

Trace CERT® **New**

定量NMR(qNMR) 内部標準物質用 認証標準物質

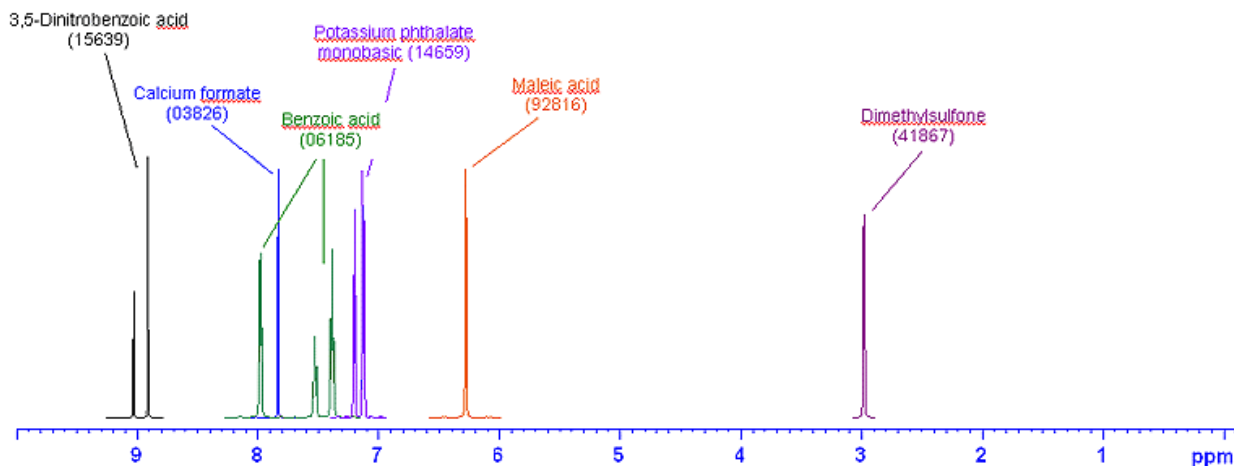
この製品はquantitative ¹H-NMRの内部標準用認証標準物質です。

ISO/IEC 17025 と ISO Guide 34の認証標準物質。NIST SRMIにトレーサブルです。

ISO Guide 31に沿って作られた認証書付。

製品番号	Substance	Chemical Shifts*	Solubility				Pack Size	価格(円)
			D ₂ O	MeOD	CDCl ₃	DMSO-d ₆		
15639	3,5,-Dinitrobenzoic acid	9.2 ppm				●	5g	16,000
06185	Benzoic acid	8.2 - 7.4 ppm		●		●	5g	16,000
41867	Dimethylsulphone	3.2 ppm	●		●	●	5g	16,000
14659	Potassium phthalate monobasic	~ 7 - 8 ppm	●	●			5g	16,000
03826	Calcium formate	~ 7.5 - 8.5 ppm	●				5g	16,000
92816	Maleic acid	6.2 ppm	●	●		●	5g	16,000

*chemical shifts may slightly vary depending on the experimental conditions



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SIGMA-ALDRICH®

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SIGMA-ALDRICH®

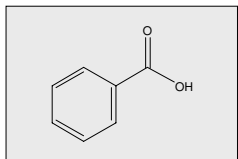
Certificate

Produced in double accredited laboratory fulfilling
ISO/IEC 17025 and
ISO Guide 34

This certificate is designed in accordance with ISO Guide 31^[1].

Substance: **Benzoic acid**

Product no.: **06185**
Lot no.: **BCBC1484**
Formula: **C₇H₆O₂**
Molecular mass: **122.12**
Traceability^[2]: **NIST SRM 350b (Benzoic acid)**
Certificate issue date: **June 16, 2010**
Expiry: **Jun/2012**



Certified value and uncertainty according to ISO Guide 35 ^[3] and Eurachem/CITAC Guide ^[4]		
Substance	Certified value	Expanded uncertainty, $U = k \cdot u_c$ ($k = 2$)
Benzoic acid	99.98 % (g/g)	0.10 % (g/g)

Minimum sample: 60 mg is recommended as the minimal sample amount. If less material is used, it is recommended to increase the certified uncertainty by a factor of two for half of sample and a factor of four for a quarter of sample.

