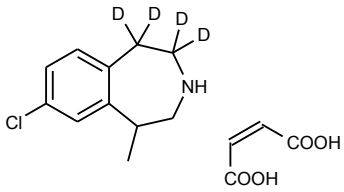
BDG SYNTHESIS

# Certificate of Analysis

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

Neil Beare, PhD, Director
31 August 2014

| | |
|---|---|
| **Name:** | **Lorcaserin-$d_4$ Maleate** |
| **CAS Number:** | 616202-92-7 (unlabelled free base) |
| **Structure:** |  |
| **Molecular Weight:** | $C_{11}H_{10}D_4ClN \cdot C_4H_4O_4$ = 315.79 |
| **Lot Number:** | BDG 9276 |
| **Appearance:** | White, crystalline solid |
| **Corrected Purity:** | 99.1 % (HPLC) - 0.6 % (diethyl ether) = 98.5 % |
| **Isotopic Purity:** | Under 0.5 % $d_0$ |
| **Re-test Date:** | 31 August 2019 |

**Storage and Handling:**

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|---|---|
| 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.
Isotopic Labelling: signals at the site of deuteration are greatly diminished, compared with what would be expected for unlabelled material. Some H/D exchange resulted in a small signal for residual CH at 3.2 ppm.
Residual Solvents: a small amount of diethyl ether (0.6 % *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 site of deuteration have collapsed to small multiplets compared with what would be expected for unlabelled material.

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

Found *m/z* 200.1139. $C_{11}H_{11}D_4ClN$ $[M+H]^+$ requires *m/z* 200.1144. The deviation of 2.5 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 %). A small signal is observed for $d_3$ material.

### HPLC

A somewhat broadened, tailing peak is observed (99.1 %). The peak at 3.2 min has been identified as Maleic acid.
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 57.05, H 4.39, D 2.51, N 4.41 % |
| $C_{11}H_{10}D_4ClN{\cdot}C_4H_4O_4$ | Requires: | C 57.05, H 4.47, D 2.55, N 4.44 % |

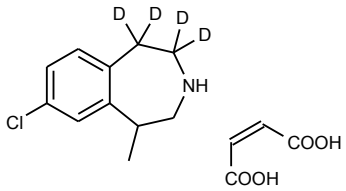
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

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.

BDG SYNTHESIS

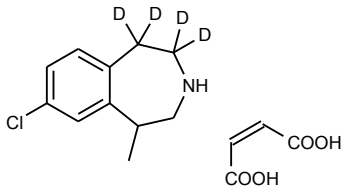
Proton NMR Spectrum of Lorcaserin-$d_4$ Maleate in Methanol-$d_4$




Lot Number: BDG 9276

BDG SYNTHESIS

# Carbon-13 NMR Spectrum of Lorcaserin-$d_4$ Maleate in Methanol-$d_4$

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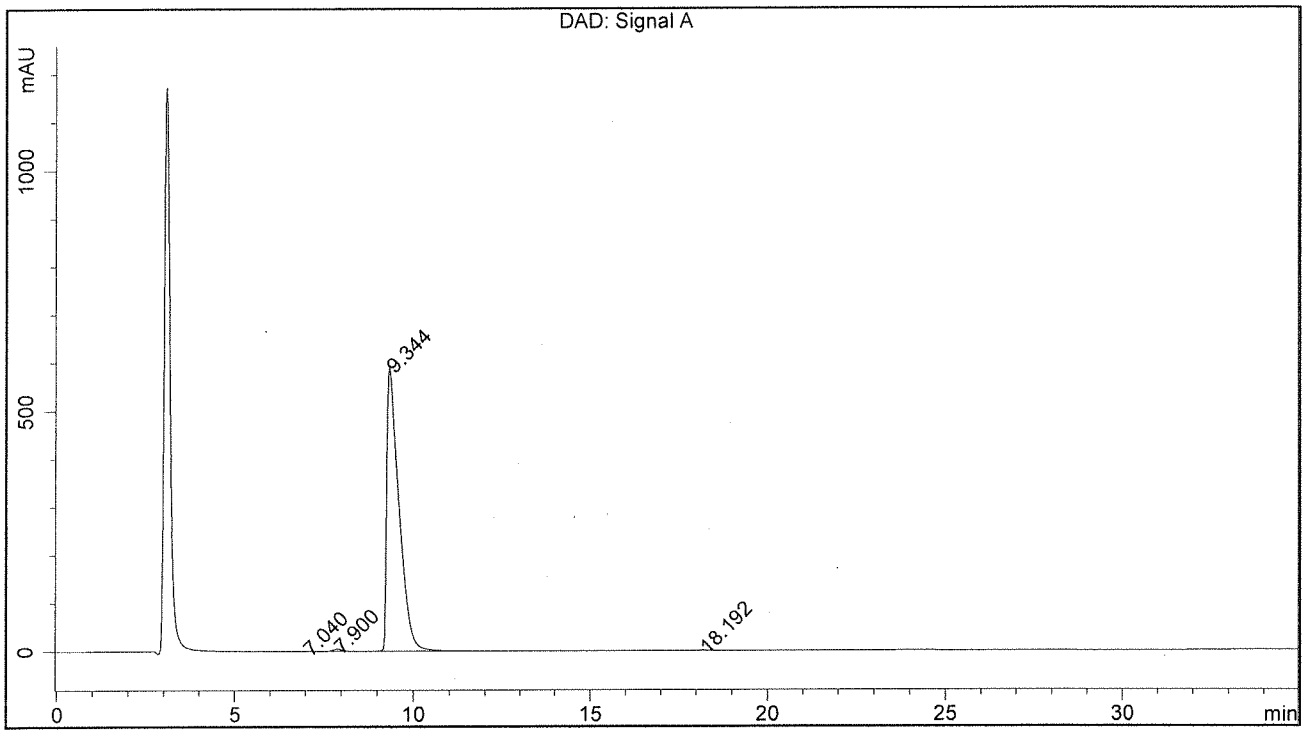
BDG - Analysis of Lorcaserin-d4 maleate

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 75:25:0.05 Water : Acetonitrile : Trifluoroacetic Acid
Flow Rate : 1.0 mL/min
Sample Solvent : 80:20 Water : Acetonitrile
Injection Volume : 10 uL
Detection: UV 218 nm

| Sample Name | BDG 9276 | Instrument | AnalyticalLC01 |
|---|---|---|---|
| Acquisition | 31/08/2014, 18:18:17 | Method (rev.) | LC10626b ( 18) |
| Sequence | BDG_31Aug2014g - Reprocessed | Vial Position | 21 |
| Operator | solvation010\cerityadmin | Injection | 2 of 2 |




**Area Percent Report**

| Peak# | RT | Peak Height | Peak Area | Width | Area % |
|---|---|---|---|---|---|
| 1 | 7.04 min | 0.3744 | 5.3386 | 0.2104 min | 0.038 % |
| 2 | 7.90 min | 4.7179 | 81.0810 | 0.2605 min | 0.575 % |
| 3 | 9.34 min | 587.3786 | 13984.6831 | 0.3418 min | 99.132 % |
| 4 | 18.19 min | 1.1440 | 35.9924 | 0.4031 min | 0.255 % |