



BDG SYNTHESIS

Certificate of Analysis

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

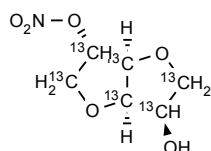
Neil Beare

Neil Beare, PhD, Director
3 September 2015

Name: Isosorbide-¹³C₆ 2-Nitrate

CAS Number: 16106-20-0 (unlabelled)

Structure:



Molecular Weight: ¹³C₆H₉NO₆ = 197.09

Lot Number: BDG 17195.2

Appearance: Off-white, crystalline solid

Corrected Purity: 98.7 % (HPLC) - 0.9 % (ethyl acetate) = 97.8 %

Isotopic Purity: Under 0.5% M-6

Re-test Date: 3 September 2020

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.

Isotopic Labelling: the spectrum is of little value in determining isotopic purity. All proton signals are split by ^{13}C - ^1H coupling as expected.

Residual Solvents: a small amount of ethyl acetate (0.9 % w/w) is observed.

Impurities: traces of unidentified impurities are seen in the baseline.

Carbon-13 NMR Spectrum

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

Isotopic Labelling: the spectrum is of little use in determining isotopic purity although all signals are massively enhanced and contain ^{13}C - ^{13}C coupling as expected.

Impurities: traces of unidentified impurities are seen in the baseline.

High-resolution Mass Spectrum (ESI+)

Found m/z 220.0534. $^{13}\text{C}_6\text{H}_9\text{NNaO}_6$ $[\text{M}+\text{Na}]^+$ requires m/z 220.0529. The deviation of 2.3 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for M-6 material was seen (detection limit about 0.5 %).

HPLC

A sharp, symmetrical peak is observed (98.7 %). 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:	H 4.56, N 7.10, C 39.85 %
$^{13}\text{C}_6\text{H}_9\text{NO}_6$	Requires:	H 4.60, N 7.11, C 39.59 %

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).

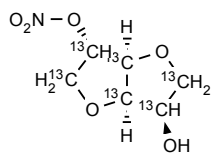
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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.

