

5.30 SPICES - MAXIMUM RESIDUE LEVEL RECOMMENDATIONS

The Thirty-sixth Session of CCPR decided (Alinorm 04/24A) to schedule by the JMPR the review of the monitoring data available for the elaboration of MRLs on spices for pesticides already in the Codex system.

Subsequently the 2004 JMPR developed the general principles for evaluation of monitoring data for recommending maximum residue levels, median and high residues depending on the number of residue data available for a given pesticide residue and commodity combination.

In accordance with the decision of the Forty-sixth Session of the CCPR, India submitted monitoring data from 2009-2014 for several pesticide residues in cardamom, black pepper, cumin, fennel and coriander for review by the 2015 JMPR.

Sampling and analytical methods

Cumin, fennel and coriander seed samples (250–500 g) were collected from the retail outlets. No information was provided on sampling of cardamom and black pepper.

The residues in/on cardamom and black pepper were extracted with a mixture of acetonitrile/water. The dried extract was purified with a primary secondary amine (PSA) adsorbent in the presence of MgSO₄, and the residues were identified and quantified by GC-MS/MS or LC-MS/MS.

Seed spices were extracted with the mixture of acetonitrile/water and further determined with a modified QuEChERS multiresidue method using GC-MS/MS and/or LC-MS/MS.

For both methods, the recoveries were within the acceptable range, and reported LOQ was 0.1 mg/kg for all pesticide residue commodity combinations.

Agricultural practices for growing spice producing plants

Cumin, cardamom, coriander, pepper and fennel are minor crops which are mainly cultivated in the southern and western parts of India. The spices need to be protected against several pests and diseases which require repeated application of pesticides around the year.

The capsule of cardamom and unripe drupes for pepper are harvested up to 6 to 8 times a year. Cumin and coriander are harvested only once and fennel, up to 3 to 4 times.

No information was available on registered or approved uses or application conditions of the pesticides.

Principles of evaluation of residues derived from monitoring programmes

Principles for evaluation of monitoring data elaborated by the 2004 JMPR were followed:

- It is assumed that the laboratories reported only valid results. Therefore, all residue data are taken into account without excluding any value as an outlier.
- When residue values were reported as <LOQ, it does not necessarily mean that the sampled commodity was not treated with or exposed to the pesticide. While, it is unlikely that all the sampled commodities were treated with the pesticides looked for with the multi residue procedure, it cannot be assumed to be a 'nil' residue situation.
- When no sample contained detectable residues, the highest reported LOQ value is used as the maximum residue level. When justified based on the consumption, the high and median residue value are taken from the reported LOQ values.
- Distribution-free statistics are used in estimating the maximum residue level, covering the 95th percentile of the residue population at the 95% confidence level. Thus, the estimated maximum residue level encompasses at least 95% of the residues with 95% probability (in

95% of cases). To satisfy this requirement, a minimum of 58–59 samples is required. In such cases the uncertainty derived from the limited number of data points are taken into account in recommending maximum residue levels.

- When > 120 samples contain detected residues, the sample size is sufficiently large to calculate the upper 95% one-tailed confidence limit of the 95th percentile of the population of residues, which should be used for estimation of maximum residue level after rounding up to the next value of the scale of expressing residues according to the OECD MRL calculator.
- Monitoring results are not used for estimating maximum residue levels that reflect post-harvest use.

Furthermore, the Meeting decided that:

Maximum residue levels would only be estimated for those pesticide residues which were determined according to the definition of residues for enforcement purposes. Consequently, the reported residues of carbofuran and imidacloprid were not considered.

Residues resulting from monitoring programmes

Black pepper

Of the 284 samples analysed for acetamiprid, cypermethrin, lambda-cyhalothrin, profenofos, and triazophos, none were found to contain residues at or above the LOQ of 0.1 mg/kg.

The Meeting concluded that the reported LOQ values are higher than those which can be obtained with current analytical methods. Consequently, the Meeting agreed there was no reason to revise its previous recommendations for maximum residue levels for cypermethrin, lambda cyhalothrin, profenofos and triazophos.

The Meeting estimated a maximum residue level and median residue of 0.1 mg/kg for acetamiprid.

Cardamom seed

Results of analyses of 487 samples were reported for acetamiprid, cypermethrin, lambda-cyhalothrin, imidacloprid, profenofos and triazophos.

