

Tebuconazole (CIPAC 494)

CIPAC Method Extension

CIPAC Method extension
of an analytical method for the determination of tebuconazole
in tebuconazole EC formulations
by Gas Liquid Chromatography

Report to CIPAC
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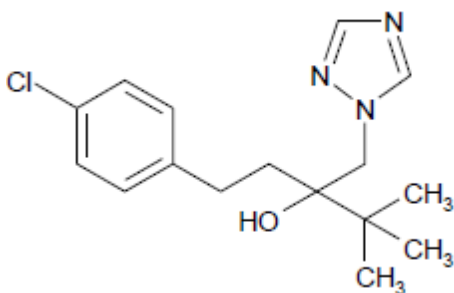
1 Introduction

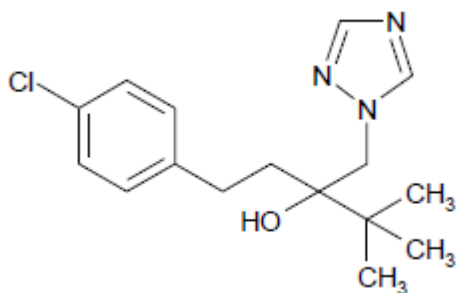
1.1 Scope

The results of the outcome of the method extension to demonstrate that the CIPAC method 494 is suitable for the determination of tebuconazole in tebuconazole emulsifiable concentrate (EC) formulations are reported.

The method extension was conducted by two independent laboratories. Five batches of a EC formulation were selected to be used for this method extension

1.2 Analyte to be determined

Analyte Name	tebuconazole
Synonyms	HWG 1608, AE F069623 , BCS-AA31476
Three Letter Code	TBZ
Structural Formula	



Empirical Formula	C ₁₆ H ₂₂ Cl N ₃ O
Molecular Weight	307.8 g/mol
CAS No	107534-96-3

1.3 Samples

Tebuconazole, Emulsifiable Concentrate EC 250 g/
Declared content: 250 g/L, 26.0 % (w/w)

Batches:

2020-002262
EDFL052343
EDFL052344
EDFL052752
EDFL053105

Blank formulation:

Declared content: 0 g/L, 0 % (w/w)
Batch 2020-002263

Certified Reference Material:

tebuconazole, reference standard
AZ 23472, purity 99.7% w/w, Expiry Date: 2025-06-18

1.4 Participants

Friedhelm Schulz	Bayer AG, Crop Science Division Alfred-Nobel-Strasse 50 40789, Monheim am Rhein Germany
Dr. Michael Haustein	Currenta GmbH & Co. OHG CUR-ANT-PDA 41538, Dormagen Germany

2 Analytical Method

2.1 Outline of the Method

Gas Chromatography (GC) hyphenated to flame ionization detector was used for quantitation of tebuconazole.

The tebuconazole is separated from formulation components and active substances using a capillary column and isocratic elution. The quantitative evaluation is carried out by comparing the peak areas with those of reference items, using an internal standard method.

2.2 Method Extension

The method extension was conducted in two laboratories accordingly the following procedure.

1) Specificity

Interferences at the retention time of the analyte in the formulation were checked by comparing the chromatograms of the reference item tebuconazole and the test solutions of formulations and blank formulation.

2) Repeatability

Lab 1: Measurement of each individual batch at two different days, double determination and single injection each.

Lab 2: Measurement of each individual batch in one day, four determinations and double injection each. With the exception of 6 determinations for batch EDFL052343

3 Remarks of the participants

Laboratory	Remarks
Lab 1	n.a.
Lab 2	n.a.

4 Results and Discussion

4.1 Method Extension

1) Specificity

The specificity of the analytical method for tebuconazole is assessed sufficient as no significant interfering compounds were detected in the chromatograms at the retention time of the analyte.

2) Repeatability

The repeatability of the method was satisfactory with RSD_r values of 0.15 – 0.63 % as shown in Table 1 to Table 5

5 Conclusion

Based on the relative standard deviation results $RSD (r)$ obtained for the five individual EC batches, the CIPAC method is regarded suitable for the extension to EC formulation types. The repeatability results (ranging from 0.15 – 0.63 % relative) are far below the modified Horwitz criterion.

In conclusion, the proposed method was considered appropriate for the determination of tebuconazole in EC formulations.

6 Determination of tebuconazole in emulsifiable concentrate (EC) formulations

6.1 Tables of results

Table 1: Results Batch 2020-002262

	Laboratory 1		Laboratory 2	
	tebuconazole		tebuconazole	
	[% w/w]		[% w/w]	
	Day 1, Weighing no. 1	25.90	Weighing no. 1	25.5725 ¹
	Day 1, Weighing no. 2	25.80	Weighing no. 2	25.6115 ¹
	Day 2, Weighing no. 1	25.61	Weighing no. 3	25.559 ¹
	Day 2, Weighing no. 2	25.39	Weighing no. 4	25.5145 ¹
Mean value	25.68		25.56	
SD	0.2249		0.0400	
RSD [%]	0.88		0.016	
Horwitz-Value RSD (r) _{max}	1.64		1.65	
Horrat value H _r	0.54		0.10	
Outliers	no		no	
Mean Value	25.62			
s _r	0.1615			
RSD (r)	0.63			

¹ Mean value of 2 injections

Table 2: Results Batch EDFL052343

	Laboratory 1		Laboratory 2	
	tebuconazole		tebuconazole	
	[% w/w]		[% w/w]	
	Day 1, Weighing no. 1	26.01	Weighing no. 1	25.624 ¹
	Day 1, Weighing no. 2	25.95	Weighing no. 2	25.605 ¹
	Day 2, Weighing no. 1	25.55	Weighing no. 3	25.7155 ¹
	Day 2, Weighing no. 2	25.60	Weighing no. 4	25.741 ¹
			Weighing no. 5	25.7495 ¹
			Weighing no. 6	25.7715 ¹
Mean value		25.78		25.70
SD		0.2360		0.0697
RSD [%]		0.92		0.27
Horwitz-Value RSD (r) _{max}		1.64		1.64
Horrat value H _r		0.56		0.16
Outliers		no		no
Mean Value				25.73
s _r				0.1547
RSD (r)				0.60

¹ Mean value of 2 injections

Table 3: Results Batch EDFL052344

	Laboratory 1		Laboratory 2	
	tebuconazole		tebuconazole	
	[% w/w]		[% w/w]	
	Day 1, Weighing no. 1	25.70	Weighing no. 1	25.482 ¹
	Day 1, Weighing no. 2	25.43	Weighing no. 2	25.391 ¹
	Day 2, Weighing no. 1	25.37	Weighing no. 3	25.453 ¹
	Day 2, Weighing no. 2	25.46	Weighing no. 4	25.423 ¹
Mean value		25.49		25.44
SD		0.1449		0.0391
RSD [%]		0.57		0.15
Horwitz-Value RSD (r) _{max}		1.65		1.65
Horrat value H _r		0.35		0.09
Outliers		no		no
Mean Value			25.46	
s _r			0.1061	
RSD (r)			0.42	

