



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.

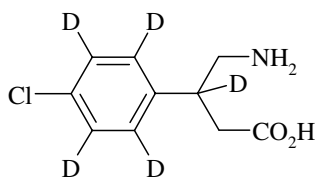
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director
10 March 2006

Name: Baclofen-d₅
CAS Number: none (1134-47-0 unlabelled)

Structure:



Molecular Weight: C₁₀H₇D₅ClNO₂ = 218.69
Lot Number: BDG 7025.0
Appearance: White, crystalline solid
Purity by HPLC: 97.7 %
Isotopic Purity: Under 0.5 % d₀
Expiry Date: 10 March 2011
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: 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.

Isotopic labelling: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

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.

Isotopic labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (ESI+): found m/z 219.0947. $C_{10}H_8D_5ClNO_2$ $[M+H]^+$ requires m/z 219.0943. The deviation of 2.0 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 %).

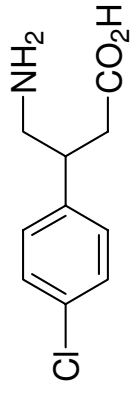
HPLC: A sharp, symmetrical peak is observed (97.7 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 54.81, H 3.16, D 4.52, N 6.30 %
 $C_{10}H_7D_5ClNO_2$ requires: C 54.92, H 3.22, D 4.60, N 6.40 %

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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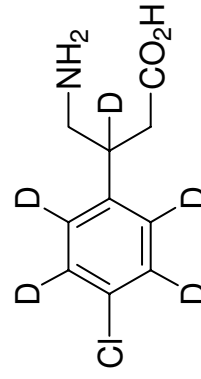
Proton NMR spectrum of Baclofen
(BDG 7025.3) in D₂O.

4xCH
3.83 H

CH₂+CH
3.01 H

CH₂
2.00 H

NMR
Solvent



Proton NMR spectrum of Baclofen-d₅
(BDG 7025.0) in D₂O.

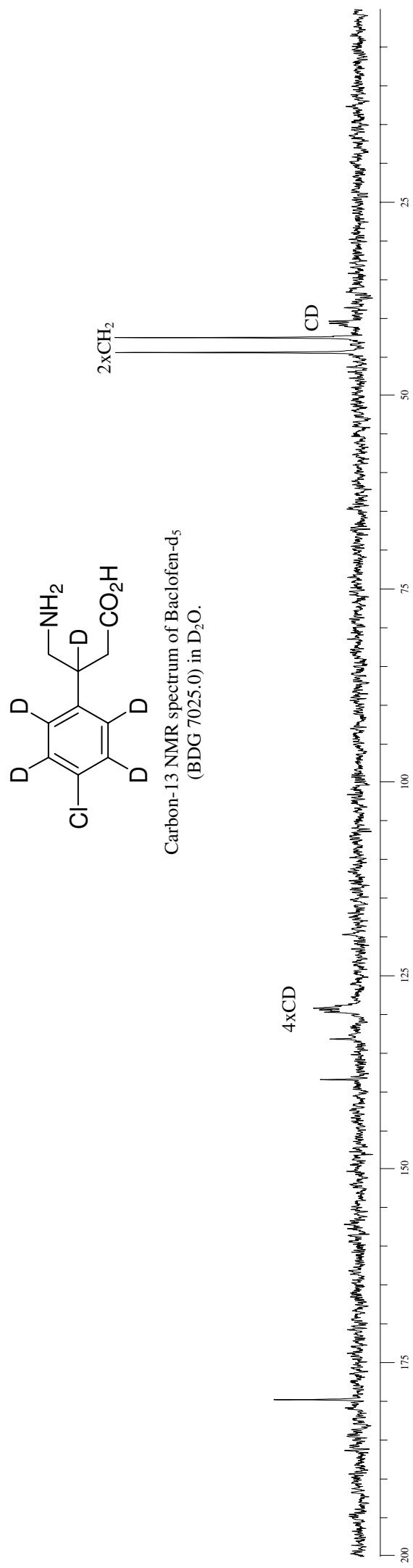
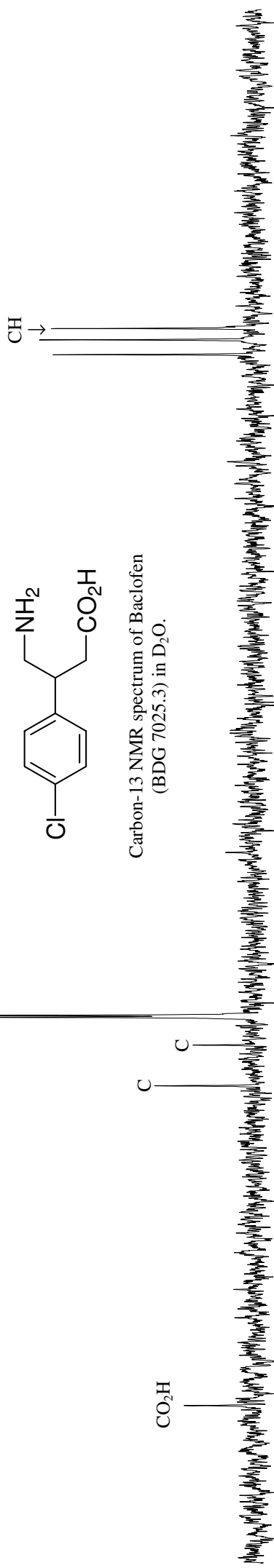
CH₂
2.01 H

