

# NMR Solvent Data Chart

More Solvents, More Sizes, More Solutions

	<sup>1</sup> H Chemical Shift (ppm from TMS) (multiplicity) ※	JHD (Hz)	<sup>13</sup> C Chemical Shift (ppm from TMS) (multiplicity) ※	JCD (Hz)	<sup>1</sup> H Chemical Shift of HOD (ppm from TMS) ●	Density at 20°C □	Melting point (°C) □	Boiling point (°C) □	Dielectric Constant	Molecular Weight □
Acetic acid-d <sub>4</sub>	11.65 (1) 2.04 (5)	2.2	178.99 (1) 20.0 (7)	20	11.5	1.12	16.7	118	6.1	64.08
Acetone-d <sub>6</sub>	2.05 (5)	2.2	206.68 (1) 29.92 (7)	0.9 19.4	2.8*	0.87	-94	56.5	20.7	64.12
Acetonitrile-d <sub>3</sub>	1.94 (5)	2.5	118.69 (1) 1.39 (7)	21	2.1*	0.84	-45	81.6	37.5	44.07
Benzene-d <sub>6</sub>	7.16 (1)		128.39 (3)	24.3	0.4	0.95	5.5	80.1	2.3	84.15
Chloroform-d	7.24 (1)		77.23 (3)	32.0	1.5*	1.50	-63.5	61-62	4.8	120.38
Cyclohexane-d <sub>12</sub>	1.38 (1)		26.43 (5)	19	0.8	0.89	6.47	80.7	2.0	96.24
Deuterium oxide	4.80 (DSS) 4.81 (TSP)		NA	NA	4.8	1.11	3.81	101.42	78.5	20.03
N, N-Dimethyl-formamide-d <sub>7</sub>	8.03 (1) 2.92 (5) 2.75 (5)	1.9 1.9	163.15 (3) 34.89 (7) 29.76 (7)	29.4 21.0 21.1	3.5	1.03	-61	153	36.7	80.14
Dimethyl sulfoxide-d <sub>6</sub>	2.50 (5)	1.9	39.51 (7)	21.0	3.3*	1.19	18.55	189	46.7	84.17
1,4-Dioxane-d <sub>8</sub>	3.53 (m)		66.66 (5)	21.9	2.4	1.13	11.8	101.1	2.2	96.16
Ethanol-d <sub>6</sub>	5.19 (1) 3.56 (1) 1.11 (m)		56.96 (5) 17.31 (7)	22 19	5.3	0.89	-114.1	78.5	24.5	52.11
Methanol-d <sub>4</sub>	4.78 (1) 3.31 (5)	1.7	49.15 (7)	21.4	4.9	0.89	-97.8	64.7	32.7	36.07
Methylene chloride-d <sub>2</sub>	5.32 (3)	1.1	54.00 (5)	27.2	1.5	1.35	-95	39.75	8.9	86.95
Pyridine-d <sub>5</sub>	8.74 (1) 7.58 (1) 7.22 (1)		150.35 (3) 135.91 (3) 123.87 (3)	27.5 24.5 25	5	1.05	-41.6	115.2-115.3	12.4	84.13
1,1,2,2-Tetrachloroethane-d <sub>2</sub>	6.0		73.78 (3)			1.62	-44	146.5	8.20	169.86
Tetrahydrofuran-d <sub>8</sub>	3.58 (1) 1.73 (1)		67.57 (5) 25.37 (5)	22.2 20.2	2.4-2.5	0.99	-108.5	66	7.6	80.16
Toluene-d <sub>8</sub>	7.09 (m) 7.00 (1) 6.98 (5) 2.09 (5)	2.3	137.86 (1) 129.24 (3) 128.33 (3) 125.49 (3) 20.4 (7)	23 24 24 19	0.4	0.94	-95	110.6	2.4	100.19
Trifluoroacetic acid-d	11.50 (1)		164.2 (4) 116.6 (4)		11.5	1.49	-15.4	72.4		115.03
Trifluoroethanol-d <sub>3</sub>	5.02 (1) 3.88 (4x3)	2(9)	126.3 (4) 61.5 (4x5)	22	5	1.41	-43.5	74.05		103.06

M.J. O'Neil, P.E. Heckelman, C.B. Koch, K.J. Roman, *The Merck Index*, an Encyclopedia of Chemicals, Drugs, and Biologicals – Fourteenth Edition, Merck Co., Inc. Whitehouse Station, NJ 2006.

※ The <sup>1</sup>H spectra of the residual protons and <sup>13</sup>C spectra were obtained on a Varian Gemini 200 spectrometer at 295°K. The NMR solvents used to acquire these spectra contain a maximum of 0.05% and 1.0% TMS (v/v) respectively. Since deuterium has a spin of 1, triplets arising from coupling to deuterium have the intensity ratio of 1:1:1. 'm' denotes a broad peak with some fine structures. It should be noted that chemical shifts can be dependent on solvent, concentration and temperature.

- Approximate values only; may vary with pH, concentration and temperature.
- Melting and boiling points are those of the corresponding unlabeled compound (except for D<sub>2</sub>O). These temperature limits can be used as a guide to determine the useful liquid range of the solvents. Information gathered from the Merck Index – Fourteenth Edition.

