



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.

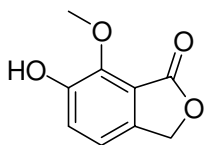
Barry Dent

Barry R. Dent, PhD, Director
15 February 2006

Name: 6-Desmethylmeconine

CAS Number: 78213-30-6

Structure:



Molecular Weight: $C_9H_8O_4 = 180.16$

Lot Number: BDG 6318.4

Appearance: Off-white, crystalline solid

Corrected Purity: 99.1% (HPLC) – 0.7% (ethyl acetate) = 98.4%

Expiry Date: 15 February 2007

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

Storage and Handling:

Temperature: ambient laboratory temperature; may be refrigerated.

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

Light: store in an amber vial and protect from bright light.

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. Residual solvents: a small amount of ethyl acetate (0.7 % 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.

High-resolution mass spectrum (ESI+): found m/z 383.0746. $(C_9H_8O_4)_2Na$ $[2M+Na]^+$ requires m/z 383.0743. The deviation of 0.8 ppm is within normally accepted limits for the establishment of identity by HRMS.

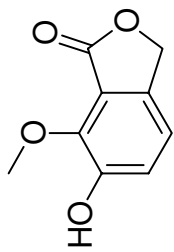
HPLC: A sharp, symmetrical peak is observed (99.1 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 60.09, H 4.51 %
C₉H₈O₄ requires: C 60.00, H 4.48 %

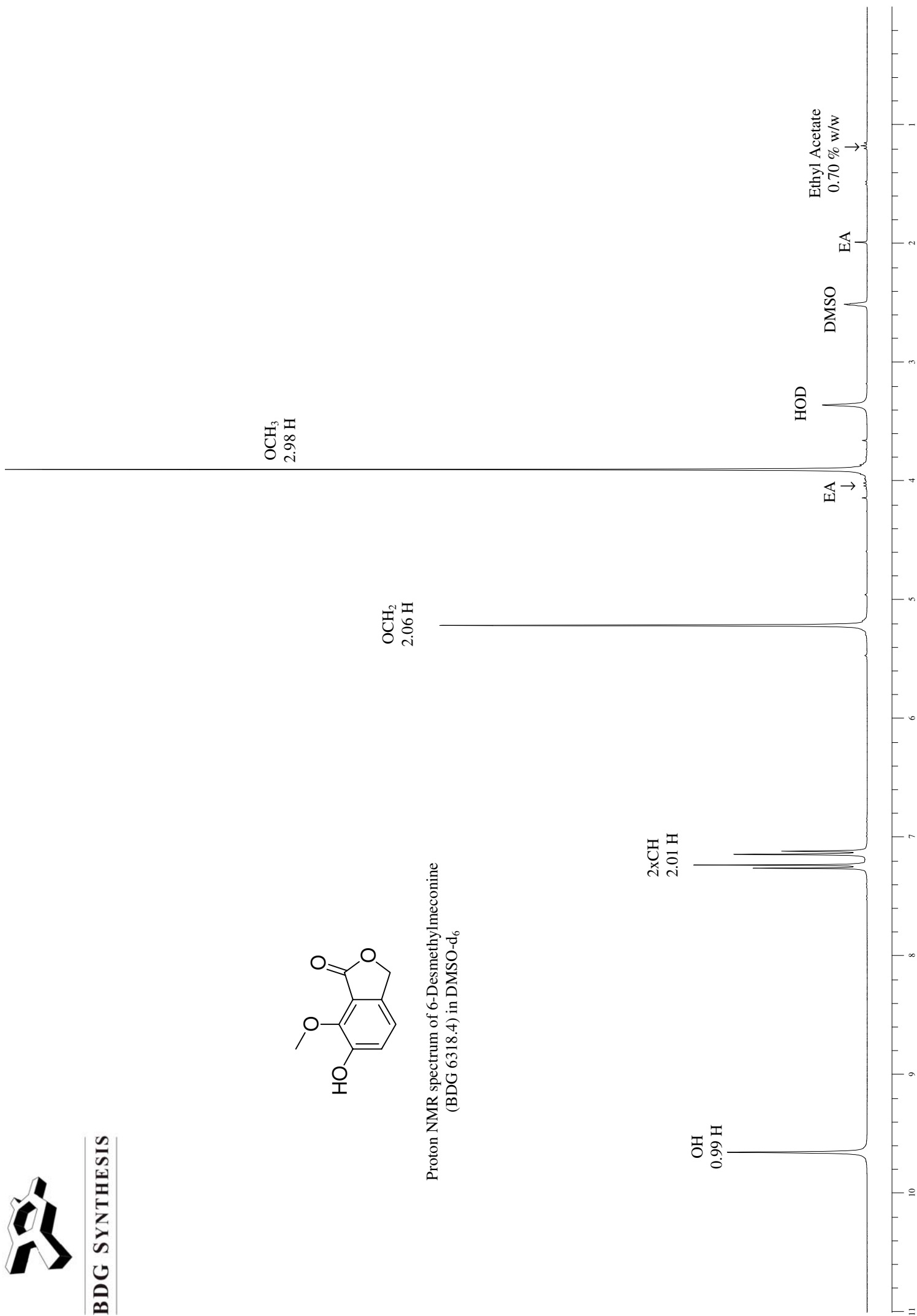
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



