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Ring test
Pyrrolizidine alkaloids in rooibos tea
P2307-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
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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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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 rooibos tea. 21 pyrrolizidine alkaloids as well as 14 co-eluting pyrrolizidine alkaloids according to Commission Regulation 2020/2040 are within the scope of the test.

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

The rooibos tea contains incurred residues of *retrorsine-N-oxide,* senecionine-N-oxide, *and usaramine-N*-oxide as well as trace levels of further PAs.

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

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

According to Commission Regulation (EU) 2020/2040 coelution is known for the PAs mentioned above as follows:

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

Europine, retrorsine, senecionine, seneciphylline, senkirkine and trichodesmine are evaluation with respect to the individual spiked PAs. Senecivernine-N-oxide is evaluated as individual PA with respect to the trueness and as sum of senecionine-N-oxide, senecivernine-N-oxide, and integerrimine-N-oxide with respect to the comparability criterion. The incurred residues of retrorsine-N-oxide and usaramine-N-oxide are evaluated as sum of retrorsine-N-oxide and usaramine-N-oxide with respect to the comparability criterion.

10 laboratories across five countries (Belgium, France, Germany, Poland, and South Africa) took part in the test. All 10 laboratories reported results and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct *identification* of 8 PAs (6 spiked and 2 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 all PAs except trichodesmine.
- 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.
- Trichodesmine is not within the scope as defined by Commission Regulation (EU) 2020/2040 and the evaluation is thus presented for information only.

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	26	25.5	10	10	9
Retrorsine	53	47.7	10	10	9
Senecionine	35	32.2	10	10	8
Seneciphylline	18	13.8	10	10	9
Senecivernine-N-oxide	44	-	6	Not applicable	6
Sum of senecionine-N-oxide / senecivernine-N-oxide / integerrimine-N-oxide	-	68.2	10	10	Not applicable
Senkirkine	65	52.3	10	10	8
Sum of retrorsine-N-oxide / usaramine-N-oxide	incurred	12.1	8	8	Not applicable
Trichodesmine*	33	-	6	Not applicable	4

* Trichodesmine is provided as an additional parameter. The results are not considered for the evaluation of the performance of the labs.