BDG SYNTHESIS

Certificate of Analysis

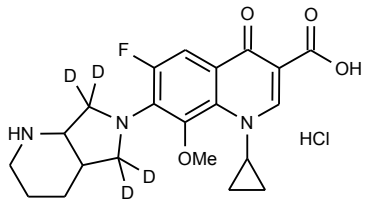
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
8 March 2016

Name: **Moxifloxacin-d_4 HCl**

CAS Number: 186826-86-8 (unlabelled)

Structure:



Molecular Weight: $C_{21}H_{20}D_4FN_3O_4 \cdot HCl = 441.92$

Lot Number: BDG 10383.1-01

Appearance: Yellow, crystalline solid

Corrected Purity: 99.6 % (HPLC) - 1.1 % (ethanol) - 4.7 % (water) = 93.8 %

Isotopic Purity: Under 0.5 % d_0

Re-test Date: 8 March 2021

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.

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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.
Isotopic Labelling: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.
Residual Solvents: a small amount of ethanol (1.1 % *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 the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 406.2072. $C_{21}H_{21}D_4FN_3O_4$ $[M+H]^+$ requires *m/z* 406.2075. The deviation of 0.6 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 (99.6 %). 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 54.39, H 4.94, D 1.88, N 8.95 %
$C_{21}H_{20}D_4FN_3O_4{\cdot}HCl{\cdot}1.2H_2O$	Requires:	C 54.41, H 5.09, D 1.74, N 9.07 %, H_2O 4.66 %
$C_{21}H_{20}D_4FN_3O_4{\cdot}HCl$	Requires:	C 57.08, H 4.79, D 1.82, N 9.51 %

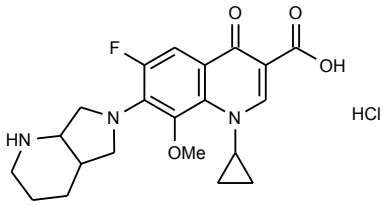
The elemental analyses fall substantially outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the “best-fit” hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the “best-fit” water content be used when determining corrected purity.

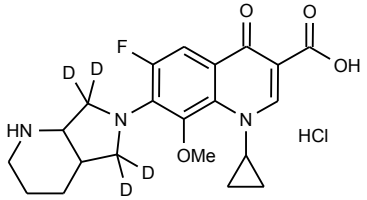
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

BDG SYNTHESIS

Proton NMR Spectrum of Moxifloxacin HCl (top) and Moxifloxacin-d_4 HCl (bottom) in DMSO-d_6

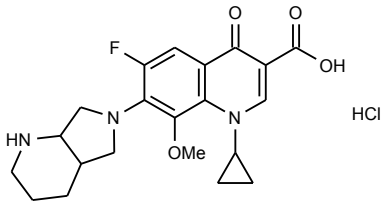


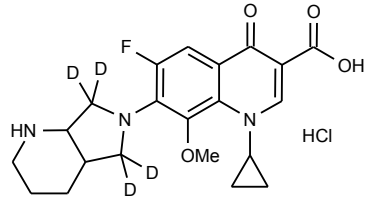


Lot Number: BDG 10383.1

BDG SYNTHESIS

Carbon-13 NMR Spectrum of Moxifloxacin HCl (top) and Moxifloxacin-d_4 HCl (bottom) in DMSO-d_6



