



## BDG SYNTHESIS

### Certificate of Analysis

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

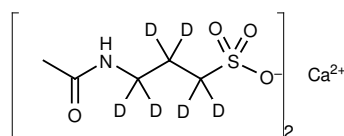
Barry Dent

Barry R. Dent, PhD, Director  
23 June 2006

**Name:** Acamprosate-d<sub>6</sub> Calcium Salt

**CAS Number:** 77337-73-6 (unlabelled)

**Structure:**



**Molecular Weight:**  $2C_5H_4D_6NO_4S \cdot Ca = 412.56$

**Lot Number:** BDG 8096

**Appearance:** White, crystalline solid

**Corrected Purity:** 90.0 % (NMR)

**Isotopic Purity:** Under 0.5 % d<sub>0</sub>

**Re-test Date:** 23 June 2011

**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

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

Purity Determination: a corrected chemical purity was determined by a quantitative proton NMR experiment using taurine as an internal standard, and was measured at 90 % *w/w*.

### 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* 186.0718.  $C_5H_4D_6NO_4S [M]^-$  requires *m/z* 186.0702. The deviation of 9.0 ppm is somewhat outside normally accepted limits for the establishment of identity by HRMS, and the mass spectral data should be considered in conjunction with other identity criteria. No signal for  $d_0$  material was seen (detection limit about 0.5 %).

### HPLC

Because of the lack of chromophores in the material, a useful chromatogram was unable to be recorded.

### Elemental Analysis

	Found:	C 28.96, H 1.99, D 5.98, N 6.71 %
$2C_5H_4D_6NO_4S \cdot Ca$	Requires:	C 29.11, H 1.95, D 5.86, N 6.79 %

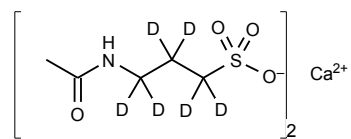
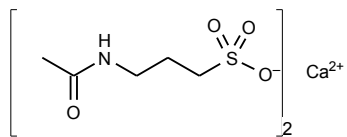
The elemental analyses fall within generally accepted limits for establishing the molecular formula given.

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.

