



## BDG SYNTHESIS

### Certificate of Analysis

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

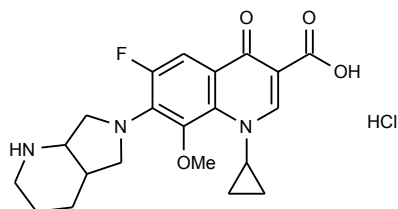
*Barry Dent*

Barry R. Dent, PhD, Director  
3 July 2013

**Name:** Moxifloxacin HCl

**CAS Number:** 186826-86-8

**Structure:**



**Molecular Weight:**  $C_{21}H_{24}FN_3O_4 \cdot HCl = 437.89$

**Lot Number:** BDG 10180.1

**Appearance:** Pale yellow, crystalline solid

**Corrected Purity:** 99.9 % (HPLC) - 4.1 % (water) = 95.8 %

**Re-test Date:** 3 July 2018

**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:       | store in an amber vial and protect from bright light.                                 |
| 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: no residual solvents are 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. A methine signal is obscured by the NMR solvent.

### High-resolution Mass Spectrum (ESI+)

Found  $m/z$  402.1815.  $C_{21}H_{25}FN_3O_4$   $[M+H]^+$  requires  $m/z$  402.1824. The deviation of 2.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A sharp, symmetrical 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

|   |           |                           |
|---|-----------|---------------------------|
|   | Found:    | C 55.84, H 5.76, N 9.23 % |
| $C_{21}H_{24}FN_3O_4 \cdot HCl \cdot 0.8H_2O$ | Requires: | C 55.76, H 5.93, N 9.29 % |
| $C_{21}H_{24}FN_3O_4 \cdot HCl$               | Requires: | C 57.60, H 5.75, N 9.60 % |

The elemental analyses fall somewhat 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 <sub>2</sub> O 4.1 % |
| $C_{21}H_{24}FN_3O_4 \cdot HCl \cdot 0.8H_2O$ | Requires: | H <sub>2</sub> O 3.2 % |

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.

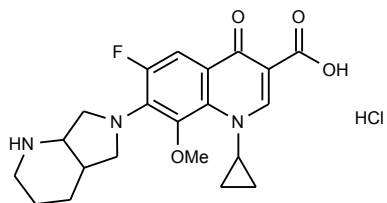
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

