BDG SYNTHESIS

Certificate of Analysis

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

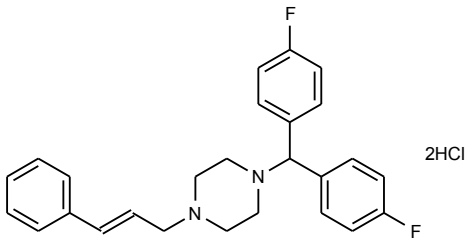
Barry Dent

Barry R. Dent, PhD, Director
18 August 2014

Name: **Flunarizine Dihydrochloride**

CAS Number: 30484-77-6

Structure:



Molecular Weight: $C_{26}H_{26}F_2N_2 \cdot 2HCl = 477.42$

Lot Number: BDG 6030

Appearance: White, crystalline solid

Purity By HPLC: 99.6 %

Re-test Date: 18 August 2019

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 trace (under 0.1 % *w/w*) of ethanol 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* 405.2140. $C_{26}H_{27}F_2N_2$ $[M+H]^+$ requires *m/z* 405.2142. The deviation of 0.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (99.6 %). 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 65.06, H 5.97, Cl 14.52, N 5.85 %
$C_{26}H_{26}F_2N_2 \cdot 2HCl$	Requires:	C 65.41, H 5.91, Cl 14.85, N 5.87 %

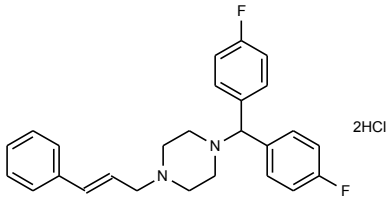
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

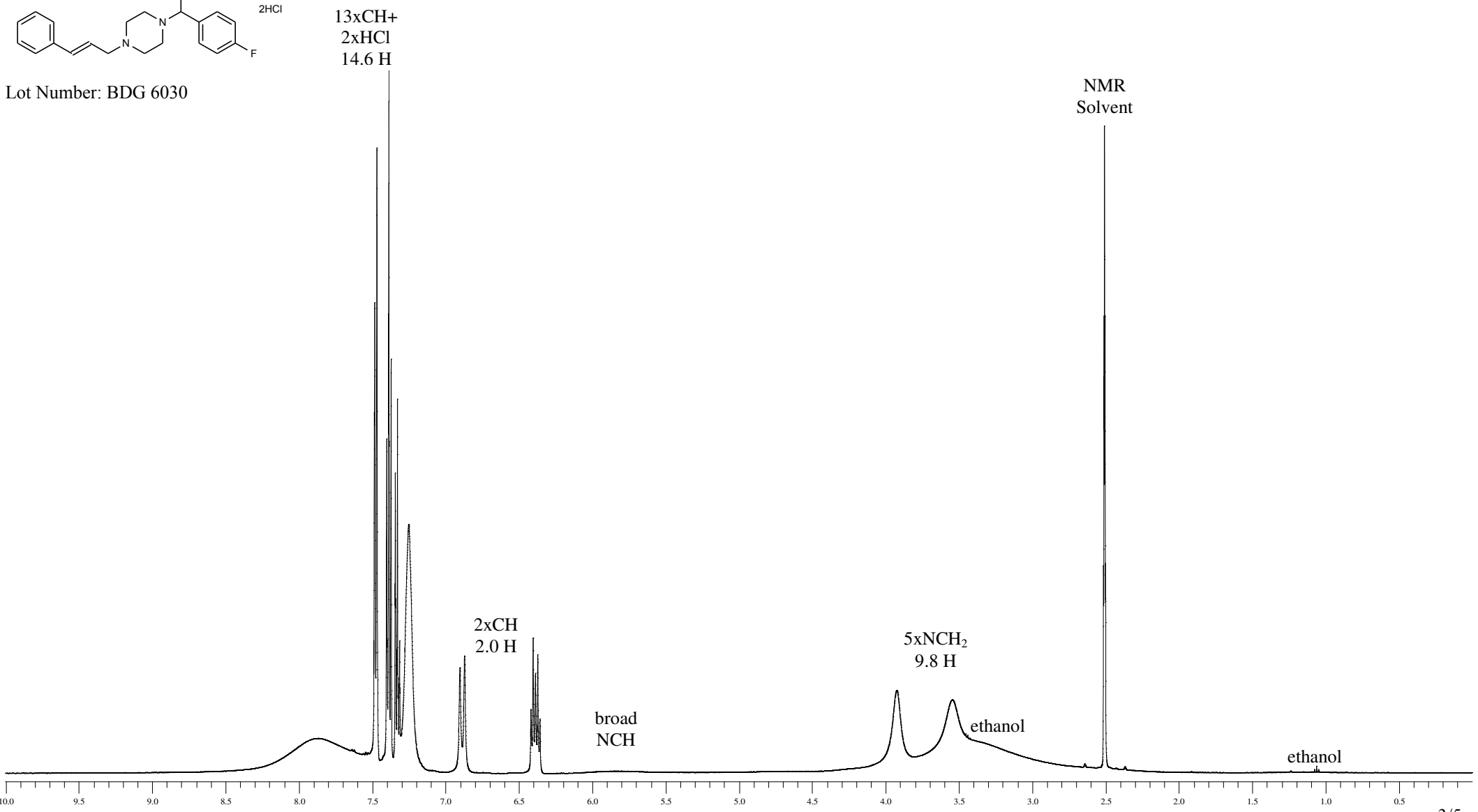
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