¹ Mean value of 2 injections

Table 4: Results Batch EDFL052752

	Laboratory 1		Laboratory 2	
	tebuconazole		tebuconazole	
	[% w/w]		[% w/w]	
	Day 1, Weighing no. 1	25.85	Weighing no. 1	25.7825 ¹
	Day 1, Weighing no. 2	25.88	Weighing no. 2	25.782 ¹
	Day 2, Weighing no. 1	25.79	Weighing no. 3	25.805 ¹
	Day 2, Weighing no. 2	25.78	Weighing no. 4	25.8315 ¹
Mean value		25.83		25.80
SD		0.0480		0.0234
RSD [%]		0.19		0.09
Horwitz-Value RSD (r) _{max}		1.64		1.64
Horrat value H _r		0.12		0.05
Outliers		no		no
Mean Value			25.81	
s _r			0.0377	
RSD (r)			0.15	

¹ Mean value of 2 injections

Table 5: Results Batch EDFL053105

	Laboratory 1		Laboratory 2	
	tebuconazole		tebuconazole	
	[% w/w]		[% w/w]	
	Day 1, Weighing no. 1	25.99	Weighing no. 1	25.9305 ¹
	Day 1, Weighing no. 2	26.23	Weighing no. 2	25.8865 ¹
	Day 2, Weighing no. 1	25.82	Weighing no. 3	25.9415 ¹
	Day 2, Weighing no. 2	25.88	Weighing no. 4	25.868 ¹
Mean value	25.98		25.91	
SD	0.1809		0.0350	
RSD [%]	0.70		0.14	
Horwitz-Value RSD (r) _{max}	1.64		1.64	
Horrat value H _r	0.43		0.09	
Outliers	no		no	
Mean Value			25.94	
s _r			0.1303	
RSD (r)			0.50	

¹ Mean value of 2 injections

Table 6: Summary of Results

	Batch 2020-002262	Batch EDFL052343	Batch EDFL052344	Batch EDFL052752	Batch EDFL053105
x [% w/w]	25.62	25.73	25.46	25.81	25.94
L	2	2	2	2	2
s _r [% w/w]	0.1615	0.1547	0.1061	0.0377	0.1303
RSD _r [%]	0.63	0.60	0.42	0.15	0.50
r [% w/w]	0.4522	0.4330	0.2972	0.1057	0.3649
Horwitz-value RSD (r) _{max}	1.64	1.64	1.65	1.64	1.64
Horrat value H _r	0.38	0.37	0.25	0.09	0.30

Where

x = average

L = number of laboratories

s_r = repeatability standard deviation

RSD_r = repeatability relative standard deviation

r = repeatability (s_r * 2.8)

6.2 Chromatograms

Figure 1: Chromatograms of analytical standard tebuconazole and internal standard DHCP - Lab 1