\* HOD Peaks – NMR spectra of "neat" deuterated solvent always exhibit a peak due to H<sub>2</sub>O in addition to the residual solvent peak. When the exchange rate between H<sub>2</sub>O and HDO is slow on the NMR timescale the water peak appears as two peaks, a singlet corresponding to H<sub>2</sub>O and a 1:1:1 triplet corresponding to HDO.

## NMR Solvent Storage and Handling Information

Please note that some packaging sizes of some solvents may require special handling not given below. The bottle or ampoule packaging information should be reviewed for further instructions.

**Acetic Acid-d<sub>4</sub> / Acetone-d<sub>6</sub> / Benzene-d<sub>6</sub> / Cyclohexane-d<sub>12</sub> / Deuterium Oxide / N,N-Dimethylformamide-d<sub>7</sub> / Dimethyl Sulfoxide-d<sub>6</sub> / 1,4-Dioxane-d<sub>8</sub> (p-Dioxane) / Ethanol-d<sub>6</sub> / Methanol-d<sub>4</sub> / Methylene Chloride-d<sub>2</sub> / Pyridine-d<sub>5</sub> / 1,1,2,2-Tetrachloroethane-d<sub>2</sub> / Toluene-d<sub>8</sub> / Trifluoroacetic Acid-d / 2,2,2-Trifluoroethanol-d<sub>3</sub>**

Store at room temperature away from light and moisture. The above products are stable if stored under recommended conditions.

### Acetonitrile-d<sub>3</sub>

Store at room temperature away from light and moisture. This product is stable for one year after receipt of order if stored under above conditions (unopened). After one year, the solvent should be re-analyzed for chemical purity before use.

### Chloroform-d / Tetrahydrofuran-d<sub>8</sub>

Store refrigerated (-5° to 5°C) away from light and moisture. These products are stable for six months after receipt of order if stored under above conditions (unopened). After six months, the solvent should be re-analyzed for chemical purity before use.

## Deuterium Exchange of Labile Protons in Deuterated Solvents Containing Residual D<sub>2</sub>O

Some deuterated solvents are prepared by catalytic exchange of protonated solvent with deuterium oxide and carefully purified by distillation. Residual water (H<sub>2</sub>O in equilibrium exchange with D<sub>2</sub>O) is kept to a minimum of 20-200 ppm, the higher value corresponds to the amount in the more hygroscopic solvents. The labile deuterons (and protons) of water are available to exchange with labile protons in the chemist's sample and can result in inaccurate integration ratios. The example below shows that just 100 ppm of D<sub>2</sub>O can cause problems when studying dilute solutions of analytes. A significant decrease in the integral of 1 labile proton may be observed in a sample containing 5 mg organic compound, MW~200, dissolved in 1g DMSO-d<sub>6</sub> containing 100 ppm D<sub>2</sub>O. The problem becomes worse as the molecular weight of the analyte increases.

### Solution

Water (as H<sub>2</sub>O, HDO or D<sub>2</sub>O) can be minimized by adding molecular sieves to the solvent, agitating the mixture and allowing it to stand for a few hours. The water content may be reduced to about 10-20 ppm in this manner. If exchange still causes a problem, it is recommended to use a less hygroscopic solvent, such as chloroform, methylene chloride or acetonitrile.

X – residual solvent      \* – residual water

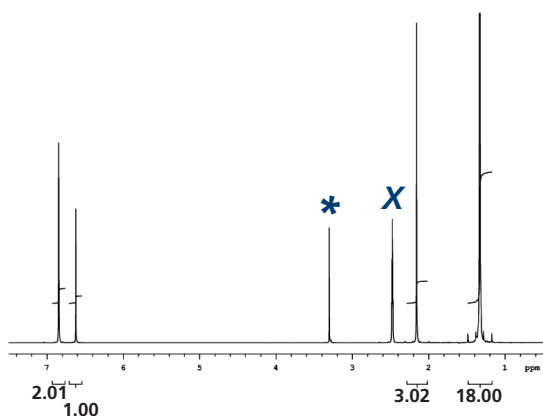


Figure 1. <sup>1</sup>H NMR spectrum of 5.0 mg 2,6-di-tert-butyl-4-methylphenol (MW 220.36g / mole) in dry DMSO-d<sub>6</sub>. Note the proper integral ratios of 18:3:1:2 (t-butyl: methyl: ring-H: -OH). Note the single H<sub>2</sub>O peak at 3.3 ppm.

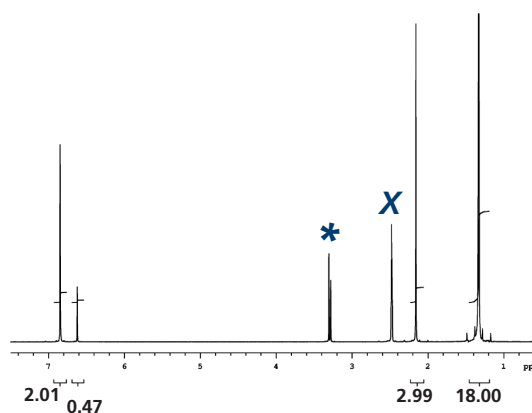


Figure 2. <sup>1</sup>H NMR spectrum of 5.3 mg of 2,6-di-tert-butyl-4-methylphenol in DMSO-d<sub>6</sub> with 100 ppm D<sub>2</sub>O added. Note the reduced ratio of the phenolic proton 18:3:2:0.47 (t-butyl: methyl: ring-H: -OH). Note that the HOH and HOD peaks are separated in the spectrum.

