Determination of Ethalfluralin in Canola Seed, Meal, and Refined Oil by Capillary Gas Chromatography with Mass Selective Detection

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Ethalfluralin is a herbicide that is effective for weed control on a wide variety of crops, including canola. A method is described for the determination of ethalfluralin residues in canola seed, meal, and refined oil. Residues are extracted from canola sample matrixes with acetonitrile. An aliquot of the extract is diluted with water and purified by C_{18} solid-phase extraction prior to analysis by capillary gas chromatography with mass selective detection. For all three sample matrixes, the method has a validated limit of quantitation of 0.02 μg/g and a limit of detection of 0.006 μg/g. Recoveries averaged 96 ± 7% for canola seed, 87 ± 6% for canola meal, and 89 ± 5% for refined oil. In a magnitude-of-residue study, canola seed from field plots that had been treated with ethalfluralin at one to three times the maximum label rate for weed control were found to contain no detectable residue of the herbicide.

Keywords: *Ethalfluralin; dinitroaniline; residues; canola; GC/MSD*

INTRODUCTION

Ethalfluralin, *N*-ethyl-*N*-(2-methyl-2-propenyl)-2,6-dinitro-4-(trifluoromethyl)benzenamine, is a dinitroaniline herbicide that is effective on a wide variety of crops, including soybeans, drybeans, dry peas, sunflowers, peanuts, pumpkins, squash, and canola. Ethalfluralin is the active ingredient in Sonalan herbicide, which primarily controls annual grasses such as foxtail spp., barnyardgrass, fall panicum, and crabgrass spp. At medium to high rates, it also controls certain annual broadleaf weeds such as redroot pigweed, kochia, and black nightshade (Weed Science Society of America, 1994). The environmental fate of ethalfluralin has been widely studied and has been the subject of a review (Wolt, 1997). The structure of ethalfluralin is shown in Figure 1.

Magnitude-of-residue studies conducted under actual field conditions are required by the U.S. Environmental Protection Agency (EPA) for the registration of pesticides on crops. As a result, analytical methodology is needed to determine residues in the raw agricultural and processed commodities of canola treated with ethalfluralin. A general residue method for ethalfluralin in crops by gas chromatography with electron capture detection has been published (Day, 1978). However, the published method did not provide adequate purification and recovery of ethalfluralin in the canola matrixes. A residue method for ethalfluralin in soil by gas chromatography with mass selective detection (GC/MSD) has also been published (West et al., 1988). Previous studies using radiolabeled (^{14}C) material demonstrated that no significant metabolites of ethalfluralin occurred in various crops (*WSSA Herbicide Handbook*, 1994). Thus, the following method is presented for the determination of ethalfluralin in canola seed, meal, and oil by GC/MSD. Results from using the analytical methodology to determine residues of ethalfluralin in canola samples from field residue trials are also presented.

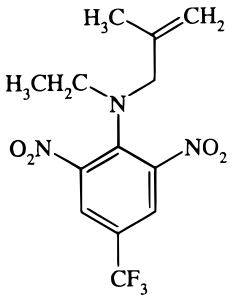

Figure 1. Structure of ethalfluralin.

MATERIALS AND METHODS

Test Substance. Two formulations of ethalfluralin were utilized for the field residue study: Sonalan 10G (a granular formulation) and Sonalan HFP (an emulsifiable concentrate formulation).

Field Sites. Two sets of residue trials were conducted. The first set of trials utilized the highest recommended application rate for Sonalan (1X rate) to determine the magnitude of ethalfluralin residues in the canola raw agricultural commodity when using the maximum label rate. The second set of trials used applications of Sonalan at three times the maximum label rate (3X rate) in order to determine if ethalfluralin residues in canola seed would concentrate in the processed products (canola meal and oil). Residue trials at the 1X rate were conducted in Georgia, Idaho (2 sites), Minnesota, North Dakota (2 sites), South Dakota, and Washington. The trials at the 3X rate were conducted in Idaho and North Dakota. Soil samples were taken at each field site to a depth of 30 cm, and the soils were characterized for chemical and physical properties (Table 1).

