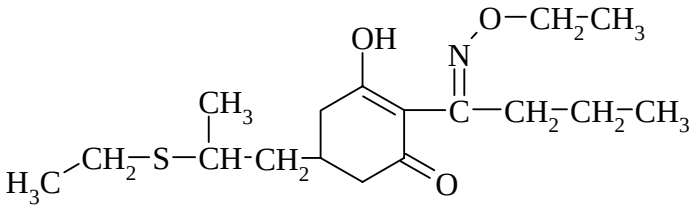
SETHOXYDIM
401



ISO common name	Sethoxydim
Chemical name	(±)-2-(1-Ethoxyiminobutyl)-5-[2-(ethylthio)propyl]-3-hydroxycyclohex-2enone (IUPAC)M 2-[1-(ethoxy-imino)butyl-5-2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one (CA; *74051-80-2*)
Empirical formula	$C_{17}H_{29}NO_3S$
RMM	327.5
b.p.	More than 90 °C at 5.32 mPa
v.p.	Less than 1.33×10^{-4} Pa at 20 °C
Solubility	In water: 25 mg/l at 20 °C and at pH=4; readily soluble in most organic solvents
Description	Colourless oily liquid
Stability	Unstable in acidic solutions and to heat
Formulations	Emulsifiable concentrates

SETHOXYDIM TECHNICAL
[*]**401**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of sethoxydim with respect to the internal standard for the sample solution should not deviate by more than 2% from that for the calibration solution.

[*] CIPAC method 1988. Prepared by the Japanese Committee (JAPAC). Chairman: Dr T Suzuki. Based on a method supplied by Nippon Soda Co., Ltd.

2.2 Infrared. Separate sethoxydim from solvent of the sample by extraction (sethoxydim dissolves in alkaline solutions but does not in acidic solutions). Prepare standard sethoxydim from standard sethoxydim-Li. Scan the sethoxydim from 4000 - 400 cm^{-1}. The spectrum of the sample should not differ significantly from that of the standard.

3 Sethoxydim

OUTLINE OF METHOD Sethoxydim is extracted with *n*-hexane after adding acetic acid aqueous solution to the sample. The concentration of sethoxydim is determined by normal phase high performance liquid chromatography with UV detection using thymol as internal standard.

REAGENTS

n-Hexane HPLC grade
Ethyl acetate HPLC grade
Acetic acid HPLC grade
Ethanol HPLC grade
Water purified by distillation or demineralization, free from organic contaminants
Eluent Mix *n*-hexane (1000 ml) with ethyl acetate (10 ml), acetic acid (10 ml) and ethyl alcohol (0.5 ml).
The retention time for sethoxydim tends to fluctuate comparatively more than for thymol (internal standard). Therefore incomplete separation of both peaks may occur depending on the analytical conditions. In this case the use of a little more ethanol is recommended.
Sethoxydim-Li analytical standard of known purity, prepared from pure sethoxydim and lithium hydroxide hydrate
5-Methyl-2-isopropyl-1-phenol (thymol) internal standard
Internal standard solution Weigh (to the nearest 0.01 g) 2 g (*r* g) of thymol into a 100 ml volumetric flask. Dissolve in *n*-hexane (40 ml), make up to the mark, and mix thoroughly.
Acetic acid aqueous solution 20% Weight 20 g of acetic acid into a 100 ml volumetric flask. Dilute with water to the mark.
Calibration solution. Weigh (to the nearest 0.1 mg) about 200 mg (*s* mg) sethoxydim-Li analytical standard into a 50 ml glass-stoppered Erlenmeyer flask, add 20% acetic acid aqueous solution (1 ml), and let it stand for 5 minutes. Then add by pipette internal standard solution (20.0 ml) and *n*-hexane (20.0 ml). Stopper the flask and shake for 5 minutes. Let stand this solution for about 1 h until the *n*-hexane layer becomes transparent. Transfer by a micropipette 200 µl

of the *n*-hexane layer into a 4 ml sample vial and dilute with *n*-hexane (1 ml). Mix thoroughly.

APPARATUS

High performance liquid chromatograph equipped with a constant flow pump, an injector and a variable wavelength UV detector
Column stainless steel, 250 × 4.0 (i.d.) mm, packed with Lichrosorb CN, 5 µm, with a theoretical plate number of at least 5,000.
Electronic integrator
Recorder

PROCEDURE

(a) Operating conditions (typical):

Flow rate	2 ml/min
Detector wavelength setting	280 nm
Detector sensitivity	Select a setting so that the peaks produce a recorder deflection of 80-90% full scale.
Injection volume	3 µl
Chart speed	0.5 cm/min
Retention times	sethoxydim: 3.3 min thymol: 4.4 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample (*w* mg) to contain about 200 mg of pure sethoxydim into a 50 ml glass-stoppered Erlenmeyer flask, and add 20% acetic acid aqueous solution (1 ml). Then add by pipette internal standard solution (20.0 ml) and *n*-hexane (20.0 ml). Stopper the flask and shake for 5 minutes. Let stand this solution for about 1 h until the *n*-hexane layer becomes transparent. Transfer by a micropipette 200 l of the *n*-hexane layer into a 4 ml sample vial and dilute with *n*-hexane (1 ml). Mix thoroughly.

(c) Determination. Let stabilize the HPLC conditions (retention time, calibration factors) for more than 1 h. Inject 3 µl aliquots of the calibration solution, then inject in duplicate 3 µl aliquots of sample solution followed by an injection of the calibration solution. Determine the peak areas by electronic integration.

(d) Calculation. Determine the response factor from the peak areas of the calibration solution injections preceding and following the two sample injections. Average these values. The response factor is:

$$f = \frac{I_r \times s}{H_s \times r}$$

where:

I_r = peak area of thymol in the calibration solution
H_s = peak area of sethoxydim in the calibration solution
r = mass of thymol (g)
s = mass of sethoxydim-Li analytical standard (mg)

$$\text{Sethoxydim content} = \frac{H_w \times r \times f \times P \times 0.9822}{I_q \times w} \text{ g/kg}$$

where:

H_w = peak area of sethoxydim in the sample solution
I_q = peak area of thymol in the sample solution
r = mass of thymol (g)
w = mass of sample taken (mg)
f = average response factor
P = purity of sethoxydim-Li analytical standard (g/kg)
0.9822 = the ratio of molecular mass of sethoxydim to that of sethoxydim-Li

Repeatability r = 8.9 g/kg at 510 g/kg active ingredient content
Reproducibility R = 11.0 g/kg at 510 g/kg active ingredient content

SETHOXYDIM EMULSIFIABLE CONCENTRATES
[*]**401**/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests.
2.2 HPLC. As for sethoxydim technical **401**/TC/M/2
2.3 Infrared. Prepare an *n*-hexane solution of the sample. Separate sethoxydim from interfering ingredients of the formulation by filtration with silica gel, and continue as for sethoxydim technical **401**/TC/M/2.

3 Sethoxydim As for sethoxydim technical **401**/TC/M/3
Repeatability r = 3.6 g/kg at 207 g/kg active ingredient content
2.1 g/kg at 132 g/kg active ingredient content

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Reproducibility R = 5.6 g/kg at 207 g/kg active ingredient content
4.6 g/kg at 132 g/kg active ingredient content