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Ring test
Polar pesticides and contaminants in black tea
P2615-RT

Summary

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

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Project coordinator

The report is 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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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 performances of laboratories with respect to their ability to quantify polar pesticides and contaminants in black tea. 11 laboratories across three countries (Germany, Italy, and Spain) took part in the proficiency test.

The test material is prepared of organic black tea. The raw material is milled in a Retsch ultra-centrifugal mill ZM200. The resulting powder is homogenised intensively and tested for incurred residues thereafter. The raw material contains incurred residues of nicotine and trimesium as well as trace levels of anthraquinone.

The milled and homogenised raw material is provided to all participants as blank material. The raw material is spiked with anthraquinone, biphenyl, chlorate, perchlorate, glyphosate, AMPA, diquat, paraquat, matrine, oxymatrine, 2-chloroethanol, and bromide to prepare the test material. It was up to the laboratories to quantify the full set of 14 parameters or a selection of it. The laboratories are asked to analyse both materials, the test material, and the blank material and to mark parameters, which they did not analyse as “n.a.” (not analysed).

All labs submitted results and are considered for evaluation.

The report contains an assessment related to

- 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 parameters except nicotine and trimesium (incurred residues).
- 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 anthraquinone, biphenyl, chlorate, perchlorate, nicotine, and glyphosate. The comparability criterion is not applicable to all other parameters due to the limited number of reported results.

Results

Parameter	Spiked level [mg/kg]	Assigned value [mg/kg]	Assigned value in % of the spiked level	No. of results	No. of results with a |z-score| ≤ 2	No. of results within 70-120 % of the spiked level
Anthraquinone	0.036	0.0400	111	9	7	7
Biphenyl	0.059	0.0599	102	9	9	8
Chlorate	0.042	0.0432	103	8	8	7
Perchlorate	0.065	0.0644	99	8	8	7
Nicotine	incurred	0.212	-	9	9	not applicable
Glyphosate	0.093	0.0914	98	7	7	7
AMPA	0.044	-	-	7	not applicable	5
Trimesium	incurred	-	-	5	not applicable	not applicable
Diquat	0.088	-	-	4	not applicable	3
Paraquat	0.057	-	-	4	not applicable	3
Matrine	0.053	-	-	6	not applicable	3
Oxymatrine	0.076	-	-	6	not applicable	5
Ethylene oxide (sum)	0.099	-	-	4	not applicable	4
Inorganic bromide	85	-	-	2	not applicable	2

To summarise:

- 11 laboratories took part in the tests. The laboratories were free to choose if they report results related to all 14 parameters or a selection of it. Only one lab (lab 4) reported results related to all 14 parameters.
- 11 labs ordered the basic module, while 8 labs ordered glyphosate, AMPA, and trimesium, 5 labs ordered diquat and paraquat, 7 labs ordered matrine and oxymatrine, 5 labs ordered ethylene oxide, and 2 labs ordered inorganic bromide.
- Nicotine and trimesium are incurred residues, while all other parameters are spiked to the material. Nicotine is evaluated with respect to the comparability criterion. Trimesium is not included in the evaluation of the performance of the labs due to the limited number of reported results.
- Comparability:

  Anthraquinone, biphenyl, chlorate, perchlorate, nicotine, and glyphosate are evaluated with respect to the comparability criterion.
- Trueness:

  The trueness criterion is applied to all parameters except nicotine and trimesium.
- The overall performance of the labs is good, even though the quantification of polar pesticides is challenging in complex matrices like black tea. Most of the labs are able to provide reliable results at least related to the more common parameters of the basic module.
- The most challenging parameters are matrine and oxymatrine as well as diquat and paraquat.