Herbicide Application. Sonalan 10G was applied directly to the soil surface, whereas Sonalan HFP was diluted with water and applied in a spray volume of 150−215 L/ha. Both formulations were applied using applicators that simulated

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Table 1. Soil Characteristics at the Residue Field Sites

soil characteristics				
location	type	pH	OM[a]	CEC[b]
Meigs, GA	loamy sand	7.1	1.2	7
Ashton, ID	loam	6.1	2.9	18
American Falls, ID	silt loam	7.4	2.2	22
Theilman, MN	loam	6.1	1.9	13
Northwood, ND	loam	7.6	4.6	21
Velva, ND	sandy clay loam	6.6	2.4	19
Britton, SD	loam	7.8	3.4	33
Ephrata, WA	sandy loam	7.1	1.5	14
Ashton, ID	loam	6.2	2.9	18
Velva, ND	loam	6.4	2.5	21

[a] Percent organic matter. [b] Cation exchange capacity, meq/100 g.

Table 2. Application, Planting, and Harvesting Information

location	rate, kg/ha	date		
		application	planting	harvesting
Meigs, GA	0.84	11/06/96	11/11/96	06/10/97
Ashton, ID	1.4	06/05/96	06/10/96	10/11/96
American Falls, ID	1.4	05/09/96	05/14/96	08/29/96
Theilman, MN	1.4	05/22/96	05/27/96	09/15/96
Northwood, ND	1.4	05/29/96	06/03/96	09/09/96
Velva, ND	1.4	05/15/96	05/20/96	08/23/96
Britton, SD	1.4	06/07/96	06/12/96	09/09/96
Ephrata, WA	0.84	05/28/96	05/31/96	08/23/96
Ashton, ID	4.2[a]	06/05/96	06/10/96	10/11/96
Velva, ND	4.2[a]	05/14/96	05/20/96	08/23/96

[a] Exaggerated (3X) rate for the canola processing residue study.

commercial applications. Treatments were incorporated into the top 5−8 cm of soil within 6 h of application. A second incorporation occurred 3−6 days after application, just prior to canola planting. Herbicide application dates and canola planting dates are included in Table 2.

The application rates made at the maximum recommended label rate at the various field sites ranged from 0.84 to 1.4 kg/ha, depending on soil texture, soil organic matter, and the time of application (Table 2). The exaggerated application rate (3X) was made at 4.2 kg/ha for the canola that was grown for use in the processing study. Except for the Georgia site, all applications and canola crop plantings were made in the spring of 1996. In Georgia, ethalfluralin was applied and canola was planted in the fall of 1996.

Climatological Conditions. Temperature and rainfall measurements were collected at each site and were compared to the 30-year averages from the NOAA weather station nearest to each site (Owensby and Ezell, 1992). Supplemental irrigation was used at the sites in Georgia, Idaho, and Washington.

Sample Collection, Handling, Shipment, and Storage. Mature canola seed was sampled at the normal harvest time for each field trial (Table 2). For the spring-planted canola, the time from application to harvest ranged from 87 to 128 days. For the fall-planted crop, the planting-to-harvest interval was 216 days. Sampling followed Codex guidelines, which recommend collecting samples from at least 12 representative areas within the plot while avoiding the plot borders. For the trials conducted at the maximum labeled rates, 0.9−3.2 kg of mature seed was collected in all but the Minnesota trial. In Minnesota, hot weather during flowering reduced the crop yield, and only 0.1 kg of seed was collected. For the processing study, grain samples were collected from canola treated at the 3X rate, and a separate 23-kg sample of canola seed was also collected for processing into meal and oil.

