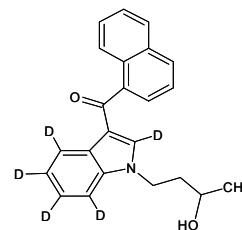


Cerilliant Quality
ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

# Certificate of Analysis

## JWH-073 3-Hydroxybutyl metabolite-D<sub>5</sub> (indole-D<sub>5</sub>)

**Catalog Number:** S-040  
**Solution Lot:** FE081511-02  
**Retest Date:** July 2016  
**Solvent:** Methanol  
**Volume per Ampule:** Not less than 1 mL  
**Storage:** Store in Freezer. See Stability Section for short term storage.  
**Shipping:** Ambient. See Stability Section  
**Intended Use:** For R&D/ analytical purposes only. Not suitable for human or animal consumption.  
**Regulatory:** Canadian TK# 61-644



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

- Retest Date - stability studies ongoing. Certificate of Analysis will be updated upon completion of retest.
- Ampules are overfilled to ensure a minimum 1 mL volume fill. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.
- For MS Applications, we advise laboratories not to mix lots during a single sequence.
- For quantitative applications, the minimum sample size for intended use is 1  $\mu$ L.

Component	Chromatographic Purity	Certified Concentration
JWH-073 3-Hydroxybutyl metabolite-D <sub>5</sub>	99.4%	100.0 $\pm$ 0.6 $\mu$ g/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 purity factor, material density, and balance and weighing technique.</li> <li>• This standard is prepared gravimetrically and mass results are reported on the conventional basis for weighing in air. Concentration is calculated based on: the actual measured mass; 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.</li> </ul>		

### Solution Standard Verification and Homogeneity

Standard Solution	Lot Number	Verified Concentration ( $\mu$ g/mL)		%RSD - Homogeneity	
		Actual Results	Acceptance Criteria	Actual Results	Acceptance Criteria
New Lot	FE081511-02	97.39	$\pm$ 3%	0.2	$\leq$ 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>• Homogeneity of the New Lot is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. The % RSD of samples pulled from across the lot demonstrate homogeneity of the New Lot.</li> </ul>					

### Traceability

- Gravimetrically prepared using qualified balances calibrated semi-annually by Mettler Toledo using NIST traceable weights. Calibration verification performed weekly and prior to each use utilizing NIST traceable weights. Each balance has been assigned a minimum weighing by Mettler Toledo taking into consideration the balance and installed environmental conditions to ensure weighing complies with USP tolerances of no more than 0.1% relative error.
- Concentration is verified against an independently prepared calibration solution gravimetrically prepared using balances calibrated to NIST.
- In addition, each neat material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques. Spectral data is provided on subsequent pages of the COA.

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 11, 2015

Date

### Standard Solution Assay Parameters

**Analysis Method:** UV/Vis  
**Wavelength:** 315 nm  
**Slit Width:** 1.0 nm  
**Response:** 0.5 s

### Calibration Curve

**Calibration Curve:** Linear Regression  
**Number of Points:** 4  
**Linearity (r):** 1.000

### Neat Material Data

**Compound Name:** JWH-073 3-Hydroxybutyl metabolite-D<sub>5</sub> (indole-D<sub>5</sub>)  
**Compound Lot:** FN071811-03  
**Chemical Formula:** C<sub>23</sub>H<sub>16</sub>D<sub>5</sub>NO<sub>2</sub>  
**CAS Number:** NA  
**Molecular Weight:** 348.45

