, sunflower seed, tomato, turnip, wheat grain and tobacco. Additionally, this method was validated with recoveries at three levels of fortification in 9 different matrices by a contract laboratory\textsuperscript{4}. Matrices validated in 1988 included many of the above as well as sweet corn, cabbage and cantaloupe.

1.2 Principles

1.2.1 This method can be used for the determination of parent oxadixyl (M-1) and a metabolite (M-3) in raw agricultural commodities (RAC's) down to 0.01 ppm for each compound. Their structures, chemical names, empirical formulas and molecular weights are given in Figure 1.

1.2.2 A known weight of crop sample (10-20 gm depending upon sensitivity needed) is homogenized with methanol, centrifuged, and the methanol decanted from the insoluble material.
1.2.3 The methanol is removed at reduced pressure and the residue dissolved in 2N HCl.

1.2.4 The mixture is refluxed for 4 hr, cooled to room temperature and transferred to a 100-ml graduated cylinder.

1.2.5 For foliage samples, this acid hydrolysate is extracted once with hexane and the hexane discarded.

1.2.6 The aqueous hydrolysate is centrifuged and an aliquot is applied to a preconditioned C-18 Bond Elute column.

1.2.7 The column is washed with 10 ml 10% methanol/90% water, 10 ml hexane and 10 ml 70% hexane/30% toluene. All of these washes are discarded.

1.2.8 Residues are eluted from the column with ethyl ether. To the combined ethyl ether eluate is added 1 ml of toluene and the mixture is concentrated until only toluene remains, using a gentle stream of nitrogen. The residues are then diluted in toluene for gas chromatographic (GC) analysis using a megabore capillary column and nitrogen-phosphorous detector.

1.2.9 The limit of detection for both oxadixyl (M-1) and the M-3 metabolite is 0.01 ppm, depending upon the sensitivity of the GC instrumentation and the final dilution volume of the toluene solution. Recoveries of fortified oxadixyl and M-3 metabolite are given in Tables I - III.

2. SAFETY

2.1 The oral LD$_{50}$ of oxadixyl in rats is between 500-1000 mg/kg.

2.2 Normal laboratory precautions are required for the safe handling of this pesticide.

2.3 Hexane, ethyl ether, acetone, methanol, and toluene are flammable and should not be used near heat, sparks, or open flames.

2.4 All solvents should be used only in well ventilated laboratories.

2.5 Protective glasses should be worn during extraction and analysis.
2.6 Disposal of samples and standards must be done in compliance with on-site safety policies and procedures.

3. MATERIALS/METHODS

3.1 Equipment

3.1.1 Glassware; Volumetric pipets (1 to 25-mL); Volumetric flasks (5 to 100-mL); Glass beads (2 mm); Vials (2-mL); Pasteur pipets, 9" disposable; from any general laboratory equipment supplier.

3.1.2 Blender, a Hobart chopper or a Waring blender.

3.1.3 Bottles, 8 oz. glass, with poly-seal® screw cap, (two per sample).

3.1.4 Homogenizer, Brinkmann Polytron.

3.1.5 Centrifuge, Model EXD, International Equipment Company, Needham Hts., Maryland, or equivalent.

3.1.6 60º Filter funnel.

3.1.7 Glass Wool.

3.1.8 Flat bottom flask, 250-mL.

3.1.9 Rotary Vacuum Evaporator, Buchi Rotovapor-RE, Switzerland, or equivalent.

3.1.10 Reflux Condenser.

3.1.11 Hot plate.

3.1.12 Graduated cylinder, 100-mL.

3.1.13 Separatory Funnel, 250-mL.

3.1.14 Bond Elut® column, 1000 mg C-18, Analytichem International, Harbor City, California (catalog no. 607406), or equivalent.

3.1.15 Visiprep Solid Phase Extraction Vacuum Manifold, Supelco, Inc. Belleville, PA 16823-0048, (catalog no. 7121-03), or equivalent.

