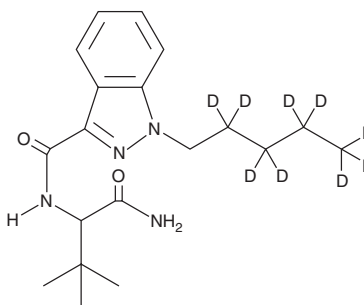


Reference Material

ACCREDITED
ISO 17034 #AR-1774



Approval: Robert Franklin

Confirmation Date: 09OCT2019

ANAB
ANSI National Accreditation Board
ACCREDITED
ISO 17034
REFERENCE MATERIAL
PRODUCER

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Qualifier	Method	Result
Appearance	Visual inspection	White solid
Chromatographic Purity, HPLC	Cayman Method TST SD85	98.53% $\pm$ 0.18%
Identity, LC-MS	Cayman Method TST SD13, +ESI	354.2 amu
Identity, GC-MS	Cayman Method TST SD12	Conforms
Identity, FTIR	USP<854> (diamond ATR)	Conforms
% LOD	Cayman Method TST SD153	0.58% $\pm$ 0.51%
% ROI	Cayman Method TST SD06	0.17% $\pm$ 0.20%
Isotopic Purity, LC-MS	Cayman Method TST SD154	<0.1% d ₀ vs. d ₉
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)

HPLC-UV

DAD1 A, Sig=304,8 Ref=450,100 (C:\CAYMAN\15112\15112 PURITY 2019-09-23 12-14-06\15112-0571172 INJ-1.D)

The chromatogram displays a baseline with several small peaks and one dominant peak. The x-axis represents time in minutes, ranging from 0 to 14. The y-axis represents mAU (milliabsorbance units), ranging from 0 to 1200. The peaks are labeled with their retention times: 5.800, 6.020, 6.336, 6.707, and 8.303. The peak at 6.707 is the most intense, reaching nearly 1200 mAU.

Retention Time (min)	Approximate mAU
5.800	50
6.020	40
6.336	60
6.707	1150
8.303	50

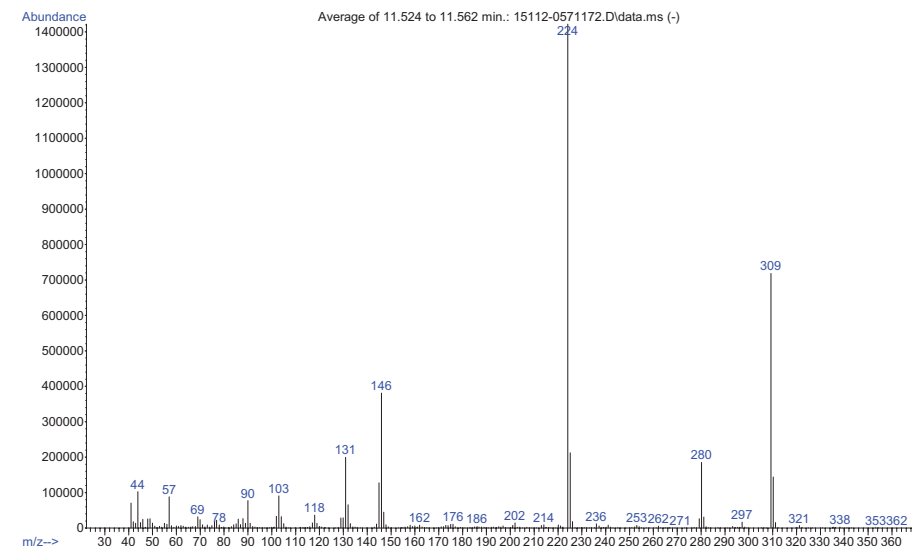
Conditions

Instrument	Agilent 1100/1200 Series								
Column	4.6 x 150 mm, 5 μm Zorbax Bonus-RP								
Mobile Phase	A: 10:90:10 mM Methanol:Water:Ammonium Acetate B: 90:10:10 mM Methanol:Water:Ammonium Acetate								
Gradient	<table><tr><th>Time (min)</th><th>%B</th></tr><tr><td>0-5</td><td>50-100%</td></tr><tr><td>5-13</td><td>100%</td></tr><tr><td>13.1-20</td><td>50%</td></tr></table>	Time (min)	%B	0-5	50-100%	5-13	100%	13.1-20	50%
Time (min)	%B								
0-5	50-100%								
5-13	100%								
13.1-20	50%								
Flow Rate	1 ml/min								
Column Temp	30°C								
Wavelength	UV monitored at 304 nm								