CH₂
2.00 H





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BDG - Analysis of Baclofen-d5

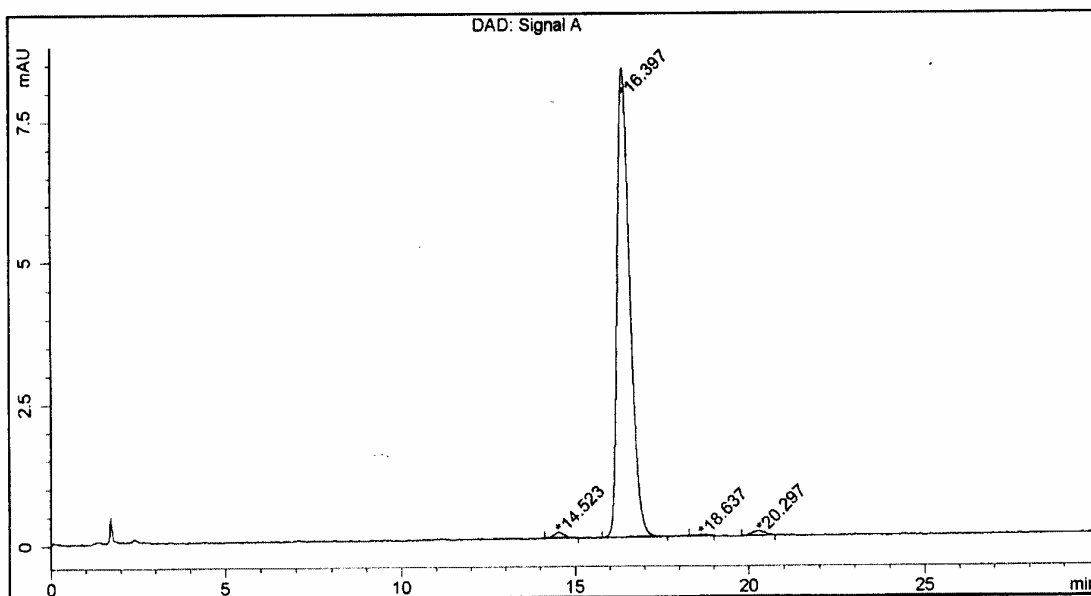
Column : Phenomenex Luna C18, 5um 250 x 4.6mm
 Guard : Phenomenex Security Guard C18 4 x 3
 Mobile Phase : 0.01M sodium heptane sulphonate
 in 56:44:5 H2O:MeOH:HOAc
 Flow Rate : 1.5 mL/min
 Sample Solvent : Initial Mobile Phase
 Column Temp : 25 C
 Injection Volume : 10 ul
 Detection : UV at 266 nm

Sample identification

Sample name	BDG 7025.0	
Sample type	Sample	
Date	Start	09-Mar-06, 10:05:22
	End	09-Mar-06, 10:05:22
Instrument	Analytical LC 01	
Acquisition	08-Mar-06, 11:44:33	
Injection	1 of 1	
Run Revision	5	

Sample method description

Method (rev.)	LC10021q (9)	Calibration created	23-Feb-06, 08:57:22
Sample scheduler	solvation010\cerityadmin	Calibration modified	02-Mar-06, 10:41:43
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
14.52	MM m	2.2136	0.1039	0.3552	14.12	15.10	0.738
16.40	MM m	210.3214	8.3050	0.4221	15.77	17.66	0.637
18.64	MM m	0.5237	0.0246	0.3551	18.29	19.00	0.888
20.30	MM m	2.1262	0.0827	0.4282	19.80	20.74	1.167

Summary

Peak Retention (minutes)	Area % (266 nm)
14.52	1.03
16.40	97.74
18.64	0.24
20.30	0.99