3.1.16 Nitrogen gas evaporator, N-EVAP, Organization Association, South Berlin, MA. 01549-0159, or equivalent.

3.1.17 Gas Chromatograph (HP 5880A), equipped with
Autoinjector (HP 7673A) and Nitrogen/Phosphorous Detector, or equivalent.

3.1.18 Soxhlet Extractor, equipped with hot plate and extraction cylinder to hold a 33 mm x 80 mm extraction thimble, American Scientific Products, Chicago, Illinois (optional).

3.2 Reagents

3.2.1 Methanol, high purity, Burdick and Jackson, Muskegon, Michigan 49442.

3.2.2 Sodium sulfate, anhydrous granular, reagent grade.

3.2.3 Water-Delonized, Milli-Q water purification system, Millipore Corporation, Bedford, Maryland.

3.2.4 2N Hydrochloric acid, (made from 16.5 mL of reagent grade acid plus 83.5 mL of deionized water).

3.2.5 Hexane, non-spectro high purity, Burdick and Jackson, Muskegon, Michigan 49442.

3.2.6 Toluene, Baker resi-analyzed, J.T. Baker Chemical Company, Phillipsburg, New Jersey 08865.

3.2.7 Ethyl ether, Baker resi-analyzed, J.T. Baker Chemical Company, Phillipsburg, New Jersey 08865.

3.2.8 Acetone, Baker resi-analyzed, J.T. Baker Chemical Co., Phillipsburg, New Jersey 08865. (Required only if soxhlet extraction is used).

3.3 Preparation of Standards

3.3.1 Oxadiaryl (M-1): N-(2,6-dimethylphenyl)-2-methoxy-N-(2-oxo-3-oxazolidinyl)-acetamide, Sandoz Crop Protection Analytical Reference Standard.

3.3.2 M-3 metabolite: N-(2,6-dimethylphenyl)-2-hydroxy-N-(2-oxo-3-oxazolidinyl)-acetamide, Sandoz Crop Protection Analytical Reference Standard.

3.3.3 Oxadiaryl Stock Solution - 1 mg/mL. Accurately prepare a stock solution containing 100 mg oxadiaryl in acetone in a 100-mL volumetric flask, giving a 1 mg/mL solution.

3.3.4 M-3 Metabolite Stock Solution - 1 mg/mL. Accurately prepare a stock solution containing 100 mg M-3 in acetone in a 100-mL volumetric flask, giving a 1 mg/mL solution.
3.3.5 Oxadixyl plus M-3 Fortification Standard
10 μg/mL Each Compound: Transfer 1 mL of the
Oxadixyl stock solution and 1 mL of the M-3
metabolite stock solution to the same 100-mL
volumetric flask and dilute to the mark with
acetone. This solution contains 10 μg/mL of each
compound. This standard is used for laboratory
fortifications of control samples.

3.3.6 Oxadixyl plus M-3 "Working Solution A" - 10 μg/mL
Each Compound. Transfer 1 mL of the Oxadixyl
stock solution and 1 mL of the M-3 metabolite stock
solution to the same 100-mL volumetric flask and
dilute to the mark with Toluene. This gives a
combined standard of 10 μg/mL of each compound.

3.3.7 Oxadixyl plus M-3 "Working Solution B" - 1 μg/mL
Each Compound. Transfer 25 mL of "working
solution A" to a 250-mL volumetric flask and dilute
to the mark with Toluene. This gives a combined
standard of 1 μg/mL of each compound.

3.3.8 Oxadixyl plus M-3, G.C. Standard Solutions - 0.01,
0.05, 0.1, 0.25, 0.50 and 0.75 μg/mL. Transfer
aliquots of "Working Solution B" (1 μg/mL of each
compound) to 100-mL volumetric flasks and dilute to
the mark with Toluene, as shown below. These are
the GC standard solutions used for quantitation.