No residues (< 0.1 mg/kg) of acetamiprid were detected.

Based on the results, the Meeting estimated a maximum residue and median residue of 0.1 mg/kg for acetamiprid.

Out of 487 samples 133 contained cypermethrin residues which were in rank order: 0.10, 0.11 (3), 0.12, 0.13, 0.14 (4), 0.16, 0.18 (2), 0.19 (3), 0.20 (3), 0.21, 0.22 (2), 0.23 (3), 0.24 (2), 0.25, 0.26 (4), 0.27, 0.28 (2), 0.29 (2), 0.30 (2), 0.31 (2), 0.32 (6), 0.34 (5), 0.35(4), 0.36, 0.37 (3), 0.38, 0.39 (2), 0.41 (2), 0.43 (2), 0.44 (4), 0.45 (2), 0.46, 0.47, 0.49, 0.50 (2), 0.52, 0.53 (2), 0.54 (2), 0.55 (2), 0.56, 0.58 (2), 0.59 (2), 0.60, 0.63, 0.64, 0.65, 0.66, 0.69 (2), 0.70 (3), 0.71 (2), 0.73, 0.75 (2), 0.76, 0.77, 0.79, 0.81, 0.86, 0.87(2), 0.91, 0.92, 0.93, 0.99, 1.03, 1.12, 1.16, 1.34, 1.41, 1.54, 1.62, 1.65, 1.67, 1.76, 1.85, 1.94, 1.98, 2.00, 2.24, and 2.97(2) mg/kg.

The upper 95% confidence limit of the detected residues is 2.24 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and a median residue of 0.43 mg/kg for cypermethrin which replaces its previous recommendations.

Out of 487 samples 146 contained lambda cyhalothrin residues which were in rank order: 0.10 (5), 0.11 (4), 0.12 (7), 0.13 (5), 0.14, 0.15 (4), 0.16 (3), 0.18 (3), 0.19 (7), 0.20 (6), 0.21 (5), 0.22, 0.23 (4), 0.24 (6), 0.25 (2), 0.26 (5), 0.27 (3), 0.28 (4), 0.29, 0.31 (2), 0.32 (3), 0.34 (5), 0.35 (3), 0.36 (2), 0.37 (3), 0.38, 0.40 (2), 0.41 (2), 0.42 (3), 0.43, 0.44, 0.45, 0.46, 0.49 (2), 0.50 (2), 0.51, 0.52 (3), 0.53, 0.54, 0.55, 0.57, 0.58 (2), 0.59, 0.61, 0.62 (2), 0.63, 0.67, 0.68, 0.69, 0.71, 0.73, 0.74 (2), 0.79, 0.82 (2), 0.86, 0.96, 0.99, 1.02, 1.04, 1.06, 1.20, 1.33, 1.87, 1.94, and 3.06 mg/kg.

The upper 95% confidence limit of the residues is 1.87 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and a median residue of 0.28 mg/kg for cyhalothrin, which replaces its previous recommendations.

Out of 487 samples 68 contained profenofos residues which were in rank order: 0.10 (3), 0.11 (5), 0.12 (3), 0.13 (2), 0.14 (5), 0.16, 0.17, 0.19 (2), 0.21, 0.22 (3), 0.24 (2), 0.25, 0.27, 0.28, 0.29, 0.30 (3), 0.31, 0.32 (2), 0.34 (2), 0.36, 0.38, 0.39, 0.42 (2), 0.43, 0.44, 0.47 (2), 0.50 (2), 0.53 (2), 0.55, 0.63, 0.65, 0.66, 0.78, 0.79, 0.82, 0.91, 1.08, 1.19, 1.26, 1.54, 1.76, 1.9, and 3.06 mg/kg.

The 95th percentile of the residues is 1.4 mg/kg. The database is insufficient for calculation of the upper confidence limit.

Taking into account the limited database, the Meeting estimated a maximum residue level of 3 mg/kg and a median residue of 0.3 mg/kg for profenofos which replaces its previous recommendations.

