

## BDG SYNTHESIS

### Certificate of Analysis

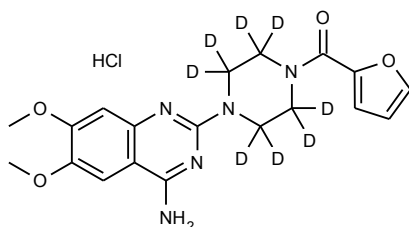
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

*Neil Beare*

Neil Beare, PhD, Director  
15 July 2019

**Name:** Prazosin-d<sub>8</sub> HCl  
**CAS Number:** 19237-84-4 (unlabelled)

**Structure:**



**Molecular Weight:** C<sub>19</sub>H<sub>13</sub>D<sub>8</sub>N<sub>5</sub>O<sub>4</sub>·HCl = 427.91  
**Lot Number:** BDG 17527.4  
**Appearance:** Off-white, crystalline solid  
**Corrected Purity:** 99.3 % (HPLC) - 2.3 % (methanol) - 2.1 % (water) = 94.9 %  
**Isotopic Purity:** Under 0.5 % d<sub>0</sub>  
**Re-test Date:** 15 July 2024  
**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: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of methanol (2.3 % 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: signals at the site of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

### High-resolution Mass Spectrum (TOF MS ES+)

Found  $m/z$  392.2180.  $C_{19}H_{14}D_8N_5O_4$   $[M+H]^+$  requires  $m/z$  392.2174. The deviation of 1.5 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for  $d_0$  material was seen (detection limit about 0.5 %).

### HPLC

A sharp, symmetrical peak is observed (99.3 %). 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 52.02, H 3.28, D 3.69 %
$C_{19}H_{13}D_8N_5O_4 \cdot HCl \cdot 0.5H_2O$	Requires:	C 52.23, H 3.46, D 3.69 %, $H_2O$ 2.06 %
$C_{19}H_{13}D_8N_5O_4 \cdot HCl$	Requires:	C 53.33, H 3.30, D 3.77 %

The elemental analyses fall slightly 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.

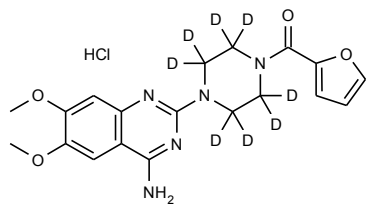
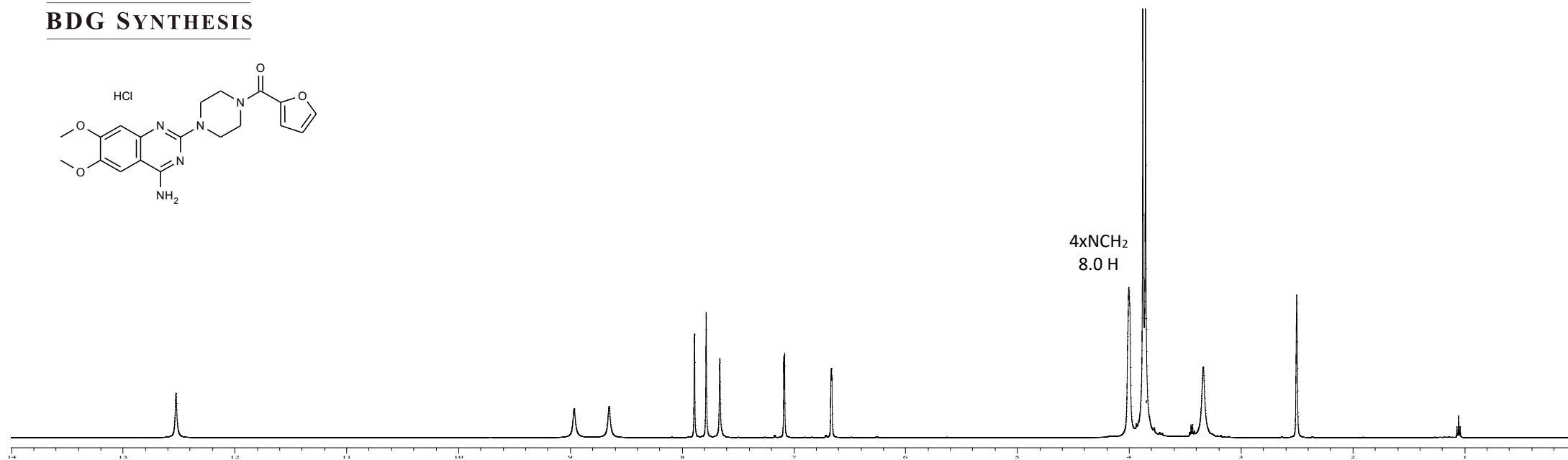
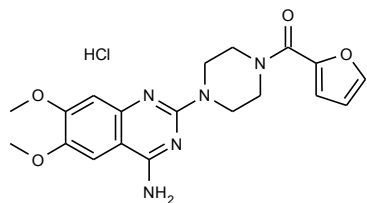
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