#### Neat Material Characterization Summary

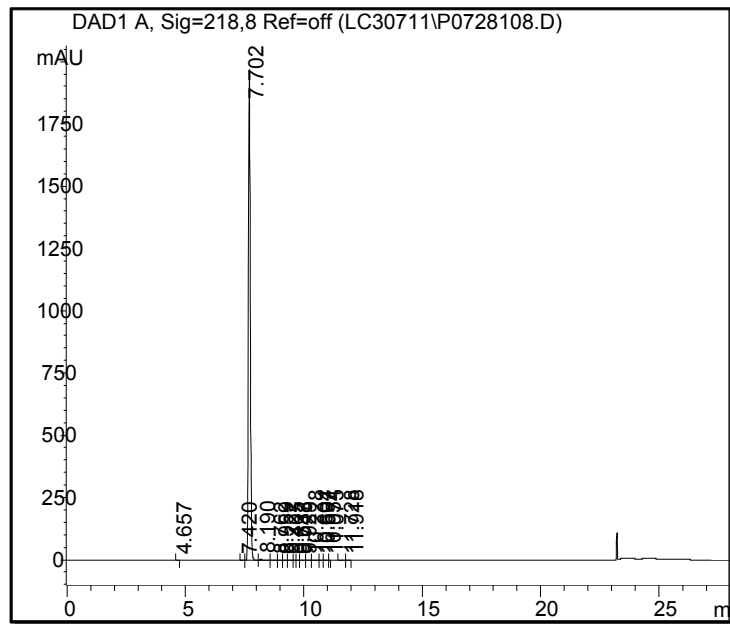
Analytical Test	Method	Results
Primary Chromatographic Purity by HPLC/UV Analysis	SP10-0102	99.4%
Secondary Chromatographic Purity by GC/FID Analysis	SP10-0101	99.5%
Identity by LC/MS Analysis	SP10-0107	Consistent with Structure
Isotopic Purity and Distribution by LC/MS SIM Analysis	SP10-0107	0.00% D <sub>0</sub> vs D <sub>5</sub>
		0.00% D <sub>0</sub>   0.26% D <sub>3</sub>
		0.00% D <sub>1</sub>   6.41% D <sub>4</sub>
		0.08% D <sub>2</sub>   93.24% D <sub>5</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>1</sup>	0.34%
Residual Water Analysis by Karl Fischer Coulometry	USP <921>, SP10-0103	0.16%
Inorganic Content by Microash Analysis	SP10-0135	< 0.2%
Purity Factor		98.93%

- ♦ Primary 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.
- ♦ The primary chromatographic purity value is used to calculate the Purity Factor.
- ♦ A secondary chromatographic purity method is utilized as a control.
- ♦ Purity Factor = [(100 - wt% residual solvent - wt% residual water - wt% residual inorganics) x Chromatographic Purity/100].
- ♦ Purity factor does not include adjustment for chiral and/or isotopic purity.

<sup>1</sup> Validated analytical method

*Spectral and Physical Data*

**HPLC/UV**



**Column:** Prodigy ODS 3, 5  $\mu$ m, 4.6 x 250 mm  
**Mobile Phase:** A: Acetonitrile B: 0.1% H<sub>3</sub>PO<sub>4</sub>  
**Gradient**

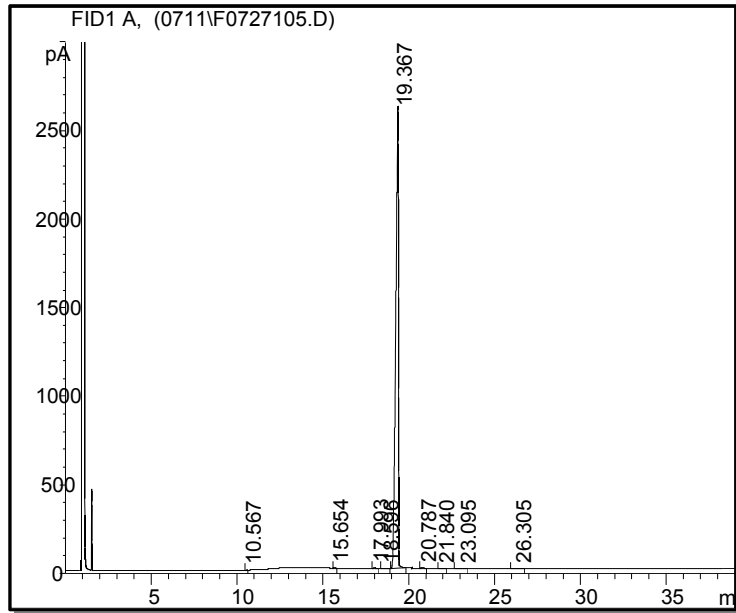
Time (mins)	%A	%B
0.0	55	45
10.0	90	10
20.0	90	10
20.1	55	45

**Program:**  
**Flow Rate:** 1.0 mL/min  
**Wavelength:** 218 nm  
**Data File Name:** S:\HPLC\HPLC3\2011\LC30711\P0728108.D  
**Instrument:** LC#3  
**Sample Name:** FN071811-03  
**Acquired:** July 28, 2011

Peak #	Ret Time	Area	Height	Area %
1	4.66	0.13	0.03	0.00
2	7.42	2.30	0.45	0.02
3	7.70	11255.50	1978.52	99.24
4	8.19	39.79	4.66	0.35
5	8.76	15.34	1.80	0.14
6	8.96	4.16	0.69	0.04
7	9.23	3.29	0.56	0.03
8	9.41	5.46	0.66	0.05
9	9.64	1.17	0.26	0.01
10	9.75	4.65	0.70	0.04
11	9.93	5.72	0.67	0.05
12	10.23	0.44	0.04	0.00
13	10.60	0.54	0.08	0.00
14	10.70	0.32	0.05	0.00
15	10.95	1.63	0.26	0.01
16	11.08	0.08	0.03	0.00
17	11.73	0.19	0.03	0.00
18	11.92	0.79	0.14	0.01

Spectral and Physical Data (cont.)

