CIPAC

COLLABORATIVE INTERNATIONAL PESTICIDES ANALYTICAL COUNCIL LIMITED

Commission Internationale des Méthodes d'Analyse des Pesticides (CIMAP)

CIPAC Free relevant impurities methods:

Methods for relevant impurities becoming more and more important in the quality control of TK/TC and FAO-specifications. In order to meet an urgent need for methods to characterize TK/TC in a.i. and formulations, CIPAC provides selected methods as a download. By downloading these methods, you accept the following conditions of use.

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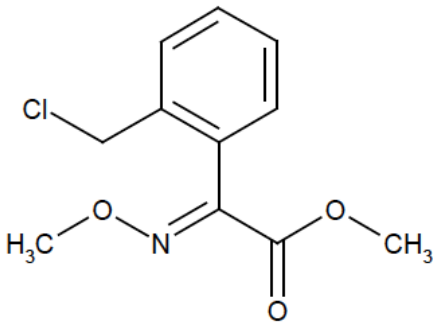
TRIFLOXYSTROBIN
617

See CIPAC O, *p* 161

relevant impurity CGA 344605



ISO common name:	CGA 344605
Chemical name:	methyl (E)-(2-chloromethylphenyl)-methoxy-imino-acetate
CAS No.	189813-45-4
Empirical formula:	$C_{11}H_{12}ClNO_3$
Molecular mass:	241.68 g/mol

TRIFLOXYSTROBIN
617

See CIPAC O, *p* 161.

TRIFLOXYSTROBIN TECHNICAL
*617/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC-UV. Use the HPLC method described below. The relative retention time of CGA 344605 in the sample solution should not deviate by more than 5% from that of the calibration solution. The UV-Spectrum obtained from the sample and reference item should not differ significantly.

5 CGA 344605

OUTLINE OF METHOD

The content of CGA 344605 (% w/w) is determined by reversed phase high performance liquid chromatography using UV detection and external standard calibration.

REAGENTS

CGA 344605: Reference standard of known content

Acetonitrile: HPLC grade or higher

Purified water: HPLC grade or higher

Phosphoric acid: 85%

Eluent A: purified water + 0.1% (v/v) phosphoric acid

Eluent B: acetonitrile+ 0.1% (v/v) phosphoric acid

* CIPAC method 2021. Based on a method supplied by Bayer CropScience, Germany.

APPARATUS

High performance liquid chromatograph, equipped with an injection system capable of injecting 5 µl and an UV/VIS or DAD detector.

Chromatographic column, stainless steel, 100 x 4.6 (i.d.) mm, packed with Ascentis Express C18; 2.7 µm or equivalent with the same selectivity.

Data system

Ultrasonic bath

Centrifuge

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Temperature 35 °C

Injection volume 5 µl

Mobile phase and flow rate

Time [min]	purified water + 0.1% (*v/v*) phosphoric acid	acetonitrile+ 0.1% (*v/v*) phosphoric acid	Flow rate [ml/min]
0.0	54	46	1.8
6.0	40	60	1.8
6.2	5	95	1.8
8.0	5	95	1.8
8.2	54	46	1.8
13.0	54	46	1.8

Retention time: approximately 1.7 minutes

Measurement wavelength 210 nm

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

(c) Preparation of calibration solutions. To prepare a stock solution, weigh (to the nearest 0.01 mg) 20 – 30 mg of the reference item CGA 344605 (*s* in mg) into a volumetric flask (50.0 ml) and add acetonitrile (approx. 40 ml).

Place the flask in an ultrasonic bath for about 10 minutes to dissolve the reference item. Then, fill up the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill the flask to the calibration mark with acetonitrile and mix thoroughly.

Prepare the calibration solutions either as described above or by successive dilution of the respective stock or calibration solutions with acetonitrile.
Prepare at least two independent stock solutions for preparation of the calibration solutions.

A single-point calibration can be used, if the CGA 344605 response in the sample is similar to (~within 50%) calibration solution C_1 and calibration check solution C_2.

For samples with varying or unknown CGA 344605 content, a multi-point calibration curve should be used, covering a suitable concentration range of the maximum expected CGA 344605 content in the sample solution, e.g. 10, 50, 100 and 120% of the max. expected CGA 344605 content (calibration solutions C_1, C_2, C_3,…, C_n).

