

# NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist

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Tables of <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts have been compiled for common organic compounds often used as reagents or found as products or contaminants in deuterated organic solvents. Building upon the work of Gottlieb, Kotlyar, and Nudelman in the *Journal of Organic Chemistry*, signals for common impurities are now reported in additional NMR solvents (tetrahydrofuran-*d*<sub>8</sub>, toluene-*d*<sub>8</sub>, dichloromethane-*d*<sub>2</sub>, chlorobenzene-*d*<sub>5</sub>, and 2,2,2-trifluoroethanol-*d*<sub>3</sub>) which are frequently used in organometallic laboratories. Chemical shifts for other organics which are often used as reagents or internal standards or are found as products in organometallic chemistry are also reported for all the listed solvents.

Hanging above the desk of most every chemist whose work relies heavily on using NMR spectroscopy<sup>1</sup> is *NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities* by Gottlieb, Kotlyar, and Nudelman.<sup>2</sup> By compiling the chemical shifts of a large number of contaminants commonly encountered in synthetic chemistry, the publication has become an essential reference, allowing for easy identification of known impurities in a variety of deuterated organic solvents. However, despite the utility of Gottlieb et al.'s work,<sup>3</sup> the chemical shifts of impurities in a number of NMR solvents often used by organometallic chemists were not included. Tetrahydrofuran-*d*<sub>8</sub> (THF-*d*<sub>8</sub>), toluene-*d*<sub>8</sub>, dichloromethane-*d*<sub>2</sub> (CD<sub>2</sub>Cl<sub>2</sub>), chlorobenzene-*d*<sub>5</sub> (C<sub>6</sub>D<sub>5</sub>Cl), and 2,2,2-trifluoroethanol-*d*<sub>3</sub> (TFE-*d*<sub>3</sub>) are commonplace in laboratories practicing inorganic syntheses. Therefore, we have expanded the spectral data compilation with the inclusion of chemical shifts of common impurities recorded in the deuterated solvents heavily employed in our organometallic laboratories. The chemical shifts of various gases (hydrogen, methane, ethane, propane,

ethylene, propylene, and carbon dioxide) often encountered as reagents or products in organometallic reactions, along with organic compounds relevant to organometallic chemists (allyl acetate, benzaldehyde, carbon disulfide, carbon tetrachloride, 18-crown-6, cyclohexanone, diallyl carbonate, dimethyl carbonate, dimethyl malonate, furan, Apiezon H grease, hexamethylbenzene, hexamethyldisiloxane, imidazole, pyrrole, and pyrrolidine), have also been added to this expanded list.

## Experimental Section

All deuterated solvents were obtained commercially through Cambridge Isotope Laboratories, Inc. NMR spectra were recorded at 298 K using 300, 500, or 600 MHz spectrometers (<sup>13</sup>C{<sup>1</sup>H} NMR frequencies of 75.5, 126, or 151 MHz, respectively). Adopting the previously reported strategy,<sup>2</sup> standard solutions of mixtures of specific impurities were used to reduce the number of necessary individual NMR experiments. The combinations of organic compounds were chosen in a way in which intermolecular interactions and resonance convolution would be minimized. Unless otherwise stated, the standard solutions were prepared with qualitatively equal molar amounts of the following compounds: (solution 1) acetone, dimethylformamide, ethanol, toluene; (solution 2) benzene, dimethyl sulfide, ethyl acetate, methanol; (solution 3) acetic acid, chloroform, diethyl ether, 2-propanol, tetrahydrofuran; (solution 4) acetonitrile, dichloromethane, 1,4-dioxane, *n*-hexane, hexamethylphosphoramide (HMPA); (solution 5) 1,2-dichloroethane, *n*-pentane, pyridine, hexamethylbenzene; (solution 6) *tert*-butyl alcohol, 2,6-di-*tert*-butyl-4-methylphenol (BHT), cyclohexane,

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(1) For general information on <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectroscopy, see: Balci, M. *Basic <sup>1</sup>H- and <sup>13</sup>C-NMR Spectroscopy*; Elsevier: Amsterdam, 2005.

(2) Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. *J. Org. Chem.* **1997**, *62*, 7512.

(3) According to ACS Publications as of December 2009 (<http://pubs.acs.org/>), Gottlieb et al.'s publication<sup>2</sup> is the most downloaded *Journal of Organic Chemistry* article over the preceding 12 months.

