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# Ring test
# Pyrrolizidine alkaloids in cumin
# P2114-RT

# Summary

The entire report is available to participants only.

Designed, realised and evaluated by


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The proficiency test evaluates the performances of laboratories with respect to their ability to quantify pyrrolizidine alkaloids (PA) in cumin. 21 Pyrrolizidine alkaloids as well as 14 co-eluting pyrrolizidine alkaloids according to Commission Regulation 2020/2040 are within the scope of the test.

Cumin powder with incurred residues of pyrrolizidine alkaloids (PA) was used as raw material for the preparation of the test material.

The cumin powder contains incurred residues of five PAs:

*europine, europine-N-oxide, heliotrine, heliotrine-N-oxide, and lasiocarpine-N-oxide.*

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

*indicine, integerrimine, intermedine-N-oxide, lasiocarpine, retrorsine-N-oxide, senecionine-N-oxide, and usaramine.*

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

- Indicine: echinatine, rinderine, lycopsamine
- Integerrimine: senecivernine, senecionine
- Intermedine-N-oxide: indicine-N-oxide, rinderine-N-oxide
- Retrorsine-N-oxide: usaramine-N-oxide
- Usaramine: retrorsine

Consequently, results, which were reported for co-eluting PAs were considered during evaluation of the PAs.

10 laboratories across seven countries (Belgium, France, Germany, Greece, Netherlands, Turkey, Switzerland) 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 12 pyrrolizidine alkaloids (7 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 12 pyrrolizidine alkaloids.
- 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.

## 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) |
|---|---|---|---|---|---|
| Indicine*1 | 90 | 95.1 | 9 | 8 | 6 |
| Integerrimine*2 | 22 | 23.5 | 7 | 5 | 4 |
| Intermedine-N-oxide*3 | 22 | 22.3 | 10 | 9 | 6 |
| Lasiocarpine | 39 | 43.4*6 | 10 | 9 | 9 |
| Retrorsine-N-oxide*4 | 28 | 32.1 | 10 | 7 | 5 |
| Senecionine-N-oxide | 34 | 32.9 | 10 | 9 | 7 |
| Usaramine*5 | 16 | 15.2 | 9 | 8 | 6 |
| Europine | incurred | 14.8 | 10 | 8 | - |
| Europine-N-oxide | incurred | 45.1 | 10 | 9 | - |
| Heliotrine | incurred | 16.4 | 10 | 6 | - |
| Heliotrine-N-oxide | incurred | 36.2 | 10 | 10 | - |
| Lasiocarpine-N-oxide | incurred | 20.3 | 10 | 9 | - |

*1 Indicine: indicine and lycopsamine are considered for evaluation.

*2 Integerrimine: integerrimine and senecionine are considered for evaluation.

*3 Intermedine-N-oxide: intermedine-N-oxide and indicine-N-oxide are considered for evaluation.

*4 Retrorsine-N-oxide: retrorsine-N-oxide and usaramine-N-oxide are considered for evaluation.

*5 Usaramine: usaramine and retrorsine are considered for evaluation.

*6 The raw material contains incurred residues of lasiocarpine (about 8.4 µg/kg). The incurred residues are considered during evaluation of the trueness criterion.