



BDG SYNTHESIS

Certificate of Analysis

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

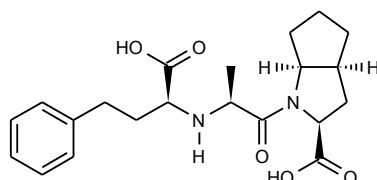
Barry Dent

Barry R. Dent, PhD, Director
20 February 2013

Name: Ramiprilat

CAS Number: 87269-97-4

Structure:



Molecular Weight: $C_{21}H_{28}N_2O_5 = 388.46$

Lot Number: BDG 2655.4

Appearance: White powder

Corrected Purity: 99.6 % (HPLC) - 1.5 % (water) = 98.1 %

Re-test Date: 20 February 2018

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
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

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. The complexity of the spectrum indicates two conformers of the product are present in solution.

Residual Solvents: no residual solvents are 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. The majority of signals are duplicated indicating that two conformers of the product are present in solution.

High-resolution Mass Spectrum (FAB+)

Found m/z 389.2074. $C_{21}H_{29}N_2O_5$ $[M+H]^+$ requires m/z 389.2076. The deviation of 0.7 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A somewhat broadened, symmetrical peak is observed (99.6 %). The small peak at 4 minutes and the broader peak at 22 minutes are excluded from the integration analysis as they are present in the blank run (solvent only). 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 63.16, H 7.18, N 7.03 %
$C_{21}H_{28}N_2O_5 \cdot 0.5H_2O$	Requires:	C 63.46, H 7.35, N 7.05 %
$C_{21}H_{28}N_2O_5$	Requires:	C 64.93, H 7.27, N 7.21 %

The elemental analyses fall somewhat 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.

Karl-Fischer Analysis

	Found:	H_2O 1.5 %
$C_{21}H_{28}N_2O_5 \cdot 0.5H_2O$	Requires:	H_2O 2.3 %

Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

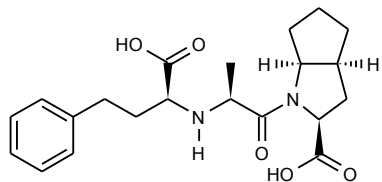
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