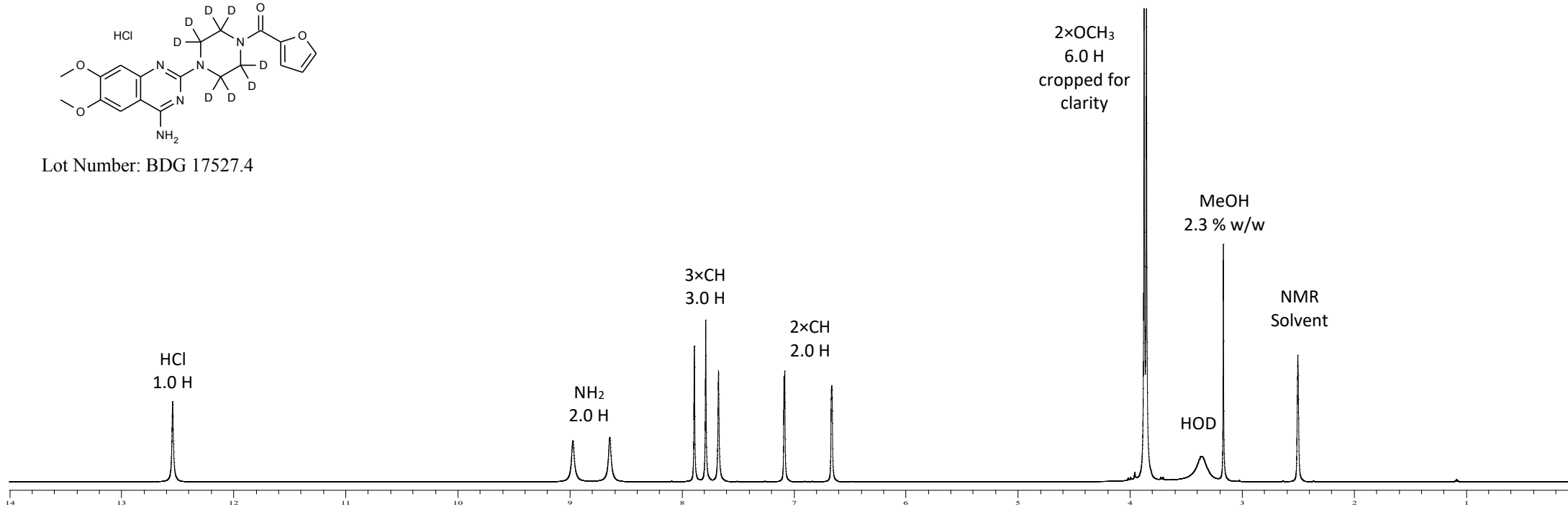


# Proton NMR Spectrum of Prazosin HCl (top) and Prazosin-d<sub>8</sub> HCl (bottom) in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



