

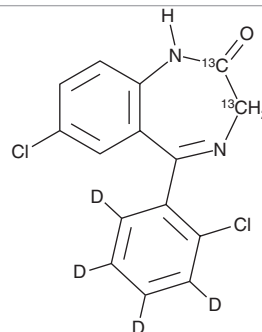
CERTIFICATE of ANALYSIS



ACCREDITED
ISO 17034 #AR-1774

Delorazepam- $^{13}\text{C}_2\text{-d}_4$ (CRM)

Certified Reference Material



Item No.:	42199
Batch No.:	0722632
Molecular Formula:	$\text{C}_{13}[^{13}\text{C}]_2\text{H}_6\text{Cl}_2\text{D}_4\text{N}_2\text{O}$
Formula Weight:	311.20 amu
Expiry Date:	09OCT2029 (valid from date of certification)
Supplied as:	A 100 µg/ml (nominal) solution in acetonitrile
Volume per Ampule:	Not less than 1 ml. Ampules are overfilled.
Storage:	Unopened at $-20^\circ\text{C} \pm 10^\circ\text{C}$
Safety:	Refer to Safety Data Sheet
Intended Use:	For analytical testing purposes only, not intended for human or animal use.
Instructions for Use:	This product is designated for one-time use and should be used immediately after opening. It is advised that laboratories warm the vial to room temperature prior to opening and use measured volumes.

Certified Concentration · 100.0 µg/ml $\pm$ 0.7 µg/ml

Concentration is calculated based on product mass, solution mass, corrected purity, and density at 20°C. It is traceable to SI units through an unbroken chain of measurements. Uncertainty of concentration is expressed as an expanded uncertainty in accordance with ISO standards for Testing Laboratories and Reference Material Producers at the approximate 95% confidence interval using a coverage factor of $k=2$ and incorporates uncertainties from the corrected purity, solution preparation, homogeneity, and long- and short-term stability. Concentration was verified by comparison to an independently prepared calibration standard.

Corrected Purity · 99.47%

Corrected purity is determined as follows: Corrected Purity = $[(100 - \% \text{ LOD} - \% \text{ ROI}) \times \text{Chromatographic Purity} / 100]$ or $[(100 - \% \text{ KF} - \% \text{ RS} - \% \text{ ROI}) \times \text{Chromatographic Purity} / 100]$. Where applicable, optical rotation, chiral purity, and/or isotopic purity testing are performed to support the identification of the reference material, therefore the uncertainty is considered null.

Approval: 
Roxanne Franckowski

Title: Senior Manager of ISO Quality

Certification Date: 09OCT2024

Cayman Chemical certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration date when stored unopened as recommended.



CERTIFICATE of ANALYSIS



CRM Assay

Method Parameters											
Cayman Method	F0579										
Column	3.0 x 150 mm, 2.7 µm Ascentis C18										
Mobile Phase	A: 20 mM Ammonium Formate and 0.1% Formic Acid in Water B: 0.1% Formic Acid in Acetonitrile										
Gradient	<table><tr><td>Time (min)</td><td>%B</td></tr><tr><td>0-2</td><td>20%</td></tr><tr><td>2-12</td><td>20-45%</td></tr><tr><td>12-16</td><td>45%</td></tr><tr><td>16.1-20</td><td>20%</td></tr></table>	Time (min)	%B	0-2	20%	2-12	20-45%	12-16	45%	16.1-20	20%
Time (min)	%B										
0-2	20%										
2-12	20-45%										
12-16	45%										
16.1-20	20%										
Flow Rate	0.7 ml/min										
Column Temp	40°C										
Wavelength	UV monitored at 254 nm										

Homogeneity

A minimum sample size of 0.1 µg was used to determine homogeneity. Homogeneity was determined by HPLC using ampoules selected from a random sampling plan from early, middle, and late fill positions.

%RSD	Acceptance Criteria
0.75%	≤3%

The recommended minimum quantity for use is 0.1 µg. Quantities below this have not been evaluated.

Neat Material Quality Information (Item No.: 40992, Batch No.: 0714269)

Qualifier	Method	Result
Chromatographic Purity, HPLC	Cayman Method F0579	>99.90%
Identity, LC-MS	Cayman Method TST SD13, +ESI	311.0 amu
Identity, GC-MS	Cayman Method TST SD12	Conforms
% LOD	Cayman Method TST SD24	0.23%
% ROI	Cayman Method TST SD06	0.20%
Isotopic Purity, LC-MS	Cayman Method TST SD154	<0.10% M ₀ vs M ₆
Identity, NMR	¹ H NMR	Conforms

