

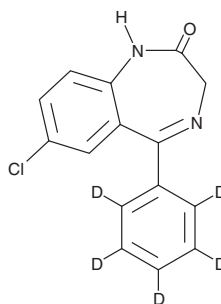
CONFIRMATION of ANALYSIS



ACCREDITED
ISO 17034 #AR-1774

Nordiazepam-d₅

Reference Material



Item No.:	15831
Batch No.:	0526461
CAS Registry No.:	65891-80-7
Molecular Formula:	C ₁₅ H ₆ ClD ₅ N ₂ O
Formula Weight:	275.8 amu
UV λ _{max} :	203, 228 nm
Expiry Date:	27AUG2033 (valid from date of certification)
Supplied as:	A neat solid
Storage:	Unopened at -20°C
Safety:	Refer to Safety Data Sheet
Intended Use:	For analytical testing purposes only, not intended for human or animal use.
Instructions for Use:	Once opened this material should be minimally exposed to ambient conditions and returned to recommended storage conditions immediately after use. Ongoing stability testing supports a negligible decrease in purity over a series of thaw-refreeze cycles. It is recommended that laboratories perform periodic testing to verify the material remains fit for the intended use.

Approval:

Title: ISO Quality Manager

Confirmation Date: 27AUG2018

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.



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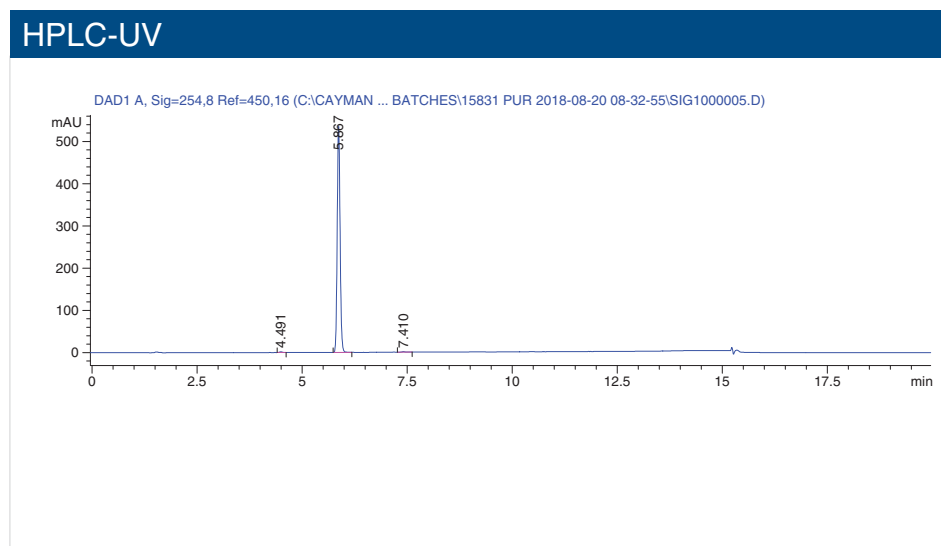
Qualifier	Method	Result
Appearance	Visual inspection	White solid
Chromatographic Purity, HPLC	Cayman Method TST SD101	99.37% $\pm$ 0.18%
Identity, LC-MS	Cayman Method TST SD13, +ESI	276.2 amu
Identity, GC-MS	Cayman Method TST SD12	Conforms
Identity, FTIR	USP<854> (diamond ATR)	Conforms
% LOD	Cayman Method TST SD24	0.20% $\pm$ 0.48%
% ROI	Cayman Method TST SD06	<0.10% $\pm$ 0.22%
Isotopic Purity, LC-MS	Cayman Method TST SD154	<0.10% d ₀ vs d ₅
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.

Measurement Uncertainty

All measurement uncertainties are expressed as expanded uncertainties in accordance with ISO standards for Testing Laboratories and Reference Material Producers at the approximate 95% confidence interval using an appropriate coverage factor. 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.