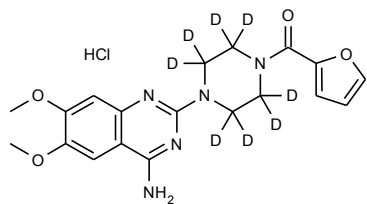
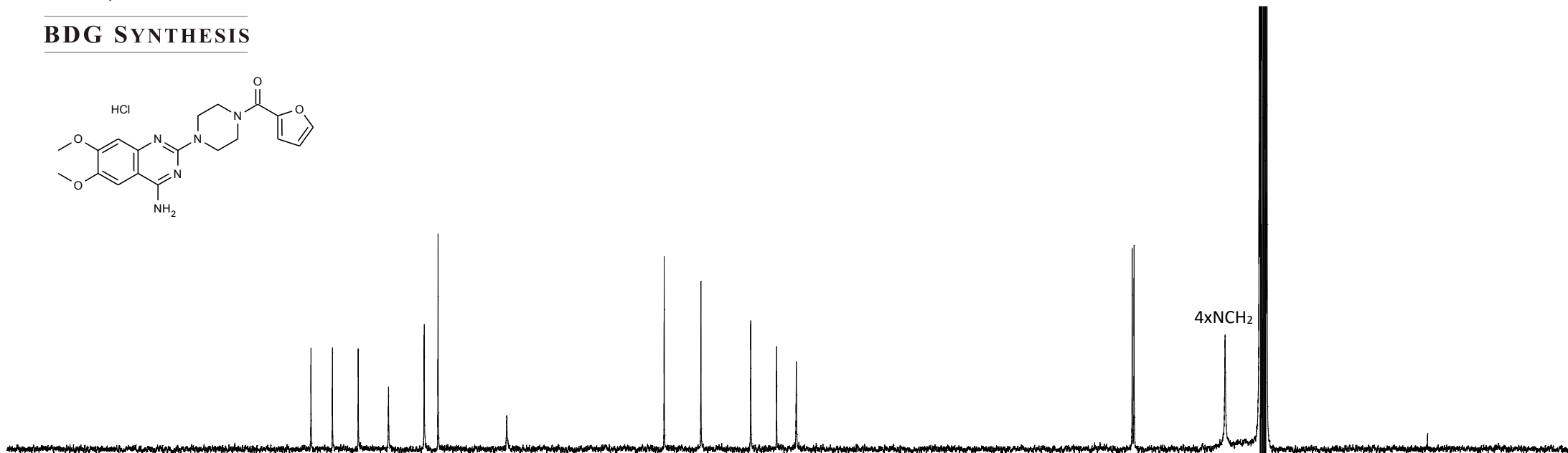
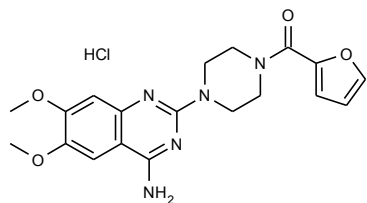
Lot Number: BDG 17527.4



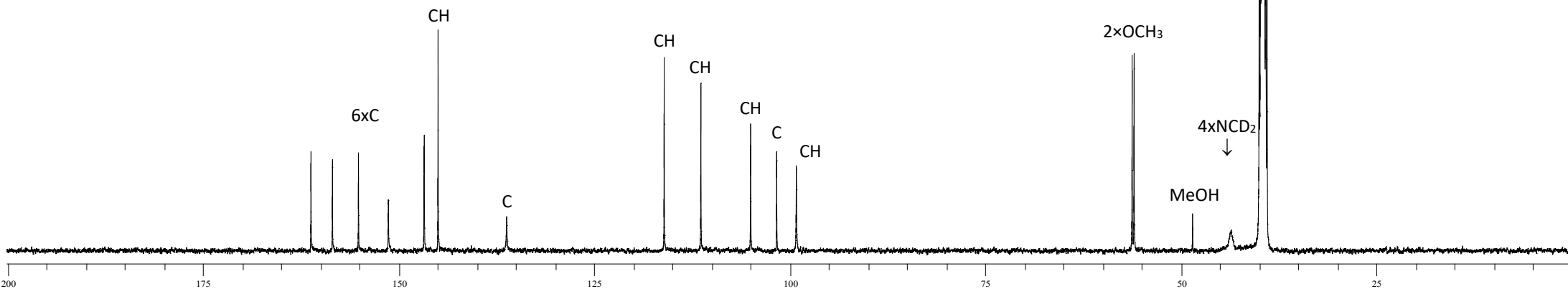


Carbon-13 NMR Spectrum of Prazosin HCl (top) and Prazosin-d<sub>8</sub> HCl (bottom) in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