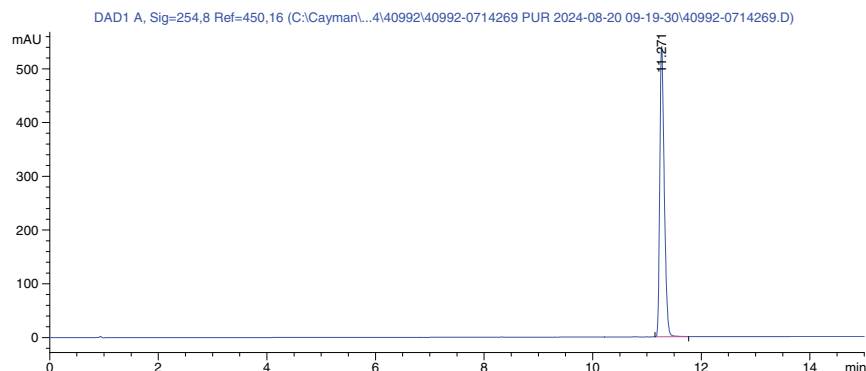
NMR and optical rotation (if applicable) are provided as supplemental information but are not within scope of ISO accreditation. Property values are traceable to SI units through an unbroken chain of measurements.

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Supplemental Data (Neat Material)

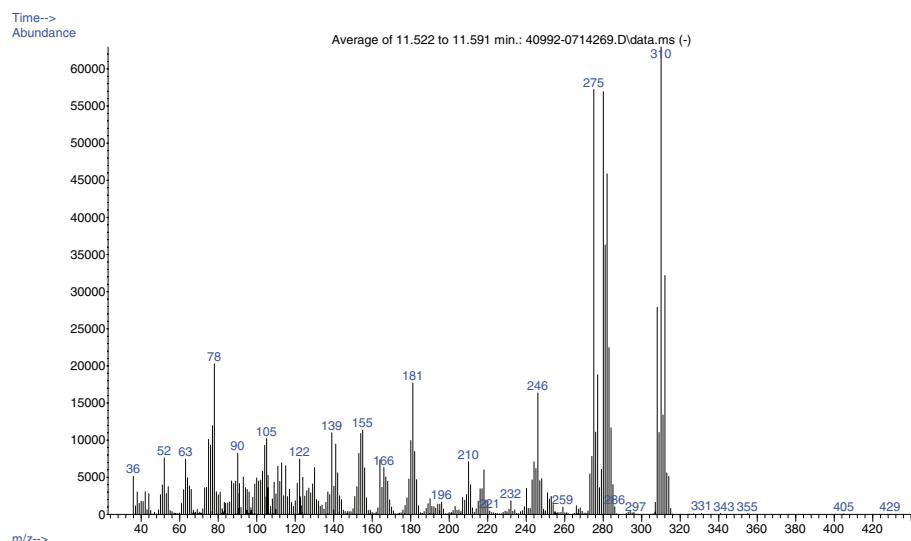
HPLC-UV



Conditions

Instrument	Agilent 1100/1200 Series										
Column	3.0 x 150 mm, 2.7 µm Ascentis C18										
Mobile Phase	A: 20 mM Ammonium Formate and 0.1% Formic Acid in Water B: 0.1% Formic Acid in Acetonitrile										
Gradient	<table border="1"> <thead> <tr> <th>Time (min)</th> <th>%B</th> </tr> </thead> <tbody> <tr> <td>0-2</td> <td>20%</td> </tr> <tr> <td>2-12</td> <td>20-45%</td> </tr> <tr> <td>12-16</td> <td>45%</td> </tr> <tr> <td>16.1-20</td> <td>20%</td> </tr> </tbody> </table>	Time (min)	%B	0-2	20%	2-12	20-45%	12-16	45%	16.1-20	20%
Time (min)	%B										
0-2	20%										
2-12	20-45%										
12-16	45%										
16.1-20	20%										
Flow Rate	0.7 ml/min										
Column Temp	40°C										
Wavelength	UV monitored at 254 nm										

