Triflumuron

rel. Impurities

1,3-bis(4-trifluoromethoxyphenyl)urea

and

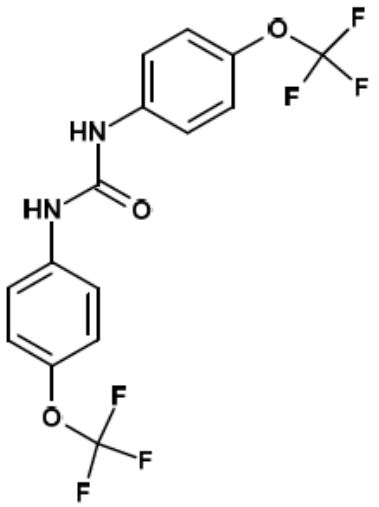
4-(trifluoromethoxy)aniline

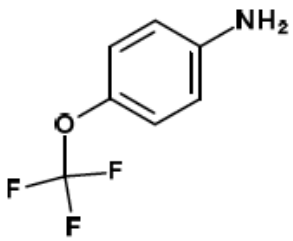
HPLC Method 5091/m

CIPAC Peer Validation

TRIFLUMURON, REL. IMPURITIES

1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline

Name	1,3-bis(4-trifluoromethoxyphenyl)urea
Synonyms	N,N'-bis-[4-(trifluoromethoxy)phenyl]urea AE B143886, BCS-AD26894
Three letter code	-
Structural formula	
Empirical Formula	$C_{15}H_{10}F_6N_2O_3$
Molecular Weight	380.2 g/mol
CAS no	78015-49-3

Name	4-(trifluoromethoxy)aniline
Synonyms	AE F069069, BCS-AC49934
Three letter code	-
Structural formula	
Empirical Formula	$C_7H_6F_3NO$
Molecular Weight	177.1 g/mol
CAS no	461-82-5

TRIFLUMURON TECHNICAL
*548/TC/M/-

1 Sampling. Take at least 100 g*. Grind the sample thoroughly in a mortar.
*(for this trial, less amount is provided; please grind the entire sample)

2 Identity tests.

2.1 HPLC. Use the HPLC method described below. The relative retention time of 1,3- bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline in the sample solution should not deviate by more than 2% from that of the calibration solution.

2.2 UV spectrometry. Record the UV spectrum during the HPLC determination. The UV spectrum obtained from the sample should not differ significantly from that of the standard. (Fig. 1 and Fig. 2)

3 1,3-bis(4-trifluoromethoxyphenyl)urea and 4- (trifluoromethoxy) aniline.

OUTLINE OF THE METHOD.

1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline contents are determined (g/kg) by reversed phase high performance liquid chromatography using UV detection at 226 nm and 258 nm and external standard calibration.

REAGENTS

1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline reference standards of known content
Acetonitrile (HPLC grade)
Purified water (HPLC grade)
Eluent A: purified water 40% (*v/v*) Eluent B: acetonitrile 60% (*v/v*)

* Based on a method supplied by Bayer Crop Science, Germany

APPARATUS

High performance liquid chromatograph equipped with an injection system capable to inject 5 µL and an UV spectrophotometric detector operated at 226 nm and 258 nm.

Liquid chromatography column, stainless steel, 125 x 4 (i.d.) mm, packed with Nucleosil 120-3 C 18; 3 µm or equivalent with the same selectivity.

Electronic integrator or data system Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Flow rate:	1 mL/min
Column temperature:	40 °C
Injection volume:	5 µL
Detector wavelength:	0.0 – 3.0 min: 226 nm (for 4-(trifluoromethoxy) aniline) 3.0 – 3.7 min: 258 nm 3.7 – 5.0 min: 300 nm (for triflumuron) 5.0 – 9.0 min: 258 nm (for 1,3-bis(4-trifluoro-methoxyphenyl)urea)
Retention time:	approx. 2.2 min for 4-(trifluoromethoxy)aniline approx. 5.2 min for 1,3-bis(4-trifluoromethoxy phenyl)urea
Total run time:	approx. 8 min

(b) Equilibration of the system. Pump sufficient mobile phase through the column to equilibrate the system. Inject 5 µL portions of the calibration solution C1 and repeat the injections until retention times and peak areas deviate by less than ± 1 % from the mean for three successive injections.