Table 1. <sup>1</sup>H NMR Data<sup>a</sup>

	proton	mult	THF- <i>d</i> <sub>8</sub>	CD <sub>2</sub> Cl <sub>2</sub>	CDCl <sub>3</sub>	toluene- <i>d</i> <sub>8</sub>	C <sub>6</sub> D <sub>6</sub>	C <sub>6</sub> D <sub>5</sub> Cl	(CD <sub>3</sub> ) <sub>2</sub> CO	(CD <sub>3</sub> ) <sub>2</sub> SO	CD <sub>3</sub> CN	TFE- <i>d</i> <sub>3</sub>	CD <sub>3</sub> OD	D <sub>2</sub> O
solvent residual signals			1.72	5.32	7.26	2.08	7.16	6.96	2.05	2.50	1.94	5.02	3.31	4.79
			3.58			6.97		6.99				3.88		
						7.01		7.14						
						7.09								
water	OH	s	2.46	1.52	1.56	0.43	0.40	1.03	2.84 <sup>b</sup>	3.33 <sup>b</sup>	2.13	3.66	4.87	
acetic acid	CH <sub>3</sub>	s	1.89	2.06	2.10	1.57	1.52	1.76	1.96	1.91	1.96	2.06	1.99	2.08
acetone	CH <sub>3</sub>	s	2.05	2.12	2.17	1.57	1.55	1.77	2.09	2.09	2.08	2.19	2.15	2.22
acetonitrile	CH <sub>3</sub>	s	1.95	1.97	2.10	0.69	0.58	1.21	2.05	2.07	1.96	1.95	2.03	2.06
benzene	CH	s	7.31	7.35	7.36	7.12	7.15	7.20	7.36	7.37	7.37	7.36	7.33	7.33
<i>tert</i> -butyl alcohol	CH <sub>3</sub>	s	1.15	1.24	1.28	1.03	1.05	1.12	1.18	1.11	1.16	1.28	1.40	1.24
	OH	s <sup>c</sup>	3.16			0.58	0.63	1.30			4.19	2.18	2.20	
chloroform	CH	s	7.89	7.32	7.26	6.10	6.15	6.74	8.02	8.32	7.58	7.33	7.90	
18-crown-6	CH <sub>2</sub>	s	3.57	3.59	3.67	3.36	3.39	3.41	3.59	3.51	3.51	3.64	3.64	3.80
cyclohexane	CH <sub>2</sub>	s	1.44	1.44	1.43	1.40	1.40	1.37	1.43	1.40	1.44	1.47	1.45	
1,2-dichloroethane	CH <sub>2</sub>	s	3.77	3.76	3.73	2.91	2.90	3.26	3.87	3.90	3.81	3.71	3.78	
dichloromethane	CH <sub>2</sub>	s	5.51	5.33	5.30	4.32	4.27	4.77	5.63	5.76	5.44	5.24	5.49	
diethyl ether	CH <sub>3</sub>	t, 7	1.12	1.15	1.21	1.10	1.11	1.10	1.11	1.09	1.12	1.20	1.18	1.17
	CH <sub>2</sub>	q, 7	3.38	3.43	3.48	3.25	3.26	3.31	3.41	3.38	3.42	3.58	3.49	3.56
diglyme	CH <sub>2</sub>	m	3.43	3.57	3.65	3.43	3.46	3.49	3.56	3.51	3.53	3.67	3.61	3.67
	CH <sub>2</sub>	m	3.53	3.50	3.57	3.31	3.34	3.37	3.47	3.38	3.45	3.62	3.58	3.61
	OCH <sub>3</sub>	s	3.28	3.33	3.39	3.12	3.11	3.16	3.28	3.24	3.29	3.41	3.35	3.37
dimethylformamide	CH	s	7.91	7.96	8.02	7.57	7.63	7.73	7.96	7.95	7.92	7.86	7.97	7.92
	CH <sub>3</sub>	s	2.88	2.91	2.96	2.37	2.36	2.51	2.94	2.89	2.89	2.98	2.99	3.01
	CH <sub>3</sub>	s	2.76	2.82	2.88	1.96	1.86	2.30	2.78	2.73	2.77	2.88	2.86	2.85
1,4-dioxane	CH <sub>2</sub>	s	3.56	3.65	3.71	3.33	3.35	3.45	3.59	3.57	3.60	3.76	3.66	3.75
DME	CH <sub>3</sub>	s	3.28	3.34	3.40	3.12	3.12	3.17	3.28	3.24	3.28	3.40	3.35	3.37
	CH <sub>2</sub>	s	3.43	3.49	3.55	3.31	3.33	3.37	3.46	3.43	3.45	3.61	3.52	3.60
ethane	CH <sub>3</sub>	s	0.85	0.85	0.87	0.81	0.80	0.79	0.83	0.82	0.85	0.85	0.85	0.82
ethanol	CH <sub>3</sub>	t, 7	1.10	1.19	1.25	0.97	0.96	1.06	1.12	1.06	1.12	1.22	1.19	1.17
	CH <sub>2</sub>	q, 7 <sup>d</sup>	3.51	3.66	3.72	3.36	3.34	3.51	3.57	3.44	3.54	3.71	3.60	3.65
	OH	s <sup>c,d</sup>	3.30	1.33	1.32	0.83	0.50	1.39	3.39	4.63	2.47			
ethyl acetate	CH <sub>3</sub> CO	s	1.94	2.00	2.05	1.69	1.65	1.78	1.97	1.99	1.97	2.03	2.01	2.07
	CH <sub>2</sub> CH <sub>3</sub>	q, 7	4.04	4.08	4.12	3.87	3.89	3.96	4.05	4.03	4.06	4.14	4.09	4.14
	CH <sub>2</sub> CH <sub>3</sub>	t, 7	1.19	1.23	1.26	0.94	0.92	1.04	1.20	1.17	1.20	1.26	1.24	1.24
ethylene	CH <sub>2</sub>	s	5.36	5.40	5.40	5.25	5.25	5.29	5.38	5.41	5.41	5.40	5.39	5.44
ethylene glycol	CH <sub>2</sub>	s <sup>e</sup>	3.48	3.66	3.76	3.36	3.41	3.58	3.28	3.34	3.51	3.72	3.59	3.65
