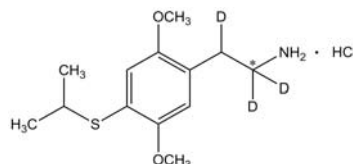


## Certified Reference Material - Certificate of Analysis

### 2,5-Dimethoxy-4-isopropylthiophenethylamine-<sup>13</sup>C,<sub>3</sub>D<sub>3</sub>, Primary Standard

(2C-T-4-<sup>13</sup>C,<sub>3</sub>D<sub>3</sub> HCl)

**Catalog Number:** T-090  
**Lot:** FE03241403  
**Retest:** June 2015  
**Description:** 2,5-Dimethoxy-4-isopropylthiophenethylamine-<sup>13</sup>C,<sub>3</sub>D<sub>3</sub> HCl  
in Methanol. Nominal concentration is adjusted for HCl content.



Cerilliant Quality
ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

**Packaging:** Solution in 2 mL amber USP Type I glass ampoule containing not less than 1 mL of certified solution.

**Storage:** Store unopened in freezer (-10 °C to -25 °C). See Stability Section for short-term storage.

**Shipping:** Ambient. See Stability Section.

**Intended Use:** This Certified Reference Material is suitable for the *in vitro* identification, calibration, and quantification of the analyte(s) in analytical and R&D applications. Not suitable for human or animal consumption.

**Instructions for Use:** Users should quantitatively transfer desired volume using established good laboratory practices to spike into matrix or to dilute to the desired concentration. Each ampoule is intended for one-time use.

**Regulatory:** USDEA Exempt

**Safety: Danger. See Safety Data Sheet**

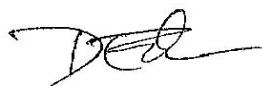
- Retest Date - stability studies ongoing. Certificate of Analysis will be updated upon completion of retest.
- Ampoules are overfilled to ensure a minimum 1 mL volume can be transferred when using a 1 mL Class A volumetric pipette
- For MS Applications, we advise laboratories not to mix lots during a single sequence.

Analyte	Certified Concentration Value
2,5-Dimethoxy-4-isopropylthiophenethylamine- <sup>13</sup> C, <sub>3</sub> D <sub>3</sub> HCl	100.0 ± 0.5 µg/mL
<ul style="list-style-type: none"> <li>Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and Guide 34 at the approximate 95% confidence interval using a coverage factor of k = 2 and has been calculated by statistical analysis of our production system and incorporates uncertainty of the mass balance purity factor, material density, balance, and weighing technique.</li> <li>This standard is prepared gravimetrically and mass results are reported on the conventional basis for weighing in air. Nominal concentration is calculated based on: the actual measured mass; Mass Balance Purity Factor of the analyte(s); measured mass of the solution; and the density of the pure diluent at 20 °C.</li> <li>Concentration is corrected for chromatographic purity, residual water, residual solvents and residual inorganics. Nominal concentration is adjusted for HCl content. No adjustment required before use.</li> <li>Additional certification information available upon request.</li> </ul>	

### Metrological Traceability

- This standard has been prepared and certified under the ISO Guide 34, ISO/IEC 17025, ISO 9001 and ISO 13485 standards. This standard meets the requirements of a Certified Reference Material and a Primary Standard as defined by ISO and is traceable to the SI and higher order standards through an unbroken chain of comparisons.
- This standard has been gravimetrically prepared using balances that have been fully qualified and calibrated to ISO 17025 requirements. All calibrations utilize NIST traceable weights which are calibrated externally by a qualified ISO 17025 accredited calibration laboratory to NIST standards. Qualification of each balance includes the assignment of a minimum weighing by a qualified and ISO 17025 accredited calibration vendor taking into consideration the balance and installed environmental conditions to ensure compliance with USP tolerances of NMT 0.1% relative uncertainty. Balance calibration adjustments are performed weekly utilizing the balance's internal adjustment mechanism. Calibration verifications are performed pre-use. Weigh tapes from the calibration verification are included in the production batch record for this standard. Production data package available upon request.
- Fill volume is gravimetrically verified throughout the dispensing process using qualified and calibrated balances.
- Concentration is verified against an independently prepared calibration solution gravimetrically prepared.
- Each raw material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques. Spectral data is provided on subsequent pages of this COA. The density and material Mass Balance Purity Factor is traceable to the SI and higher order reference standards through mass measurement and instrument qualification and calibrations.

