

## BDG SYNTHESIS

### Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of research-grade materials. Research materials often contain higher levels of residual solvents and/or water, and we urge you to use the corrected purity where needed rather than the raw HPLC purity.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

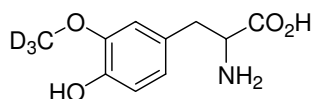
*Barry Dent*

Barry R. Dent, PhD, Director  
25 June 2007

**Name:** 3-(Methoxy-d<sub>3</sub>)-tyrosine

**CAS Number:** 586954-09-8

**Structure:**



**Molecular Weight:** C<sub>10</sub>H<sub>10</sub>D<sub>3</sub>NO<sub>4</sub> = 214.24

**Lot Number:** BDG 6751.7

**Appearance:** Off-white, crystalline solid

**Corrected Purity:** 99.3 % (HPLC) – 0.2 % (methanol) – 8.5 % (water) = 90.6 %

**Isotopic Purity:** Under 0.5 % d<sub>0</sub>

**Expiry Date:** 25 June 2012

This expiry date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage stability studies because of the small amount of material available.

**Storage and Handling:**

Temperature: ambient laboratory temperature; may be refrigerated.

Humidity: not believed to be hygroscopic; may be handled in normal laboratory atmosphere.

Light: protect from strong sunlight.

Caution: Only experienced laboratory personnel should handle the material.

## Identity and Purity:

### Source of Material

The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

### Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. The spectrum is recorded in NaOD/D<sub>2</sub>O and hence represents the spectrum of the derived sodium carboxylate salt.

Isotopic labelling: signals at the site of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual solvents: a small amount of methanol (0.2 % w/w) is observed.

Impurities: no significant impurities are evident in the spectrum.

### Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Isotopic labelling: the signal at the site of deuteration has collapsed into a small multiplet compared with what would be expected for unlabelled material, indicating clean deuteration.

**High-resolution mass spectrum (ESI+):** found  $m/z$  215.1113. C<sub>10</sub>H<sub>11</sub>D<sub>3</sub>NO<sub>4</sub> [M+H]<sup>+</sup> requires  $m/z$  215.1106. The deviation of 3.5 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d<sub>0</sub> material was seen (detection limit about 0.5 %).

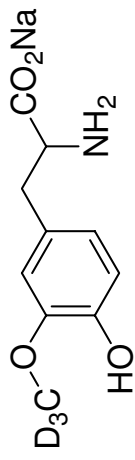
**HPLC:** A sharp, symmetrical peak is observed (99.3 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

<b>Elemental Analysis:</b>	Found:	C 51.25, H 5.43, D 2.67, N 6.00 %
C <sub>10</sub> H <sub>10</sub> D <sub>3</sub> NO <sub>4</sub> •1.1 H <sub>2</sub> O	requires:	C 51.31, H 5.25, D 2.58, N 5.98 %, H <sub>2</sub> O 8.5 %
C <sub>10</sub> H <sub>10</sub> D <sub>3</sub> NO <sub>4</sub>	requires:	C 56.06, H 4.70, D 2.82, N 6.54 %

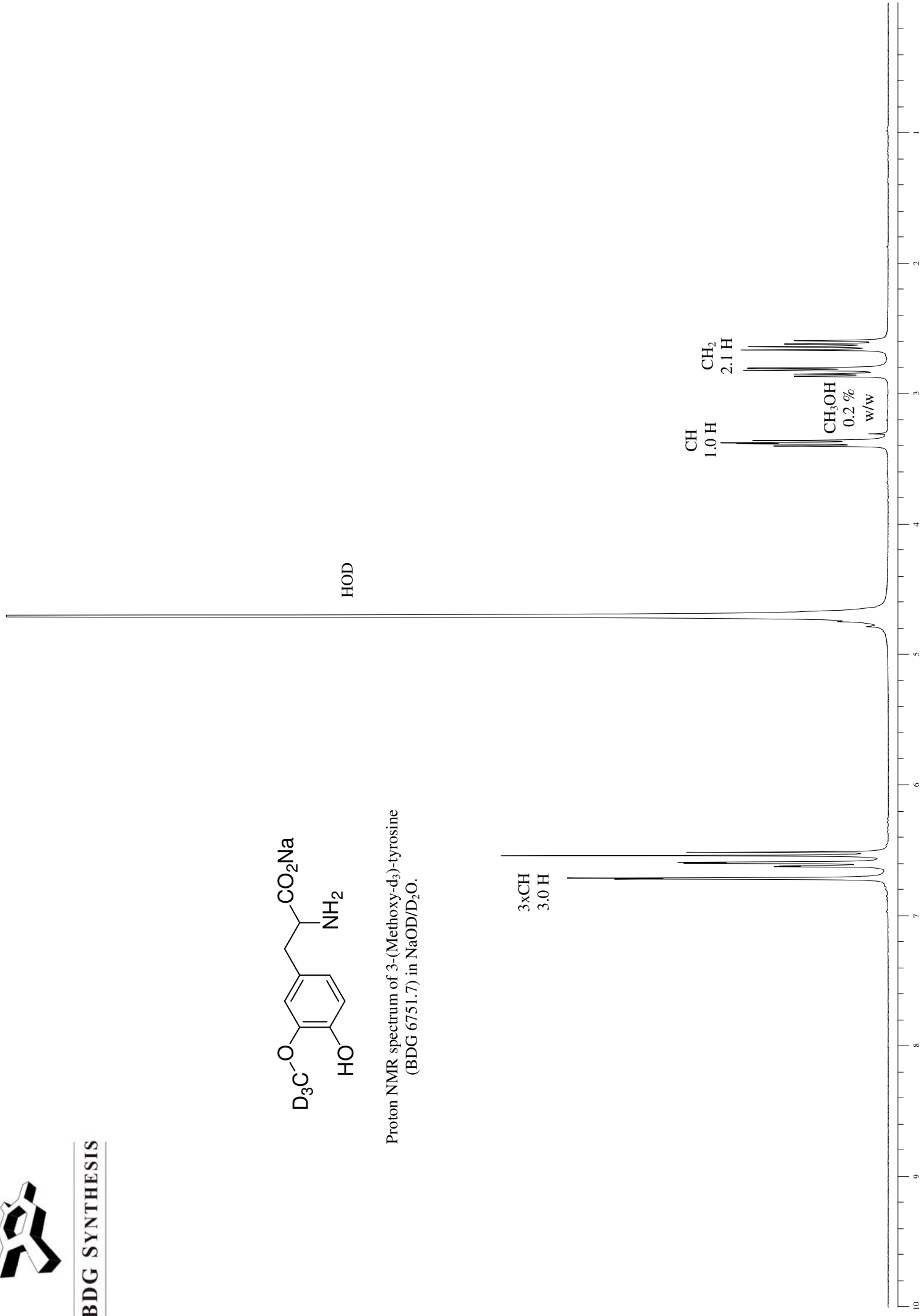
The elemental analyses fall substantially outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the “best-fit” water content be used when determining corrected purity.



