

BDG SYNTHESIS

Certificate of Analysis

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

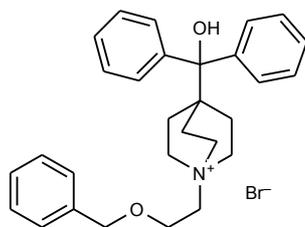
Neil Beare

Neil Beare, PhD, Director
27 July 2015

Name: Umeclidinium Bromide

CAS Number: 869113-09-7

Structure:



Molecular Weight: $C_{29}H_{34}NO_2 \cdot Br = 508.49$

Lot Number: BDG 15051.4

Appearance: White, crystalline solid

Purity By HPLC: 99.1 %

Re-test Date: 27 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:	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.
Residual Solvents: a trace (under 0.1 % w/w) of dichloromethane 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 428.2591. $C_{29}H_{34}NO_2$ $[M]^+$ requires m/z 428.2590. The deviation of 0.2 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A somewhat broadened, tailing peak is observed (99.1 %). The peak at 2.5 min has been identified as the bromide counterion. 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 68.42, H 6.76, N 2.65 %
$C_{29}H_{34}NO_2 \cdot Br$	Requires:	C 68.50, H 6.74, N 2.75 %

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).

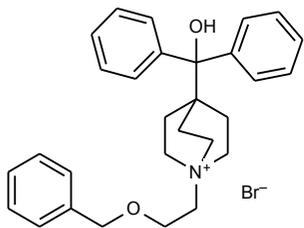
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials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