H grease <sup>f</sup>	CH <sub>3</sub>	m	0.85–0.91	0.84–0.90	0.84–0.87	0.89–0.96	0.90–0.98	0.86–0.92	0.90	0.82–0.88		0.88–0.94	0.86–0.93	
	CH <sub>2</sub>	br s	1.29	1.27	1.25	1.33	1.32	1.30	1.29	1.24		1.33	1.29	
hexamethylbenzene	CH <sub>3</sub>	s	2.18	2.20	2.24	2.10	2.13	2.10	2.17	2.14	2.19	2.24	2.19	
<i>n</i> -hexane	CH <sub>3</sub>	t, 7	0.89	0.89	0.88	0.88	0.89	0.85	0.88	0.86	0.89	0.91	0.90	
	CH <sub>2</sub>	m	1.29	1.27	1.26	1.22	1.24	1.19	1.28	1.25	1.28	1.31	1.29	
HMDSO	CH <sub>3</sub>	s	0.07	0.07	0.07	0.10	0.12	0.10	0.07	0.06	0.07	0.08	0.07	0.28
HMPA	CH <sub>3</sub>	d, 9.5	2.58	2.60	2.65	2.42	2.40	2.47	2.59	2.53	2.57	2.63	2.64	2.61
hydrogen	H <sub>2</sub>	s	4.55	4.59	4.62	4.50	4.47	4.49	4.54	4.61	4.57	4.53	4.56	
imidazole	CH(2)	s	7.48	7.63	7.67	7.30	7.33	7.53	7.62	7.63	7.57	7.61	7.67	7.78
	CH(4,5)	s	6.94	7.07	7.10	6.86	6.90	7.01	7.04	7.01	7.01	7.03	7.05	7.14
methane	CH <sub>4</sub>	s	0.19	0.21	0.22	0.17	0.16	0.15	0.17	0.20	0.20	0.18	0.20	0.18
methanol	CH <sub>3</sub>	s <sup>g</sup>	3.27	3.42	3.49	3.03	3.07	3.25	3.31	3.16	3.28	3.44	3.34	3.34
	OH	s <sup>c,g</sup>	3.02	1.09	1.09			1.30	3.12	4.01	2.16			
nitromethane	CH <sub>3</sub>	s	4.31	4.31	4.33	3.01	2.94	3.59	4.43	4.42	4.31	4.28	4.34	4.40
<i>n</i> -pentane	CH <sub>3</sub>	t, 7	0.89	0.89	0.88	0.87	0.87	0.84	0.88	0.86	0.89	0.90	0.90	
	CH <sub>2</sub>	m	1.31	1.30	1.27	1.25	1.23	1.23	1.27	1.27	1.29	1.33	1.29	
propane	CH <sub>3</sub>	t, 7.3	0.90	0.90	0.90	0.89	0.86	0.84	0.88	0.87	0.90	0.90	0.91	0.88
	CH <sub>2</sub>	sept, 7.3	1.33	1.32	1.32	1.32	1.26	1.26	1.31	1.29	1.33	1.33	1.34	1.30
2-propanol	CH <sub>3</sub>	d, 6	1.08	1.17	1.22	0.95	0.95	1.04	1.10	1.04	1.09	1.20	1.50	1.17
	CH	sept, 6	3.82	3.97	4.04	3.65	3.67	3.82	3.90	3.78	3.87	4.05	3.92	4.02
propylene	CH <sub>3</sub>	dt, 6.4, 1.5	1.69	1.71	1.73	1.55	1.55	1.58	1.68	1.68	1.70	1.70	1.70	1.70
	CH <sub>2</sub> (1)	dm, 10	4.89	4.93	4.94	4.92	4.95	4.91	4.90	4.94	4.93	4.93	4.91	4.95
	CH <sub>2</sub> (2)	dm, 17	4.99	5.03	5.03	4.98	5.01	4.98	5.00	5.03	5.04	5.03	5.01	5.06
	CH	m	5.79	5.84	5.83	5.70	5.72	5.72	5.81	5.80	5.85	5.87	5.82	5.90
pyridine	CH(2,6)	m	8.54	8.59	8.62	8.47	8.53	8.51	8.58	8.58	8.57	8.45	8.53	8.52
	CH(3,5)	m	7.25	7.28	7.29	6.67	6.66	6.90	7.35	7.39	7.33	7.40	7.44	7.45
	CH(4)	m	7.65	7.68	7.68	6.99	6.98	7.25	7.76	7.79	7.73	7.82	7.85	7.87
pyrrole	NH	br t	9.96	8.69	8.40	7.71	7.80	8.61	10.02	10.75	9.27			
	CH(2,5)	m	6.66	6.79	6.83	6.43	6.48	6.62	6.77	6.73	6.75	6.84	6.72	6.93
	CH(3,4)	m	6.02	6.19	6.26	6.27	6.37	6.27	6.07	6.01	6.10	6.24	6.08	6.26
pyrrolidine <sup>h</sup>	CH <sub>2</sub> (2,5)	m	2.75	2.82	2.87	2.54	2.54	2.64		2.67	2.75	3.11	2.80	3.07
	CH <sub>2</sub> (3,4)	m	1.59	1.67	1.68	1.36	1.33	1.43	1.55	1.61	1.93	1.72	1.87	
silicone grease	CH <sub>3</sub>	s	0.11	0.09	0.07	0.26	0.29	0.14	0.13	–0.06	0.08	0.16	0.10	
tetrahydrofuran	CH <sub>2</sub> (2,5)	m	3.62	3.69	3.76	3.54	3.57	3.59	3.63	3.60	3.64	3.78	3.71	3.74
	CH <sub>2</sub> (3,4)	m	1.79	1.82	1.85	1.43	1.40	1.55	1.79	1.76	1.80	1.91	1.87	1.88
toluene	CH <sub>3</sub>	s	2.31	2.34	2.36	2.11	2.11	2.16	2.32	2.30	2.33	2.33	2.32	
	CH(2,4,6)	m	7.10	7.15	7.17	6.96–7.01	7.02	7.01–7.08	7.10–7.20	7.18	7.10–7.30	7.10–7.30	7.16	
	CH(3,5)	m	7.19	7.24	7.25	7.09	7.13	7.10–7.17	7.10–7.20	7.25	7.10–7.30	7.10–7.30	7.16	
triethylamine	CH <sub>3</sub>	t, 7	0.97	0.99	1.03	0.95	0.96	0.93	0.96	0.93	0.96	1.31	1.05	0.99
	CH <sub>2</sub>	q, 7	2.46	2.48	2.53	2.39	2.40	2.39	2.45	2.43	2.45	3.12	2.58	2.57