(c) Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) approximately 20 mg (*s* mg) of the reference standard of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline into separate volumetric flasks (50 mL). Add 20 mL acetonitrile and place the flasks in an ultrasonic bath for 15 min. Make up the flasks with acetonitrile to just below the calibration mark and allow to cool to

ambient temperature. Fill to the mark with acetonitrile and mix thoroughly.

Transfer 5 mL of this solution into a separate volumetric flask (50 mL) and make up the flasks with acetonitrile to just below the calibration mark. Allow to cool to ambient temperature, fill to the mark with acetonitrile and mix thoroughly.

Depending on the final concentrations of the analytes in the sample solution, transfer 3 mL of this solution into a separate volumetric flask (50 mL) and make up the flasks with acetonitrile to just below the calibration mark. Allow to cool to ambient temperature, fill to the mark with acetonitrile and mix thoroughly (Calibration solutions C1, C2) (Fig. 3).

(d) Sample preparation. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about approximately 0.12 mg (*w* mg) of 1,3-bis(4-trifluoromethoxy-phenyl)urea and 4-(trifluoromethoxy)aniline into separate volumetric flasks (50 mL). Add 20 mL acetonitrile and place the flasks in an ultrasonic bath for 15 min. Make up the flasks with acetonitrile to just below the calibration mark and allow to cool to ambient temperature. Fill to the mark with acetonitrile and mix thoroughly (Sample solutions S1, S2) (Fig. 4).

(e) Determination. Inject in duplicate each sample solution and bracket a series of sample solution injections by injections of the calibration solutions as follows: calibration solution 1, calibration solution 2, calibration solution 1, sample solution 1, sample solution 1, sample solution 2, sample solution 2, calibration solution 1, … (C1, C2, C1, S1, S1, S2, S2, C1, …).

Determine the peak areas of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline.

(f) Calculation: Calculate the response factors from the calibration solutions bracketing the injections of the sample solutions. Average the response factors of the calibration solutions preceding and following the sample solution injections. These must agree within ±1 % of the average otherwise repeat the determination. Calculate the content of the sample solutions.

$$f_i = \frac{sxP}{Hs}$$

1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline

$$\text{content (g/kg)} = \frac{Hw \times f}{w}$$

Where:

f_i = single response factor
f = average response factor
H_S = peak area of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline standard in the calibration solution
H_W = peak area of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline in the sample solution
S = weight of the 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline standard in the calibration solution (mg)
w = weight of the sample (mg)
P = purity of the 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline standard (g/kg)

TRIFLUMURON SUSPENSION CONCENTRATE
*548/SC/M/-

1 Sampling. Take at least 500 mL*. Shake the sample well prior to weighing.
*(for this trial less amount is provided)

2 Identity tests.

2.1 HPLC. As for 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline in Triflumuron 548/TC/M/-

2.2 UV spectrometry. As for 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline in Triflumuron 548/TC/M/-

3 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy) aniline.

As for 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline in Triflumuron 548/TC/M/- except

Disposable PTFE syringe filter compatible with organic solvents and a 0.45 µm pore diameter or centrifuge.

(d) Sample preparation. As for 1,3-bis(4-trifluoromethoxyphenyl)urea and 4- (trifluoromethoxy)aniline in Triflumuron 5091/TC/M/- except filter the sample solution through a disposable filter or centrifuge the sample solution (Sample solutions S3, S4) (Fig. 5).