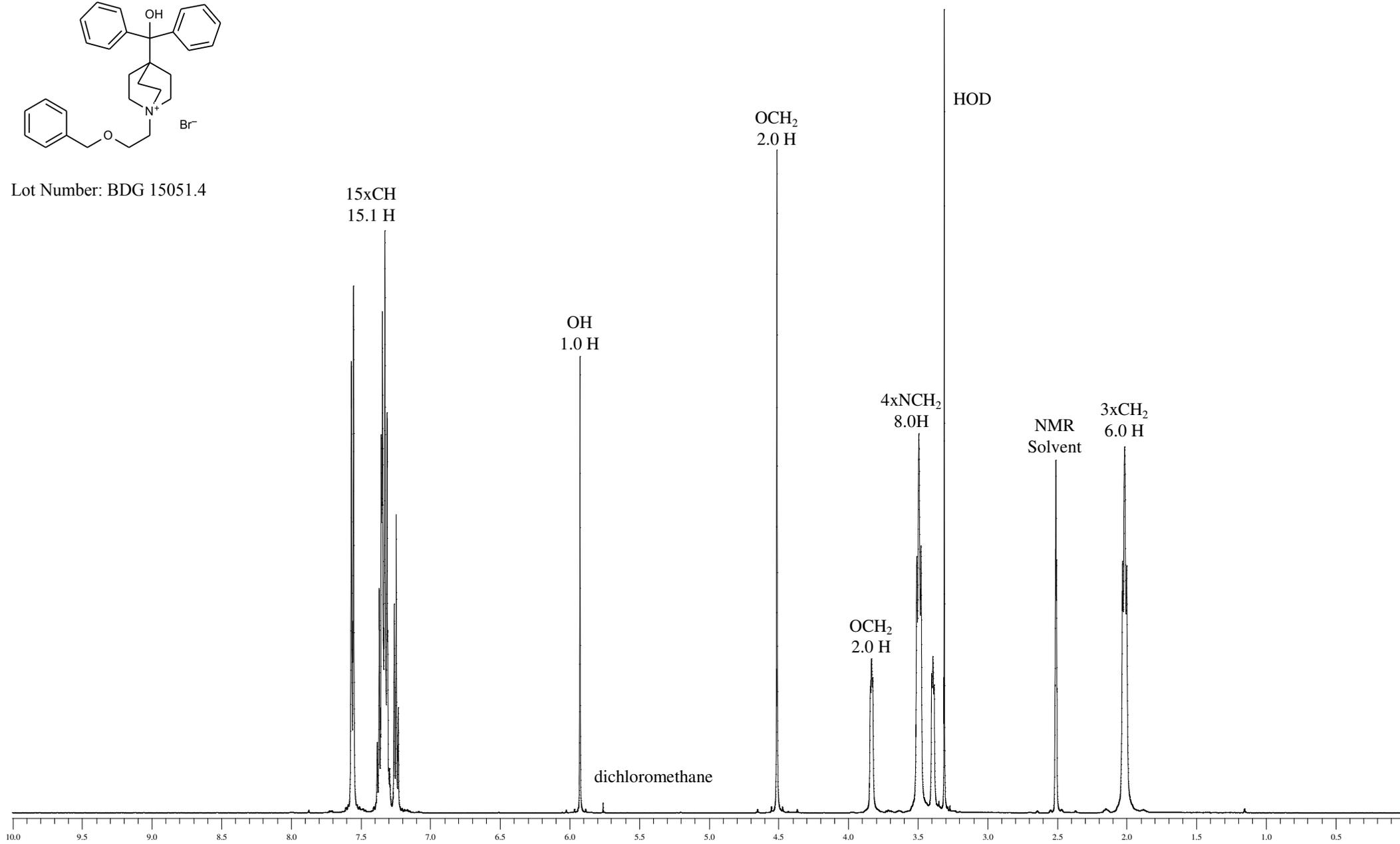


Proton NMR Spectrum of Umeclidinium Bromide in DMSO-d₆

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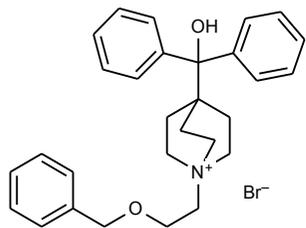
Lot Number: BDG 15051.4