Cerilliant certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration/retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.

Darron Ellsworth, Quality Assurance Manager

May 7, 2014

Date

## Solution Standard Verification

Concentration accuracy and within- and between-bottle homogeneity are analytically verified against an independently prepared calibration solution.

Solution standard verification demonstrates confirmation that the specified requirements for the Primary Standard have been fulfilled and validated under ISO 13485.

Standard Solution Assay Parameters		Calibration Curve			
<b>Analysis Method:</b>	UV/Vis	<b>Calibration Curve:</b>	Linear Regression		
<b>Wavelength:</b>	220 nm	<b>Number of Points:</b>	4		
<b>Slit Width:</b>	1.0 nm	<b>Linearity (r):</b>	1.000		
<b>Response:</b>	0.5 s				

Standard Solution	Lot Number	Verified Concentration (µg/mL)		%RSD - Homogeneity	
		Actual Results	Acceptance Criteria	Actual Results	Acceptance Criteria
New Lot	FE03241403	101.5	± 3%	1.1	≤ 3%

<ul style="list-style-type: none"> <li>Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration solution.</li> <li>Within-sample and between-sample homogeneity of the New Lot is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. Multiple samples pulled from across the lot using a random stratified sampling plan were analyzed to verify homogeneity. % RSD results shown above for the New Lot demonstrate ampoule-to-ampoule homogeneity.</li> </ul>
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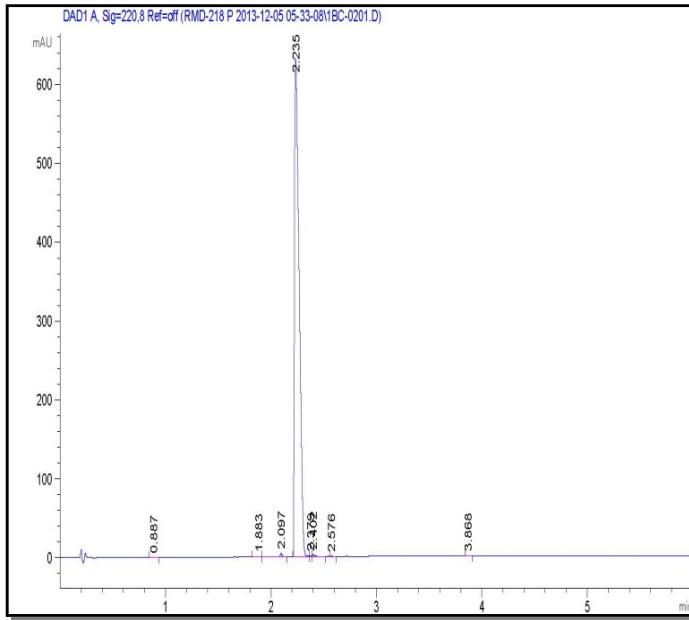
### Analyte Certification - Mass Balance Purity Factor

Each analyte is thoroughly identified and characterized using an orthogonal approach. A mass balance purity factor is assigned incorporating chromatographic purity and residual impurities. The mass balance purity factor and salt adjustment are utilized to calculate the weighing adjustment necessary to ensure accuracy of the solution standard concentration.

