Facing analytical quality.

proof acs

Ring test
Pyrrolizidine alkaloids in rooibos tea
P2116-RT

Summary

The entire report is available to participants only.

Designed, realised and evaluated by

PROOF-ACS GmbH
Bremen, Germany

September / October 2021,

Schindler

Dr. Birgit Schindler

The proficiency test evaluates the performances 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 (EU) 2020/2040 amending Regulation (EC) No 1881/2006 are within the scope of the test. Rooibos tea with incurred residues of pyrrolizidine alkaloids (PA) was used as raw material for the preparation of the test material.

Incurred residues of five PAs are considered for evaluation:

retrorsine-N-oxide, senecionine, senecionine-N-oxide (sum of senecionine-N-oxide, senecivernine-N-oxide, and integerrimine-N-oxide), seneciphylline, and seneciphylline-N-oxide.

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

heliosupine-N-oxide, indicine, lasiocarpine-N-oxide, retrorsine, and senkirkine.

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

- Heliosupine-N-oxide: echimidine-N-oxide
- Indicine: echinatine, intermedine, lycopsamine, rinderine
- Lasiocarpine-N-oxide: no co-elution
- Retrorsine: usaramine
- Senkirkine: no co-elution

Co-eluting PAs are considered for indicine and senecionine-N-oxide during evaluation.

18 laboratories across eight countries (Croatia, France, Germany, Italy, Norway, South Africa, Switzerland, Turkey) took part in the test. 16 laboratories reported results and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct *identification* of 10 pyrrolizidine alkaloids (5 spiked and 5 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 pyrrolizidine alkaloids except heliosupine-N-oxide.
- 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 heliosupine-N-oxide, indicine and lasiocarpine and is provided for information related to retrorsine and senkirkine due to incurred residues in the blank material.

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-N-oxide	26	-	7	Not applicable	5
Indicine (sum) *[1]	93	92.2	14	13	11
Lasiocarpine-N-oxide	12	12.4	11	10	9
Retrorsine *[2]	75	70.1	16	15	For information only
Senkirkine *[3]	85	78.9	16	14	For information only
Retrorsine-N-oxide	incurred	39.4	16	11	Not applicable
Senecionine	incurred	35.3	15	12	Not applicable
Senecionine-N-oxide (sum) *[4]	incurred	155	15	12	Not applicable
Seneciphylline	incurred	25.4	15	13	Not applicable
Seneciphylline-N-oxide	incurred	93.6	15	12	Not applicable

*[1] Indicine (sum): indicine and lycopsamine are considered for evaluation.

*[2] The raw material contains incurred residues of retrorsine (assigned value 12 µg/kg). The evaluation with respect to the trueness criterion is provided for information only.

*[3] The raw material contains incurred residues of senkirkine (assigned value 15 µg/kg). The evaluation with respect to the trueness criterion is provided for information only.

*[4] Senecionine-N-oxide (sum): senecionine-N-oxide, senecivernine-N-oxide and integerrimine-N-oxide are considered for evaluation.