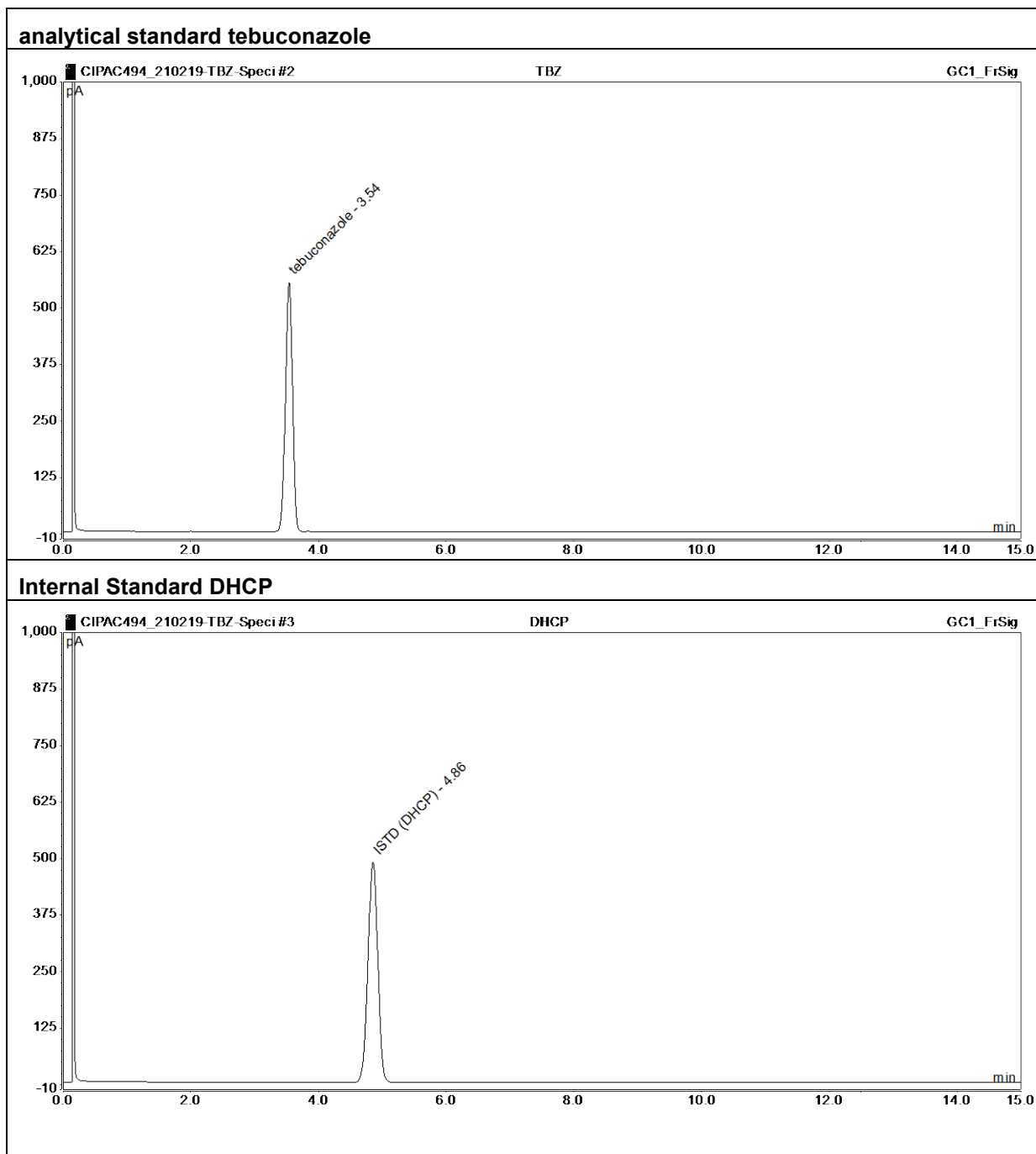


Figure 1. continuation

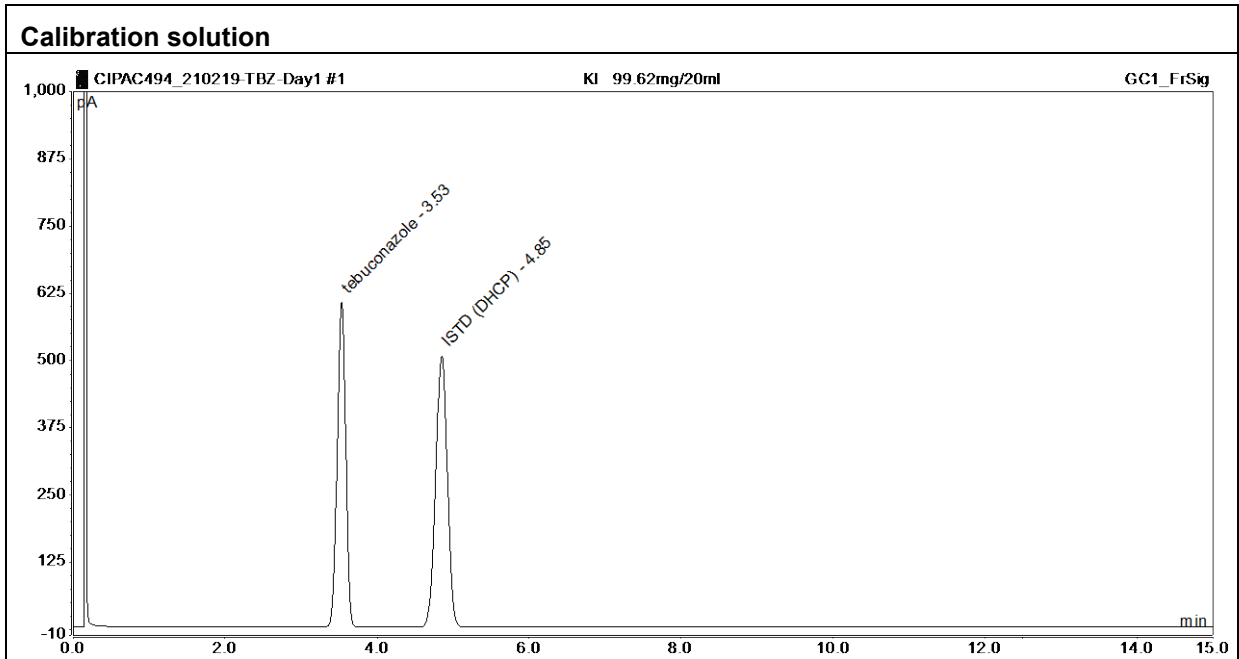


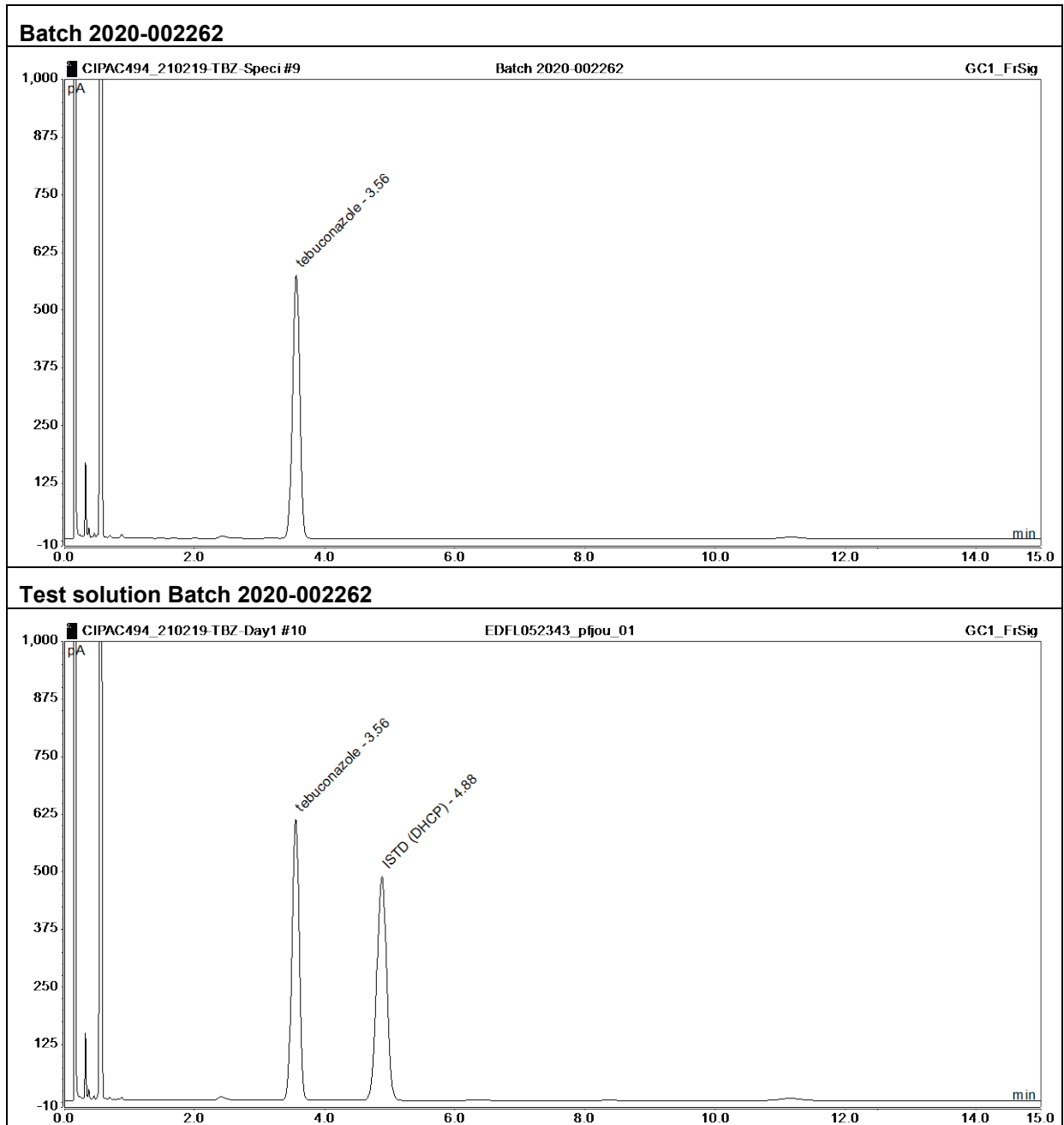
Figure 2: Chromatogram of tebuconazole EC - Lab 1