<table>
<thead>
<tr>
<th>Std. Conc. (μg/mL)</th>
<th>Milliliters of &quot;Working Soln B&quot;</th>
<th>Toluene</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>0.05</td>
<td>5</td>
<td>95</td>
</tr>
<tr>
<td>0.10</td>
<td>10</td>
<td>90</td>
</tr>
<tr>
<td>0.25</td>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td>0.50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>0.75</td>
<td>75</td>
<td>25</td>
</tr>
</tbody>
</table>

3.4 Analytical Procedure (for all crop matrices)

3.4.1 Preparation of sample

A representative sample is prepared for analysis by
blending in a Hobart chopper or a Waring blender until
the sample is homogeneous. All samples should be
stored frozen before and after homogenization.

3.4.2 Extraction

3.4.2.1 Weigh 10-20 gm of homogeneous sample
(depending on the GC instrumentation
sensitivity and necessary level of detection)
into a 8 oz. glass jar. Fortifications are made at this point.

NOTE: When fortifying samples, add a known amount of oxadixyl and M-3 metabolite (see Section 3.3 for preparation of standards) to the sample and allow 15 minutes for the solvent to evaporate before adding the extraction solvent (methanol).

3.4.2.2 Add 100 mL of methanol to the sample and homogenize for a minimum of 3 minutes at maximum speed using a polytron homogenizer. The sample should be thoroughly pulverized into a uniform mixture.

3.4.2.3 Centrifuge the mixture for 10 minutes at 2,000 rpm. Place about 2 grams of anhydrous sodium sulfate in a 60° filter funnel containing a small plug of glass wool. Decant the solvent through the sodium sulfate into a 250-mL flat bottom flask. Gently rinse the 8 oz. glass bottle with 20 mL of fresh methanol and pour it through the sodium sulfate into the 250-mL flask. Further rinse the sodium sulfate with 2 x 5 mL of methanol.

3.4.2.4 Remove most of the methanol at reduced pressure (rotary evaporation) using a 50°C water bath. Before the sample goes to dryness interrupt the evaporation, add 10 mL of deionized water to the flask, and continue to rotary evaporate the sample removing the last trace of methanol. When the remaining methanol has been removed an abrupt change in the rate of solvent condensation in the rotary evaporator collection flask can be observed. Do not evaporate the sample to dryness.

3.4.3 Acid Hydrolysis

3.4.3.1 Add 80 mL of 2N hydrochloric acid (see Section 3.2.4 for acid preparation) to the flask containing the aqueous extract from section 3.4.2.4. Attach the flask to a condenser (Section 3.1.10) and reflux on a hot plate for 4 hours. Wrapping glass wool or aluminum foil around the flask may be necessary to maintain reflux.

3.4.3.2 Cool the flask to room temperature and transfer the acid solution to a 100-mL graduated cylinder. Rinse the flask with a few milliliters of deionized water, adding
this to the graduated cylinder, and adjust the final volume to the 100-mL mark with deionized water.

3.4.4 Foliage Samples - Hexane Partition Clean-up

3.4.4.1 For foliage samples only, extract the 100 mL of aqueous acid hydrolysate (from section 3.4.3.2) with 50 mL of hexane using a 250-mL separatory funnel. Discard this hexane fraction.

3.4.5 C-18 Column Clean-up

3.4.5.1 Transfer this aqueous hydrolysate from section 3.4.3.2 or section 3.4.4.1 to a clean 8 oz. glass bottle and centrifuge for 5 minutes at 2,000 rpm.

3.4.5.2 Attach a C-18 Bond Elute column (1000 mg) to a Solid Phase Extraction vacuum manifold processing station. Condition the C-18 Bond Elute column by washing sequentially with 10-15 mL each of methanol and then water. Discard both of these washings. Apply 50 mL of the aqueous hydrolysate solution from section 3.4.5.1 to the C-18 column. Sequentially pass the following solutions through the C-18 column: 10 mL of 10% methanol/90% water; 10 mL of hexane; and 10 mL of 70% hexane/30% toluene. Discard all eluates up to this point. Avoid letting the column go to dryness until all of these solutions have passed through the column.

