

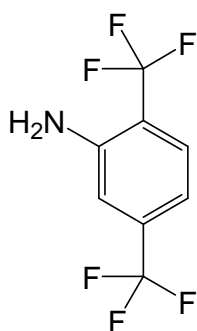
Certificate of Analysis

LGC Quality

ISO Guide 34:2009
DAkkS D-RM-14176-01-00
ISO/IEC 17025:2005
DAkkS D-PL-14176-01-00
ISO 9001:2008
DQS 102448 QM08

Primary Reference Standard

2,5-Bis-(trifluoromethyl)aniline



Molecular Formula: C₈H₅F₆N
Molecular Weight: 229.12
CAS Number: [328-93-8]

Catalogue Number: LGCQUANT1424.01

Lot Number: 21311

Long-term Storage: 15 to 25 °C, dark

Appearance: yellow-brown liquid

Assay¹ 'as is': 99.9 %

Uncertainty² U: 0.4 %

Date of shipment: **2014-May-26**

This certificate is valid for one year from the date of shipment provided the substance is stored under the recommended conditions.

¹ The value is based on the results of analytical techniques. Calibration and verification were carried out with standards traceable to SI-units. The value is expressed on an "as is" basis. The identity is verified by data from international scientific literature.

² 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 %. Coverage factor $k = 2$.

