Supelco®
Analytical Products

MERCK

Quantitative NMR

Traceable Internal Standards

Certification Concept and Product List

In recent years, quantitative NMR (qNMR) spectroscopy has become one of the most important tools for the content determination of organic substances and the quantitative evaluation of impurities.

Since 2009, we have been using the qNMR method under ISO/IEC 17025 accreditation for the development of organic certified reference materials (CRMs).

All CRMs are produced under ISO 17034. They comprise a set of over 20 different qNMR standards for ^{1}H, ^{13}P and ^{19}F measurements, and a considerable portfolio of CRMs for further analytical testing.

The graphics to the right and below are illustrating the workflow we follow for the certification of our CRMs.

This flyer can also be downloaded as a pdf at **SigmaAldrich.com/qnmr**

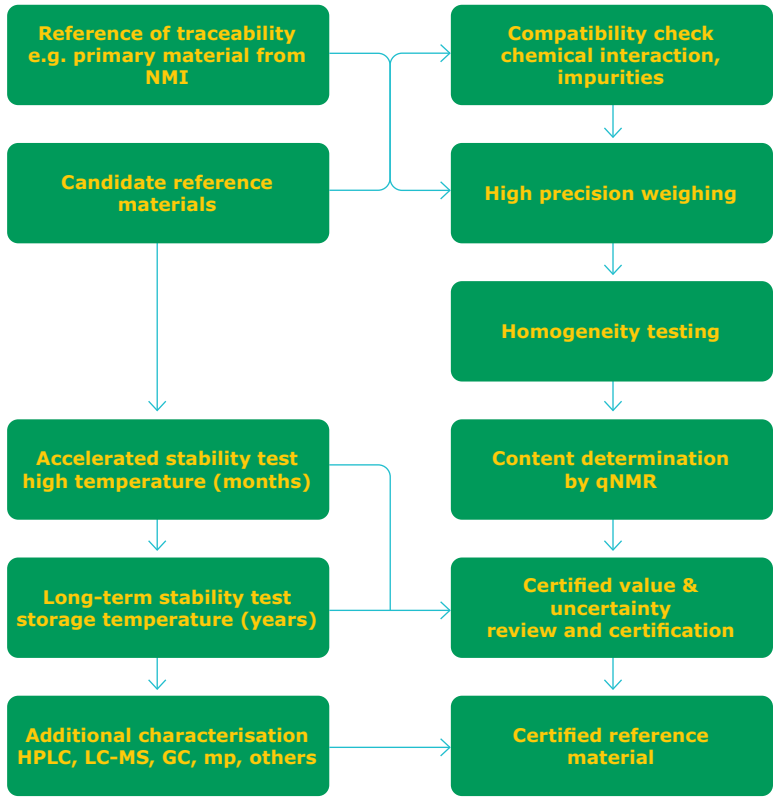
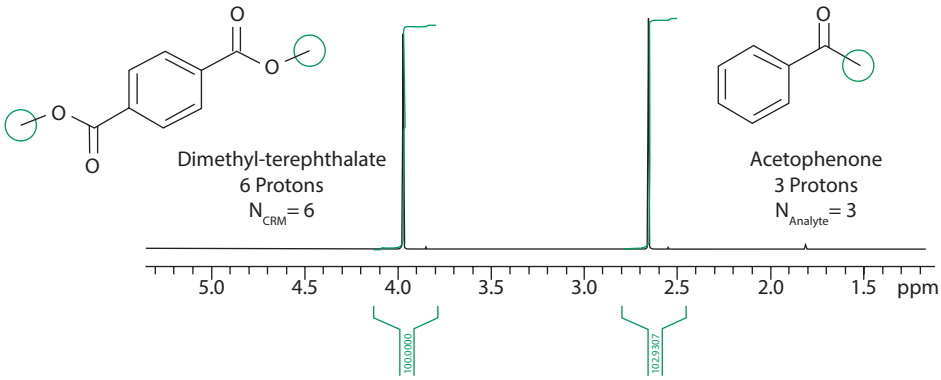


Figure 1. Workflow of the entire process under ISO/IEC 17025 and ISO 17034.



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

Figure 2. Example of a qNMR spectrum of acetophenone (analyte) with internal standard (dimethyl terephthalate) and the formula used to calculate the sample content
(P = purity, I = integral,
N = number of protons,
M = molecular mass,
m = mass)

