Facing analytical quality.

rolling proof 2016

Module tea and spices

Black pepper – P1614-RT

Summary

The entire report is made available to participants only.

Designed, realised and evaluated by

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The aim of ***rolling proof*** is to offer laboratories the opportunity to test the applied pesticide multi-residue methods for the most relevant pesticides and in different matrices within one cycle of accreditation.

Thus, ***rolling proof*** is developed to support laboratories in meeting the requirements of accreditation bodies. According to advisory document EA-4/18:2010 analytical laboratories are requested to establish a PT participation plan for accredited analytical methods. ***rolling proof*** is an on-going scheme of ring tests.

The module "tea and spices" of ***rolling proof*** is designed for difficult or unique commodities (according to SANTE 11945/2015, Annex A) and includes

- teas like black tea, green tea, herbal tea, fruit tea, rooibos tea etc., and
- spices like pepper, curry powder, paprika powder, etc.

The module "tea and spices" covers all in all a minimum of 150 of the most relevant pesticides. The scope of pesticides covered by ***rolling proof*** is defined in a provided list. All pesticides are tested within a period of five years. Thus, the laboratories that take part in ***rolling proof*** are able to test their pesticide multi-methods for a large number of pesticides and a variety of matrices within one cycle of accreditation. However, it is up to the participants to join all tests of the 5-year programme of ***rolling proof***, or to book the tests individually.

rolling proof evaluates the performance of laboratories according to:

- The correct *identification* of the spiked pesticides.
- 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 *comparability* of the results. The evaluation of the comparability is based on the z-score model. The z-score should be at least ≤ |2|.

In 2016, black pepper is chosen as matrix for ***rolling proof*** – module "tea and spices". Twelve laboratories across four countries (Austria, Germany, Spain and Switzerland) took part in the test. Eleven of them reported results and were considered for evaluation.

The test material was prepared of ground organic black pepper. The raw material was homogenised, tested for incurred residues and spiked with 34 pesticides thereafter. The identity of the pesticides, the spiked levels and a summary of the overall performance of the laboratories are provided in the table below.

Pesticide	Spiked level [mg/kg]	Assigned value [mg/kg]	Total number of results	No. of participants, which pass the **comparability criterion** (z-score ≤ |2|)	No. of participants which pass the **trueness criterion** (70-120 % recovery of the spiked level)
2,4-D	0.081	0.0794	9	9	9
2,4`-DDT	0.049	0.0366	9	5	4
4,4`-DDD	0.015	0.0139	10	8	8
Aldicarb	0.092	0.0849	9	9	9
Azoxystrobin	0.22	0.203	9	9	9
Boscalid	0.061	0.0638	8	8	7
Carbaryl	0.055	0.0496	10	10	9
Chlorpyrifos-methyl	0.067	0.0664	11	11	11
β-Cyfluthrin	0.12	0.108	9	9	7
λ-Cyhalothrin	0.052	0.0462	9	9	7
DEET	0.058	0.0595	10	10	9
Demeton-S-methyl	0.060	0.0525	10	10	6
Diazinon	0.082	0.0814	11	11	11
Dichlofluanid	0.097	0.0884	9	8	6
Dieldrin	0.054	0.0512	11	11	10
Difenoconazole	0.36	0.358	11	9	9
Dimethoate	0.11	0.106	11	11	11
α-Endosulfan	0.050	0.0442	10	10	9
Epoxiconazole	0.032	0.0332	9	9	8
Fenamiphos	0.079	0.0838	11	11	10
Fludioxonil	0.038	0.0376	9	9	7
β-HCH	0.035	0.0270	7	7	5
γ-HCH (Lindane)	0.024	0.0248	9	8	7
Imidacloprid	0.16	0.152	11	11	11
Metolachlor	0.042	0.0416	10	10	9
Parathion	0.091	0.0873	10	10	10
Permethrin	0.16	0.151	10	10	10
Piperonyl butoxide	0.95	0.924	11	10	10
Propamocarb	0.062	0.0616	10	10	9
Tebuconazole	0.021	0.0222	7	6	6
Tecnazene	0.055	0.0510	11	10	10
Thiabendazole	0.088	0.0818	10	10	9
Thiophanate-methyl	0.073	0.0584	11	8	6
Vinclozolin	0.046	0.0457	11	10	10

Summary of the performances of the laboratories:

- **Dried pepper is a challenging matrix**
- **Three laboratories *identified all 34 pesticides* correctly, while three more laboratories still *identified >90 % of the pesticides* (31 resp. 33 pesticides) correctly.**
- **One of the participants reported a *false positive* result of parathion-methyl as parathion-ethyl was spiked to the test material. None of the other participants reported false positive results.**
- ***False negative* results of one or more pesticides were reported by eight out of eleven participants.**
- **None of the laboratoried quantified all 34 pesticides correctly.**
- **Three laboratories quantified *32 resp. 33 out of 34 pesticides* correctly and thus provided the best results with respect to the *comparability*.**
- **With respect to the *trueness* the best results were provided by the same three labs, which quantified *30 resp. 31 out of 34 pesticides* correctly.**