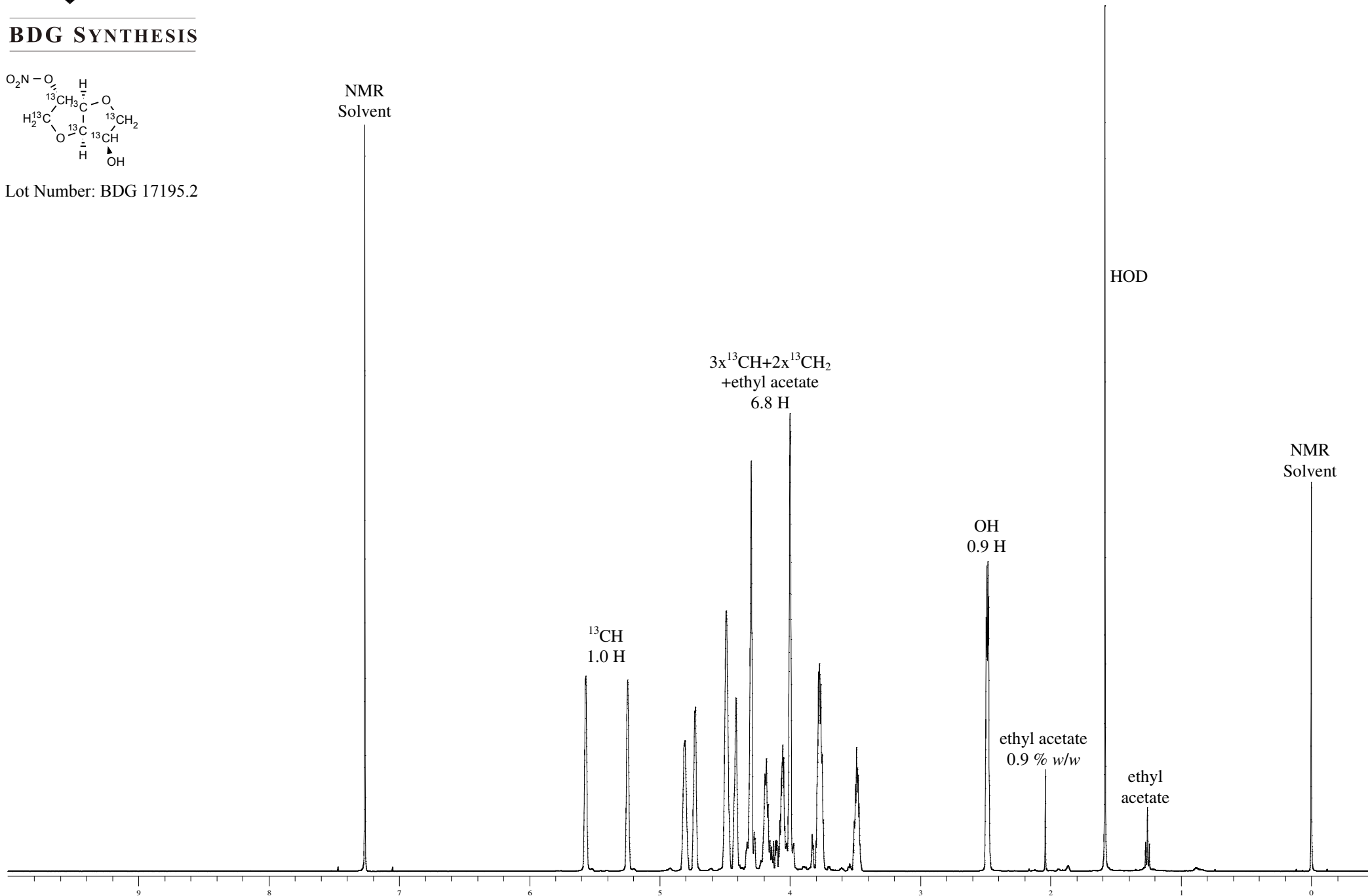


Proton NMR Spectrum of Isosorbide-¹³C₆ 2-Nitrate in CDCl₃

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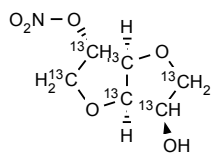
Lot Number: BDG 17195.2



