



BDG SYNTHESIS

Certificate of Analysis

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

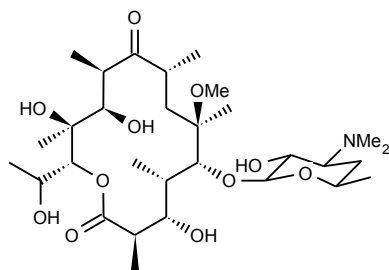
Barry Dent

Barry R. Dent, PhD, Director
30 November 2011

Name: Clarithromycin M8

CAS Number: none

Structure:



Molecular Weight: $C_{30}H_{55}NO_{11} = 605.76$

Lot Number: BDG 13315

Appearance: White, crystalline solid

Corrected Purity: 91.8 % (HPLC) - 0.4 % (pentane) - 0.7 % (diethyl ether) - 2.0 % (water) = 88.7 %

Re-test Date: 30 November 2012

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.

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Residual Solvents: small amounts of pentane (0.4 % w/w) and diethyl ether (0.7 % w/w) 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.

High-resolution Mass Spectrum (ESI+)

Found m/z 606.3857. $C_{30}H_{56}NO_{11}$ $[M+H]^+$ requires m/z 606.3853. The deviation of 0.7 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, slightly tailing peak is observed (91.8 %). 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 58.06, H 9.36, N 2.17 %
$C_{30}H_{55}NO_{11} \cdot 0.7H_2O$	Requires:	C 58.27, H 9.19, N 2.27 %, H_2O 2.04 %
$C_{30}H_{55}NO_{11}$	Requires:	C 59.48, H 9.15, N 2.31 %

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. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

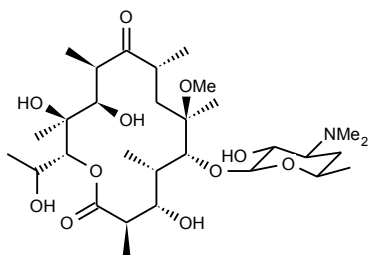
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

