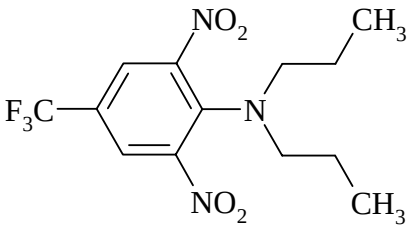
TRIFLURALIN
183



ISO common name	Trifluralin
Chemical name	2,6-Dinitro-*N*,*N*-dipropyl-4-trifluoromethyl-aniline (IUPAC); 2,6-dinitro-*N*,*N*-dipropyl-4-(trifluoromethyl) benzeneamine (CA; *1582-09-8*)
Empirical formula	$C_{13}H_{16}F_3N_3O_4$
RMM	335.3
m.p	48.5 - 49 °C
v.p.	1.4×10^{-4} Pa at 25 °C
Solubility	In water: less than 1 mg/l; acetone: 400 g/l; xylene: 580 g/l
Description	Orange crystalline solid
Stability	Stable but susceptible to photodecomposition
Formulations	Emulsifiable concentrates and granules

TRIFLURALIN TECHNICAL
[*]**183**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 GLC. Use the GLC method below. The relative retention time of trifluralin with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.
2.2 Infrared. Prepare potassium bromide discs from the sample and from trifluralin standard. Scan the discs from 400-4000 cm^{-1}. The spectrum from the sample should not differ significantly from that of the standard.

3 Trifluralin

3.1 Gas chromatographic method

OUTLINE OF METHOD The sample is dissolved in acetone and trifluralin is determined gas chromatography using flame ionisation detection and internal standardisation.

REAGENTS

Trifluralin standard of known purity
Acetone HPLC
Diisobutyl phthalate internal standard
Internal standard solution. Weigh into a volumetric flask (250 ml) diisobutyl phthalate (625 mg), dissolve in and dilute to volume with acetone, and mix.
Calibration solution. Weigh (to the nearest 0.1 mg) 160 mg of trifluralin standard (*s* mg) into a volumetric flask (100 ml). Dissolve in and dilute to volume with acetone and mix. Transfer by pipette 25.0 ml of this solution to a volumetric flask, add by pipette internal standard solution (10.0 ml), fill to the mark with acetone, and mix.

APPARATUS

Gas chromatograph fitted with a flame ionisation detector; capable of temperature programming
Column glass, 1.5 m × 6 mm (i.d.) packed with 5 % DC 200 on Chromosorb W-HP, 80 to 100 mesh. Condition new columns at 230 °C overnight with nitrogen carrier gas

[*]AOAC-CIPAC method 1975.

Electronic integrator or *data system*

PROCEDURE

(a) Operating conditions (typical):

Oven temperature	programmed from 133 to 190 °C at 8 °C per min
Injection port temperature	205 °C
Detector temperature	275 °C
Injection volume	2.5 µl
Flow rate carrier gas	nitrogen, 60 ml/min
Flow rates other gases	as recommended for the particular detector
Retention time	trifluralin: 5.5 min internal standard: 7.5 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain 160 mg of trifluralin (*w* mg). Dissolve in and dilute to volume with acetone and mix. Transfer by pipette 25.0 ml of this solution to a volumetric flask, add by pipette internal standard solution (10.0 ml), fill to the mark with acetone, and mix.

(c) Determination. Inject into the gas chromatograph 2.5 µl portions of the calibration solution. Start temperature programming to give symmetrical peaks about 70 % full scale and retention times as indicated under *(a)*. Repeat the injection until the ratio of the trifluralin to the internal standard peak areas agree to within 1% of their mean. Without changing conditions inject 2.5 µl of the sample solution. Determine the peak areas and calculate the trifluralin to internal standard peak area ratios for the calibration and sample solutions (*R'* and *R* respectively).

(d) Calculation

$$\text{Trifluralin content} = \frac{R \times s \times P}{R' \times w} \text{1g/kg}$$

where:

R = trifluralin to internal standard peak area ratio in the sample solution
R' = trifluralin to internal standard peak area ratio in the calibration solution
s = mass of trifluralin in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of trifluralin standard (g/kg)

3.2 Ultraviolet spectroscopic method

OUTLINE OF METHOD The sample is dissolved in *n*-hexane, purified by chromatography on a Florisil column and determined by ultraviolet spectroscopy at 376 nm.

REAGENTS

n-Hexane UV spectroscopic quality
Trifluralin standard of known purity
Calibration solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) 125 mg trifluralin standard (*s* mg). Dissolve in and fill to the mark with *n*-hexane, and mix thoroughly.
Florisil 100 to 200 mesh, tested by adding 5 ml of the calibration solution to the prepared column and eluting as below. The elution volume should be between 80 and 100 ml. If the elution volume does not fall in this range, adjust the water content by trial and error to obtain the proper elution volume (add water to decrease, dry at 130°C to increase the elution time; about 4 % water normally gives the correct elution time).
Sodium sulphate anhydrous

APPARATUS

Ultraviolet spectrophometer
Quartz cell pathlength 1 cm
Chromatographic columns 25 × 400 mm glass tubes with PTFE stopcocks

PROCEDURE

(a) Preparation of column. Insert a glass wool plug in the bottom of the column (two columns required) and add with constant tapping of the columns sodium sulphate (5 g), standardised Florisil to a height of 50 mm, and sodium sulphate (5 g). With the stopcock open, add *n*-hexane (50 ml) and let drain the top of the column. Close the stopcock.

