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Ring test
Quinolizidine alkaloids
in lupin products
P2309-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 performances of laboratories with respect to their ability to quantify quinolizidine alkaloids at two different concentration levels and in two different typical matrices.

A lupin flour with incurred residues as well as a lupin yogurt with spiked and incurred residues are provided as test materials.

The lupin flour contains angustifoline, lupanine and α-i-lupanine, 13-α-OH-lupanine, and multiflorine. The lupin yogurt is spiked with angustifoline, lupanine, and multiflorine, and contains incurred residues of 13-α-OH-lupanine.

13 laboratories across four countries (Belgium, Germany, Ireland, and Netherlands) took part in the test. 11 labs reported results. The results of 10 labs refer to the quinolizidine alkaloids above for the matrix lupin flour and are considered for evaluation. 8 of the labs reported results related to the quinolizidine alkaloids above for lupine yogurt and are considered for evaluation.

The performance of laboratories in the test is evaluated according to

- 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 alkaloids and both matrices.
- 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 angustifoline, lupanine, and multiflorine in lupin yogurt.

Results

Matrix	Parameter	Spiked level [mg/kg]	Assigned value [mg/kg]	Total number of results	Compara-bility criterion passed*	Trueness criterion passed**
Lupin flour	Angustifoline	incurred	18.4	10	7	Not applicable
	Sum of lupanine, α-iso-lupanine	incurred	148	10	7	Not applicable
	13-α-OH-lupanine	incurred	82.5	10	7	Not applicable
	Multiflorine	incurred	10.3	8	5	Not applicable
Lupin yogurt	Angustifoline	2.2	2.29	8	6	7
	Lupanine	1.9	2.24	8	6	6
	Multiflorine	2.3	2.19	8	6	6
	13-α-OH-lupanine	incurred	0.240	7	5	Not applicable

* no. of participants, with z-score ≤ |2|
** no. of participants with results within 70-120 % recovery of the spiked level

To summarise,

- The applied analytical methods are suitable for the quantification of quinolizidine alkaloids in lupin flour and lupin yogurt.
- The quantification of the quinolizidine alkaloids in the matrix lupin yogurt is more challenging due to the lower concentration levels.
- The overall performance should be improved. The robust standard deviations are higher than the target standard deviations according to Horwitz for both matrices and all alkaloids.
- The overall performance with respect to the trueness in lupin yogurt is good. The assigned values correspond to 95 to 118 % of the spiked levels.