



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA MX030: Pesticides in Tomato Puree

Certified values

Pesticide	CAS No.	Mass fraction (mg/kg)
Bifenthrin	82657-04-3	0.289 ± 0.021
Buprofezin	69327-76-0	0.182 ± 0.017
Chlorpyrifos	2921-88-2	0.781 ± 0.035
Endosulfan sulfate	1031-07-8	0.615 ± 0.052
Imidacloprid	138261-41-3	0.351 ± 0.029
Pirimicarb	23103-98-2	0.670 ± 0.052

The reported uncertainty is expanded to provide a level of confidence of 95% using coverage factors of 2.0.

Expiry: 30 April 2026

Batch No.: 2022.01

When stored in accordance with the instructions given on this certificate.

Description: This reference material consists of approximately 100 g of pureed tomato fortified with a number of pesticides and is contained in an amber screw capped bottle.

Intended use: The reference material is intended to be used to validate analytical methods for the measurement of pesticides in fruit and vegetable matrices. It may also be used to calibrate secondary reference materials of similar composition or as a matrix calibration standard.

Instructions for use: The material should be thawed at room temperature and thoroughly mixed by vigorous shaking for at least 30 s immediately before use. Do not allow to settle while sub-sampling. The minimum sample size for which homogeneity has been determined is 5 g.

The material can be refrozen and resampled, however the time the material spends unfrozen should be minimised, as it has not been sterilised and does not contain preservatives.

Storage: The material should be stored at or below -18 °C in the closed container as issued.

Metrological traceability: The certified values for mass fraction are traceable to the SI unit for mass (kg). Gravimetric preparation using calibrated analytical balances is traceable to the SI kg through the Australian national standard for mass. Mass fractions were determined by isotope dilution mass spectrometry and are traceable to SI units through the pure substance reference materials used for calibration, certified by NMIA (bifenthrin, chlorpyrifos and endosulfan sulfate) or by the National Institute for Measurement, China (buprofezin, imidacloprid and pirimicarb).

Stability: The stability of the material when stored below -18 °C for up to 20 months, when stored at room temperature or 4 °C for 2 weeks, and when subjected to repeated freeze-thaw cycles was demonstrated by isochronous stability trials conducted in accordance with ISO Guide 35 [1]. The uncertainties in the mass fractions were expanded to incorporate transport stability at room temperature for 14 days and storage stability for the period until the expiration date of this certificate. Stability will continue to be monitored.

Homogeneity: Assessment of homogeneity of the material was conducted in accordance with ISO Guide 35 [1]. Duplicate 5 g sub-samples from 10 randomly selected bottles of the CRM were analysed and the results used to calculate the within-bottle and between-bottle variances. The uncertainties in the certified values incorporate these variances.

Safety: CRM NMIA MX030 is intended for in-vitro diagnostic analysis only.

Production: The material was prepared in June 2022. Fresh tomatoes were pureed and sieved (850 microns), and fortified with bifenthrin, buprofezin, chlorpyrifos, endosulfan sulfate, imazalil, imidacloprid and pirimicarb. The puree was dispensed into bottles in 100 g aliquots and frozen. Imazalil was added to the sample at approximately 0.7 mg/kg on preparation. After 20 months storage the mass fraction had decreased to 0.5 mg/kg and for this reason imazalil mass fraction has not been certified.

Analytical method: The pesticide mass fractions were measured by isotope dilution [2] with two-dimensional liquid chromatography tandem mass spectrometry (2DLC-MSMS). Bifenthrin, chlorpyrifos, endosulfan sulfate and pirimicarb were also measured by gas chromatography (GC)-MSMS. Isotopically labelled analogues of the pesticides were added to the tomato prior to extraction with acidified acetonitrile and dispersive solid-phase extraction (dSPE) using QuEChERS methodology [3]. Extracts were diluted for 2DLC-MSMS, or subjected to preparative HPLC prior to GC-MSMS.

Measurement uncertainty: Standard uncertainties were estimated and combined as described in the JCGM Guide to the Expression of Uncertainty in Measurement [4]. The individual components contributing to the measurement uncertainty estimates were the mass fractions assigned to calibration standards, gravimetric mass measurements, precision of the analytical method, batch homogeneity, long-term storage stability of the material at -20 °C, stability of the material during transportation and potential sources of bias in the reference analytical procedure. The combined standard uncertainties were expanded to a level of confidence of 95% using a coverage factor calculated from the effective degrees of freedom obtained from the Welch-Satterthwaite equation.



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Manager, Chemical Reference Values
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References:

1. Reference Materials – Guidance for characterisation and assessment of homogeneity and stability. ISO Guide 35:2017
2. L.G. Mackay, C.P. Taylor, R.B. Myors, R. Hearn and B. King; *Accred. Qual. Assur.*, **8**, 2003, 191-194
3. M. Anastassiades, S. Lehotay, D. Stajnbaher and F. J. Schenk; *J. AOAC Int.*, **86** (2), 2003, 412-431
4. Joint Committee for Guides in Metrology; Evaluation of measurement data — Guide to the Expression of Uncertainty in Measurement; JCGM 100:2008

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

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