Food Authenticity — Determination of 16-O-Methylcafestol Content of Green and Roasted Coffee HPLC-Method Validation Study of CEN/TC-460 WG 3

S8-

Karl Speer, S. Uhlig[1], K. Simon[1], K. Frost[1], A. Schlierf[1]

TECHNISCHE UNIVERSITÄT DRESDEN

Introduction / Methods

In blended coffees of *Coffea arabica* and *Coffea canephora var. robusta*, the botanical origin can be determined by analyzing the concentration of the diterpene 16-O-methylcafestol (16-OMC) [1]. Whereas Arabica coffees contain no detectable or only very small amounts of 16-OMC (less than 20 mg/kg), the concentrations in Robusta coffees are significantly higher in the approximate range of 800 to 2500 mg/kg [2].

On behalf of the German Federal Office of Consumer Protection and Food Safety (BVL), an interlaboratory study for method validation was conducted for the determination of 16-OMC in coffee by means of HPLC. The ring trial was organized by members of the working-group “Coffee and coffee products” (CEN/TC-460 WG 3) and the working-group “NMR analysis” (CEN/TC-460 WG 4). The aim was to shorten and simplify the proven DIN 10779 HPLC-method [3].

DIN 10799	New DIN EN 18003
Lipid extraction Soxhlet	
Saponification Round bottom flask	Saponification Test tube
Liquid-liquid extraction Separation funnel	Liquid-liquid extraction Test tube
HPLC-UV	HPLC-UV
2½ days	8 hours

Validation data

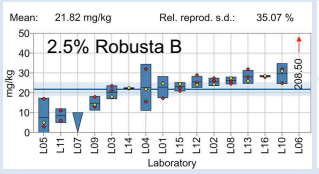
16 European laboratories (Italy 2, France 2, Germany 12) analyzed 8 coffee samples in triplicates each:

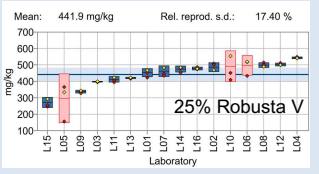
- Arabica + Robusta Brazil (B) with Robusta proportion of 2.5%, 5%, 7.5%, 10%
- Arabica + Robusta Vietnam (V) with Robusta proportion of 25%, 35%, 50%
- Green coffee beans of the Robusta Vietnam

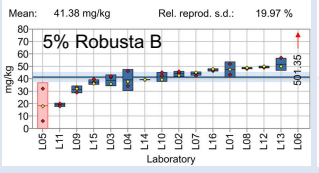
The statistical analyses were performed by **QuoData GmbH[1], Dresden, Germany,** in accordance with ASU § 64 LFGB on the basis of statistical approaches according to DIN ISO 5725-3. The graphics of the results are shown.

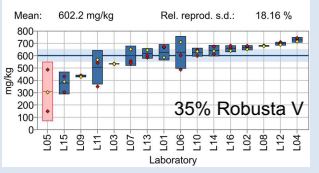
Legend for the graphs:

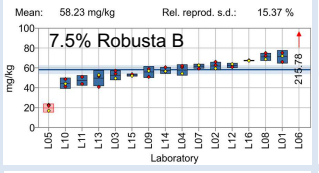
L01 – L16:	laboratory number
Blue boxes:	measurement values and variability for the corresponding laboratory
Red diamonds:	analysis day 1
Yellow diamonds:	analysis day 2
Horizontal blue line:	laboratory mean value
Red boxes:	measurement values of the outlier laboratories (values were not considered)
Red arrow:	Laboratories whose values lie outside the y-axis, the mean value is provided
Triangle:	below the quantification limit (LOQ), upper edge indicates the LOQ
Blue horizontal line:	overall mean value
Light blue band:	expanded measurement uncertainty (± U)

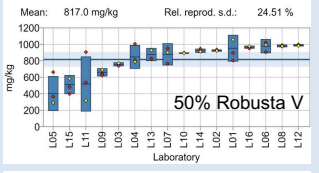


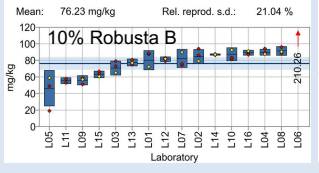


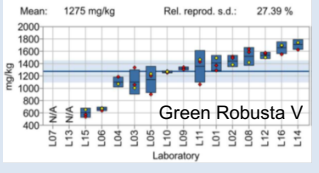












References

[1] Speer K, Tewis R, Montag A (1991), 16-O-Methylcafestol: a quality indicator for coffee. *Proc.14th ASIC Coll.*, ASIC, Paris, 237-244.

[2] Speer K, Kölling-Speer I (2006), The lipid fraction of the coffee bean. Braz. J. Plant. Physiol.,18, 201-216.

[3] DIN 10779: 2011-03

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Conclusions

- The new method is much faster and fulfills the actual environmental requirements.
- The new method is successfully validated for the determination of 16-OMC in green and roasted coffee in the range from 40 to 1500 mg/kg.
- 16-OMC levels around 20 mg/kg were also successfully analyzed by a number of laboratories.