BDG SYNTHESIS

# Certificate of Analysis

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

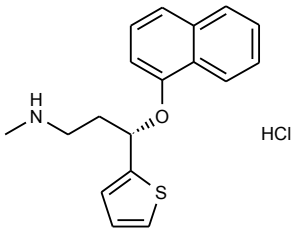
Barry Dent

Barry R. Dent, PhD, Director
6 August 2013

**Name:** **Duloxetine HCl**

**CAS Number:** 136434-34-9

**Structure:**




**Molecular Weight:** $C_{18}H_{19}NOS \cdot HCl = 333.88$

**Lot Number:** BDG 9124

**Appearance:** White, crystalline solid

**Corrected Purity:** 99.7 % (HPLC) - 1.7 % (2-propanol) = 98.0 %

**Re-test Date:** 6 August 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: | protect from strong sunlight. |
| Caution: | only experienced laboratory personnel should handle the material. |



• Analytical reference standards, metabolites, stable isotope labelled compounds • Custom synthesis • FTE contract research

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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.
Residual Solvents: a small amount of 2-propanol (1.7 % *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.

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

Found *m/z* 298.1272. $C_{18}H_{20}NOS$ $[M+H]^+$ requires *m/z* 298.1266. The deviation of 2.0 ppm is within normally accepted limits for the establishment of identity by HRMS.

### HPLC

A somewhat broadened, symmetrical peak is observed (99.7 %). 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 64.95, H 6.09, N 4.21 % |
| $C_{18}H_{19}NOS{\cdot}HCl$ | Requires: | C 64.75, H 6.04, N 4.20 % |

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.

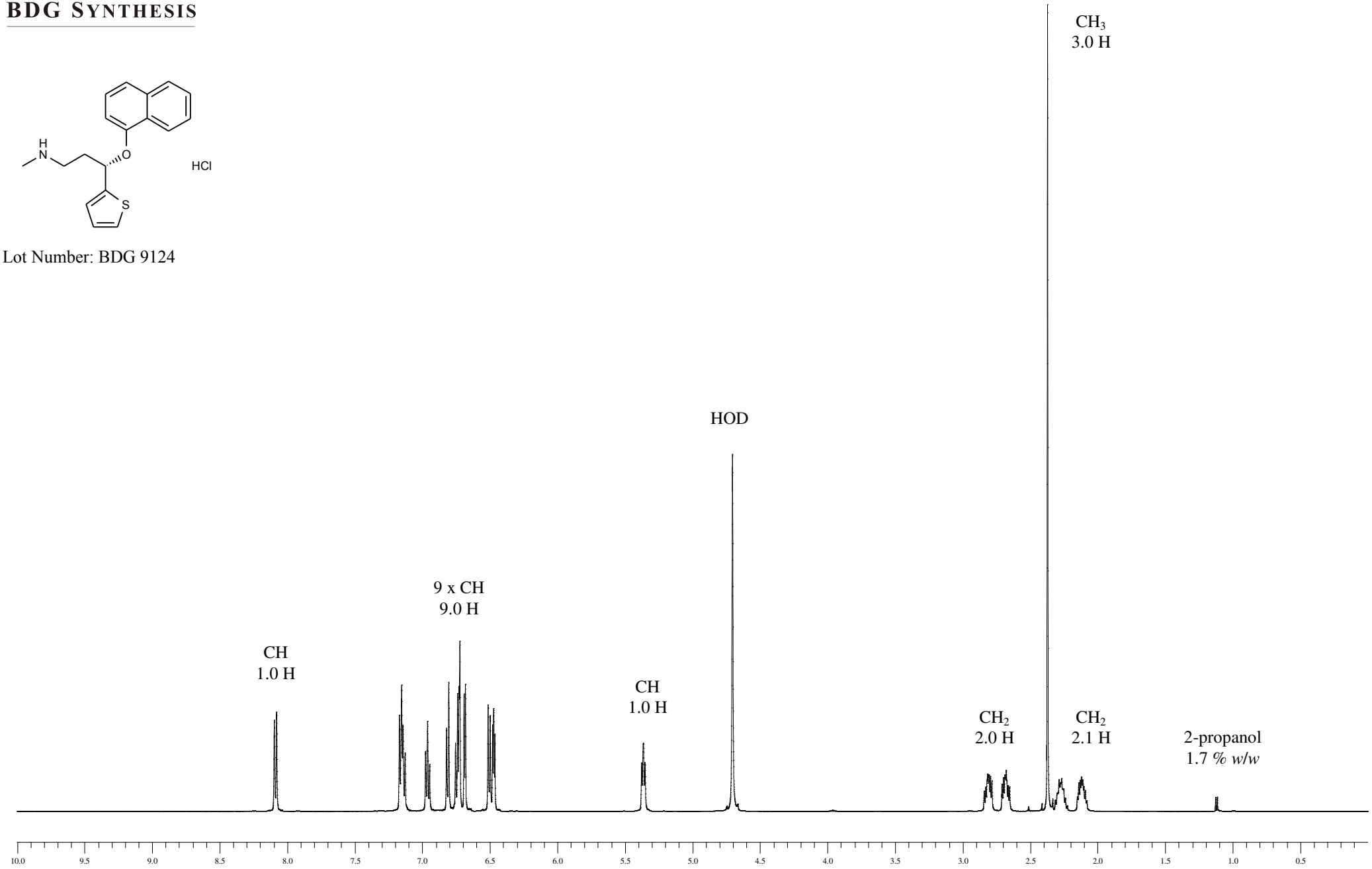
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