All seed samples were placed in clean, polypropylene-lined bags and were frozen within 4 h of collection. Samples were shipped to the analytical laboratory in a freezer truck or were packed in dry ice for overnight express delivery.

Upon receipt at the analytical lab, the samples were stored in a freezer at approximately −20 °C except when removed for preparation or analysis.

Initial Sample Preparation and Storage. *(a) Canola Seed and Meal.* Canola seed samples were passed through a sieve to remove field trash. The seed samples were frozen with liquid nitrogen, chopped, and then ground through a homoloid mill with a 2-mm screen size (Model J, The Fitzpatrick Co., Elmhurst, IL). After grinding, the samples were manually mixed in a plastic bag and then transferred to high-density polyethylene freezer cartons for storage at −20 °C. Canola meal samples that were prepared for method validation were prepared in the same manner as the seed.

(b) Refined Canola Oil. Canola oil samples that were used for method validation did not require sample preparation prior to frozen storage.

Analytical Reagents. Water was purified using a purification system (Milli-Q UV Plus, Millipore Corp., Milford, MA), and solvents were OmniSolv grade (EM Science, Gibbstown, NJ). The helium carrier gas was 99.995% pure (Airco Gas and Gear, Indianapolis, IN). The purity of the ethalfluralin analytical standard was determined chromatographically to be 99.8%. The analytical standard may be obtained from the Test Substance Coordinator, Dow AgroSciences LLC, 9330 Zionsville Road, Building 306/A1, Indianapolis, IN 46268.

Analytical Standard Preparation. A stock solution of the ethalfluralin analytical standard was prepared at 1000 μg/mL in toluene, and intermediate solutions were prepared at 10.0 and 2.0 μg/mL in methanol by further dilution of the stock solution. Appropriate dilutions of the 2.0-μg/mL solution were then made with methanol to result in fortification solutions at concentrations of 0.060, 0.20, 0.40, and 2.0 μg/mL.

GC/MSD calibration standards were also prepared from appropriate dilutions of the 1000-μg/mL stock solution. Intermediate solutions were prepared at 10.0 and 1.0 μg/mL in hexane. Calibration standards were then prepared by diluting appropriate aliquots of the 1.0- and 0.1-μg/mL intermediate hexane solutions with hexane to obtain concentrations of 0.003, 0.005, 0.01, 0.02, 0.05, and 0.1 μg/mL.

Weighing and Fortification of Recovery Samples. Untreated control samples (10 ± 0.1 g) were weighed into 4-oz (118-mL) glass bottles (Qorpak, with PTFE-lined lids, Fisher Scientific, Pittsburgh, PA). Fortified recovery samples were prepared by adding 1.0 mL of the appropriate fortification solution to untreated control samples. Preparation of the fortified samples, an untreated control sample, and a reagent blank sample was then continued as described under Sample Homogenization.

Sample Homogenization. *(a) Canola Seed and Meal.* Using a graduated cylinder, 40 mL of acetonitrile was added to each bottle, and the samples were blended on high speed for approximately 1 min using a homogenizer (Polytron Model PT-MR 3000, Brinkmann Instruments, Inc., Westbury, NY). The analyses were continued as described under Sample Extraction.

(b) Refined Canola Oil. Homogenization was not required for canola oil samples. Acetonitrile (40 mL) was added, and the analyses were continued as described under Sample Extraction.

Sample Extraction. Canola seed, meal, and oil samples were extracted by sealing the sample bottles with PTFE-lined lids and shaking on a variable-speed reciprocating shaker with a box carrier (Model 6000, Eberbach Corp., Ann Arbor, MI) for 30 min at 250 excursions/min. The samples were then centrifuged at 2250 rpm for 10 min.