Figure 2 continuation

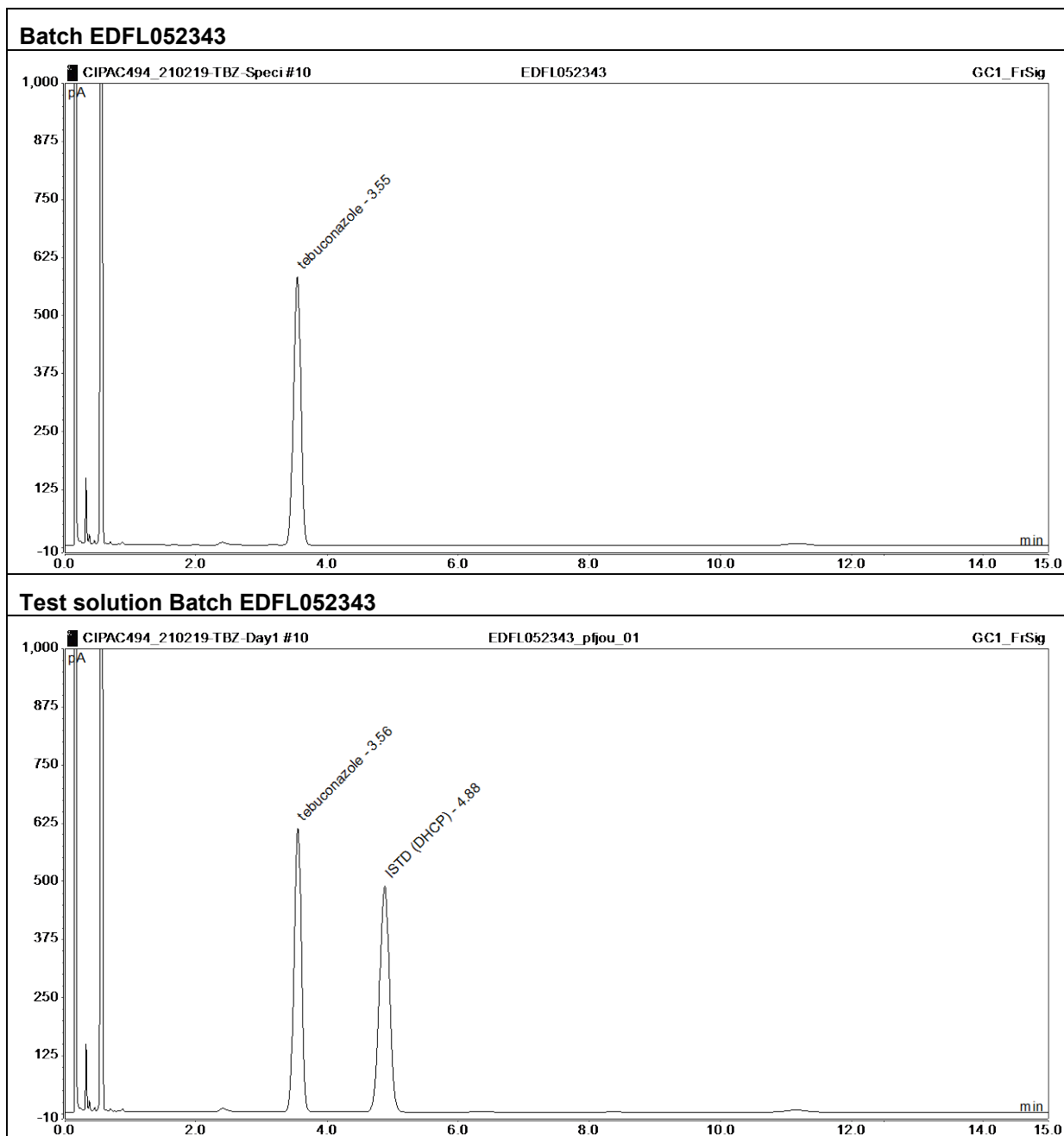


Figure 2 continuation

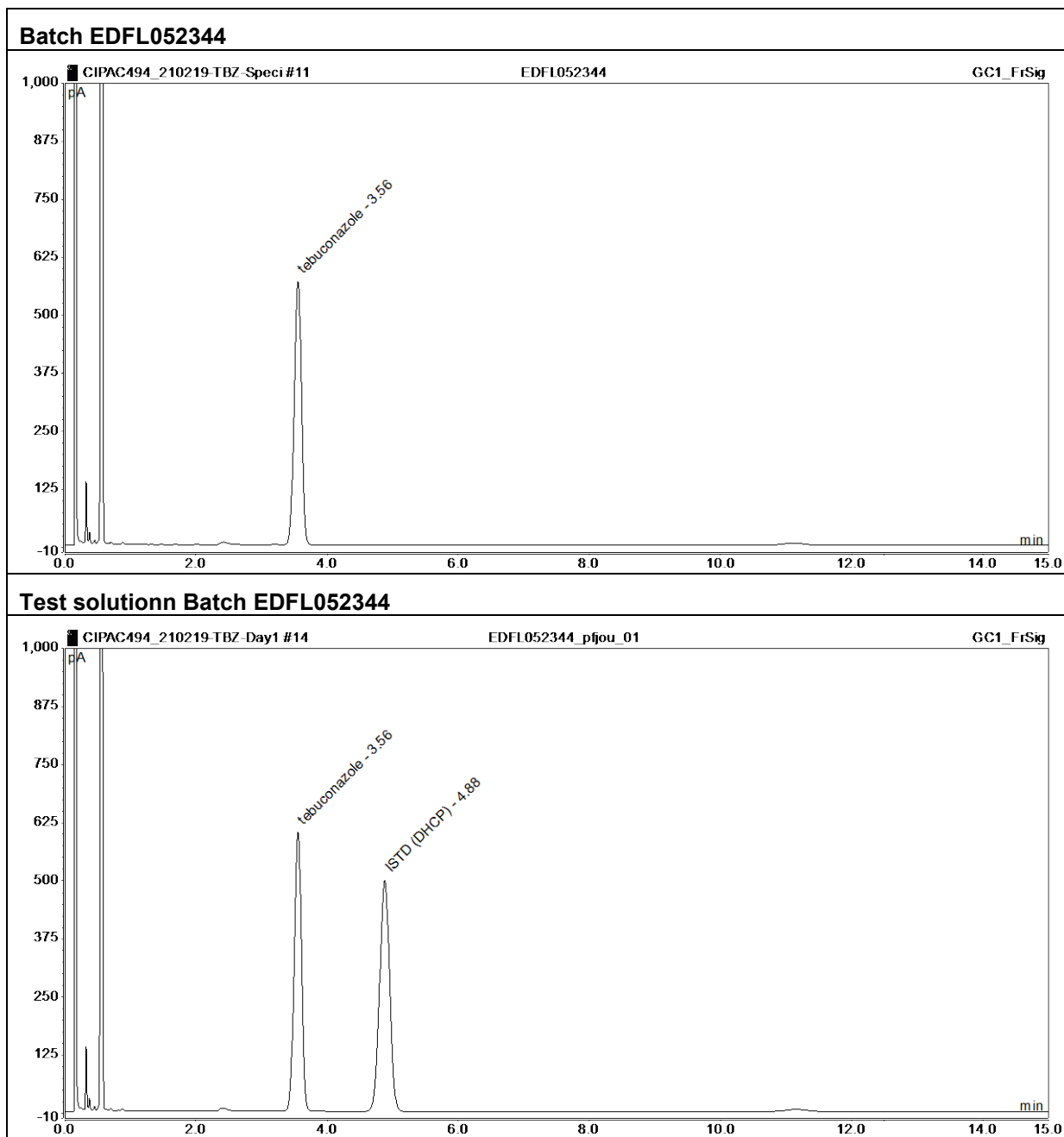