**BDG SYNTHESIS**

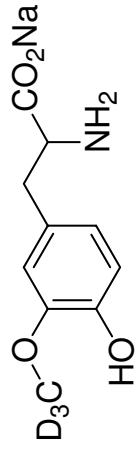


Proton NMR spectrum of 3-(Methoxy-d<sub>3</sub>)-tyrosine (BDG 6751.7) in NaOD/D<sub>2</sub>O.

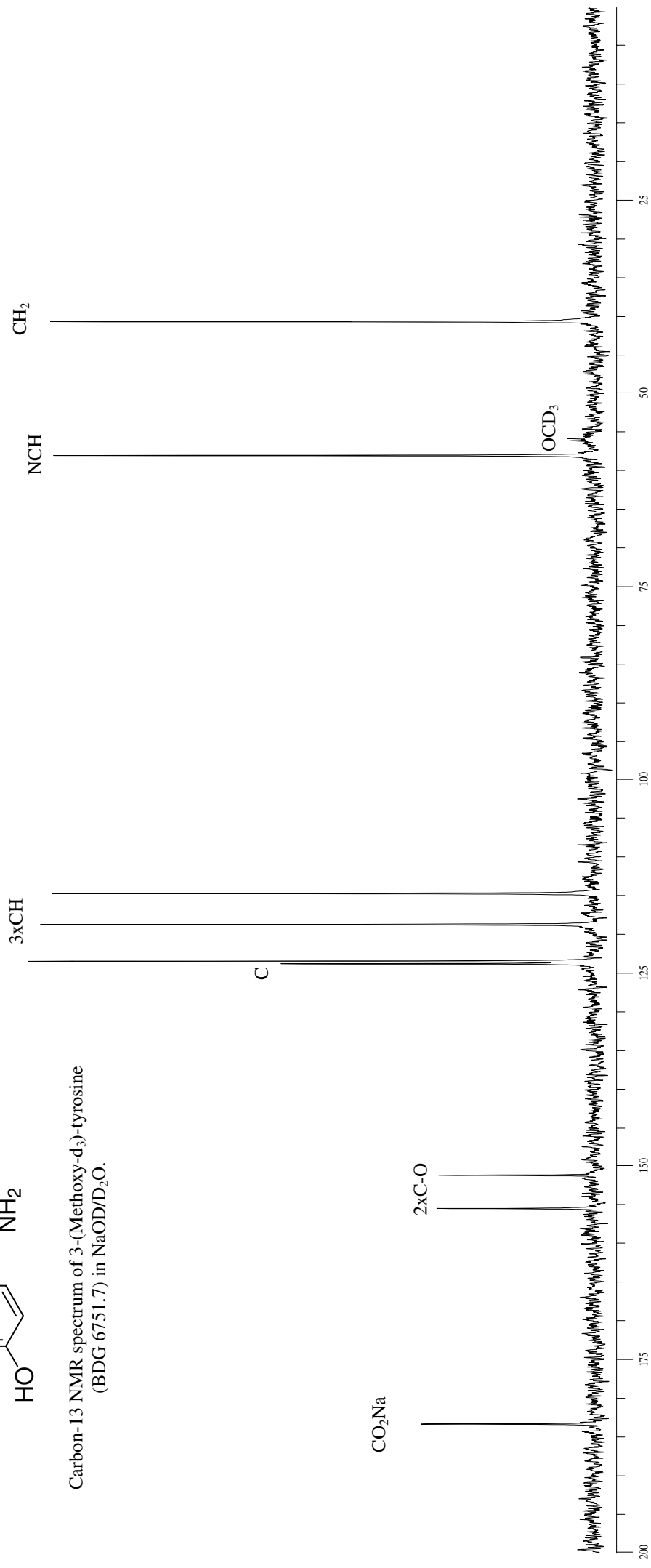




## BDG SYNTHESIS



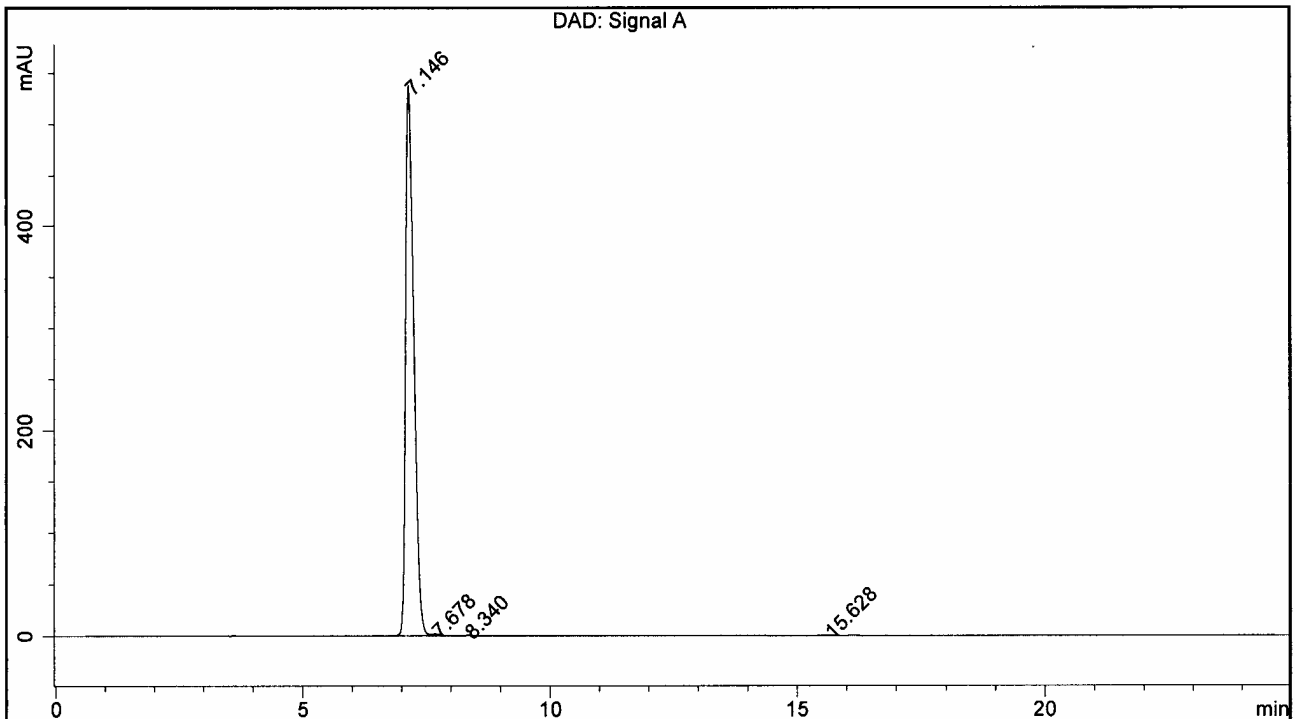
Carbon-13 NMR spectrum of 3-(Methoxy-d<sub>3</sub>)-tyrosine (BDG 6751.7) in NaOD/D<sub>2</sub>O.



BDG - Analysis of 3-(Methoxy-d3)-Tyrosine

Column : Phenomenex Luna C18(2) 5 um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase A : Water + 0.1% Trifluoroacetic Acid  
 Mobile Phase B : Acetonitrile + 0.1% Trifluoroacetic Acid  
 Gradient : T0=90:10, T20=80:20, T22=90:10, T25=90:10  
 Flow Rate : 1.0 mL/min  
 Sample Solvent : 80:20 A:B  
 Column Temperature : 20C  
 Injection Volume : 10 uL  
 Detection : UV at 280 nm

Sample Name	BDG 6751.7	Instrument	AnalyticalLC01
Acquisition	18/06/2007, 15:54:45	Method (rev.)	LC10147a ( 16)
Sequence	BDG_18Jun2007b	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.15 min	537.9921	6217.1443	0.1788 min	99.336 %
2	7.68 min	1.7253	29.0307	0.2343 min	0.464 %
3	8.34 min	0.5203	6.6275	0.1886 min	0.106 %
4	15.63 min	0.4151	5.8775	0.2232 min	0.094 %