Sample Filtration. *(a) Canola Seed and Meal.* Aliquots (approximately 10 mL) of the seed and meal sample extracts were filtered using 10-cc disposable syringes (Becton Dickinson & Co., Franklin Lakes, NJ) and 0.45-μm glass microfiber syringe filters (Whatman, Inc., Fairfield, NJ). Using gravity flow or by applying a slight positive pressure, the extracts were slowly filtered to retain the particulates, and the filtrates were collected in clean 40-mL vials.

Aliquots (5.0 mL) of the filtered extracts were transferred to clean 40-mL vials. Purified water (3.0 mL) was added to

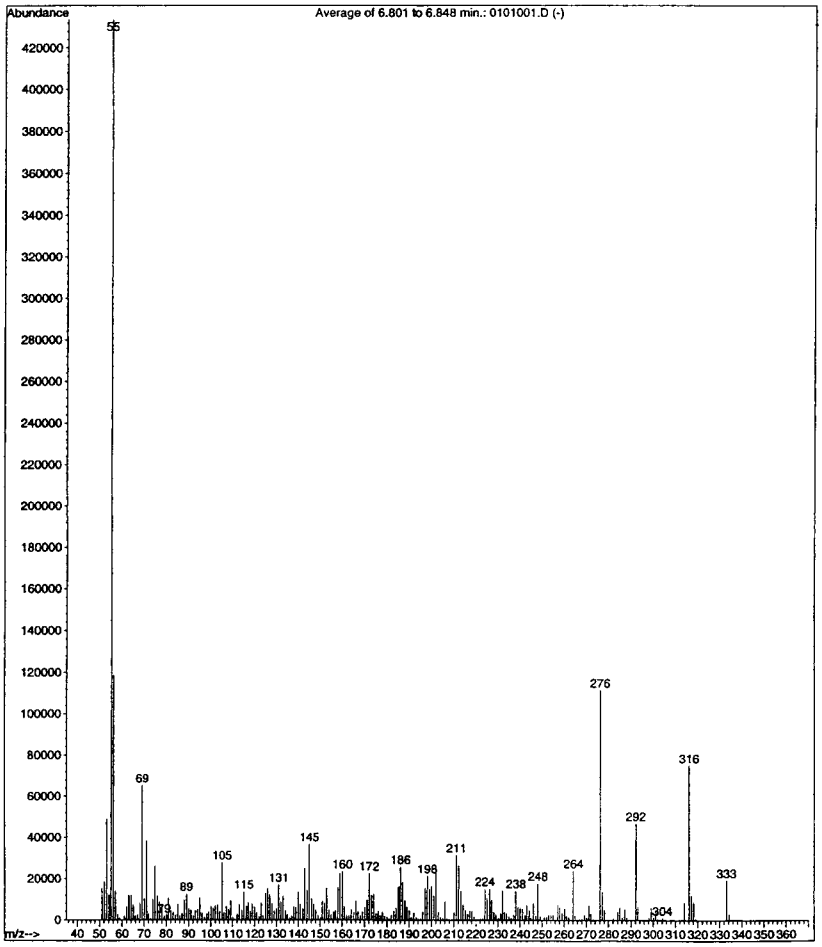

Figure 2. Mass spectrum of ethalfluralin.

the vials, and the contents were mixed using a vortex mixer (Model G-560, Scientific Industries, Inc., Bohemia, NY). The analyses were continued as described under Sample Purification.

(b) Refined Canola Oil. Oil sample extracts did not require filtration. A 5.0-mL aliquot of the acetonitrile extract was diluted with 3.0 mL of purified water and vortex mixed, and the analyses were continued as described under Sample Purification.

Sample Purification. The extracts of all of the canola matrixes were purified using C_{18} solid-phase extraction (SPE) columns (catalog no. P479, Fisher Scientific, Pittsburgh, PA). Prior to using each new lot of C_{18} SPE columns, an elution profile was determined by adding 0.2 μg of ethalfluralin and collecting fractions of the column eluants as described below for analysis by GC/MSD.