<sup>a</sup> Except for the compounds in solutions 8–10, as well as the gas samples, hexamethylbenzene, and the corrected values mentioned in the Supporting Information, all data for the solvents CDCl<sub>3</sub>, C<sub>6</sub>D<sub>6</sub>, (CD<sub>3</sub>)<sub>2</sub>CO, (CD<sub>3</sub>)<sub>2</sub>SO, CD<sub>3</sub>CN, CD<sub>3</sub>OD, and D<sub>2</sub>O were previously reported in ref 2. <sup>b</sup> A signal for HDO is also observed in (CD<sub>3</sub>)<sub>2</sub>SO (3.30 ppm) and (CD<sub>3</sub>)<sub>2</sub>CO (2.81 ppm), often seen as a 1:1:1 triplet (<sup>2</sup>J<sub>H,D</sub> = 1 Hz). <sup>c</sup> Not all OH signals were observable. <sup>d</sup> In some solvents, the coupling interaction between the CH<sub>2</sub> and the OH protons may be observed (*J* = 5 Hz). <sup>e</sup> In CD<sub>3</sub>CN, the OH proton was seen as a multiplet at 2.69 ppm, as well as extra coupling to the CH<sub>3</sub> resonance. <sup>f</sup> Apiezon brand H grease. <sup>g</sup> In some solvents, a coupling interaction between the CH<sub>3</sub> and the OH protons may be observed (*J* = 5.5 Hz). <sup>h</sup> Pyrrolidine was observed to react with (CD<sub>3</sub>)<sub>2</sub>CO.

