

## BDG SYNTHESIS

### Certificate of Analysis

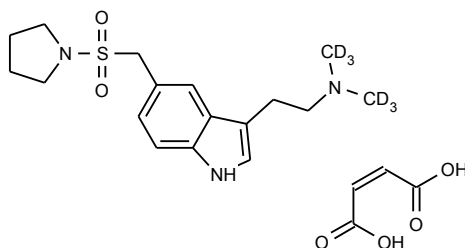
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

*Barry Dent*

Barry R. Dent, PhD, Director  
15 March 2005

**Name:** Almotriptan-d<sub>6</sub> Maleate  
**CAS Number:** 154323-57-6 (unlabelled free base)

**Structure:**



**Molecular Weight:**  $C_{17}H_{19}D_6N_3O_2S \cdot C_4H_4O_4 = 457.57$   
**Lot Number:** BDG 4736.5  
**Appearance:** Cream, crystalline solid  
**Corrected Purity:** 98.8 % (HPLC) - 1.3 % (ethanol) = 97.5 %  
**Isotopic Purity:** Under 0.5 % d<sub>0</sub>  
**Re-test Date:** 15 March 2010  
**Storage and Handling:** Temperature: ambient laboratory temperature; may be refrigerated.  
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.

Isotopic Labelling: signals at the sites of deuteration are absent, compared with what would be expected for unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of ethanol (1.3 % 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.

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  $m/z$  342.2111.  $C_{17}H_{20}D_6N_3O_2S$   $[M+H]^+$  requires  $m/z$  342.2116. The deviation of 1.5 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 (98.8 %). The peak at 2.62 minutes is assumed to be maleic acid and is not included in the integration. 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 55.06, H 5.17, D 2.70, N 9.08 %
$C_{17}H_{19}D_6N_3O_2S \cdot C_4H_4O_4$	Requires:	C 55.12, H 5.07, D 2.64, N 9.18 %

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).

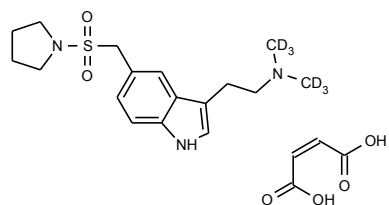
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

