Facing analytical quality.

# rolling proof 2016

## Module vegetables and fruits

## Spinach – P1610-RT

## Kiwi fruit – P1611-RT

# Summary

The entire report is made available to participants only.

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***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 11945/2015, 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 2016, spinach and kiwi fruit 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 2016, ten laboratories across five countries (Austria, Germany, Italy, Spain and Switzerland) took part in ***rolling proof*** module "vegetables and fruits" for one or both matrices.

The test materials were prepared of organic spinach resp. kiwi fruit. 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 spinach (P1610-RT)

The test material spinach was spiked with 28 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) |
|---|---|---|---|---|---|
| 2,4-DB | 0.036 | 0.0340 | 8 | 6 | 6 |
| Chlorpropham | 0.020 | 0.0192 | 9 | 9 | 9 |
| Chlorpyrifos | 0.045 | 0.0403 | 9 | 9 | 9 |
| Clothianidin | 0.18 | 0.169 | 9 | 8 | 7 |
| Cyazofamid | 0.018 | 0.0179 | 9 | 9 | 8 |
| Diazinon | 0.032 | 0.0276 | 9 | 9 | 9 |
| Dodine | 0.35 | 0.299 | 9 | 8 | 7 |
| Epoxiconazole | 0.042 | 0.0409 | 9 | 8 | 7 |
| Etridiazole | 0.070 | 0.0613 | 9 | 9 | 9 |
| Fenpropidin | 0.085 | 0.0829 | 9 | 9 | 9 |
| Fenpropimorph | 0.068 | 0.0693 | 9 | 9 | 8 |
| Fluazifop-P-butyl | 0.25 | 0.238 | 9 | 9 | 8 |
| Imidacloprid | 0.076 | 0.0753 | 9 | 9 | 8 |
| Isoproturon | 0.017 | 0.0173 | 9 | 8 | 8 |
| Lenacil | 0.13 | 0.104 | 9 | 8 | 8 |
| Linuron | 0.038 | 0.0376 | 9 | 9 | 7 |
| Mandipropamid | 0.44 | 0.470 | 9 | 9 | 9 |
| MCPB (free acid) | 0.072 | 0.0680 | 9 | 8 | 8 |
| Metalaxyl-M | 0.080 | 0.0751 | 9 | 9 | 9 |
| Metamitron | 0.095 | 0.0808 | 8 | 8 | 8 |
| Metobromuron | 0.040 | 0.0383 | 9 | 9 | 8 |
| Metribuzin | 0.16 | 0.144 | 9 | 9 | 8 |
| Pendimethalin | 0.051 | 0.0457 | 9 | 9 | 8 |
| Pentachloroaniline | 0.022 | 0.0197 | 8 | 8 | 8 |
| Propamocarb | 0.80 | 0.793 | 9 | 8 | 8 |
| Spiroxamine | 0.048 | 0.0479 | 9 | 9 | 9 |
| Terbuthylazine | 0.022 | 0.0208 | 9 | 9 | 8 |
| Trifluralin | 0.060 | 0.0519 | 9 | 8 | 8 |

**Spinach - summary of the performances of participating laboratories:**

- **Six out of nine laboratories *identified all 28 pesticides* correctly.**
- ***No false positive results* were reported.**
- **Three laboratories *quantified all 28 pesticides* correctly with respect to the *comparability criterion*, while three more laboratory *quantified 27 out of 28 pesticides* correctly.**
- **Two laboratories *quantified all 28 pesticides* correctly with respect to the *trueness criterion*, while the three more labs *quantified 27 out of 28 pesticides correctly*.**

## Test material kiwi fruit (P1611-RT)

The test material kiwi fruit was spiked with 31 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) |
|---|---|---|---|---|---|
| 2,4-DB | 0.023 | 0.0216 | 9 | 9 | 9 |
| 2,4'-DDT | 0.042 | 0.0368 | 9 | 6 | 5 |
| Abamectin B1a | 0.030 | 0.0282 | 8 | 8 | 7 |
| Ametryn | 0.045 | 0.0430 | 10 | 9 | 8 |
| Bifenthrin | 0.025 | 0.0245 | 10 | 10 | 10 |
| Bromacil | 0.025 | 0.0233 | 9 | 9 | 9 |
| Buprofezin | 0.54 | 0.455 | 10 | 10 | 10 |
| Carbaryl | 0.035 | 0.0319 | 10 | 10 | 9 |
| Chlorpyrifos-methyl | 0.085 | 0.0773 | 10 | 10 | 9 |
| Clopyralid | 0.29 | 0.241 | 9 | 8 | 7 |
| α-Cypermethrin | 0.065 | 0.0632 | 10 | 10 | 9 |
| Difenoconazole | 0.071 | 0.0645 | 10 | 10 | 8 |
| Diflubenzuron | 0.098 | 0.101 | 10 | 9 | 8 |
| Diphenylamine | 0.16 | 0.129 | 10 | 9 | 9 |
| Diuron | 0.022 | 0.0205 | 10 | 10 | 9 |
| Fenitrothion | 0.040 | 0.0354 | 10 | 9 | 9 |
| HCB | 0.026 | 0.0257 | 10 | 10 | 8 |
| Imazalil | 0.33 | 0.288 | 10 | 9 | 9 |
| MCPA | 0.064 | 0.0586 | 10 | 10 | 10 |
| Paclobutrazol | 0.18 | 0.159 | 10 | 10 | 9 |
| 2-Phenylphenol | 0.044 | 0.0402 | 10 | 10 | 10 |
| Piperonyl butoxide | 0.12 | 0.105 | 10 | 10 | 10 |
| Prochloraz | 0.095 | 0.0674 | 10 | 9 | 6 |
| Pyrethrins (sum) | 0.45 | 0.456 | 10 | 6 | 4 |
| Spirodiclofen | 0.026 | 0.0262 | 10 | 9 | 9 |
| Tebuconazole | 0.027 | 0.0229 | 10 | 10 | 9 |
| Tebufenozide | 0.35 | 0.339 | 10 | 10 | 10 |
| Triadimefon | 0.14 | 0.126 | 10 | 10 | 9 |
| Triclopyr | 0.13 | 0.114 | 9 | 9 | 8 |
| Triflumuron | 0.056 | 0.0572 | 10 | 10 | 7 |
| Vinclozolin | 0.049 | 0.0434 | 10 | 10 | 10 |

**Kiwi fruit - summary of the performance of the laboratories:**

- **Five laboratories *identified all 31 pesticides* correctly.**
- ***No false positive results* were reported.**
- **Three out of ten laboratories *quantified all 31 pesticides* correctly with respect to the *comparability criterion*, while four more laboratory quantified 28 to 30 out of 31 pesticides correctly (> 90 % of the parameters).**
- **Six laboratories quantified *28 to 30 out of 31 pesticides* (> 90 % of the parameters) correctly with respect to the *trueness criterion*, while none of the laboratories quantified *all 31 pesticides* correctly.**