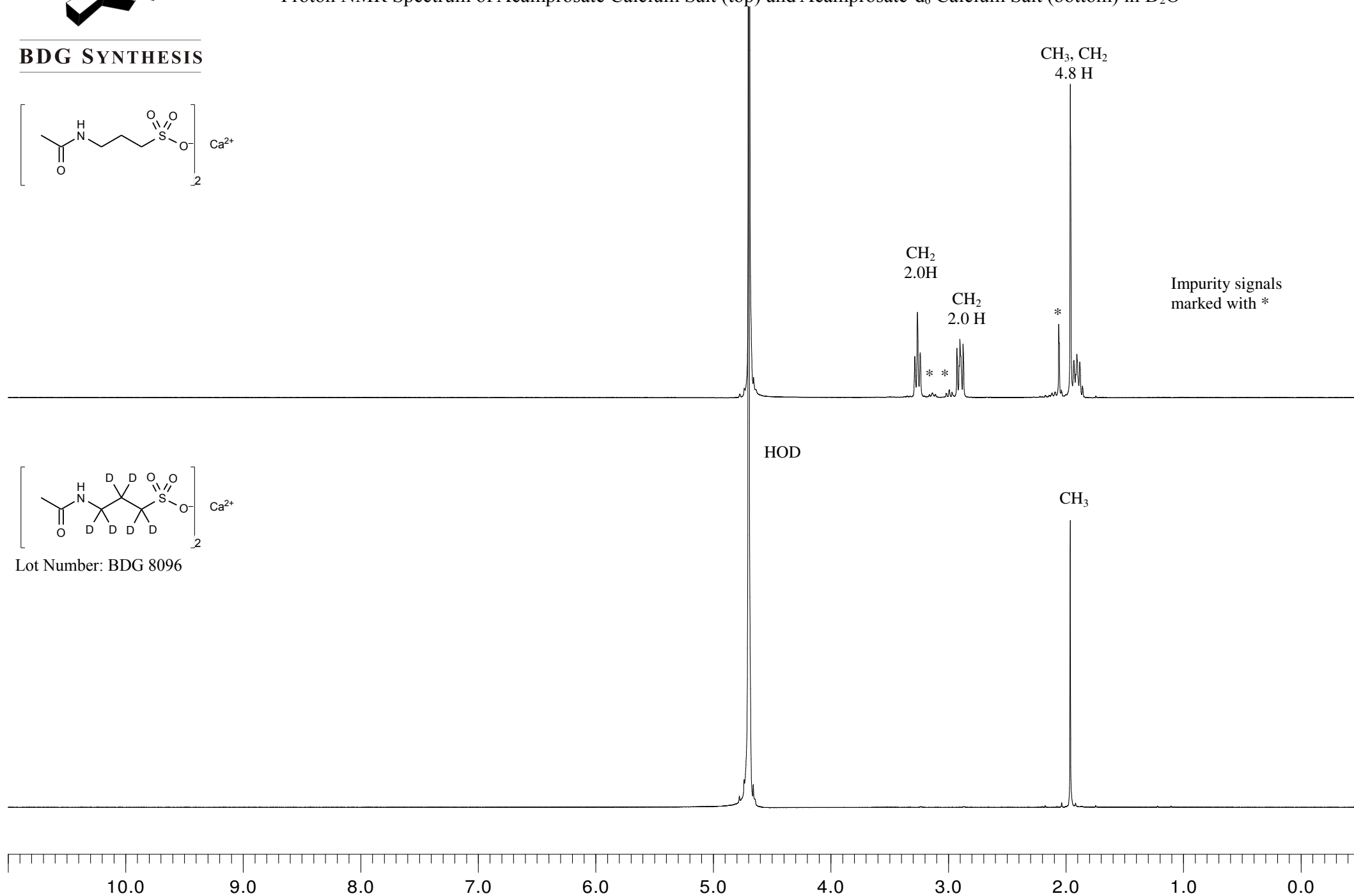


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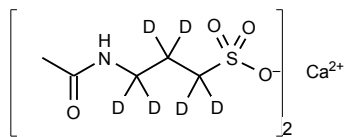
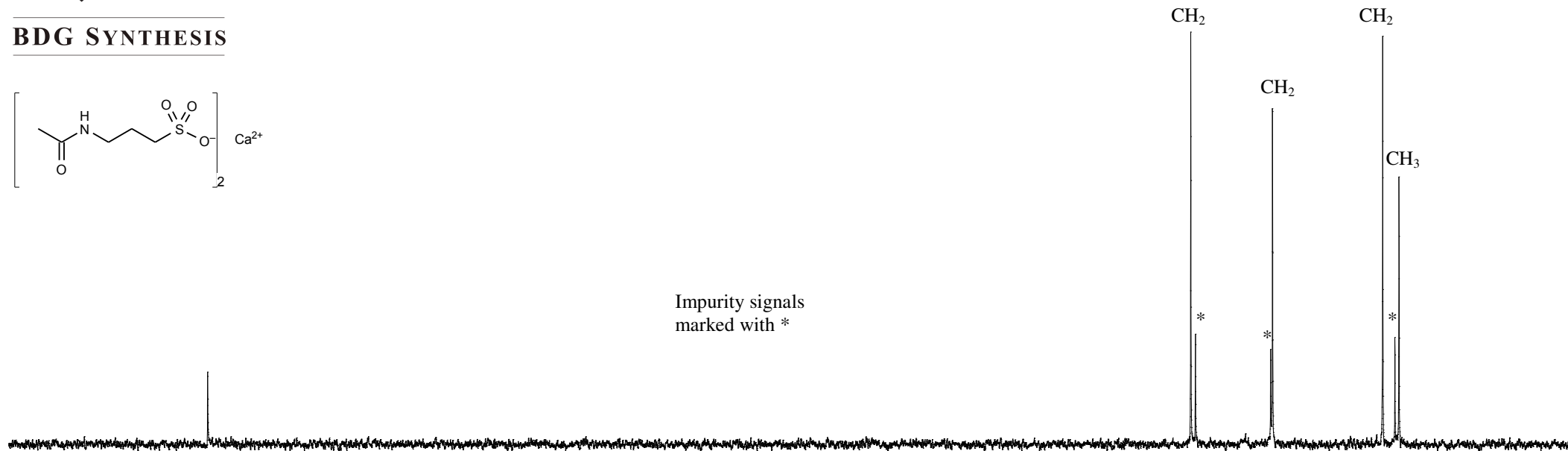
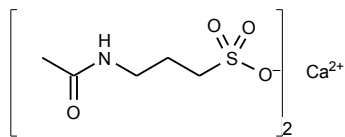
Proton NMR Spectrum of Acamprosate Calcium Salt (top) and Acamprosate-d<sub>6</sub> Calcium Salt (bottom) in D<sub>2</sub>O



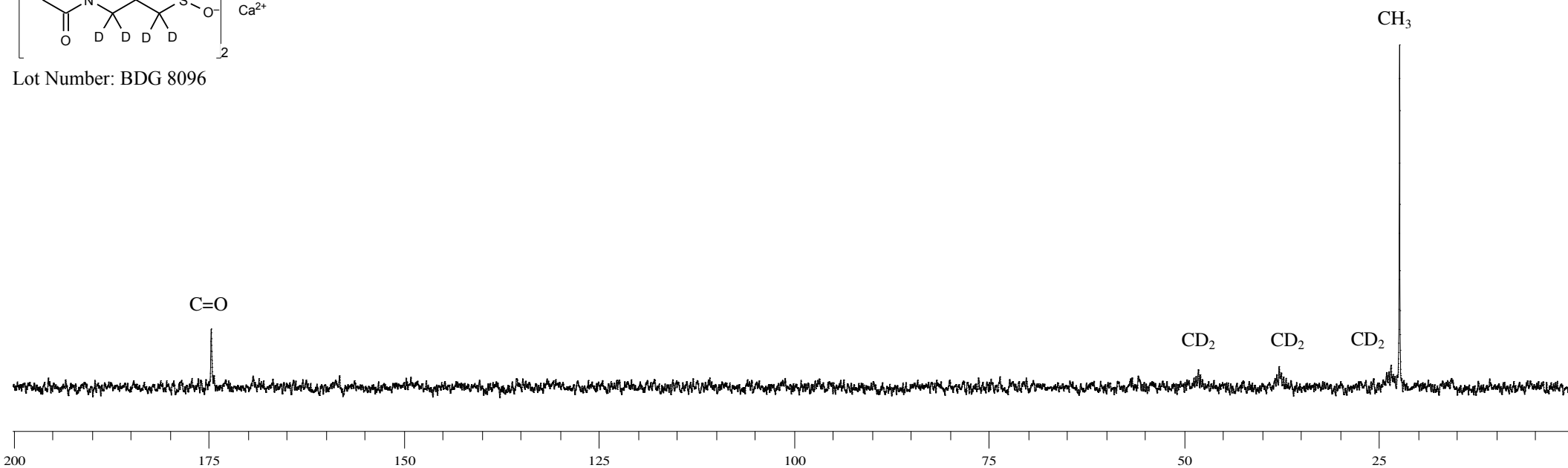


Carbon-13 NMR Spectrum of Acamprosate Calcium Salt (top) and Acamprosate-d<sub>6</sub> Calcium Salt (bottom) in D<sub>2</sub>O

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