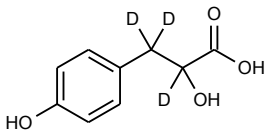
BDG SYNTHESIS

Certificate of Analysis

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

Neil Beare

Neil Beare, PhD, Director
2 May 2017

Name:	**4-Hydroxyphenyllactic acid-d_3**
CAS Number:	none
Structure:	
Molecular Weight:	$C_9H_7D_3O_4$ = 185.19
Lot Number:	BDG 1806.5
Appearance:	White, crystalline solid
Corrected Purity:	98.8 % (HPLC) - 4.6 % (water) = 94.2 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	2 May 2022

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.

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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 greatly diminished, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual Solvents: no residual solvents are observed.
Impurities: traces of unidentified impurities are seen in the baseline.

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 what would be expected for unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 208.0664. $C_9H_7D_3O_4$ $[M+Na]^+$ requires *m/z* 208.0660. The deviation of 1.9 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 (98.8 %). 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 55.38, H 3.93, D 3.37 %
$C_9H_7D_3O_4 \cdot 0.5H_2O$	Requires:	C 55.66, H 4.15, D 3.11 %, H_2O 4.64 %
$C_9H_7D_3O_4$	Requires:	C 58.37, H 3.81, D 3.26 %

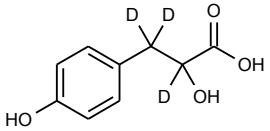
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

BDG SYNTHESIS

Proton NMR Spectrum of 4-Hydroxyphenyllactic acid-d_3 in Methanol-d_4



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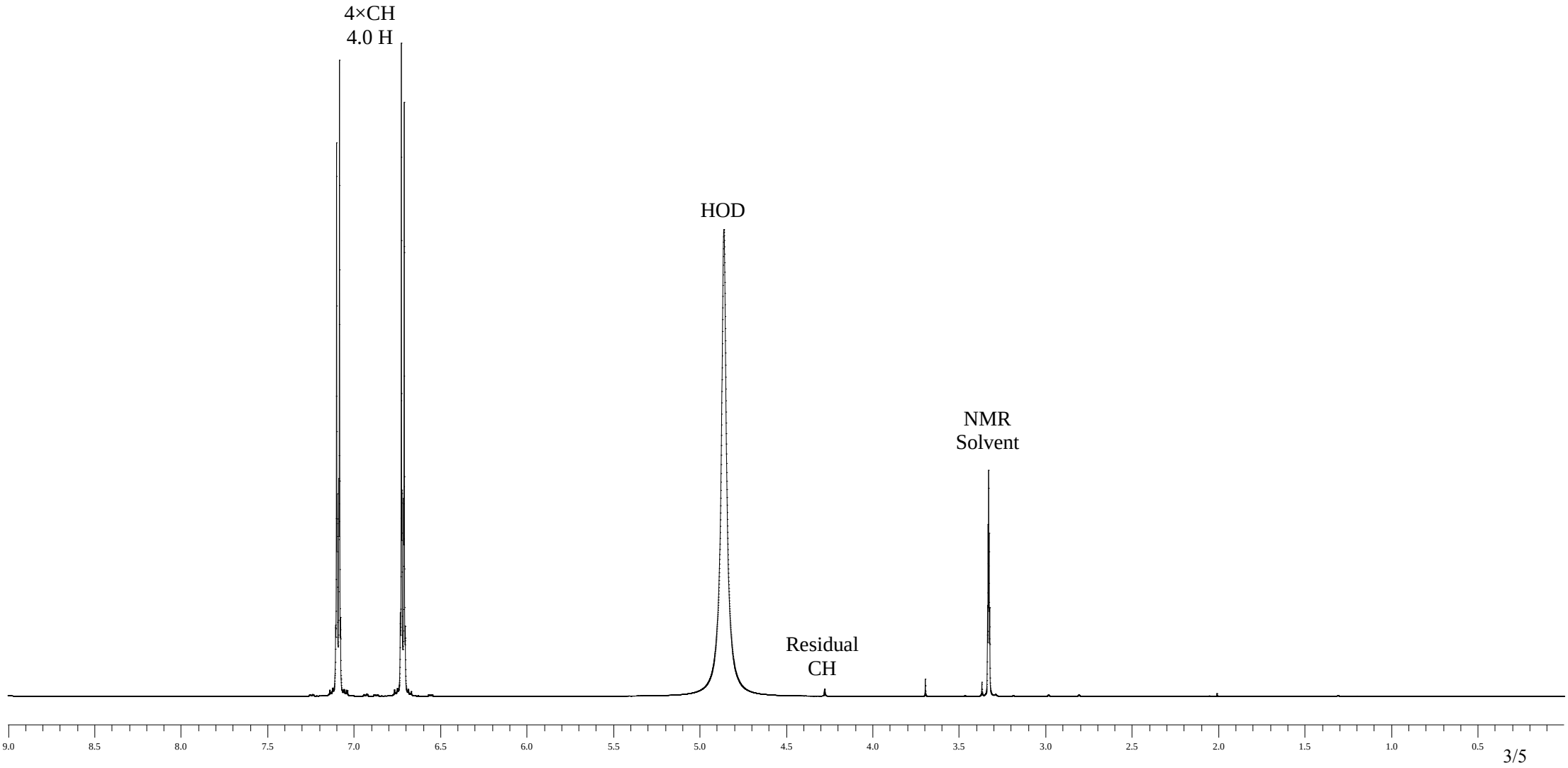
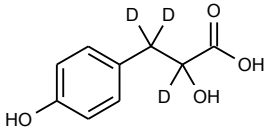


