ORGANICS LABORATORY
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Method No.	Edition	Revision
L35011	1	

Subject:

Determination of Linuron in Soil using High Performance Liquid Chromatography

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References: J. Ag. Food Chem., 1980, 28, 974-978

1.0 SCOPE

This method describes the procedure for extracting and analyzing soil samples for Linuron residue. The screening limit for this residue is 0.01 ppm in soil.

2.0 PRINCIPLE

Linuron is extracted from soil by shaking with methanol and water. The extract is then concentrated by rotary evaporation until near dryness. The remaining extract is diluted to 5ml with 45/55 acetonitrile/water and filtered through a 45 micron filter. The extract is then analyzed by HPLC.

3.0 CHEMICALS AND SOLUTIONS

Acetonitrile, J.T. Baker HPLC grade, or equivalent

Linuron Standard Reference Material

Methanol, J.T. Baker reagent grade, or equivalent

Water, deionized

4.0 APPARATUS

4.1 Equipment

4.1.1 Centrifuge, capable of holding 250ml bottles and capable of 1500rpm

4.1.2 Centrifuge bottle, 250ml polypropylene, w/lid

4.1.3 Cotton, absorbent

4.1.4 Filter, 45 micron, for 4.1.13

4.1.5 Funnel, 65mm

4.1.6 Graduated cylinder, 10ml

4.1.7 Graduated cylinder, 100ml

4.1.8 Rotary Film Evaporator

4.1.9 Ultrasonic bath

4.1.10 Shaker, Reciprocating, capable of 200rpm

4.1.11 Syringe, 2.5-5ml capacity disposable

4.2 Glassware

4.2.1 Boiling Flask, 500ml

4.2.2 Pipettes, Disposable Pastuer

4.2.3 Vial, Auto Sampler (4ml), w/screw cap and 7mm thin Teflon seal

4.2.4 Vial, Solvent Saver (7ml), w/screw cap

4.2.5 Volumetric Flask, 5ml, w/lid

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4.3 HPLC

4.3.1 Column: Dupont Zorbax ODS, number 880952.702, 4.6mm X 25cm, or equivalent.

4.3.2 Detector: Shimadzu SPO-6A variable wavelength UV, operable at 254nm, or equivalent.

4.3.3 Injector: Shimadzu SIL-6B, capable of reproducibly injecting 20uL.

4.3.4 Pump: Waters model 510 or equivalent, capable of operating at pressures up to 2500psi with a solvent flow rate of 1.5 ml/minute.

4.3.5 Recorder: Shimadzu C-R3A Chromatopac, capable of reporting peak height, area and retention time, or equivalent.

5.0 ANALYTICAL PROCEDURE

5.1 Extraction

5.1.1 Weigh 50 grams (to 0.005 g) of well homogenized, air dried soil into a 250 ml polypropylene centrifuge bottle fitted with a rubber ringed screw cap.

5.1.2 Add 10 ml of deionized water and 100 ml of methanol to the soil sample and shake for one hour at 200rpm on the reciprocating shaker.

5.1.3 Centrifuge for fifteen minutes at 1500 rpm. Place a small plug of absorbent cotton in a 65mm funnel and decant the solution through the cotton into a 500ml boiling flask. Keep the funnels and flasks together and set aside.

5.1.4 Add 100 ml of methanol to the 250 ml centrifuge bottle, cap and shake to break up soil then return to shaker for one hour.

5.1.5 Centrifuge again for fifteen minutes at 1500rpm. Decant the solution again through the cotton into the 500 ml boiling flask, combining this decantant with the decantant from step 5.1.3.

5.1.6 Concentrate the combined solutions on a rotary evaporator at 40 degrees C., until about 1ml of solution remains.

5.1.7 Using a disposable pastuer pipette, quantitatively transfer the remaining solution to a 5ml volumetric flask, then using about 1ml aliquotes of acetonitrile/water (45/55) and the sonic cleaner, remove as much of the remaining residue from the boiling flask as possible and add it to the volumetric flask. Bring the sample to 5ml with the acetonitrile /water.

5.1.8 Filter the resulting solution from 5.1.7 through a 0.45 micron filter, using a disposable syringe, into a 7 ml saver vial. Transfer 2ml of the filtered solution to an autosampler vial for analysis and archive the remaining extract.

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6.0 High Performance Liquid Chromatographic Analysis

The final determination of Linuron is performed on a HPLC equipped with a reverse phase column and a UV detector at 254nm, a pump flow rate of 1.5ml/minute of mobile phase (45/55 acetonitrile/water) and a chart speed of 1cm/minute.

6.1 Standardization

6.1.1 Make up standard solutions by serially diluting a known amount of the reference standard in mobile phase.

6.1.2 Inject constant volumes (20uL) of known amounts of standard on the HPLC.

6.1.3 Measure the standard peak areas.

6.1.4 Plotting peak area vs. concentration, calculate the best fit line using linear regression.

6.2 Residue Determination

6.2.1 Inject the solution from 5.1.8 at the same volume used for the standards (20uL).

6.2.2 Measure the peak area.

6.2.3 Determine the concentration (ppm) of Linuron in the sample aliquot injected by inserting the peak area into the equation of the line obtained in 6.1.4.

6.2.4 Calculate the residue as follows:

$$ppm(sample) = \frac{(\text{extraction conc ug/mL})(\text{dilution ml})(\text{dilution factor})}{(\text{grams of sample})}$$

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