Table 2.  $^{13}\text{C}\{^1\text{H}\}$  NMR Data<sup>a</sup>

	carbon	THF- <i>d</i> <sub>6</sub>	CD <sub>2</sub> Cl <sub>2</sub>	CDCl <sub>3</sub>	toluene- <i>d</i> <sub>8</sub>	C <sub>6</sub> D <sub>6</sub>	C <sub>6</sub> D <sub>5</sub> Cl	(CD <sub>3</sub> ) <sub>2</sub> CO	(CD <sub>3</sub> ) <sub>2</sub> SO	CD <sub>3</sub> CN	TFE- <i>d</i> <sub>3</sub>	CD <sub>3</sub> OD	D <sub>2</sub> O
solvent signals		67.21	53.84	77.16	137.48	128.06	134.19	29.84	39.52	1.32	61.50	49.00	
		25.31			128.87		129.26	206.26		118.26	126.28		
					127.96		128.25						
					125.13		125.96						
acetic acid	CO	171.69	175.85	175.99	175.30	175.82	175.67	172.31	171.93	173.21	177.96	175.11	177.21
	CH <sub>3</sub>	20.13	20.91	20.81	20.27	20.37	20.40	20.51	20.95	20.73	20.91	20.56	21.03
acetone	CO	204.19	206.78	207.07	204.00	204.43	204.83	205.87	206.31	207.43	32.35	209.67	215.94
	CH <sub>3</sub>	30.17	31.00	30.92	30.03	30.14	30.12	30.60	30.56	30.91	214.98	30.67	30.89
acetonitrile	CN	116.79	116.92	116.43	115.76	116.02	115.93	117.60	117.91	118.26	118.95	118.06	119.68
	CH <sub>3</sub>	0.45	2.03	1.89	0.03	0.20	0.63	1.12	1.03	1.79	1.00	0.85	1.47
benzene	CH	128.84	128.68	128.37	128.57	128.62	128.38	129.15	128.30	129.32	129.84	129.34	
<i>tert</i> -butyl alcohol	(CH <sub>3</sub> ) <sub>3</sub> C	67.50	69.11	69.15	68.12	68.19	68.19	68.13	66.88	68.74	72.35	69.40	70.36
	(CH <sub>3</sub> ) <sub>2</sub> C	30.57	31.46	31.25	30.49	30.47	31.13	30.72	30.38	30.68	31.07	30.91	30.29
carbon dioxide	CO <sub>2</sub>	125.69	125.26	124.99	124.86	124.76	126.08	125.81	124.21	125.89	126.92	126.31	
carbon disulfide	CS <sub>2</sub>	193.37	192.95	192.83	192.71	192.69	192.49	193.58	192.63	193.60	196.26	193.82	197.25
carbon tetrachloride	CCl <sub>4</sub>	96.89	96.52	96.34	96.57	96.44	96.38	96.65	95.44	96.68	97.74	97.21	96.73
chloroform	CH	79.24	77.99	77.36	77.89	77.79	77.67	79.19	79.16	79.17	78.83	79.44	
18-crown-6	CH <sub>2</sub>	71.34	70.47	70.55	70.86	70.59	70.55	71.25	69.85	71.22	70.80	71.47	70.14
cyclohexane	CH <sub>2</sub>	27.58	27.38	26.94	27.31	27.23	26.99	27.51	26.33	27.63	28.34	27.96	
1,2-dichloroethane	CH <sub>2</sub>	44.64	44.35	43.50	43.40	43.59	43.60	45.25	45.02	45.54	45.28	45.11	
dichloromethane	CH <sub>2</sub>	54.67	54.24	53.52	53.47	53.46	53.54	54.95	54.84	55.32	54.46	54.78	
diethyl ether	CH <sub>3</sub>	15.49	15.44	15.20	15.47	15.46	15.35	15.78	15.12	15.63	15.33	15.46	14.77
	CH <sub>2</sub>	66.14	66.11	65.91	65.94	65.94	65.79	66.12	62.05	66.32	67.55	66.88	66.42
diglyme	CH <sub>3</sub>	58.72	58.95	59.01	58.62	58.66	58.42	58.77	59.98	58.90	59.40	59.06	58.67
	CH <sub>2</sub>	71.17	70.70	70.51	70.92	70.87	70.56	71.03	69.54	70.99	73.05	71.33	70.05
dimethylformamide	CH <sub>2</sub>	72.72	72.25	71.90	72.39	72.35	72.07	72.63	71.25	72.63	71.33	72.92	71.63
	CH	161.96	162.57	162.62	161.93	162.13	162.01	162.79	162.29	163.31	166.01	164.73	165.53
	CH <sub>3</sub>	35.65	36.56	36.50	35.22	35.25	35.45	36.15	35.73	36.57	37.76	36.89	37.54
	CH <sub>3</sub>	30.70	31.39	31.45	30.64	30.72	30.71	31.03	30.73	31.32	30.96	31.61	32.03
1,4-dioxane	CH <sub>2</sub>	67.65	67.47	67.14	67.17	67.16	66.95	67.60	66.36	67.72	68.52	68.11	67.19
DME	CH <sub>3</sub>	58.72	59.02	59.08	58.63	58.68	58.31	58.45	58.03	58.89	59.52	59.06	58.67
	CH <sub>2</sub>	72.58	72.24	71.84	72.25	72.21	71.81	72.47	71.17	72.47	72.87	72.72	71.49
ethane	CH <sub>3</sub>	6.79	6.91	6.89	6.94	6.96	6.91	6.88	6.61	6.99	7.01	6.98	
ethanol	CH <sub>3</sub>	18.90	18.69	18.41	18.78	18.72	18.55	18.89	18.51	18.80	18.11	18.40	17.47
	CH <sub>2</sub>	57.60	58.57	58.28	57.81	57.86	57.63	57.72	56.07	57.96	59.68	58.26	58.05
ethyl acetate	CH <sub>3</sub> CO	20.45	21.15	21.04	20.46	20.56	20.50	20.83	20.68	21.16	21.18	20.88	21.15
	CO	170.32	171.24	171.36	170.02	170.44	170.20	170.96	170.31	171.68	175.55	172.89	175.26
