

Ring test  
Pyrrolizidine alkaloids  
in sage  
P2609-RT



## Summary

The entire report is available to participants only.

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

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

The report is approved by

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](mailto:proof@proof-acs.de)  
[www.proof-acs.de](http://www.proof-acs.de)



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.

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The proficiency test evaluates the performance of laboratories with respect to their ability to quantify pyrrolizidine alkaloids (PA) in dried sage. 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.

Dried sage is used as raw material for the preparation of the test material and the blank material. The sage is milled to a fine powder and homogenised thereafter.

The dried sage (blank material) contains incurred residues of *europine*, *europine-NO*, and *lycopsamine-NO* at low concentration levels. Homogeneity is not confirmed for the incurred residues. The incurred PAs are not considered for evaluation.

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

*indicine-NO*, *intermedine*, *lycopsamine*, *retrorsine-NO*, *senecionine-NO*, *seneciphylline*, *seneciphylline-NO*, and *senkirkine*.

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

- *indicine-NO* with *intermedine-NO*, *lycopsamine-NO*, *echinatine-NO*, and *rinderine-NO*.
- *intermedine* and *lycopsamine*, with each other and with *indicine*, *echinatine*, and *rinderine*.
- *senecionine-NO* with *senecivernine-NO*, and *integerrimine-NO*.
- *seneciphylline* with *spartioidine*.
- *seneciphylline-NO* with *spartioidine-NO*.
- *retrorsine-NO* with *usaramine-NO*.

No coelution is described for *senkirkine*.

11 laboratories across six countries (Cyprus, France, Germany, Greece, Italy, and South Africa) took part in the test. 10 labs reported results, kept the deadline for reporting of results, and are considered for evaluation.

The performance of laboratories is evaluated according to:

- the correct identification of 8 spiked PAs.
- 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  $\leq 2$ . The comparability criterion is applied to all spiked 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 all spiked PAs.

## Results

Pyrrrolizidine alkaloid	Spiked level [µg/kg]	Assigned value [µg/kg]	Total number of results	Comparability criterion: no. of participants, which pass the criterion ( $ z\text{-score}  \leq 2$ )	Trueness criterion: no. of participants which pass the criterion (70-120 % recovery of the spiked level)
Indicine-NO* <sup>1</sup>	36	33.6	9	9	9
Intermedine	22	-	6	Not applicable	5
Lycopsamine	11	-	4	Not applicable	3
Sum of intermedine and lycopsamine* <sup>2</sup>	33	32.8	9	9	8
Retrorsine-NO* <sup>3</sup>	13	12.9	10	10	7
Senecionine-NO* <sup>4</sup>	65	54.7	10	9	6
Seneciphylline* <sup>5</sup>	41	33.6	10	9	8
Seneciphylline-NO* <sup>6</sup>	33	29.7	10	9	9
Senkirkine	28	24.4	10	9	8

\*<sup>1</sup> The sum of indicine-NO, intermedine-NO, lycopsamine-NO, echinatine-NO, and rinderine-NO is considered for evaluation.

\*<sup>2</sup> The sum of intermedine, lycopsamine, indicine, echinatine, and rinderine is considered for evaluation.

\*<sup>3</sup> The sum of retrorsine-NO and usaramine-NO is considered for evaluation.

\*<sup>4</sup> The sum of senecionine-NO, senecivernine-NO, and integerrimine-NO is considered for evaluation.

\*<sup>5</sup> The sum of seneciphylline and spartioidine is considered for evaluation.

\*<sup>6</sup> The sum of seneciphylline-NO and spartioidine-NO is considered for evaluation.