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GC-MS

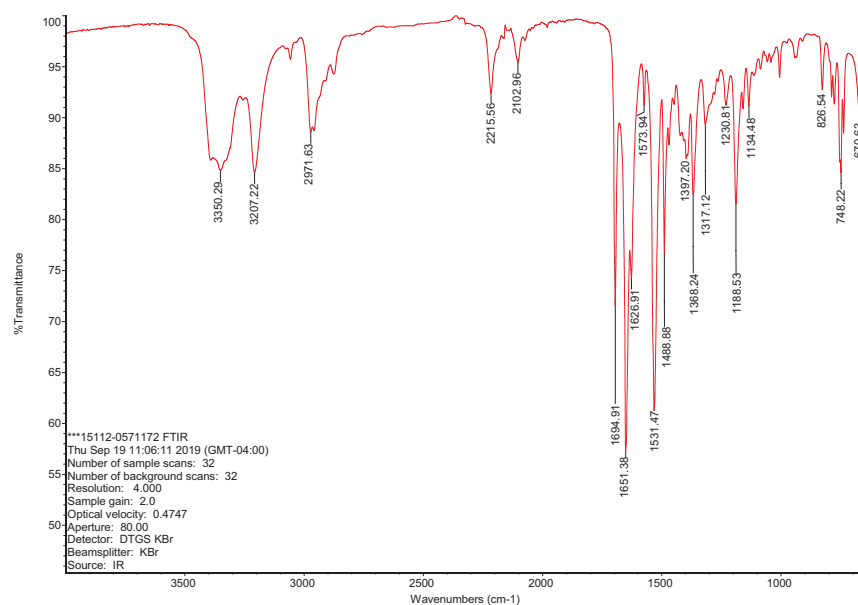


Conditions

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

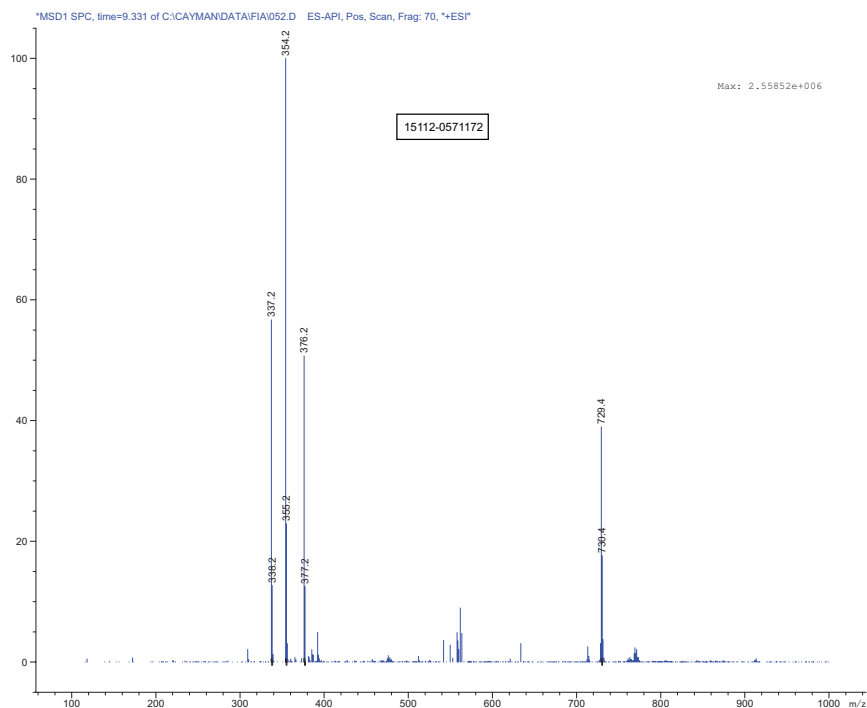
Instrument	Thermo Nicolet iS10 FTIR / Diamond SmartATR (single bounce)
Scans	32 scans / 32 background scans
Range	650-4,000 cm ⁻¹
Resolution	4.000

