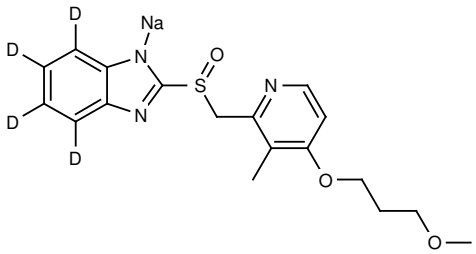
BDG SYNTHESIS

Certificate of Analysis

BD[illegible] t this reference material meets or exceeds the specifications stated herein.

B[illegible] Barry Dent

Barry R. Dent, PhD, Director
15 March 2012

Name:	**Rabeprazole-d_4 Sodium Salt**
CAS Number:	117976-90-6 (unlabelled)
Structure:	
Molecular Weight:	$C_{18}H_{16}D_4N_3NaO_3S$ = 385.45
Lot Number:	BDG 13416.1
Appearance:	Tan powder
Corrected Purity:	98.6 % (HPLC) - 0.4 % (methanol) - 0.6 % (methyl *t*-butyl ether) - 6.6 % (water) = 91.1 %
Isotopic Purity:	Under 0.5 % d_0
Re-test Date:	15 March 2017
Storage and Handling:	Temperature: refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
	Humidity: may be hygroscopic; store desiccated.
	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 absent, compared with what would be expected for unlabelled material, indicating clean deuteration.
Residual Solvents: small amounts of methanol (0.4 % *w/w*) and methyl *t*-butyl ether (0.6 % *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.
Isotopic Labelling: signals at the sites 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* 386.1446. $C_{18}H_{17}D_4N_3NaO_3S$ $[M+H]^+$ requires *m/z* 386.1452. The deviation of 1.6 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 (98.6 %). 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 52.49, H 4.33, D 1.82, N 10.08 %
$C_{18}H_{16}D_4N_3NaO_3S{\cdot}1.5H_2O$	Requires:	C 52.41, H 4.64, D 1.95, N 10.19 %, H_2O 6.55 %
$C_{18}H_{16}D_4N_3NaO_3S$	Requires:	C 56.09, H 4.18, D 2.09, N 10.90 %

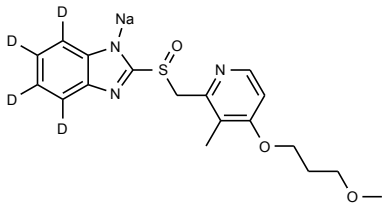
The elemental analyses fall substantially outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we 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.

BDG SYNTHESIS

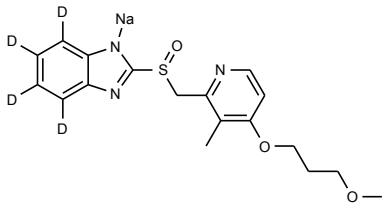
Proton NMR Spectrum of Rabeprazole-d_4 Sodium Salt in DMSO-d_6



Lot Number: BDG 13416.1

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Rabeprazole-d_4 Sodium Salt in DMSO-d_6

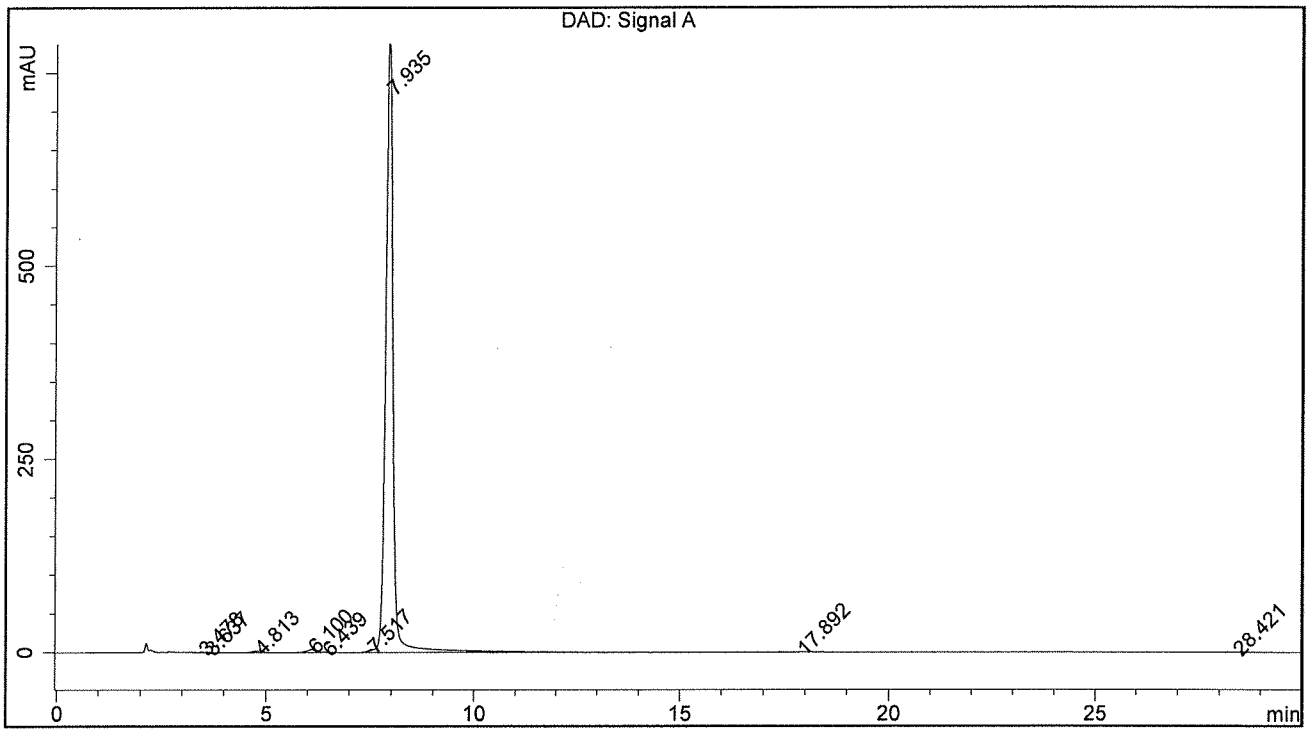


Lot Number: BDG 13416.1

BDG - Analysis of Rabeprazole-d4 Sodium Salt

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex SecurityGuard C18 4 x 3mm
Mobile Phase : 65:35 20 mM diPotassium Hydrogen Phosphate pH=8.0 : Acetonitrile
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 286 nm

Sample Name	BDG 13416.1	Instrument	AnalyticalLC01
Acquisition	15/03/2012, 14:16:21	Method (rev.)	LC10496a (8)
Sequence	BDG_15Mar2012c - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.48 min	0.4857	3.7389	0.1178 min	0.037 %
2	3.64 min	0.7138	4.2682	0.0918 min	0.043 %
3	4.81 min	2.1268	30.9876	0.2380 min	0.311 %
4	6.10 min	3.9958	54.0045	0.1937 min	0.541 %
5	6.44 min	0.4498	9.1075	0.2422 min	0.091 %
6	7.52 min	0.9935	6.6790	0.1124 min	0.067 %
7	7.93 min	809.1780	9834.7619	0.1838 min	98.571 %
8	17.89 min	0.3384	20.4578	0.7153 min	0.205 %
9	28.42 min	0.3750	13.3090	0.4240 min	0.133 %