



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.

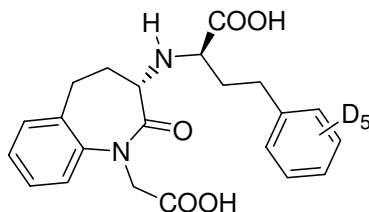
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director
18 January 2007

Name: Benazeprilat-d₅
CAS Number: none (86541-78-8 unlabelled)

Structure:



Molecular Weight: C₂₂H₁₉D₅N₂O₅ = 401.47
Lot Number: BDG 6627.1
Appearance: White, crystalline solid
Corrected Purity: 98.9 % (HPLC) – 0.2 % (triethylsilanol) = 98.7 %
Isotopic Purity: Under 0.5 % d₀
Expiry Date: 18 January 2012

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

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: store in an amber vial and protect from bright light.
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 what would be expected for unlabelled material, indicating clean deuteration.

Residual solvents: no residual solvents are observed.

Impurities: a small amount of triethylsilanol (0.2 % w/w) is seen.

Carbon-13 NMR Spectrum

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

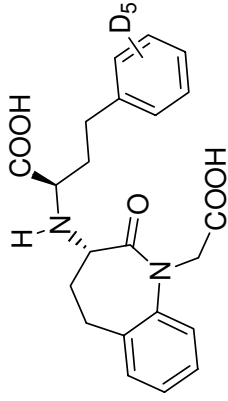
Isotopic labelling: signals at the sites of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material, indicating clean deuteration.

High-resolution mass spectrum (ESI-): found m/z 400.1921. $C_{22}H_{18}D_5N_2O_5$ $[M-H]^-$ requires m/z 400.1915. The deviation of 1.4 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 %).

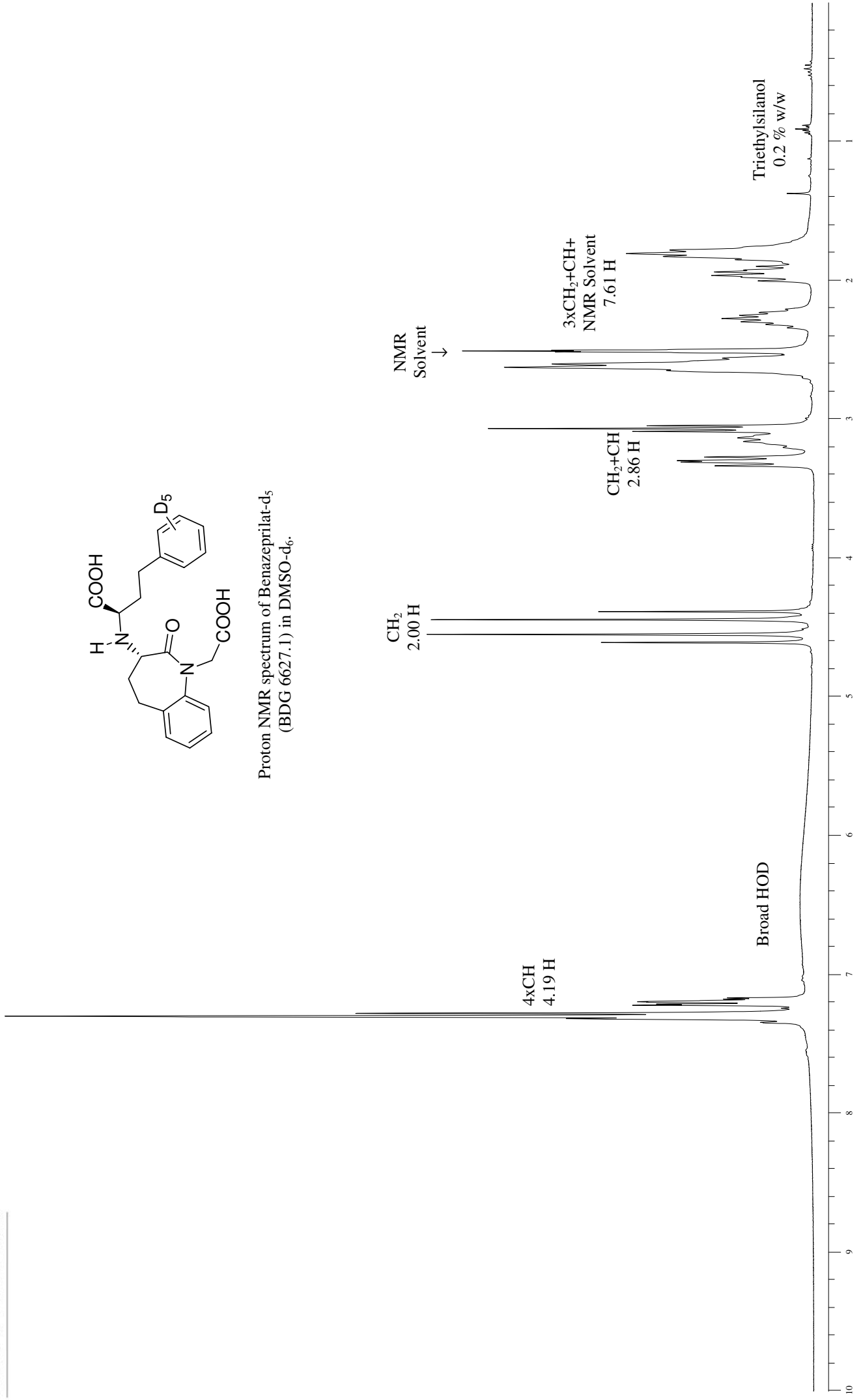
HPLC: A somewhat broadened, slightly tailing peak is observed (98.9 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 65.42, H 4.86, D 2.56, N 6.84 %
 $C_{22}H_{19}D_5N_2O_5$ requires: C 65.81, H 4.77, D 2.51, N 6.98 %

