

BDG SYNTHESIS

Certificate of Analysis

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

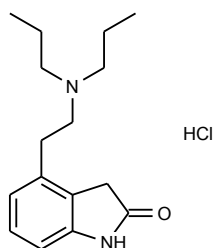
Barry Dent

Barry R. Dent, PhD, Director
20 October 2009

Name: Ropinirole HCl

CAS Number: 91374-20-8

Structure:



Molecular Weight: $C_{16}H_{24}N_2O \cdot HCl = 296.84$

Lot Number: BDG 2653.1

Appearance: Pale yellow, powder

Corrected Purity: 97.9 % (HPLC) - 0.6 % (water) = 97.3 %

Re-test Date: 20 October 2010

Storage and Handling:

Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
Humidity:	may be hygroscopic; store desiccated; recommended to determine water content periodically.
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: a trace (under 0.1 % w/w) of diethyl ether 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.

High-resolution Mass Spectrum (FAB+)

Found m/z 261.1974. $C_{16}H_{25}N_2O$ $[M+H]^+$ (free base) requires m/z 261.1967. The deviation of 2.7 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (97.9 %). 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 63.65, H 8.78, N 9.25 %
$C_{16}H_{24}N_2O \cdot HCl \cdot 0.3H_2O$	Requires:	C 63.58, H 8.54, N 9.27 %
$C_{16}H_{24}N_2O \cdot HCl$	Requires:	C 64.74, H 8.49, N 9.44 %

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.

Karl-Fischer Analysis

	Found:	H_2O 0.6 %
$C_{16}H_{24}N_2O \cdot HCl \cdot 0.3H_2O$	Requires:	H_2O 1.8 %

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.

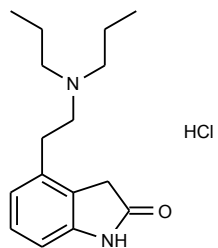
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

