

5.12 DIPHENYLAMINE (030)

RESIDUE AND ANALYTICAL ASPECTS

The CCPR at its 36th Session advanced the MRL for cattle milk, 0.0004 (*) F mg/kg, to Step 8. Subsequently the 38th Session requested JMPR to review the basis for setting the cattle milk MRL. The issue was referred to the JMPR Secretariat with a direction to separate the MRLs for milk and cream.

The CCPR at its 40th Session decided that where separate MRLs are established for whole milk and milk fat, regulation and monitoring of fat-soluble pesticides in milk should be in reference to whole milk. That is, whole milk should be analysed and the result compared to the MRL of whole milk.

Relevant studies on analytical method, livestock metabolism, and livestock feeding were supplied by the Northwest Horticultural Council (USA). All were studies considered during the periodic re-evaluation of diphenylamine (2001 Report). No new data were made available.

The residue definition is diphenylamine. The residue is fat soluble (JMPR Report 2001).

In the Holstein cow feeding study, diphenylamine was detectable at the 0.005 mg/kg LOD only occasionally at the 30 and 90 ppm dosing levels. At the 300 ppm dosing level, the diphenylamine was equal to or above the level of detection in all three cows (day 24): 0.005, 0.012, and 0.008 mg/kg, but below the demonstrated level of quantitation (LOQ), 0.01 mg/kg. The dietary burdens for the estimated MRL and STMR values are the same, 11.5 ppm (as determined in 2001).

Cream samples were also analysed from day 14, but the method of separation (mechanical, chemical) of the cream (milk fat) is not described. The maximum residue level at the 30 ppm feeding level was 0.011 mg/kg and the average was 0.0098 mg/kg, range 0.008 to 0.011 mg/kg for three cows at two milking sessions on day 14. The separation technique is questionable given that one sample of skim milk contained 0.011 mg/kg diphenylamine (whole milk < 0.005 mg/kg). This is an unexpected result for a fat-soluble pesticide, or at least indicates factors other than fat solubility entering into the distribution of the pesticide. The maximum residue level estimate for milk fat would be 0.01* mg/kg, based on the limit of quantitation of the analytical method, which would be the recommended maximum residue level estimate of 0.0004* mg/kg for whole milk at 4% fat (0.04 × 0.01).

Based on the dietary burden of 11.2 ppm for dairy cattle (JMPR 2001) and average residues at a 30 ppm feeding level < 0.005 mg/kg (0.005 mg/kg maximum), the current Meeting estimated a maximum residue level of 0.01 (*) mg/kg and an STMR of 0.0019 mg/kg (0.005 mg/kg × 11.2 / 30) for whole milk (ML 106). This replaces a previous maximum residue recommendation for 0.0004 (*) mg/kg for cattle milk. The Meeting further recommended a maximum residue level of 0.01 mg/kg for milk fats (0.011 mg/kg × 11.5 / 30 × 2 = 0.0084, detectable but < LOQ) and an STMR of 0.0075 mg/kg (0.0098 × 11.5 / 30 × 2 = 0.0075). This assumes that cream is 50% milk fat.