* Based on a method supplied by Bayer Crop Science, Germany

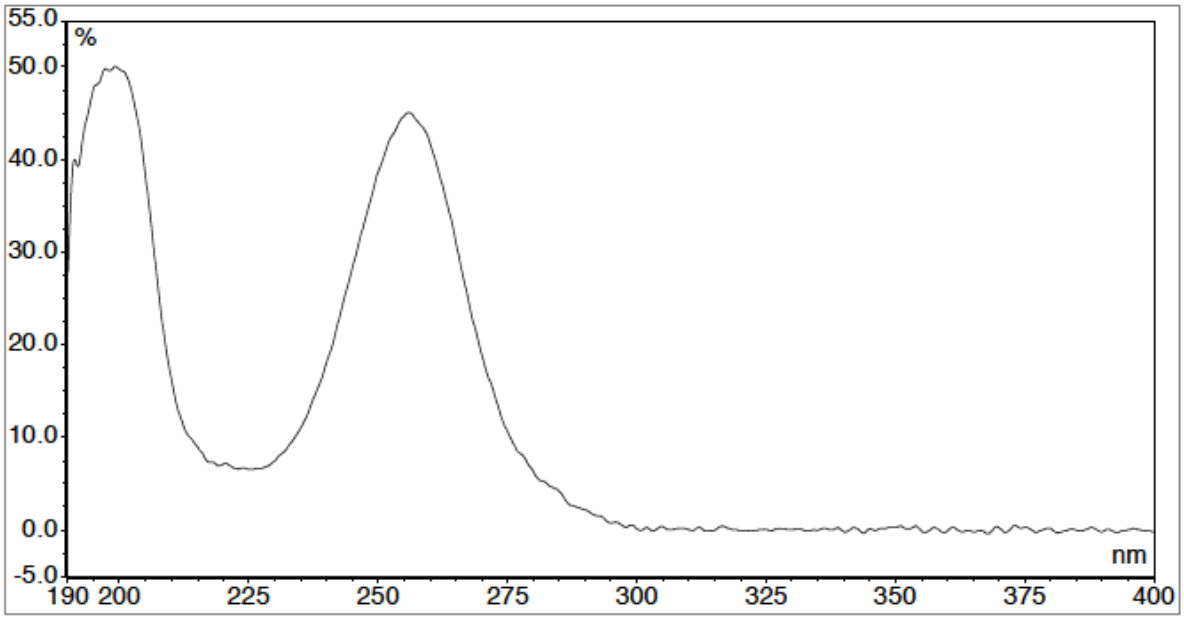


Fig. 1 UV-Spectrum of 1,3-bis(4-trifluoromethoxyphenyl)urea

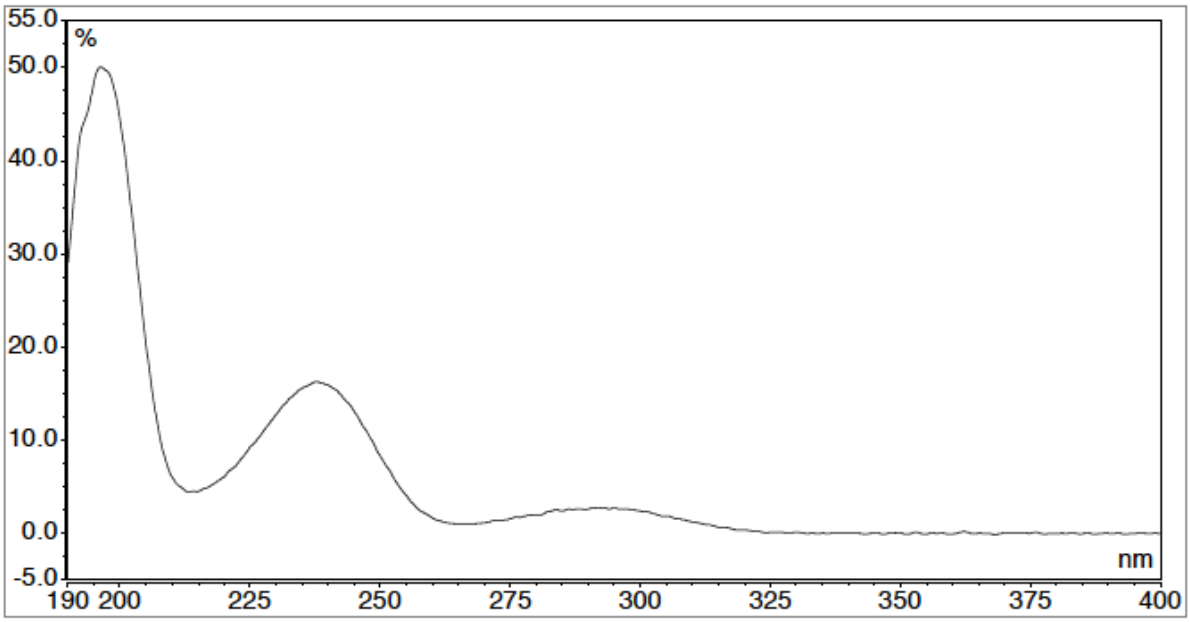