Proton NMR Spectrum of Flunarizine Dihydrochloride in DMSO-d_6



Lot Number: BDG 6030



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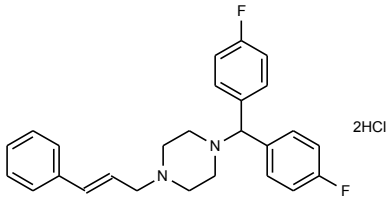
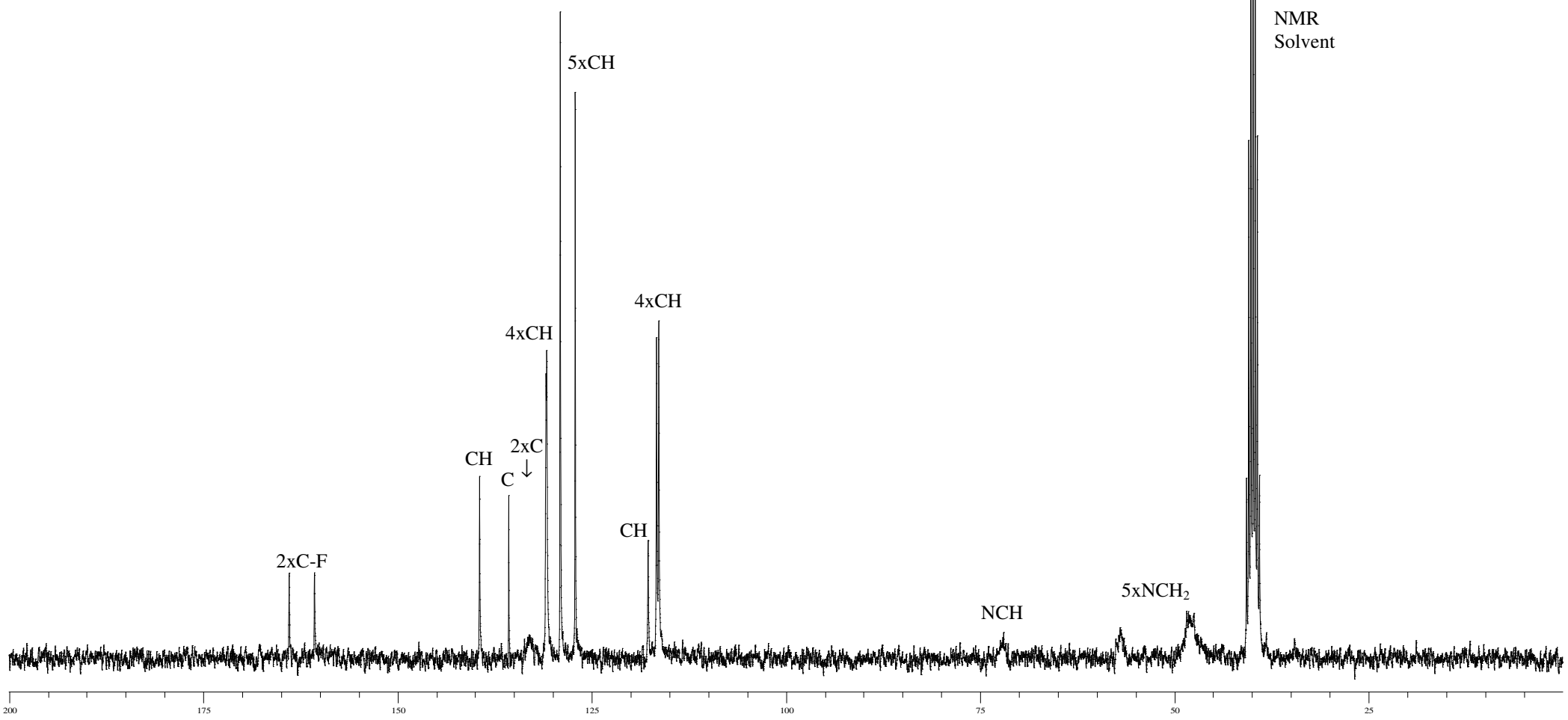
Carbon-13 NMR Spectrum of Flunarizine Dihydrochloride in DMSO-d_6



