



BDG SYNTHESIS

Certificate of Analysis

This material is a research-grade material prepared by custom synthesis. The quantity available is limited, 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 research-grade materials. Research 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.

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated in this data sheet.

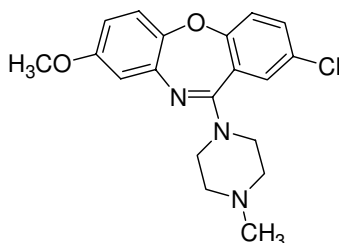
Barry Dent

Barry R. Dent, PhD, Director
4 September 2006

Name: 8-Methoxyloxapine

CAS Number: 70020-54-1

Structure:



Molecular Weight: $C_{19}H_{20}ClN_3O_2 = 357.84$

Lot Number: BDG 5494

Appearance: Pale yellow, crystalline solid

Corrected Purity: 99.9 % (HPLC) – 0.5 % (acetonitrile) = 99.4 %

Expiry Date: 4 September 2007

Because of the small amount of material available it is not possible to perform formal storage stability studies. This expiry date is assigned from experience gained with the material in the laboratory and/or on storage.

Storage and Handling:

Temperature: ambient laboratory temperature; may be refrigerated.

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.

Identity and Purity:**Source of Material**

The material was made by an unambiguous synthetic route, using literature procedures where possible; starting materials were purchased from reputable sources and all intermediates were checked for identity by NMR.

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 acetonitrile (0.5 % 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 358.1333. $C_{19}H_{21}ClN_3O_2$ $[M+H]^+$ requires m/z 358.1317. The deviation of 4.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

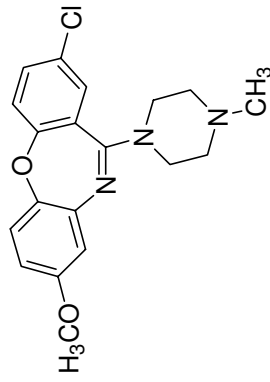
HPLC: A sharp, symmetrical peak is observed (99.9 area %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis: Found: C 64.04, H 5.70, N 11.83 %
 $C_{19}H_{20}ClN_3O_2$ requires: C 63.77, H 5.63, N 11.74 %

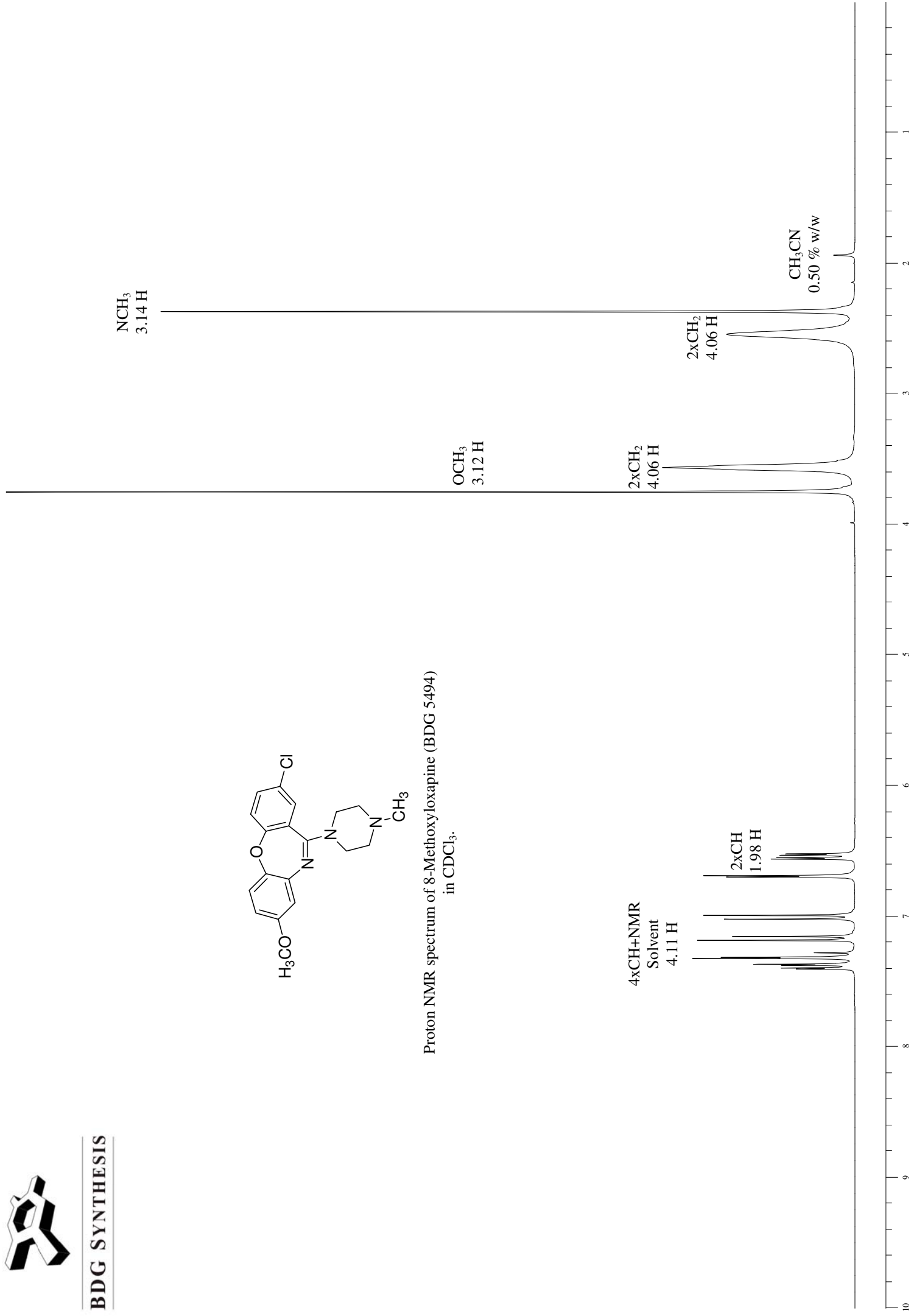
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).