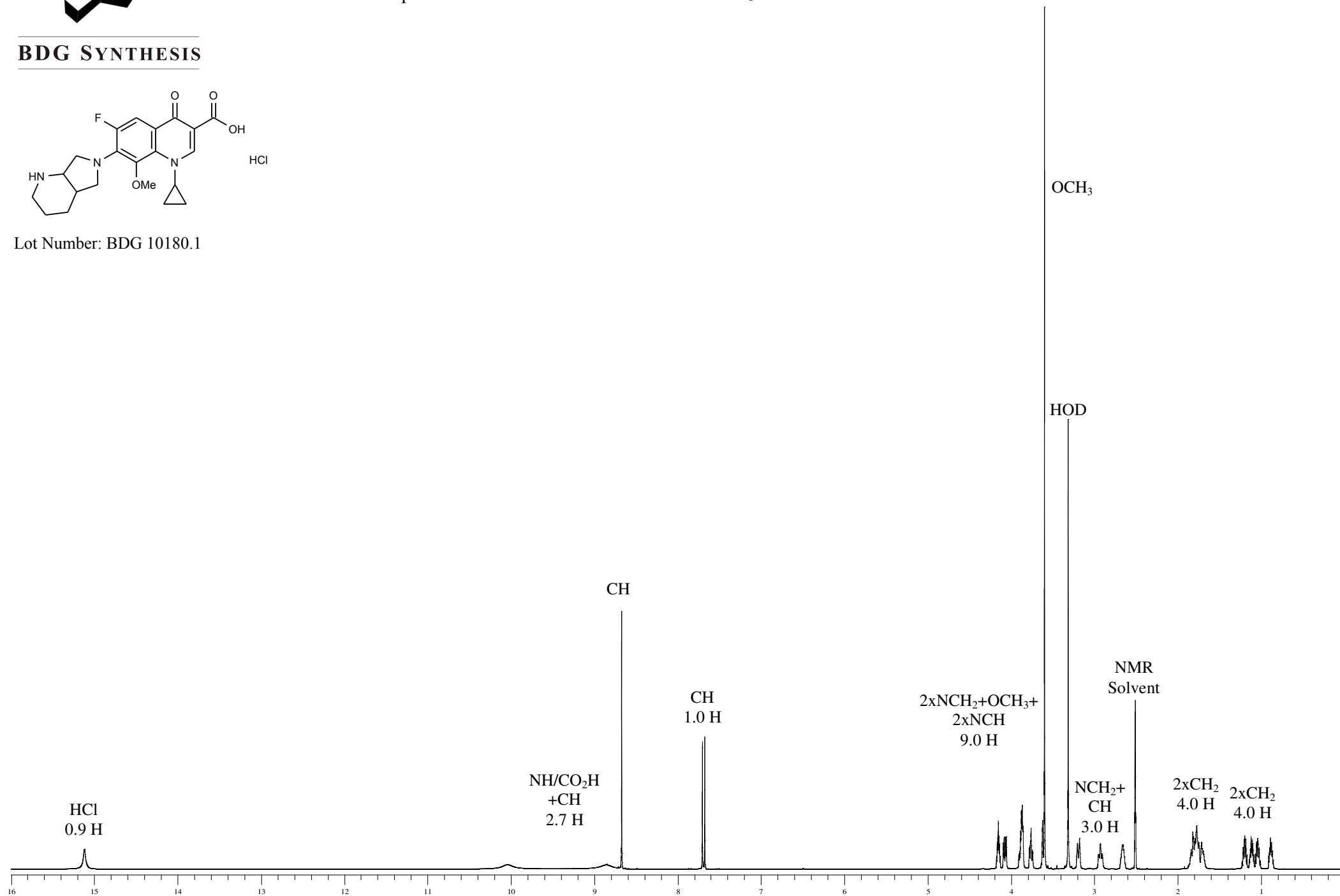


Proton NMR Spectrum of Moxifloxacin HCl in DMSO-d<sub>6</sub>

**BDG SYNTHESIS**



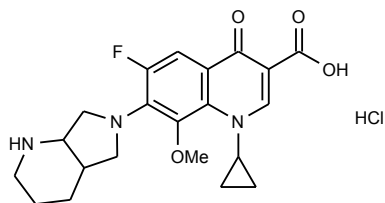
Lot Number: BDG 10180.1



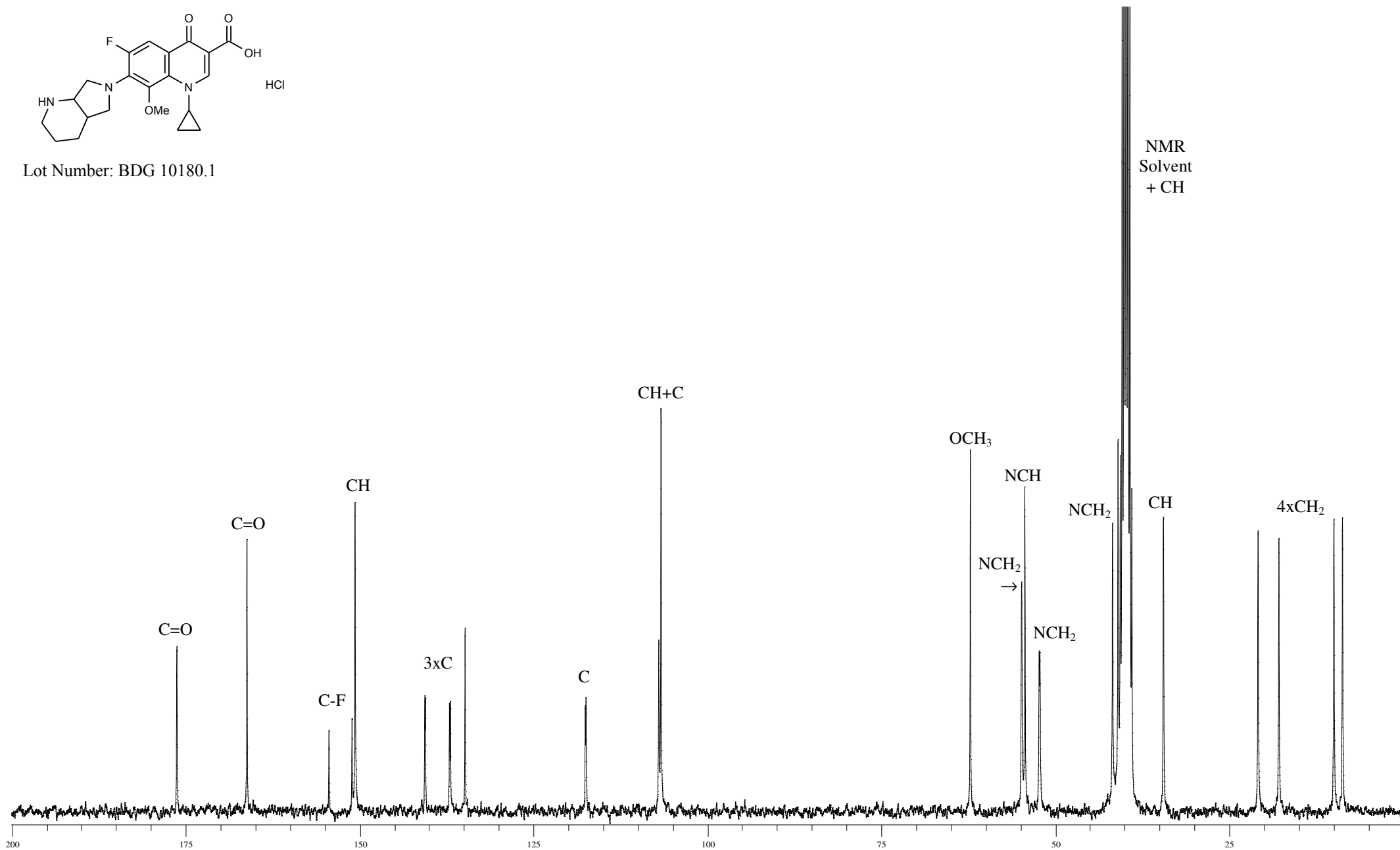


# Carbon-13 NMR Spectrum of Moxifloxacin HCl in DMSO-d<sub>6</sub>

## BDG SYNTHESIS



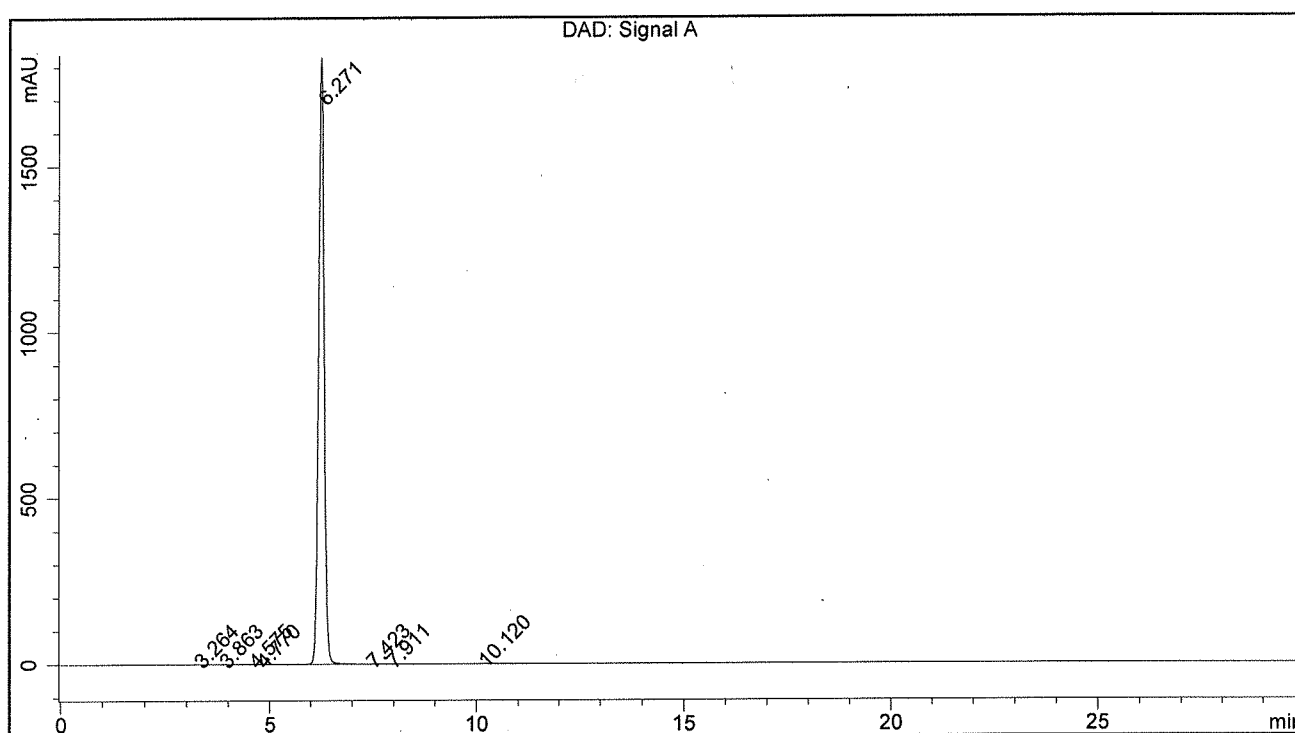
Lot Number: BDG 10180.1



BDG - Analysis of Moxifloxacin Hydrochloride

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm  
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm  
 Mobile Phase : 50:50 25 mM Potassium diHydrogen Phosphate Buffer pH = 3.0, then 10 mM Sodium Dodecyl Sulphate : Acetonitrile  
 Flow Rate : 1 mL/min  
