Facing analytical quality.

Ring test
Pyrrolizidine alkaloids in honey
P2115-RT

Summary

The entire report is available to participants only.

Designed, realised and evaluated by

PROOF-ACS GmbH
Bremen, Germany

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Dr. Birgit Schindler

The proficiency test evaluates the performances of laboratories with respect to their ability to quantify pyrrolizidine alkaloids (PA) in honey. 21 pyrrolizidine alkaloids as well as 14 co-eluting pyrrolizidine alkaloids according to Commission Regulation 2020/2040 are within the scope of the test.

Honey with incurred residues of pyrrolizidine alkaloids (PA) was used as raw material for the preparation of the test material and was provided as blank material.

The honey contains incurred residues of four PAs:

europine, senecionine, seneciphylline, and usaramine/retrorsine.

Furthermore, four PAs are spiked to the raw material in order to prepare the test material:

heliosupine, intermedine, senkirkine, and usaramine.

According to Commission Regulation (EU) 2020/2040 co-elution is known for the spiked PAs as follows:

- heliosupine: echimidine
- intermedine: echinatine, indicine, lycopsamine, and rinderine.
- usaramine: retrorsine

Results, which were reported as retrorsine and/or usaramine were considered as sum of retrorsine and usaramine for evaluation. Results, which were reported as echimidine and heliosupine are considered for evaluation of heliosupine. Results, which were reported as senecionine, senecivernine and integerrimine are considered for evaluation of senecionine. Co-elution was not relevant for evaluation of the other parameters.

8 laboratories across four European countries (Austria, France, Germany, and Spain) took part in the test. All laboratories reported results and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct *identification* of 7 pyrrolizidine alkaloids (4 spiked and 3 incurred).
- the *comparability* of the results. The evaluation of the comparability is based on the z-score model. The z-score should be at least ≤ |2|. The comparability criterion is applied to 6 out of 7 pyrrolizidine alkaloids.
- the *trueness* of the results. The trueness is expressed as the coverage of the spiked level in %. The coverage should be at least between 70 and 120 % of the spiked level. The trueness criterion is applied to 3 out of 4 spiked PAs.

Results

Pyrrolizidine alkaloid	Spiked level [µg/kg]	Assigned value [µg/kg]	Total number of results	**Comparability criterion:** no. of participants, which pass the criterion (z-score ≤ |2|)	**Trueness criterion:** no. of participants which pass the criterion (70-120 % recovery of the spiked level)
Heliosupine	12	-	4	Not applicable	3
Intermedine	18	16.7	8	8	7
Senkirkine	5.0	4.65	8	8	8
Usaramine *[1, 3]	3.0	5.94	8	8	Not applicable
Europine	incurred	2.33	8	8	Not applicable
Senecionine	incurred	7.12	8	7	Not applicable
Seneciphylline	incurred	15.7	8	7	Not applicable

*1 Usaramine: usaramine and retrorsine are considered for evaluation.
*2 The comparability criterion is not applicable to heliosupine due to the limited number of reported results.
*3 The trueness criterion is not applied for evaluation of usaramine. The raw (blank) material contains incurred residues of retrorsine/usaramine and thus the assigned value corresponds to the concentration level of the incurred residues (assigned value: 2.9 µg/kg) plus the spiked concentration (3.0 µg/kg).