(d) Preparation of sample. Weigh (at least to the nearest 0.1 mg) an amount (*w* in mg) of homogeneous sample containing approx. 50 mg of the active substance trifloxystrobin into a volumetric flask (50 ml) and add acetonitrile (approx. 40 ml). Place the flask in an ultrasonic bath for about 10 minutes to dissolve the sample. Then fill the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill the flask to the calibration mark with acetonitrile and mix thoroughly. If cloudy, the solution should be centrifuged or filtered prior to analysis (sample solutions S_1 and S_2)

(e) Determination. *Single-point calibration:* Inject a blank solution (e.g. sample solvent) and the calibration solution five times before bracketing a series of sample solutions by injections of a calibration check as follows:

- blank B,
- calibration solution C_1 x 5
- calibration check C_2
- sample solution S_1,
- sample solution S_2,
- calibration check C_1 or C_2
- ... (B, C_1-C_1, C_2, S_1, S_2, C_2, …)

Multi-point calibration curve: Inject a blank solution (e.g. sample solvent), the calibration solutions and each sample solution (single injections or in duplicate) and bracket a series of sample solutions by injecting a calibration check as follows:

- blank B,
- calibration solutions C_1 – C_n
- calibration check C_x
- sample solution S_1,
- sample solution S_2,
- calibration check C_x
- ... (B, C_1 – C_n, C_x, S_1, S_2, C_x, …)

Determine the peak area of CGA 344605.

(f) Calculation. For each sample solution, calculate the content of CGA 344605.

Single-point calibration curve: Calculate the response factors of the calibration solutions. Average the response factors of the calibration solutions. These must agree within ± 2% of the average, otherwise repeat the determination.

Calculate the content of the sample solutions

$$f_i = \frac{Hs \times 100}{s \times P}$$

$$\text{CGA 344605} = \frac{Hw \times 100}{w \times f}\,[\%\ (\text{w/w})]$$

where:

f_i	=	single response factor
H_s	=	area of CGA 34405 in the calibration solution
s	=	mass of reference item CGA 344605 in the calibration solution (mg)
P	=	Purity of CGA 344605 reference item [% (w/w)]
CGA 344605	=	concentration of CGA 344605 in the sample, e.g. [% (w/w)]
H_w	=	area of CGA 344605 in the sample solution
w	=	mass of sample taken (mg)
f	=	average response factor

Multi-point calibration curve: Calculate the calibration function by plotting the resulting peak area of the analyte versus the nominal concentration of the analyte in calibration solution. A suitable calibration function should be applied to the data. A linear regression (1st order) function is preferred, however, if necessary, quadratic regression functions (2nd order) can also be used.

Calculate the analyte content in the sample solution using the calibration function and the determined peak area of the analyte in sample solution.

Calculate the analyte content in the sample, expressed in weight percent [% (*w/w*)], considering the total sample weight:

$$Hs = m \times x + b$$

$$CGA\ 344605\ [\text{mg/l}] = \frac{(Hw - b)}{m}$$

$$CGA\ 344605\ [\%\ (\text{w/w})] = \frac{CGA\ 344605\ [mg/l]}{c_w\ [mg/l]} \times 100\%\ (\text{w/w})$$

where:

H_s	=	area of CGA 34405 in the calibration solution
m	=	slope of calibration function
x	=	Cs = concentration of CGA 344605 in calibration solution, e.
b	=	intercept of calibration function
CGA 344605	=	concentration of CGA 344605 in the sample solution, e.g. [mg/l]
H_w	=	area of CGA 344605 in sample solution
CGA 344605	=	concentration of CGA 344605 in the sample, e.g. [% (w/w)]
c_w	=	concentration of sample in sample solution e.g. [mg/l]

TRIFLOXYSTROBIN EMULSIFIABLE CONCENTRATE

***617**/EC/M/-

1 Sampling. As for trifloxystrobin technical concentrate **617**/TC/M/1

2 Identity tests. As for trifloxystrobin technical concentrate **617**/TC/M/2

5 CGA 344605. As for trifloxystrobin technical concentrate **617**/TC/M/5

* CIPAC method 2021. Based on a method supplied by Bayer CropScience, Germany.

