Facing analytical quality.

rolling proof 2015

Module vegetables and fruits

Tomato – P1512-RT

Blueberry – P1513-RT

Summary

The entire report is made available to participants only.

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Summary

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.

Two commodity groups (according to SANCO 12571/2013, Annex A) are included in the ***rolling proof*** module "vegetables and fruits":

- vegetables and fruits (high water content),
- citrus fruits, small fruits and berries (high acid content).

Two test materials are provided for the module "vegetables and fruits" in each year, one for each of the two commodity groups above.

In 2015, tomato and blueberries are chosen as matrices for ***rolling proof*** – module "vegetables and fruits".

A list of pesticides is provided to the participating laboratories, which defines the scope of pesticides, covered by ***rolling proof***. The module "vegetables and fruits" covers all in all a minimum of 300 pesticides. All pesticides are tested within a period of six 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.

rolling proof evaluates the performance of laboratories with respect to their ability to identify and quantify pesticides in vegetables and fruits. It is up to the participants to join all tests of the 6-year programme of ***rolling proof***, or to book the tests individually.

In 2015, eight laboratories across five European countries (Austria, Germany, Italy, Netherlands, and Spain) took part in ***rolling proof*** module "vegetables and fruits" for one or both matrices.

The test materials were prepared of organic tomatoes resp. blueberries. The raw materials were homogenised, tested for incurred residues and spiked with pesticides thereafter.

rolling proof evaluates the results 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|.

Test material tomato (P1512-RT)

The test material tomato was spiked with 34 pesticides. 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	**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)
Acrinathrin	0.028	0.0286	7	7	7
Benalaxyl	0.032	0.0313	7	7	6
Bifenazate	0.47	0.215	7	6	Not evaluated
Bitertanol	0.16	0.148	7	7	7
Chloranthraniliprole	0.26	0.365	7	6	Not evaluated
Clofentezine	0.022	0.0154	7	7	5
Cymoxanil	0.051	0.0471	7	7	7
Cyprodinil	0.39	0.349	7	7	7
Deltamethrin	0.31	0.277	7	7	7
Dimethomorph	0.067	0.0604	7	7	7
Etofenprox	0.042	0.0368	7	7	7
Famoxadone	0.055	0.0500	7	7	7
Fenazaquin	0.077	0.0684	7	7	6
Flonicamid	0.031	0.0271	7	7	7
Fluopicolide	0.43	0.357	7	7	7
Hexythiazox	0.51	0.473	7	7	7
Imazalil	1.2	0.566	7	7	Not evaluated
Indoxacarb	0.079	0.0702	7	7	6
Mepanipyrim	0.064	0.0573	7	7	7
Methomyl	0.029	0.0243	7	7	7
Methoxyfenozide	0.082	0.0668	7	7	6
Myclobutanil	0.026	0.0223	7	7	7
Oxamyl	0.046	0.0383	7	7	7
Procymidone	0.11	0.0976	7	7	7
Propargite	0.14	0.140	7	7	7
Pyridaben	0.060	0.0556	7	7	7
Pyriproxyfen	0.039	0.0344	7	7	7
Spinosad	0.086	0.0884	7	7	6
Spiromesifen	0.056	0.0500	7	7	6
Tebufenpyrad	0.43	0.378	7	7	7
Tetraconazole	0.22	0.178	7	7	6
Thiophanate-methyl	0.54	0.460	7	7	7
Triadimenol	0.17	0.156	7	7	7
Zoxamide	0.023	0.0188	7	7	6

Tomato - summary of the performances of participating laboratories:

- **All laboratories reported results.**
- **All laboratories *identified all 34 pesticides* correctly.**
- **The laboratories reported *no false positive results*.**
- **Six out of seven laboratories *quantified all 34 pesticides* correctly with respect to the *comparability criterion*, while one laboratory *quantified 32 out of 34 pesticides* correctly.**
- **Three out of seven laboratories *quantified all 31 pesticides** correctly with respect to the *trueness criterion*, while the other four labs *quantified ≥ 90 % of the evaluated pesticides** correctly.**

  *For scientific reasons, bifenazate, chloranthraniliprole, and imazalil were evaluated with respect to the comparability criterion only.

Test material blueberry (P1513-RT)

The test material blueberry was spiked with 29 pesticides. 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	**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)
Acetamiprid	0.025	0.0239	7	7	7
Azinphos-methyl	0.31	0.329	7	7	5
Azoxystrobin	0.024	0.0220	7	7	7
Boscalid	0.19	0.178	7	7	7
Carbendazim	0.42	0.354	7	7	6
Chlorothalonil	0.078	0.0394	7	7	Not evaluated
λ-Cyhalothrin	0.022	0.0235	7	7	5
DEET	0.056	0.0530	7	7	7
Dimethoate	0.16	0.155	7	7	7
Dodine	0.053	0.0507	7	7	6
Fenbuconazole	0.082	0.0764	7	7	7
Fenhexamid	3.4	3.53	7	6	6
Fludioxonil	0.29	0.275	7	7	7
Flufenacet	0.042	0.0372	7	7	7
Imidacloprid	0.031	0.0297	7	7	6
Iprodione	0.94	0.826	7	7	6
Kresoxim-methyl	0.65	0.613	7	7	6
Phenmedipham	0.22	0.0527	7	6	Not evaluated
Phosmet	0.059	0.0641	7	7	5
Pirimicarb	0.26	0.238	7	7	7
Propyzamide	0.064	0.0603	7	7	7
Pyraclostrobin	0.051	0.0461	7	7	7
Pyrimethanil	0.28	0.258	7	7	7
Rotenone	0.13	0.148	7	5	5
Tebuconazole	0.26	0.235	7	7	7
Thiabendazole	0.073	0.0589	7	7	7
Thiacloprid	0.11	0.102	7	7	7
Tolylfluanid (sum)	0.065	0.0608	7	7	6
Trifloxystrobin	0.043	0.0400	7	7	6

Blueberry - summary of the performance of the laboratories:

- **All laboratories *identified all 29 pesticides* correctly.**
- ***No false positive results* were reported.**
- **Three out of seven laboratories *quantified all 29 pesticides* correctly with respect to the *comparability criterion*, while four more laboratory quantified 28 out of 29 pesticides correctly.**
- **Three out of seven laboratories *quantified 26 out of 27 pesticides** correctly with respect to the *trueness criterion*, while the other four laboratories *quantified > 80 % of the evaluated pesticides** correctly.**

* For scientific reasons, chlorothalonil and phenmedipham were evaluated with respect to the comparability only.