Drying instruction: This material does not require drying before use.

Intended use: This CRM shall be used as internal standard for quantitative ¹H-NMR measurements.

Storage and handling: The CRM should be stored in the original bottle at room-temperature (20-25 °C). After use the bottle should be tightly closed and protected from excessive moisture and light. Storage under Argon is recommended.

CRM operations: *A. Rück*
A. Rück, Ph.D.

Certification body: *Klaus-Dieter Schmidt*
K.-D. Schmidt, Ph.D.

SRMS 001
ISO Guide 34

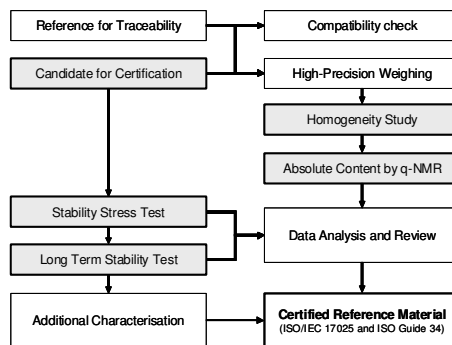
STS 490
ISO/IEC 17025

16368-02
ISO 9001

CERTIFICATION

In order to guarantee highest reliability of this TraceCERT CRM a multi-component approach was applied whereby the certified value is determined by high-resolution quantitative NMR measurements (q-NMR on a Bruker 600 MHz Avance III NMR spectrometer). This certified value is determined under double-accreditation in accordance with ISO/IEC 17025^[5] and also ISO Guide 34^[6]. Extensive stability and homogeneity tests are considered for certification.

The certified values are confirmed by extended analytical data including impurity determination. These data are not covered by the scope of accreditation but extended analytical data are determined following best practices in analytical measurements.



Candidate substances are checked for suitability in terms of purity. Only materials of highest available purity are accepted. 2D-NMR (H-H COSY) measurements are applied to guarantee that no impurities underlie to a peak of interest. Detection limit usually is below 0.1%.

Compatibility check guarantees that the candidate substance does not react neither with the solvent nor with the internal q-NMR reference (t=0 and t=24h comparison).

High precision weighing is performed under ISO/IEC 17025 accreditation with ultra-micro balances certified by DKD and calibrated with OIML Class E2 weights.

Stability stress test is performed with samples which are stored above the recommended storing temperature (mostly at 45 °C) and q-NMR double determinations for verification after 1 and 3 months.

Long term stability test is performed with samples which are stored at the recommended storing temperature and q-NMR double determination after 3, 9 and 24 or 36 months.

Homogeneity of the material is tested by q-NMR measurements using 10 subsamples which are taken from different positions in the entire bulk material. The recommended minimal sample size is taken for all the homogeneity test samples. Analysis of variance (ANOVA) results are included into the calculation of content uncertainty of this CRM.

Absolute content by q-NMR is performed using 10 separate samples of the candidate substance which are each spiked with an adequate amount of internal reference and then immediately dissolved in deuterated solvent. In most cases 32 scans are recorded for every sample, whereby ¹H relaxation time (T1) is defined depending on the chemical structure of the candidate. Quantification of the candidate content is directly calculated from the ¹H-NMR peak areas and the initial weights of the candidate and reference substance. After ANOVA the resulting standard deviation is included into the uncertainty calculation of the certified value.

UNCERTAINTY CALCULATION

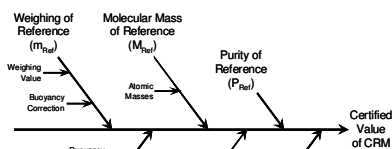
All uncertainties are calculated according to Eurachem/CITAC Guide and reported as combined expanded uncertainties. The uncertainty contributions are illustrated by the following cause-effect diagram.

