



## BDG SYNTHESIS

### Certificate of Analysis

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

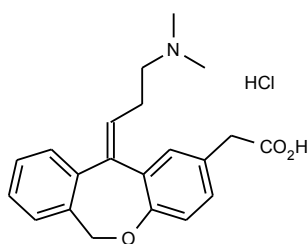
*Barry Dent*

Barry R. Dent, PhD, Director  
13 February 2014

**Name:** Olopatadine HCl

**CAS Number:** 140462-76-6

**Structure:**



**Molecular Weight:**  $C_{21}H_{23}NO_3 \cdot HCl = 373.87$

**Lot Number:** BDG 10881.1

**Appearance:** White, crystalline solid

**Purity By HPLC:** 99.9 %

**Re-test Date:** 13 February 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.

## 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 acetone 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  $m/z$  338.1759.  $C_{21}H_{24}NO_3$   $[M+H]^+$  requires  $m/z$  338.1756. The deviation of 0.9 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, slightly tailing peak is observed (99.9 %). 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

$C_{21}H_{23}NO_3 \cdot HCl$	Found:	C 67.74, H 6.56, N 3.72 %
	Requires:	C 67.46, H 6.47, N 3.75 %

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).

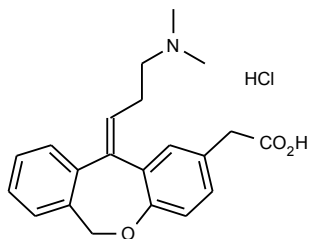
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

