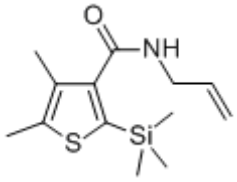
SILTHIOFAM

635



ISO Common name	Silthiofam
Chemical name	N-allyl-4,5-dimethyl-2-(trimethylsilyl)thiophene-3-carboxamide (IUPAC) 4,5-Dimethyl-N-(2-propenyl)-2-(trimethylsilyl)-3-Thiophenecarboxamide (CA)
CAS No.	175217-20-6
Empirical formula	$C_{13}H_{21}NOSSi$
RMM	267,46
m.p.	88°C (361K)
v.p.	(8.1±0.7) x 10^{-2} Pa (20°C)
Solubility	Water: 39.9mg/l n-heptane: 15.5g/l p-xylene: >250g/l 1,2-dichloromethane: >250g/l methanol: >250g/l acetone: >250g/l ethyl acetate: >250g/l At 19.5 ± 0.5°C
Description	White crystalline powder
Formulations	Flowable concentrate for seed treatment (FS)

SILTHIOFAM

635/TC/m/-

1. Sampling

Take at least 10 g.

2. Identity tests

2.1. HPLC

Use the HPLC method described below. The retention time of silthiofam for the sample solution should not deviate by more than 0.2 min from that of the calibration solution.

2.2. UV-spectrum

Use the HPLC method described below using a photodiode array detector (PAD) and scan from 210 to 400nm. The spectrum obtained from the sample should not differ significantly from that of the standard (Figure 1).

3. Silthiofam

3.1. Outline of method

Silthiofam is determined by means of reversed phase high performance liquid chromatography using UV-detection at 260nm and internal standardization.

3.2. Reagents

Acetonitrile: HPLC grade

Water: Milli-Q grade

Silthiofam: standard of known purity

Diethyl phthalate (DEP): standard of known purity (minimum requirement 99%)

Mobile phase: Acetonitrile + Milli-Q water (50+50) (v/v)

Calibration Solution: Weigh in duplicate (to the nearest 0.1 mg) 100 mg of silthiofam and 400 mg DEP into separate volumetric flasks (50 ml). Fill to the mark with acetonitrile. Mix thoroughly by shaking for 5 min. 5 ml of this solution is transferred into a volumetric flask of 25 ml and filled to the mark with acetonitrile (calibration Solutions C_1 and C_2).

3.3. Apparatus

High performance liquid chromatography equipped with a detector suitable for operation at 260 nm, constant temperature column compartment and an injector capable of delivering 5µl.

Column: Stainless steel, 125 x 4.6mm (i.d.) packed with Lichrospher 100 C18 5µm, or equivalent

Electronic integrator of data system

Mechanical shaker

Filtration unit: equipped with a PTFE membrane, 0.2 µm

3.4. Procedure

a) Liquid chromatographic conditions (typical)

Column	Stainless steel, 125 x 4.6mm (i.d.) packed with Lichrospher 100 C18 5µm, or equivalent
Mobile phase	Acetonitrile + Milli-Q water (50+50)(v/v)
Temperature	40°C
Flow rate	Isocratic: 1.2 ml/min
Detection wavelength	260 nm
Injection volume	5 µl
Retention time	Approximately: Silthiofam: 7.5 min DEP: 3.5 min

b) Linearity check:
Check the linearity of the detector response by injecting 5µl of silthiofam reference solutions (enriched with the same amount of DEP as in the calibration solution) at 0.5, 1 and 2 times that of the calibration solution. A minimum correlation (r) of >0.99 should be obtained.

c) System equilibration:
Inject 5µl portions of the calibration solution C_1 and repeat the injections until the peak areas (of both silthiofam and DEP) deviate by less than ±1.0% of the mean of two consecutive injections. Then inject consecutively two 5µl portions of the second calibration solution C_2 the mean response factor for this calibration solution should not deviate by more than 1.0% from that of the first calibration solution (C_1), otherwise prepare new calibration solutions.

d) Sample preparation:
Prepare sample solutions in duplicate. Weigh (to the nearest 0.1 mg) 100 mg of silthiofam technical into separate 50 ml volumetric flasks and add 400 mg of DEP internal standard (to the nearest 0.1mg). Add acetonitrile to the mark and shake the solution for 5 minutes.
Transfer 5 ml of these solutions in separate 25ml volumetric flasks and dilute with acetonitrile to the mark. Mix thoroughly and filter the solution trough a 0.2 µm PTFE-membrane filter (S_{TC1}, S_{TC2})

e) Determination:
Inject in duplicate 5 µl portions of each sample solution bracketing them by injections of the

calibration solution as follows:

