

PYRETHRINS (063)

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EXPLANATION

Pyrethrins were last evaluated for residues under the Periodic Review Program by the 2000 JMPR. The 2000 JMPR agreed that the residue definition for compliance with the MRL and for estimating dietary intake is total pyrethrins, calculated as the sum of the six biologically active pyrethrin esters: pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2 after calibration with the World Standard Pyrethrum Extract. Pyrethrins were last evaluated toxicologically by the 2003 JMPR. It confirmed the ADI of 0.04 mg/kg bw established by the 1972 JMPR and reaffirmed by the 1999 JMPR, and the ARfD of 0.2 mg/kg bw established by the 1999 JMPR.

The 2000 JMPR concluded that the existing CXL of 1 mg/kg Po for tree nuts should be withdrawn because no information had been submitted. The 34th Session of the CCPR decided to maintain the CXL for tree nuts for 4 years, as the Government of Australia had indicated its intention to submit new residue data to the JMPR.

The 2005 JMPR received reports of studies on analytical method and supervised residue trials and information on GAP for tree nuts from the Government of Australia (Bodnaruk, K. 2005, NT03007).

METHODS OF RESIDUE ANALYSIS

Analytical methods for tree nuts

A new method was reported to the Meeting for determining residues of pyrethrins in tree nuts in supervised residue trials.

Samples were extracted with acetone and water with the homogenate filtered using a separator funnel. Hexane was then added to the separator funnel followed by a sodium chloride solution. Liquid-liquid partition was repeated with the hexane layer collected. The combined hexane extract was then dried using anhydrous sodium sulfate into a rotary evaporation flask. Hexane was evaporated to dryness. The residue was then taken up in hexane and membrane-filtered prior to analysis. Determination was carried out by HPLC with a fluorescence detector at 223 nm. The limits of quantification (LOQ) as total pyrethrins (the sum of pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2) were 0.2 mg/kg for almond and 0.5 mg/kg for macadamia nuts. The results of validation experiments are shown in table 1.

Table 1. Recovery of pyrethrins from fortified nut samples.

| Crops | Fortification levels of pyrethrins, mg/kg | Average recovery, % | %RSD |
|---------------|-------------------------------------------|---------------------|------|
| Almond | 0.2 | 97.7 | 8.2 |
| | 0.8 | 97.4 | 5.8 |
| Macadamia nut | 1.0 | 93.4 | 6.0 |

Stability of residues in stored analytical samples

Almond and Macadamia nuts samples were stored frozen at -18°C until preparation and analysis. Data on the stability of residues in stored analytical samples were not provided. However, valid storage stability data on peanuts exists (from 2000 JMPR) with the results of this study considered applicable to tree nuts.

USE PATTERN

Post-harvest uses of pyrethrins have been approved for control of various insects in Australia. The use on stored tree nuts is not specified on product labels. However, the use is acknowledged by the regulator as being covered by the current product labels.

Table 2. Post-harvest uses of pyrethrins on tree nuts in Australia.

| Situation | Formulation (Concentration) | Application rate (g ai/100m ³ space) | Interval and number of applications |
|-----------------------------------|-----------------------------|----------------------------------------------------------|-------------------------------------|
| Food storage and processing areas | Gas (4 g a.i./kg) | 0.067 (for flying insects) 0.2 (for crawling insects) | Not mentioned |

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised residue trial data from post-harvest use on almonds and macadamia nuts. All trials were carried out using a gas formulation containing pyrethrins (4 g/L) and piperonyl butoxide (20 g/L). The formulation used was applied by fixed spray installation using programmed time release. The size of storage room used for almond and macadamia nuts were 4036 – 10335m³ and 162.5 – 10436m³ respectively. For both almonds and macadamia nuts, the nut kernels were placed in open condition (e.g. in open mesh bags) and exposed to pyrethrins. The nuts kernels were collected as samples. Samples were stored at -18°C until preparation and analysis. The interval between sampling and analysis was 2 – 12 months. The results of the trials on almonds (2005; Study No. COD0301a) and macadamia nuts (2005; Study No. COD0301b) are summarized in Tables 3 and 4.

Almond

In three trials on almonds in 2005 (Study No. COD0301a), residues in kernels were below the LOQ, 0.2 mg/kg (Table 3).

Table 3. Residues of pyrethrins in almond kernels after post-harvest use.

| Location, Year | Formulation | Application | | | Days after last application (days) | Residues (mg/kg) |
|---------------------------------|-----------------|----------------------|---------------------------------|---------------------------|------------------------------------|------------------|
| | | Dates of application | Rate (g /ai/100m ³) | Total No. of applications | | |
| Loxton,SA Australia, 2005 | Gas formulation | 13 April 2004 | 0.2 | 3 | 4 | < <u>0.2</u> |
| | | 14 April 2004 | 0.2 | | 5 | < 0.2 |
| | | 15 April 2004 | 0.2 | | 6 | < 0.2 |
| Renmark,SA Australia 2005 | Gas formulation | 22 May 2004 | 0.2 | 3 | 0 | < <u>0.2</u> |
| | | 26 May 2004 | 0.2 | | | |
| | | 28 May 2004 | 0.2 | | | |
| Loxton,SA Australia 2005 | Gas formulation | 13 May 2004 | 0.2 | 3 | 0 | < <u>0.2</u> |
| | | 14 May 2004 | 0.2 | | | |
| | | 15 May 2004 | 0.2 | | | |

Macadamia nut

In three trials on macadamia nuts in 2005 (Study No. COD0301b), pyrethrin residues in nut kernels were below the LOQ, 0.5 mg/kg (Table 4).