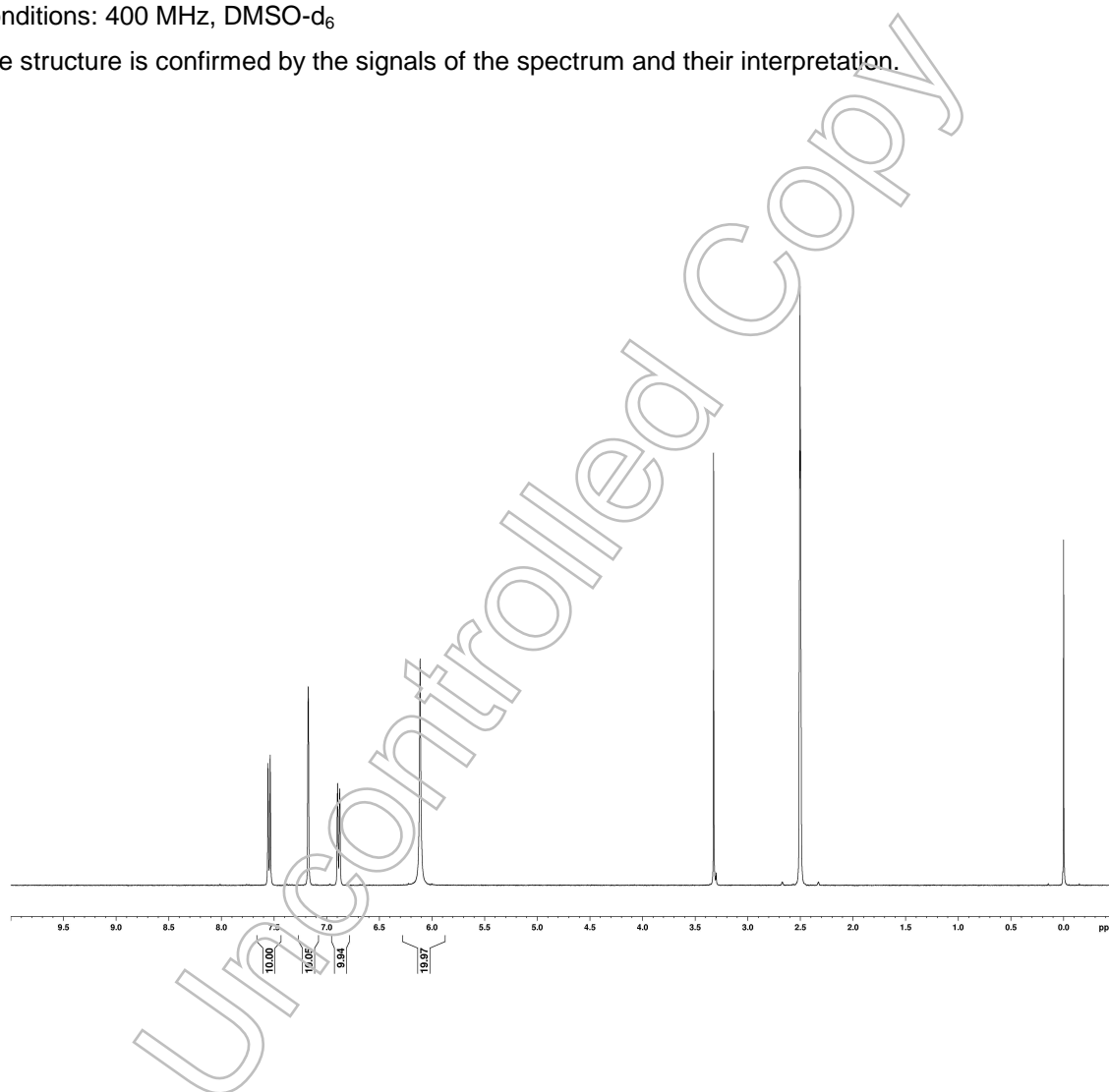
I. Identity

The identity of the reference material was established by following analyses.

Ia. $^1\text{H-NMR}$ Spectrum

Conditions: 400 MHz, DMSO- d_6

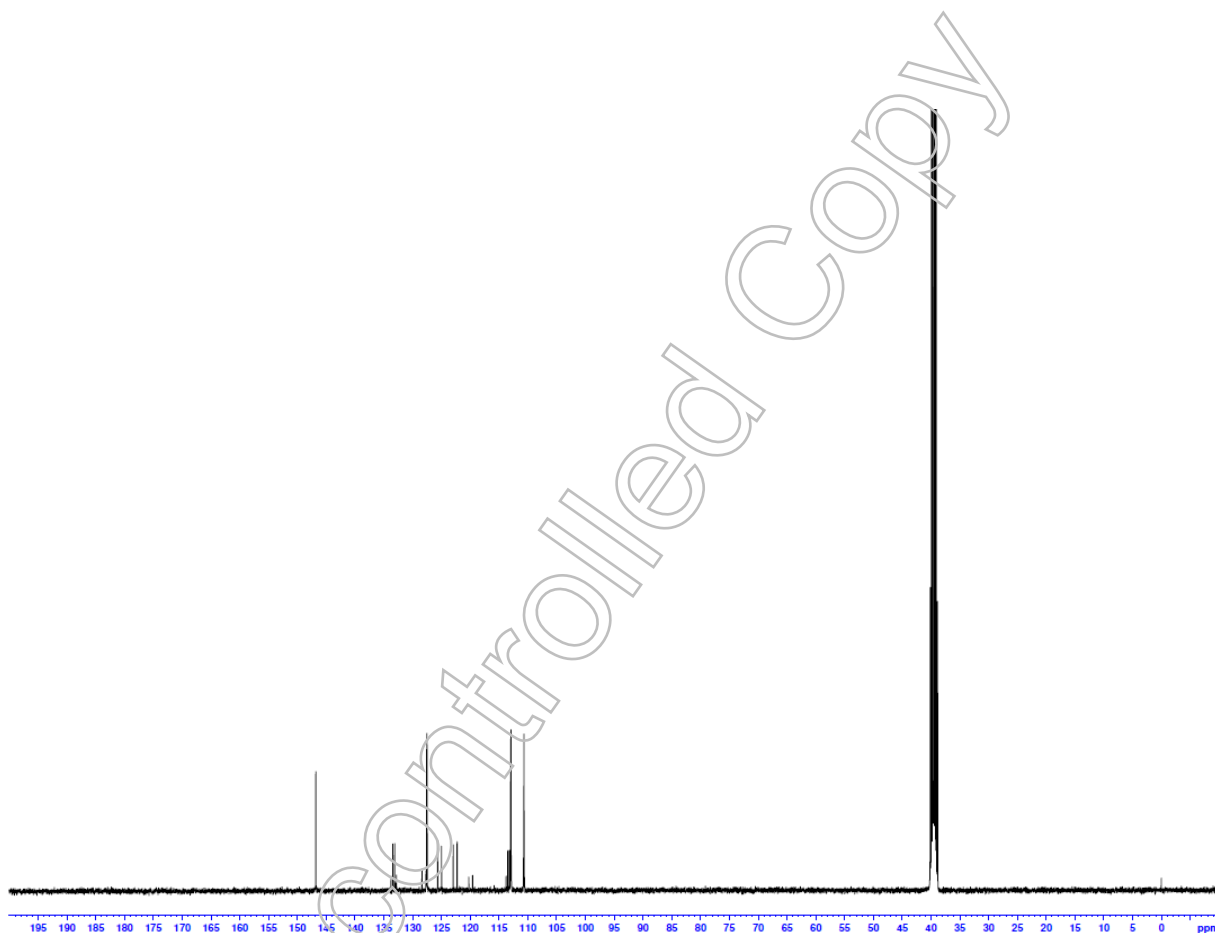
The structure is confirmed by the signals of the spectrum and their interpretation.