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

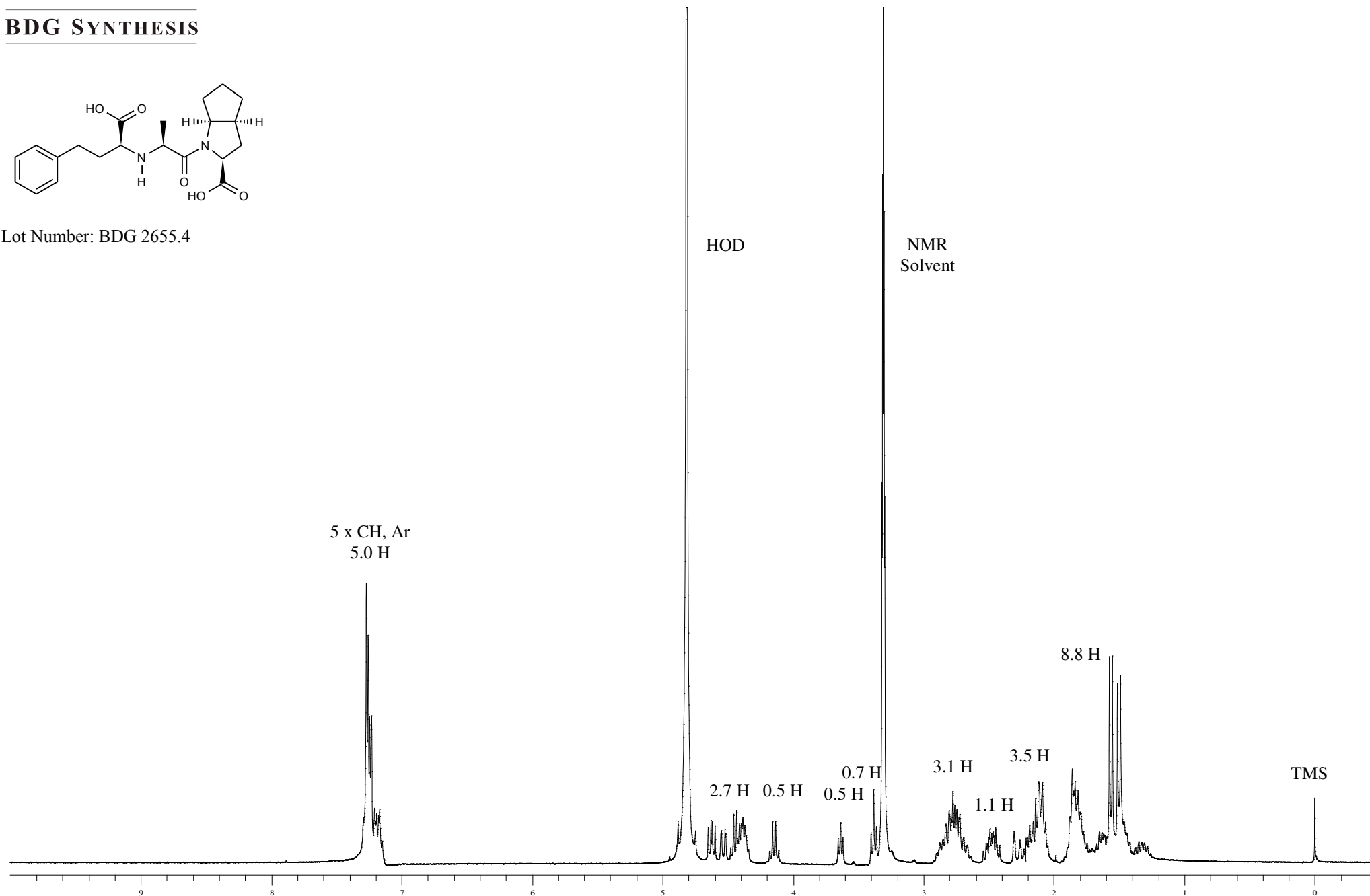


Proton NMR Spectrum of Ramiprilat in Methanol-d₄

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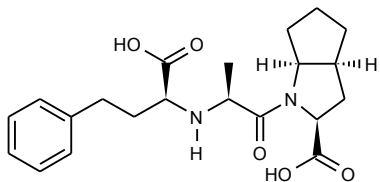
Lot Number: BDG 2655.4