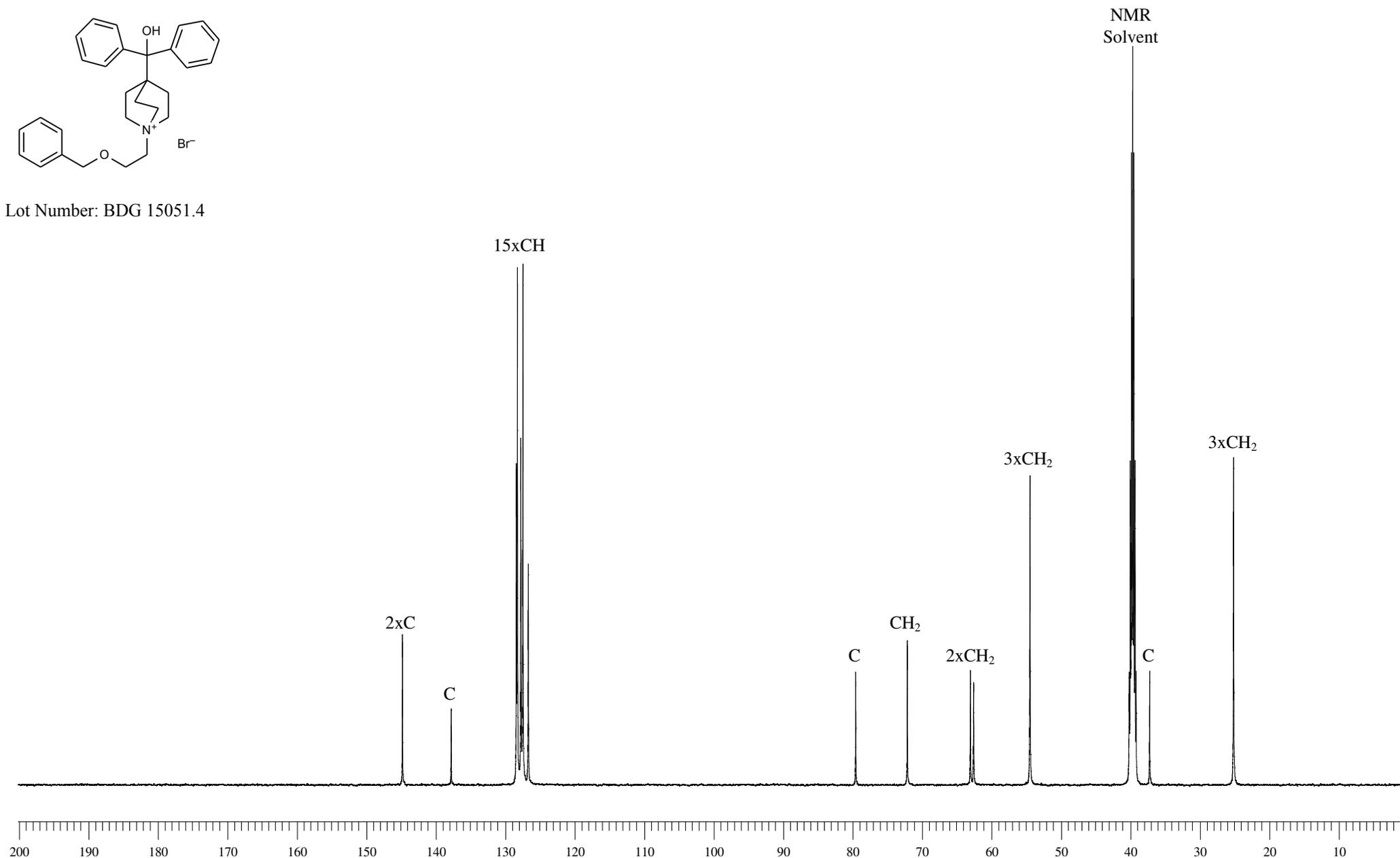


Carbon-13 NMR Spectrum of Umeclidinium Bromide in DMSO-d₆

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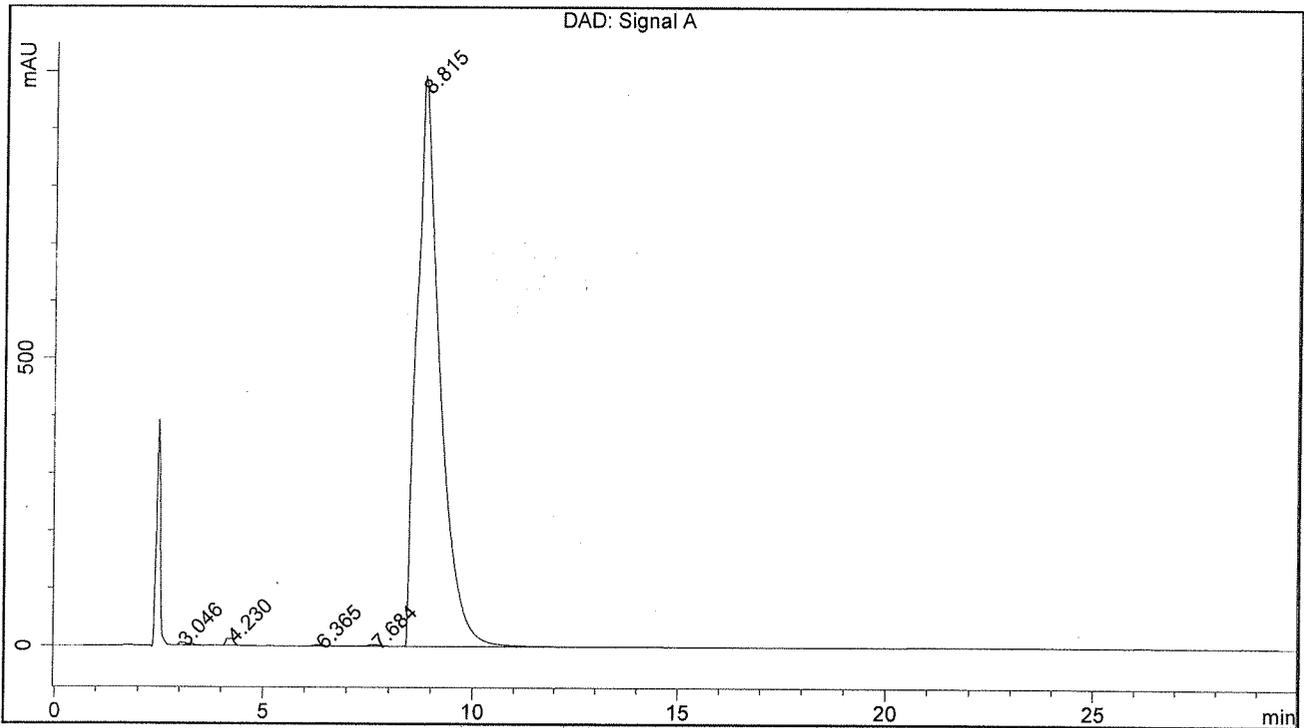
Lot Number: BDG 15051.4



BDG - Analysis of Umeclidinium Bromide

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 55:45:0.05 Water : Acetonitrile : Trifluoroacetic Acid
 Column Temperature : 20 C
 Flow Rate : 1.0 mL/min
 Sample Solvent : 70:30 Water : Acetonitrile
 Injection Volume : 10 uL
 Detection: UV 214 nm

Sample Name	BDG 15051.4	Instrument	AnalyticalLC01
Acquisition	27/07/2015, 15:12:35	Method (rev.)	LC10622a (16)
Sequence	BDG_27Jul2015b - Reprocessed	Vial Position	14
Operator	solvation010\cerityadmin	Injection	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.05 min	5.1263	89.9987	0.2248 min	0.233 %
2	4.23 min	12.3711	167.0821	0.2176 min	0.433 %
3	6.36 min	2.1730	44.6297	0.2756 min	0.116 %
4	7.68 min	2.5705	55.4883	0.2878 min	0.144 %
5	8.81 min	991.7836	38208.9599	0.5095 min	99.074 %