ATR and background corrected

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ESI-MS



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

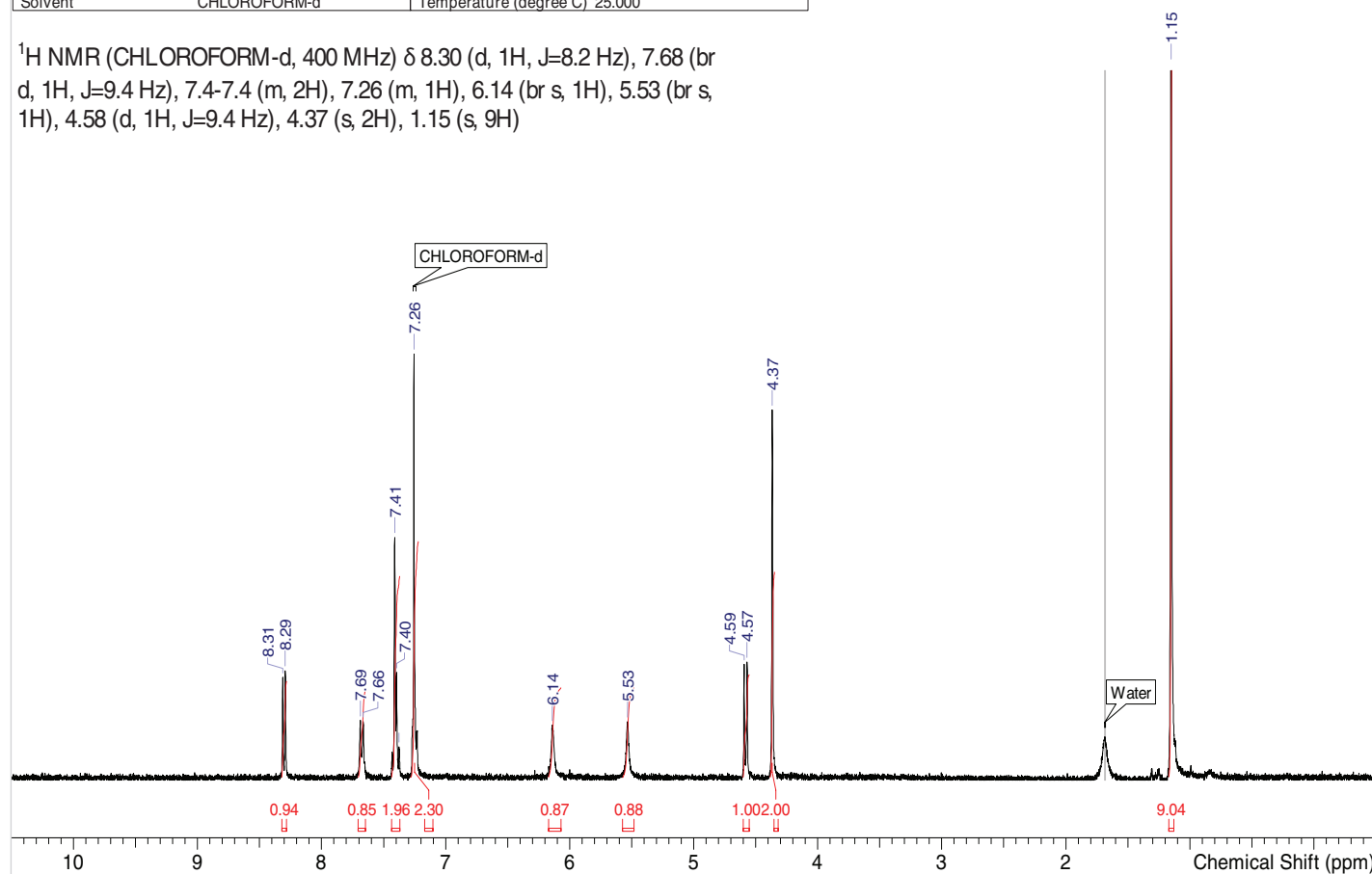
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NMR (not within scope of ISO accreditation)

Date	Sep 6 2019	
File Name	\\sulfur\private\nmrdata\ 2019\15112-0571172_20190906_1\15112-0571172_20190906_01\15112-0571172_20190906_001.fid\fid	
Frequency (MHz)	399.9661	Nucleus 1H
Solvent	CHLOROFORM-d	Temperature (degree C) 25.000
		Number of Transients 64

^1H NMR (CHLOROFORM-d, 400 MHz) δ 8.30 (d, 1H, J=8.2 Hz), 7.68 (br d, 1H, J=9.4 Hz), 7.4-7.4 (m, 2H), 7.26 (m, 1H), 6.14 (br s, 1H), 5.53 (br s, 1H), 4.58 (d, 1H, J=9.4 Hz), 4.37 (s, 2H), 1.15 (s, 9H)



Conditions

Instrument	Varian Inova 400 MHz NMR
Scans	64 scans

Homogeneity

A minimum sample size of 3.0 μg was used to determine homogeneity of the bulk solid. The recommended minimum quantity for use is 3.0 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 15 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.

CONFIRMATION of ANALYSIS



Revision History

Revision No.	Date	Reason for Revision
01	09OCT2019	Initial version
02	24JAN2020	Expiry date extension
03	28MAR2022	Updated format, accreditation symbols, and expiry date
04	24JAN2023	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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