GC/FID

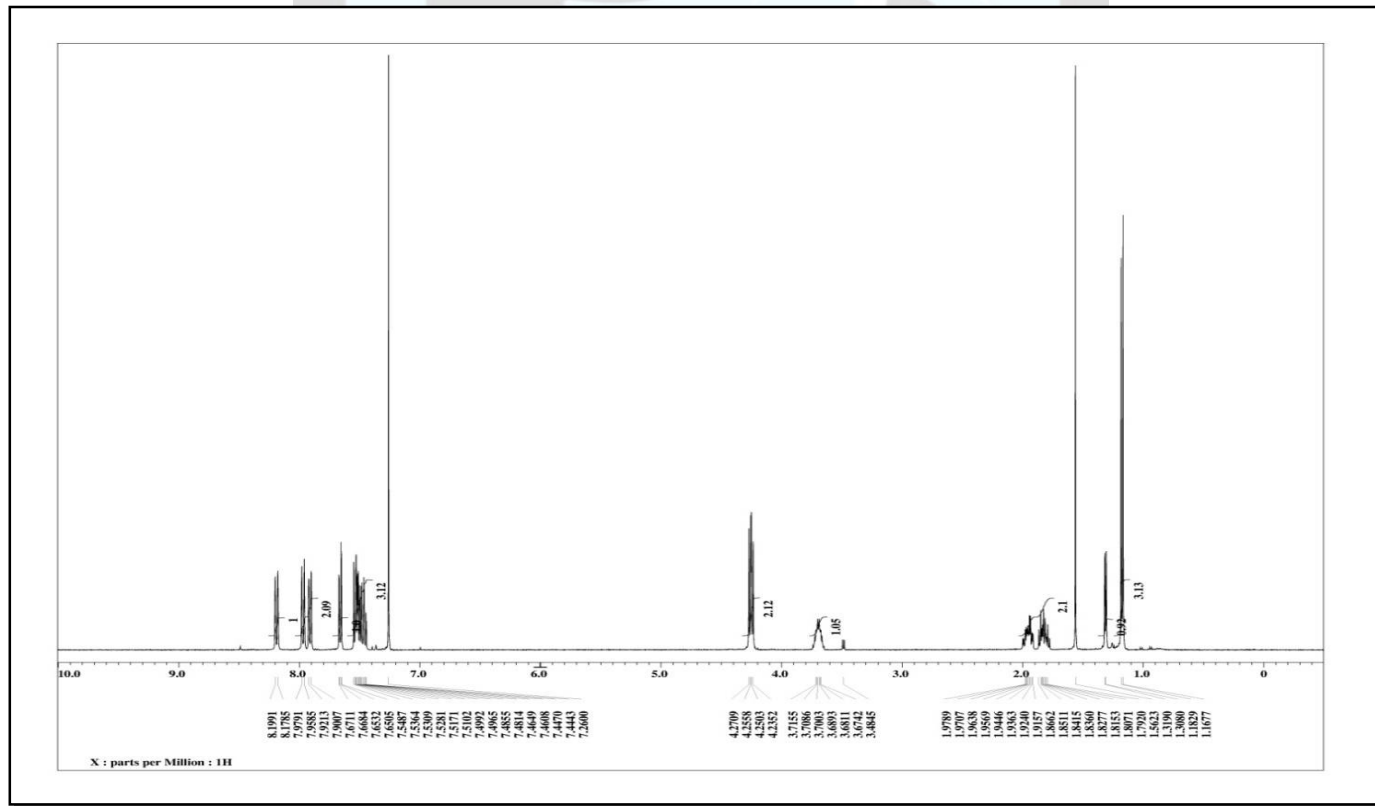


**Column:** DB-5ms, 30 m x 0.53 mm ID, 1.5 µm film thickness  
**Temp Program:** 60°C to 300°C at 20°C/min hold 27 mins  
**Injector Temp:** Cool-on-Column  
**Detector Temp:** 325°C  
**Data File Name:** S:\GC\GC6\2011\0711\F0727105.D  
**Instrument:** GC#6  
**Sample Name:** FN071811-03  
**Acquired:** July 27, 2011

