



REFERENCE MATERIAL ANALYSIS REPORT

Report ID: P1373b.2017.01 (Bottled 151130)

This batch of bottles was prepared from the bulk material on 30th November 2015.

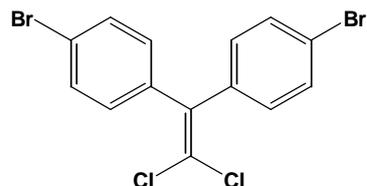
Compound Name: **Dibromo-DDE**

Collection Number: P1373b

Chemical Formula: C₁₄H₈Br₂Cl₂

CAS Number: 21655-73-2

Structure:



Description: Colourless, needle-like crystals

Batch Number: 04-AV-03

Molecular Weight: 406.9

Release date: November 2004

Synonyms: 2,2-Bis (*p*-bromophenyl)-1,1-dichloroethene
p, p'-DDE dibromide
p, p'-DDBrE

Purity (mass fraction): 99.9 ± 0.4% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques by subtraction from 100% of total impurities by GC-FID, Karl Fischer analysis and ¹H NMR spectroscopy. Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890N
Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 170 °C (1 min), 10 °C/min to 300 °C (3 min)
Injector: 250 °C Detector Temp: 320 °C
Carrier: Helium Split ratio: 20/1
Relative peak area response of main component:
Initial analysis: Mean = 99.95%, s = 0.01% (7 sub samples in duplicate, September 2004)
Re-analysis: Mean = 99.91%, s = 0.03% (5 sub samples in duplicate, February 2007)
Re-analysis: Mean = 99.90%, s = 0.004% (5 sub samples in duplicate, May 2009)
Re-analysis: Mean = 99.91%, s = 0.002% (5 sub samples in duplicate, April 2012)
Re-analysis: Mean = 99.93%, s = 0.004% (5 sub samples in duplicate, March 2017)

Karl Fischer analysis: Moisture content < 0.1% mass fraction (July 2006)
Moisture content 0.4% mass fraction (May 2009)
Moisture content < 0.1% mass fraction (April 2012 and March 2017)

Thermogravimetric analysis: Volatile content not determined due to volatility of the material

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Column:	ZB-5, 30 m x 0.25 mm x 0.25 μ m
	Program:	180 °C (1 min), 15 °C/min to 280 °C, then 20 °C/min to 300 °C
	Injector:	250°C Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min Split ratio: 20/1
	The retention time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass to charge ratios and (in brackets) as a percentage relative to the base peak.	
	8.76 min: 408 (M^+ , 89), 406 (100), 338 (11), 336 (22), 334 (11), 292 (35), 290 (27), 246 (38), 176 (55) m/z	
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Elution with hexane Single spot observed, $R_f = 0.40$
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm ⁻¹ , KBr pellet
	Peaks:	3083, 3065, 1903, 1584, 1485, 1392, 1071, 1011, 858, 824, 790 cm ⁻¹
¹ H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz Solvent: CDCl ₃ (7.26 ppm)
	Spectral data:	δ 7.15 (1H, d, $J = 8.5$ Hz), 7.48 (1H, d, $J = 8.5$ Hz) ppm 0.2% Isopropanol and traces of methanol observed in ¹ HNMR (June 2009)
¹³ C NMR:	Instrument:	Bruker DMX600
	Field strength:	150 MHz Solvent: CDCl ₃ (77.16 ppm)
	Spectral data:	δ 120.5, 122.5, 131.0, 131.6, 137.8, 138.3 ppm
Melting point:	121-122 °C	
Microanalysis:	Found: C = 41.5 %, H = 2.1 % (October 2004) Calc: C = 41.3 %, H = 2.0 % (Calculated for C ₁₄ H ₈ Br ₂ Cl ₂)	

Expiration of certification

The property values are valid till 29th March 2022, i.e. five years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body.

The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

Homogeneity assessment

The homogeneity of the material was assessed using purity assay by gas chromatography with flame ionization detection on seven randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Recommended storage

When not in use this material should be stored at or below 25 °C in a closed container in a dry, dark area.

Intended use

For *in vitro* laboratory analysis only.

Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 4 April 2017

Characterisation data and property values specified in this report supersede those in all reports issued prior to 4th April 2017.