3.4.5.3 Aspirate the column for about 1 minute to ensure removal of previous solvents. Release the vacuum and put the 10-mL receivers into the manifold. Reapply the vacuum and elute oxadiazyl and M-3 residues from the C-18 column into the 10-mL volumetric flask with 3 x 1-mL portions of ethyl ether. Three consecutive 1-mL elutions, instead of one 3-mL elution, are necessary for high recoveries. Allow the column to go dry between each ethyl ether elution. Add 1 mL of toluene to the volumetric flask and evaporate the combined ethyl ether eluate, leaving the toluene, with a gentle stream of nitrogen (N-EVAP evaporator Section 3.1.16). Add toluene to bring the total volume to 10 mL. Sometimes there is a small amount of water which remains at the bottom of the flask. Thoroughly shake the flask to extract any residue from the water
and then let any water droplets settle to the bottom before taking a sample of this solution for GC analysis. Dilution to a lesser volume may be necessary if the GC instrumentation used for analysis is less sensitive than indicated in this method.

3.4.5.4 The sample is now ready for G.C. analysis or dilution if necessary.

3.5 Analysis

3.5.1 Gas Chromatograph Conditions

The following instrument and operating conditions are suitable for analysis of oxadixyl and its M-3 metabolite. Other conditions may be acceptably provided that oxadixyl and its M-3 metabolite are separated from sample interferences and response is linear over the range of interest. Verify that the detector response is linear over the desired range and the retention time is stable, on a daily basis. This is preferably done by injecting standard solutions prior to the analysis of samples and after each 2-4 samples during analysis.

3.5.1.1 GC/NPD Analysis

Instrument: Hewlett-Packard, model 5880A Gas Chromatograph equipped with a 7673A autosampler and a Nitrogen/Phosphorous Detector.

Columns: Column: HP-1 megabore (SE-30, crosslinked methyl silicone gum), 30 m x 0.53 mm ID, 0.88 μm film thickness. Hewlett-Packard catalog no. 19095Z-023.

Optional Column: HP-5 megabore (SE-54, crosslinked 5% phenyl-methyl gum), 30 m x 0.53 mm ID, 0.88 μm film thickness. Hewlett-Packard catalog no. 19095J-023.

Optional Column: HP-5 capillary (SE-54, crosslinked 5% phenyl-methyl gum), 25 m x 0.31 mm ID, 0.52 μm film thickness. Hewlett-Packard catalog no. 19091B-112.
HP-5 DB-5°
Megabore

Temperatures in °C
Oven Temperature: 215 210
Injector Temp.: 250 250
Detector Temp.: 350 350

Gas Flows in mL/min.
Carrier Gas: helium 27 4
Detector Gas: air 100 150
hydrogen 4 12
Make-Up Gas: helium -- 27

Oxadixyl Retention Time:(min.) 3.2 4.7
M-3 Metabolite Retention Time:(min.) 3.5 5.1

These retention times will vary with the exact G.C. operating conditions and length and condition of the column.

3.5.1.2 Another optional GC column that can be used is a 1 m x 2 mm ID glass column packed with 4% SE-30 on ultrabond (Applied Science Laboratories).

3.5.1.3 Operating conditions are: column temperature = 230°C, injection port temperature = 250°C, detector temperature = 300°C, and nitrogen carrier gas at a flow rate of 30 mL/min.

3.5.2 Quantitation

3.5.2.1 Prepare a calibration curve by injecting standards of known concentration (Section 3.3.8) and plotting peak height versus concentration of injected standard on log-log paper, or using an equivalent computer program.

3.5.2.2 Determine the concentration of oxadixyl and its metabolite M-3 in an injected aliquot of sample from the peak height for the sample and the standard curve obtained in section 3.5.2.1.