C_1, $S_{TC1\text{-}1}$, $S_{TC1\text{-}2}$, C_2, $S_{TC2\text{-}1}$, $S_{TC2\text{-}2}$, C_1 …

Determine the relevant peak areas

f) Calculation:

Calculate the mean value of each pair of response factors bracketing the injections of a sample and use this value for calculation the silthiofam contents of the bracketed sample injection.

$$f_i = \frac{H_S}{I_r} \times \frac{r \times P}{s}$$

$$\text{Silthiofam content} = \frac{\frac{H_w}{I_q} \times q}{f \times w} \times 100\ (\%\,m/m)$$

Where:

f_i	= individual response factor
H_S	= Area silthiofam in the calibration solution
I_r	=Area internal standard in the calibration solution
r	=Mass of the internal standard in the calibration solution (g)
s	=Mass of silthiofam in the calibration solution (g)
P	=Purity of the silthiofam standard
H_w	=Area of silthiofam in the sample solution
I_q	=Area of the internal standard in the sample solution
q	=Mass of the internal standard in the sample solution (g)
w	=Mass of sample taken (g)
f	=mean response factor.

Repeatability r = 0.341 % (m/m) at 99.41 % (m/m) active ingredient content.

Reproducibility R = 0.422 % (m/m) at 99.41 % (m/m) active ingredient content.

SILTHIOFAM Flowable Concentrate For Seed treatment

635/FS/m/-

1. Sampling

Take at least 10 g.

2. Identity tests

2.1 HPLC

As for silthiofam technical 635/TC/m/2.1

2.2 IR-spectrum

Scan a droplet of the flowable concentrate for seed treatment from 4000 to 520 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard spectrum (Figure 3).

3. Silthiofam

As for 635/TC/m/3 except:

Change 'PROCEDURE (d) sample preparation' as follows:

Prepare sample solutions in duplicate. Weigh (to the nearest 0.1 mg) 850 mg of FS formulation into separate 50 ml volumetric flasks, add 2 ml water to dissolve the suspension concentrate. Add 400 mg of DEP internal standard (to the nearest 0.1mg). Add acetonitrile to the mark and shake the solution for 15 minutes.

Transfer 5 ml of these solutions in separate 25ml volumetric flasks and dilute with acetonitrile to the mark. Mix thoroughly and filter the solution trough a 0.2 µm PTFE-membrane filter (STC1, STC2)

Repeatability r = 0.068 % (m/m) at 11.666 % (m/m) active ingredient content.

Reproducibility R = 0.115 % (m/m) at 11.666099 % (m/m) active ingredient content.

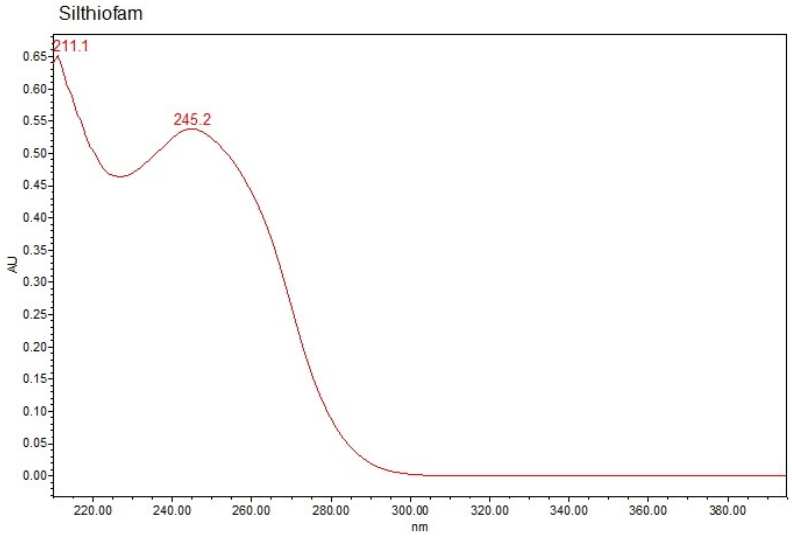


Figure 1: UV-spectrum of silthiofam.