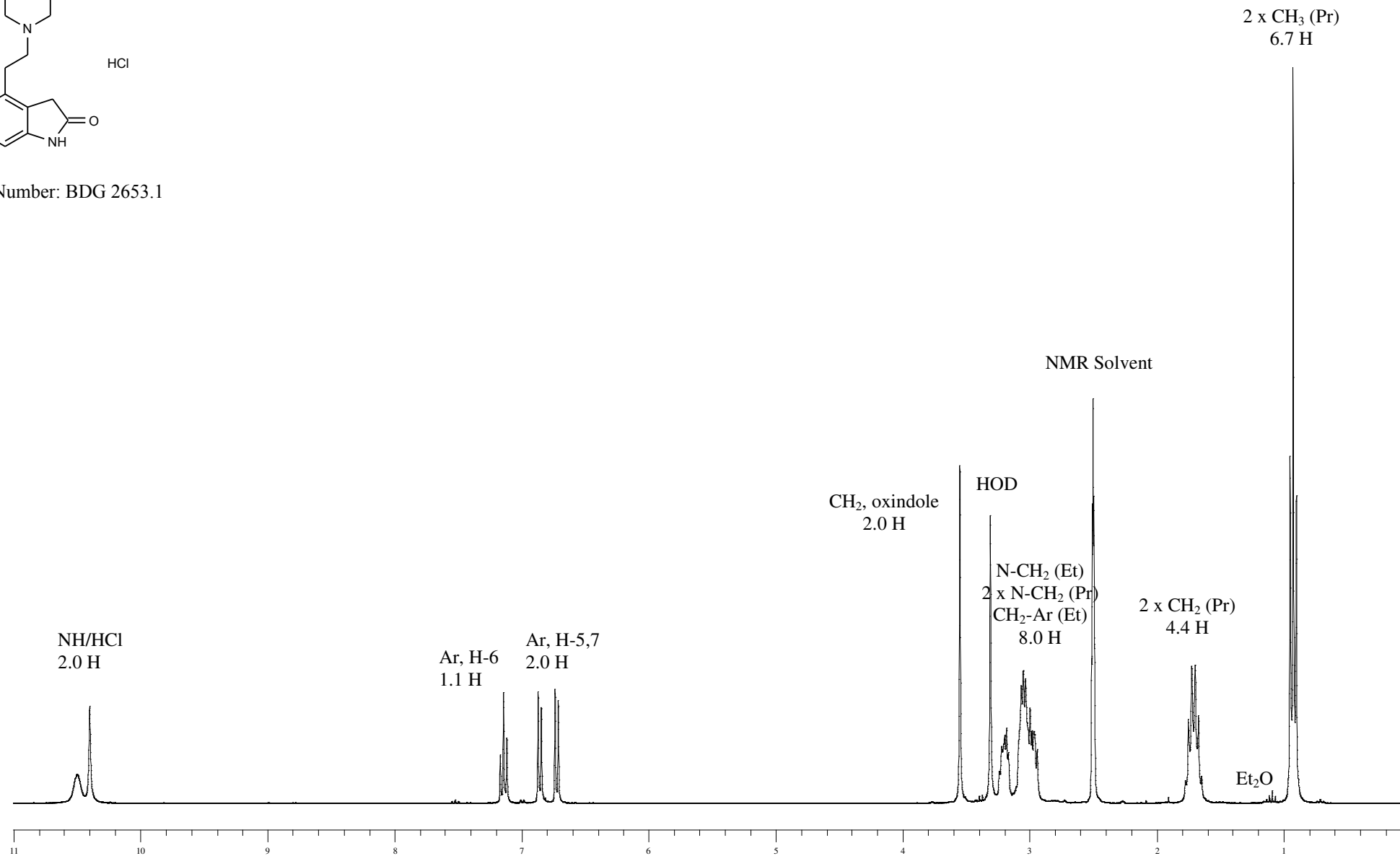


Proton NMR Spectrum of Ropinirole HCl in DMSO-d₆

BDG SYNTHESIS



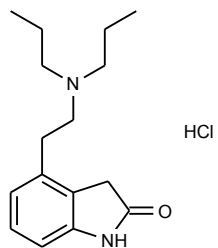
Lot Number: BDG 2653.1



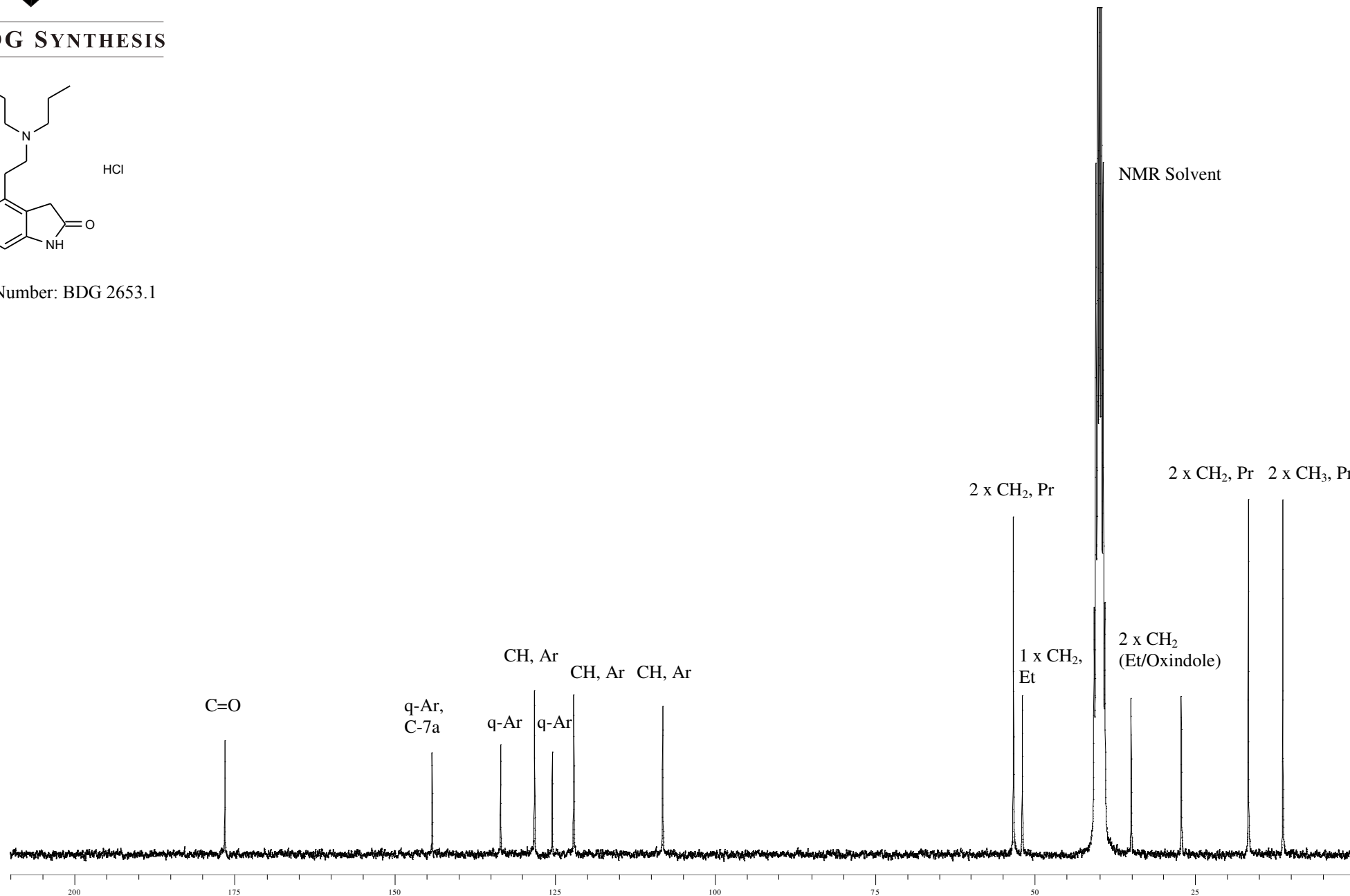


Carbon-13 NMR Spectrum of Ropinirole HCl in DMSO-d₆

BDG SYNTHESIS



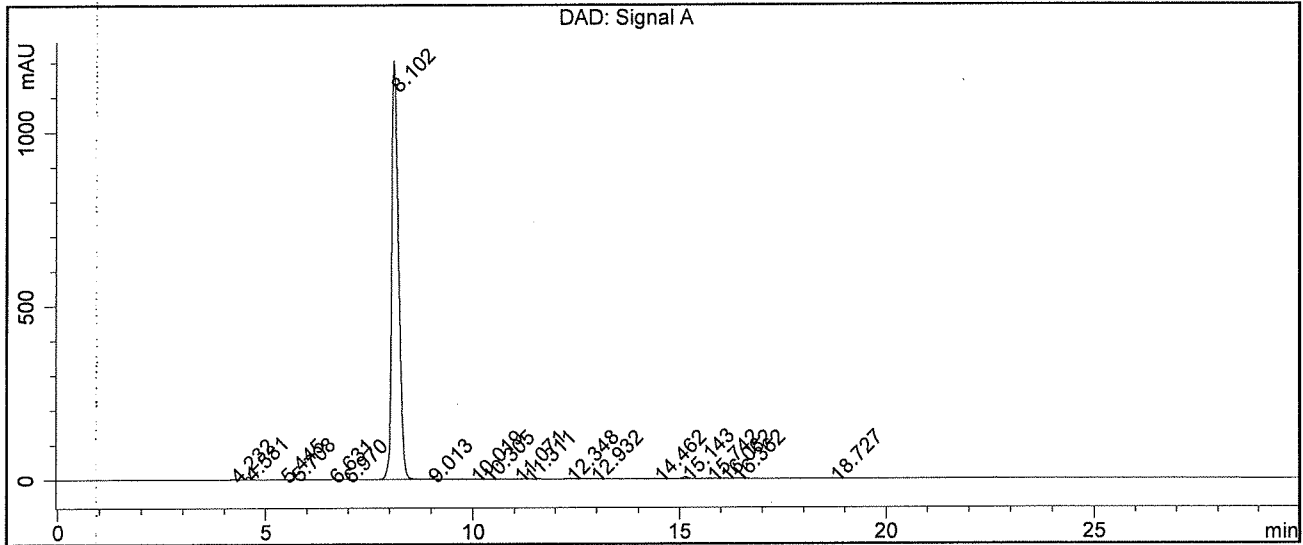
Lot Number: BDG 2653.1



BDG - Analysis of Ropinirole HCl

Column: Phenomenex Luna C18(2) 5um 250 x 4.6 mm + SecurityGuard C18 4 x 3mm
 Mobile Phase A: 80:20 Water :Acetonitrile + 0.1% TFA . . . B: 60:40 Water : Acetonitrile + 0.1% TFA
 Gradient (A:B) : T0=100:0, T20=0:100, T24=0:100, T27=100:0, T30=100:0
 Flow Rate : 1.0 mL/min Sample Solvent : 80:20 Water:Acetonitrile
 Column Temperature : 20C Injection Volume : 10 uL Detection : UV at 250 nm

Sample Name	BDG 2653.1	Instrument	AnalyticalLC01
Acquisition	20/10/2009, 18:47:35	Method (rev.)	LC10061f (8)