3.5.2.3 Calculate the concentration of residue in the sample using the formula given in section 3.9.

3.6 Interferences

3.6.1 Sample matrices

More than 15 crop types have been analyzed with control chromatograms (matrix not treated with oxadixyl) showing no significant interfering peaks
in the regions of oxadixyl (M-1) and metabolite M-3 peaks.

3.6.2 Other Pesticides

Crops treated with pesticides such as Ambush, Bravo, Captan, Lannate, Mancozeb, Prast, Prowl, Lorox, Sencor, and Temik were analyzed and found to be free of interferences from these chemicals in the regions of oxadixyl (M-1) and metabolite M-3 peaks.

3.6.3 Solvents and labware

Interferences have not been a problem when using the high quality solvents listed under section 3.2 of this method and carefully cleaned labware.

3.7 Time Required For Analysis

3.7.1 Once the materials and instrumentation are set-up, 8 samples can be prepare for GC analysis in a 10 hour day. GC analysis time, with standards, can take an additional 2 hours. Several sets can be overlapped, performing the extraction and cleanup of a set during the 4 hour reflux of a subsequent set, shortening the overall time per sample.

3.8 Modifications

3.8.1 An optional extraction procedure using a soxhlet extraction instead of the polytron homogenization can be used. Weigh a 20 gram sample into a 33 mm x 80 mm extraction thimble, add 175 mL of acetone and a few glass beads to the boiling flask and reflux (soxhlet extract) overnight at a rate of about 6 solvent turnovers per hour. After cooling, transfer the acetone to a 250-mL flask, concentrate the acetone at reduced pressure as described in section 3.4.2.4 and follow the rest of the method.

3.9 Methods of Calculation

3.9.1 Calculate the concentration of the residue in the sample using the following expression:

$$\text{ppm (ng/mg)} = \frac{C_e (\text{ng/µL}) \times V_s (\text{µL})}{W (\text{mg})}$$

Where:

- ppm = concentration of analyte in the sample in parts per million (ng/mg).
- $C_e$ = concentration of analyte in extract (ng/µL)
determined from the standard curve.

\[ V_s = \text{volume of final sample extract in microliter taking into account all dilutions.} \]

\[ W = \text{weight of sample taken for analysis, in milligrams.} \]

4. RESULTS/DISCUSSION

4.1 Accuracy

4.1.1 Table I, which is from the 1983 method CBK 3092/63014, shows recoveries from various fortification levels in 6 different crop matrices with average recoveries of 99% ± 5.6 and 88% ± 10.2 for oxadixyl (M-1) and metabolite M-3, respectively. The range of recoveries is 72% for M-3 in potato to 108% for M-1 in lettuce. Most recoveries are between 83% and 104%.

4.1.2 Table II, which is from a 1984 report CBK 11000/84007, shows recoveries from 0.1 ppm and 0.2 ppm fortification levels in 13 different crop matrices with average recoveries of 94% ± 19.8 and 92% ± 19.5 for oxadixyl (M-1) and metabolite M-3, respectively. Most recoveries are between 74% and 110%.

4.1.3 Table III, which is from an outside lab validation (EPL Bio-Analytical Services), gives recoveries from 9 different matrices fortified at three different levels (0.05, 0.1 and 0.2 ppm). Recoveries for all matrices except cotton seed, averaged between 80 to 100%. Cotton seed recoveries averaged 66% for Oxadixyl and 74% for M-3. Additional substrates are being tested with some possible minor modifications essentially dealing with cleanup techniques.

4.2 Precision

4.2.1 The coefficients of variation for the 14 recoveries in Table I are 5.6% for oxadixyl (M-1) and 11.5% for metabolite M-3.

4.2.2 The coefficients of variation for recoveries from 13 different crops in Table II are 21% for oxadixyl (M-1) and 21.2% for metabolite M-3.