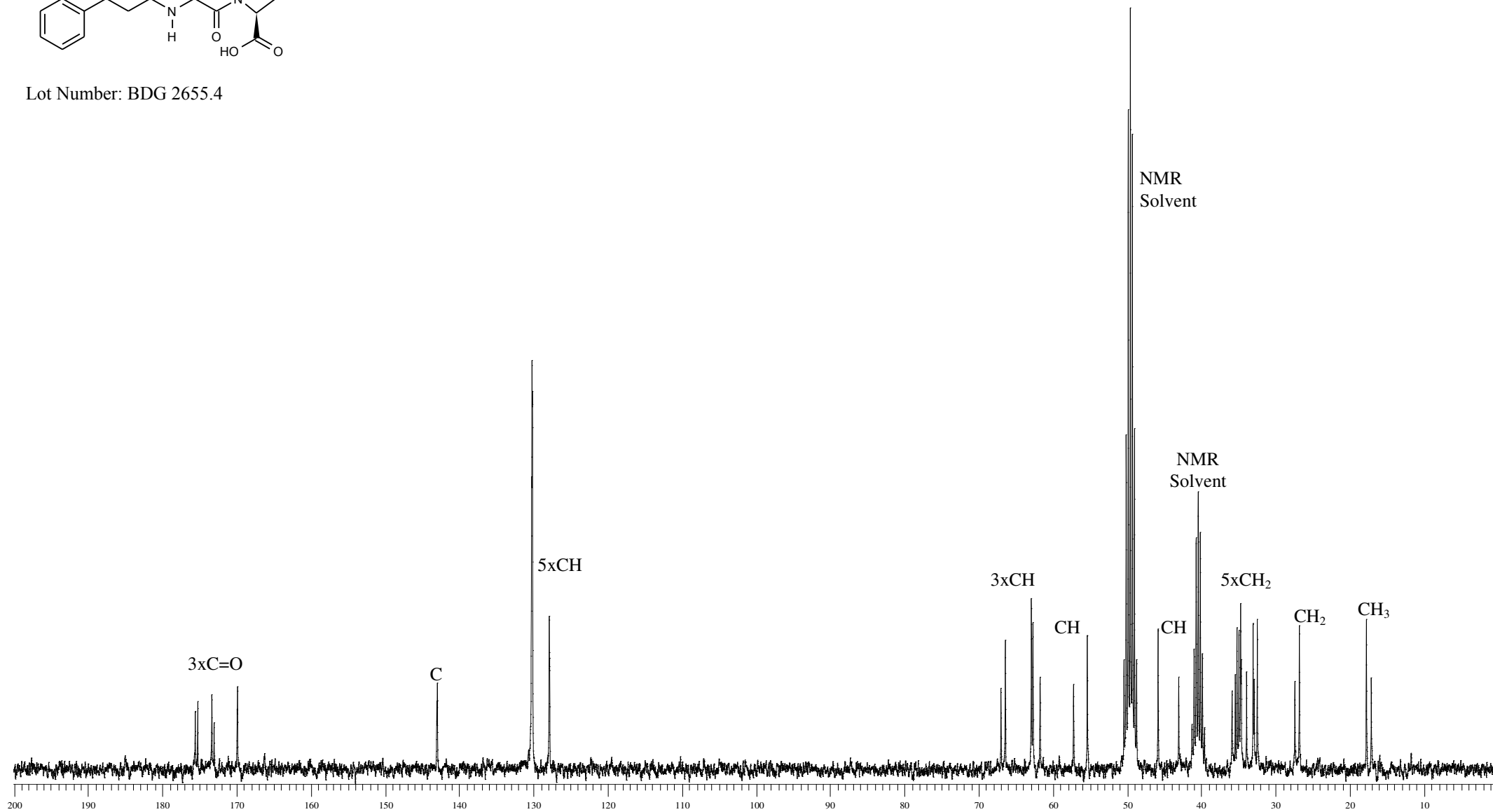


Carbon-13 NMR Spectrum of Ramiprilat in Methanol-d₄ and DMSO-d₆

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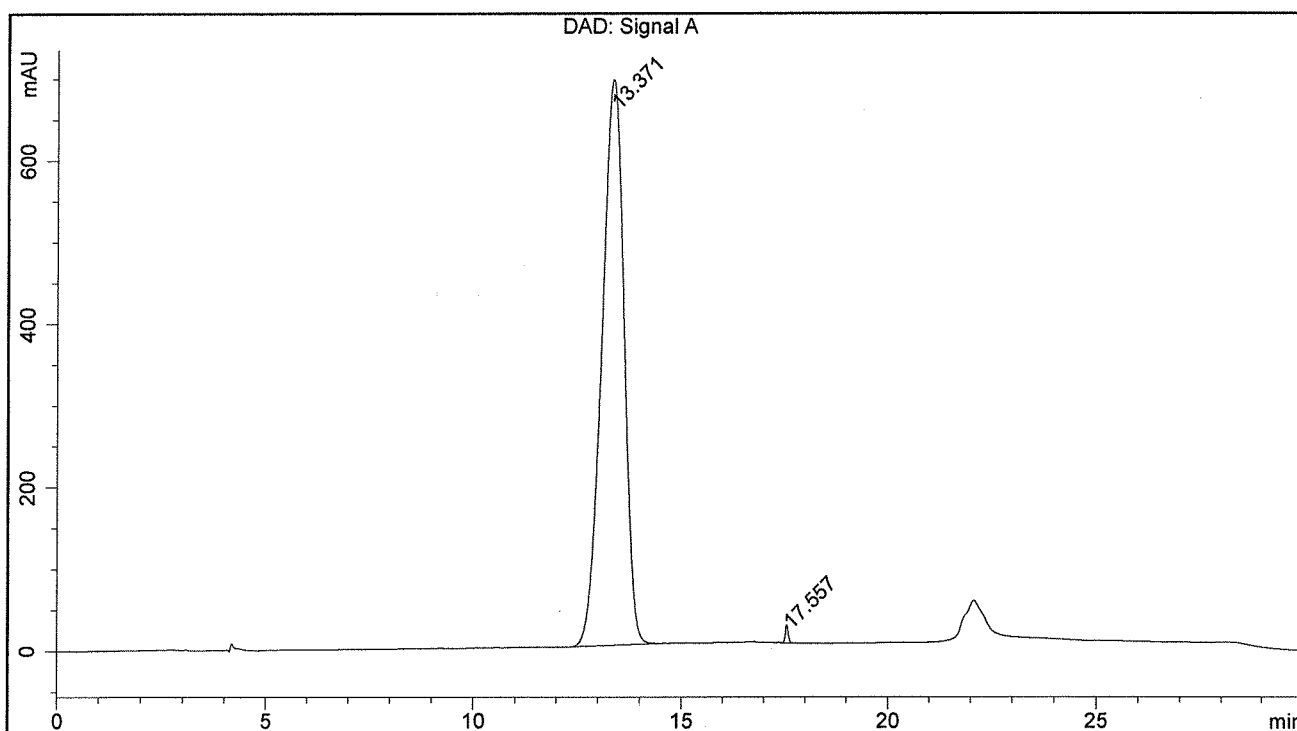
Lot Number: BDG 2655.4



BDG - Analysis of Ramiprilat

Column : Phenomenex Luna C18 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 4 x 3 mm
 Mobile Phase : 80:20 0.2% Sodium Perchlorate 0.05% Triethylamine pH=3.6 : Acetonitrile
 Mobile Phase : 30:70 0.2% Sodium Perchlorate 0.05% Triethylamine pH=2.6 : Acetonitrile
 Gradient (A:B) : T0=100:0, T8=90:10, T20=0:100, T25=0:100, T27=100:0, T30=100:0
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 60C
 Injection Volume : 10 uL
 Detection : UV at 210 nm

Sample Name	BDG 2655.4	Instrument	AnalyticalLC01
Acquisition	20/02/2013, 20:26:34	Method (rev.)	LC10241c (5)
Sequence	BDG_20Feb2013b - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	13.37 min	691.6542	25485.3923	0.5779 min	99.572 %
2	17.56 min	22.3033	109.4312	0.0752 min	0.428 %