Peak #	Ret Time	Area	Height	Area %
1	10.57	2.91	1.28	0.01
2	15.65	12.35	2.02	0.04
3	17.99	19.37	2.44	0.07
4	18.60	18.51	1.08	0.07
5	19.37	27444.70	2608.75	99.47
6	20.79	57.72	6.54	0.21
7	21.84	5.51	0.50	0.02
8	23.10	13.48	0.71	0.05
9	26.31	16.67	0.73	0.06

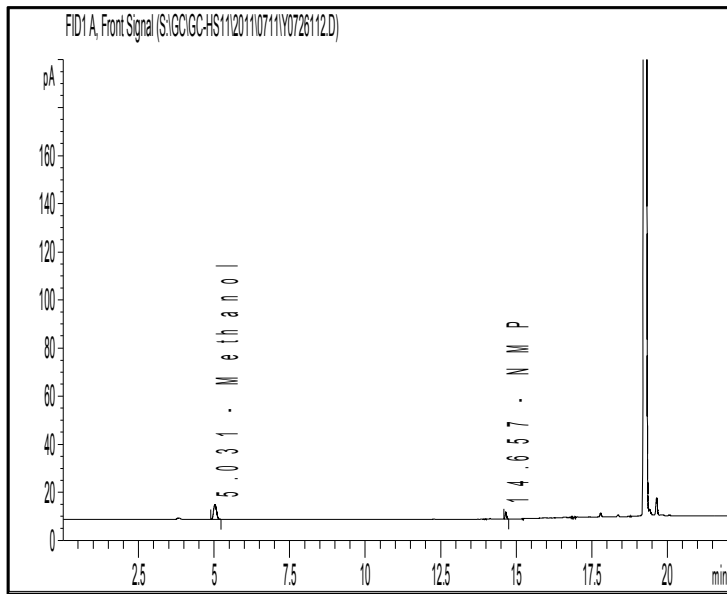
<sup>1</sup>H NMR

**Instrument:** JEOL ECS 400  
**Solvent:** Chloroform-D



*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:** S:\GC\GC-HS11\2011\0711\Y0726112.D  
**Instrument:** GC#11  
**Sample Name:** FN071811-03  
**Acquired:** July 26, 2011