Figure 2 continuation

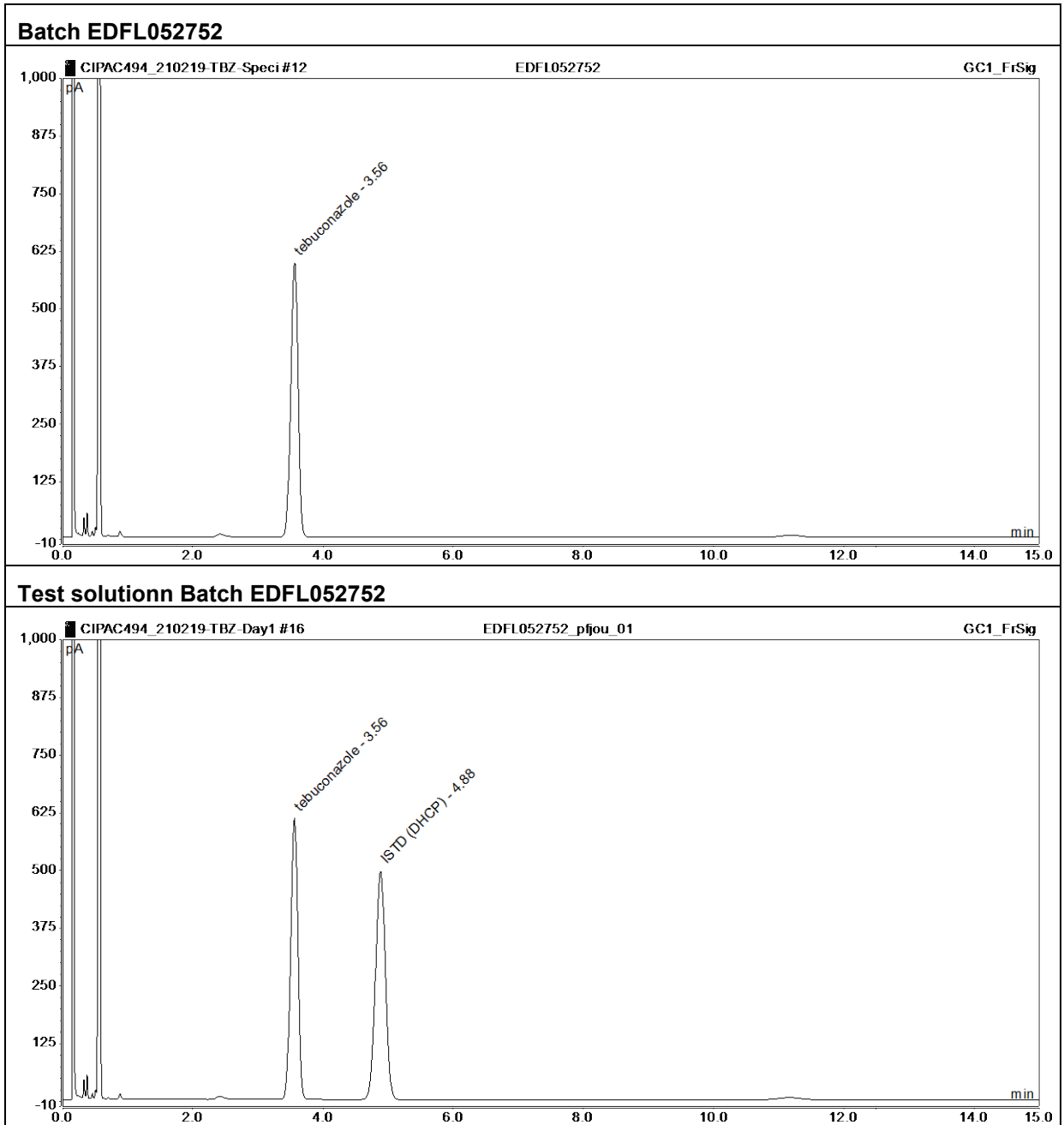


Figure 2 continuation