Supplemental Data (Neat Material)

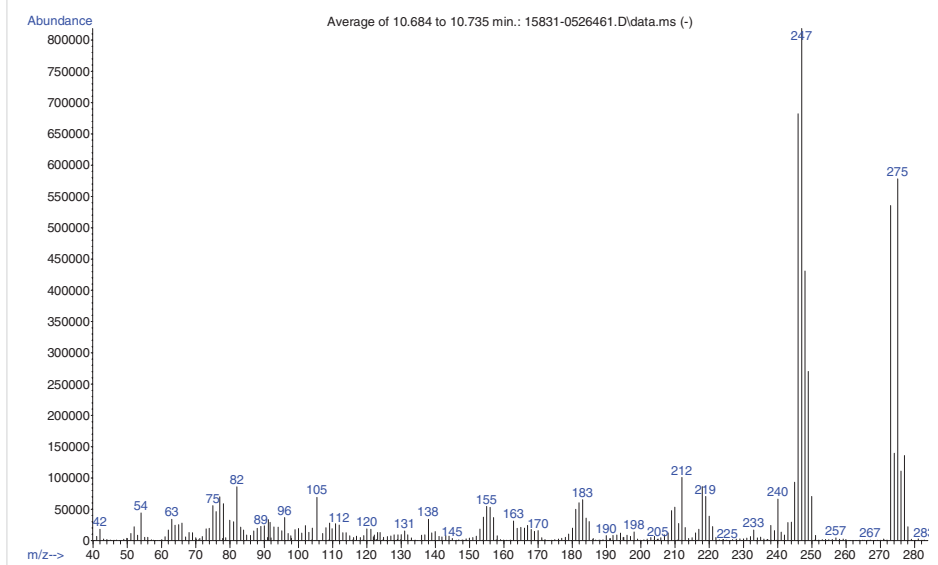


Conditions									
Instrument	Agilent 1100/1200 Series								
Column	4.6 x 150 mm, 5 μ m Zorbax Bonus-RP								
Mobile Phase	A: 10:90:10 mM Acetonitrile: Water:Ammonium Formate B: 90:10:10 mM Acetonitrile: Water:Ammonium Formate								
Gradient	<table border="1"> <thead> <tr> <th>Time (min)</th><th>%B</th></tr> </thead> <tbody> <tr> <td>0-10</td><td>31.2-100%</td></tr> <tr> <td>10-13</td><td>100%</td></tr> <tr> <td>13.1-20</td><td>31.2%</td></tr> </tbody> </table>	Time (min)	%B	0-10	31.2-100%	10-13	100%	13.1-20	31.2%
Time (min)	%B								
0-10	31.2-100%								
10-13	100%								
13.1-20	31.2%								
Flow Rate	1 ml/min								
Column Temp	30°C								
Wavelength	UV monitored at 254 nm								

