

### Product name

Including concentration and solvent, where relevant.

### CAS number

The unique identifier assigned by the Chemical Abstracts Service.

### Molecular weight and molecular formula

If the neat material is in salt form these parameters are for the salt not free base.

### Volume per ampoule

All are overfilled to ensure the minimum stated is recoverable.

### Expiry date

Our expiry dates are determined by real time and accelerated stability testing. NOTE 3

### Quality bar

Quality accreditation under which that specific material was produced, not LGC's full scope of quality accreditation.

# LoGiCal®

produced by LGC

## Certificate of Analysis

### Reference Material - Primary Standard

Product Name: Amitriptyline Hydrochloride 1.0 mg/ml in Methanol (as free base)

Catalogue Number: LGCAMP0062.00-01

Lot Number: 7836

CAS Number: 549-18-8

Molecular Formula:  $C_{20}H_{23}N \cdot HCl$

Molecular Weight: 313.86

Solvent: Methanol

Volume per Ampoule: Not less than 1 ml<sup>1</sup>

Long-term Storage: 2 to 8 °C, dark

Expiry Date: August-2015

Intended Use: The primary aim of this material is for identification, calibration and quantification.

Component	Concentration ("free base")	Uncertainty
see product name	1.000 mg/ml <sup>2</sup>	U = 0.009 mg/ml <sup>3</sup>

Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and Guide 34 at the about 95 % level of confidence using a coverage factor of  $k = 2$  and has been calculated by statistical analysis of our production system and incorporates uncertainty of the purity, material density and balance and weighing technique.  
Concentration based on material weighings and material purity factor (assay of the neat material).

The solution's concentration and homogeneity are verified by independent method.

LGC certifies that this standard meets the specification stated in this certificate and warrants this product to meet the stated acceptance criteria through the retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.

Release Date:  
Luckenwalde, April 2012

Signed:

Dr. Sabine Schröder  
Unit for Reference Materials

<sup>1</sup> Ampoules are overfilled to ensure a minimum 1 ml volume fill. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.

<sup>2</sup> The value is based on the results of analytical techniques, which calibration and verification was carried out with standards traceable to SI-units. The value is expressed on a "free base" basis.  
The concentration with its uncertainty is valid in the range between 19 °C and 25 °C.

The identity is verified by data from international scientific literature.  
Gravimetrically prepared using qualified balances calibrated annually by accredited calibration service. Calibration verification performed daily prior to use utilizing weights traceable to SI via other mass standards.

<sup>3</sup> The uncertainty "U" is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM). It is corresponding to a level of confidence of about 95 %. Standard uncertainties are indicated with "u".

LGC Quality - ISO Guide 34:2009 DAKS D-RM-14176-01-00 ISO/IEC 17025:2005 DAKS D-PL-14176-01-00 ISO 9001:2008 DQS 10248 QM09

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**LGC** Standards  
Excellence through measurement

### Catalogue number

LGCAMP prefix for solutions and LGCFOR for powders.

### Chemical structure

Indicating exact position of isotopic labels and freely downloadable.

### Long term storage

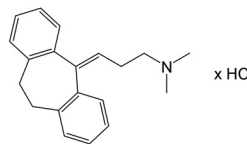
The minimum specified storage requirements and those under which real time stability testing is performed.  
NOTE 1

### Uncertainty

The statistical parameter that is used to describe the possible fluctuation in concentration.  
NOTE 2

### Concentration

Based on material weighings and purity factor (assay of the neat material).  
NOTE 4



### Concentration 'as is'

If the neat material is in salt form the concentration of the salt is also provided.

### Verification of concentration

Depending on the analyte, the concentration is verified by an appropriate technique such as GCMS or HPLC.

### Identity

We use a range of analytical techniques to confirm the identity of our reference substance (neat material); <sup>1</sup>H-NMR, MS, IR, HPLC-DAD, GC.

### Purity

Purity testing is performed by HPLC or GC, water content is evaluated by Coulometric Karl Fischer titration, and residual solvent by <sup>1</sup>H-NMR.

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produced by LGC

Additional information			
Concentration ("as is")		1.131 mg/ml	
Verification of Concentration and Homogeneity			
Lot Number	Verified Concentration (mg/ml)		% RSD - Homogeneity
7836	Result	Acceptance Criteria	Result    Acceptance Criteria
	1.029	± 3 %	0.664    ≤ 3 %
Concentration verified by GCMS			
Solution Standard Assay Parameters		External Calibration (100 % amount)	
Analysis Method	GCMS		
Column:	HP-5MS, 30 m x 0.25 mm x 0.25 µm	Number of Measurements:	6
Injector:	220 °C		
Flow:	1.50 ml/min		
Oven Program:	Rate	°C	Time
		70	3 min
	30 °C/min	250	11 min (run time 20 min)
Detector:	280 °C		
Neat Material Data			
Product Name:	Amitriptyline Hydrochloride		
CAS Number:	549-18-8		
Molecular Formula:	C <sub>20</sub> H <sub>23</sub> N HCl		
Molecular Weight:	313.86		
Compound Lot:	EN 7391		
Test	Method	Result	
Melting Point (°C)*	SOP 06-010	199 °C	
<sup>1</sup> H-NMR Spectrum*	SOP 06-053	conform / complies to structure	
IR Spectrum*	SOP 06-036	conform / complies to structure	
Mass Spectrum (ESI)*	SOP 06-022	conform / complies to structure	
Assay by carbon titration (free base)*		Elementary Analysis	88.52 %
The expanded uncertainty according to the assay is U = 0.83 % (about 95 % level of confidence using a coverage factor of k = 2).			
Assay by carbon titration ("as is")*		100.18 %	

\*: Validated method performed by ISO/IEC 17025 accredited testing lab

The assay of the neat material is verified by the 100 % method using HPLC, corrected with water (KFT) and residual solvents.

### Homogeneity

Homogeneity is assured by validated procedures, including ampoulation, and is verified by HPLC.

### Physical properties

Where other physical properties are known then we also use these as an identity check (e.g. melting point, appearance, and polarimetry).

### Absolute methods

We use elemental analysis (carbon titration) and/or quantitative NMR in the characterisation of our reference materials.

LGCAMP0062.00-01 Lot Number 7836

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