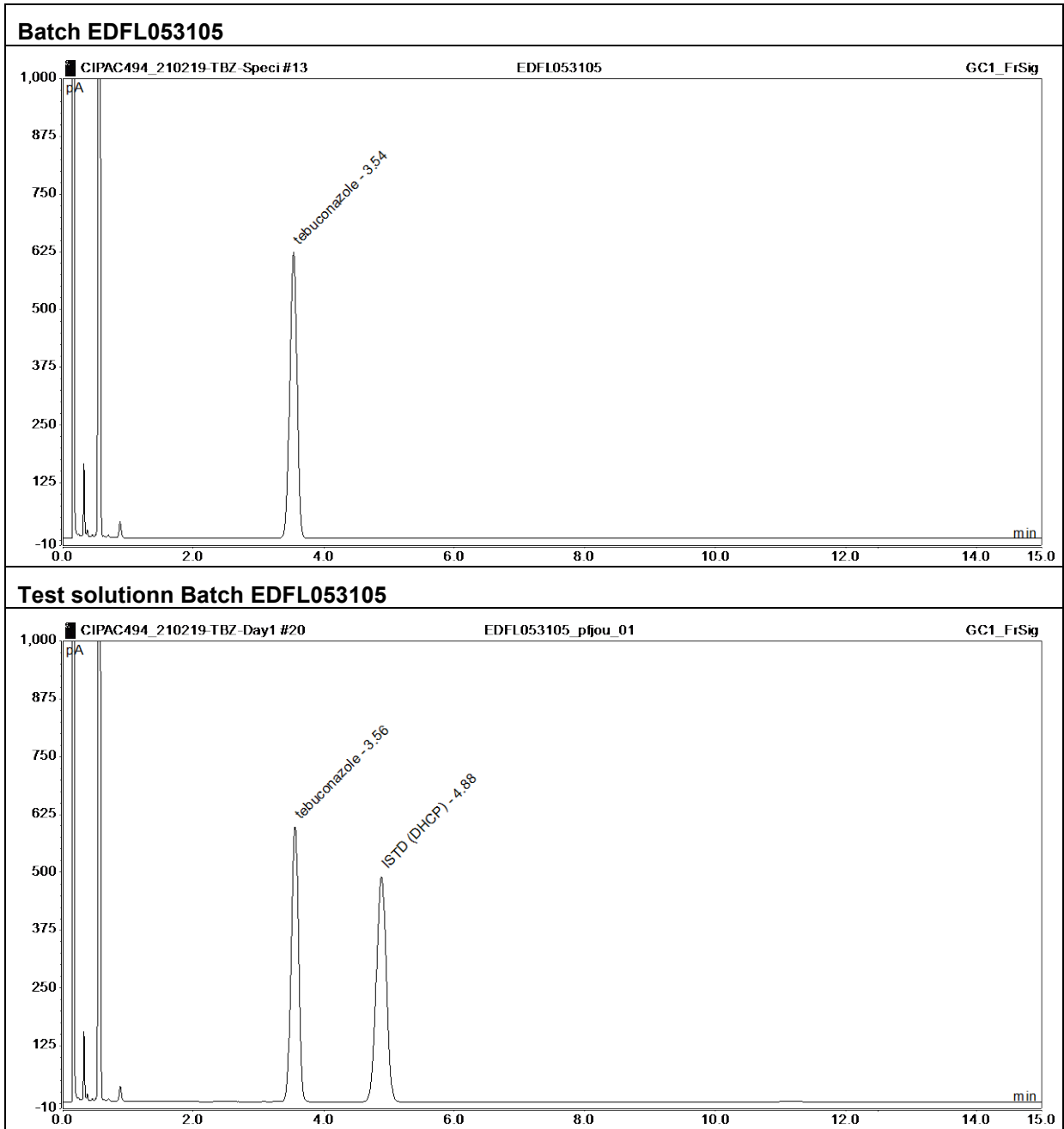


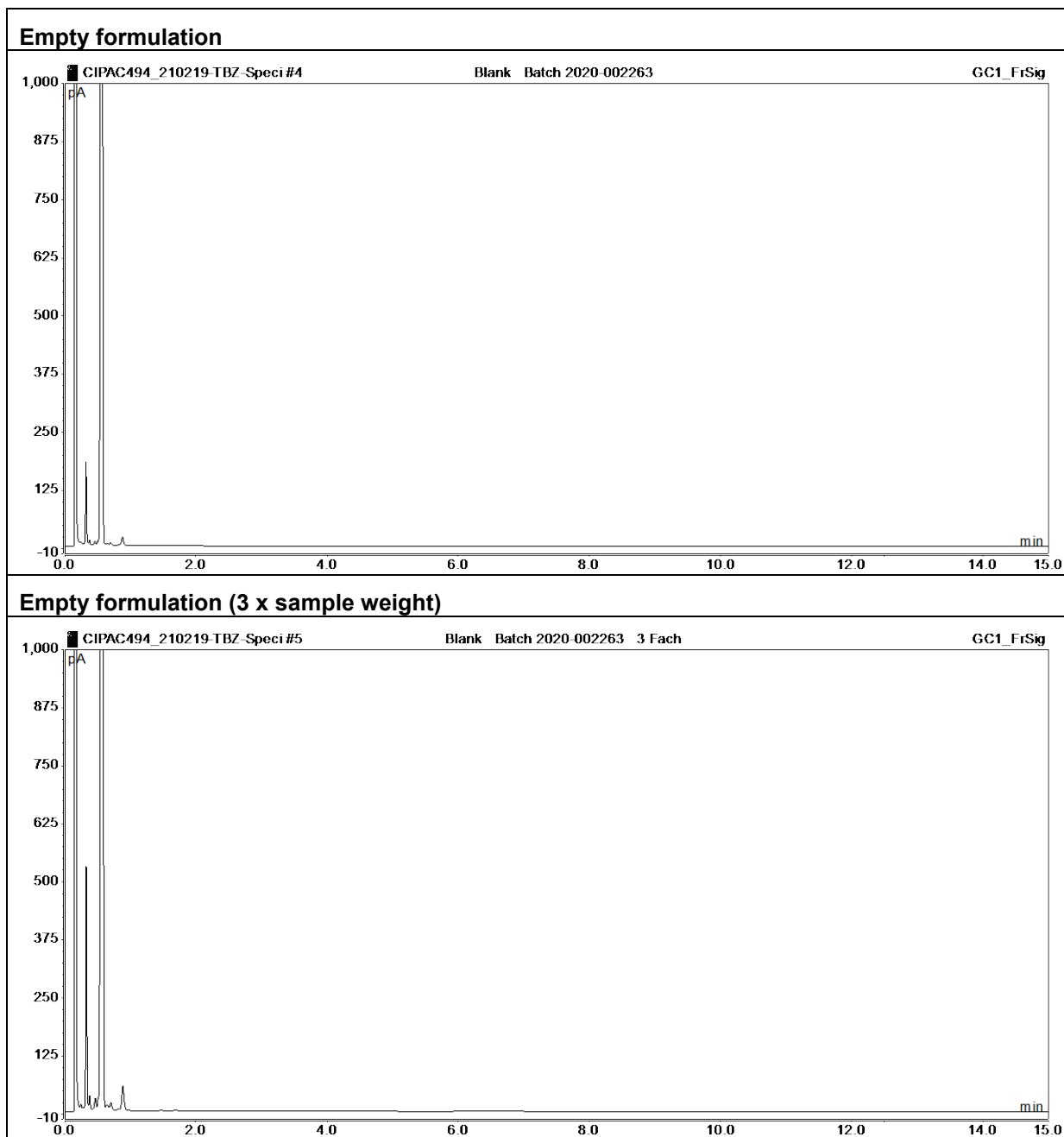
Figure 3: Chromatogram of empty formulation - Lab 1