Material	Lot Number	CAS Number	Chemical Formula	Molecular Weight (salt)	Molecular Weight (base)	Salt Adjustment
2C-T-4- <sup>13</sup> C, <sub>3</sub> D <sub>3</sub> HCl	FC10151301	NA	C <sub>12</sub> <sup>13</sup> CH <sub>18</sub> D <sub>3</sub> NO <sub>2</sub> S•HCl	295.85	259.39	1.141
<b>Material Characterization Summary</b>						
<b>Analytical Test</b>		<b>Method</b>		<b>Results</b>		
Primary Chromatographic Purity by HPLC/UV Analysis		SP10-0102		99.4%		
Secondary Chromatographic Purity by GC/FID Analysis		SP10-0101		99.7%		
Identity by GC/MS Analysis		SP10-0105		Consistent with Structure		
Isotopic Purity by GC/MS		SP10-0105		0.11% M <sub>0</sub> vs M <sub>4</sub>		
				0.11% M <sub>0</sub>	5.99% M <sub>3</sub>	
				0.29% M <sub>1</sub>	93.29% M <sub>4</sub>	
				0.32% M <sub>2</sub>		
Identity by LC/MS Analysis		SP10-0107		Consistent with Structure		
Isotopic Purity by LC/MS SIM or High Resolution MS <sup>1</sup>		SP10-0107		0.00% M <sub>0</sub> vs M <sub>4</sub>		
				0.00% M <sub>0</sub> to M <sub>1</sub>	91.47% M <sub>4</sub>	
				0.04% M <sub>2</sub>	5.03% M <sub>5</sub>	
				3.46% M <sub>3</sub>	0.00% M <sub>6</sub>	
Identity by <sup>1</sup> H-NMR Analysis		USP <761>, SP10-0116		Consistent with Structure		
Residual Solvent Analysis by GC/FID Headspace		AM1087 <sup>2</sup>		Below Quantitation Limit		
Residual Water Analysis by Karl Fischer Coulometry		AM1346 <sup>2</sup>		Below Quantitation Limit		
Inorganic Content by Microash Analysis		SP10-0135		< 0.2%		
Mass Balance Purity Factor				99.44%		
<ul style="list-style-type: none"> <li>♦ The primary chromatographic purity is calculated as the average of two independently performed analyses utilizing two different methods. Acceptance criteria requires the purity values to be within 0.5% of each other.</li> <li>♦ The primary chromatographic purity value is used to calculate the Mass Balance Purity Factor.</li> <li>♦ A secondary chromatographic purity method is utilized as a control.</li> <li>♦ Mass Balance Purity Factor = [(100 - wt% residual solvent - wt% residual water - wt% residual inorganics) x Chromatographic Purity/100].</li> <li>♦ Mass Balance Purity Factor does not include adjustment for chiral and/or isotopic purity.</li> </ul> <p><sup>1</sup> Isotopic distribution values are adjusted for the natural abundance of isotopes (M + 1 adjusted 20.18%; M + 2 adjusted 2.55%)</p> <p><sup>2</sup> Validated analytical method</p>						

**Spectral and Physical Data**

**HPLC/UV**



**Column:** Ascentis Express C18, 2.7  $\mu$ , 2.1 x 50 mm  
**Mobile Phase:** A::Acetonitrile B::0.1% Phosphoric acid  
**Gradient:**

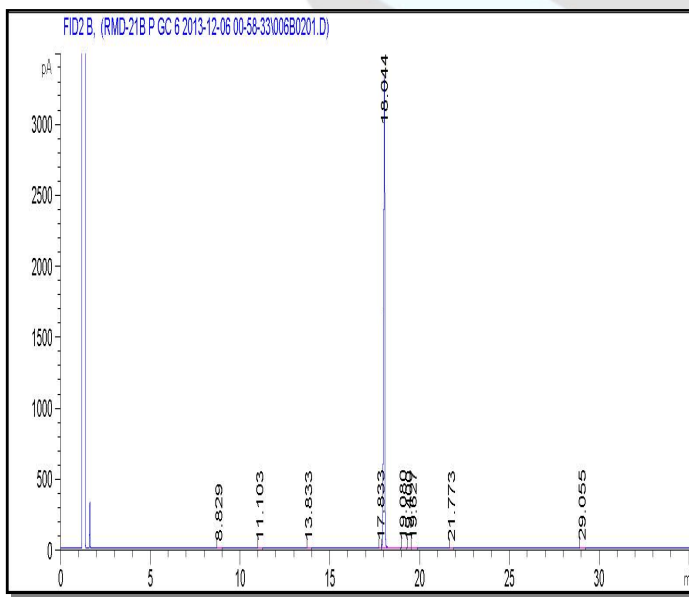
Time	A	B
0.0	10	90
3.0	70	30
5.0	70	30
5.1	10	90
6.0	10	90

**Flow Rate:** 0.6 mL/min  
**Wavelength:** 220 nm

**Data File Name:** RMD-218 P 2013-12-05 05-33-08\1BC-0201.D  
**Instrument:** LC#13  
**Sample Name:** FC10151301  
**Acquired:** December 5, 2013

Peak #	Ret Time	Area	Height	Area %
1	0.89	0.21	0.08	0.01
2	1.88	0.29	0.18	0.01
3	2.10	4.87	4.11	0.25
4	2.24	1931.26	630.97	99.43
5	2.38	1.27	1.22	0.07
6	2.40	3.51	2.60	0.18
7	2.58	0.38	0.37	0.02
8	3.87	0.54	0.39	0.03