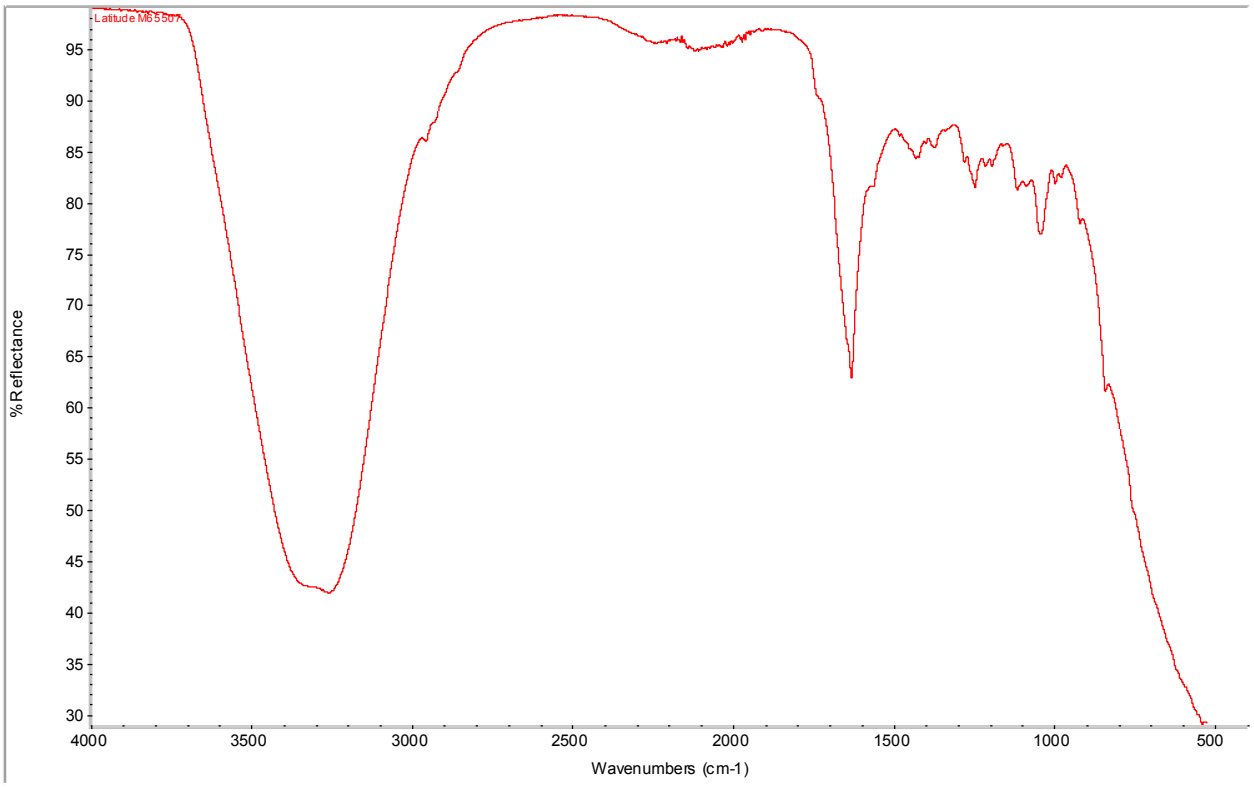


Figure 2: Infrared spectrum of silthiofam FS.

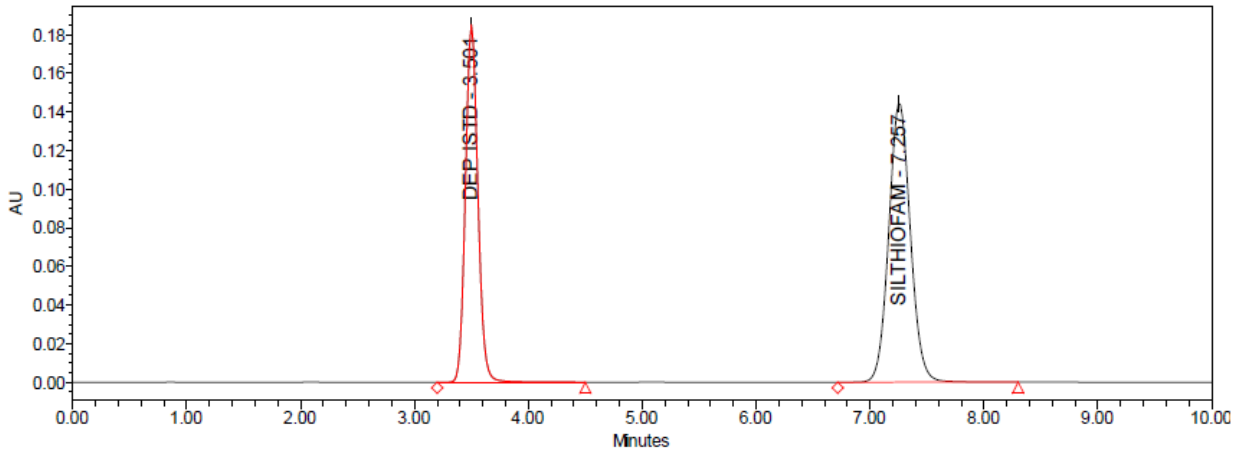


Figure 3: Example chromatogram of thiofam FS.