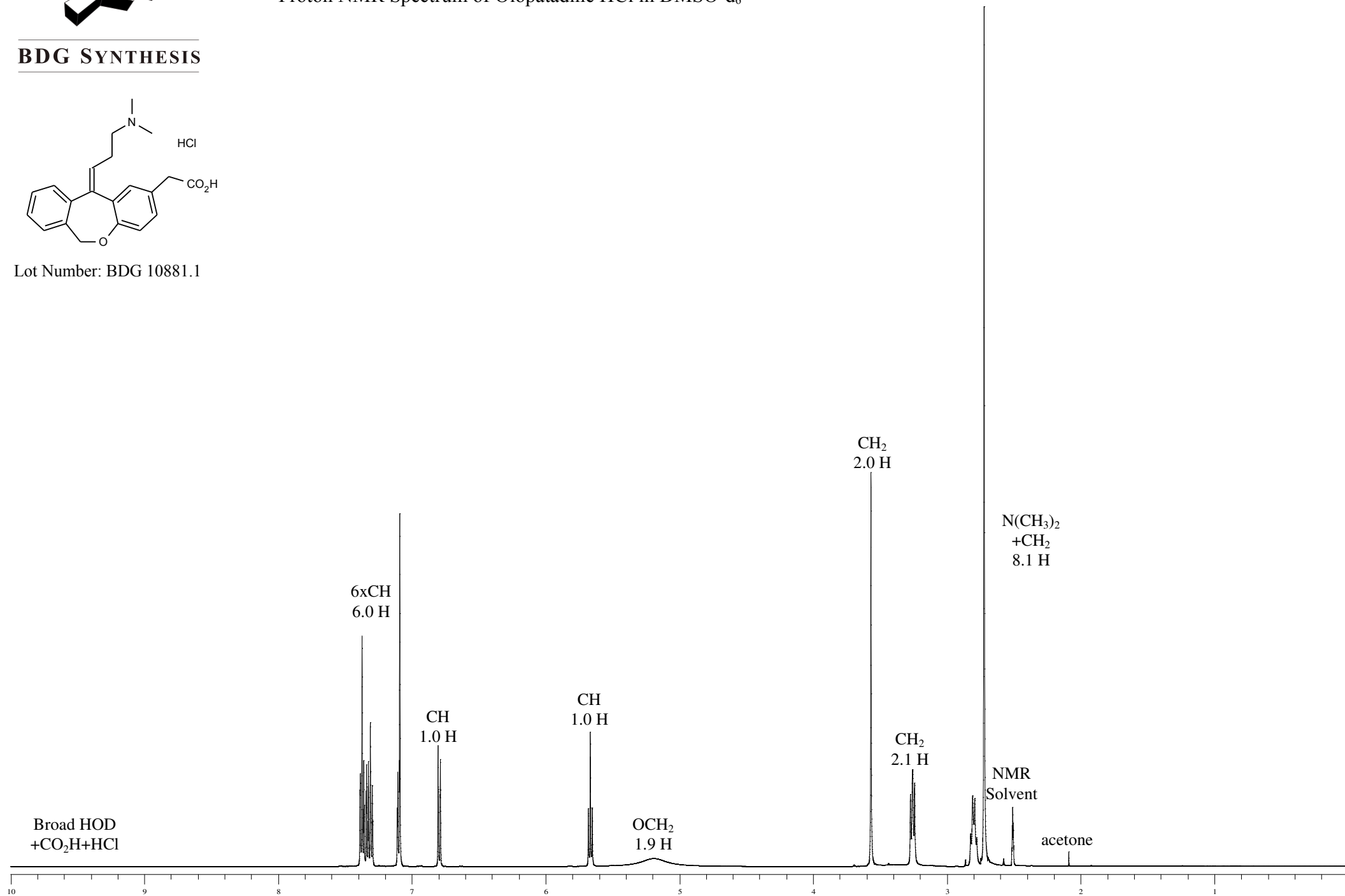


Proton NMR Spectrum of Olopatadine HCl in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



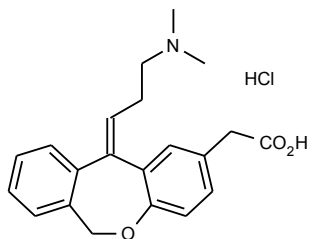
Lot Number: BDG 10881.1



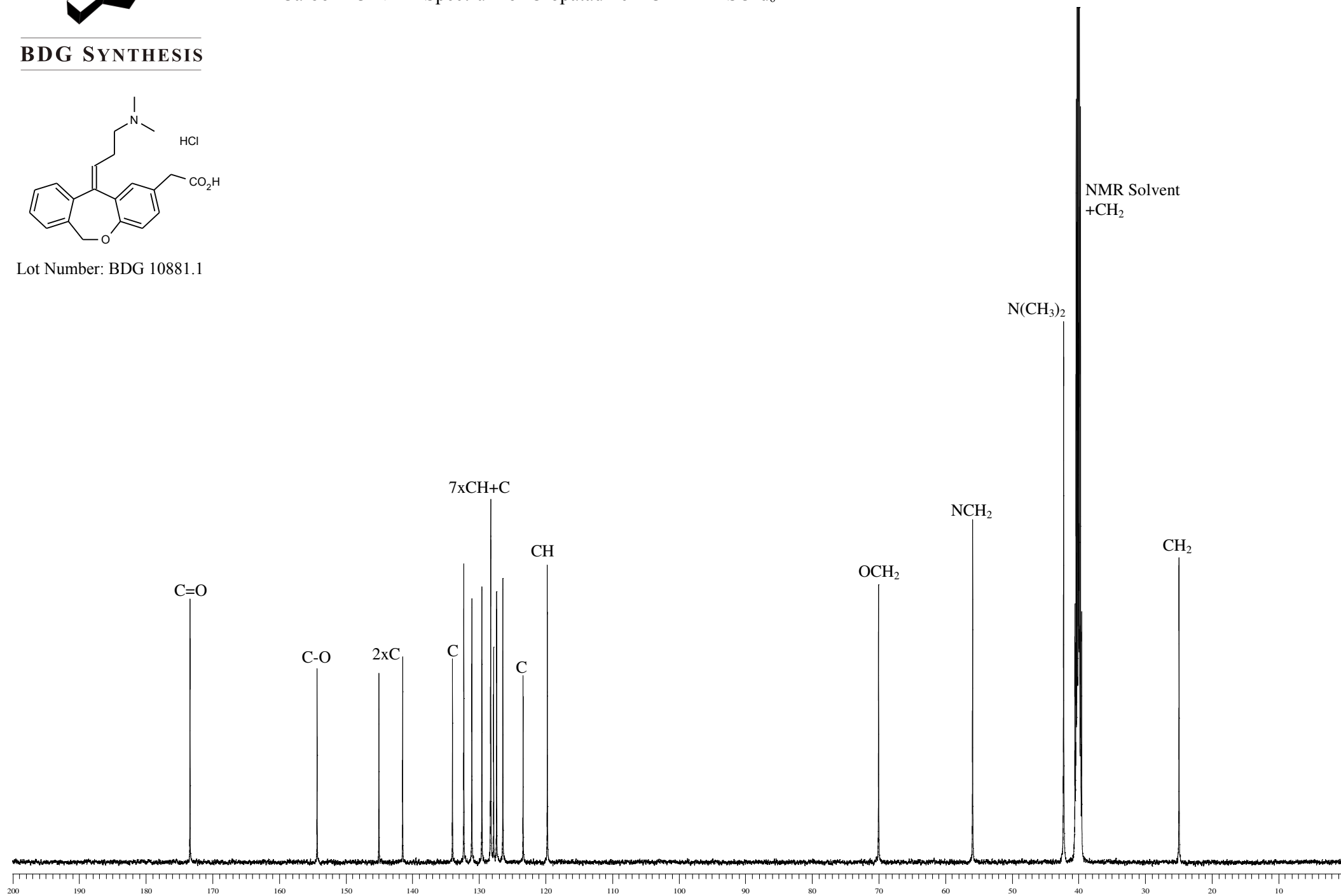


Carbon-13 NMR Spectrum of Olopatadine HCl in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