Ib. ^{13}C -NMR Spectrum

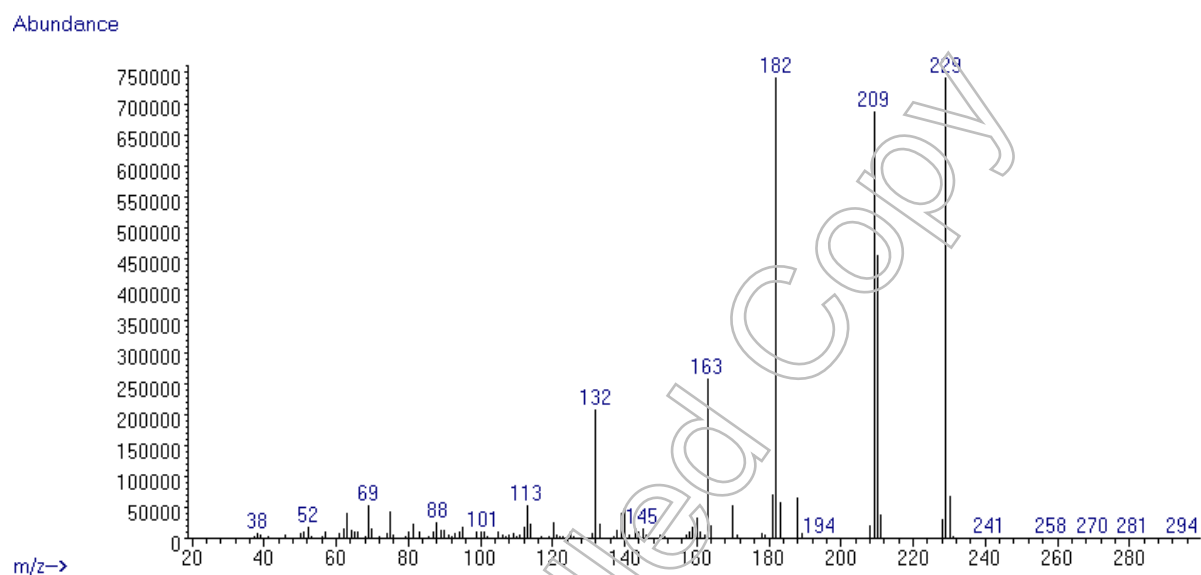
Conditions: 100 MHz, DMSO- d_6

The structure is confirmed by the signals of the spectrum and their interpretation.



Ic. Mass Spectrum

Method: EI, 70eV, detector temperature: 280 °C



The signals of the MS spectrum and their interpretation are consistent with the structural formula.

II. Purity

The purity of the reference material was determined by high performance liquid chromatography (HPLC).

HPLC Conditions:

Column:

Kinetex C18
2.6 µm, 150 x 4.6 mm

Conditions:

1.0 ml/min, 40 °C
0-12 min Water/Acetonitrile 50/50
12-15 min Water/Acetonitrile to 20/80
15-18 min Water/Acetonitrile to 50/50
18-28 min Water/Acetonitrile 50/50 (v/v);
0.1 % H₃PO₄

Detector:

DAD
245 nm

Injector:

