

BDG SYNTHESIS

Certificate of Analysis

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

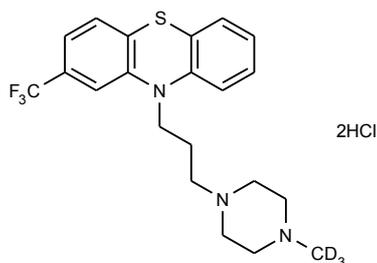
Barry Dent

Barry R. Dent, PhD, Director
6 July 2012

Name: Trifluoperazine-d₃ Dihydrochloride

CAS Number: 440-17-5 (unlabelled)

Structure:



Molecular Weight: $C_{21}H_{21}D_3F_3N_3S \cdot 2HCl = 483.44$

Lot Number: BDG 1461.1

Appearance: White, crystalline solid

Corrected Purity: 99.8 % (HPLC) - 0.2 % (chloroform) = 99.6 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 6 July 2017

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.

Isotopic Labelling: signals at the site of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: a small amount of chloroform (0.2 % w/w) and a trace (under 0.1 % w/w) of diethyl ether 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 site 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 411.1906. $C_{21}H_{22}D_3F_3N_3S$ $[M+H]^+$ requires m/z 411.1910. The deviation of 1.0 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 somewhat broadened, slightly tailing peak is observed (99.8 %). 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 51.91, H 4.84, D 1.26, Cl 14.45, N 8.62 %
$C_{21}H_{21}D_3F_3N_3S \cdot 2HCl$	Requires:	C 52.17, H 4.80, D 1.25, Cl 14.67, N 8.69 %

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).

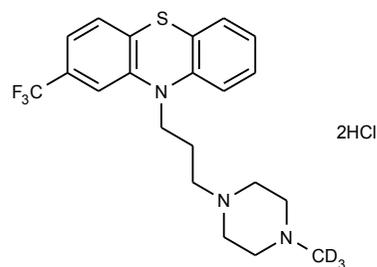
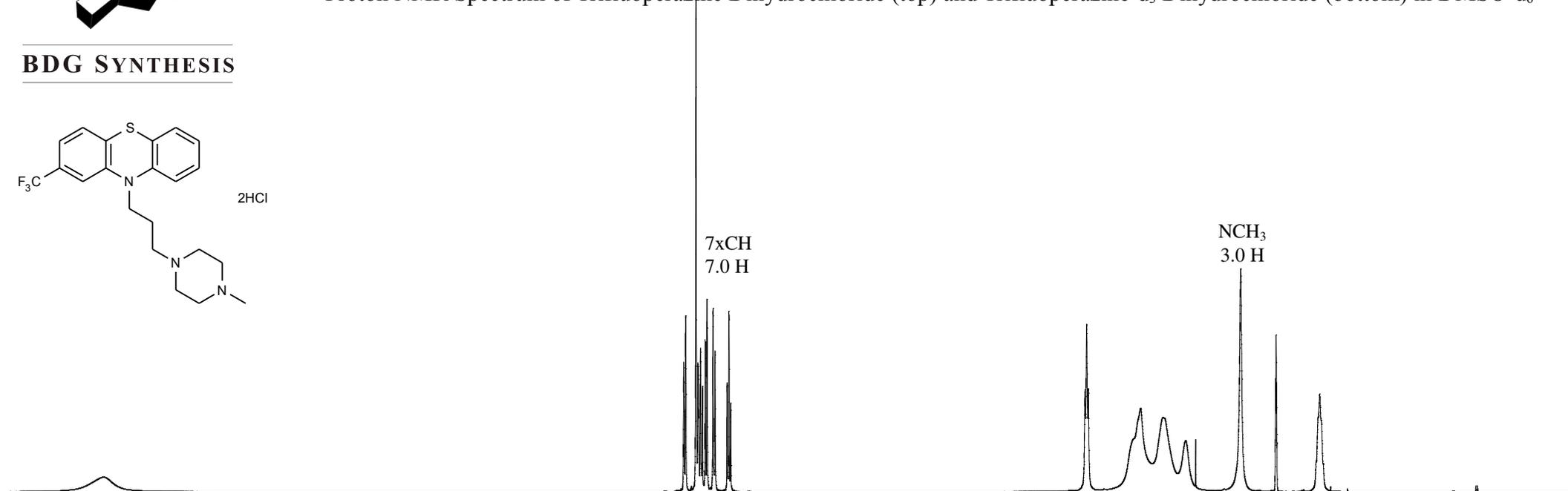
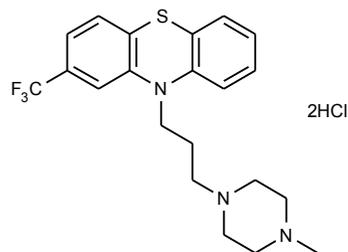
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

