



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.

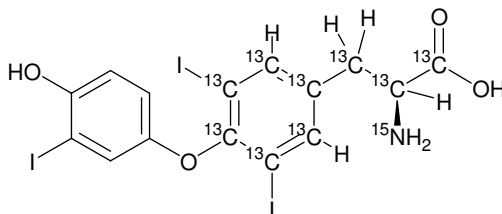
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

Barry Dent

Barry R. Dent, PhD, Director
18 April 2006

Name: Liothyronine-¹³C₉, ¹⁵N
CAS Number: none (6893-02-3 unlabelled)

Structure:



Molecular Weight: C₆¹³C₉H₁₂I₃¹⁵NO₄ = 660.90
Lot Number: BDG 4991.3
Appearance: White, crystalline solid
Corrected Purity: 99.5 % (HPLC) – 6.1 % (water) = 93.4 %
Isotopic Purity: Under 0.5 % ¹³C₀
Expiry Date: 18 April 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 isotopic labelling show typical ^1H - ^{13}C coupling.

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.

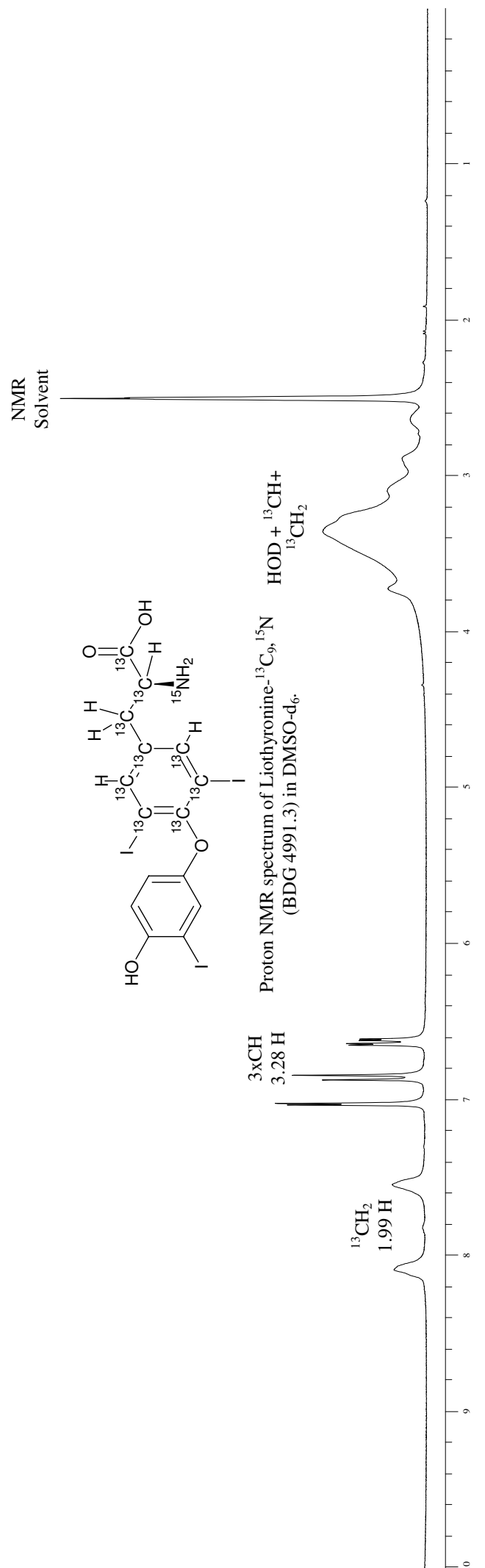
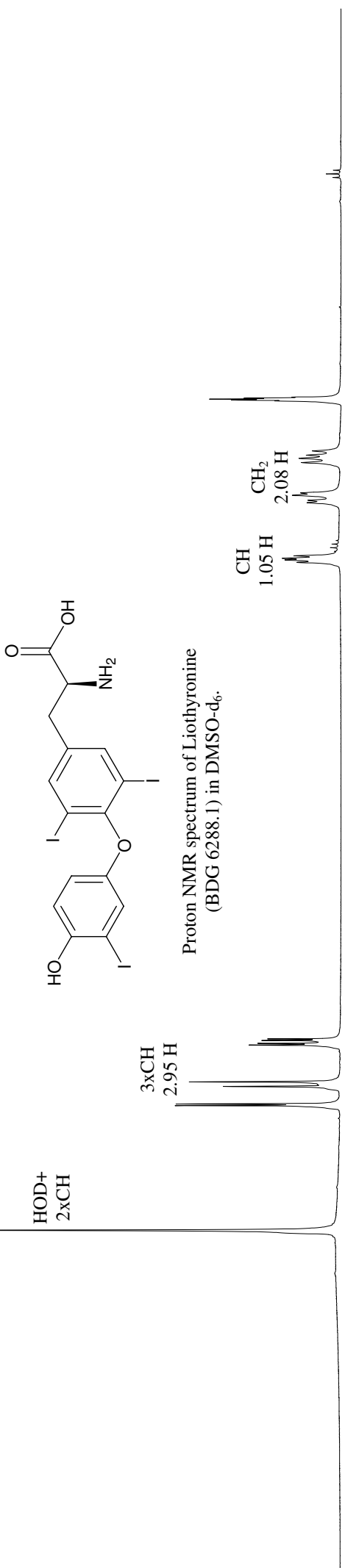
Isotopic labelling: signals at the sites of isotopic labelling show typical ^{13}C - ^{13}C coupling.

