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CERTIFICATE OF ANALYSIS

REFERENCE MATERIAL No. IPO 119

p,p'-DDE

1,1-dichloro-2,2-bis(4-chlorophenyl)ethylene

$C_{14}H_8Cl_4$ (318.0) CAS REG. No. [*72-55-9*]

Series No. 6A/11 Valid to: December 2019

Purity: 99.8 ± 0.2% (*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 °C ± 4°C until it is required to use. Allow to equilibrate to ambient temperature before opening. 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 p,p'-DDE literature spectrum [1,2]. 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 [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 methods:

- differential scanning calorimetry (DSC)
- gas chromatography (GC)
- high performance liquid chromatography (HPLC)
- thin layer chromatography (TLC)
- determination of sulphated ash
- determination of water content

The uncertainties quoted below are the half-width of a 95% confidence interval based on the standard deviation of the results obtained.

Determination by DSC

Range of temperature: 68°C to 98°C, heating rate: 2°C/min. The results indicated a purity value of 99.84 ± 0.19 mole% (n=14).

Determination by GC

Column I: fused-silica DB-1 (30 m x 0.53 mm i.d.), film thickness 3.0 µm.
Column II: fused-silica DB-17 (30 m x 0.53 mm i.d.), film thickness 1.5 µm.

Temperature conditions:

- Column: 60°C (5 min) to 210°C (5°/min), 210°C (15 min) to 240°C (3°/min)
- Injector: 230°C
- Detector (flame ionisation): 270°C

Carrier gas: 5 ml/min.
Injection volume: 0.2 µl of sample solutions in acetone (0.8-1.6% *m/V*).
The purity was calculated by peak area normalisation of the chromatograms.
Result I: Three impurities were detected with a total concentration of less than 0.05% (*m/m*) (n = 5).
Result II: Two impurities were detected with a total concentration of less than 0.05% (*m/m*) (n = 5).

Determination by HPLC

- Column: Nucleosil 100-5 C_{18}, 5 µm, 250 x 4 mm i.d.
- Mobile phase: methanol + water (5:1, *V/V*)
- Flow rate: 0.5 ml/min
- UV detection at 206 nm.
- Injection volume: 20 µl of sample solutions in methanol (1.0% *m/V*).

The purity was determined assuming equal detector response factors for all constituents. No impurities were detected with a concentration of more than 0.03% (*m/m)* (n=3).

Estimation by TLC

- Adsorbent: silica gel 60 GF_{254}
- Mobile phase:
  - I n-hexane
  - II n-hexane + ethyl acetate (9:1, *V/V*)
- Visualisation: UV at 254 nm, silver nitrate + UV irradiation
- Sample loading: 100; 1000 µg of sample in n-hexane.

Results: No additional spots were observed in any solvent system.

Determination of sulphated ash

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

Determination of water content

By the Karl Fischer method. Result: 0.03% (*m/m*) (n=2).

CONCLUSIONS

On the basis of the results of three independent quantitative methods (DSC, GC and HPLC) and other determinations, the purity of this batch of p,p'-DDE was assessed as 99.8 ± 0.2% (*m/m*).

The analytical measurements were coordinated by:

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1. G. Zweig (Ed.), Analytical Methods for Pesticides and Plant Growth Regulators, Vol. IX, p. 204, Academic Press, Inc. (1977).
2. MS, NMR, IR, UV Atlas, Spectra Collection of Pesticides, Drugs and Pollutants, Spectral Service GmbH, Köln, Riedel-de Haën AG, Seelze, Germany, Vol. P2 (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 Departament of Commerce USA, September 2004 , p.22