

Analytical Study
HOE9236V

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OBJECTIVES

The objectives of this study were to validate the method DFG S 19 for the determination of the residues of fenpyroximate (HOE 094552) and metabolite M-1 (HOE 112573) in soil.

The analyses were performed with untreated soil originating from project number ER 92 DEU 820.

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MATERIALS AND METHODS

TEST SUBSTANCES

The analytical standards used for fortification and quantification in this study, supplied by HOECHST AG, are described below.
(Data as provided by the sponsor)

HOE 094552, NNI-850:

Molecular formula: $C_{24}H_{27}N_3O_4$

Molecular mass: 421.5 g/mol

Common name: fenpyroximate

Chemical name (IUPAC): tert-butyl (E)- α -(1,3-dimethyl-5-phenoxy-pyrazol-4-methyleneamino-oxy)-p-toluate

Lot No. NN-002

Purity 99.76 %

HOE 112573, NNI-850Z:

Molecular formula: $C_{24}H_{27}N_3O_4$

Molecular mass: 421.5 g/mol

Common name: M-1

Chemical name (IUPAC): tert-butyl (Z)- α -(1,3-dimethyl-5-phenoxy-pyrazol-4-methyleneamino-oxy)-p-toluate

Lot No. 1AA0107P

Purity 97.7 %

METHOD

DFG-method S 19

Title: Organochlorine, Organophosphorus, Nitrogen-Containing and Other Pesticides
Gas-chromatographic determination after cleanup by gel permeation chromatography and silica gel minicolumn chromatography

Authors: W. Specht and H.P. Thier

Literature: Manual of Pesticide Residue Analysis
DFG, Dt. Forschungsgemeinschaft, Pesticides Comm.,
edited by H.P. Thier and H. Zeumer
Weinheim, New York, Vol.1 (1987)
ISBN 3-527-27010-8 (VCH Verlagsgesellschaft)
ISBN 0-89573-592-X (VCH Publishers)

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Outline of method

Soil is extracted with acetone. Water is added beforehand in an amount that takes full account of the natural water content of the sample so that during extraction the acetone : water ratio remains constant at 2 : 1 v/v. The extract is saturated with sodium chloride and diluted with dichloromethane, resulting in separation of excess water. The evaporation residue of the organic phase is cleaned up by gel permeation chromatography on Bio Beads S-X3 polystyrene gel, using a mixture of cyclohexane and ethyl acetate (1+1) as eluant and an automated gel permeation chromatograph. The residue-containing fraction is concentrated and after supplemental cleanup on a small silica gel column analyzed by gas chromatography using a widebore capillary column and a nitrogen sensitive detector.

Elution volumes for gel permeation chromatography:

Substance specific collect volumes:

Fenpyroximate: 90 - 130 ml
M-1 : 95 - 130 ml

Adjusted collect volume: 90 - 150 ml

Elution from the mini silica gel column with Eluat 4, a mixture of toluene and acetone (80 + 20).

Modifications to the method were not necessary for the determination of soil.

Calculations were performed by the peak height method using external standard.

Limit of determination: 0.01 mg/kg

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GLC CONDITIONS

Gas chromatograph: VARIAN 3700 with autosampler

Column: fused silica capillary column DB 1,
15 m
internal diameter 0.53 mm
film thickness 0.15 μm

Column temperature: initial 150°C, hold for 2 min,
with a rate of 10°C/min to 240°C,
hold for 5 min

Injection port temperature: 230°C

Detector: alkali flame ionisation detector
(N-FID)
temperature 250°C

Gas flow rates: carrier : helium 30 ml/min
detector: air 175 ml/min
hydrogen 4.5 ml/min

Injection volume: 5 μl splitless

Integrator: HP 3365 ChemStation

Standard solutions: fenpyroximate: 0.0600 and 0.600 $\mu\text{g/ml}$
metabolite M-1: 0.0594 and 0.594 $\mu\text{g/ml}$
(in toluene)