4.2.3 The coefficients of variation for the 27 recoveries
in Table III for oxadixyl (M-1) and the 27 recoveries for metabolite M-3 (including all three fortification levels) are 18.2% and 23.1%, respectively.

4.3 Limit of Detection/Quantification

4.3.1 The limit of detection was 0.01 ppm for both oxadixyl (M-1) and metabolite M-3. EPL Bio-Analytical Services needed to use a much more concentrated final solution (section 3.4.5.3) to obtain adequate detection of oxadixyl and M-3, because of their instrument sensitivity.

4.3.2 Quantitation limits can vary depending on column efficiency, instrument sensitivity and background GC peaks. Only a few matrices showed lower recoveries at lower fortification levels. However, these recoveries were still acceptable. The optional HP-5 capillary column is suggested for use if minimum quantification levels are deemed necessary.

5. CONCLUSION

This method is better than the 1983 and 1984 methods since the megabore and capillary gas chromatographic columns are superior to the packed columns used in the previous methods. A large number of recoveries (9 matrices at three different levels of fortification given in Table III) have been generated using this method and a DB-5 megabore column, with good results.

Since these 9 crop matrices cover 9 different crop groupings, this method is expected to give adequate enforcement data on almost any crop matrix.

The analysis of oxadixyl (M-1) and metabolite M-3 using this method has also been performed by an contract laboratory (EPL Bio-Analytical Services) with good results and little interaction from Sandoz. As a result, this method is expected to give acceptable performance at any location by trained personnel.

6. CERTIFICATION

This method has been compiled and prepared by M.M. Graben, T.R. Bade, and W.H. Cahill.
7. **TABLES**

**Table I. Recovery Data from 1983 Method - CBK 3092/83014**

<table>
<thead>
<tr>
<th>Crop Matrixes</th>
<th>Fortification Level (ppm)</th>
<th>Mean % Recovery Oxadixyl</th>
<th>M-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tomato Fruit (2)</td>
<td>0.025</td>
<td>94%</td>
<td>108%</td>
</tr>
<tr>
<td>Lettuce (1)</td>
<td>0.05</td>
<td>99%</td>
<td>94%</td>
</tr>
<tr>
<td>Carrot Root (1)</td>
<td>0.05</td>
<td>104%</td>
<td>83%</td>
</tr>
<tr>
<td>Tomato Fruit (3)</td>
<td>0.10</td>
<td>101%</td>
<td>88%</td>
</tr>
<tr>
<td>Potato (2)</td>
<td>0.10</td>
<td>93%</td>
<td>72%</td>
</tr>
<tr>
<td>Lettuce (2)</td>
<td>0.10</td>
<td>108%</td>
<td>86%</td>
</tr>
<tr>
<td>Carrot Top (1)</td>
<td>0.10</td>
<td>92%</td>
<td>88%</td>
</tr>
<tr>
<td>Carrot Root (2)</td>
<td>0.10</td>
<td>101%</td>
<td>84%</td>
</tr>
</tbody>
</table>

**Overall Average and Standard Deviation**

<table>
<thead>
<tr>
<th>Oxadixyl</th>
<th>M-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>99%±5.6</td>
<td>88%±10.2</td>
</tr>
</tbody>
</table>

**Coefficient of Variation**

<table>
<thead>
<tr>
<th>Oxadixyl</th>
<th>M-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.6%</td>
<td>11.6%</td>
</tr>
</tbody>
</table>
Table II. Recovery Data from 1984 Field Residue Report
CBK 11000/84007 (using method CBK 3092/83014)