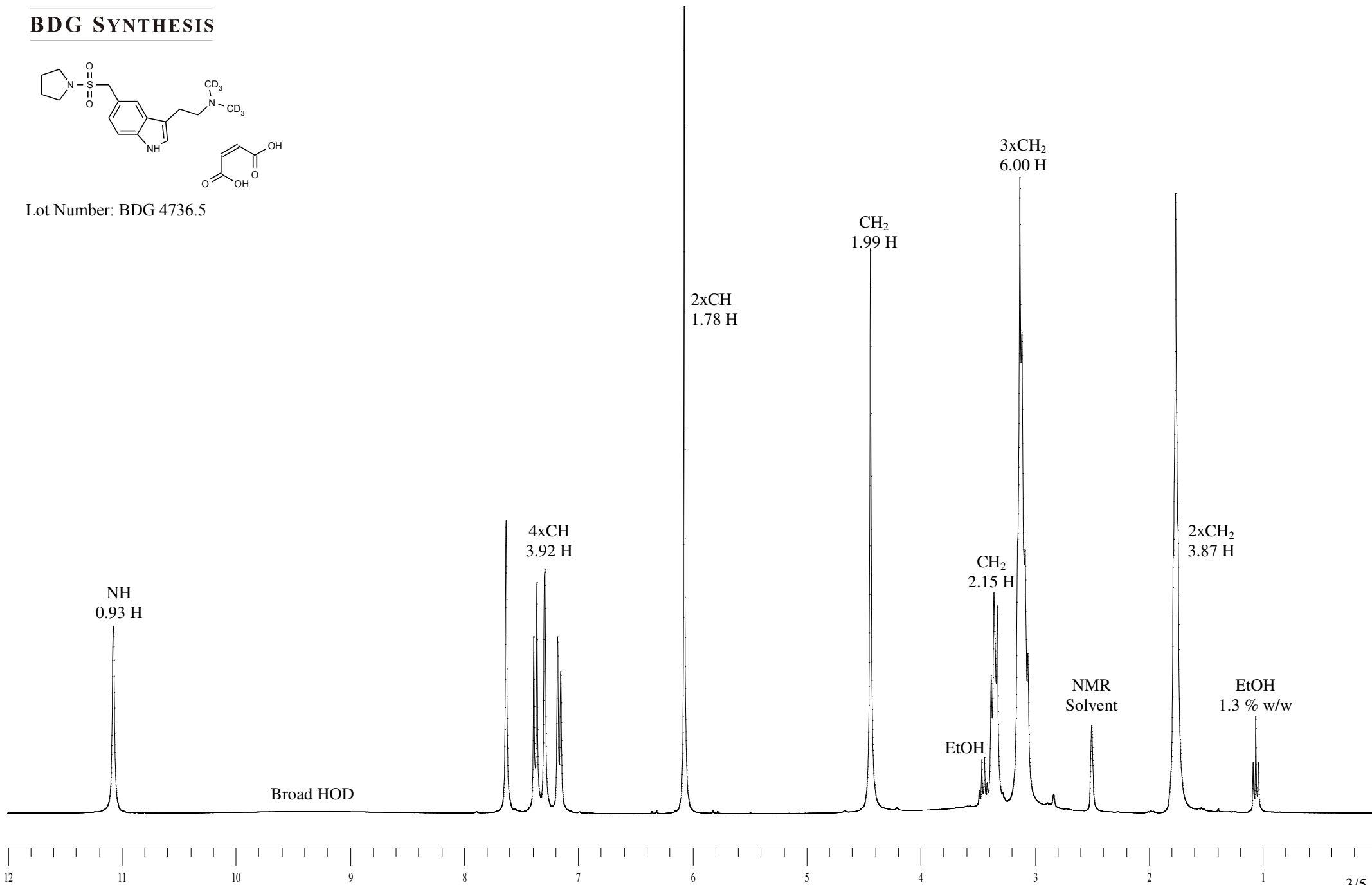


# Proton NMR Spectrum of Almotriptan-d<sub>6</sub> Maleate in DMSO-d<sub>6</sub>

## BDG SYNTHESIS



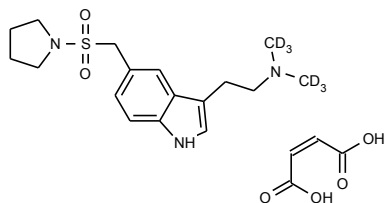
Lot Number: BDG 4736.5



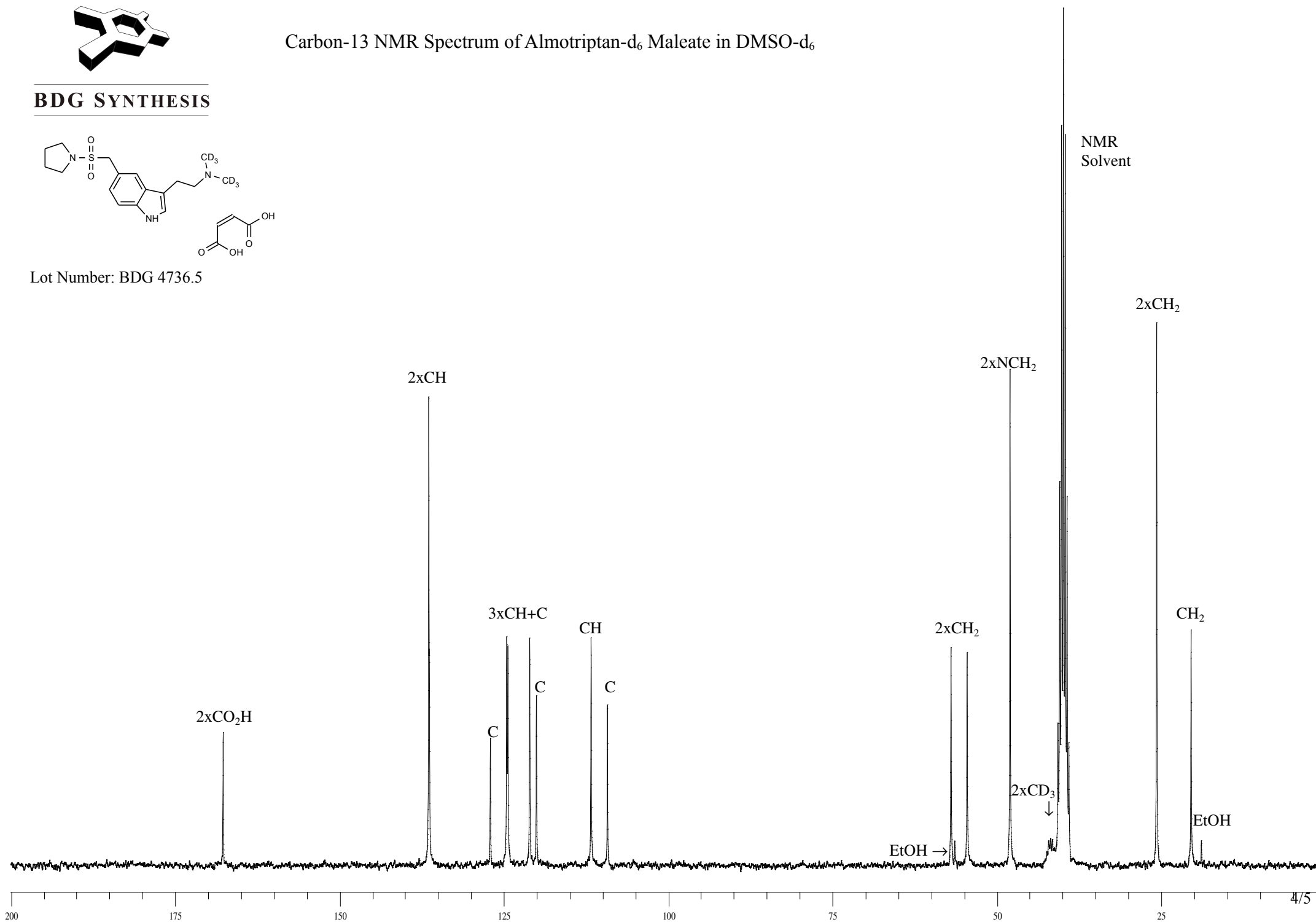


# Carbon-13 NMR Spectrum of Almotriptan-d<sub>6</sub> Maleate in DMSO-d<sub>6</sub>

## BDG SYNTHESIS



Lot Number: BDG 4736.5

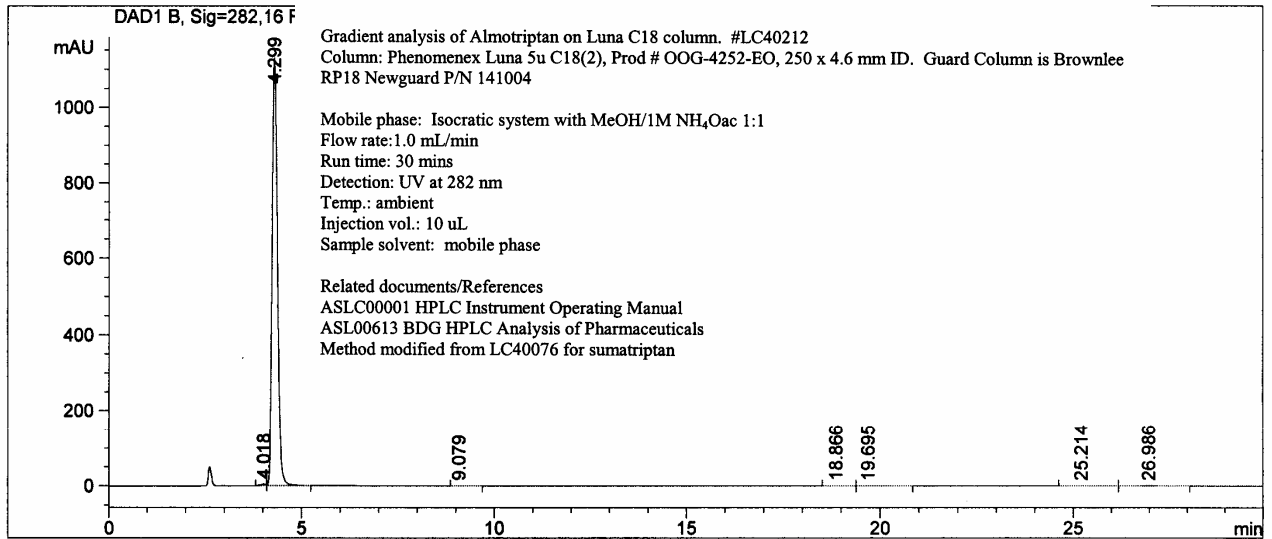


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Injection Date   : 3/14/05 6:40:47 PM           Seq. Line :    6
Sample Name     : BDG4736.5                     Location  : Vial 12
Acq. Operator  : YRLman                          Inj       :    1
                                                    Inj Volume: 10 µl

Acq. Method    : N:\LC1100_2\1\METHODS\LC40212A.M
Last changed   : 3/14/05 4:23:49 PM by YRLman
Analysis Method: N:\LC1100_2\1\METHODS\LC40212A.M
Last changed   : 3/15/05 10:08:00 AM by YRLman
                (modified after loading)
    
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Isocratic analysis of almotriptan-d6 maleate on Luna C18 with MeOH/ammonium acetate, # LC40212



Area Percent Report

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Sorted By      :      Signal
Multiplier    :      1.0000
Dilution      :      1.0000
    
```

Signal 1: DAD1 B, Sig=282,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.018	MF	0.1686	49.29826	4.87403	0.4448
2	4.299	FM	0.1615	1.09473e4	1129.72534	98.7650
3	9.079	MM	0.3664	6.43112	2.92558e-1	0.0580
4	18.866	MF	0.4838	8.81194	3.03561e-1	0.0795
5	19.695	FM	0.7755	6.68362	1.43632e-1	0.0603
6	25.214	MF	0.7023	24.19116	5.74108e-1	0.2182
7	26.986	FM	0.7514	41.47004	9.19821e-1	0.3741

Totals : 1.10842e4 1136.83305

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*