Lot Number: BDG 17527.4



Sample Name: BDG 175277.4  
Injection Date : 7/15/2019 8:36:11 PM

Inj : 1

Inj Volume : 10 µl

Sequence File : C:\CHEM32\1\SEQUENCE\BDG\_15JUL2019C.S

Acq. Method : C:\CHEM32\1\METHODS\2018\LC20107A.M

Last changed : 7/15/2019 6:58:36 PM by Bruce Hamilton

Analysis Method : C:\CHEM32\1\METHODS\2018\LC20107A.M

Last changed : 7/16/2019 10:13:42 AM by Bruce Hamilton

Method Info : BDG - Analysis of Prazosin-d8 HCl

Column : Phenomenex Luna C18(2) 5 µm 250 x 4.6 mm

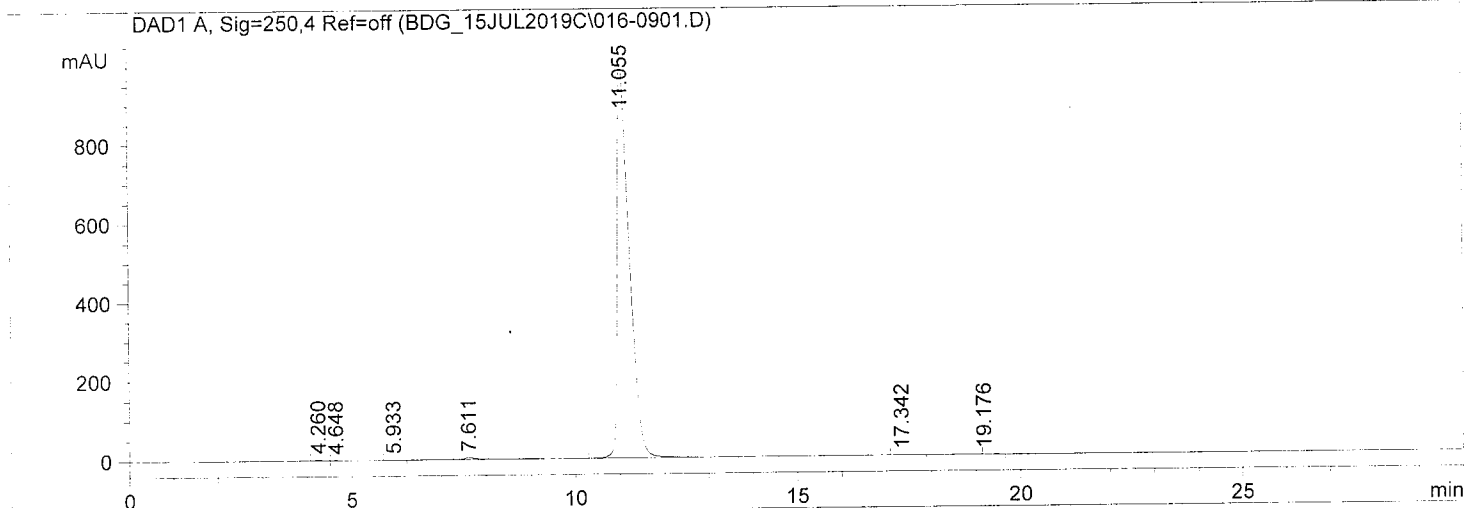
Guard : Phenomenex SecurityGuard C18 4 x 3 mm

Mobile Phase A : 80:20 20 mM KH<sub>2</sub>PO<sub>4</sub> pH=3.00 : Acetonitrile

Mobile Phase B : Acetonitrile

Sample Solvent : 70:30 Water : Acetonitrile, Detection : UV 250 nm,

Flow : 1 ml/min., Column Temperature : 30 C, Injection : 10 µl.



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Area Percent Report  
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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.260	BV R	0.0994	17.82019	2.75705	0.0942
2	4.648	VB	0.1270	5.00226	5.89749e-1	0.0265
3	5.933	BV R	0.1424	3.25446	3.63834e-1	0.0172
4	7.611	BV R	0.3046	96.64516	4.46292	0.5111
5	11.055	BV R	0.2789	1.87799e4	1010.24652	99.3201
6	17.342	BB	0.2438	2.98941	1.49146e-1	0.0158
7	19.176	VB	0.2305	2.85033	1.49426e-1	0.0151

Totals : 1.89085e4 1018.71864

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\*\*\* End of Report \*\*\*