

# State of the art of the detection of glyceroltriheptanoate (GTH) in fats by GC-MS in the European Union

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## Introduction

In the framework of the global feed ban against the spreading of TSEs, end point marking of derived animal by-products (ABPs) from category 1 and 2 with glyceroltriheptanoate (GTH) at a minimum mass fraction of 250 mg kg<sup>-1</sup> related to the fat content is compulsory in the European Union. The objective of this marking is to ensure traceability and to prevent re-entering of such products into the feed and food chain. Controls for detection of GTH are based on the use of gas chromatography coupled to mass spectrometry (GC-MS) since 2010<sup>a,b</sup>. In 2019, the European Commission mandated the EURL-AP to conduct an inter laboratory study to 1) assess the implementation of the method in laboratories designated by the competent authorities of the Member States and 2) evaluate whether a revision of the protocol would be required and if the method as such is suitable for GTH detection in feed matrices.

## Study organization

- 19 participating laboratories with expertise in GTH detection
- 6 homogeneous and stable samples from category 3 poultry fat (*blank + 4 levels of adulteration with industrial GTH with purity of 91%*) and category 2 fat marked with unknown GTH mass fraction.

	Industrial GTH content [mg kg <sup>-1</sup> ]	Target assigned value x <sub>pt</sub> [mg kg <sup>-1</sup> ]	u(x <sub>pt</sub> ) (k=1) [mg kg <sup>-1</sup> ]	σ <sub>pt</sub>	u(x <sub>pt</sub> )/σ <sub>pt</sub> (>0.3?)	score to use
Blank Cat 3	0	/	/	/	/	/
GTH25	25	22.77	1.29	4.55	0.28	z
GTH50	50	45.55	2.57	6.83	0.38	z'
GTH100	100	91.10	5.14	13.66	0.38	z'
GTH250	250	227.70	12.85	34.15	0.38	z'
Cat 2 fat	unknown	737.46*	41.61	110.61	0.38	z'

u(x<sub>pt</sub>) is the measurement uncertainty of the assigned value

σ<sub>pt</sub> is the standard deviation for the proficiency assessment

\* estimated from robust mean calculated from the results of the laboratories that participated in this study