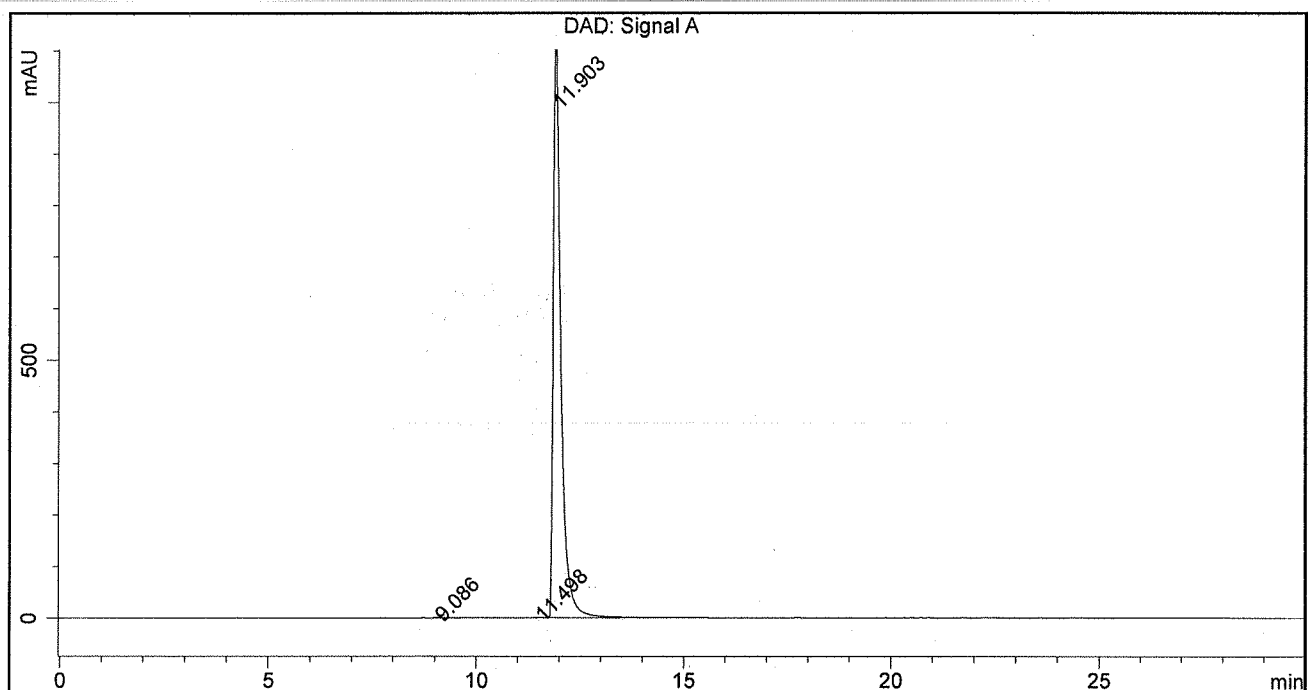
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BDG - Analysis of olopatadine derivatives

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase A : 10 mM diPotassium Hydrogen Phosphate pH = 7.0  
 Mobile Phase B : Acetonitrile  
 Gradient ( A:B ) : T0=80:20, T20=50:50, T24=50:50, T27=80:20, T30=80:20  
 Flow Rate : 1.0 mL/min . . . . . Sample Solvent : 2:1 Water : Acetonitrile  
 Column Temperature : 20C . . . . . Injection Volume : 10 uL . . . . . Detection : UV at 254 nm

Sample Name	BDG 10881.1	Instrument	AnalyticalLC01
Acquisition	13/02/2014, 11:55:20	Method (rev.)	LC10016I ( 5)
Sequence	BDG_13Feb2014a - Reprocessed	Vial Position	1
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	9.09 min	0.7003	9.0493	0.2008 min	0.062 %
2	11.50 min	0.4439	3.9361	0.1234 min	0.027 %
3	11.90 min	1142.6964	14621.8651	0.1893 min	99.911 %