**GC/FID**



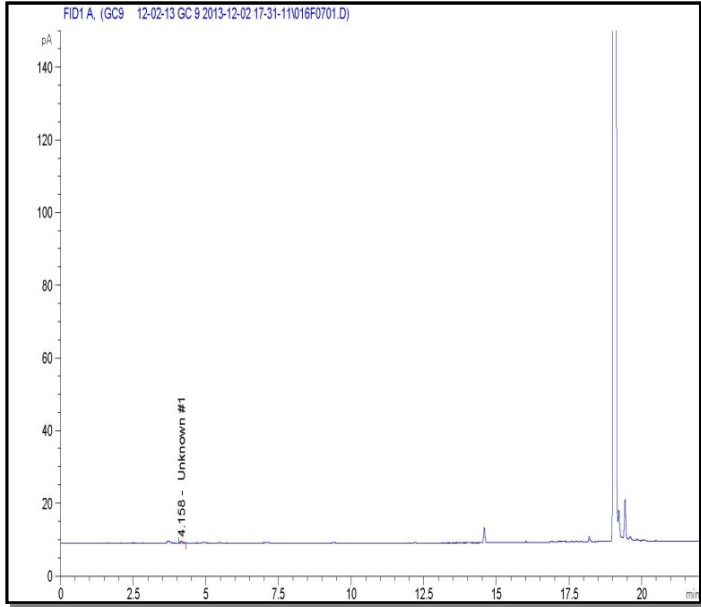
**Column:** DB-35ms, 30 m x 0.53 mm ID, 1.0  $\mu$ m film thickness  
**Temp Program:** 40°C to 140°C at 40°C/min  
 140°C to 280°C at 5°C/min hold 5 min  
**Injector Temp:** Cool-on-Column  
**Detector Temp:** 325°C

**Data File Name:** RMD-218 P GC 6 2013-12-06 00-58-33\006B0201.D  
**Instrument:** GC#6  
**Sample Name:** FC10151301  
**Acquired:** December 6, 2013

Peak #	Ret Time	Area	Height	Area %
1	8.83	1.19	0.18	0.01
2	11.10	0.56	0.09	0.00
3	13.83	6.58	1.37	0.04
4	17.83	27.03	5.37	0.17
5	18.04	15944.00	3301.38	99.71
6	19.08	2.40	0.46	0.02
7	19.40	1.27	0.24	0.01
8	19.63	5.39	0.96	0.03
9	21.77	0.53	0.10	0.00
10	29.06	1.47	0.24	0.01

*Spectral and Physical Data (cont.)*

**Residual Solvent Analysis by GC/FID Headspace**



**Column:** DB-ALC1 30 m x 0.53 mm, 3 µm film thickness  
**Temp Program:** 40°C (12 min) to 220°C at 40°C/min (5.5 min)  
**Carrier Gas:** Helium  
**Flow Rate:** 2.0 mL/min  
**Detector Heater Temp:** 250°C  
**Injector:** Headspace Sampler  
**HS Oven Temp:** 60°C  
**Vial Equilibration:** 10 minutes