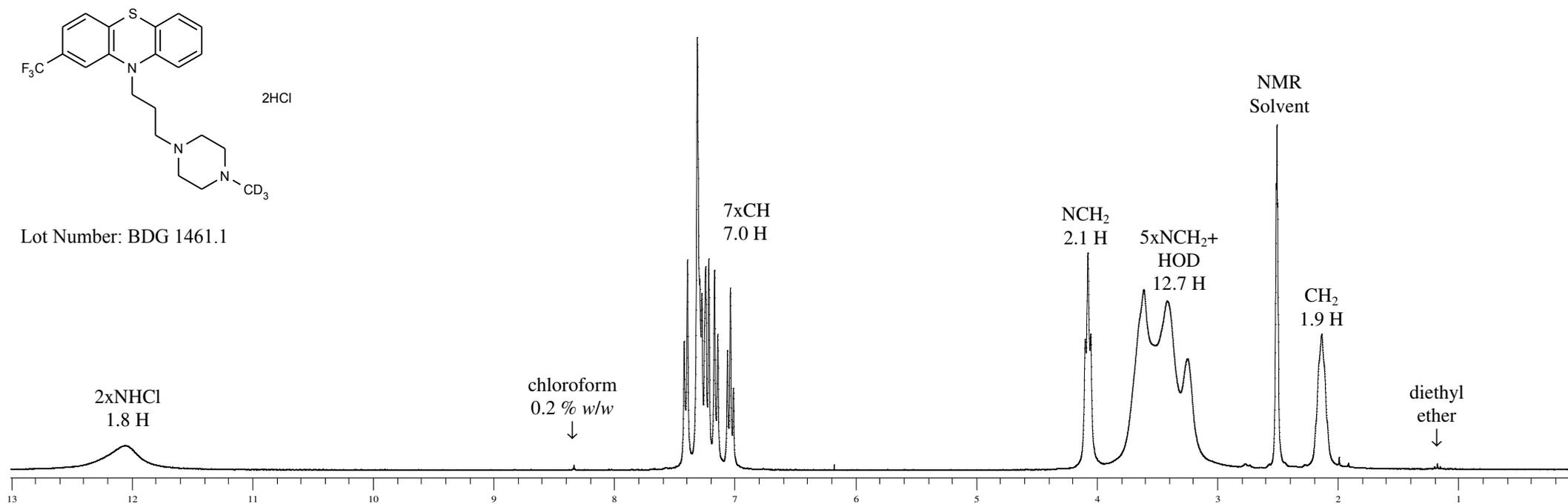


Proton NMR Spectrum of Trifluoperazine Dihydrochloride (top) and Trifluoperazine-d₃ Dihydrochloride (bottom) in DMSO-d₆