# Proton NMR Spectrum of Duloxetine HCl in $D_2O$

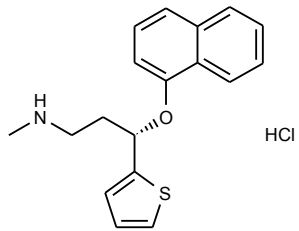



Lot Number: BDG 9124

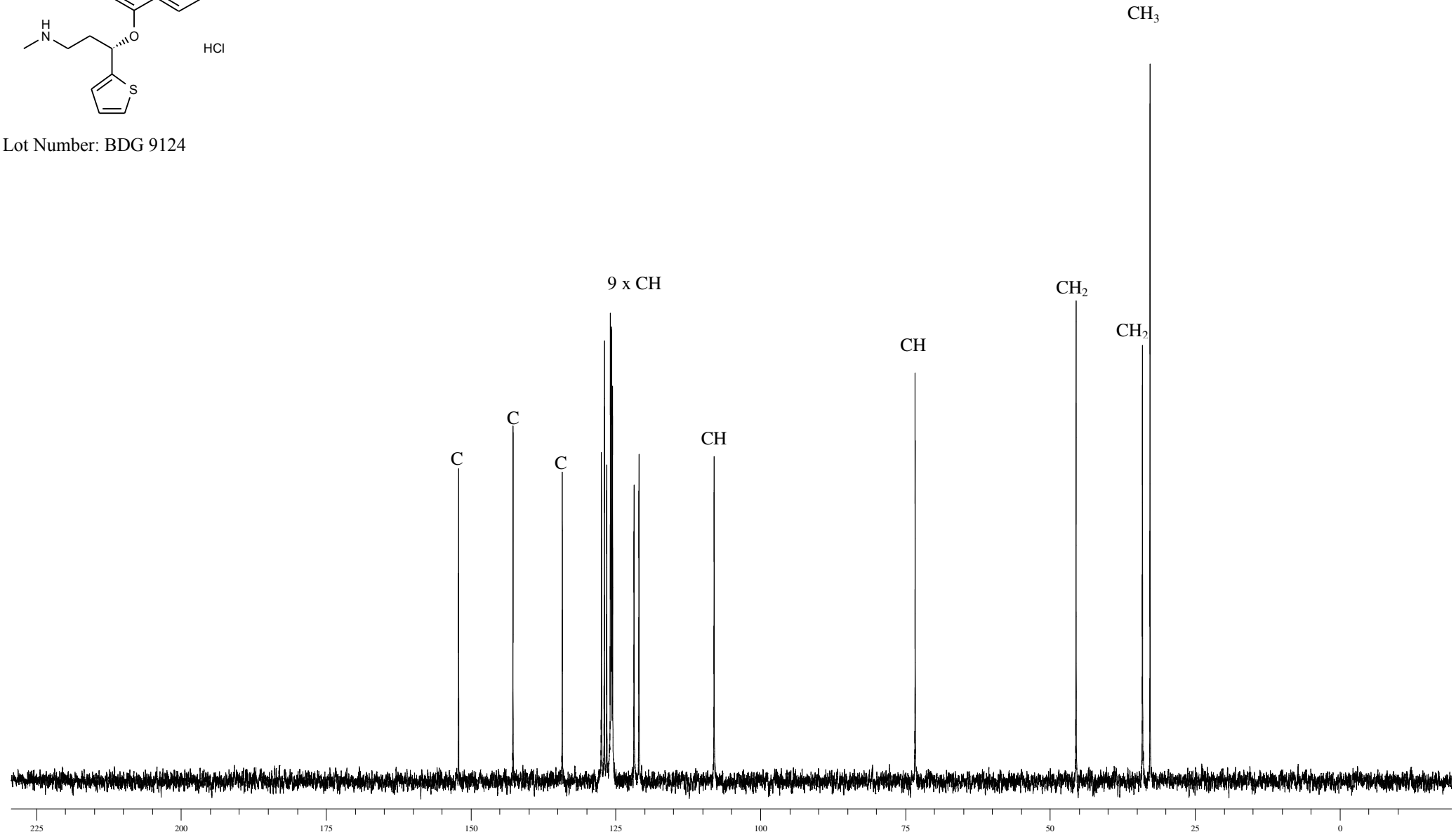


BDG SYNTHESIS

Carbon-13 NMR Spectrum of Duloxetine HCl in $D_2O$



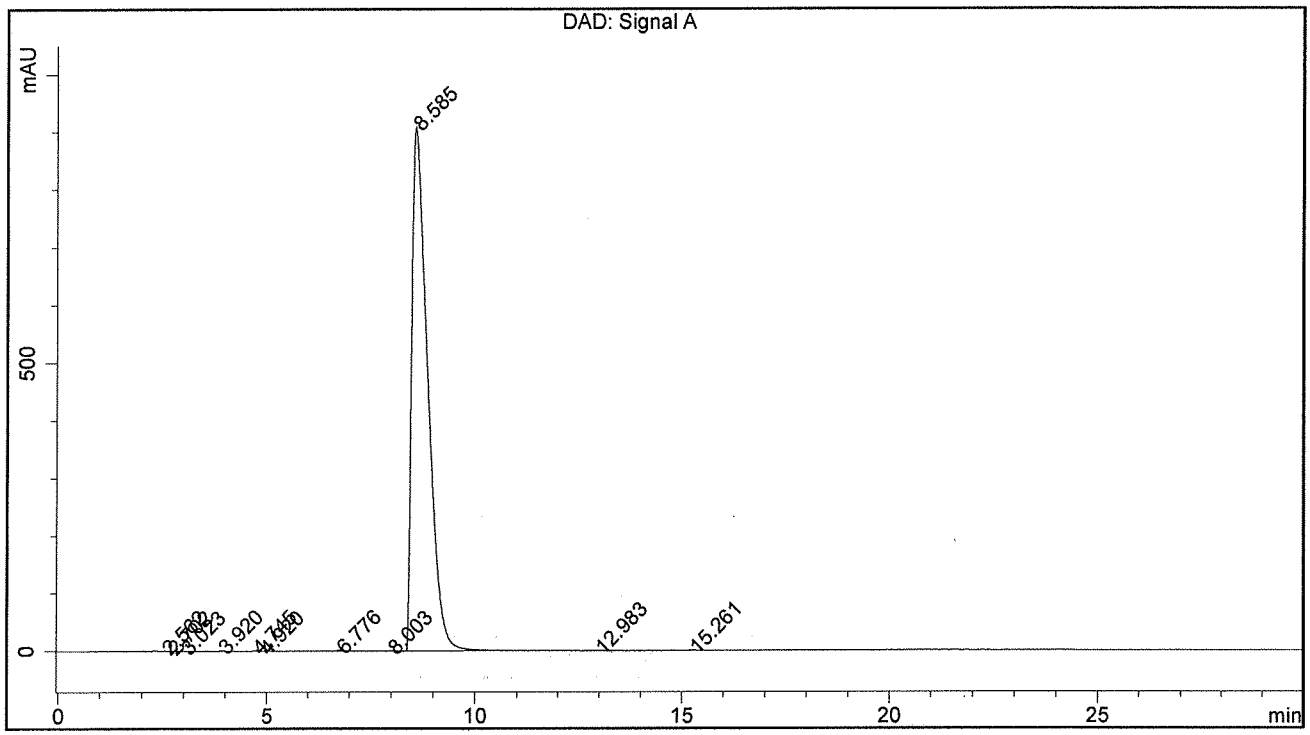

Lot Number: BDG 9124



BDG - Analysis of Duloxetine HCl

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 60:40 10mM Potassium diHydrogen Phosphate pH = 5.5: Acetonitrile
Flow Rate : 1.0 mL/min . . . . . Sample Solvent : 70/30 H2O/CH3CN
Column Temperature : 35C . . . . . Injection Volume : 10 uL
Detection : UV at 231 nm

| Sample Name | BDG 9124 | Instrument | AnalyticalLC01 |
|---|---|---|---|
| Acquisition | 06/08/2013, 13:09:27 | Method (rev.) | LC10581a ( 5) |
| Sequence | BDG_06Aug2013c - Reprocessed | Vial Position | 3 |