Out of 487 samples 79 contained triazophos residues which were in rank order: 0.10, 0.11(2), 0.12(2), 0.14, 0.15, 0.16, 0.17 (4), 0.19 (2), 0.21(5), 0.22, 0.23 (2), 0.25, 0.26, 0.28, 0.29 (3), 0.32, 0.33, 0.34, 0.37(2), 0.39(2), 0.40 (2), 0.43, 0.45 (3), 0.46, 0.47, 0.48, 0.49, 0.5 (2), 0.53 (2), 0.55 (2), 0.58, 0.59, 0.6, 0.61, 0.62, 0.63, 0.64, 0.69, 0.77, 0.82 (2), 0.84, 0.85 (2), 0.86, 1.06, 1.09, 1.11, 1.13, 1.34, 1.38, 1.42, 1.49, 1.67, 1.68, 1.71, 2.30, and 3.64 mg/kg.

The 95th percentile of the residues is 1.7 mg/kg. The database is insufficient for calculation of the upper confidence limit.

Taking into account the limited database, the Meeting estimated a maximum residue level of 4 mg/kg and a median residue of 0.45 mg/kg for triazophos, which replaces the previous recommendations.

Coriander seed

Altogether 223 samples were analysed (positive results in brackets) for acetamiprid (0.02 mg/kg), profenofos (0), phorate (0) and triazophos (0). The reported LOQ was 0.1 mg/kg.

The residue data was not sufficient to estimate a maximum residue level for acetamiprid.

The Meeting estimated maximum and median residue levels of 0.1 mg/kg for profenofos, phorate and triazophos in coriander seed.

Cumin seed

The results of analyses of 447 samples were reported for acetamiprid, phorate and profenofos.

Out of 447 samples acetamiprid (33) and phorate (7) residues were detected above the LOQ of 0.1 mg/kg.

As the number of detected residues is lower than the minimum required (58), no recommendations could be made for maximum residue levels for acetamiprid and phorate.

Out of 447 samples 76 contained profenofos residues which were in rank order: 0.10 (2), 0.11 (2), 0.12, 0.13 (4), 0.14 (2), 0.15 (2), 0.16 (2), 0.17, 0.18, 0.19(2), 0.20, 0.22 (2), 0.24 (2), 0.25, 0.27, 0.31 (2), 0.32, 0.34, 0.38, 0.39, 0.41, 0.42, 0.44, 0.47, 0.56, 0.63, 0.64, 0.65 (2), 0.66, 0.68 (2), 0.73, 0.77, 0.80, 0.82, 0.85, 0.86, 0.94, 0.95, 0.99, 1.03, 1.05, 1.07, 1.10, 1.21, 1.22 (2), 1.26(2), 1.30, 1.38, 1.51, 1.52, 1.61, 1.85, 1.98, 2.11, 2.32, 2.47, 2.69, 2.90, 3.83, and 4.12 mg/kg.

The 95th percentile of the residues is 2.52 mg/kg. The database is insufficient for calculation of the upper confidence limit. Taking into account the limited database, the Meeting estimated a maximum residue level of 5 mg/kg and median residue of 0.635 mg/kg for profenofos.

Fennel, seed

Altogether 255 samples were analysed (positive results in brackets) for acetamiprid (0.023, 0.03 mg/kg), profenofos (0), phorate (0), and triazophos (0).

The Meeting estimated maximum and median residue levels 0.1 mg/kg for profenofos, phorate and triazophos.

DIETARY RISK ASSESSMENT*Long-term intake*

The contribution of residues present in the pepper, black white to the long-term-intake of acetamiprid and lambda-cyhalothrin was addressed in the evaluation of these compounds. No consumption data is available for cardamom, coriander, cumin and fennel seeds in the 17 GEMS/Food Cluster diets to estimate the contribution of the residues present in these spices to the long-term-intake of acetamiprid, cypermethrin, lambda-cyhalothrin, profenofos, phorate and triazophos.

Short-term intake

The International Estimated Short-Term Intake (IESTI) of acetamiprid and lambda-cyhalothrin from the consumption of pepper, black white and cardamom seed was addressed in the evaluation of these compounds.

The IESTIs for profenofos, phorate and triazophos from the consumption of the spices considered by the current Meeting were estimated. The results are shown in Annex 4 to the 2015 Report. The IESTI represented 0% of the ARfD of cypermethrin and profenofos, a maximum of 10% of the ARfD of phorate and a maximum of 7% of the ARfD of triazofos. The Meeting concluded that the short-term intake of cypermethrin, profenofos, phorate and triazophos residues from the uses considered by the current Meeting was unlikely to present a public health concern.