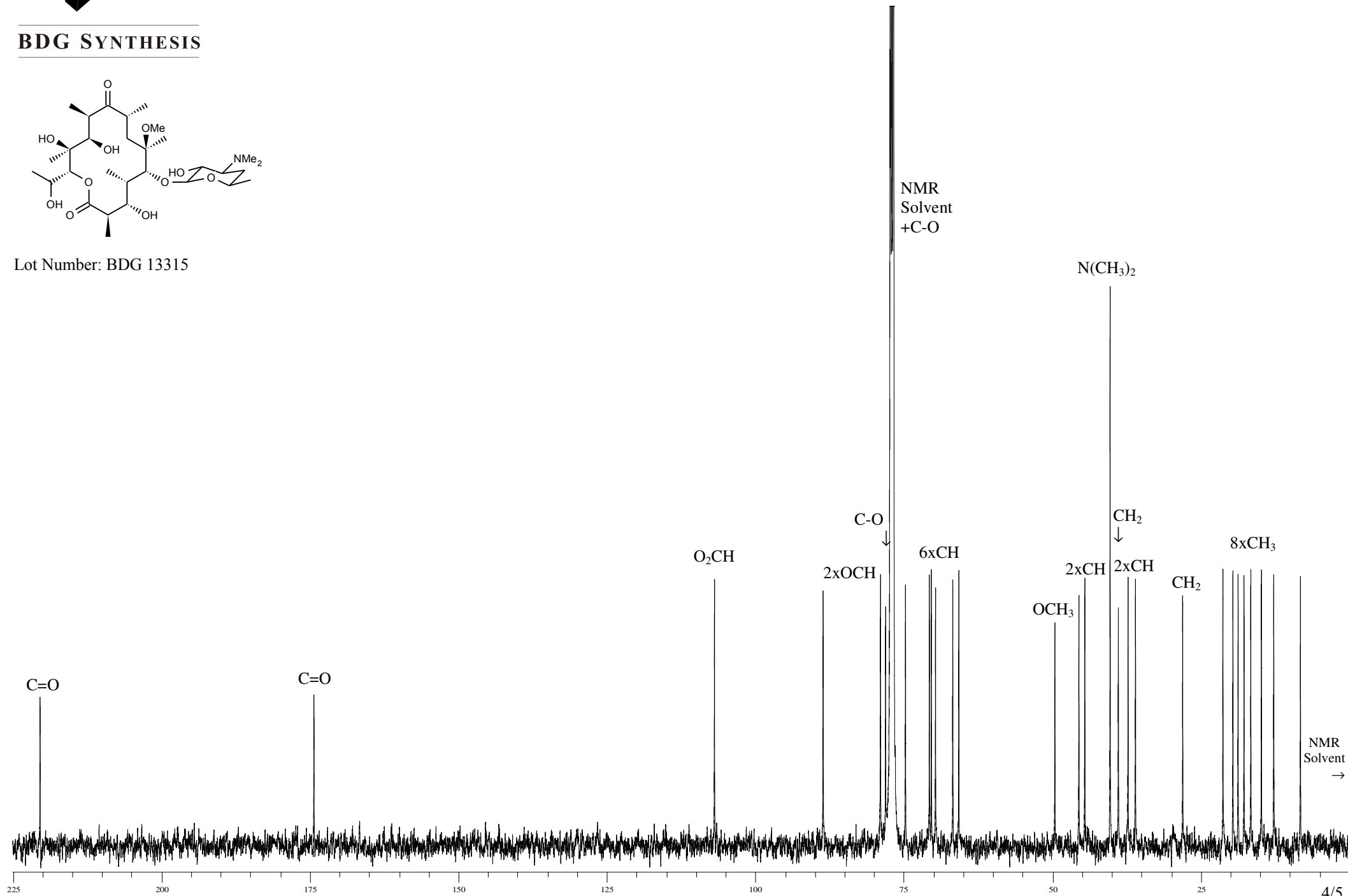


Carbon-13 NMR Spectrum of Clarithromycin M8 in CDCl₃

BDG SYNTHESIS



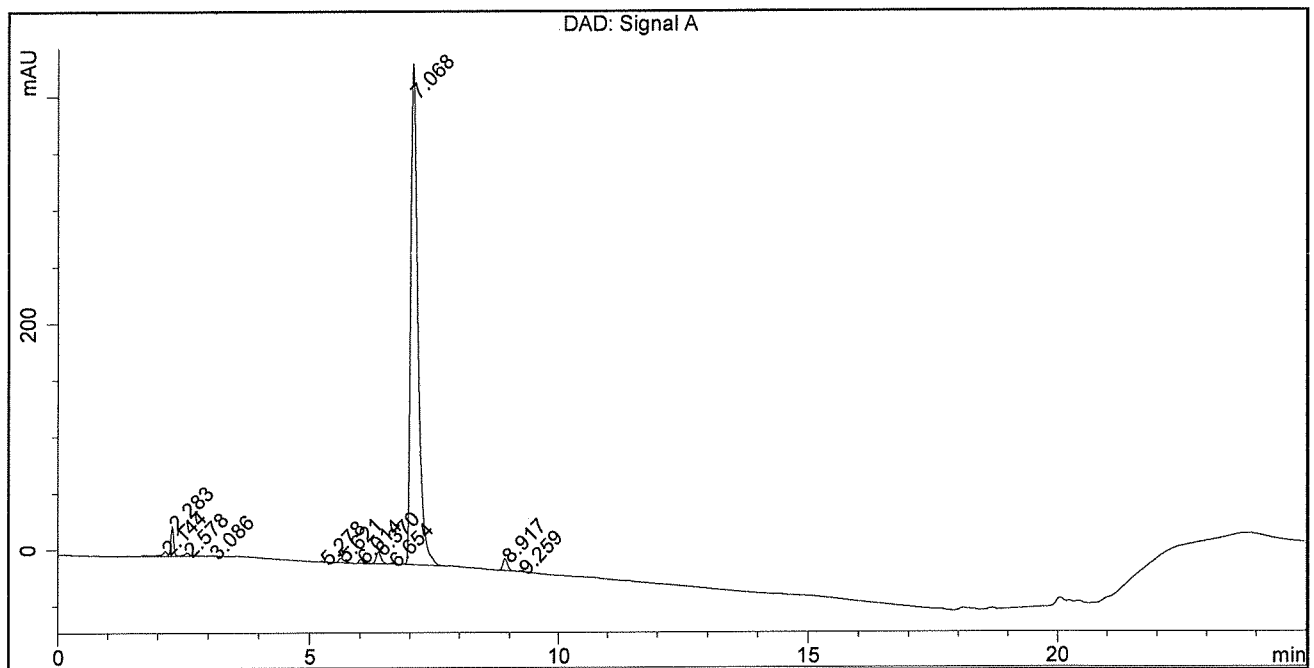
Lot Number: BDG 13315



BDG - Analysis of Clarithromycin M8

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase A : 75:25 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
 Mobile Phase B : 40:60 10 mM diPotassium Hydrogen Phosphate pH=7.0 : Acetonitrile
 Gradient (A:B) : T0=100:0, T15=0:100, T18=0:100, T20=100:0. T25=100:0
 Flow Rate : 1.0 mL/min
 Sample Solvent : Initial Mobile Phase
 Column Temperature : 30C
 Injection Volume : 20 uL
 Detection : UV at 205 nm

Sample Name	BDG 13315	Instrument	AnalyticalLC01
Acquisition	30/11/2011, 11:26:51	Method (rev.)	LC10207s (8)
Sequence	BDG_30Nov2011c - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	2.14 min	4.4926	44.6633	0.1340 min	0.923 %
2	2.28 min	26.3103	106.1265	0.0631 min	2.193 %
3	2.58 min	2.8821	26.3486	0.1267 min	0.545 %
4	3.09 min	0.6613	6.8372	0.1346 min	0.141 %
5	5.28 min	0.7953	6.3487	0.1151 min	0.131 %
6	5.62 min	4.0560	33.5350	0.1225 min	0.693 %
7	6.01 min	3.5825	23.6004	0.0989 min	0.488 %
8	6.37 min	10.4513	71.6679	0.1040 min	1.481 %
9	6.65 min	0.8306	5.2260	0.0994 min	0.108 %
10	7.07 min	442.5811	4443.0202	0.1491 min	91.821 %
11	8.92 min	10.4188	65.0705	0.0969 min	1.345 %
12	9.26 min	1.0455	6.3237	0.0946 min	0.131 %