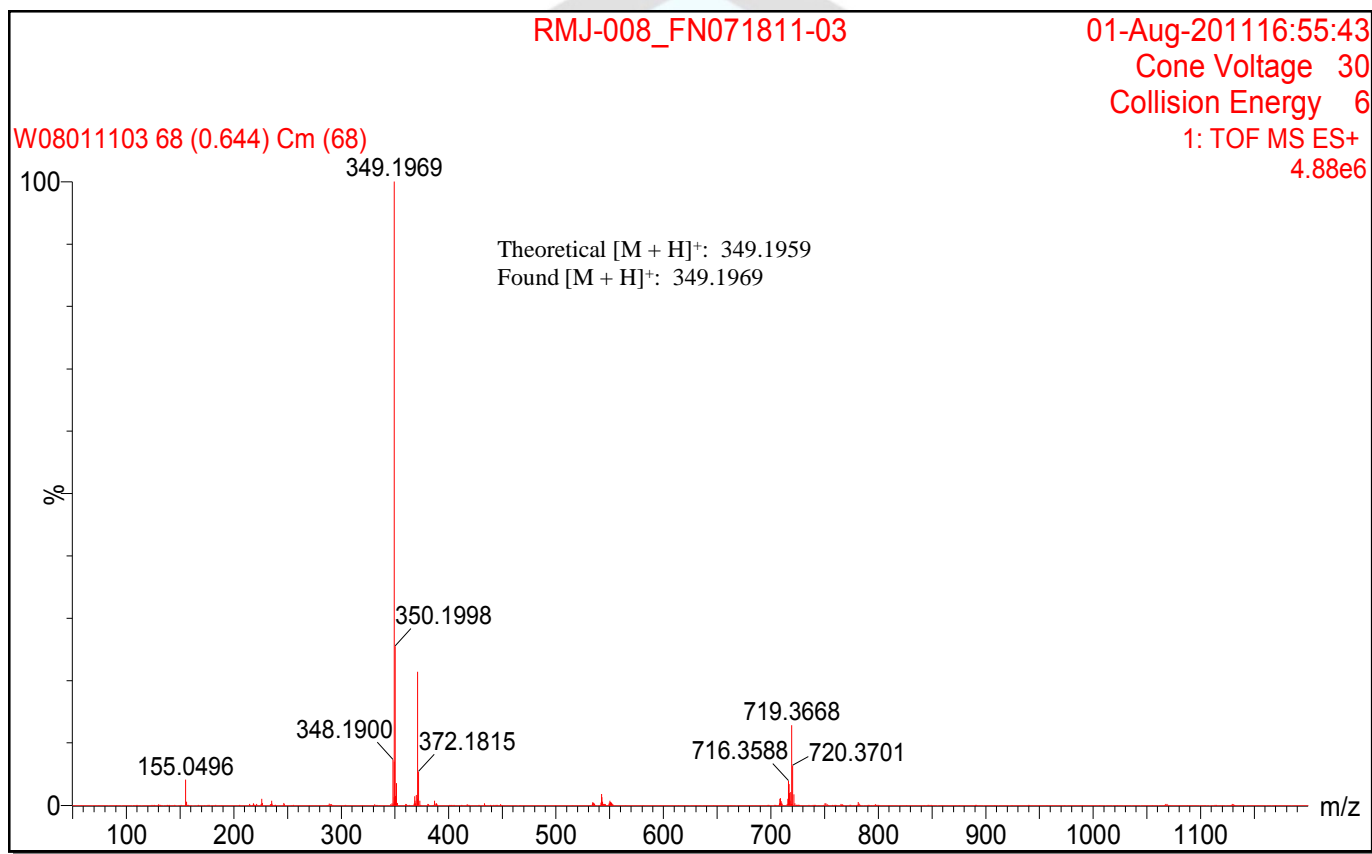
Peak	Compound	Area	Weight %
1	Methanol	42.08	0.34
2	NMP	NA	NA
<b>Total</b>			<b>0.34</b>

*Spectral and Physical Data (cont.)*

**LC/MS**

**Column:** Zorbax Eclipse Plus C18 RRHD 1.8 $\mu$ , 2.1 x 50 mm  
**Mobile Phase:** 0.1% Formic acid in Acetonitrile::0.1% Formic acid in Water (70::30)  
**Flow Rate:** 0.4 mL/min  
**Scan Range:** 50-1200 amu  
**Ionization:** Electrospray, Positive Ion

**Data File Name:** W08011103  
**Instrument:** Waters XEVO G2 QTOF  
**Sample Name:** FN071811-03  
**Acquired:** August 01, 2011