Figure 3 continuation

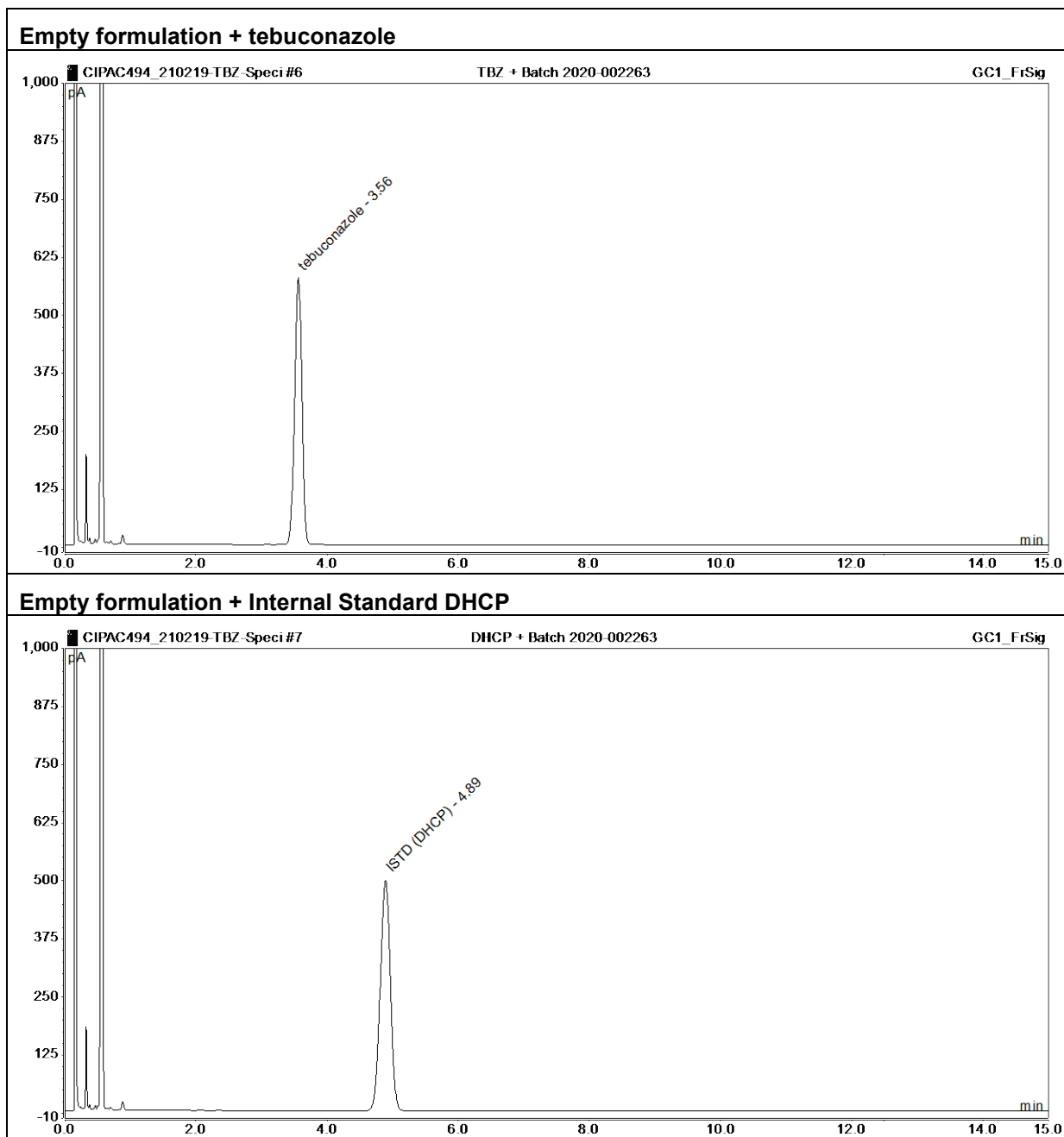


Figure 3 continuation

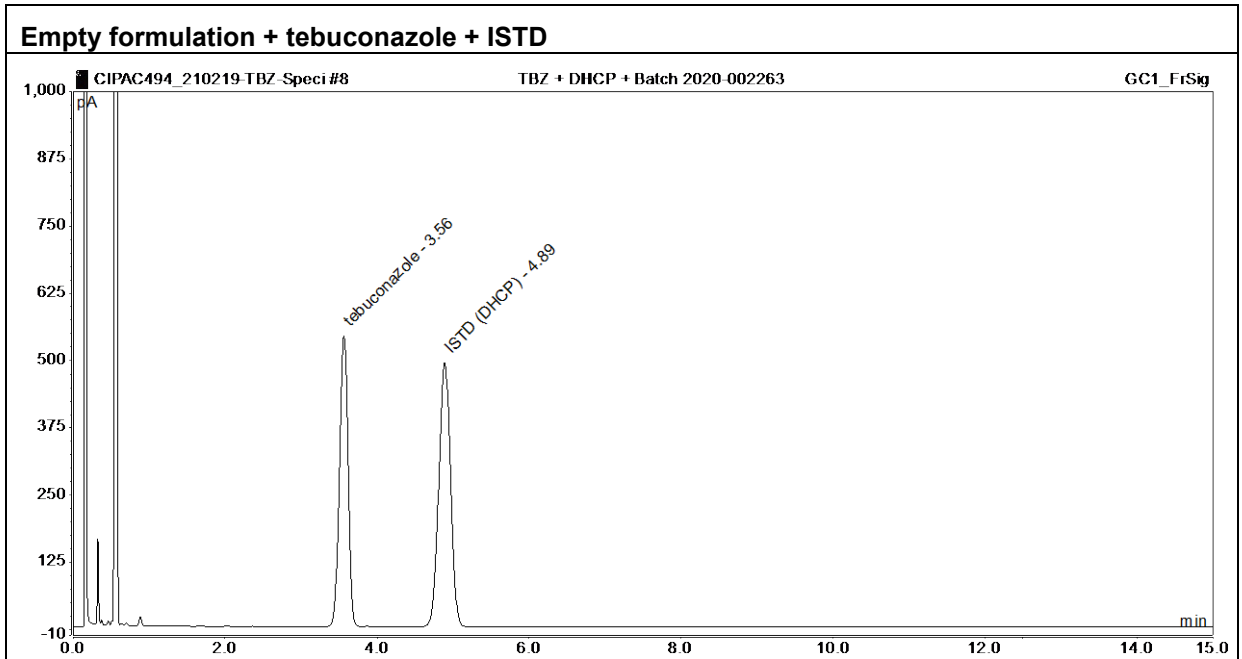


Figure 4: Chromatogram of solvent (acetone) - Lab 1

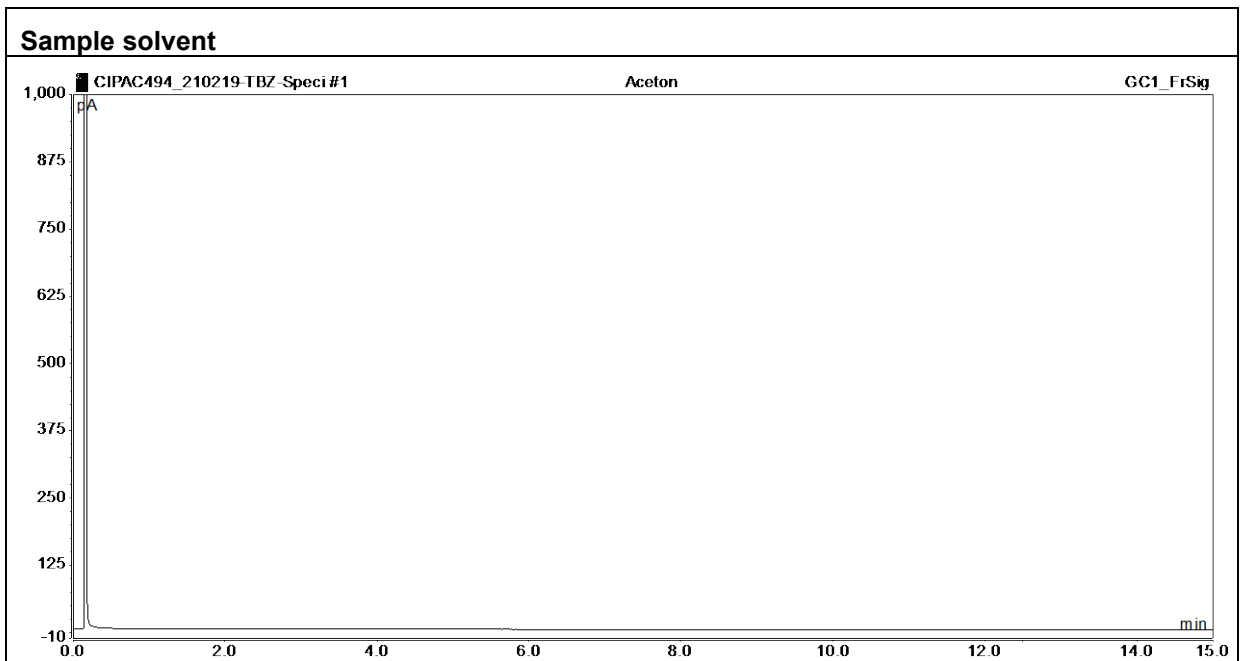


Figure 5: Chromatograms of analytical standard tebuconazole and internal standard DHCP - Lab 2