GC-MS



Conditions

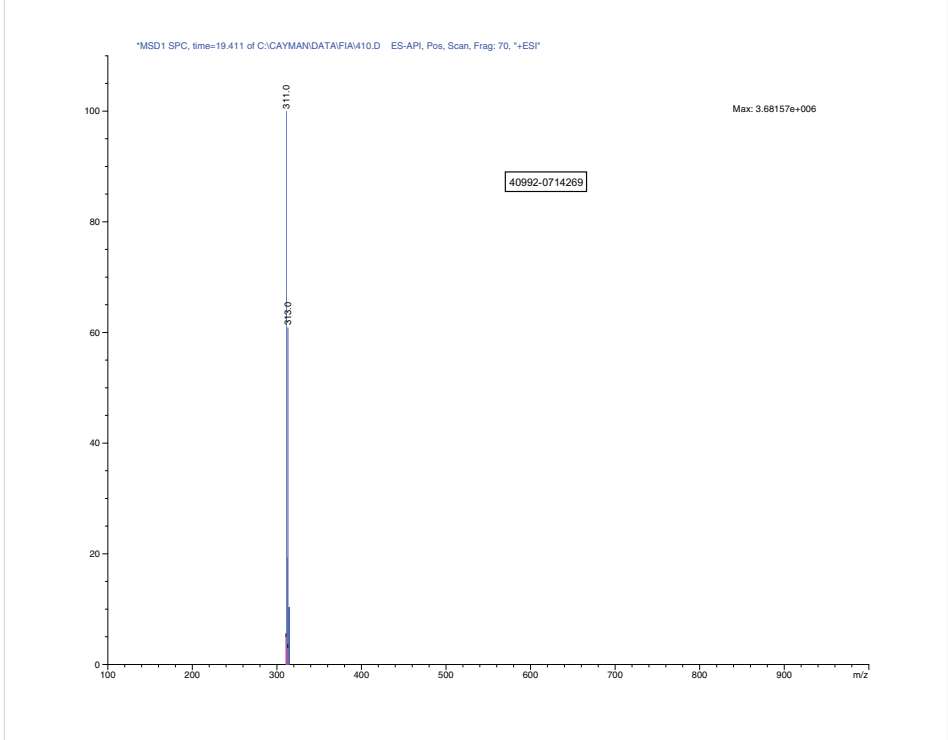
Instrument	Agilent GC MSD
Column	30 m x 0.32 mm, 0.5 µm Rtx-5MS
Carrier Gas	He
Flow Rate	2 ml/min
Inlet Temp	300°C
Split Ratio	15:1
Oven Program	50°C hold for 1 min, ramp to 300°C at 30°C per min, hold at 300°C to 25 minutes
Transfer Line Temp	300°C
Voltage	70eV EI MS
Scan Range	35-500 m/z
Tune File	stune

Apex spectrum – background (1 min window in front of peak)

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ESI-MS



Conditions

Instrument	Agilent HPLC MSD
Mobile Phase	50:50:0.1 Methanol/Water/Acetic Acid
Flow Rate	0.5 ml/min
Ionization Mode	+ESI
Mass Range	100-1,000 m/z
Nebulizer	60 psi
Desolvation Gas	13 L/min
Desolvation Temp	350°C
Electrospray Voltage	4kV

MS collected across peak width at half height

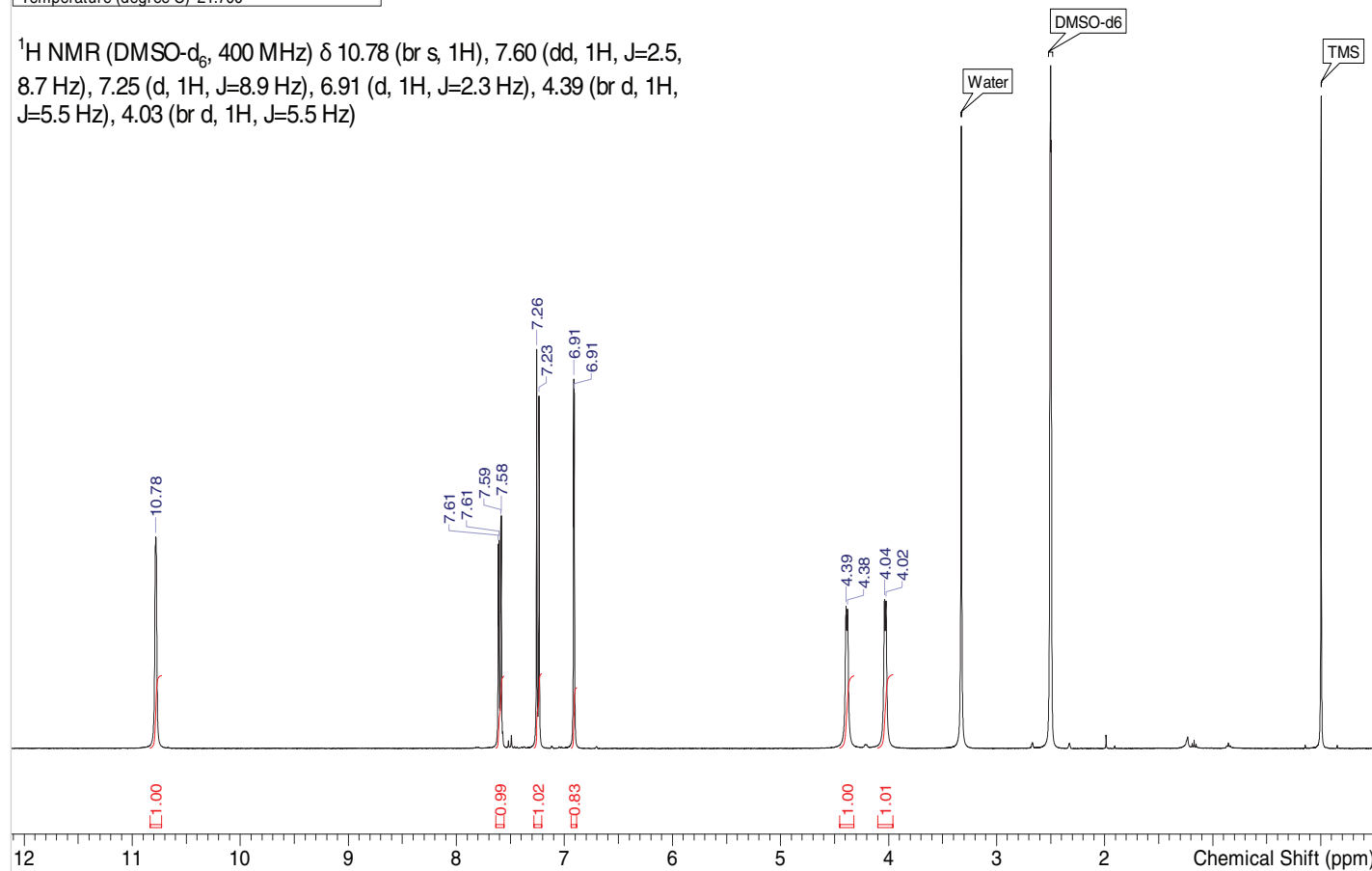
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NMR (not within scope of ISO accreditation)