	CH <sub>2</sub>	60.30	60.63	60.49	60.08	60.21	60.06	60.56	59.74	60.98	62.70	61.50	62.32
ethylene glycol	CH <sub>3</sub>	14.37	14.37	14.19	14.23	14.19	14.07	14.50	14.40	14.54	14.36	14.49	13.92
	CH <sub>2</sub>	123.09	123.20	123.13	122.92	122.96	122.95	123.47	123.52	123.69	124.08	123.46	
ethylene glycol	CH <sub>2</sub>	64.35	64.08	63.79	64.29	64.34	64.03	64.26	62.76	64.22	64.87	64.30	63.17
H grease <sup>b</sup>	CH <sub>2</sub>	30.45	30.14	29.71	30.31	30.22	30.11						
hexamethylbenzene	C	131.88	132.09	132.21	131.72	131.79	131.54	132.22	131.10	132.61	134.04	132.53	
	CH <sub>3</sub>	16.71	16.93	16.98	16.84	16.95	16.68	16.86	16.60	16.94	17.04	16.90	
<i>n</i> -hexane	CH <sub>3</sub>	14.22	14.28	14.14	14.34	14.32	14.18	14.34	13.88	14.43	14.63	14.45	
	CH <sub>2</sub> (2,5)	23.33	23.07	22.70	23.12	23.04	22.86	23.28	22.05	23.40	24.06	23.68	
	CH <sub>2</sub> (3,4)	32.34	32.01	31.64	32.06	31.96	31.77	32.30	30.95	32.36	33.17	32.73	
HMDSO	CH <sub>3</sub>	1.83	1.96	1.97	1.99	2.05	1.92	2.01	1.96	2.07	2.09	1.99	2.31
HMPA <sup>c</sup>	CH <sub>3</sub>	36.89	36.99	36.87	36.80	36.88	36.64	37.04	36.42	37.10	37.21	37.00	36.46
imidazole	CH(2)	135.72	135.76	135.38	135.57	135.76	135.50	135.89	135.15	136.33	136.58	136.31	136.65
	CH(4,5)	122.20	122.16	122.00	122.13	122.16	121.96	122.31	121.55	122.78	122.93	122.60	122.43
methane	CH <sub>4</sub>	-4.90	-4.33	-4.63	-4.34	-4.29	-4.33	-5.33	-4.01	-4.61	-5.88	-4.90	
methanol	CH <sub>3</sub>	49.64	50.45	50.41	49.90	49.97	49.66	49.77	48.59	49.90	50.67	49.86	49.50 <sup>d</sup>
nitromethane	CH <sub>3</sub>	62.49	63.03	62.50	61.14	61.16	61.68	63.21	63.28	63.66	63.17	63.08	63.22
<i>n</i> -pentane	CH <sub>3</sub>	14.18	14.24	14.08	14.27	14.25	14.10	14.29	13.28	14.37	14.54	14.39	
	CH <sub>2</sub> (2,4)	23.00	22.77	22.38	22.79	22.72	22.54	22.98	21.70	23.08	23.75	23.38	
	CH <sub>2</sub> (3)	34.87	34.57	34.16	34.54	34.45	34.26	34.83	33.48	34.89	35.76	35.30	
propane	CH <sub>3</sub>	16.60	16.63	16.63	16.65	16.66	16.56	16.68	16.34	16.73	16.93	16.80	
	CH <sub>2</sub>	16.82	16.63	16.37	16.63	16.60	16.48	16.78	15.67	16.91	17.46	17.19	
2-propanol	CH <sub>3</sub>	25.70	25.43	25.14	25.24	25.18	25.14	25.67	25.43	25.55	25.21	25.27	24.38
	CH	66.14	64.67	64.50	64.12	64.23	64.18	63.85	64.92	64.30	66.69	64.71	64.88
propylene	CH <sub>3</sub>	19.27	19.47	19.50	19.32	19.38	19.32	19.42	19.20	19.48	19.63	19.50	
	CH <sub>2</sub>	115.74	115.70	115.74	115.89	115.92	115.86	116.03	116.07	116.12	116.38	116.04	
	CH	134.02	134.21	133.91	133.61	133.69	133.57	134.34	133.55	134.78	136.00	134.61	
pyridine	CH(2,6)	150.57	150.27	149.90	150.25	150.27	149.93	150.67	149.58	150.76	149.76	150.07	149.18
	CH(3,5)	124.08	124.06	123.75	123.46	123.58	123.49	124.57	123.84	127.76	126.27	125.53	125.12
	CH(4)	135.99	136.16	135.96	135.17	135.28	135.32	136.56	136.05	136.89	139.62	138.35	138.27
pyrrole	CH(2,5)	118.03	117.93	117.77	117.61	117.78	117.65	117.98	117.32	118.47	119.61	118.28	119.06
	CH(3,4)	107.74	108.02	107.98	108.15	108.21	108.03	108.04	107.07	108.31	108.85	108.11	107.83
pyrrolidine <sup>e</sup>	CH <sub>2</sub> (2,5)	45.82	47.02	46.93	47.12	46.86	46.75		46.51	47.57	47.43	47.23	46.83
	CH <sub>2</sub> (3,4)	26.17	25.83	25.56	25.75	25.65	25.59		25.26	26.34	25.73	26.29	25.86
silicone grease	CH <sub>3</sub>	1.20	1.22	1.19	1.37	1.38	1.09	1.40			2.87	2.10	
	CH <sub>2</sub> (2,5)	68.03	68.16	67.97	67.75	67.80	67.64	68.07	67.03	68.33	69.53	68.83	68.68
tetrahydrofuran	CH <sub>2</sub> (3,4)	26.19	25.98	25.62	25.79	25.72	25.68	26.15	25.14	26.27	26.69	26.48	25.67
	CH <sub>3</sub>	21.29	21.53	21.46	21.37	21.10	21.23	21.46	20.99	21.50	21.62	21.50	
toluene	C(1)	138.24	138.36	137.89	137.84	137.91	137.65	138.48	137.35	138.90	139.92	138.85	
	CH(2,6)	129.47	129.35	129.07	129.33	129.33	129.12	129.76	128.88	129.94	130.58	129.91	