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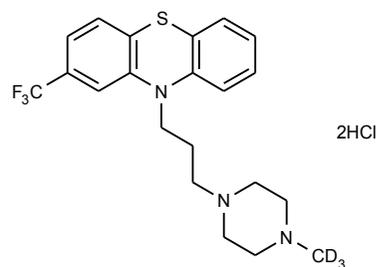
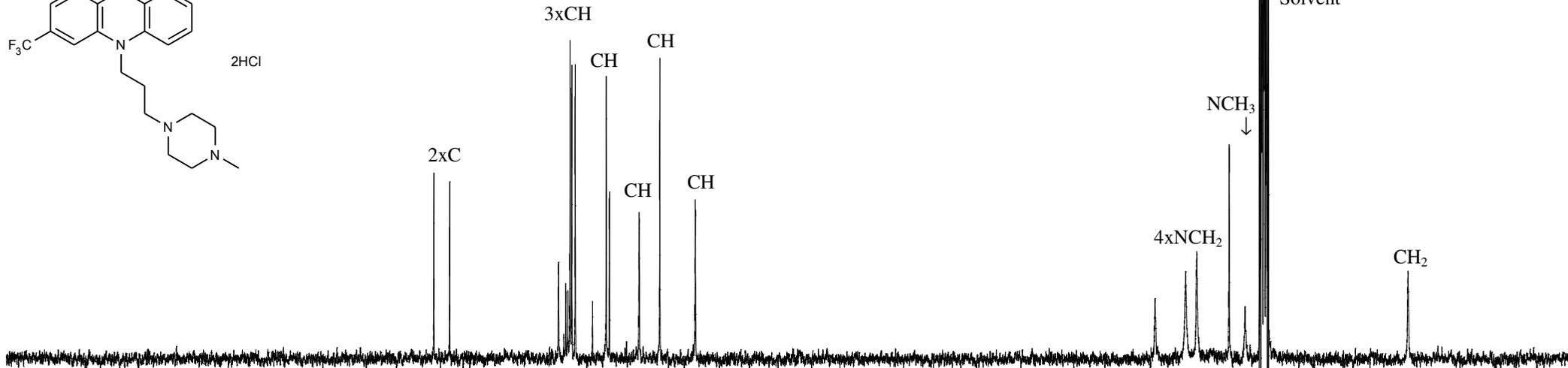
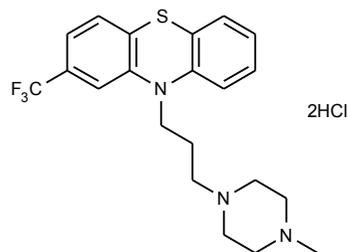
Lot Number: BDG 1461.1



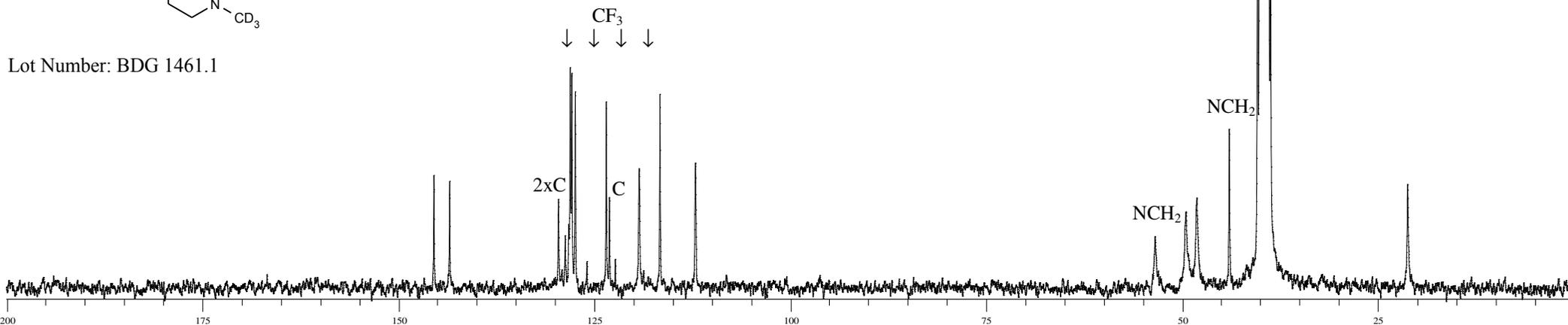


BDG SYNTHESIS

Carbon-13 NMR Spectrum of Trifluoperazine Dihydrochloride (top) and Trifluoperazine-d₃ Dihydrochloride (bottom) in DMSO-d₆



