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CERTIFICATE OF ANALYSIS

REFERENCE MATERIAL No. IPO 005

ATRAZINE

6-chloro-N²-ethyl-N⁴-isopropyl-1,3,5-triazine-2,4-diamine

C₈H₁₄ClN₅ (215.68)

CAS REG. No. [1912-24-9]

Series No. 6B/17

Valid to: December 2023

Purity: 98.8 ± 0.1 % (m/m)

Unit: 0.25 g of crystalline solid in a brown glass vial.

Storage: The material should be stored in the original closed vial in a refrigerator at 5 ± 4 °C until it is required to use. Allow to equilibrate to ambient temperature before opening. Protect from moisture. It is intended for use as a reference material for the calibration of measuring equipment, for the evaluation of analytical procedures and for the study of biological activity.

CONFIRMATION OF THE IDENTITY

The identity of the product was established by infrared spectroscopy and mass spectrometry. The IR spectrum (KBr disc technique, scanning from 4000 to 400 cm⁻¹) of sample was compared with atrazine literature spectrum¹. Significant differences were not observed. The mass spectrum of sample was also recorded (EI, 70 eV, temperature of ion source 250 °C) and no significant differences were observed in comparison with the literature spectrum^{1,2,3}.

DETERMINATION OF THE PURITY

Representative samples were drawn from the bulk material. The purity value was based on determinations made on these representative samples using the following techniques:

- gas chromatography (GC)
- high performance liquid chromatography (HPLC)
- determination of sulphated ash (gravimetric)
- determination of water content (KF)

The uncertainties quoted below are the half-width of a 95 % confidence interval based on the standard deviation of the results obtained. The certified uncertainty is the combined uncertainty calculated according to the methodology described⁴ with a coverage factor $k = 2$. It corresponds to a confidence level of 95 %.

Determination by GC

Column I: fused-silica ZB-1 (30 m x 0.53 mm i.d.), film thickness 3.0 µm.

Temperature conditions:

- Column: 40 °C (5 min) to 180 °C (10 °C/min), 180 °C (20 min) to 240- °C (5 °C/min)
- Injector: 220 °C
- Detector (flame ionisation): 270 °C

Carrier gas: 5.2 mL/min.

Injection volume: 0.5 µL of sample solutions in methanol (0.1-0.4 % *m/V*).

The purity was calculated by peak area normalisation method. Two impurities were detected with a total concentration of 1.12 ± 0.05 % (*m/m*) (n = 5).

Column II: fused-silica DB-17 (30 m x 0.53 mm i.d.), film thickness 1.5 µm.

Temperature conditions:

- Column: 50 °C (3 min) to 190 °C (5 °C/min), 190 °C (15 min) to 240 °C (5 °C/min).
- Injector: 230 °C
- Detector (flame ionisation): 270 °C

Carrier gas: 6.3 mL/min.

Injection volume: 0.2 µL of sample solutions in methanol (0.3-0.6 % *m/V*).

The purity was calculated by peak area normalisation method. Two impurities were detected with a total concentration of 1.07 ± 0.03 % (*m/m*) (n = 4).

Determination by HPLC

- Column: Luna C₁₈, 5 µm, 250 x 4.6 mm i.d.,
- Mobile phase: methanol + water (50 + 50, V/V)
- Flow rate: 1.4 mL/min
- UV detection at 206 and 220 nm
- Injection volume: 10 µL of sample solutions in methanol (0.02-0.05 % *m/V*).

The purity was determined assuming equal detector response factors for all constituents.

One impurity was detected with a total concentration of 1.09 ± 0.04 % (*m/m*) (n = 8).

Determination of sulphated ash

Result: not more than 0.02 % (*m/m*) (n = 2).

Determination of water content

By the Karl Fischer technique. Result: 0.04 % (*m/m*) (n = 3).

CONCLUSIONS

On the basis of the results of two independent quantitative techniques (GC and HPLC) and other determinations, the purity of this batch of atrazine was assessed as 98.8 ± 0.1 % (*m/m*).

The analytical measurements were coordinated by:



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2. K. Pflieger, H. H. Maurer, A. Weber, Mass Spectral and GC Data of Drugs, Poisons, Pesticides, Pollutants and Their Metabolites, VCH, Weinheim, 2nd Ed., part 2, p. 957 (1992).
3. NIST/EPA/NIH Mass Spectral Library (1995).
4. NIST Special Publication 1012. An approach to the metrologically sound traceable assessment of the chemicals purity of organic reference materials David L. Duever and others, US Department of Commerce USA, September 2004, p.22