High-resolution mass spectrum (ESI+): found m/z 661.8248. $\text{C}_6^{13}\text{C}_9\text{H}_{13}\text{I}_3^{15}\text{NO}_4$ $[\text{M}+\text{H}]^+$ requires m/z 661.8245. The deviation of 0.4 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for $^{13}\text{C}_0$ material was seen (detection limit about 0.5 %).

HPLC: A somewhat broadened, slightly tailing peak is observed (99.5 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.

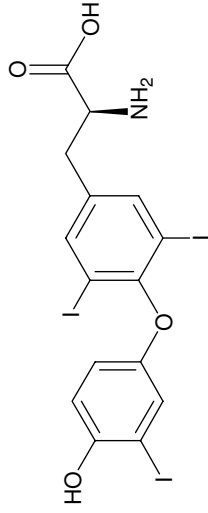
Elemental Analysis:	Found:	C 26.66, H 2.05, N 1.99 %
$\text{C}_6^{13}\text{C}_9\text{H}_{12}\text{I}_3^{15}\text{NO}_4 \bullet 2.4\text{H}_2\text{O}$	requires:	C 26.86, H 2.40, N 2.13 %, H_2O 6.1 %
$\text{C}_6^{13}\text{C}_9\text{H}_{12}\text{I}_3^{15}\text{NO}_4$	requires:	C 28.61, H 1.83, N 2.27 %

The elemental analyses substantially 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.