Auto
4 µl; 0.06 µl/ml in
Acetonitrile

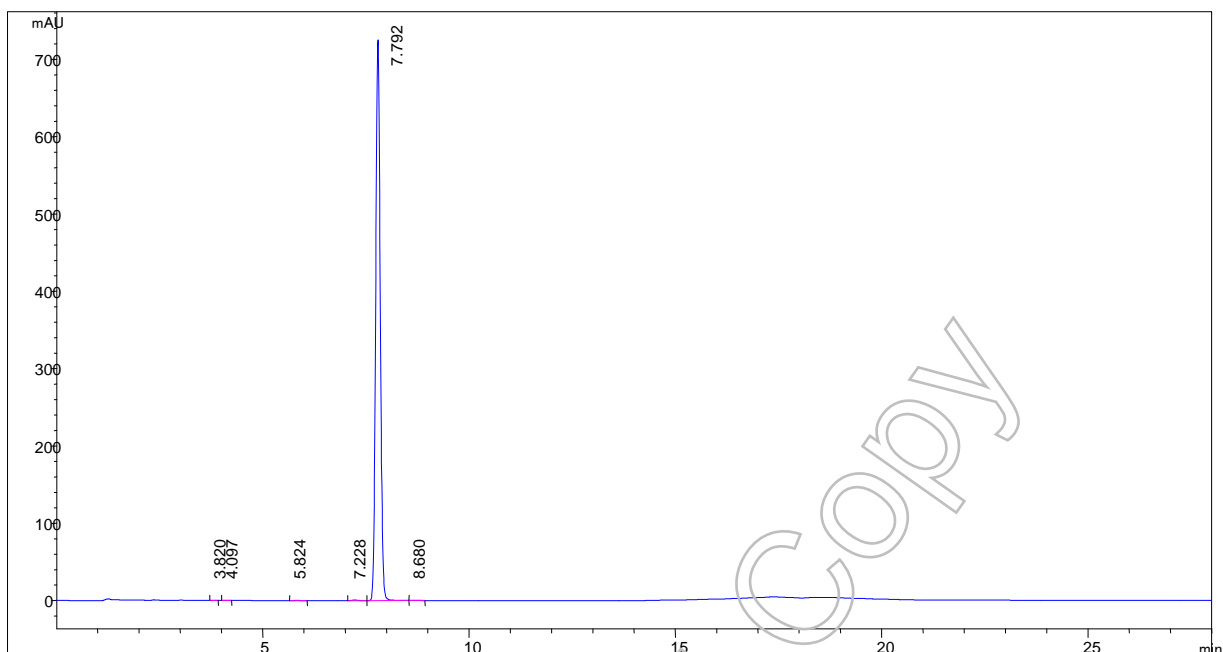


Excellence through measurement

LGCQUANT1424.01 Lot Number 21311

LGC GmbH, Im Biotechnologiepark, TGZ II, D-14943 Luckenwalde, Germany

page 4/9



Area Percent Report - Sorted by Signal

Pk #	Retention Time	Area	Area %
1	3.82	0.56	0.01
2	4.10	0.40	0.01
3	5.82	0.47	0.01
4	7.23	4.08	0.07
5	7.79	5568.96	99.89
6	8.68	0.66	0.01
Totals		5575.13	100.00

For the calculation the system peaks were ignored. The content of the analyte was determined as the ratio of the peak area of the analyte and the cumulative areas of the purities, added up to 100 %.

Results:

Average 99.89 %
Number of results n=6
Standard deviation < 0.01%



III. Water Content

Method: Karl Fischer titration (not accredited testing method)

Results:

Average	0.09 %
Number of results	n=3
Standard deviation	< 0.01 %

IV. Residual Solvents

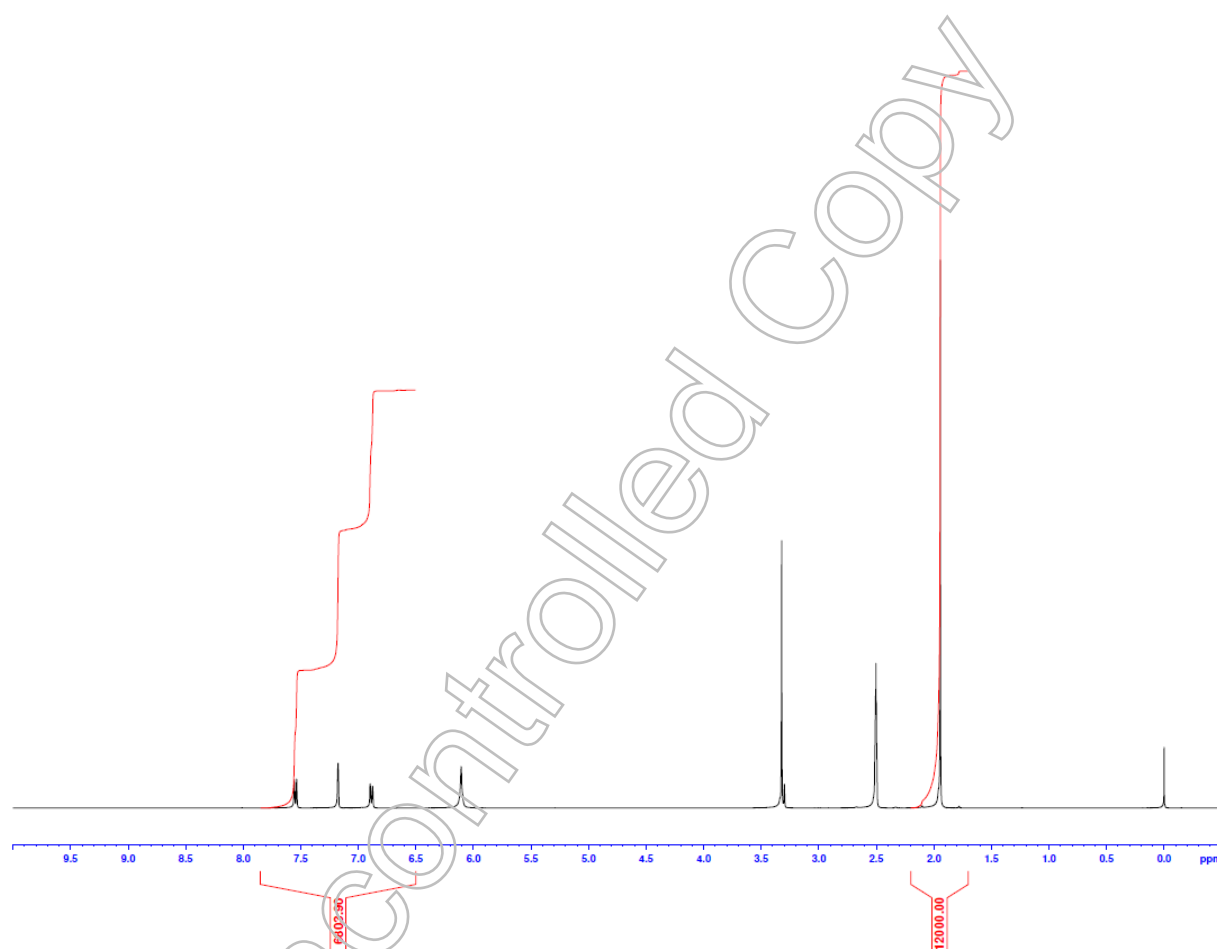
Method: ¹H-NMR (not yet accredited testing method)

