Slovenian results within JRC-IRMM organised boar taint reference method ring test

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Introduction

A reference method for the determination of skatole, indole and androstenone was developed by JRC-IRMM in Geel. The method was tested in an inter-laboratory comparison study in 2014.

15 laboratories from 9 countries (AT, BE, DE, DK, FR, IT, SI, SP, SR) participated.

The training was provided to the participants, as well as analytical procedure, calibration check standards in toluene and methanol, deuterated standards and GPC column.

Materials and methods

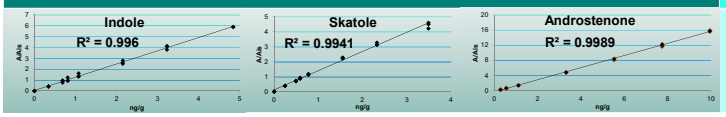
Five samples were provided: 2x tissue and 3x lard. The samples were received mill frozen, and were weighed in the test tubes and liquefied in water bath at 90°C with the addition of Na_2SO_4. Liquefied lipids were centrifuged for 5 min at 40°C and 3220g.

1 ± 0.01 g water-free liquid fat was transferred in a glass vial, isotopically labelled internal standards and GPC eluent were added. Samples were filtered through 0.5 µm PET syringe filter in a HPLC vial. 750 µL of samples were injected to GPC column (the HPLC injection loop volume had to be adopted!) and the fraction between 25 and 37 min was manually collected. For GC-MS, nonane was added and the samples were vacuum dried at 40°C, the nonane extract was analysed. For LC-APCI-MS/MS 1-octanol was added and the samples were vacuum dried at 40°C, the 1-octanol extract was analysed.

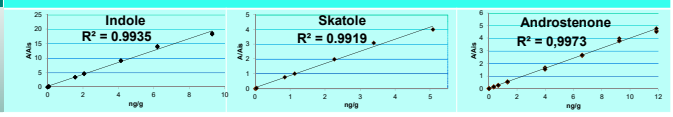
GC conditions	Compound	GC, SIM parameters	LC-MS/MS transitions		HPLC conditions
HP 5 MS, 30 m * 0,25 mm, 0.25 µm	indole	117, 90	118.0 - 65.1	118.0 - 91.0	Luna 150 x 4.6 mm, 5 µm 100 A
	Indole-D7	123	124.0 - 95.9		
Liner temperature 250°C	skatole	130, 103	132.1 - 117.0	132.1 - 89.1	0.6 mL/min, V_{inj}= 5 µL
	Skatole-D3	132	135.1 - 117		
Mobile phase: He, 1 mL/min	5-chloroindole	151	152.0 - 117		A: 0.1 % formic acid B: 0.1% formic MeOH
	Androstenone	272, 257	273.2 - 255.0	273.2 - 159.0	
	Androstenone- D4	276	277.2 - 259		

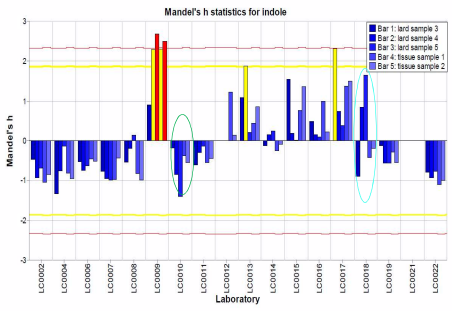
Results

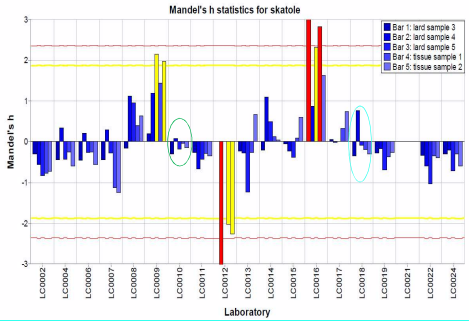
GC-MS

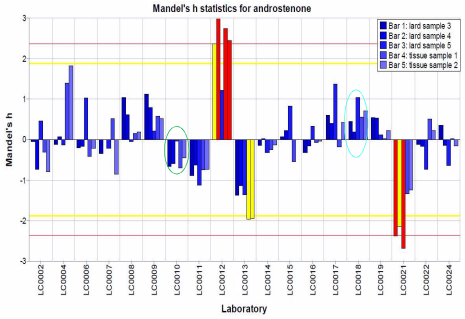


HPLC-APCI-MS/MS









Conclusions

The reference method is robust, free from matrix interferences and sensitive enough to determine the off-flavour compounds at the sensory threshold values with acceptable analytical precision. Method performance characteristics are compliant with requirements for official control methods in the area of food contaminants **but** it uses laboratory instrumentation which is not a common for a typical analytical laboratory (GPC with large volume injection, fraction collector, APCI ion source for MS/MS), isotopically labeled standards, which are very expensive and requires a lot of time.

References: Buttinger G., Wenzl T. (2014). Inter-laboratory validation of a reference method for the determination of boar taint compounds by GC-MS and LC-MSMS, JRC91075

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