1,2-dimethoxyethane (DME), nitromethane, poly(dimethylsiloxane) (silicone grease), triethylamine; (solution 7) diglyme, dimethylacetamide, ethylene glycol, ethyl methyl ketone; (solution 8) allyl acetate, 2,6-di-*tert*-butyl-4-methoxyphenol (BHA), long-chain, linear aliphatic hydrocarbons from pump oil;<sup>4</sup> (solution 9) benzaldehyde, carbon disulfide, carbon tetrachloride, cyclohexanone, dimethyl malonate, furan, Apiezon H grease (H grease); (solution 10) 18-crown-6, diallyl carbonate, dimethyl carbonate, hexamethyldisiloxane (HMDSO), imidazole, pyrrole, pyrrolidine.<sup>5</sup> In the case of TFE-*d*<sub>3</sub>, nitromethane was omitted from solution 6 and run separately, since the protons of nitromethane exchange with deuterium from TFE-*d*<sub>3</sub> in the presence of triethylamine. In the case of (CD<sub>3</sub>)<sub>2</sub>CO, pyrrolidine was omitted from solution 10, since the two compounds were observed to react with each other. The gases used in this study included hydrogen, methane, ethane, propane, ethylene, propylene, and carbon dioxide.

Before examining the various standard contaminant solutions by <sup>1</sup>H NMR spectroscopy, solvent residual signals<sup>6</sup> and chemical shifts for H<sub>2</sub>O<sup>7</sup> for each NMR solvent were referenced against tetramethylsilane (TMS, δ 0 ppm) and reported. Before collecting <sup>13</sup>C{<sup>1</sup>H} NMR spectral data, solvent signals<sup>6</sup> were recorded with reference to the signal of a TMS internal standard. For D<sub>2</sub>O, <sup>1</sup>H NMR spectra were referenced to the methyl signal (δ 0 ppm) of sodium 3-(trimethylsilyl)propane-sulfonate,<sup>8,9</sup> and <sup>13</sup>C{<sup>1</sup>H} NMR spectra were referenced to the signal for the methyl group of methanol (one drop, added as an internal standard), which was set to 49.50 ppm.<sup>2</sup>

In a typical experiment for collecting <sup>1</sup>H NMR spectral data, a 3 μL sample of a standard contaminant solution was added to an NMR tube containing approximately 0.4 mL of a deuterated solvent. For <sup>13</sup>C{<sup>1</sup>H} NMR spectral data collection, an approximately 50 μL sample of the standard contaminant solution was added. When there was any uncertainty in the assignment of a resonance, the solution was spiked with an additional 1–2 μL of the impurity in question to accurately identify its chemical shift. In cases where the chemical shifts of resonances were highly dependent on the concentration of the impurities present, ambiguous resonances were instead resolved via gradient-

(4) VWR brand vacuum pump oil #19.

(5) The components of solution 10 were stable together in dilute solution but unstable when neat mixtures were prepared. In general, it was observed that the nitrogen-containing compounds and possibly 18-crown-6 catalyzed the hydrolysis of the carbonates, reacted directly with them, or both. Therefore, for the purpose of storage, the solution was partitioned into two subsolutions: (solution 10A) 18-crown-6, imidazole, pyrrole, pyrrolidine; (solution 10B) diallyl carbonate, dimethyl carbonate, hexamethyldisiloxane. These subsolutions were stable for long periods as neat mixtures and were combined to form solution 10 by adding equal portions to an NMR tube containing the desired deuterated solvent.

(6) For <sup>1</sup>H NMR spectra, the solvent residual signals arise from the proton of isotopomers containing one less deuterium atom than the perdeuterated solvent: e.g., CDHCl<sub>2</sub> in CD<sub>2</sub>Cl<sub>2</sub>. For <sup>13</sup>C NMR spectra, the solvent signals arise from the <sup>13</sup>C atoms at natural abundance in the perdeuterated solvent.

(7) The chemical shift for H<sub>2</sub>O can vary depending on the temperature, [H<sub>2</sub>O], and the solutes present: e.g., a downfield shift may be observed in the presence of any hydrogen bond acceptors. For more information see page 75 of ref 1.