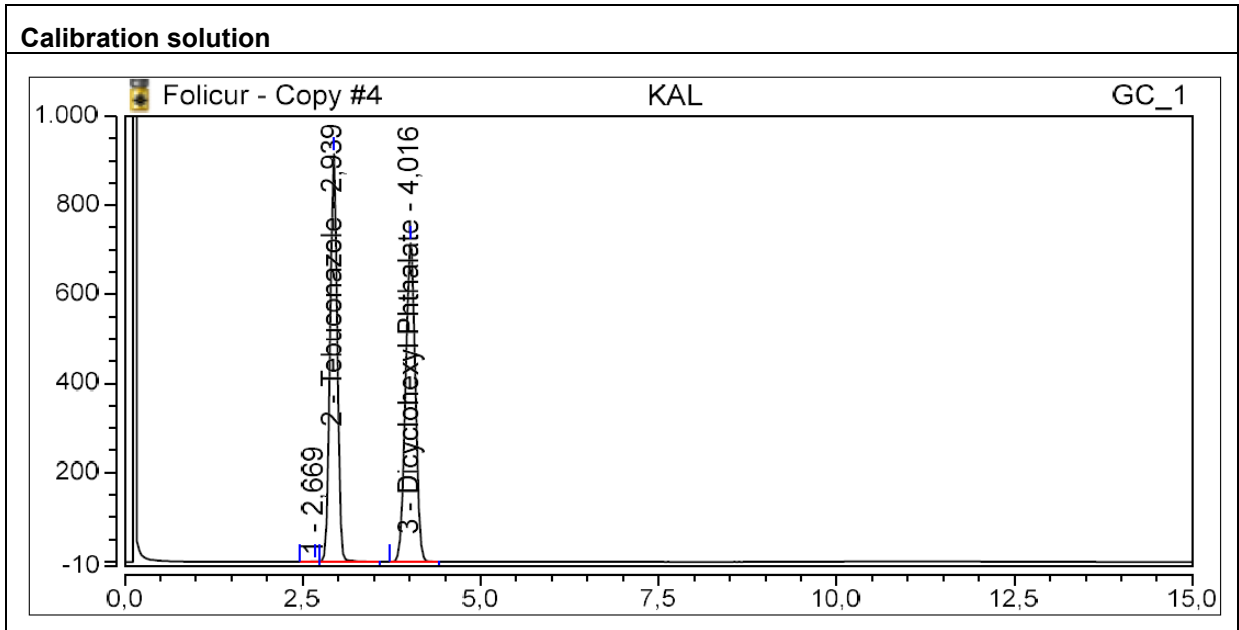


Figure 6: Chromatogram of tebuconazole EC - Lab 2

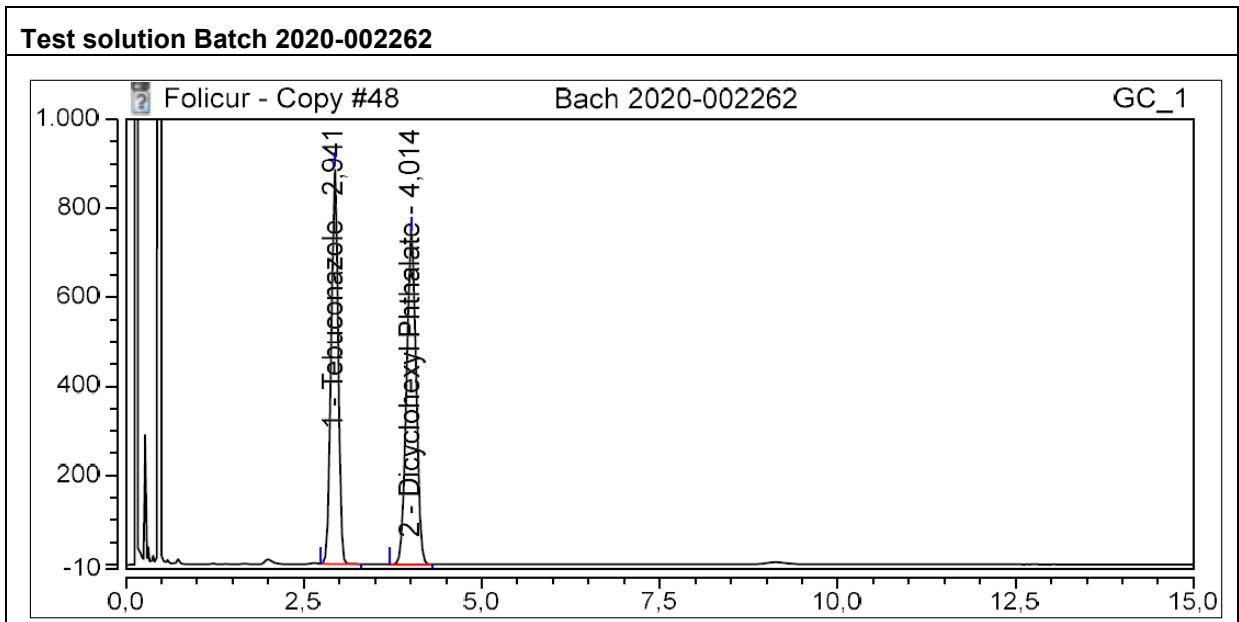


Figure 6 continuation

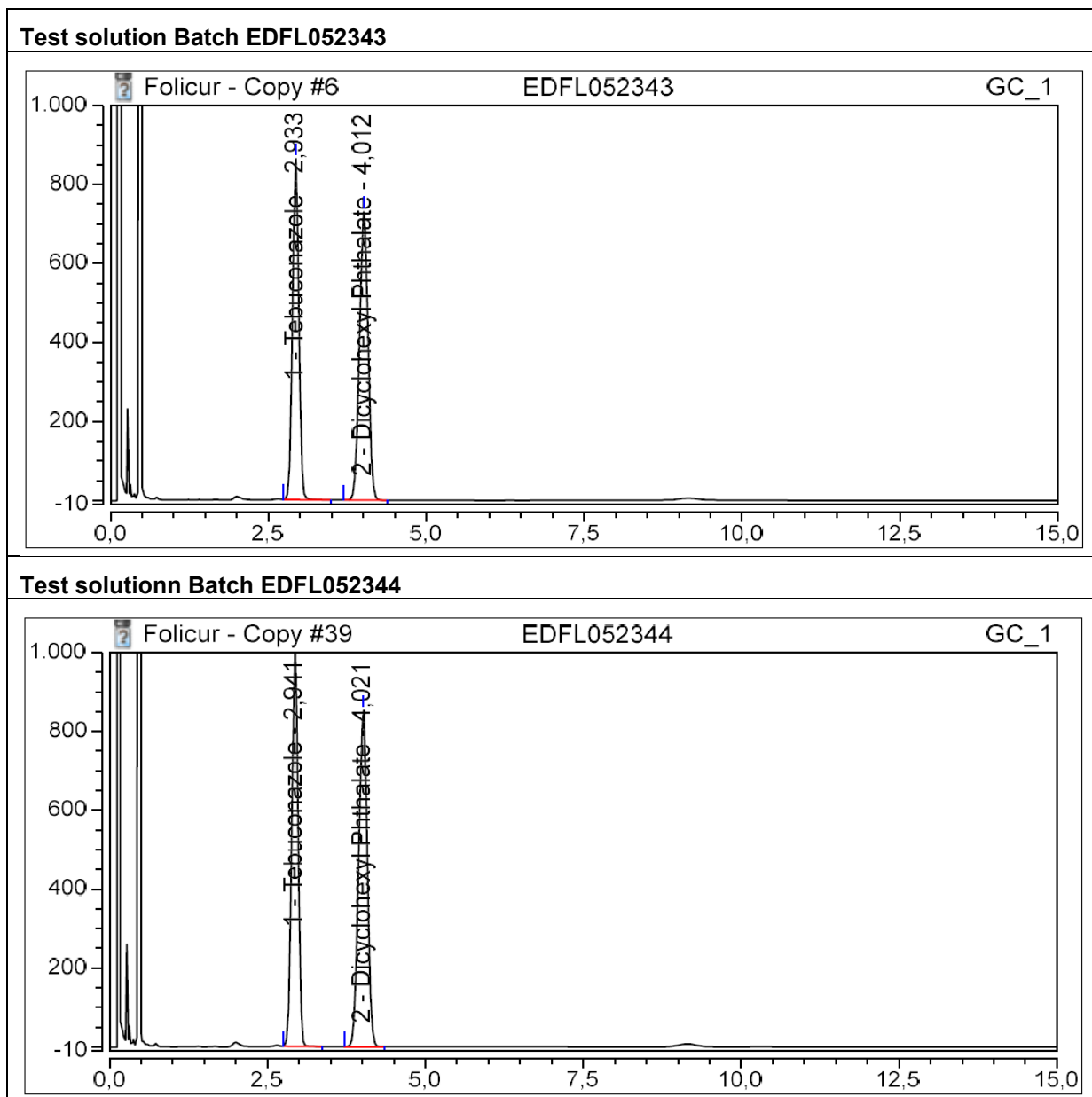


Figure 6 continuation