TRIFLOXYSTROBIN WATER DISPERSIBLE GRANULES

*617/WG/M/-

1 Sampling. As for trifloxystrobin technical concentrate **617**/TC/M/1

2 Identity tests. As for trifloxystrobin technical concentrate **617**/TC/M/2

5 CGA 344605. As for trifloxystrobin technical concentrate **617**/TC/M/5, except:

(c) Preparation of calibration solutions. To prepare a stock solution, weigh (to the nearest 0.01 mg) 20 – 30 mg of the reference item CGA 344605 (*s* in mg) into a volumetric flask (50.0 ml) and add water (10 ml) and acetonitrile (approx. 30 ml). Place the flask in an ultrasonic bath for about 10 minutes to dissolve the reference item. Then, fill the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill to the calibration mark with acetonitrile and mix thoroughly.

Prepare the calibration solutions either as described above or by successive dilution of the respective stock or calibration solutions with 80% acetonitrile and 20% water (v/v).

Prepare at least two independent stock solutions for preparation of the calibration solutions.

A single-point calibration can be used, if the CGA 344605 response in the sample is similar to (~within 50%) calibration solution C_1 and calibration check solution C_2.

For samples with varying or unknown CGA 344605 content a multi-point calibration curve should be used, covering a suitable concentration range of the maximum expected CGA 344605 content in the sample solution, e.g. 10, 50, 100 and 120% of the max. expected CGA 344605 content (calibration solutions C_1, C_2, C_3,…, C_n).

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(d) Preparation of sample. Weigh (at least to the nearest 0.1 mg) an amount (*w* in mg) of homogeneous sample containing approx. 50 mg of the active substance trifloxystrobin into a volumetric flask (50.0 ml) and add water to suspend the sample (10 ml) and acetonitrile (approx. 30 ml). Place the flask in an ultrasonic bath for about 10 minutes to dissolve the sample. Then, fill the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill the flask to the calibration mark with acetonitrile and mix thoroughly. If cloudy, the solution should be centrifuged or filtered prior to analysis (sample solutions S_1 and S_2)

TRIFLOXYSTROBIN SUSPENSION CONCENTRATE

***617**/SC/M/-

1 Sampling. As for trifloxystrobin technical concentrate **617**/TC/M/1

2 Identity tests. As for trifloxystrobin technical concentrate **617**/TC/M/2

5 CGA 344605. As for trifloxystrobin technical concentrate **617**/TC/M/5, except:

(c) Preparation of calibration solutions. To prepare a stock solution, weigh (to the nearest 0.01 mg) 20 – 30 mg of the reference item CGA 344605 (*s* in mg) into a volumetric flask (50.0 ml) and add water (10 ml) and acetonitrile (approx. 30 ml). Place the flask in an ultrasonic bath for about 10 minutes to dissolve the reference item. Following, fill the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill the flask to the calibration mark with acetonitrile and mix thoroughly.

Prepare the calibration solutions either as described above or by successive dilution of the respective stock or calibration solutions with 80% acetonitrile and 20% water (v/v).

Prepare at least two independent stock solutions for preparation of the calibration solutions.

A single-point calibration can be used, if the CGA 344605 response in the sample is similar to (~within 50%) calibration solution C_1 and calibration check solution C_2.

* CIPAC method 2021. Based on a method supplied by Bayer CropScience, Germany.

For samples with varying or unknown CGA 344605 content a multi-point calibration curve should be used, covering a suitable concentration range of the maximum expected CGA 344605 content in the sample solution, e.g. 10, 50, 100 and 120% of the max. expected CGA 344605 content (calibration solutions C_1, C_2, C_3,…, C_n).

(d) Preparation of sample. Weigh (at least to the nearest 0.1 mg) an amount (*w* in mg) of homogeneous sample containing approx. 50 mg of the active substance trifloxystrobin into a volumetric flask (50 ml) and add water to suspend the sample (10 ml) and acetonitrile (approx. 30 ml). Place the flask in an ultrasonic bath for about 10 minutes to dissolve the sample. Then, fill the flask with acetonitrile to just below the calibration mark. After the temperature is equilibrated, fill the flask to the calibration mark with acetonitrile and mix thoroughly. If cloudy, the solution should be centrifuged or filtered prior to analysis (sample solutions S_1 and S_2)

