



## BDG SYNTHESIS

### Certificate of Analysis

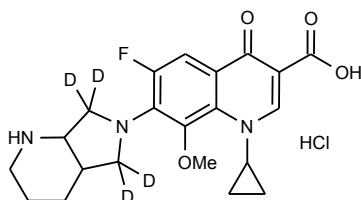
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director  
10 January 2009

**Name:** Moxifloxacin-d<sub>4</sub> HCl  
**CAS Number:** 186826-86-8 (unlabelled)

**Structure:**



**Molecular Weight:** C<sub>21</sub>H<sub>20</sub>D<sub>4</sub>FN<sub>3</sub>O<sub>4</sub>·HCl = 441.92  
**Lot Number:** BDG 10386.1  
**Appearance:** Yellow, crystalline solid  
**Corrected Purity:** 99.4 % (HPLC) - 1.1 % (ethanol) - 0.5 % (diethyl ether) - 5.4 % (water) = 92.4 %  
**Isotopic Purity:** Under 0.5 % d<sub>0</sub>  
**Re-test Date:** 10 January 2014  
**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

### 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 the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: small amounts of diethyl ether (0.5 % w/w) and ethanol (1.1 % w/w) 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.

Isotopic Labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

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

Found  $m/z$  406.2064.  $C_{21}H_{21}D_4FN_3O_4$   $[M+H]^+$  requires  $m/z$  406.2075. The deviation of 2.8 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 (99.4 %). 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 54.04, H 4.94, D 1.88, N 8.72 %
$C_{21}H_{20}D_4FN_3O_4 \cdot HCl \cdot 1.4H_2O$	Requires:	C 53.99, H 5.14, D 1.72, N 9.00 %, $H_2O$ 5.40 %
$C_{21}H_{20}D_4FN_3O_4 \cdot HCl$	Requires:	C 57.08, H 4.79, D 1.82, N 9.51 %

The elemental analyses fall substantially 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. In the absence of a Karl-Fischer water analysis, we recommend that the “best-fit” water content be used when determining corrected purity.

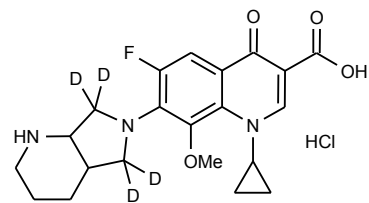
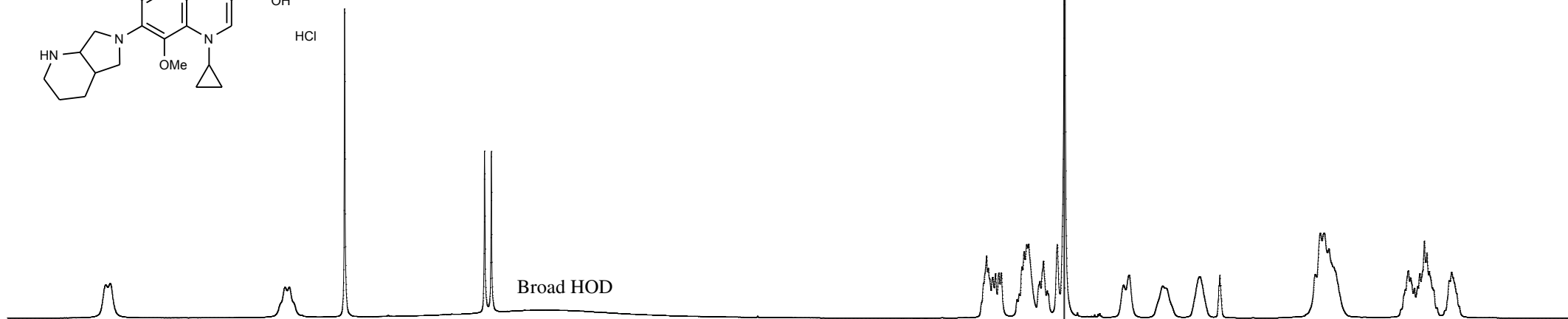
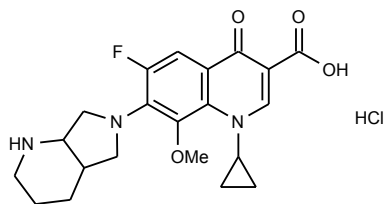
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