Fig. 2 UV-Spectrum of 4-(trifluoromethoxy)aniline

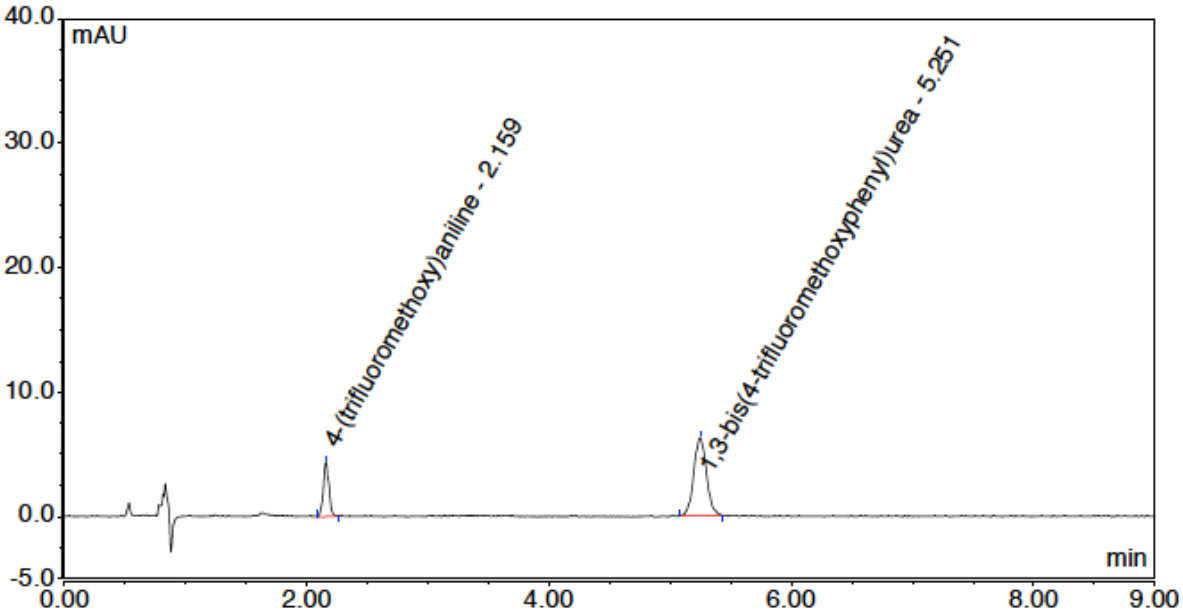


Fig. 3 Chromatogram of 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline Analytical Standards