BDG SYNTHESIS

Carbon-13 NMR Spectrum of 4-Hydroxyphenyllactic acid-d_3 in Methanol-d_4

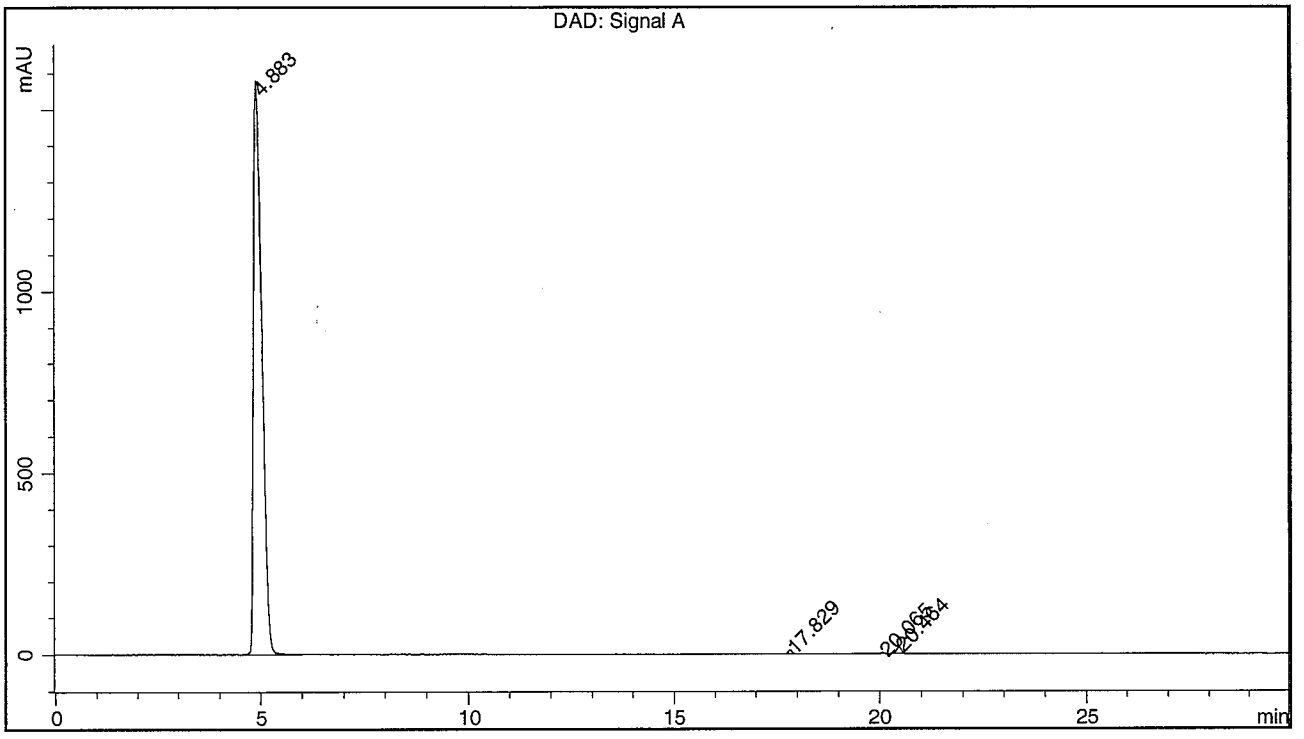


Lot Number: BDG 1806.5

BDG - Analysis of 4-Hydroxyphenyllactic acid-d3

Column : Phenomenex Luna C18 (2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A : 95:5 10 mM diPotassium Hydrogen Phosphate pH=7.00 : Acetonitrile
Mobile Phase A : 70:30 10 mM diPotassium Hydrogen Phosphate pH=7.00 : Acetonitrile
Gradient (A:B) : T0=100:0, T5=100:0, T20=0:100, T26=0:100, T27=100:0, T30=100:0
Flow Rate : 1 mL/min
Column Temperature : 20 C
Sample Solvent : Water
Injection Volume : 10 uL
Detection : UV at 224 nm

Sample Name	BDG 1806.5	**Instrument**	AnalyticalLC01
Acquisition	02/05/2017, 11:45:05	**Method (rev.)**	LC10710a (7)
Sequence	BDG_02May2017c - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.88 min	1579.0487	22444.2872	0.2298 min	98.846 %
2	17.83 min	10.7064	81.4605	0.1168 min	0.359 %
3	20.07 min	2.7976	19.4781	0.1012 min	0.086 %
4	20.46 min	18.0760	161.1431	0.1360 min	0.710 %