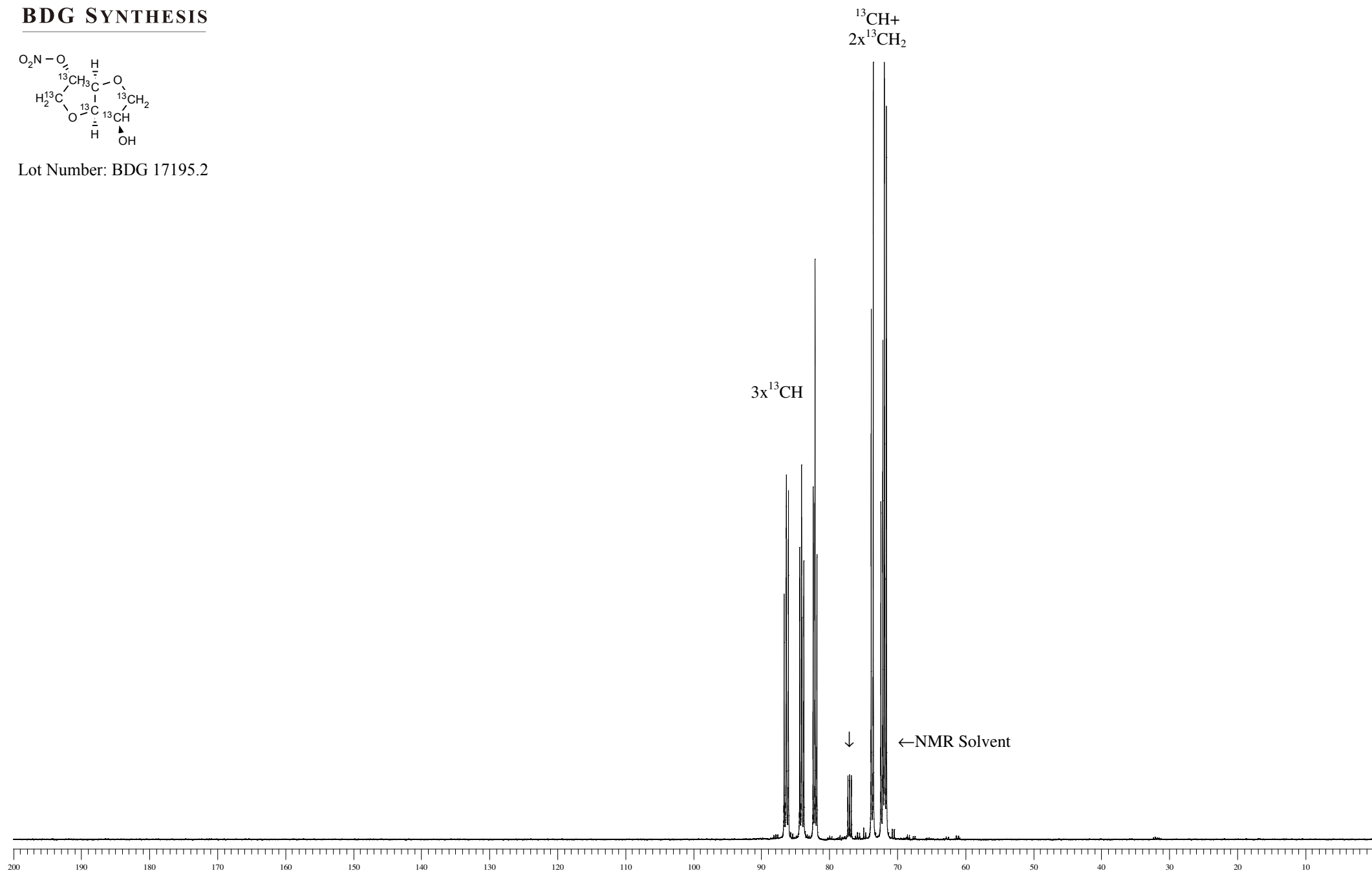


Carbon-13 NMR Spectrum of Isosorbide-¹³C₆ 2-Nitrate in CDCl₃

BDG SYNTHESIS



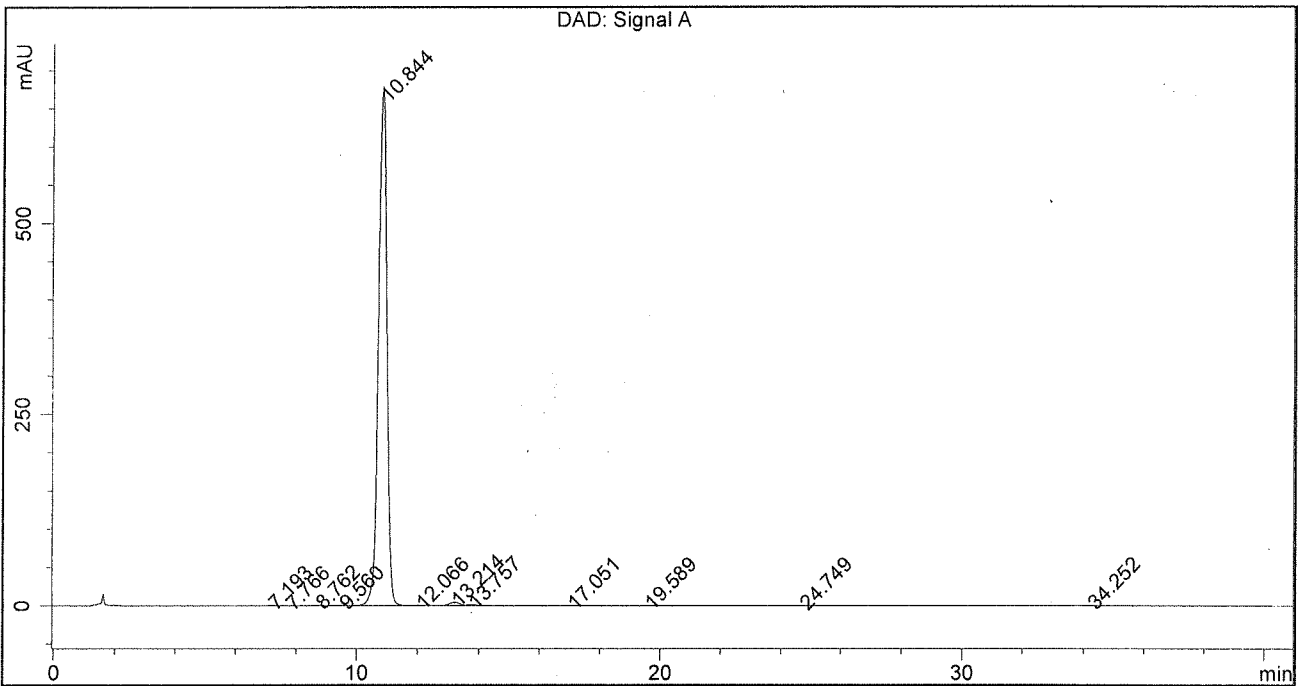
Lot Number: BDG 17195.2



BDG - Analysis of isosorbide-13C6 2-nitrate

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 90:10 10 mM Ammonia solution : Acetonitrile
 Flow Rate : 1 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 215 nm

Sample Name	BDG 17195.2	Instrument	AnalyticalLC01
Acquisition	03/09/2015, 19:10:24	Method (rev.)	LC10075c (5)
Sequence	BDG_03Sep2015d - Reprocessed	Vial Position	2
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	7.19 min	0.2582	4.2097	0.2142 min	0.032 %
2	7.77 min	0.1517	1.9788	0.1641 min	0.015 %
3	8.76 min	0.6210	9.4599	0.2417 min	0.072 %
4	9.56 min	0.3846	6.0943	0.2089 min	0.046 %
5	10.84 min	677.4978	12948.5081	0.3030 min	98.740 %
6	12.07 min	0.1762	1.9961	0.1442 min	0.015 %
7	13.21 min	4.1997	96.8211	0.3570 min	0.738 %
8	13.76 min	1.1968	26.4931	0.3463 min	0.202 %
9	17.05 min	0.1649	4.3577	0.3282 min	0.033 %
10	19.59 min	0.1207	3.8753	0.3906 min	0.030 %
11	24.75 min	0.1662	7.9548	0.5824 min	0.061 %
12	34.25 min	0.1057	1.9480	0.2310 min	0.015 %