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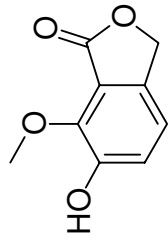


Proton NMR spectrum of 6-Desmethylmeconine (BDG 6318.4) in DMSO-d₆

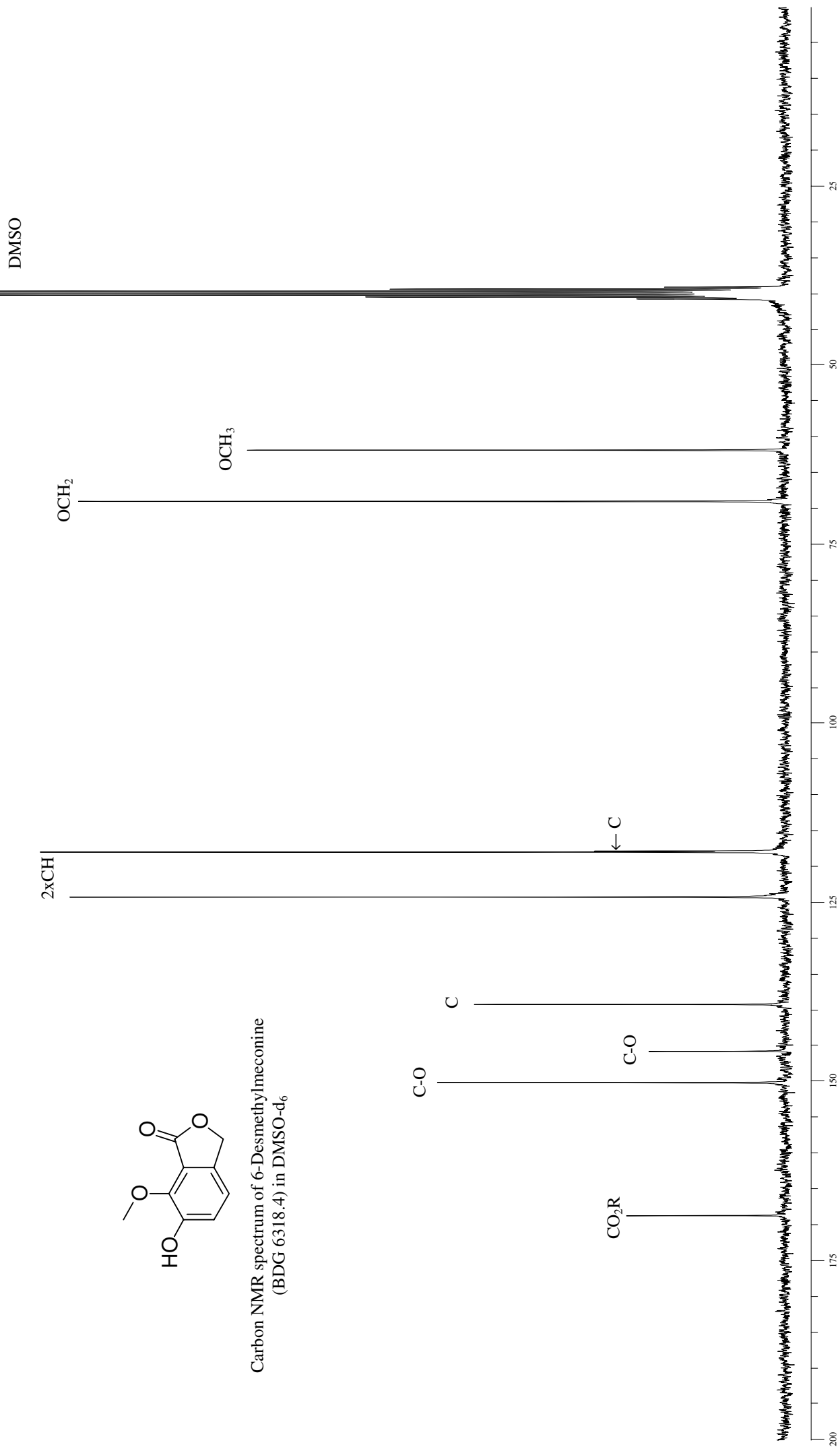




BDG SYNTHESIS



Carbon NMR spectrum of 6-Desmethylimeconine
(BDG 6318.4) in DMSO-d₆



Sample single injection

Description:

BDG - Analysis of 6-Desmethylmeconine

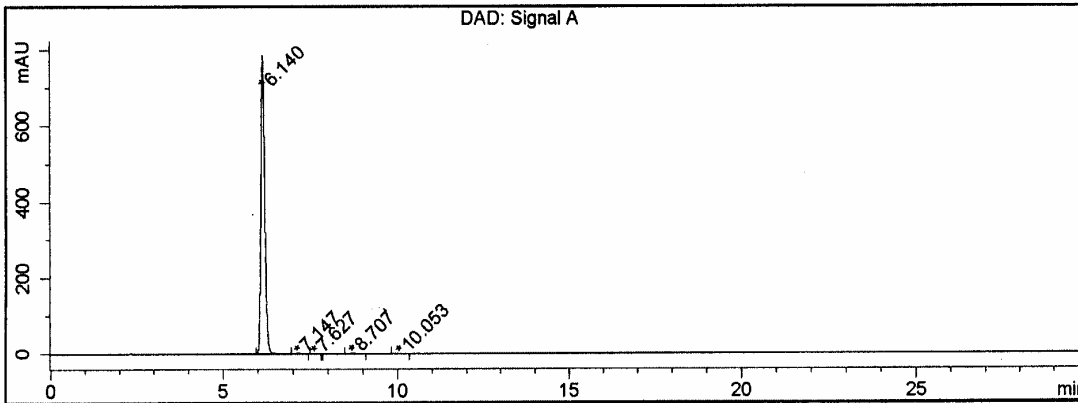
Column : Phenomenex Luna C18, 5um 250 x 4.6mm
 Guard : Phenomenex Security Guard C18 4 x 3
 Mobile Phase : 30/70 Acetonitrile/ Water
 Flow Rate : 1 mL/min
 Sample Solvent : Mobile Phase
 Column Temp : 30C
 Injection Volume : 10 ul
 Detection : UV at 220 nm

Sample identification

Sample name		BDG 6318.4
Sample type		Sample
Date	Start	15-Feb-06, 13:11:04
	End	15-Feb-06, 13:11:04
Instrument		Analytical LC 01
Acquisition		10-Feb-06, 13:01:01
Injection		1 of 1
Run Revision		6

Sample method description

Method (rev.)	LC10014 (3)	Calibration created	N/A
Sample scheduler	solvation010\cerityadmin	Calibration modified	N/A
Instrument	Analytical LC 01	Instrument rev.	2



Results Table :

RT	Peak Type and Separation Code	Peak Area	Peak Height	Peak Width	Time Start	Time End	Symmetry
6.14	MM m	6086.3729	786.4102	0.1290	5.96	7.86	0.727
7.15	MM x	18.8381	2.1491	0.1461	6.96	7.43	0.806
7.63	MM t	1.8258	0.2249	0.1353	7.48	7.81	0.767
8.71	MM m	28.8977	2.7499	0.1751	8.50	9.08	0.744
10.05	MM m	4.9988	0.3127	0.2664	9.82	10.34	0.893

Summary

Peak Retention (minutes)	Area % (220 nm)
6.14	99.11
7.15	0.31
7.63	0.03
8.71	0.47
10.05	0.08