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).

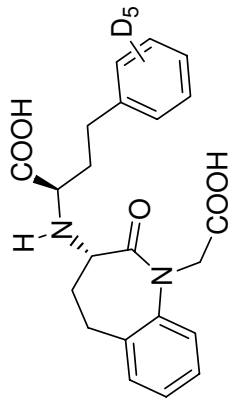


Proton NMR spectrum of Benazeprilat-d₅
(BDG 6627.1) in DMSO-d₆.

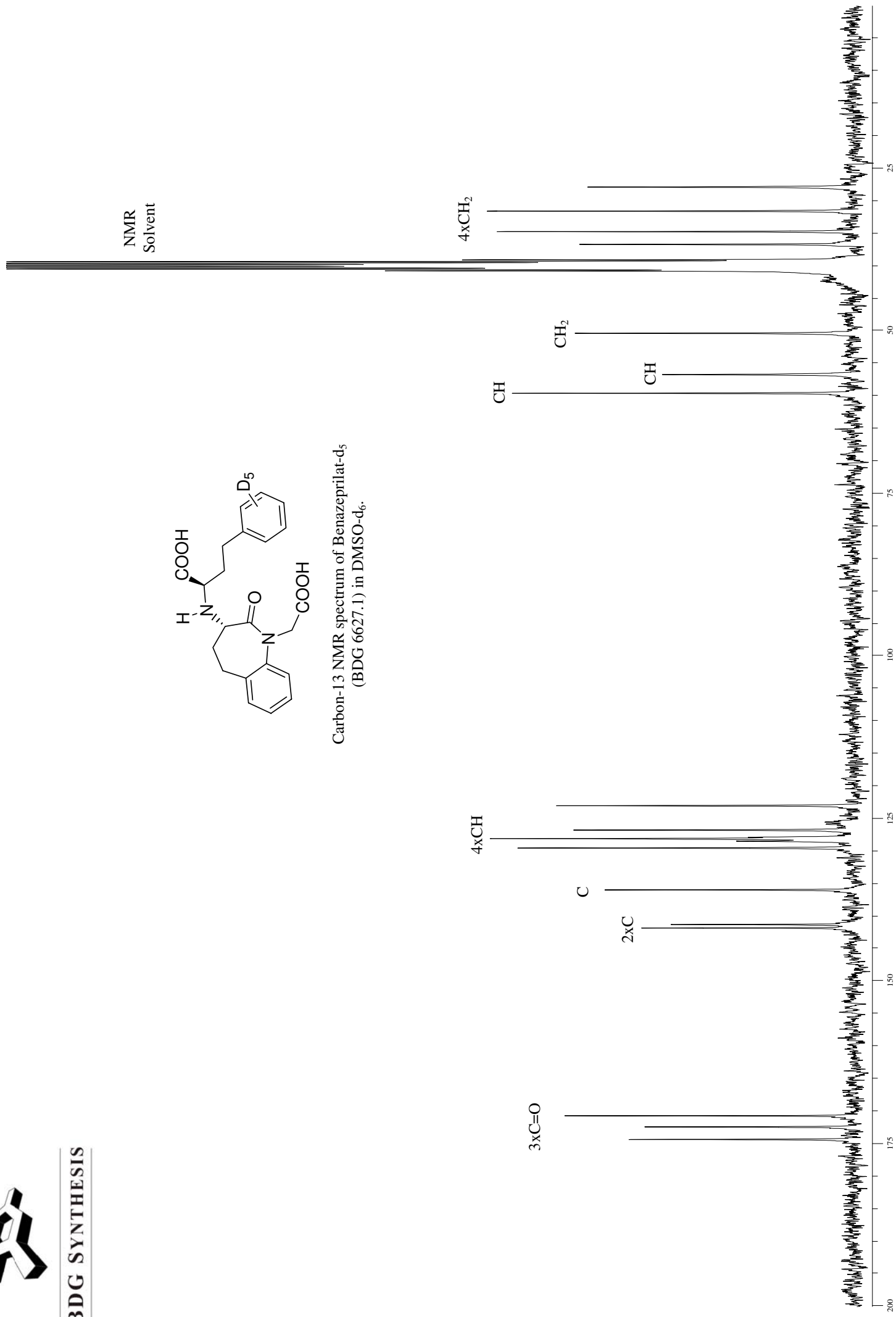




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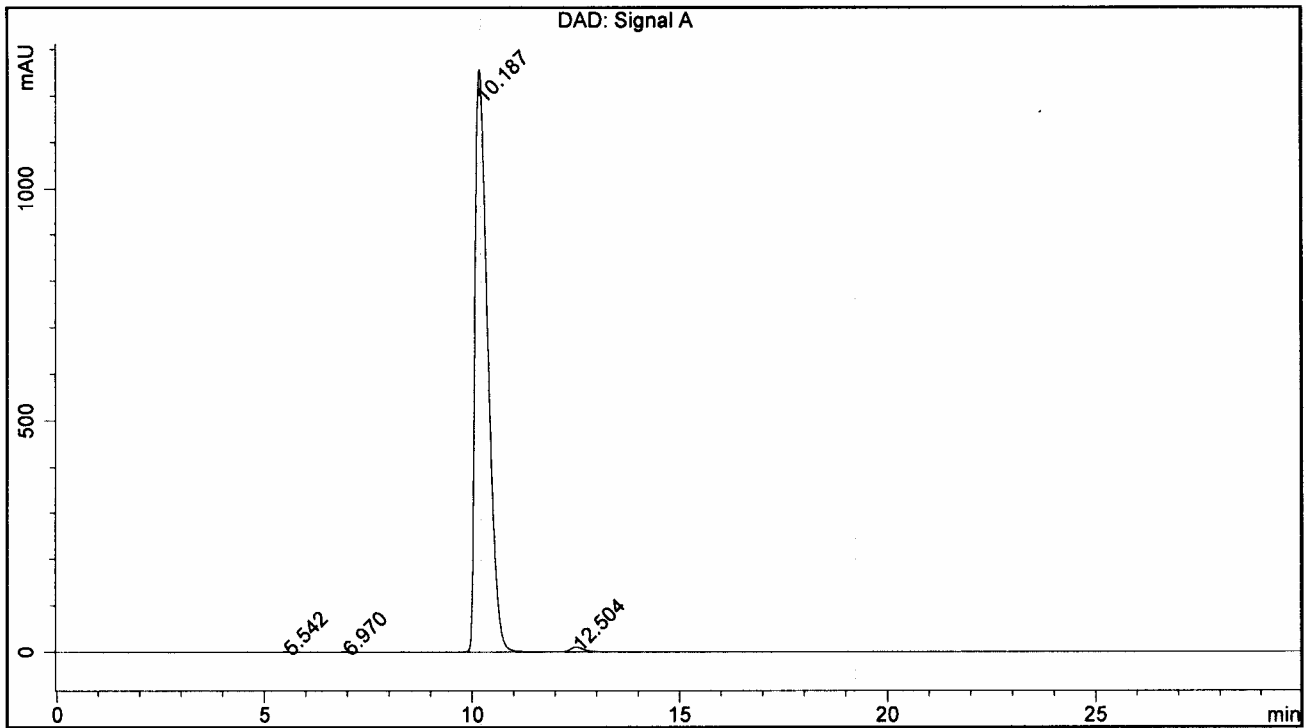
Carbon-13 NMR spectrum of Benazeprilat-d₅
(BDG 6627.1) in DMSO-d₆.



BDG - Analysis of Benazeprilat-(phenyl-d5)

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 80:20 0.02M Sodium Dihydrogen Phosphate pH=3.0 + 0.1% Triethylamine : 2-Propanol
 Flow Rate : 0.5 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 240 nm

Sample Name	BDG 6627.1	Instrument	AnalyticalLC01
Acquisition	08/01/2007, 10:25:12	Method (rev.)	LC10130a (2)
Sequence	BDG_08Jan2007a	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.54 min	0.6852	7.2261	0.1570 min	0.027 %
2	6.97 min	0.6752	11.5261	0.2392 min	0.043 %
3	10.19 min	1257.2342	26816.0203	0.3308 min	98.936 %
4	12.50 min	10.6546	269.6859	0.3713 min	0.995 %