Table 4. Residues of pyrethrins in macadamia nut kernels after post-harvest use.

| Location, year | Formulation | Application | | | Days after last application (days) | Residues (mg/kg) |
|-----------------------------|-----------------|----------------------|---------------------------------|---------------------------|------------------------------------|------------------|
| | | Dates of application | Rate (g /ai/100m ³) | Total No. of applications | | |
| Lismore, NSW Australia 2005 | Gas formulation | 25 Aug. 2003 | 0.2 | 4 | 0 | < <u>0.5</u> |
| | | 01 Sep. 2003 | 0.2 | | 1 | < 0.5 |
| | | 07 Sep. 2003 | 0.2 | | 2 | < 0.5 |
| | | 14 Sep. 2003 | 0.2 | | 4 | < 0.5 |
| Gympie, QLD Australia 2005 | Gas formulation | 11 Jan. 2004 | 0.2 | 4 | 0 | < <u>0.5</u> |
| | | 18 Jan. 2004 | 0.2 | | | |
| | | 25 Jan. 2004 | 0.2 | | | |
| | | 01 Feb. 2004 | 0.2 | | | |
| Gympie, QLD Australia 2005 | Gas formulation | 21 Feb. 2004 | 0.2 | 4 | 0 | < <u>0.5</u> |
| | | 28 Feb. 2004 | 0.2 | | | |
| | | 06 Mar. 2004 | 0.2 | | | |
| | | 13 Mar. 2004 | 0.2 | | | |

APPRAISAL

Pyrethrins were evaluated for residues in the Periodic Review Programme of the 2000 JMPR, which concluded that the existing CXL of 1 mg/kg Po for tree nuts should be withdrawn because no information was submitted. The 34th Session of the CCPR decided to maintain the CXL for tree nuts for 4 years, as the Government of Australia had indicated its intention to submit new residue data to the JMPR.

The 2005 JMPR received reports of studies on analytical methods and supervised residue trials and information on GAP for tree nuts. Reports were supplied by the government of Australia. The formulation used for trials contains not only pyrethrins but piperonyl butoxide. There is no residue information for piperonyl butoxide and a CXL for piperonyl butoxide in tree nuts has not been established.

Method of analysis

The sample was extracted with acetone and water. Extracts were assessed by HPLC with a fluorescence detector at 223nm. The limit of quantification (LOQ) was 0.2 mg/kg for almond and 0.5 mg/kg for macadamia nuts. This method was considered acceptable for supervised trials.

Definition of the residue

The Meeting agreed that the residue definition for enforcement purposes for plant commodities and for consideration of dietary intake should be total pyrethrins, calculated as the sum of pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2, determined after calibration with World Standard pyrethrum extract. The Meeting agreed that the residues are fat-soluble.

Definition of the residue for compliance with MRLs and estimation of dietary intake: total pyrethrins, calculated as the sum of pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2, determined after calibration with World Standard pyrethrum extract.

Results of supervised trials on crops

Tree nuts

The current Australian label indicates that a gas formulation of pyrethrins may be applied to stored tree nuts in food storage area. The application rate of pyrethrins for tree nuts is either 0.067 g ai/100m³ (for flying insects) or 0.2 g ai/100m³ (for crawling insects). However the frequency of applications is not specified on product labels but determined by the situation in which the product is being applied. Three supervised trials each were conducted for stored almond and macadamia nuts according to the

maximum application rate based on the Australia GAP (0.2 g ai/100m³). The number of applications to almond or macadamia nuts was 3 or 4 respectively. The residues of pyrethrins were below the respective LOQ's, < 0.2 mg/kg in almond and < 0.5 mg/kg in macadamia nuts.

The Meeting estimated a maximum residue level of 0.5 * mg/kg, an STMR of 0.2 mg/kg and an HR of 0.5 mg/kg for post-harvest use of pyrethrins on tree nuts and recommended withdrawal of the existing CXL for tree nuts of 1 mg/kg Po.

RECOMMENDATIONS

The Meeting estimated the maximum residue level, STMR value and HR value shown in table 1. The maximum residue level is recommended for use as an MRL.

Definition of the residue for compliance with MRLs and estimation of dietary intake: *total pyrethrins, calculated as the sum of pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2, determined after calibration with World Standard pyrethrum extract.*

The residue is fat-soluble.

Table 5. Summary of recommendations.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|-----------|-----------|------------|----------|-------------|-----------|
| CCN | Name | New | Previous | | |
| TN 0085 | Tree nuts | 0.5* Po | 1 Po | 0.2 | 0.5 |

* at or about the limit of quantification

Po accommodates post-harvest treatment of the commodity

FURTHER WORK OR INFORMATION

Desirable

1. Feeding studies with ruminants (from 2000 JMPR).
2. Feeding studies with laying hens (from 2003 JMPR).

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of pyrethrins, based on the STMRs estimated by the 2000, 2003 and 2005 JMPR for 12 commodities, for the five GEMS/Food regional diets was 1% of the maximum ADI of 0.04 mg/kg bw. The Meeting concluded that the long-term intake of residues of pyrethrins, resulting from the uses considered by the JMPR, is unlikely to present a public health concern. The results are shown in Annex 3 of the 2005 JMPR Report.

Short-term intake

The International Estimated Short Term Intake (IESTI) for pyrethrins was calculated for tree nuts for which the maximum residue level was estimated by the current JMPR. The results are shown in Annex 4 of the 2005 JMPR Report.

The IESTI represented 1% of the ARfD (0.2 mg/kg bw) for the general population and 0% of the ARfD for children. The Meeting concluded that the short-term intake of residues of pyrethrins, resulting from the uses considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

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