The C_{18} SPE columns were attached to a vacuum manifold (catalog no. 5−7250, Supelco, Inc., Bellefonte, PA). Before adding the sample extracts to the C_{18} SPE columns, the columns were conditioned with 5 mL of acetonitrile followed by 5 mL of purified water. Unless noted below, the column beds were not allowed to dry before adding the next solution.

The volumes of eluting solvents that are specified below were typical for the C_{18} SPE procedure, but the volumes used might require modification for a different lot of SPE columns. The sample extracts were added to the SPE columns in the acetonitrile/water solution and were slowly eluted under vacuum at a rate of approximately 1−2 mL/min. The eluate was discarded. The columns were rinsed with 5 mL of 60% acetonitrile/40% purified water, which was slowly eluted under vacuum at a rate of approximately 1−2 mL/min. The eluate was discarded, and the columns were dried for 5−10 min under a full vacuum (approximately −50 cm of Hg.)

Ethalfluralin was then eluted from the columns with 3.0 mL of hexane and collected in 5-mL volumetric flasks. The eluates in the volumetric flasks were diluted to volume with hexane and inverted several times to mix. Analyses were continued as described under GC/MSD.

GC/MSD. A Hewlett-Packard Model 5890A Series II GC with a Hewlett-Packard Model 5791A mass selective detector was used in combination with a Hewlett-Packard Model G1034B data system software for the measurement of peak area responses. The GC capillary column was a Durabond-5, 10 m × 0.18 mm i.d., 0.4-μm film thickness (J & W Scientific, Folsom, CA). The oven temperature was initially held at 80 °C for 1.5 min and was then ramped from 80 to 170 °C at 15 °C per min. After holding at 170 °C for 4 min, the temperature was again ramped from 170 to 300 °C at 30 °C per min and was then held at 300 °C for 6 min. The injector temperature was 250 °C, and the interface temperature was 300 °C. The carrier gas was helium with a head pressure of 70 kPa and a linear velocity of approximately 54 cm/sec. Charcoal, moisture, and oxygen filters (Chrompack, Inc., Raritan, NJ) were connected to the carrier gas lines to purify the helium entering the gas chromatograph. The injection mode was splitless with a purge delay of 1.4 min, a splitter flow of 50 mL/min, and a septum purge of 1.0 mL/min. The injection volume was 3 μL. Under these conditions, ethalfluralin had a GC retention time of approximately 6.8 min.

An electron-impact selected-ion monitoring detector was also utilized, and the electron multiplier was set at 1600 V (the tune setting). The maximum-sensitivity autotune (usertune) was used for the calibration program. The ions monitored for the determination of ethalfluralin were *m*/*z* 276 for quantitation and *m*/*z* 316 and 292 for confirmation. The dwell time was 75 ms. A total ion mass spectrum of ethalfluralin is illustrated in Figure 2.

Samples were analyzed by GC/MSD, and the suitability of the chromatographic system was determined using the following performance criteria. (a) It was determined that the correlation coefficient (r^2) exceeded 0.99 for the power regression equation that described the detector response as a function of the concentration of the calibration standards. (b) It was determined that the exponent for the power regression equation ranged from 0.9 to 1.10. (c) It was visually determined that baseline resolution was achieved for ethalfluralin relative to any potential interferences. (d) It was visually determined that a signal-to-noise ratio of at least 10:1 was achievable for detecting the 0.005-μg/mL calibration standard, which was equivalent to a residue at the 0.02-μg/g limit of quantitation for ethalfluralin in canola seed, meal, and refined oil.

If the peak response for any of the samples exceeded the range of the calibration curve, those samples were diluted with hexane and reanalyzed by GC/MSD.

