

Analytical approach suitable for

Determination of 2(5H)-furanone and 3-methyl-2(5H)-furanone in food (meat, cheese and fish) with smoke flavour

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The EU Reference Laboratory for Processing Contaminants was asked about suitable methods and expected achievable Limits of Quantifications (LOQs) for determination of 2(5H)-furanone in foods, including meat, cheese and fish, The compounds are relevant for smoked, via a smoking process or smoke flavour, foods.

Conclusion

The EURL-PC has tested a QUECHERS alike approach using GC-MS/MS for the determination of 2(5H)-furanone and 3-methyl-2(5H)-furanone in food (meat, cheese and fish) and has achieved acceptable results. The lowest accepted levels were 50 µg/kg and 10 µg/kg for 2(5H)-furanone and 3-methyl-2(5H)-furanone, respectively, which should therefore be considered to be the preliminary Limits of Quantification (LOQ) for the two compounds.

Limitation

This approach has been tested, but not finally validated due to a very short deadline given for the work. Therefore, appropriate validation should be performed by laboratories using the described approach.

Scope

Foods – the approach has been tested on meat, cheese and fish.

Methodology

The determination of the following compounds has been included in this document:

Compound	CAS No.
2(5H)-furanone	497-23-4
3-methyl-2(5H)-furanone	22122-36-7

1. A representative sample should be homogenised and 4 gram of sample should be mixed in a 50 mL centrifuge tube. Add two ceramic pieces and add 10 mL water and 10 mL acetonitrile (ACN).
2. Shake for 10 min.
3. Add 4 gram $MgSO_4$, 1 gram NaCl, 1 gram Na_3 citrate dihydrate and $\frac{1}{2}$ gram Na_2H citratesesquihydrate. Shake for 10 min.
4. Centrifuge using 2500g for 5 min.
5. Add 4 mL water into 15 mL EMR lipid dSPE tube. Add aliquot of 4 mL of extract.
6. Shake 10 min and centrifuge 5 min. using 2500g.
7. Supernatant is transferred into a 15 mL EMR lipid Polish tube.
8. Shake 10 min. and centrifuge using 2500g for 5 min.
9. Transfer the final extract into sampler vials and analyse by GC-MS/MS.

Instrumentation

GC-MS/MS instrument equipped with a DB-5ms alike column, 30 m, 250 μ m, 0.25 μ m
He-flow: 0.9 mL/min.

Injection: 1 μ L splitless at 260°C for 1 min. It is important to keep a clean inlet to insure sharp peaks.
Temperature program: 50°C for 2 min., 30°C/min. to 85°C, 5°C/min. to 95°C, 30°C/min. to 265°C for 5 min.

Compounds	RT [min.]	Precursor [m/z]	Product [m/z]	CE [V]
2(5H)-furanone	5.6	84	55	10
2(5H)-furanone	5.6	84	56	5
2(5H)-furanone	5.6	55	41	45
3-Methyl-2(5H)-furanone	6.2	98	69	5
3-Methyl-2(5H)-furanone	6.2	98	41.1	20

Results from preliminary test of the method

Two concentration levels (10 μ g/kg and 50 μ g/kg) in meat, fish tissue and cheese has been tested for estimating trueness, precision and LOQ. Five replicates for each sample type and concentration level are analysed and content in the blank sample material was subtracted from the spike level.

Precision is below 15% was achieved and mean recoveries are in the range 95 – 114%. For cheese, though, one value was at 123% and one value at 130% for 2(5H)-furanone and 3-methyl-2(5H)-furanone, respectively. For the 10 μ g/kg spike level for meat one value was 77% for 3-methyl-2(5H)-furanone.

2(5H)-furanone

Sample	N	Level	Repeatability	Recovery
Fish muscle	5	50 µg/kg	8%	106%
Meat	5	50 µg/kg	9%	97%
Cheese	4	50 µg/kg	10%	108%

*Acceptable results were not achieved for 10 µg/kg

3-methyl-2(5H)-furanone

Sample	N	Level	Repeatability	Recovery
Fish muscle	5	10 µg/kg	15%	104%
	5	50 µg/kg	10%	101%
Meat	5	10 µg/kg	7%	100%
	5	50 µg/kg	6%	95%
Cheese	5	10 µg/kg	5%	100%
	4	50 µg/kg	12%	114%

Ion ratio (for the qualifier ion) for all spiked samples were comparable to ion ratio of the standards (using 20% tolerance). Limit of quantification calculated as 6 times repeatability standard deviation resulted in LOQs of 24 - 29 µg/kg for 2(5H)-furanone, and 2.9 - 8.9 µg/kg for 3-methyl-2(5H)-furanone for all samples. However, as the lowest tested levels accepted were 50 µg/kg and 10 µg/kg, respectively, these levels are suggested as LOQs.

2(5H)-furanone

Sample	LOQ (6*SDr) [µg/kg]	LOQ (Lowest accepted level tested) [µg/kg]
Fish muscle	24	50
Meat	27	50
Cheese	29	50

3-methyl-2(5H)-furanone

Sample type	LOQ (6*SDr) [µg/kg]	LOQ (Lowest tested level) [µg/kg]
Fish muscle	8.9	10
Meat	4.5	10
Cheese	2.9	10

Reference

Qiong, Wu, Xiaomei, Shi, Dandan, Ren, Shuya, Wan, Haijin, Liu, Yunfeng, Xie and Liushui Yan. (2021) A rapid and sensitive method for simultaneous quantification of seven furfural compounds in milk powder based on GC-MS/MS combined with QuEChERS method. Food Science and Technology Research, 27 (4), 671–679. doi: 10.3136/fstr.27.671