Facing analytical quality.

proof acs

Ring test
Pyrrolizidine alkaloids and tropane alkaloids in cumin
P2507-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

Dr. Birgit Schindler
cn=Dr. Birgit Schindler,
o=PROOF-ACS GmbH,
ou=Managing Director,
email=proof@proof-acs.de,
c=DE
2025.12.05 13:44:07 +01'00'

Dr. Birgit Schindler

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

PROOF-ACS GmbH
Gottlieb-Daimler-Str. 1
28237 Bremen
Phone: +49 421 388 928 50
E-mail: proof@proof-acs.de
www.proof-acs.de

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

PROOF-ACS is a DAkkS accredited proficiency testing provider according to DIN EN ISO 17043:2023 (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) as well as the tropane alkaloids (TA) hyoscyamine (atropine) and scopolamine in cumin. 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.

Cumin powder with incurred residues of PAs is used as raw material for the preparation of the test material and the blank material.

The cumin powder contains incurred residues of *europine, europine-NO, heliotrine, heliotrine-NO, lasiocarpine, and lasiocarpine-NO*. Homogeneity is not confirmed for the incurred residues and thus the correct identification of the incurred PAs is considered for evaluation only.

Five PAs and two TAs are spiked to the milled and homogenised raw material to prepare the test material:

retrorsine, usaramine-NO, senecionine, senkirkine, monocrotaline, hyoscyamine, and scopolamine.

Monocrotaline is not considered to calculate the sum of PAs according to Commission Regulation (EU) 2023/915. The evaluation is presented for information only.

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

- retrorsine and usaramine,
- retrorsine-NO and usaramine-NO, and
- senecionine, senecivernine, and integerrimine.

No coelution is described for monocrotaline and senkirkine.

15 laboratories across nine countries (Belgium, France, Germany, Greece, Ireland, Italy, Poland, Serbia, and South Africa) took part in the test. 13 laboratories kept the deadline for reporting of results and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct *identification* of 10 PAs (4 spiked and 6 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 4 spiked PAs and 2 spiked TAs.
- 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 4 spiked PAs and 2 spiked TAs.

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)
Retrorsine*	44	38.2	13	11	9
Usaramine-NO**	22	20.8	13	10	9
Senecionine***	60	47.9	12	9	6
Senkirkine	17	14.9	13	13	12
Monocrotaline****	12	9.94	7	Not evaluated	
Europine	incurred	-	11	Not applicable	
Europine-NO	incurred	-	12	Not applicable	
Heliotrine	incurred	-	12	Not applicable	
Heliotrine-NO	incurred	-	12	Not applicable	
Lasiocarpine	incurred	-	12	Not applicable	
Lasiocarpine-NO	incurred	-	12	Not applicable	
Hyoscyamine	25	26.8	11	11	8
Scopolamine	17.0	17.1	12	10	9

* The sum of retrorsine and usaramine is considered for evaluation.
** The sum of usaramine-NO and retrorsine-NO is considered for evaluation.
*** The sum of senecionine, senecivernine, and integerrimine is considered for evaluation.
**** The evaluation with respect to monocrotaline is presented for information only.