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GC-MS

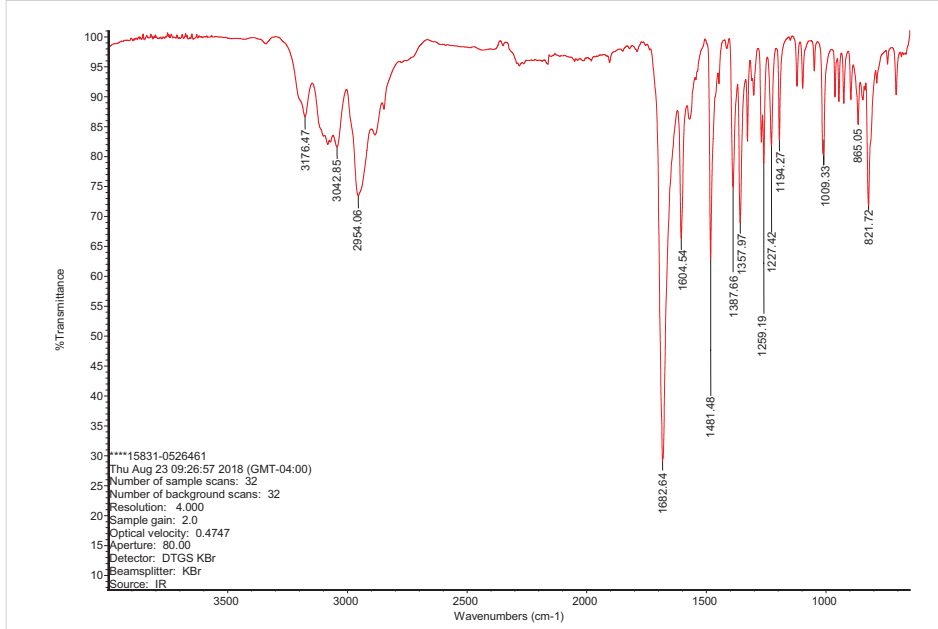


Conditions

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 15 min
Transfer Line Temp	300°C
Voltage	70eV EI MS
Scan Range	40-600 m/z
Tune File	stune

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

FTIR



Conditions

Instrument	Thermo Nicolet iS10 FTIR / Diamond SmartATR (single bounce)
Scans	32 scans / 32 background scans
Range	650-4,000 cm ⁻¹
Resolution	4.000

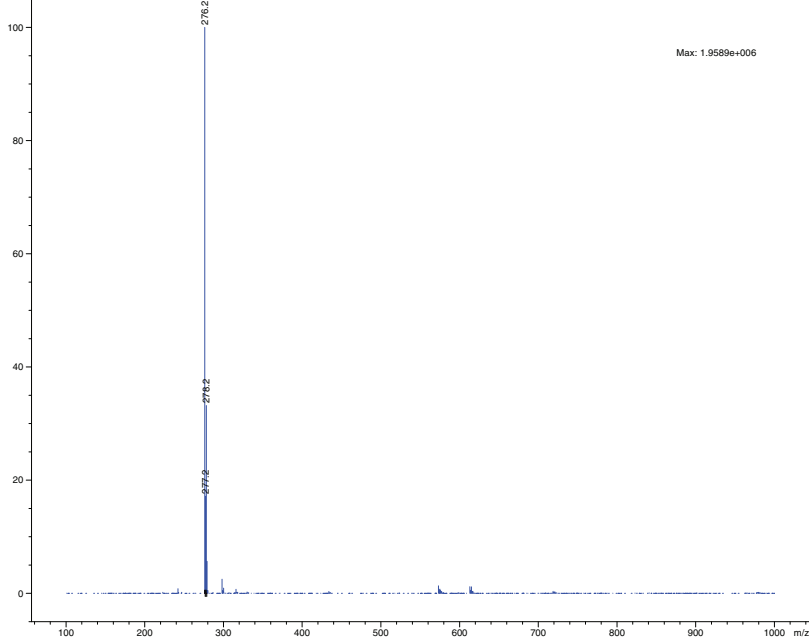
ATR and background corrected

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ESI-MS

*MSD1 SPC, time=3.265 of C:\CAYMAN\DATA\FIA\CAY010.D ES-API, Pos, Scan, Frag: 70, "+ESI"



Conditions

Instrument	Agilent HPLC MSD
Mobile Phase	50:50:0.1 MeOH/H ₂ O/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