Calculation of Results. A standard calibration curve was prepared by plotting the concentration of ethalfluralin in the calibration standards on the abscissa (x axis) and the respective peak areas on the ordinate (y axis). Using power regression analysis (Freund and Williams, 1991), the equation for the calibration curve was determined with respect to the abscissa as follows

$$y = \text{constant} \times x^{(\text{exponent})} \quad (1)$$

$$x = \left(\frac{y}{\text{constant}}\right)^{1/\text{exponent}} \quad (2)$$

The concentration (μg/g) of the analytes in the samples was calculated from the net concentration in the final solution (X), the final analysis volume (V), the weight of the sample that was extracted (W), and the aliquot factor (AF) using the following equation

$$\mu\text{g/g} = (X \times AF \times V)/W \quad (3)$$

The aliquot factor was calculated from the appropriate extraction and aliquot volumes for each sample type, i.e.

$$AF = (40\text{ mL extraction volume}/5\text{ mL aliquot volume}) = 8.0 \quad (4)$$

The percent recovery (R) was calculated from the net concentration (μg/g) found in fortified recovery samples using the following equation

$$R = [(\mu\text{g/g})/(\text{added }\mu\text{g/g})] \times 100\% \quad (5)$$

Calculated Limits of Detection and Quantitation. Using a technique described previously (Keith et al., 1983), the limits of detection (LOD) and quantitation (LOQ) for the residue method were calculated from the standard deviation (s) of the results of the ethalfluralin concentration (μg/g) found in at least 8 recovery samples fortified at 0.02 μg/g. For each sample matrix, the LOD was calculated as 3 times the standard deviation (3s), and the LOQ was calculated as 10 times the standard deviation (10s).

Confirmation of Results. For each calibration standard, the confirmation ratio was calculated as:

$$\text{Confirmation Ratio} = \frac{\text{peak area of confirmation ion}}{\text{peak area of quantitation ion}} \quad (6)$$

$$\text{Confirmation Ratio} = \frac{\text{peak area at } m/z\ 316}{\text{peak area at } m/z\ 276} \quad (7)$$

Residues of ethalfluralin were confirmed in a sample if the retention times of the analytes in the samples matched those in the calibration standards and if the confirmation ratios were within the range of ±20% of the average calculated for the standards. The mass spectrum of ethalfluralin in Figure 2 contains additional ions (e.g., m/z 292) that may also be used for confirmation, if desired.

Table 3. Recovery of Ethalfluralin from Canola Seed, Meal, and Refined Oil

sample type	added, μg/g	n	% recovery (mean ± SD)[a]
canola seed	0.006	1	NA[b]
	0.02	9	97 ± 8
	0.04	4	95 ± 7
	0.20	5	94 ± 7
	overall	18	96 ± 7
canola meal	0.006	1	NA
	0.02	10	87 ± 8
	0.04	3	87 ± 3
	0.20	3	85 ± 2
	overall	14	87 ± 6
canola oil	0.006	1	NA
	0.02	8	90 ± 6
	0.04	3	85 ± 1
	0.20	3	92 ± 3
	overall	14	89 ± 5

[a] No residues were detected in untreated control samples. [b] Peaks were detected in samples fortified at 0.006 μg/g (the method LOD), but the residues were below the limit of quantitation.

RESULTS AND DISCUSSION

Method Validation. Prior to beginning field sample analysis, the residue method was validated over the concentration range of 0.02−0.2 μg/g for all three canola matrixes. The results of the validation study are summarized in Table 3. The recoveries averaged 96 ± 7% for canola seed, 87 ± 6% for meal, and 89 ± 5% for refined oil.

Soil Characteristics. The physical and chemical properties of the soils used in the field residue trials are summarized in Table 1. Soil types included loamy sand, loam, silt loam, sandy clay loam, and sandy loam. The pH of the soils ranged from 6.1 to 7.8, the percent organic matter ranged from 1.2 to 4.6%, and the cation exchange capacity ranged from 7 to 33 meq/100 g.

Climatological Data. Temperature and rainfall data collected at the field sites were compared to 30-year averages (Owensby and Ezzel, 1992) and were considered to be within normal weather patterns encountered in canola production.