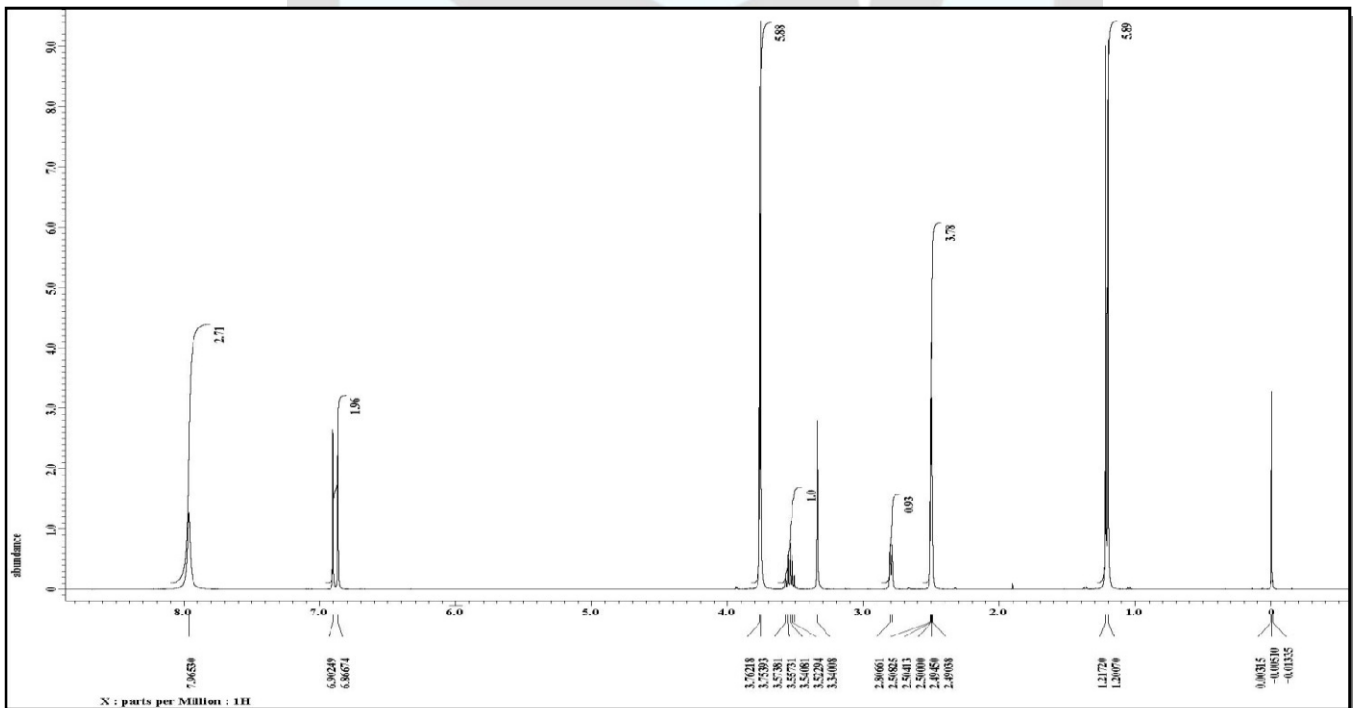
**Data File Name:** GC 9 2013-12-02 17-31-11\016F0701.D  
**Instrument:** GC#9  
**Sample Name:** FC10151301  
**Acquired:** December 2, 2013

Peak	Compound	Area	Weight %
1	Unkown #1	2.78	BQL
2	NMP	NA	NA
<b>Total</b>			<b>BQL</b>

BQL - Below Quantitation Limit

**<sup>1</sup>H NMR**

**Instrument:** JEOL ECS 400  
**Solvent:** DMSO-D<sub>6</sub>



*Spectral and Physical Data (cont.)*

**LC/MS**

**Column:** Ascentis Express C18, 2.7  $\mu$ m, 3.0 x 50 mm

**Mobile Phase:** A:: 0.1% Formic acid in water

B:: Acetonitrile

Gradient	Time (mins)	%A	%B
Program:	0.0	90	10
	0.5	90	10
	4.0	50	50
	5.8	50	50
	6.0	90	10
	8.0	90	10