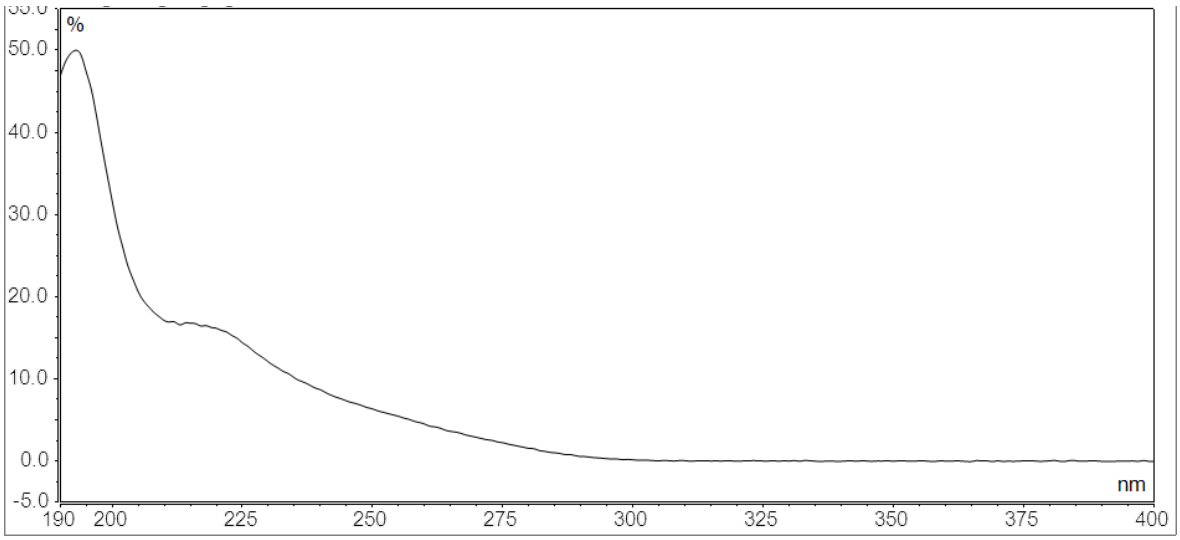

Fig. 1 UV Spectrum of CGA 344605

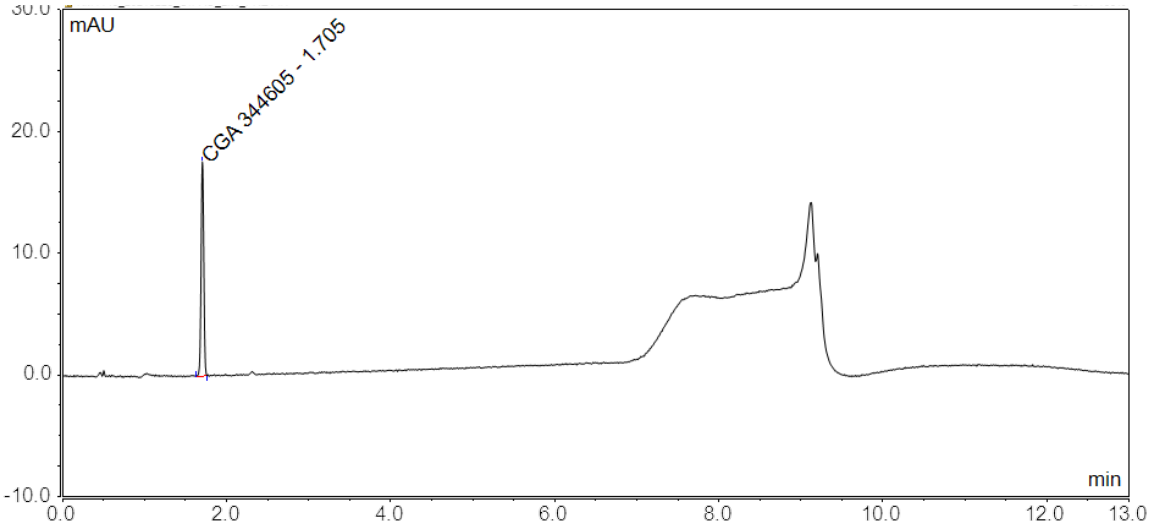

Fig. 2 Chromatogram of analytical standard CGA 344605

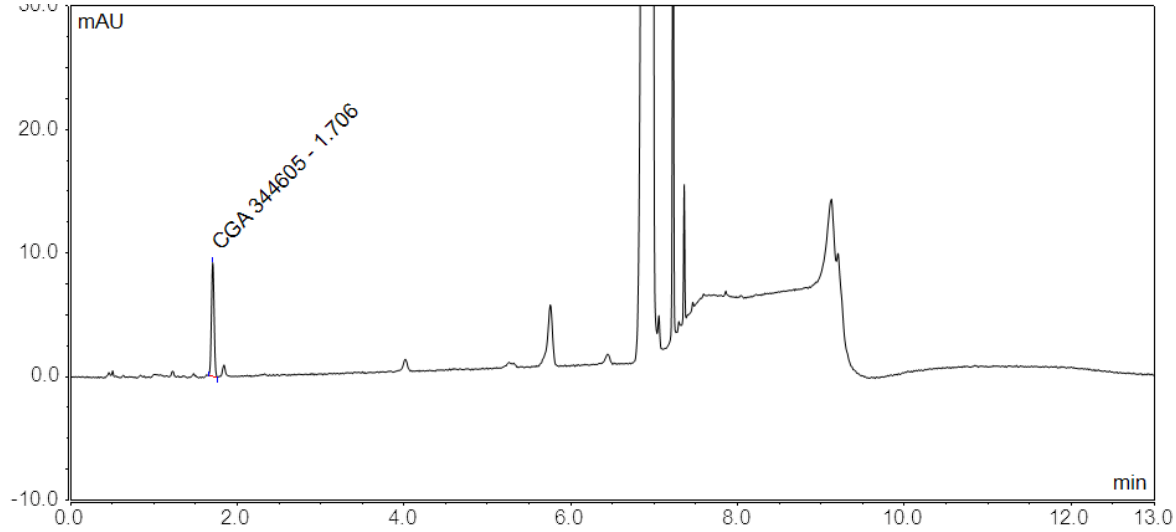