| Operator | solvation010\cerityadmin | Injection | 1 of 1 |




**Area Percent Report**

| Peak# | RT | Peak Height | Peak Area | Width | Area % |
|---|---|---|---|---|---|
| 1 | 2.56 min | 0.4604 | 2.9907 | 0.0978 min | 0.012 % |
| 2 | 2.70 min | 0.3065 | 1.9720 | 0.1051 min | 0.008 % |
| 3 | 3.02 min | 0.3326 | 3.4602 | 0.1414 min | 0.014 % |
| 4 | 3.92 min | 1.5559 | 12.3963 | 0.1169 min | 0.051 % |
| 5 | 4.75 min | 1.1124 | 8.8821 | 0.1172 min | 0.037 % |
| 6 | 4.92 min | 0.3384 | 3.3093 | 0.1221 min | 0.014 % |
| 7 | 6.78 min | 1.9330 | 22.1209 | 0.1675 min | 0.092 % |
| 8 | 8.00 min | 0.6120 | 7.2901 | 0.1790 min | 0.030 % |
| 9 | 8.59 min | 908.9448 | 24008.3719 | 0.4002 min | 99.650 % |
| 10 | 12.98 min | 0.4230 | 10.0106 | 0.2881 min | 0.042 % |
| 11 | 15.26 min | 0.6768 | 11.8875 | 0.2209 min | 0.049 % |