File Name	\\sulfur\private\nmrdata\JEOL_2024\40992-0714269\40992-0714269_PROTON_25-Jul-2024-1-1.jdf		
Date	25 Jul 2024 21:16:13	Nucleus	¹ H
Solvent	DMSO-d ₆	Frequency (MHz)	399.5822
Temperature (degree C)	21.700	Number of Transients	16
		Origin	JEOL ECZ400S Sc v601

¹H NMR (DMSO-d₆, 400 MHz) δ 10.78 (br s, 1H), 7.60 (dd, 1H, J=2.5, 8.7 Hz), 7.25 (d, 1H, J=8.9 Hz), 6.91 (d, 1H, J=2.3 Hz), 4.39 (br d, 1H, J=5.5 Hz), 4.03 (br d, 1H, J=5.5 Hz)



Conditions

Instrument	JEOL ECZ 400S
Scans	16 scans

Stability

The effect of the components of stability on the combined standard uncertainty of the CRM property value are considered negligible unless indicated in stability studies.

Short-Term Stability

No decrease in the property value was observed at ambient or 60°C after two weeks. This data supports shipping of this product at ambient temperature.

Long-Term Stability

Long-term stability data confirmed five years stability at the -20°C storage temperature.

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Quality Standard Documentation

The manufacturer of this Certified Reference Material is accredited under ISO 17034:2016 accreditation issued by ANAB. Refer to ANAB certificate and scope of accreditation AR-1774.

The manufacturer of this Certified Reference Material is accredited under ISO/IEC 17025:2017 accreditation issued by ANAB. Refer to the ANAB certificate and scope of accreditation AT-1773.

Revision History

Revision No.	Date	Reason for Revision
01	09OCT2024	Initial version

Disclaimers

Material Safety Data

This material should be considered hazardous until information to the contrary becomes available. Do not ingest, swallow, or inhale. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. This information contains some but not all of the information required for the safe and proper use of this material. Before use, review the complete Safety Data Sheet, which has been sent *via* email to your institution.

Warranty and Limitation of Remedy

Cayman Chemical Company makes no warranty or guarantee of any kind, whether written or oral, expressed or implied, including without limitation, any warranty of fitness for a particular purpose, suitability and merchantability, which extends beyond the description of the chemicals hereof. Cayman warrants only to the original customer that the material will meet our specifications at the time of delivery.

Cayman will carry out its delivery obligations with due care and skill. Thus, in no event will Cayman have any obligation or liability, whether in tort (including negligence) or in contract, for any direct, indirect, incidental or consequential damages, even if Cayman is informed about their possible existence.

This limitation of liability does not apply in the case of intentional acts or negligence of Cayman, its directors or its employees.

Buyer's exclusive remedy and Cayman's sole liability hereunder shall be limited to a refund of the purchase price, or at Cayman's option, the replacement, at no cost to Buyer, of all material that does not meet our specification.

Said refund or replacement is conditioned on Buyer giving written notice to Cayman within thirty (30) days after arrival of the material at its destination. Failure of Buyer to give said notice within thirty (30) days shall constitute a waiver of Buyer of all claims hereunder with respect to said material.

For further details, please refer to our Warranty and Limitations of Remedy located on our website and in our catalog.

This Certificate shall not be reproduced except in full, without written approval from the Cayman Chemical ISO Quality Manager.

ISO CRT SD02 v 5.1

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