Field Sample Analysis. None of the canola seed samples from the 1X or the 3X trials contained ethalfluralin residues above the LOD. Because 3X represents the maximum theoretical concentration factor for canola processed products and no residues were detected in the raw agricultural commodity, processing of the canola seed into the oil and meal fractions was not required by EPA guidelines for residue processing studies.

Chromatograms. Typical chromatograms for the determination of ethalfluralin in canola seed are illustrated in Figure 3 (ethalfluralin standard), Figure 4 (untreated control sample), Figure 5 (fortified recovery sample), and Figure 6 (treated sample). Chromatograms for meal and refined oil were similar to those for canola seed.

Linearity. The linearity of the detector was determined using six calibration standards ranging in concentration from 0.003 to 0.1 μg/mL. The correlation coefficient (r^2) for the power regression equation describing the detector response as a function of concentration of the standard curve solutions was greater than

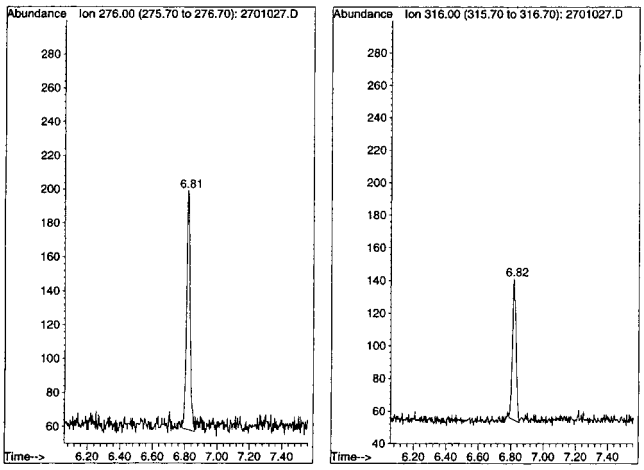

Figure 3. Typical chromatogram of a 0.005-μg/mL standard, equivalent to 0.020 μg/g of ethalfluralin in canola seed.

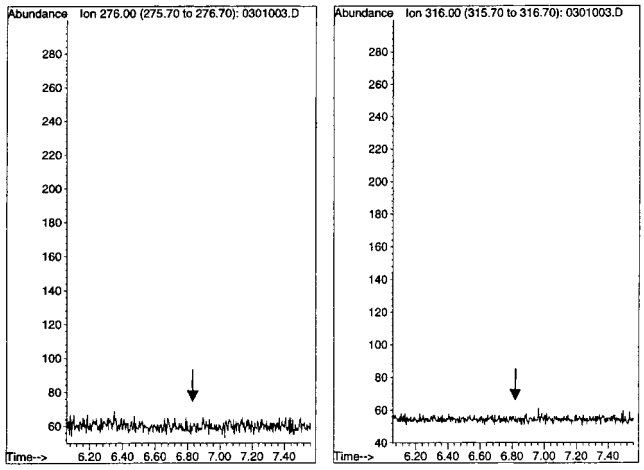

Figure 4. Typical chromatogram of an untreated control canola seed sample containing no detectable residue of ethalfluralin.

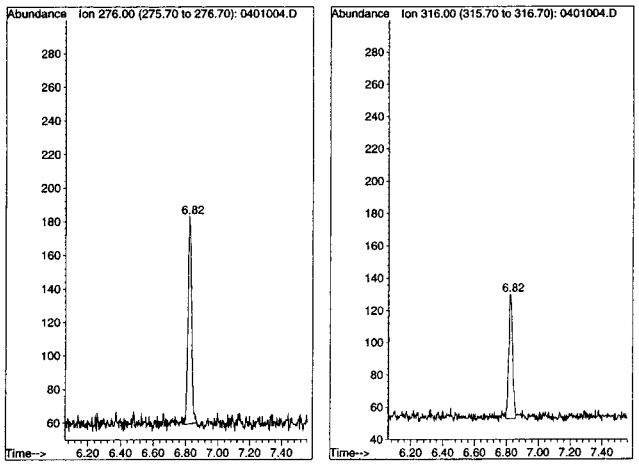

Figure 5. Typical chromatogram of an untreated control canola seed sample fortified with 0.02 μg/g of ethalfluralin, equivalent to an 88% recovery.