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 120 mg of trifluralin (*w* mg). Dissolve in and fill to the mark with *n*-hexane, and mix thoroughly.

(c) Determination. Transfer by pipette 5.0 ml of the sample solution to the column and 5.0 ml of the calibration solution to a second column. Treat each column as follows: Wash the sample into the column with small portions of *n*-hexane. Let each portion drain to the top of the column before adding the next. Fill the column with *n*-hexane, discarding the eluate until the band has moved about three-quarters of the column. Collect the eluate containing the trifluralin band (the first yellow-orange band to eluate) in a volumetric flask (100 ml). If the band requires more than 100 ml to eluate, replace the volumetric flask, evaporate, and transfer quantitatively to a volumetric flask (100 ml). Dilute to volume with *n*-hexane. Determine the absorbance of the sample and the calibration solutions (*A* and *A′* respectively) at 376 nm against

n-hexane as the references.

$$\text{Trifluralin content} = \frac{A \times s \times P}{A' \times w} \text{ 2g/kg}$$

where:
A = absorbance of trifluralin in the sample solution
A' = absorbance of trifluralin in the calibration solution
s = mass of trifluralin in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of trifluralin standard (g/kg)

TRIFLURALIN EMULSIFIABLE CONCENTRATES
*183/EC/M/-

1 Sampling. Take at least 1 l.

2 Identity tests. As for trifluralin technical **183**/TC/M/2.1.

3 Trifluralin. As for trifluralin technical **183**/TC/M/3.

TRIFLURALIN GRANULES
*183/GR/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests. As for trifluralin technical **183**/TC/M/2.1.

3 Trifluralin

3.1 Gas chromatographic method

OUTLINE OF METHOD The sample is extracted with acetone and

* AOAC-CIPAC method 1975.

determined as for the technical material.

REAGENTS AND APPARATUS As for trifluralin technical **183**/TC/M/3.1 together with:

Soxhlet apparatus
Extraction thimbles 33 x 80 mm

PROCEDURE As for trifluralin technical **183**/TC/M/3.1 except:

(b) Preparation of sample.
(i) Formulations containing more than 1 % of trifluralin. Weigh (to the nearest 0.1 mg) into an extraction thimble enough sample to contain about 160 mg (*w* mg) of trifluralin. Cover with glass wool and extract with acetone for 1 h beyond the time when no further colour is extracted. Evaporate to 60 ml on a steam bath with a stream of air directed to the flask. Quantitatively transfer to a volumetric flask (100 ml) with acetone. Dilute to volume and mix. Continue as for **183**/TC/3.1*(b)* from 'Transfer by pipette 25.0 ml ...'

(ii) Formulations containing less than 1 % of trifluralin. Weigh (to the nearest 0.1 mg) into an extraction thimble enough sample to contain about 40 mg (*w* mg) of trifluralin. Cover with glass wool and extract with acetone for 1 h beyond the time when no further colour is extracted. Evaporate to 60 ml on a steam bath with a stream of air directed to the flask. Quantitatively transfer to a volumetric flask (100 ml) with acetone, add by pipette internal standard solution (10.0 ml), fill to the mark with acetone, and mix.

(d) Calculation

$$\text{Trifluralin content} = \frac{R \times s \times P}{R' \times w \times F} \text{ 3g/kg}$$

where:
F = 1 for *(i)* and 4 for *(ii)*.

3.2 Ultraviolet spectroscopic method

OUTLINE OF METHOD The sample is extracted with chloroform and evaporated to dryness. The residue is dissolved in *n*-hexane, purified by chromatography on a Florisil column and determined by ultraviolet spectroscopy at 376 nm.

REAGENTS As for trifluralin technical **183**/TC/M/3.2 together with:

Chloroform
APPARATUS As for trifluralin technical **183**/TC/M/3.2 together with:

Soxhlet apparatus
Extraction thimbles 33 × 80 mm
Rotary evaporator

PROCEDURE As for trifluralin technical **183**/TC/M/3.2 except:

(b) Preparation of sample.
(i) Formulations containing more than 1 % trifluralin. Weigh (to the nearest 0.1 mg) into an extraction thimble enough sample to contain about 250 mg of trifluralin (*w* mg), cover with glass wool and extract with chloroform for 1 h beyond the time when no further colour is extracted. Quantitatively transfer the extract ot a volumetric flask (200 ml), dilute to volume with chloroform, and mix. Transfer by pipette 5.0 ml of this solution to a rotary evaporator and evaporate to dryness.
(ii) Formulations containing less than 1 % of trifluralin. Weigh (to the nearest 0.1 mg) into an extraction thimble enough sample to contain about 50 mg (*w* mg) of trifluralin and continue as in *(i)* except that 25.0 ml of the solution should be evaporated.

(c) Determination. Transfer the residue to the column using *n*-hexane and continue as for trifluralin technical **183**/TC/M/3.2(c).

$$\text{Trifluralin content} = \frac{A \times s \, x \, P \times F}{A' \times w} \text{ 4g/kg}$$

where:
F = 2 for *(i)* and 0.4 for *(ii).*