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Ring test
Pyrrolizidine alkaloids
in chamomile tea
P2410-RT

Summary

The entire report is available to participants only.

The ring test was designed, realised, evaluated, and authorised on behalf of PROOF-ACS GmbH by

Dr. Birgit Schindler
Managing Director PROOF-ACS GmbH
Project coordinator

The report was approved by

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Participants with any comments or concerns related to this ring test are invited to contact:

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DAkkS Deutsche Akkreditierungsstelle D-EP-22211-01-00

PROOF-ACS is a DAkkS accredited proficiency testing provider according to DIN EN ISO 17043:2010 (D-EP-22211-01-00). This ring test is covered by the scope of accreditation.

PROOF-ACS GmbH does not have any analytical laboratory facilities of its own. Homogeneity testing and stability testing are subcontracted to laboratories, accredited according to DIN EN ISO 17025. The subcontracted laboratory may also participate in the ring tests. If so, the laboratory is treated in the same way as other participants and the same rules of confidentiality apply.

The proficiency test evaluates the performance of laboratories with respect to their ability to quantify pyrrolizidine alkaloids (PA) in chamomile tea. 21 pyrrolizidine alkaloids as well as 14 co-eluting pyrrolizidine alkaloids according to Commission Regulation (EU) 2023/915 are within the scope of the test.

Chamomile tea with incurred residues of PAs was used as raw material for the preparation of the test material and the blank material.

The chamomile tea contains incurred residues of *intermedine, lycopsamine, intermedine-N-oxide, and lycopsamine-N-oxide*. The incurred residues of intermedine are considered for evaluation.

Seven PAs are spiked to the milled and homogenised raw materials to prepare the test material:

europine, heliotrine-N-oxide, retrorsine-N-oxide, senecionine, seneciphylline-N-oxide, senecivernine-N-oxide, and senkirkine.

According to Commission Regulation (EU) 2023/915 coelution is known for the PAs mentioned above as follows:

- retrorsine-N-oxide and usaramine-N-oxide,
- senecionine, integerrimine, and senecivernine,
- seneciphylline-N-oxide and spartioidine-N-oxide,
- senecivernine-N-oxide and senecionine-N-oxide.

Europine, heliotrine-N-oxide, and senkirkine are evaluation with respect to the individual spiked PAs. Retrorsine-N-oxide, senecionine, seneciphylline-N-oxide, and senecivernine-N-oxide are reported as sum values by most of the labs. The sum is considered for evaluation, as the other parameters of the sum are not present in the test material.

14 laboratories across five countries (France, Germany, Greece, Italy, and South Africa) took part in the test. All 14 laboratories reported results and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct *identification* of 8 PAs (7 spiked and 1 incurred).
- the *comparability* of the results. The evaluation of the comparability is based on the z-score model. The absolute values of z-scores should be at least ≤ 2. The comparability criterion is applied to all PAs.
- 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 the 7 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)
Europine	11	10.2	13	12	9
Heliotrine-N-oxide	23	20.8	13	13	13
Retrorsine-N-oxide *	44	38.1	14	13	11
Senecionine **	15	11.9	14	13	10
Seneciphylline-N-oxide ***	35	30.2	13	11	10
Senecivernine-N-oxide ****	12	11.7	13	12	11
Senkirkine	9.0	8.04	14	13	10
Intermedine	incurred	7.84	7	6	Not applicable

* The sum of retrorsine-NO and usaramine-NO was considered for evaluation.
** The sum of senecionine, senecivernine, and integerrimine was considered for evaluation.
*** The sum of seneciphylline-NO and spartioidine-NO was considered for evaluation.
**** The sum of senecivernine-NO and senecionine-NO was considered for evaluation.