0.996 for all of the calibration curve determinations during method validation. The power regression exponent ranged from 1.05 to 1.09.

Limits of Detection and Quantitation. The calculated values for the LOD (3*s*) and LOQ (10*s*) are presented in Table 4. For all three matrixes, the calculated LOD ranged from 0.003 to 0.005 μg/g. These calculated values support using a method LOD of 0.006 μg/g, which was further supported by the presence of detectable peaks in chromatograms of control samples fortified at 0.006 μg/g (Table 3).

Likewise, the calculated LOQ ranged from 0.011 to 0.016 μg/g (Table 4). These calculated values supported the validated method LOQ of 0.02 μg/g for all three sample matrixes. A typical chromatogram demonstrating the determination of ethalfluralin in canola seed at the validated LOQ is shown in Figure 5.

Specificity. GC/MSD is a highly specific detection system. As illustrated in the typical chromatogram for the untreated control sample (Figure 4), no interference occurred in canola samples at the retention time of ethalfluralin. In addition, GC/MSD can selectively detect ethalfluralin in the presence of similar dinitroaniline herbicides (West et al., 1988).

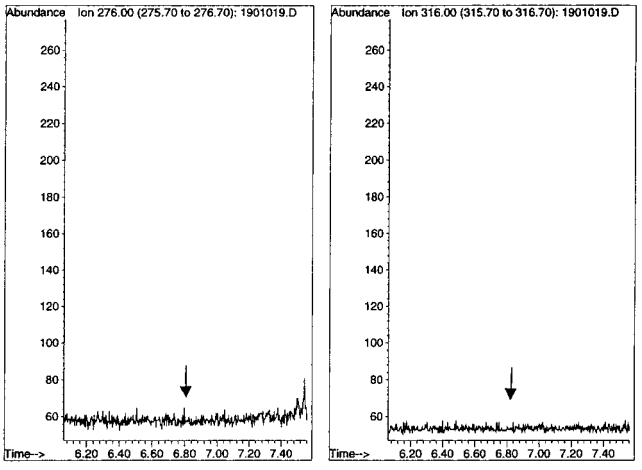

Figure 6. Typical chromatogram of a treated canola seed sample containing no detectable residue of ethalfluralin.

Table 4. Calculated Limits of Detection and Quantitation for Ethalfluralin in Canola Seed, Meal, and Refined Oil

sample type	*n*	μg/g added	μg/g found (mean ± *s*)	LOD[a]	LOQ[b]
canola seed	9	0.02	0.0195 ± 0.0016	0.0048	0.0160
canola meal	10	0.02	0.0174 ± 0.0016	0.0048	0.0160
canola oil	8	0.02	0.0180 ± 0.0011	0.0033	0.0110

[a] Calculated limit of detection (μg/g), calculated as 3*s*. [b] Calculated limit of quantitation (μg/g), calculated as 10*s*.

CONCLUSIONS

Residue methodology has been validated for the determination of ethalfluralin in canola seed, meal, and refined oil. The accuracy, precision, and specificity of the methodology make it suitable for residue monitoring or tolerance enforcement. No residues of ethalfluralin occurred in canola seed treated with Sonalan 10G or Sonalan HFP at the maximum labeled application rates. Because 3X is the maximum theoretical concentration factor for canola processed commodities and no ethalfluralin residue occurred in canola seed from 3X the maximum application rate, residues above the 0.006-μg/g LOD would not be expected to occur in canola meal or refined oil processed from treated canola seed.

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