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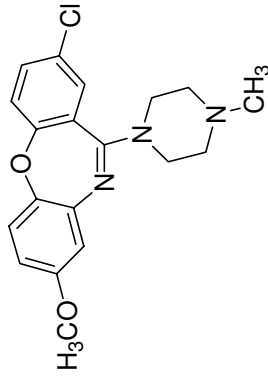


Proton NMR spectrum of 8-Methoxyloxapine (BDG 5494) in CDCl₃.





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Carbon-13 NMR spectrum of 8-Methoxyloxapine (BDG 5494) in CDCl₃.

2xCH₂

NCH₃

OCH₃

NMR
Solvent

2xCH₂

2xCH

2xCH

2xCH

3xC

2xC

C

C

200

175

150

125

100

75

50

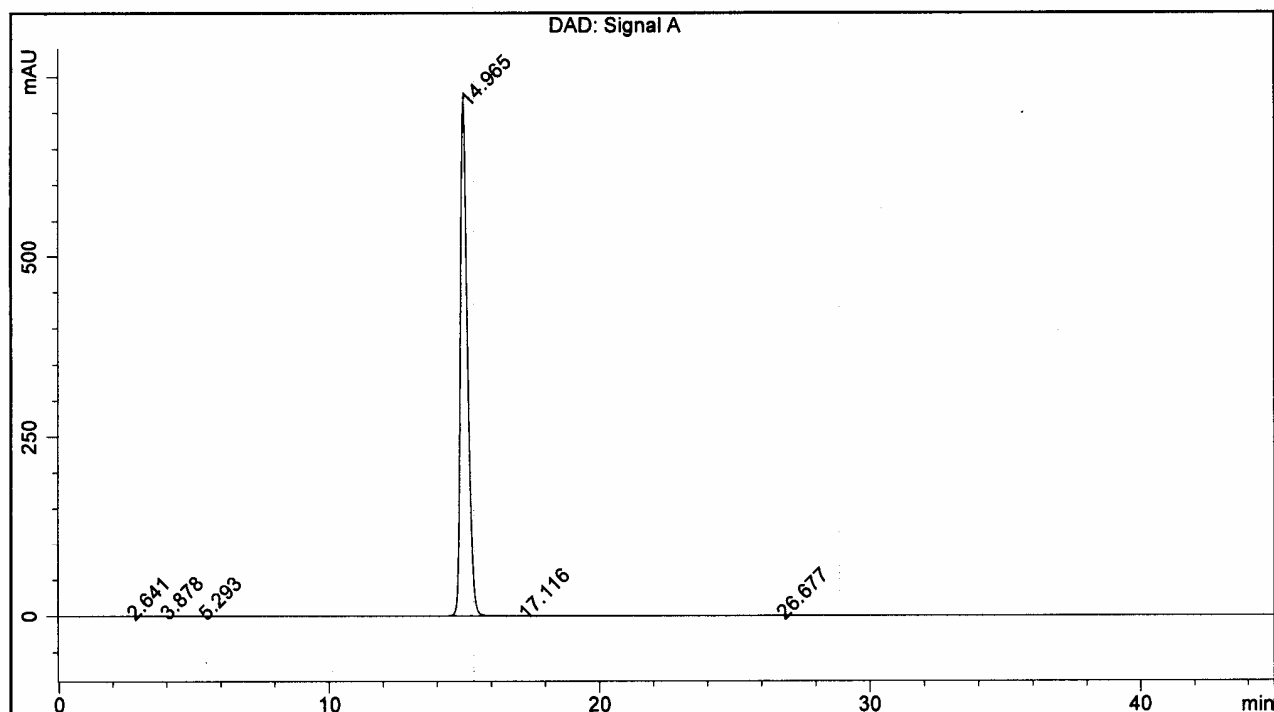
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Solvation Analytical Report

BDG - Analysis of 8-Methoxylozapine

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 52:48 5.5 mM Cetyltrimethylammonium Bromide, 5 mM Na2HPO4 pH = 7.0 : Acetonitrile
 Flow Rate : 1.5 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 50C
 Injection Volume : 10 uL
 Detection : UV at 254 nm

Sample Name	BDG 5494	Instrument	AnalyticalLC01
Acquisition	30/08/2006, 15:03:38	Method (rev.)	LC10083a
Sequence	BDG_30Aug2006b - Reprocessed	Vial Position	1
Operator	LC10083a	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	2.64 min	0.0520	0.2543	0.0750 min	0.002 %
2	3.88 min	0.3321	2.7943	0.1262 min	0.021 %
3	5.29 min	0.0708	0.6340	0.1205 min	0.005 %
4	14.97 min	716.9845	13199.2388	0.2788 min	99.952 %
5	17.12 min	0.0577	0.7691	0.1795 min	0.006 %
6	26.68 min	0.0592	1.9094	0.4081 min	0.014 %