## Results

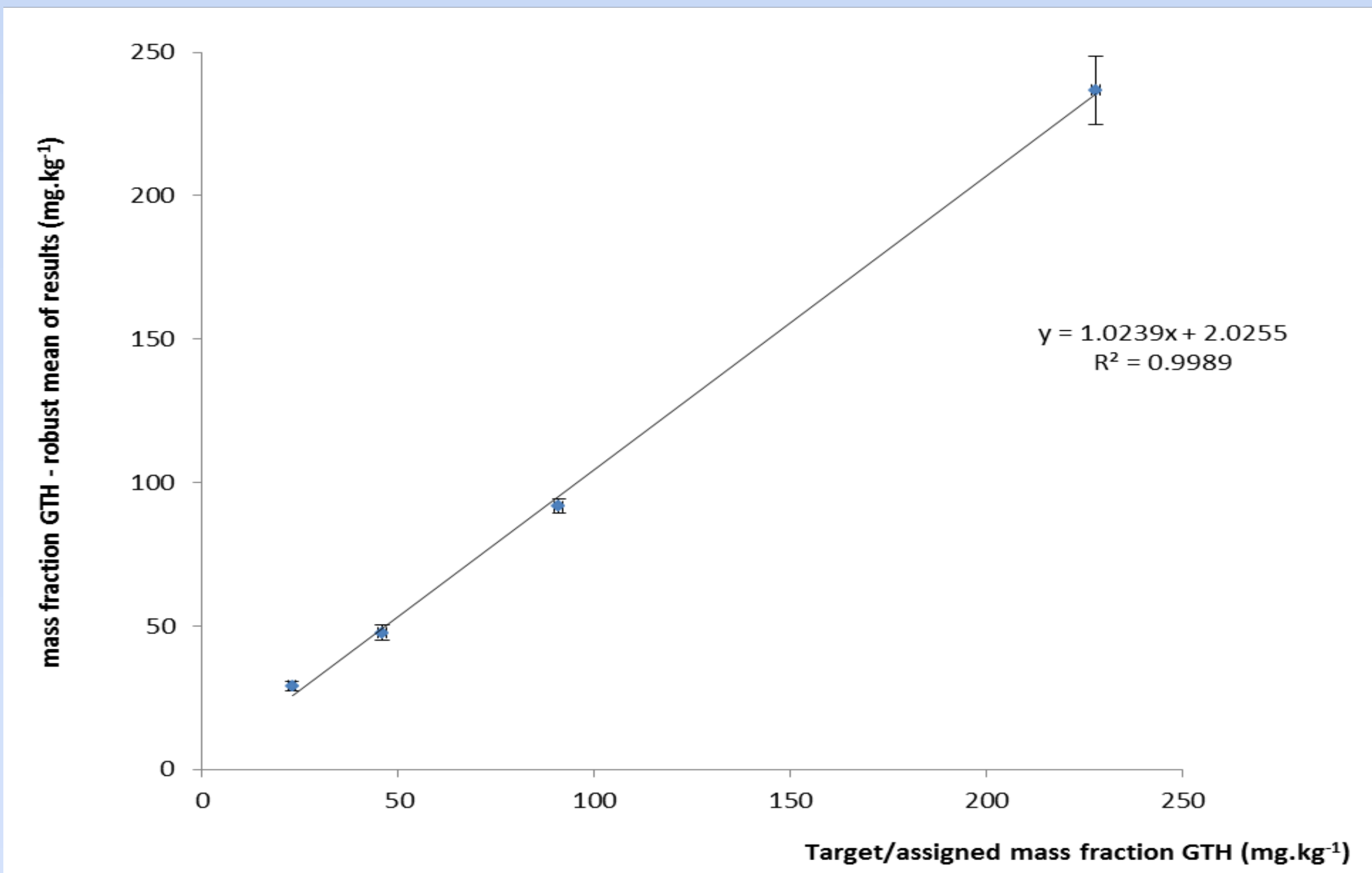
- z and z' performance scores

	GTH 25	GTH 50	GTH 100	GTH 250	Cat 2 Fat
Assigned value [mg kg <sup>-1</sup> ]	22.77	45.55	91.10	227.70	737.46
LAB CODE	z score	z' score			
L1	0.7	-0.3	-0.9	-0.8	-0.9
L2	0.3	-0.2	-0.4	0.2	0.2
L3	1.1	0.5	-0.6	-0.7	-0.7
L4	10.0	7.8	1.8	-2.1	1.1
L5	0.3	-0.1	-0.8	-1.0	-0.2
L6	0.2	1.3	0.2	0.0	-0.1
L7	7.6	4.1	0.6	1.8	3.0
L8	0.3	0.6	0.1	-0.1	0.3
L9	1.3	0.9	0.7	1.1	0.4
L10	3.1	-1.1	0.6	2.8	3.0
L11	1.4	0.5	-0.7	-0.3	-0.3
L12	1.5	-2.3	0.4	2.1	-0.5
L13	-1.3	-1.8	-0.1	5.0	-1.1
L14	2.4	1.1	0.8	0.4	0.3
L15	1.1	0.2	-0.2	0.7	0.3
L16	3.1	2.7	1.4	1.4	2.5
L17	2.7	-1.8	-2.5	-1.9	-2.0
L19	0.3	1.5	0.1	-0.7	-0.5
L20	0.4	-1.4	-0.3	-0.1	-2.4
Number of laboratories	19	19	19	19	19
Satisfactory	13	15	18	15	15
Questionable	2	2	1	3	2
Unsatisfactory	4	2	0	1	2
% satisfactory z or z'	68 %	79 %	95 %	79 %	79 %

no false positive results for the blank

- Fitness for purpose

	x <sub>pt</sub> [mg kg <sup>-1</sup> ]	σ <sub>pt</sub> [mg kg <sup>-1</sup> ]	Robust mean [mg kg <sup>-1</sup> ]	Robust standard deviation [mg kg <sup>-1</sup> ]	RSD [%]	Standard Error
GTH 25	22.77	4.55	29.16	6.74	23.1	1.55
GTH 50	45.55	6.83	47.70	12.04	25.2	2.76
GTH 100	91.10	13.66	91.72	10.56	11.5	2.42
GTH 250	227.70	34.15	236.78	51.67	21.8	11.86
Cat 2 Fat	-	110.61	737.46	140.14	19.0	32.15



## Conclusions

- The GC-MS method is well implemented as indicated by a high number of satisfactory z-scores at mass fractions at 91 mg kg<sup>-1</sup> and above this level. The few number of unsatisfactory scores are all due to overestimations of the GTH content.
- The method is fit for purpose for current legal implementation.
- No revision of the method is necessary when used to verify the correct addition of GTH to target ABPs. However, the results hint at limitations as to the detection of GTH at low levels, which would be the case for other matrices such as fertilisers or compound feed. For these matrices a thorough revision of the method seems to be necessary.