Standards for ^{1}H quantitative NMR, TraceCERT®

Cat. No.	Substance	D_2O			$CDCl_3$			DMSO-d_6			CD_3OD			CD_3CN		
		δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)
01380	Ethylene carbonate	4.5	5.5	>250	4.5	7.0	>250	4.5	2.7	>250	4.5	5.3	>250	4.5	2.0	>250
03826	Calcium formate	8.4*	17.4*	~80*	-	-	-	-	-	-	-	-	-	-	-	-
06185	Benzoic acid	-	-	-	8.1 7.7 7.5	3.7 4.0 3.4	~150	8.0 7.6 7.5	3.3 3.7 3.0	>250	8.0 7.6 7.5	4.3 4.4 3.9	>250	8.0 7.7 7.5	4.5 2.5 2.6	~80
07038	Dimethyl terephthalate	-	-	-	8.1 4.0	3.6 1.8	~160	8.1 3.9	2.9 1.1	~20	8.1 4.0	4.4 2.4	~4	8.1 3.9	4.9 2.6	~20
14659	Potassium hydrogen phthalate	7.5	2.5	>250	-	-	-	-	-	-	7.9	2.5	~ 5	-	-	-
40384	1,2,4,5-Tetrachloro-3-nitrobenzene	-	-	-	7.8	10.7	>250	8.5	12.6	>250	8.1	6.4	~ 30	8.0	9.6	~10
41867	Dimethyl sulfone	3.0	2.9	>250	3.0	2.7	~80	3.0	2.4	>250	3.0	3.3	~40	2.9	2.6	>250
42582	Ethyl 4 (dimethylamino) benzoate	-	-	-	7.9 6.7 4.3 3.1 1.4	3.8 2.4 2.8 2.0 2.5	>250	7.8 6.7 4.2 3.0 1.3	2.5 1.4 1.9 1.5 2.1	>250	7.9 6.7 4.3 3.0 1.4	3.4 2.5 3.3 2.2 2.7	>250	7.9 6.7 4.3 3.0 1.3	5.6 3.7 4.1 3.6 3.6	~50
50409	Thymol	-	-	-	7.1 6.8 6.6 3.2 2.3 1.3	3.8 4.5 4.8 4.3 3.1 1.9	>250	7.0 6.7 6.5 3.1 2.2 1.1	2.0 2.2 2.6 2.3 2.0 0.9	>250	7.0 6.6 6.5 3.2 2.2 1.2	3.7 4.4 5.8 4.0 2.8 1.8	>250	7.1 6.7 6.6 3.2 2.2 1.2	4.9 5.7 5.7 5.2 3.5 2.4	>250
74599	1,3,5-Trimethoxybenzene	-	-	-	6.1 3.8	4.7 2.2	>250	6.1 3.7	3.2 1.4	>250	6.1 3.8	4.8 2.7	>250	6.1 3.8	5.5 2.8	>250
74658	1,2,4,5-Tetramethylbenzene	-	-	-	7.0 2.2	6.1 4.0	>250	6.9 2.1	4.7 2.6	~10	6.9 2.2	5.9 4.3	~10	6.9 2.2	7.7 5.0	~50
89151	Dimethyl malonic acid	1.3	1.0	~100	-	-	-	1.3	0.7	>250	1.4	1.0	>250	1.4	2.0	~30
92816	Maleic acid	6.3	6.1	>250	-	-	-	6.3	3.0	>250	6.3	3.9	~10	6.4	6.2	~20
94681	Methyl 3,5-dinitrobenzoate	-	-	-	9.3 9.2 4.1	8.0 6.1 2.6	>250	9.1 8.9 4.0	9.4 7.6 1.5	~100	-	-	-	9.1 9.0 4.0	9.0 8.2 3.5	>250
93074	Pentachlorobenzene	-	-	-	7.6	5.8	>250	8.2	13.8	~15	7.9	8.2	~9	7.8	12.2	~15

*: The values for Calcium formate are pH dependent. For a mixture of D_2O and DCl (5:1), the values are: δ : 7.8 ppm, T1: 26.7s, s: >250 mg/mL

Standards for ^{31}P quantitative NMR, TraceCERT®

Cat. No.	Substance	D_2O			$CDCl_3$			DMSO-d_6			CD_3OD			CD_3CN		
		δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)
05498	Triphenyl phosphate	-	-	-	-17.7	2.7	>250	-17.3	1.2	~100	-17.5	3.1	~10	-17	4.3	>250
92214	Potassium phosphate monobasic	0.08	8.0	>250	-	-	-	-	-	-	-	-	-	-	-	-
96708	Phosphonoacetic acid	15.7	4.6	>250	-	-	-	14.9	1.5	>250	17.7	2.9	>250	-	-	-

Standards for ^{19}F quantitative NMR, TraceCERT®

Cat. No.	Substance	D_2O			$CDCl_3$			DMSO-d_6			CD_3OD			CD_3CN		
		δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)	δ (ppm)	T1 (s)	s (mg/mL)
07563	4,4'-Difluoro-benzophenone	-	-	-	-106	2.4	>250	-107	1.4	~150	-108	2.8	~30	-108	2.3	~140
53396	2,4-Dichloro-benzotrifluoride	-	-	-	-63	2.3	>250	-61	1.2	>250	-65	3.3	>250	-63	2.9	>250
80730	2-Chloro-4-fluorotoluene	-	-	-	-116	4.4	>250	-115	3.3	>250	-118	4.8	>250	-117	4.7	>250

The table provides an overview of spectral shifts, relaxation times and solubility in different solvents of 15 ^{1}H-qNMR standards, 3 ^{31}P-qNMR standards and 3 ^{19}F-qNMR standards. They cover the entire spectral range, allowing the quantification of almost any organic molecule by qNMR. Solubility tests were done at room-temperature using commercially available NMR solvents. Tests were performed starting from 1 mg/mL up to 250 mg/mL (mg CRM/mL solvent). T_1 relaxation times were recorded for the CRM only (c ≈ 5 to 20 mg/mL at 25 °C), but may vary in the mixture. Therefore it is recommended to check the T_1 delay prior to the qNMR experiment. (*with DCl or NaOD)

Merck KGaA
Frankfurter Strasse 250
64293 Darmstadt, Germany

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