**Flow Rate:** 0.4 mL/min

**Scan Range:** 100-1200 amu

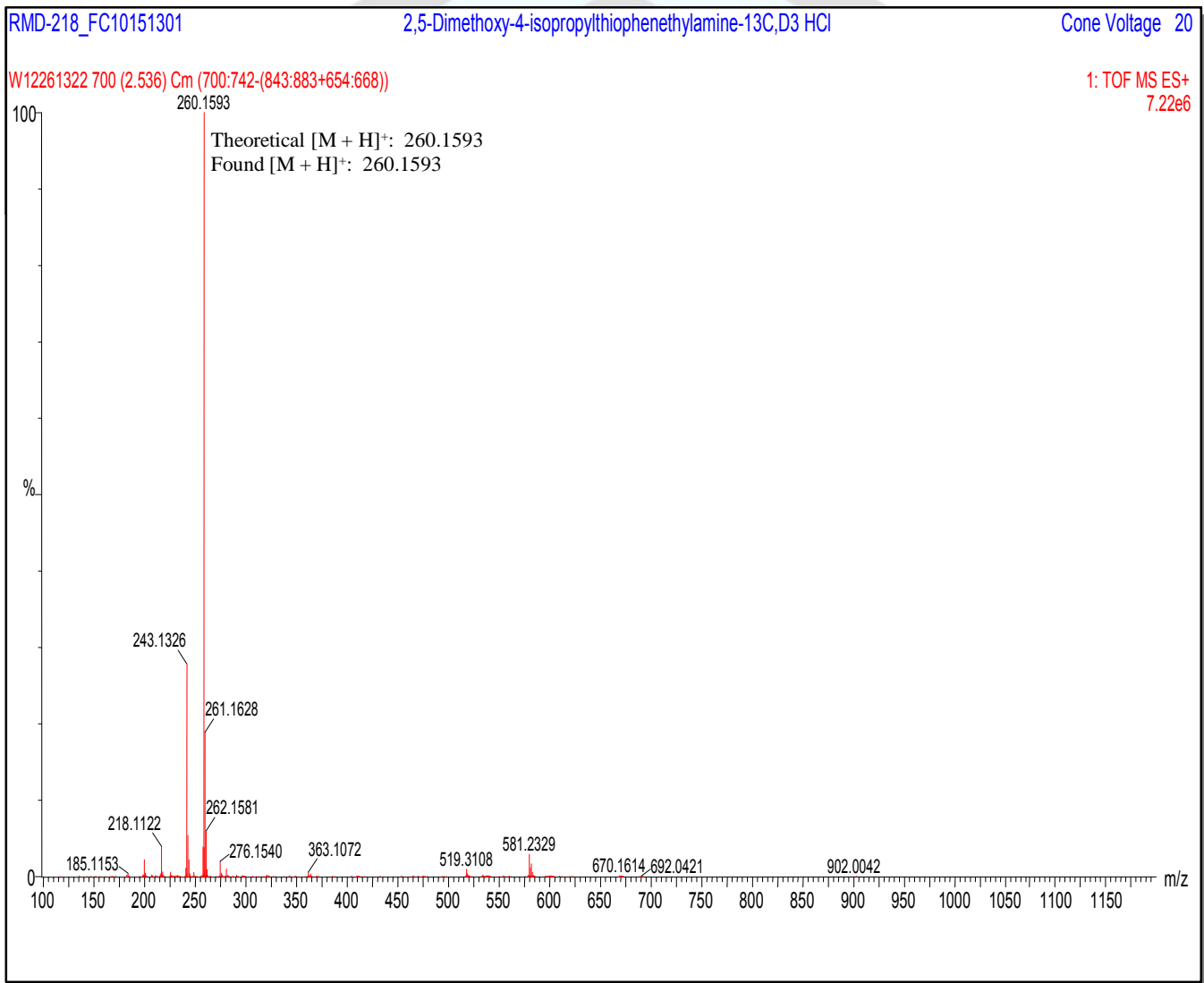
**Ionization:** Electrospray, Positive Ion