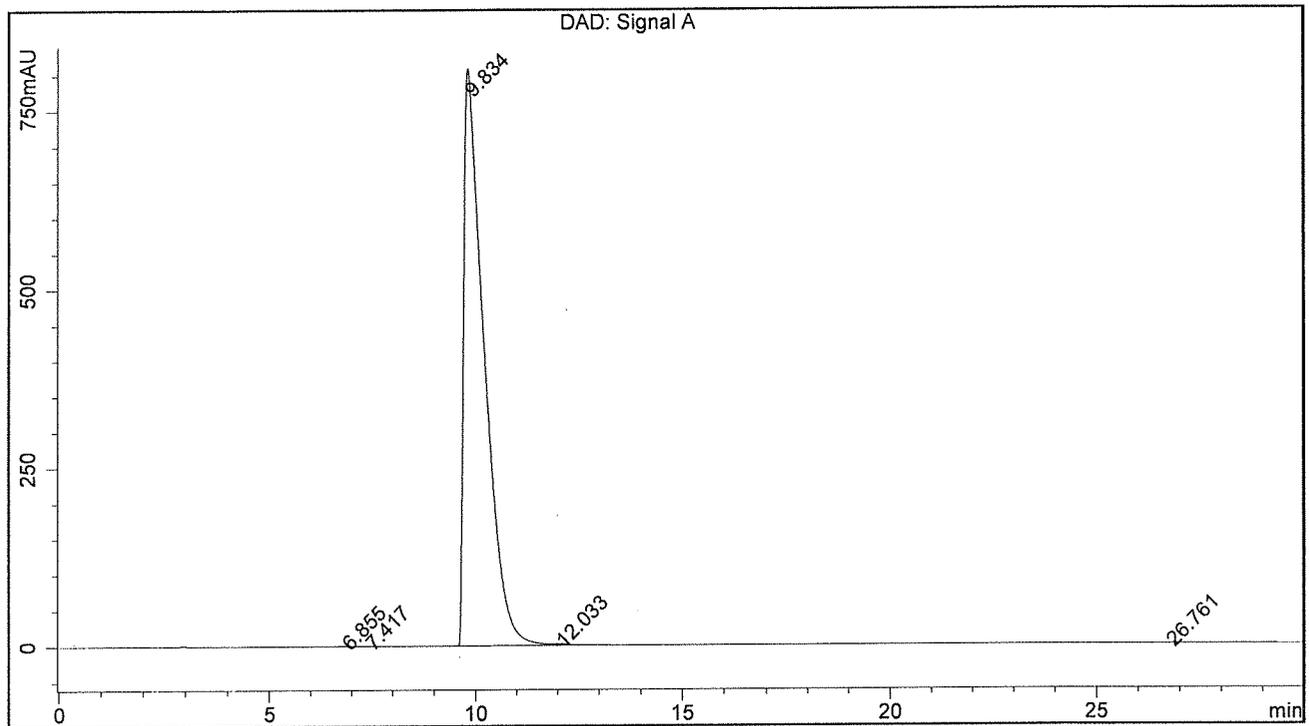
Lot Number: BDG 1461.1



BDG - Analysis of Trifluoperazine-d3 Dihydrochloride

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 4 x 3 mm
 Mobile Phase : 58:42 20mM Potassium diHydrogen Phosphate pH=3.0 : Acetonitrile
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 260 nm

Sample Name	BDG 1461.1	Instrument	AnalyticalLC01
Acquisition	06/07/2012, 12:12:20	Method (rev.)	LC10523a (8)
Sequence	BDG_06Jul2012d	Vial Position	3
Operator	solvation010\cerityadmin	Injection	1 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	6.86 min	0.3853	6.0975	0.2046 min	0.022 %
2	7.42 min	0.2947	3.6402	0.1661 min	0.013 %
3	9.83 min	808.8940	27582.9684	0.4738 min	99.846 %
4	12.03 min	1.2547	32.3525	0.3128 min	0.117 %
5	26.76 min	0.1000	0.5365	0.0746 min	0.002 %