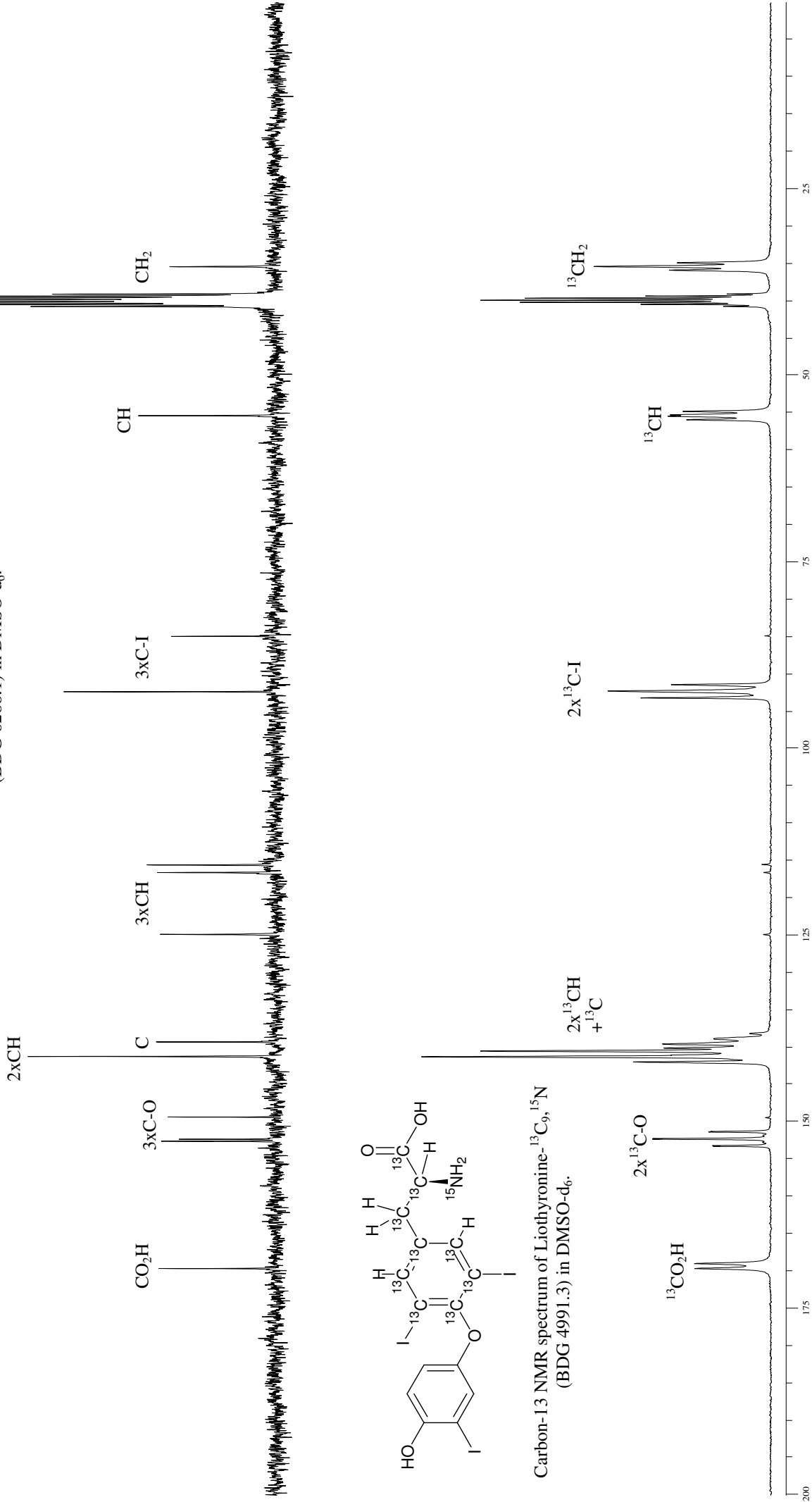




BDG SYNTHESIS



Carbon-13 NMR spectrum of Liothyronine (BDG 6288.1) in DMSO-d₆.



Solvation Analytical Report

BDG - Analysis of Liothyronine

Column : Phenomenex Synergi Max RP, 4um 250 x 4.6mm

Guard : Phenomenex Security Guard Max RP 4 x 3

Mobile Phase A: Milli-Q

Mobile Phase B: CH3CN

Flow Rate : 1.0 mL/min

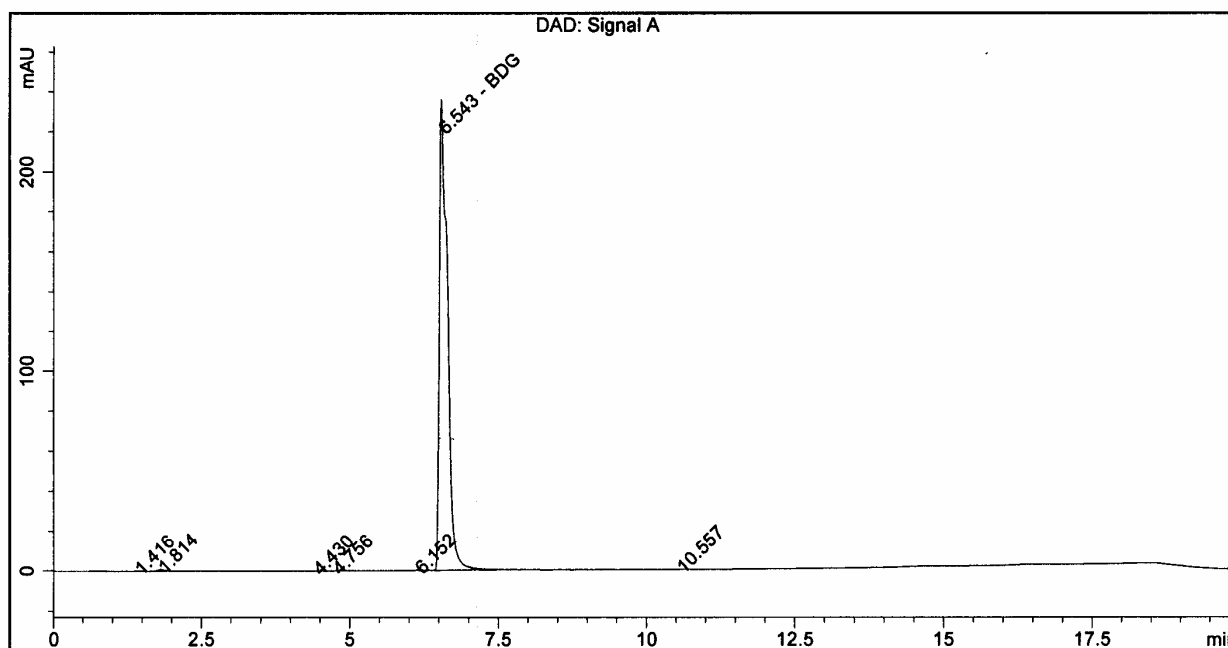
Sample Solvent : Initial Mobile Phase

Column Temp : 20 C

Injection Volume : 10 ul

Detection : UV at 230 nm

Sample Name	BDG 4991.3	Instrument	Analytical LC 01
Acquisition	13-Apr-06, 12:59:40	Method (rev.)	LC10022 (9)
Sequence	BDG_13Apr2006b	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak #	RT	Height	Area	Width	Area %
1	1.42	0.0669	0.9564	0.1807	0.042
2	1.81	0.8492	6.1243	0.1021	0.271
3	4.43	0.0520	0.3722	0.1014	0.016
4	4.76	0.1102	0.9248	0.1259	0.041
5	6.15	0.2302	2.0068	0.1237	0.089
6	6.54	236.2229	2248.1006	0.1290	99.505
7	10.56	0.0675	0.7952	0.1555	0.035