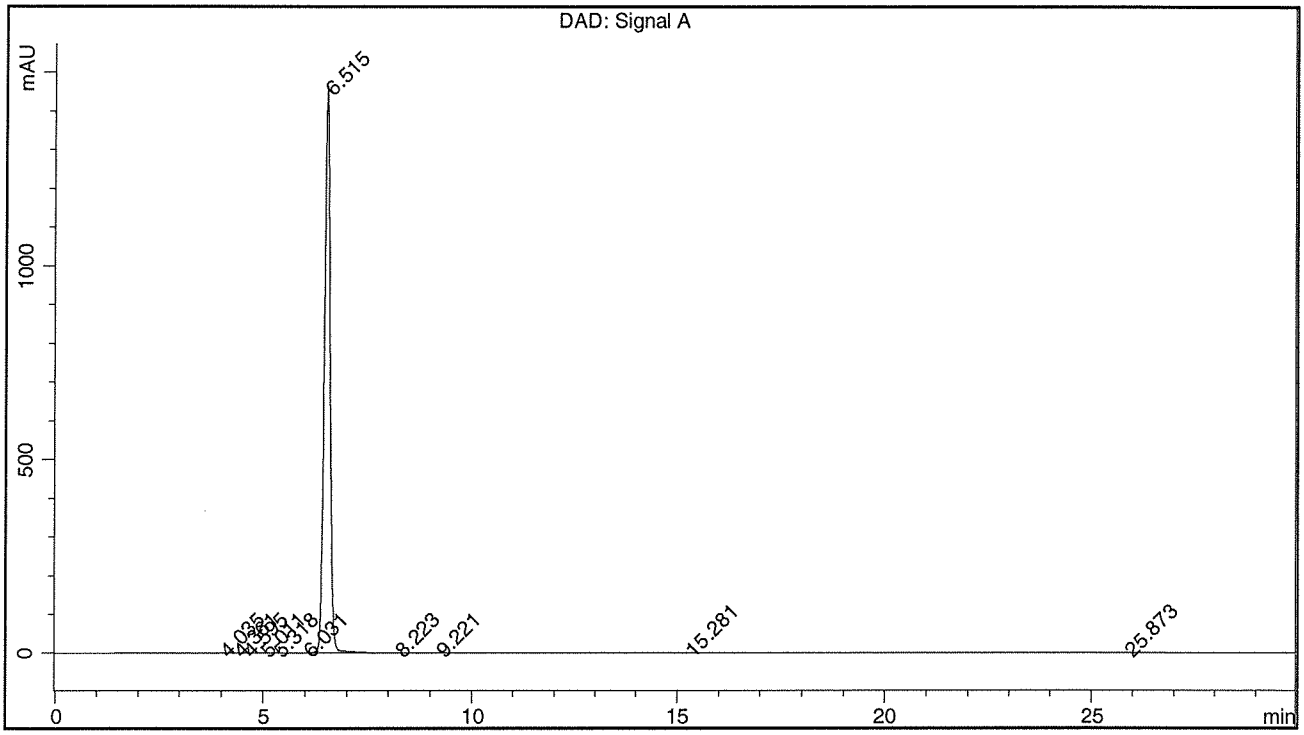


Lot Number: BDG 10383.1

BDG - Analysis of Moxifloxacin-d4 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
Column Temperature : 20 C . . . . . Flow Rate : 1 mL/min . . . . . Sample Solvent : Mobile Phase
Injection Volume : 10 uL . . . . . Detection : UV at 296 nm

Sample Name	BDG 10383.1	**Instrument**	AnalyticalLC01
Acquisition	08/03/2016, 15:31:23	**Method (rev.)**	LC10082b (9)
Sequence	BDG_08Mar2016a - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	4.03 min	0.1667	1.3561	0.1349 min	0.010 %
2	4.36 min	0.2477	2.6166	0.1472 min	0.018 %
3	4.59 min	0.7687	6.5599	0.1276 min	0.046 %
4	5.01 min	0.2261	1.7756	0.1236 min	0.012 %
5	5.32 min	0.4678	5.2531	0.1750 min	0.037 %
6	6.03 min	0.2684	2.3531	0.1383 min	0.017 %
7	6.52 min	1462.9376	14180.1131	0.1491 min	99.559 %
8	8.22 min	0.6838	12.2630	0.2572 min	0.086 %
9	9.22 min	0.3052	3.9671	0.1717 min	0.028 %
10	15.28 min	0.2157	4.8855	0.2802 min	0.034 %
11	25.87 min	0.1838	21.8096	1.6081 min	0.153 %