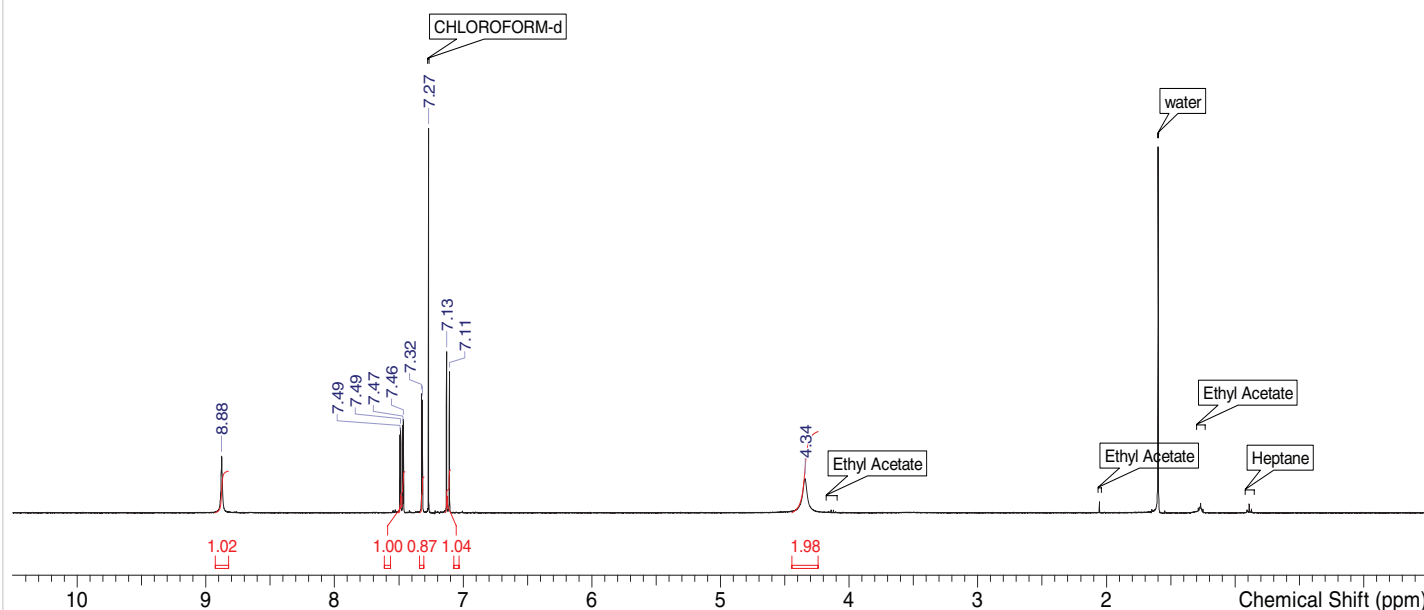
CONFIRMATION of ANALYSIS



NMR (not within scope of ISO accreditation)

Date	Aug 7 2018		
File Name	\\sulfur\private\nmrdata\ 2018\15831-0536461_20180807_15831-0536461_20180807_01\15831-0536461_20180807_001.fid\fid		
Frequency (MHz)	399.9661	Nucleus	1H
Solvent	CHLOROFORM-d	Temperature (degree C)	25.000
		Number of Transients	32

^1H NMR (CHLOROFORM-d, 400 MHz) δ 8.88 (br s, 1H), 7.48 (dd, 1H, $J=2.3, 8.6$ Hz), 7.32 (d, 1H, $J=2.3$ Hz), 7.12 (d, 1H, $J=8.6$ Hz), 4.34 (br s, 2H)



Conditions

Instrument	Varian Inova 400MHz NMR
Scans	32 scans

Homogeneity

A minimum sample size of 0.8 μg was used to determine homogeneity of the bulk solid. The recommended minimum quantity for use is 0.8 μg . Quantities below this have not been evaluated.

Short-Term Stability

No decrease in the purity 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 predicts fifteen years stability at the -20°C storage temperature. Long-term stability studies are ongoing and the Certificate of Analysis will be updated upon study completion.

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Revision History

Revision No.	Date	Reason for Revision
01	27AUG2018	Initial version
02	20SEP2018	Expiry date extension
03	07OCT2019	Expiry date extension
04	02NOV2020	Expiry date extension
05	27OCT2021	Updated format, accreditation symbols, and expiry date
06	21DEC2022	Expiry date extension

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 SD01 v 4.2

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