 Sample Solvent : Mobile Phase  
 Column Temperature : 20C  
 Injection Volume : 10 uL  
 Detection : UV at 296 nm

|                    |                          |                      |                |
|--------------------|--------------------------|----------------------|----------------|
| <b>Sample Name</b> | BDG 10180.1              | <b>Instrument</b>    | AnalyticalLC01 |
| <b>Acquisition</b> | 03/07/2013, 16:31:49     | <b>Method (rev.)</b> | LC10082a ( 11) |
| <b>Sequence</b>    | BDG_03Jul2013b           | <b>Vial Position</b> | 1              |
| <b>Operator</b>    | solvation010\cerityadmin | <b>Injection</b>     | 1 of 1         |



Area Percent Report

| Peak# | RT        | Peak Height | Peak Area  | Width      | Area %   |
|-------|-----------|-------------|------------|------------|----------|
| 1     | 3.26 min  | 0.6014      | 5.0758     | 0.1245 min | 0.030 %  |
| 2     | 3.86 min  | 0.2429      | 2.8505     | 0.1549 min | 0.017 %  |
| 3     | 4.57 min  | 0.1433      | 1.0584     | 0.1062 min | 0.006 %  |
| 4     | 4.77 min  | 0.1389      | 1.0188     | 0.0996 min | 0.006 %  |
| 5     | 6.27 min  | 1827.5946   | 16986.2370 | 0.1424 min | 99.892 % |
| 6     | 7.42 min  | 0.1908      | 1.8232     | 0.1235 min | 0.011 %  |
| 7     | 7.91 min  | 0.2775      | 2.7850     | 0.1291 min | 0.016 %  |
| 8     | 10.12 min | 0.3154      | 3.7511     | 0.1527 min | 0.022 %  |