<table>
<thead>
<tr>
<th>Crop</th>
<th>Oxadixyl</th>
<th>M-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alfalfa</td>
<td>(N=1)</td>
<td>74%</td>
</tr>
<tr>
<td>Bermuda Grass</td>
<td>(N=1)</td>
<td>68%</td>
</tr>
<tr>
<td>Corn</td>
<td>(N=6)</td>
<td>99%</td>
</tr>
<tr>
<td>Cotton</td>
<td>(N=1)</td>
<td>104%</td>
</tr>
<tr>
<td>Cucumber</td>
<td>(N=1)</td>
<td>68%</td>
</tr>
<tr>
<td>Green Beans</td>
<td>(N=1)</td>
<td>99%</td>
</tr>
<tr>
<td>Pea</td>
<td>(N=2)</td>
<td>108%</td>
</tr>
<tr>
<td>Radish</td>
<td>(N=2)</td>
<td>119%</td>
</tr>
<tr>
<td>Sorghum</td>
<td>(N=1)</td>
<td>117%</td>
</tr>
<tr>
<td>Soybean</td>
<td>(N=1)</td>
<td>101%</td>
</tr>
<tr>
<td>Sunflower</td>
<td>(N=2)</td>
<td>104%</td>
</tr>
<tr>
<td>Turnip</td>
<td>(N=3)</td>
<td>59%</td>
</tr>
<tr>
<td>Wheat</td>
<td>(N=1)</td>
<td>103%</td>
</tr>
</tbody>
</table>

Total Average: 94  92
Standard Deviation: 19.8  19.5
Coefficient Of Variation: 21%  21.2%
### Table III. Recovery Data from 1988 Method Validation by EPL BIO-ANALYTICAL SERVICES, INC.

#### Recoveries of OXADIXYL at Each Fortification Level

<table>
<thead>
<tr>
<th>Fortification Level (ppm)</th>
<th>0.05</th>
<th>0.10</th>
<th>0.20</th>
<th>% Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sweet Corn</td>
<td>106.8</td>
<td>88.2</td>
<td>88.7</td>
<td>94.6±10.6</td>
</tr>
<tr>
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<td>83.7</td>
<td>80.4</td>
<td>94.1</td>
<td>86.1±7.1</td>
</tr>
<tr>
<td>Sweet Peas</td>
<td>92.6</td>
<td>117.9</td>
<td>88.4</td>
<td>99.6±15.9</td>
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<tr>
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<td>122.2</td>
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<td>102.2±19.9</td>
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<tr>
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<td>89.8</td>
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<tr>
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<td>78.7±14.3</td>
</tr>
<tr>
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<td>84.7±10.6</td>
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<tr>
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<tr>
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<td>66.1±15.5</td>
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Avg. Recovery: 79.1 ±17.3 %

#### Recoveries of M-3 at Each Fortification Level

<table>
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<th>0.10</th>
<th>0.20</th>
<th>% Recovery</th>
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<td>87.0</td>
<td>86.4</td>
<td>89.3±4.4</td>
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<tr>
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<td>84.9</td>
<td>87.7</td>
<td>93.2±12.0</td>
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<tr>
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</tr>
<tr>
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<td>150.6</td>
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<td>83.4±27.9</td>
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<tr>
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</tr>
<tr>
<td>Carrot Tops</td>
<td>102.0</td>
<td>98.6</td>
<td>72.9</td>
<td>91.2±15.9</td>
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<td>83.3</td>
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</tbody>
</table>

Avg. Recovery: 82.0 ±21.6 %
8. FIGURES

FIGURE 1. STRUCTURES AND NAMES OF OXADIXYL (M-1) AND METABOLITE M-3

Oxadixyl (M-1): N-(2,6-dimethylphenyl)-2-methoxy-N-(2-oxo-3-oxazolidinyl)-acetamide
Molecular Weight: 278
Empirical Formulas: C_{14}H_{16}N_{2}O_{4}

M-3 Metabolite: N-(2,6-dimethylphenyl)-2-hydroxy-N-(2-oxo-3-oxazolidinyl)-acetamide
Molecular Weight: 264
Empirical Formulas: C_{13}H_{16}N_{2}O_{4}
9. REFERENCES

1. CBK Report 3097/82031.


5. CBK Report 11000/84007. SAN 371F Residues in Fifteen Crops after IN and 2H Rate Seed-Treatment Applications.