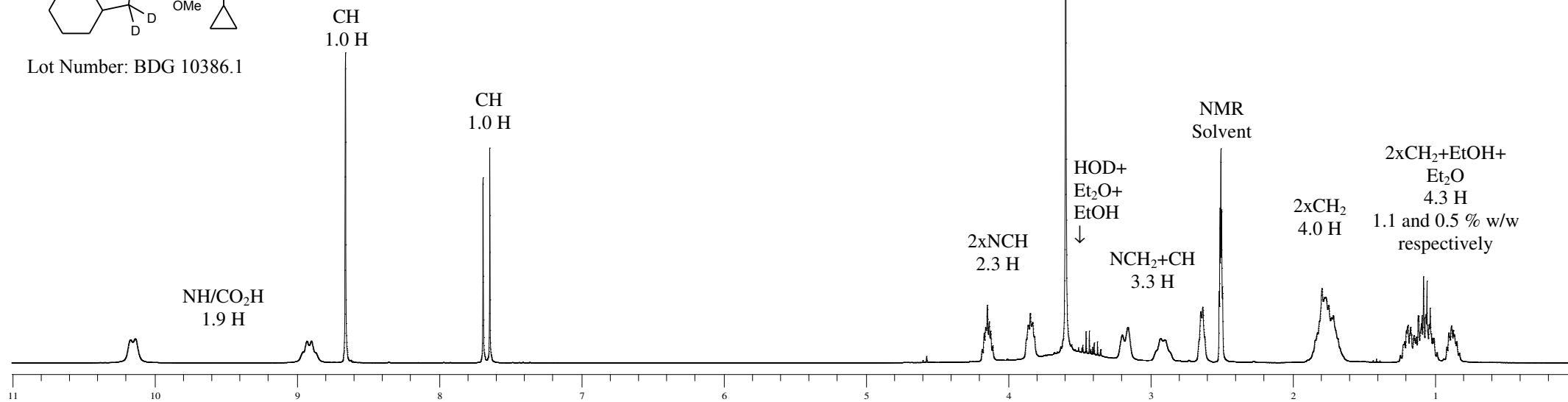


Proton NMR Spectrum of Moxifloxacin HCl (top) and Moxifloxacin-d<sub>4</sub> HCl (bottom) in DMSO-d<sub>6</sub>

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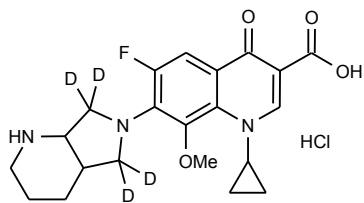
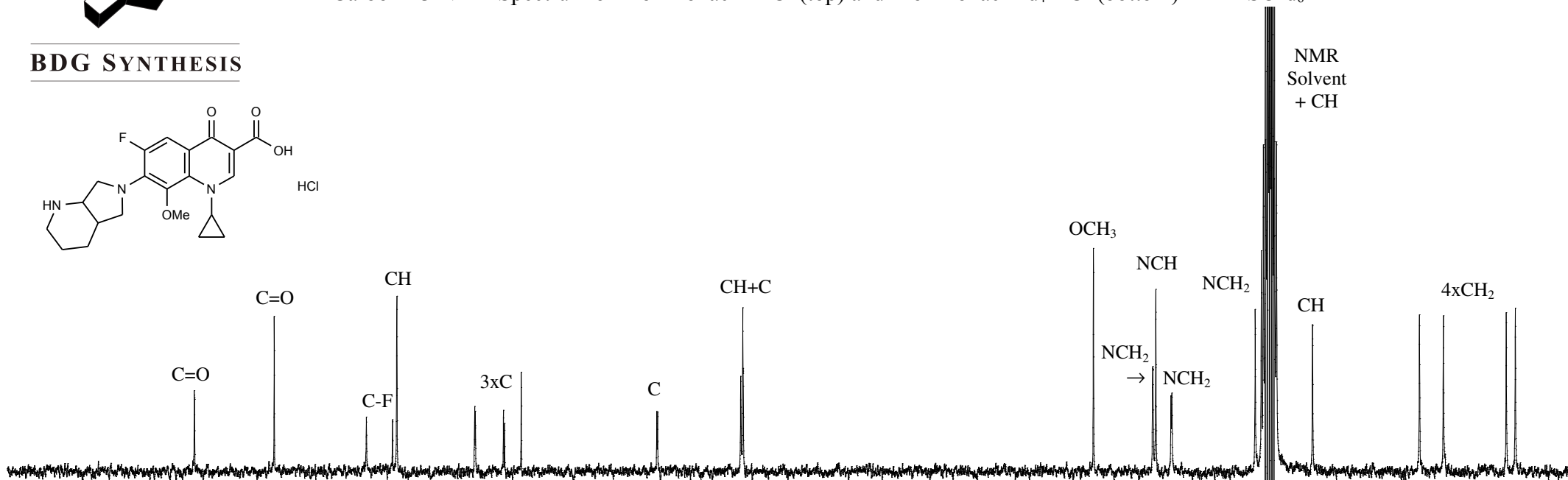
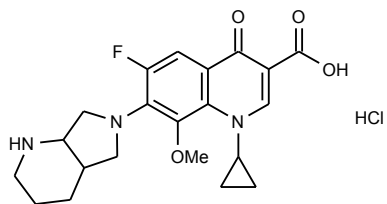
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Carbon-13 NMR Spectrum of Moxifloxacin HCl (top) and Moxifloxacin-d<sub>4</sub> HCl (bottom) in DMSO-d<sub>6</sub>

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