Fig. 3 Chromatogram of trifloxystrobin TC, spiked with CGA 344605

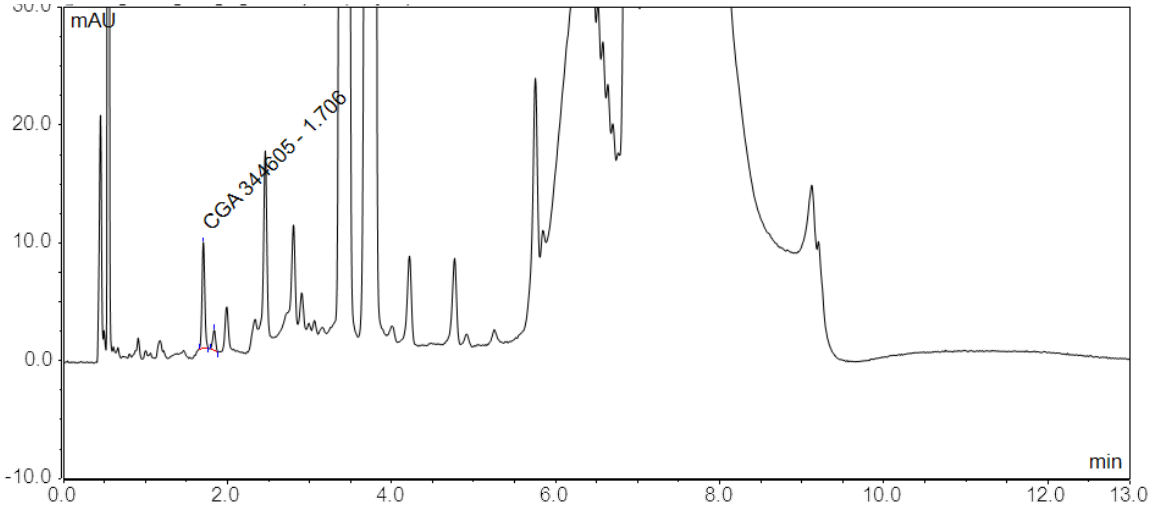

Fig. 4 Chromatogram of trifloxystrobin EC, spiked with CGA 344605

