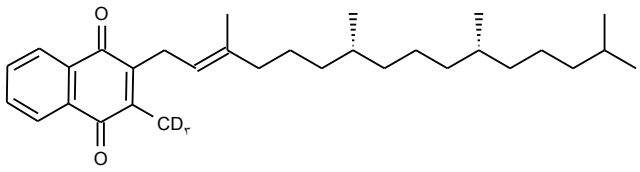
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
11 October 2011

Name:	**Phylloquinone-d_3**
CAS Number:	84-80-0 (unlabelled)
Structure:	
Molecular Weight:	$C_{31}H_{43}D_3O_2$ = 453.71
Lot Number:	BDG 12358.3
Appearance:	Yellow-orange oil
Corrected Purity:	95.9 % (HPLC) - 1.6 % (benzene) = 94.3 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	11 October 2016

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.

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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: the signal at the site of deuteration is absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: a small amount of benzene (1.6 % *w/w*) is 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: the signal at the site of deuteration has collapsed to a small multiplet compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 454.3764. $C_{31}H_{44}D_3O_2$ $[M+H]^+$ requires *m/z* 454.3764. The deviation of 0.0 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, symmetrical peak is observed (95.9 %). 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 82.23, H 9.82, D 1.37 %
$C_{31}H_{43}D_3O_2$	Requires:	C 82.06, H 9.55, D 1.33 %

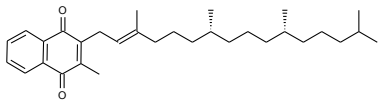
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

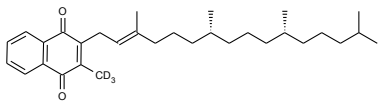
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials 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 Phylloquinone (top) and Phylloquinone-d_3 (bottom) in $CDCl_3$





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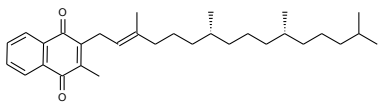
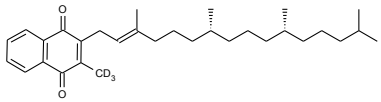
BDG SYNTHESIS

Carbon-13 NMR Spectrum of Phylloquinone (top) and Phylloquinone-d_3 (bottom) in $CDCl_3$





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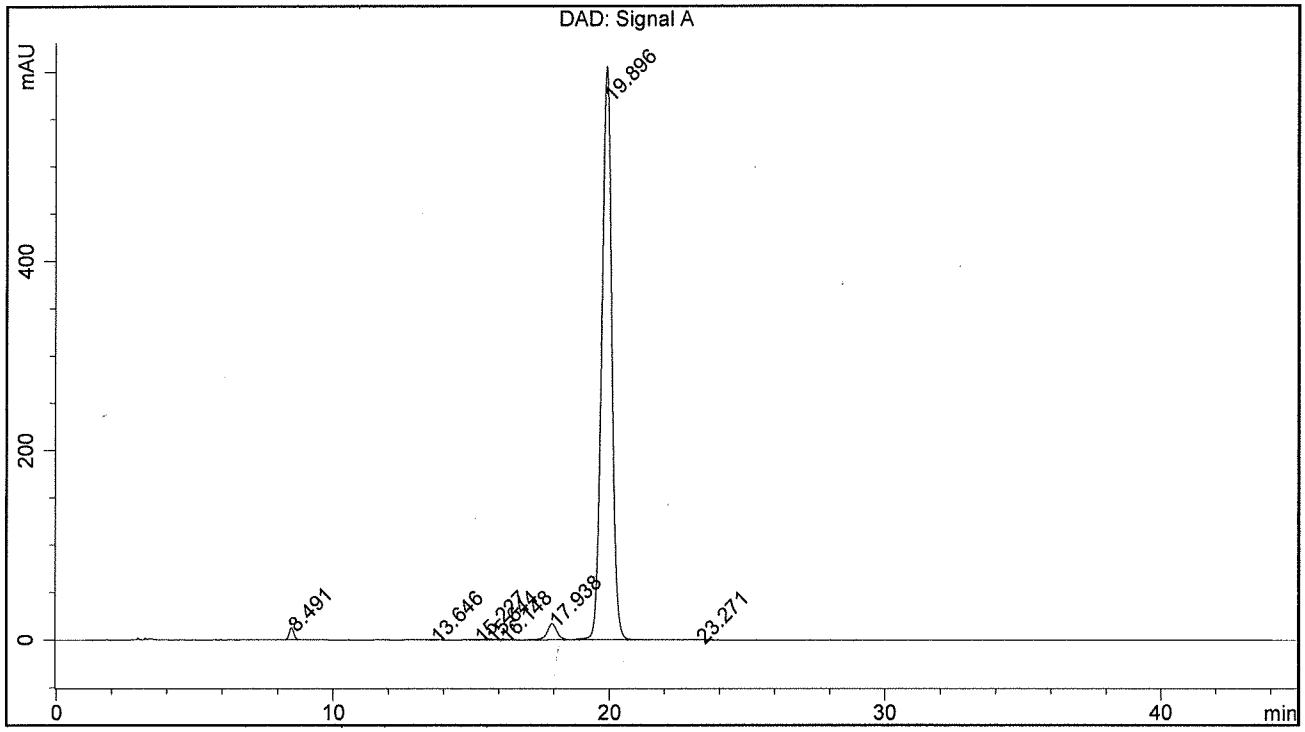
BDG - Analysis of Phylloquinone-d3

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex C18 4 x 3 mm
Mobile Phase : Methanol
Flow Rate : 1.0 mL/min
Sample Solvent : Methanol
Column Temperature : 30C
Injection Volume : 10 uL
Detection : UV at 246 nm

Sample Name	BDG 12358.3	**Instrument**	AnalyticalLC01
Acquisition	11/10/2011, 12:59:48	**Method (rev.)**	LC10269c (6)
Sequence	BDG_11Oct2011a	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	8.49 min	12.6158	150.0691	0.1828 min	0.946 %
2	13.65 min	0.5432	10.7901	0.2458 min	0.068 %
3	15.23 min	0.5946	14.5530	0.3015 min	0.092 %
4	15.64 min	0.9631	21.6382	0.3021 min	0.136 %
5	16.15 min	0.7206	18.1340	0.3016 min	0.114 %
6	17.94 min	17.0081	434.1815	0.3818 min	2.737 %
7	19.90 min	605.3521	15211.7039	0.3872 min	95.878 %
8	23.27 min	0.2538	4.6060	0.2157 min	0.029 %