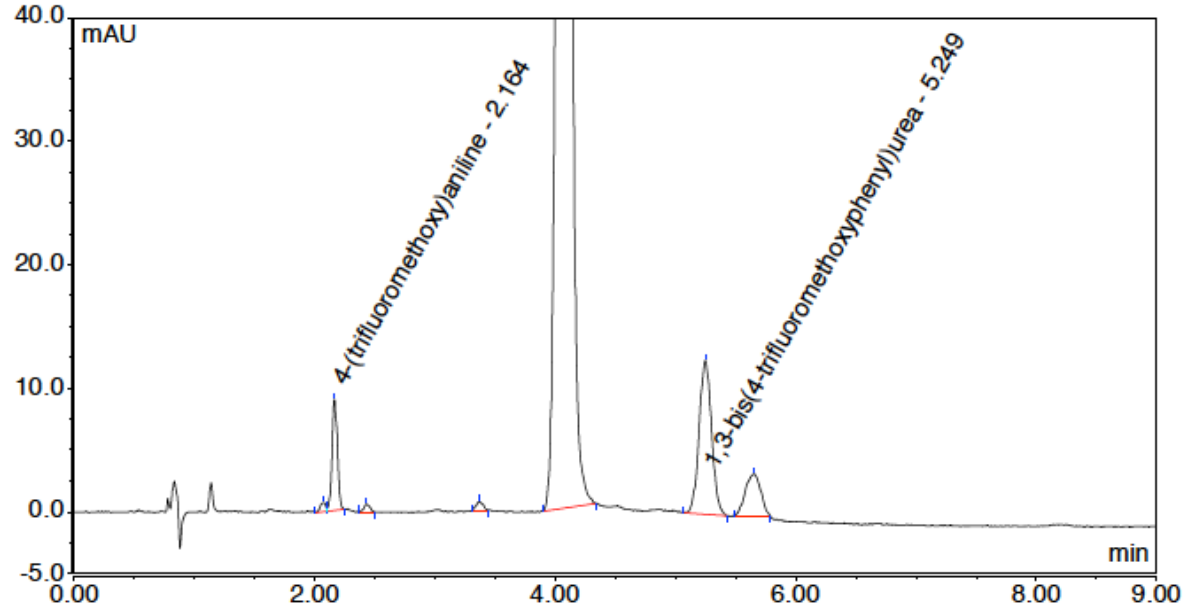


Fig. 4 Chromatogram of Triflumuron TC spiked with 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline

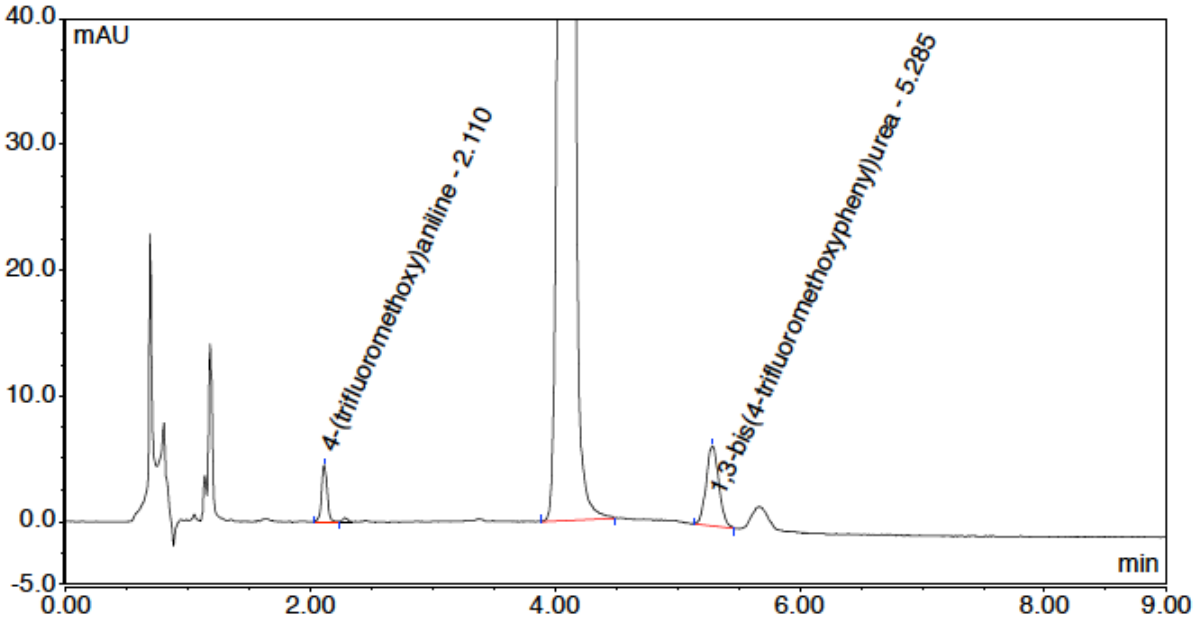


Fig. 5 Chromatogram of Triflumuron SC spiked with 1,3-bis(4-trifluoromethoxyphenyl)urea and 4-(trifluoromethoxy)aniline