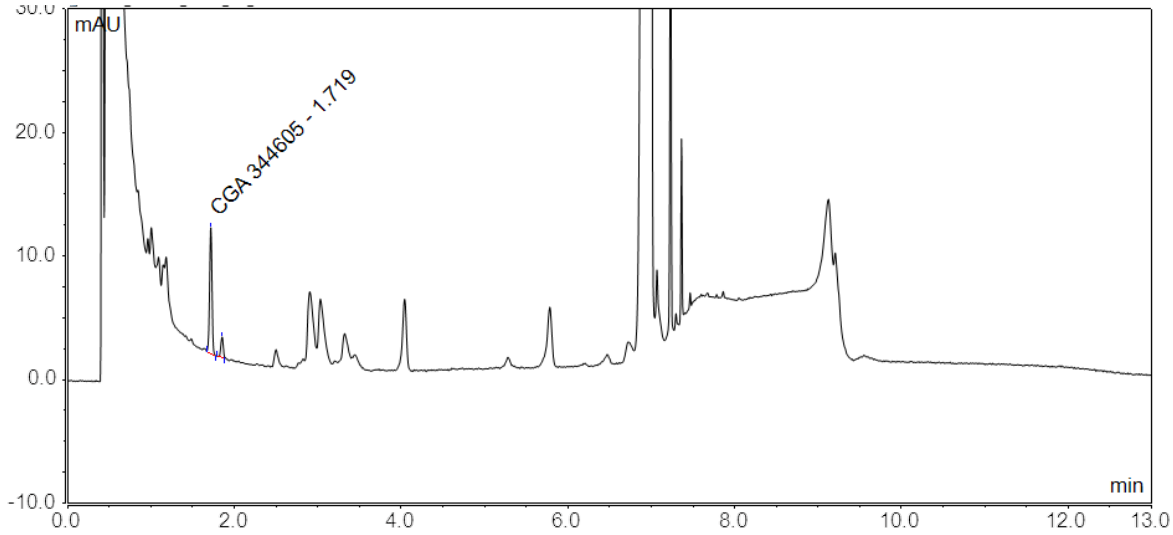

Fig. 5 Chromatogram of trifloxystrobin WG

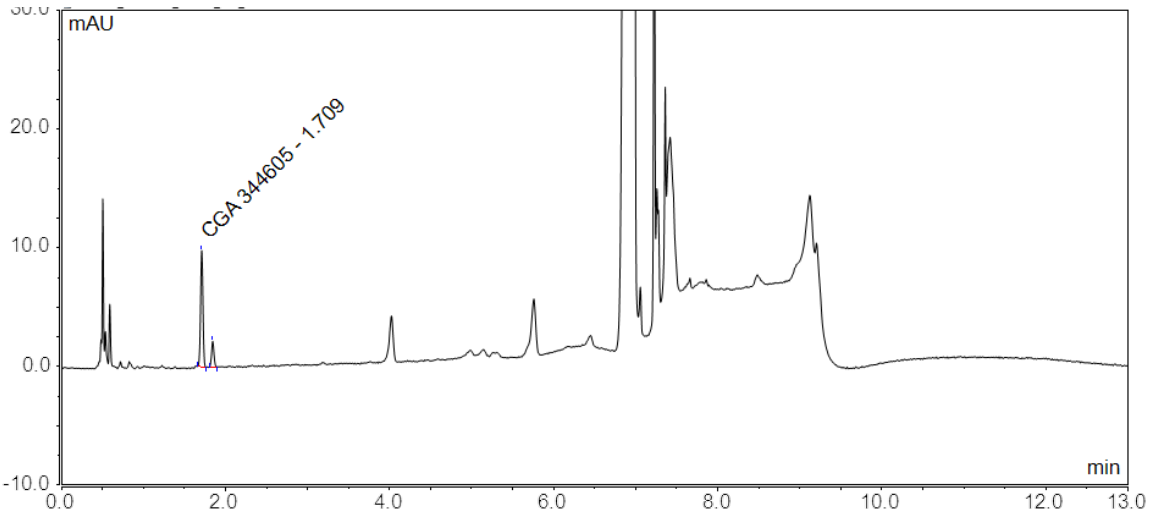

Fig. 6 Chromatogram of trifloxystrobin SC, spiked with CGA 344605