BDG SYNTHESIS

Certificate of Analysis

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

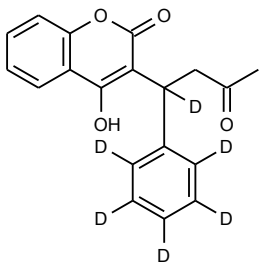
Barry Dent

Barry R. Dent, PhD, Director
17 September 2013

Name: **Warfarin-d_6**

CAS Number: 81-81-2 (unlabelled)

Structure:



Molecular Weight: $C_{19}H_{10}D_6O_4$ = 314.36

Lot Number: BDG 9029.6

Appearance: White, crystalline solid

Purity By HPLC: 99.9 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 17 September 2018

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. The complexity of the spectrum indicates that a pair of diastereoisomeric hemiketals of the product are present in solution.
Isotopic Labelling: signals at the sites of deuteration are greatly diminished, 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. The complexity of the spectrum indicates that a pair of diastereoisomeric hemiketals of the product are present in solution.
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 (FAB+)

Found *m/z* 337.1325. $C_{19}H_{10}D_6NaO_4$ $[M+Na]^+$ requires *m/z* 337.1317. The deviation of 2.2 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 (99.9 %). 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 72.40, H 3.08, D 3.69 %
$C_{19}H_{10}D_6O_4$	Requires:	C 72.59, H 3.21, D 3.84 %

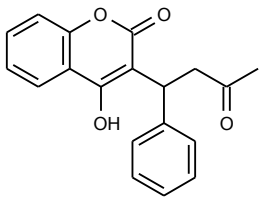
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

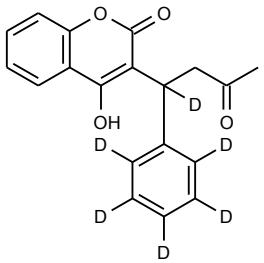
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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 Warfarin (top) and Warfarin-d_6 (bottom) in DMSO-d_6

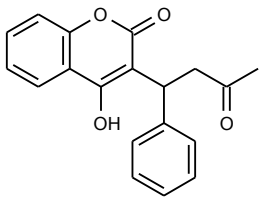


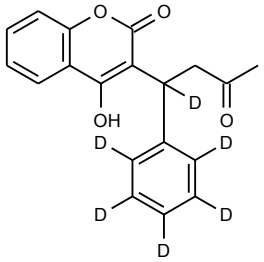


Lot Number: BDG 9029.6

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Warfarin (top) and Warfarin-d_6 (bottom) in DMSO-d_6





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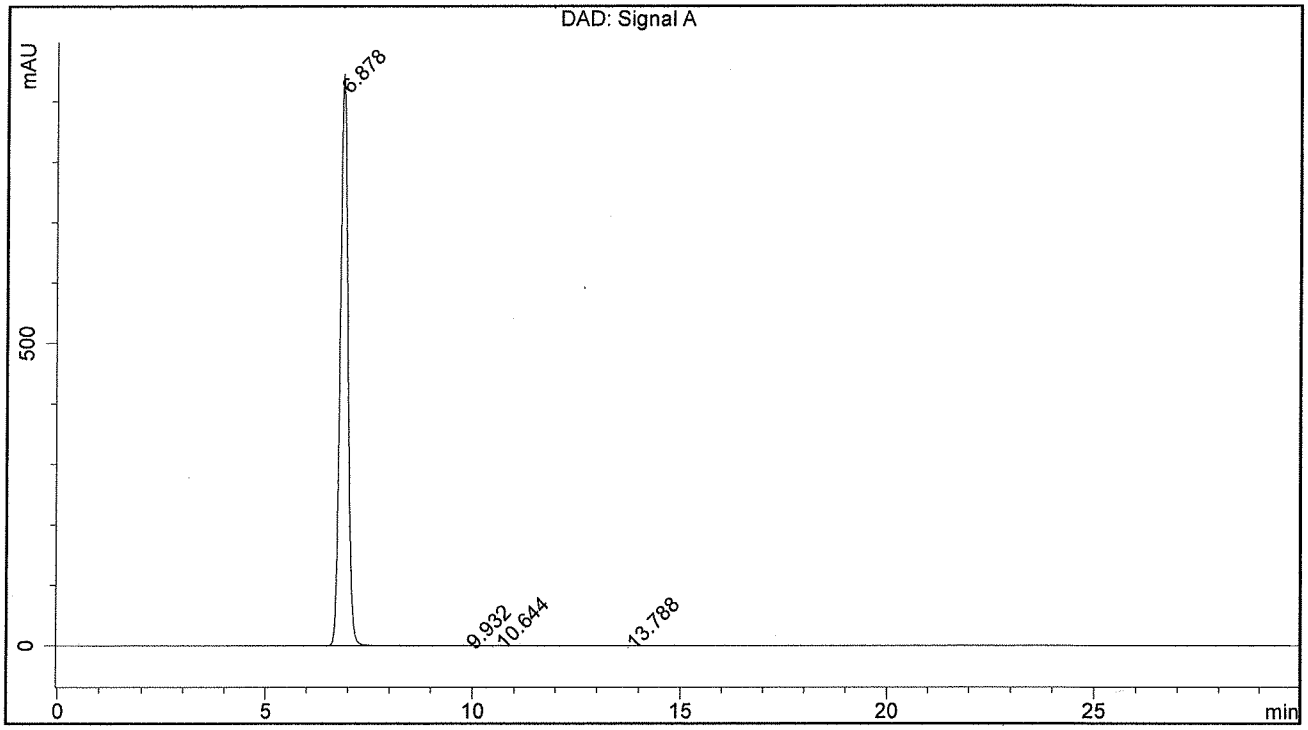
BDG - Analysis of Warfarin-d6

Column : Phenomenex Luna C8(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C8 RP 4 x 3 mm
Mobile Phase : 64:36:1 Methanol : Water : Acetic Acid
Flow Rate : 1.4 mL/min
Sample Solvent : 2:1 Methanol : Water
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 280 nm

Sample Name	BDG 9029.6	**Instrument**	AnalyticalLC01
Acquisition	17/09/2013, 17:33:47	**Method (rev.)**	LC10138a (7)
Sequence	BDG_17Sep2013d - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.88 min	945.2761	12297.5362	0.2018 min	99.900 %
2	9.93 min	0.3356	6.4314	0.2576 min	0.052 %
3	10.64 min	0.1806	3.4736	0.2424 min	0.028 %
4	13.79 min	0.1098	2.3791	0.2729 min	0.019 %