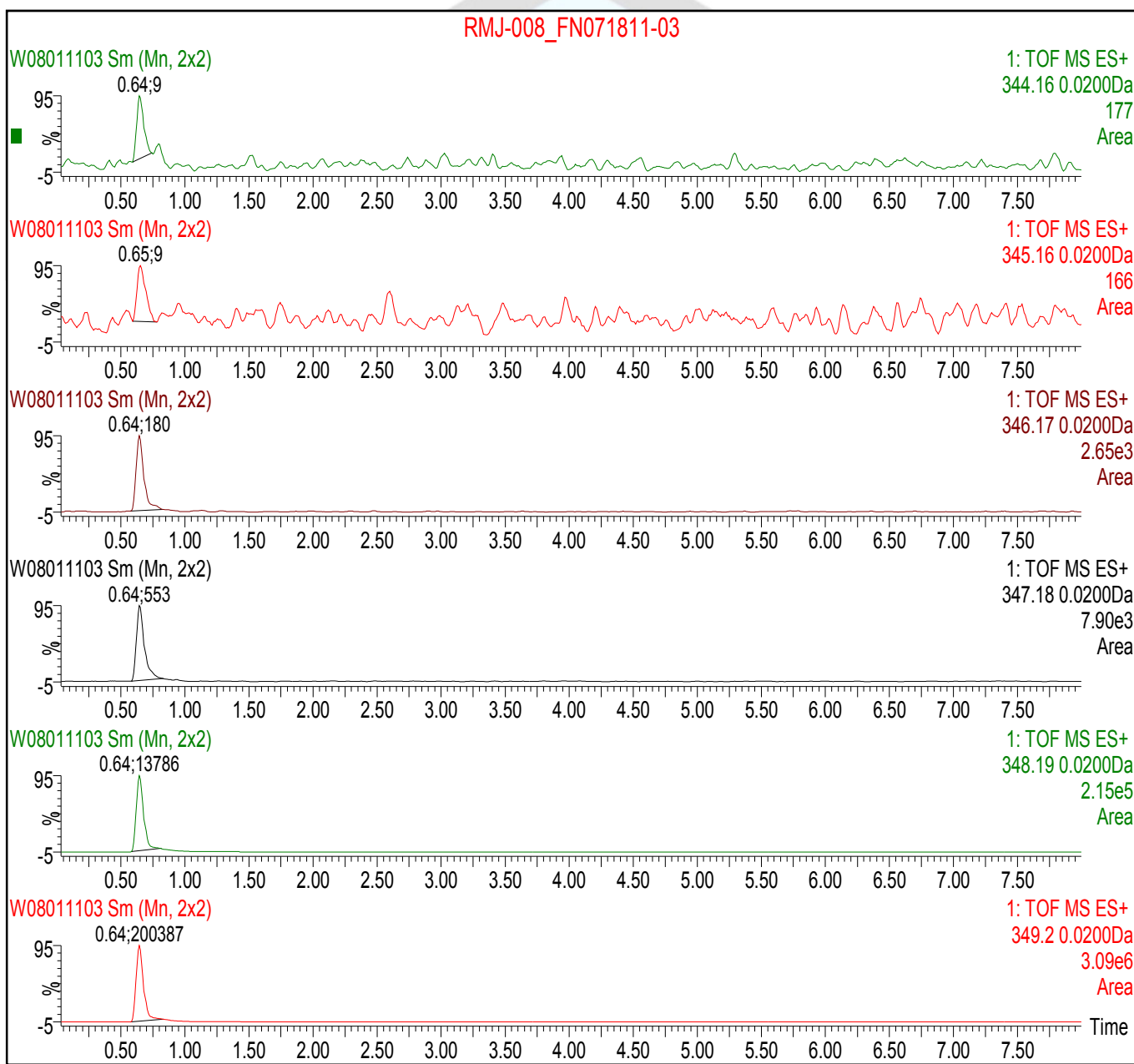


Spectral and Physical Data (cont.)

Isotopic Purity by LC/MS SIM Analysis

**Column:** Zorbax Eclipse Plus C18 RRHD 1.8 $\mu$ , 2.1 x 50 mm  
**Mobile Phase:** 0.1% Formic acid in Acetonitrile::0.1% Formic acid in Water (70::30)  
**Flow Rate:** 0.4 mL/min  
**Scan Range:** 344-349 amu  
**Ionization:** Electrospray, Positive Ion

**Data File Name:** W08011103  
**Instrument:** Waters XEVO G2 QTOF  
**Sample Name:** FN071811-03  
**Acquired:** August 01, 2011



## Stability

**Short Term Stability :** A summary of accelerated stability findings for a related product (S-037, JWH-073 N-(3-hydroxybutyl) metabolite) is listed below.

Storage Condition	Mean Kinetic Temperature (MKT)	Time Period
Freezer	-15°C	No decrease in purity was noted after four weeks.
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 of no more than 3 months at Refrigerate conditions.

**Long Term Stability:** Long term stability has been assessed for freezer storage (-10 °C to -25 °C) conditions. Stability of a minimum of 47 months has been established through real-time stability studies for a related product (S-037, JWH-073 N-(3-hydroxybutyl) metabolite).

## COA Revision History

Revision No.	Date	Reason for Revision
00	August 29, 2011	Initial version
01	September 22, 2011	Added Shipping conditions to page 1. Revised Stability Section to reference 4 week time period. Transport/Shipping: Replaced "at MKT not exceeding 40°C" with "ambient conditions". Added Short Term Storage to Stability Section.
02	September 26, 2011	Corrected "Expiration Date" to "Retest Date". Also replaced expiration date footnote on
03	July 26, 2012	Revised Retest Date from October 2012 to November 2013.
04	September 05, 2013	Revised Retest Date from November 2013 to October 2014.
05	July 29, 2014	Revised Retest date from October 2014 to September 2015. Added minimal sample size and MS application statements to front page.
06	May 11, 2015	Revised Retest Date from September 2015 to July 2016. Added 20.0 mins to HPLC/UV Gradient table. Updated Safety Section from "Flammable, Poison" to "Danger. See Safety Data Sheet"