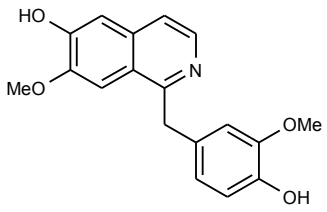
BDG SYNTHESIS

Certificate of Analysis

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

Barry Dent

Barry R. Dent, PhD, Director
21 March 2011

Name:	**4',6-Di-*O*-desmethylpapaverine**
CAS Number:	57170-09-9
Structure:	
Molecular Weight:	$C_{18}H_{17}NO_4$ = 311.33
Lot Number:	BDG 6350.2
Appearance:	Tan, crystalline solid
Corrected Purity:	98.9 % (HPLC) - 0.8 % (chloroform) - 1.4 % (water) = 96.7 %
Re-test Date:	21 March 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:	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.
Residual Solvents: a small amount of chloroform (0.8 % *w/w*) is 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.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 312.1252. $C_{18}H_{18}NO_4$ $[M+H]^+$ requires *m/z* 312.1230. The deviation of 6.9 ppm is somewhat outside normally accepted limits for the establishment of identity by HRMS, and the mass spectral data should be considered in conjunction with other identity criteria.

HPLC

A sharp, symmetrical peak is observed (98.9 %). 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 67.95, H 5.21, N 4.58 %
$C_{18}H_{17}NO_4 \cdot 0.4H_2O$	Requires:	C 67.87, H 5.63, N 4.40 %
$C_{18}H_{17}NO_4$	Requires:	C 69.44, H 5.50, N 4.50 %

The elemental analyses fall somewhat 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.

Karl-Fischer Analysis

	Found:	H_2O 1.4 %
$C_{18}H_{17}NO_4 \cdot 0.4H_2O$	Requires:	H_2O 2.3 %

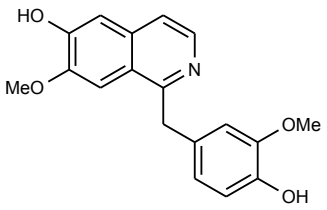
Of necessity, only a small sample could be used and only a single or duplicate analysis performed. We are unable to state what the errors in the reported water content are, but recommend that the result be used, as the best available, when determining corrected purity.

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',6-Di-*O*-desmethylpapaverine in DMSO-d_6



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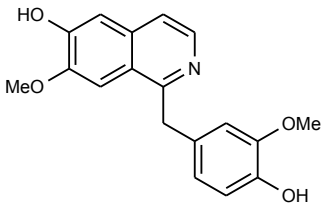
Carbon-13 NMR Spectrum of 4',6-Di-*O*-desmethylpapaverine in DMSO-d_6



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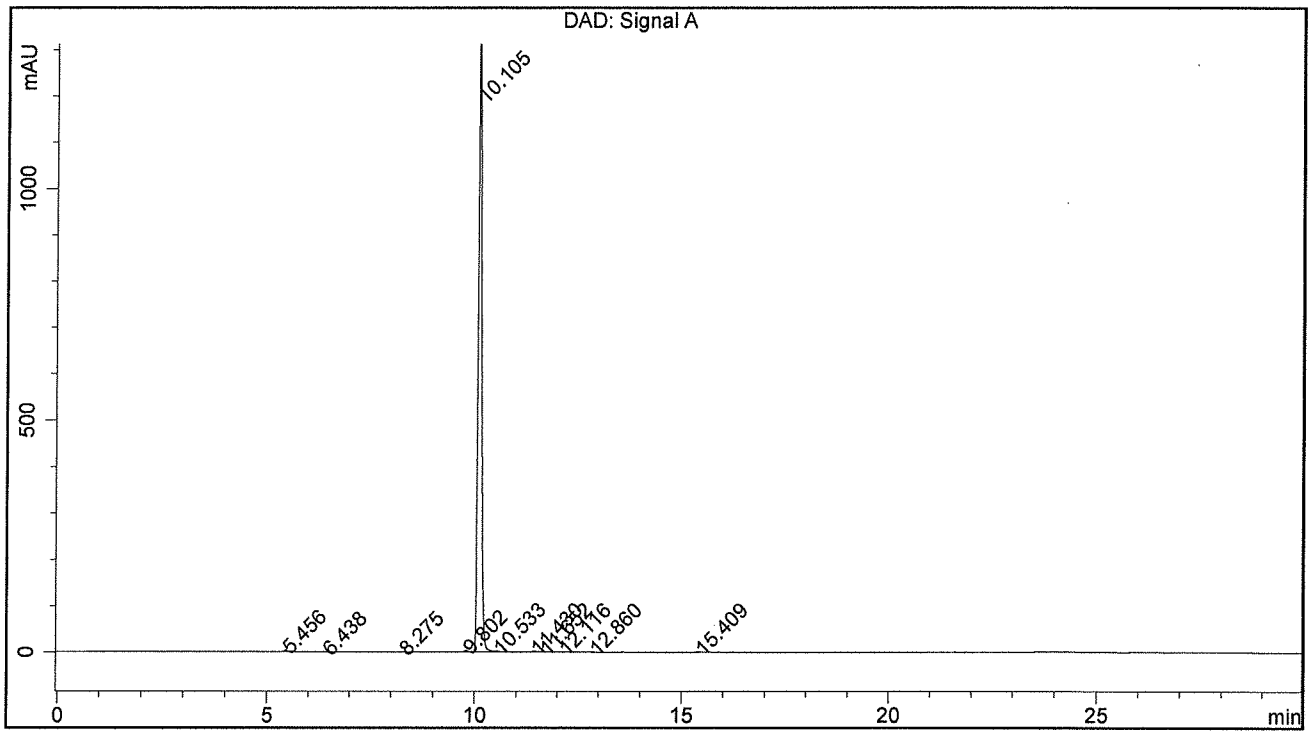
BDG - Analysis of Papaverine derivatives

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A: 90:10 25 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Mobile Phase B: 50:50 25 mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
Gradient (A:B) : T0=100:0, T20=0:100, T25=0:100, T28=100:0
Flow Rate : 1.0 mL/min
Sample Solvent : Initial Mobile Phase
Column Temperature : 35C
Injection Volume : 10 uL
Detection : UV at 238 nm

Sample Name	BDG 6350.2	**Instrument**	AnalyticalLC01
Acquisition	21/03/2011, 09:12:16	**Method (rev.)**	LC10019b (6)
Sequence	BDG_21Mar2011a - Reprocessed	**Vial Position**	2
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	5.46 min	3.5543	17.9282	0.0768 min	0.228 %
2	6.44 min	0.4425	2.5063	0.0841 min	0.032 %
3	8.28 min	0.5787	3.4927	0.0884 min	0.044 %
4	9.80 min	3.3469	20.4066	0.0891 min	0.259 %
5	10.10 min	1356.6685	7788.8614	0.0890 min	98.928 %
6	10.53 min	0.7085	4.0927	0.0874 min	0.052 %
7	11.43 min	1.6968	12.4540	0.1096 min	0.158 %
8	11.65 min	0.9945	6.5290	0.0966 min	0.083 %
9	12.12 min	0.6384	3.6228	0.0842 min	0.046 %
10	12.86 min	0.9973	5.3645	0.0808 min	0.068 %
11	15.41 min	1.3853	7.9863	0.0893 min	0.101 %