Sequence	BDG_20Oct2009d - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.23 min	1.6015	12.4688	0.1208 min	0.089 %
2	4.58 min	7.7934	56.2791	0.1122 min	0.403 %
3	5.44 min	1.2453	8.1329	0.1022 min	0.058 %
4	5.71 min	5.3982	38.0075	0.1081 min	0.272 %
5	6.63 min	1.0976	8.9628	0.1233 min	0.064 %
6	6.97 min	0.5633	5.5692	0.1454 min	0.040 %
7	8.10 min	1204.6590	13686.2223	0.1745 min	97.904 %
8	9.01 min	1.2472	12.6044	0.1459 min	0.090 %
9	10.02 min	0.9090	7.4606	0.1238 min	0.053 %
10	10.30 min	0.6009	10.2323	0.2527 min	0.073 %
11	11.07 min	0.7564	6.1855	0.1254 min	0.044 %
12	11.31 min	0.8219	8.7131	0.1577 min	0.062 %
13	12.35 min	1.2760	17.0227	0.2096 min	0.122 %
14	12.93 min	0.9132	16.4937	0.2387 min	0.118 %
15	14.46 min	0.3554	5.3670	0.2182 min	0.038 %
16	15.14 min	4.7264	45.3733	0.1460 min	0.325 %
17	15.74 min	1.1840	12.7579	0.1557 min	0.091 %
18	16.06 min	0.4878	5.1198	0.1625 min	0.037 %
19	16.36 min	0.9460	11.9775	0.1797 min	0.086 %
20	18.73 min	0.2252	4.3009	0.2528 min	0.031 %