**Data File Name:** W12261322

**Instrument:** Waters XEVO G2 QTOF

**Sample Name:** FC10151301

**Acquired:** December 26, 2013



*Spectral and Physical Data (cont.)*

**Isotopic Purity by LC/MS SIM or High Resolution MS Analysis**

**Column:** Ascentis Express C18, 2.7 µm, 3.0 x 50 mm

**Flow Rate:** 0.4 mL/min

**Mobile Phase:** A:: 0.1% Formic acid in water

**Scan Range:** 256-262 amu

B:: Acetonitrile

**Ionization:** Electrospray, Positive Ion

**Gradient**

Time (mins)	%A	%B
0.0	90	10
0.5	90	10
4.0	50	50
5.8	50	50
6.0	90	10
8.0	90	10

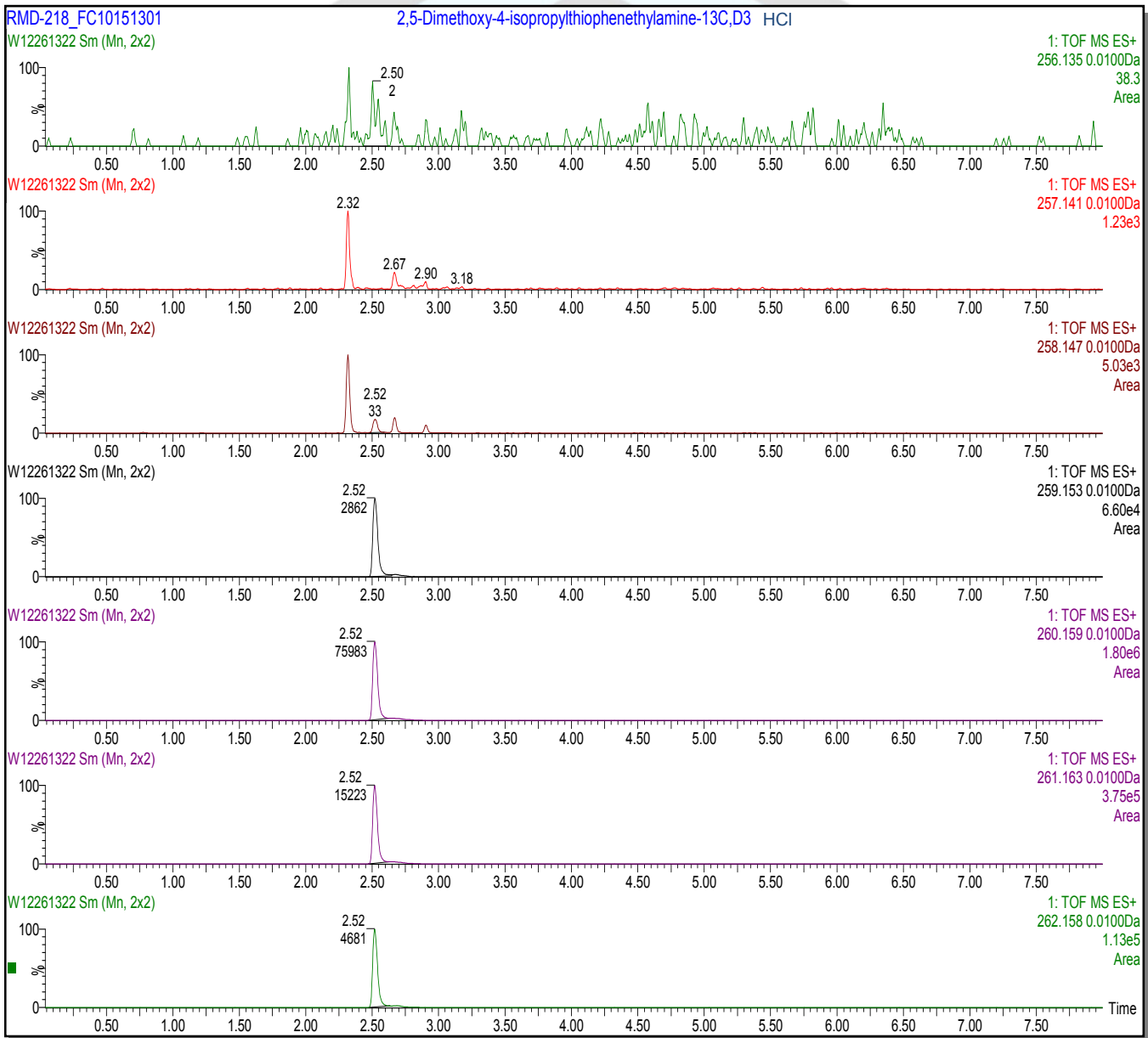
**Data File Name:** W12261322

**Program:**

**Instrument:** Waters XEVO G2 QTOF

**Sample Name:** FC10151301

**Acquired:** December 26, 2013

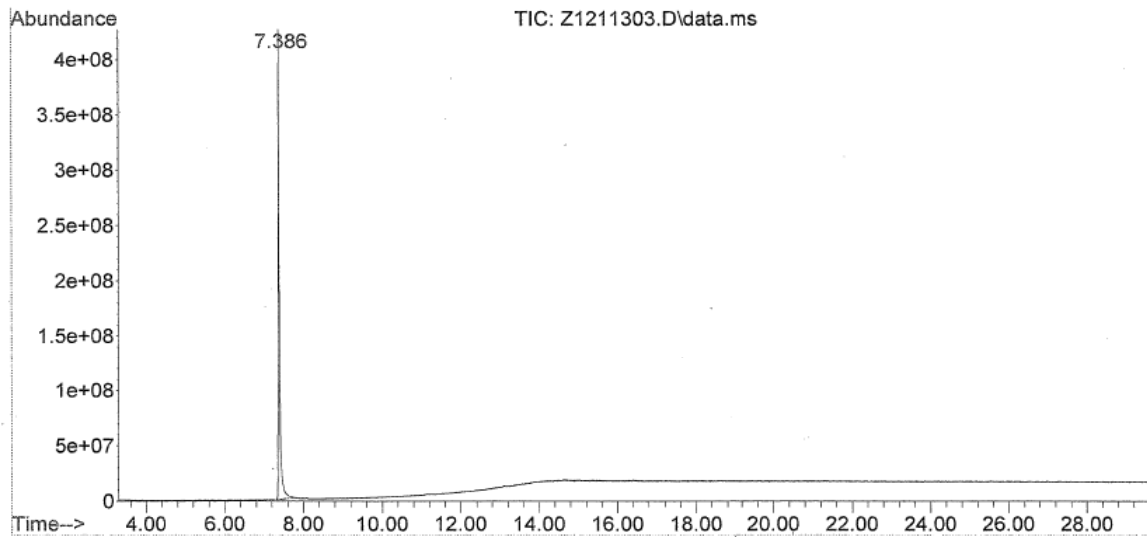


*Spectral and Physical Data (cont.)*

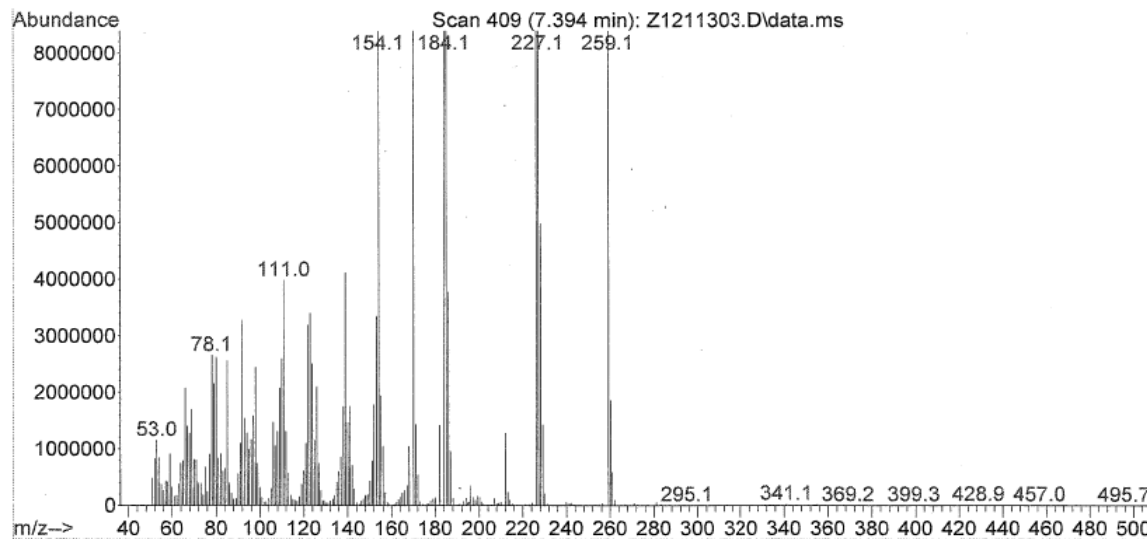
**GC/MS**

Compound Name : 2,5-Dimethoxy-4-isopropylthiophenethylamine/ *Free Base\**  
Lot Number : FC10151301  
Instrument : Agilent GCMS  
Operator : ECM(BRYAN DOCKERY)  
Date Reported : Wed Dec 11 17:08:53 2013  
Column Type : DB-5ms, 30m x 0.25mm ID, 0.25um film thickness  
Temp. Program : 50°C to 200°C@40°C/min, 200°C to 300°C@10°C/min, 16min hold  
Injector Temp. : Cool on-column  
Carrier Gas : Helium  
Flow Rate (mL/min) : 0.80 mL/min  
Transfer Line Temp. : 280°C  
Scan Range : 50-500

Total Ion Chromatogram



Mass Spectrum





## Stability

Short term stability studies have been performed under accelerated conditions for periods up to one week. Short term data is utilized to predict long term stability and to support transport conditions and normal laboratory use. Real-time stability studies are performed at the recommended storage conditions over the life of the product.

**Short Term Stability** : A summary of accelerated stability findings for a related product (T-088, 2C-T-4 HCl) is listed below.

Storage Condition	Mean Kinetic Temperature (MKT)	Time Period/Result
Freezer	-15°C	No decrease in purity was noted after one week.
Refrigerator	4°C	
Room Temperature	21°C	
40°C	40°C	

**Transport/Shipping** : Stability data supports transport of this product at ambient conditions.

**Short Term Storage**: Stability data supports short term storage for no more than 3 months at Refrigerate conditions.

## COA Revision History

Revision No.	Date	Reason for Revision
00	May 7, 2014	Initial version