No significant amounts of residual solvents were detected (< 0.05 %).

Uncontrolled Copy

V. Assay by quantitative NMR spectroscopy

The assay of the reference substance was established by quantitative NMR spectroscopy using DMSO-d₆ as the solvent and with Duroquinone (certified reference material, signal 1.7 – 2.2 ppm, 12 H) as internal standard.



Results:

Average	99.87 %
Number of results	n=6
Uncertainty U (expanded)	0.42 %

VI. Final Result

Chromatographic purity (HPLC)	99.89 %
Water content	0.09 %
Residual solvents	n. d. (not detected)

Assay:

Quantitative NMR spectroscopy	99.87 %
--------------------------------------	---------

The assay is assessed to be 99.9 % 'as is'

The assay 'as is' is equivalent to the assay based on the not anhydrous and not dried substance respectively. It is determined by quantitative NMR spectroscopy and its expanded uncertainty ($k = 2$) is 0.4 %.

The assay is verified by the 100 % method (HPLC): 99.8 % which is inside the expanded uncertainty of the assay by quantitative NMR spectroscopy.

VII. Stability and Homogeneity

Accelerated stability studies indicate no significant instability. The given validity period is based on this data. This is backed up by additional stability testing and historical data over the range of several years. Homogeneity assured by qualified process of preparation, verified by homogeneity testing.

VIII. Further Information

General

For laboratory use only. Not suitable for human or animal consumption.

This material conforms to the characteristics of a primary standard as described within ISO Guide 30 (Terms and definitions used in connection with reference materials).

The values quoted in this certificate are LGC's best estimate of the true values within the stated uncertainties and based on the techniques described in this certificate.

The production of this reference material was realized taking into account the principles of ISO Guide 34.



Excellence through measurement

Intended Use

Use for identification and quantification.

Handling of the RM

Before usage of the reference material, it should be allowed to warm to room temperature.

Assay and uncertainty

The identity and the assay are assessed by an ISO/IEC 17025 accredited testing method.

The calculation of the 100 % method (HPLC) follows the formula:

$$\text{Assay (\%)} = (100 \% - \text{KF} - \text{RES}) \quad * \quad \frac{\text{Purity HPLC (\%)}}{100 \%}$$

Water (KF) and Residual solvents (RES) are considered as absolute contributions, HPLC purity is considered as relative contribution.

Uncertainty of the assay is expressed as an expanded uncertainty in accordance with ISO/IEC 17025 at the about 95 % level of confidence.

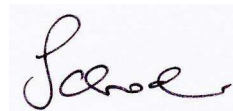
For quantitative applications use the assay as a calculation value on the as is basis. The uncertainty of the assay can be used for estimation/calculation of your own measurement uncertainty.

Quality Control Assessment

The product quality is controlled by regularly performed quality control tests (retests).

Release Date: 2013-04-26

LGC GmbH



Dr. Sabine Schröder
Product Release

Revision	Date	Reason for Revision
00	2013-04-26	Release of the Lot – initial version
01	2013-10-29	Accreditation Number added



Excellence through measurement