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CERTIFICATE OF ANALYSIS

REFERENCE MATERIAL No. IPO 116

2,4-D

(2,4-dichlorophenoxy)acetic acid

$C_8H_6Cl_2O_3$ (221.0) CAS REG. No. [*94-75-7*]

Series No. 1E/07 Valid to: December 2017

Purity: 99.7 ± 0.3% (*m/m*)

Unit: 0.25 g of crystalline solid in a brown glass vial.
Storage: at approx. + 4°C. 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 2,4-D literature spectrum [1,2]. Significant differences were not observed.
The mass spectrum of sample was also recorded (EI, 70 eV, temperature of ion source 220^{O}C) and no 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 methods:

- differential scanning calorimetry (DSC)
- 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. The certified uncertainty is the combined uncertainty calculated according to the methodology described in[4] with a coverage factor k = 2. It corresponds to a confidence level of 95 %.
The thin layer chromatography (TLC) method was used as used as semiquantitative estimation of purity.

Determination by DSC

Range of temperature: 120°C to 150°C, heating rate: 2°C/min. The results indicated a purity value of 99.66 ± 0.18 mole% (n = 4).

Determination by HPLC

- Column: Luna C_{18}, 3 µm, 150 x 4.6 mm i.d.
- Mobile phase: acetonitrile + water (55:45, *V/V*) + 0.04% (*V/V*) phosphoric acid
- Flow rate: 1.0 ml/min
- UV detection at 206 nm.
- Injection volume: 20 µl of sample solutions in acetonitrile (0.01-0.05% *m/V*).

The purity was determined assuming equal detector response factors for all constituents.
Five impurities were detected with a total concentration of 0.05 ± 0.01 % (*m/m*) (n = 6).

Estimation by TLC

- Adsorbent: silica gel 60 GF_{254}
- Mobile phase:
  - I cyclohexane + diisopropyl ether + acetic acid (50:20:3.5, *V/V/V*)
  - II chloroform
- Visualisation: UV at 254 nm; silver nitrate + UV irradiation
- Sample loading: 100; 1000 µg of sample in acetone.

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*).

CONCLUSIONS

On the basis of the results of two independent quantitative methods (DSC and HPLC) and other determinations, the purity of this batch of 2,4-D was assessed as 99.7 ± 0.3 % (*m/m*).

The analytical measurements were coordinated by:

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1. Spectral Database for organic compounds SDBS http://www.aist.go.jp/RIODB/SDBS no 5920
2. MS, NMR, IR, UV Atlas, Spectra Collection of Pesticides, Drugs and Pollutants, Spectral Service GmbH, Kö ln, Riedel-de Haën AG, Seelze, Germany, Vol. P2 (1992) # 35715
3. NIST/EPA/NIH Mass Spectral Library (1995) #10371
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