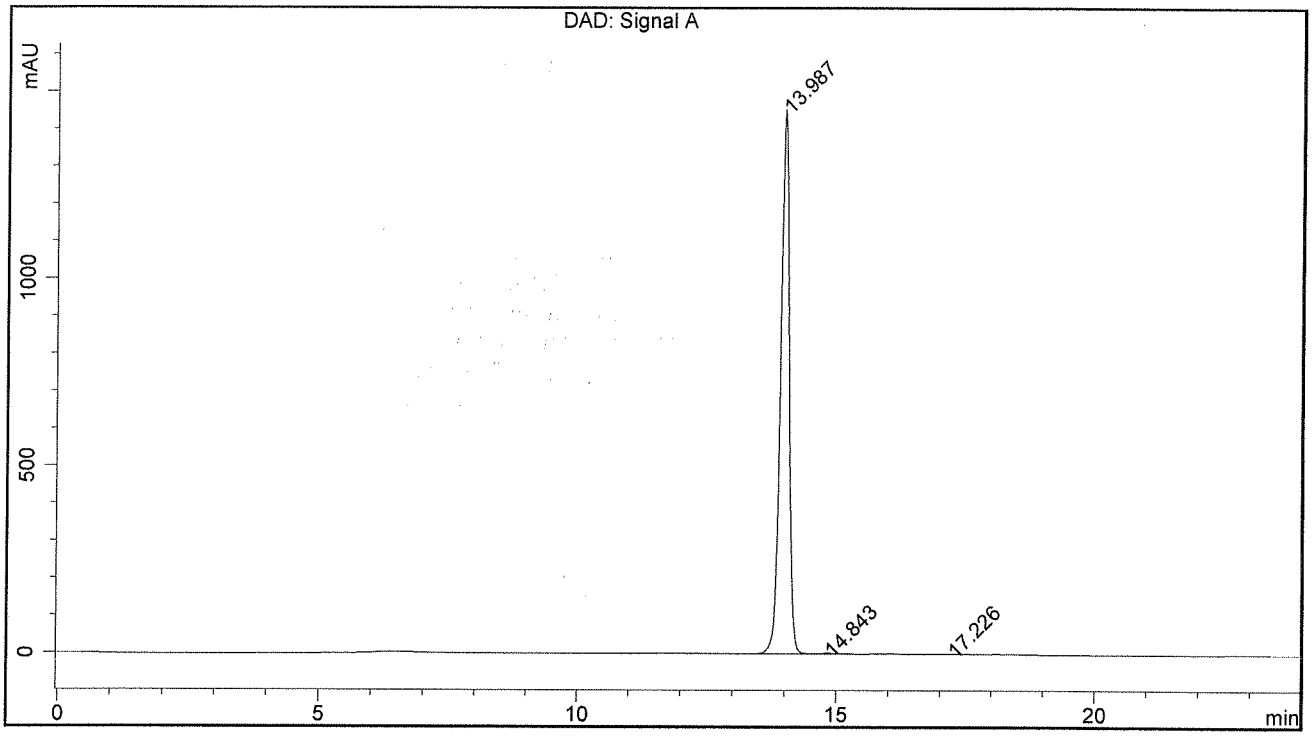
Lot Number: BDG 6030



BDG - Analysis of Flunarizine Dihydrochloride

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase A: 70:30 Ion Pair Reagent : Acetonitrile
Mobile Phase B: 30:70 Ion Pair Reagent : Acetonitrile
Ion Pair Reagent = 20mM Tetrabutylammonium Hydroxide pH=4.0 (HOAc) + 9mM Ammonium Acetate
Gradient (A:B) : T0=100:0, T1=100:0, T10=0:100, T16=0:100, T18=100:0, T24=100:0
Flow Rate : T0=1 ml/min, T1=1.5 mL/min
Sample Solvent : Initial Mobil Phase . . . . . Injection Volume : 10 uL
Column Temperature : 20C . . . . . Detection : UV at 230 nm

Sample Name	BDG 6030	**Instrument**	AnalyticalLC01
Acquisition	18/08/2014, 16:41:05	**Method (rev.)**	LC10557b (11)
Sequence	BDG_18Aug2014c - Reprocessed	**Vial Position**	36
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	13.99 min	1455.2355	16839.4977	0.1770 min	99.613 %
2	14.84 min	2.3311	54.1908	0.3192 min	0.321 %
3	17.23 min	0.6618	11.3117	0.2254 min	0.067 %