Typical relative contributions are:

- $u(P_{Ref}) < 0.05\%$
- $u(m_{Ref}) < 0.01\%$
- $u(m_{CRM}) < 0.01\%$
- $u(M_{Ref}) < 0.003\%$
- $u(M_{CRM}) < 0.003\%$
- $u(A_{Signals}) < 0.10\%$

The combined uncertainty is calculated by combination of the squared contribution values.

Expanded uncertainty is then calculated to a confidence level of 95%, typically by multiplying with a confidence level factor of $k=2$.



APPLICATION FORMULA (FOR Q-NMR USE)

$$P_{Sample} = \frac{I_{Sample}}{I_{Ref}} \cdot \frac{N_{Ref}}{N_{Sample}} \cdot \frac{M_{Sample}}{M_{Ref}} \cdot \frac{m_{Ref}}{m_{Sample}} \cdot P_{Ref}$$

- P_{Sample} : Purity of sample as mass fraction (% g/g)
- P_{Ref} : Purity of reference as mass fraction (Benzoic acid = 99.98 % g/g)
- I_{Sample} : Integral of the sample signal
- I_{Ref} : Integral of the reference signal (Benzoic acid)
- N_{Sample} : Number of sample protons
- N_{Ref} : Number of reference protons (Benzoic acid = 5)
- M_{Sample} : Molecular mass of the sample
- M_{Ref} : Molecular mass of the reference (Benzoic acid = 122.12)
- m_{Sample} : Mass of sample
- m_{Ref} : Mass of reference (Benzoic acid)

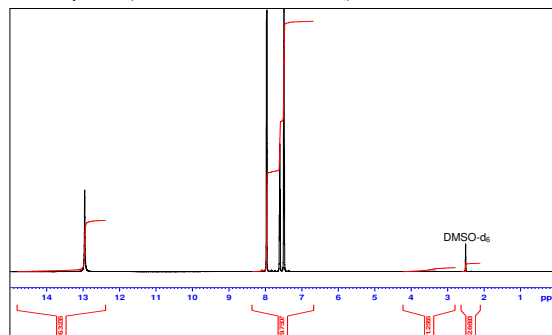
EXTENDED ANALYTICAL DATA

- Solubility**: Benzoic acid is soluble in Methanol-d₄ and DMSO-d₆
- bp**: 249 °C (lit.)
- mp**: 121-125 °C (lit.)

EXTENDED ANALYTICAL DATA (CONT.)

- Total impurities**: ≤0.002 % sulfur compounds (as S)
≤0.005 % halogenated compounds (as Cl)
- Ign. Residue**: ≤0.005 % (as SO₄)
- Transition temp.**: solidification point 122-123 °C
- Anion traces**: sulfate (SO₄²⁻): ≤20 mg/kg
- Cation traces**:
Ca: ≤50 mg/kg
Cd: ≤5 mg/kg
Co: ≤5 mg/kg
Cr: ≤5 mg/kg
Cu: ≤5 mg/kg
Fe: ≤2 mg/kg
K: ≤50 mg/kg
Mg: ≤5 mg/kg
Mn: ≤5 mg/kg
Na: ≤100 mg/kg
Ni: ≤5 mg/kg
Pb: ≤5 mg/kg
Zn: ≤5 mg/kg

¹H-NMR Spectrum (600 MHz, Benzoic acid in DMSO-d₆)



REFERENCES

- ISO Guide 31, 2nd Ed. (2000), "Reference materials - Contents of certificates and labels"
- Eurachem/CITAC Guide, 1st Ed. (2003), "Traceability in chemical measurement"
- ISO Guide 35, 3rd Ed. (2006), "Reference materials - General and statistical principles for certification"
- Eurachem/CITAC Guide, 2nd Ed. (2000), "Quantifying uncertainty in analytical measurement"
- ISO/IEC 17025, 2nd Ed. (2005), "General requirements for the competence of testing and calibration laboratories"
- ISO Guide 34, 3rd Ed. (2009), "General requirements for the competence of reference material producers"