(8) Harris, R. K.; Becker, E. D.; Cabral de Menezes, S. M.; Granger, P.; Hoffman, R. E.; Zilm, K. W. *Pure Appl. Chem.* **2008**, *80*, 59.

(9) For information on the temperature dependence of HDO chemical shifts in D<sub>2</sub>O, see ref 2.

selected heteronuclear single-quantum coherence (gs-HSQC) and gradient-selected heteronuclear multiple-quantum coherence (gs-HMQC) NMR spectroscopies. For the experiments involving gases, a J. Young NMR tube containing approximately 0.4 mL of NMR solvent was first degassed with three freeze–pump–thaw cycles. Using a vacuum line equipped with a gas manifold, 1 atm of the desired gas was added to the tube. Each gas was run separately, degassing between each gas sample.

## Results and Discussion

Chemical shifts for each of the impurities are reported in the tables: <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectral data of all substrates are presented in Tables 1 and 2, respectively. Notably, physically larger tables, containing all the data from Tables 1 and 2 as well as the chemical shifts of additional organic compounds, are provided in the Supporting Information. Unless noted otherwise, coupling constants (reported in Hz) and resonance multiplicities (abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, sept = septet, m = multiplet, br = broad) were observed to be solvent-independent.

It was noted that the amount of gas dissolved in solution gave <sup>1</sup>H NMR signal integrations that were qualitatively comparable to those for the solutions made with the 3 μL additions of the liquid or solid contaminants. However, typically in order to observe signals for the gas samples by <sup>13</sup>C{<sup>1</sup>H} NMR spectroscopy, additional time for data collection was required. The solubility of each gas in D<sub>2</sub>O was extremely limited, making <sup>13</sup>C detection impractical. Of all the gases, methane required the most number of transients in order to obtain an observable signal by <sup>13</sup>C{<sup>1</sup>H} NMR spectroscopy. In most cases, the <sup>13</sup>C chemical shift of methane was acquired through the use of gs-HMQC NMR spectroscopy to provide enhanced sensitivity. In order to reflect what would be observed in typical NMR-scale experiments, <sup>13</sup>C detection was not pursued with isotopically enriched gases. A number of misreported values were discovered in the years since the original publication<sup>10</sup> and in the preparation of this paper. These are detailed in the Supporting Information, and the values are now correctly listed in Tables 1 and 2.

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**Supporting Information Available:** Large-format tables of the all the NMR data. This material is available free of charge via the Internet at <http://pubs.acs.org>.

(10) The misreported value for acetonitrile in C<sub>6</sub>D<sub>6</sub> from the original paper<sup>2</sup> was also pointed out by Dr. Jongwook Choi, to whom we are grateful.

## NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities

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In the course of the routine use of NMR as an aid for organic chemistry, a day-to-day problem is the identification of signals deriving from common contaminants (water, solvents, stabilizers, oils) in less-than-analytically-pure samples. This data may be available in the literature, but the time involved in searching for it may be considerable. Another issue is the concentration dependence of chemical shifts (especially  $^1\text{H}$ ); results obtained two or three decades ago usually refer to much more concentrated samples, and run at lower magnetic fields, than today's practice.

We therefore decided to collect  $^1\text{H}$  and  $^{13}\text{C}$  chemical shifts of what are, in our experience, the most popular "extra peaks" in a variety of commonly used NMR solvents, in the hope that this will be of assistance to the practicing chemist.

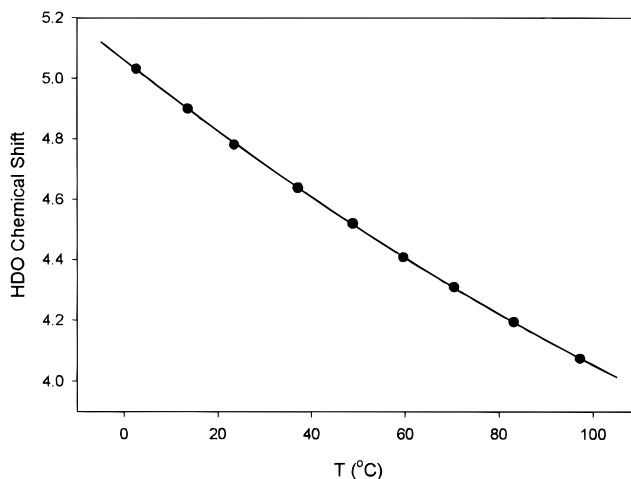
### Experimental Section

NMR spectra were taken in a Bruker DPX-300 instrument (300.1 and 75.5 MHz for  $^1\text{H}$  and  $^{13}\text{C}$ , respectively). Unless otherwise indicated, all were run at room temperature ( $24 \pm 1$  °C). For the experiments in the last section of this paper, probe temperatures were measured with a calibrated Eurotherm 840/T digital thermometer, connected to a thermocouple which was introduced into an NMR tube filled with mineral oil to approximately the same level as a typical sample. At each temperature, the  $\text{D}_2\text{O}$  samples were left to equilibrate for at least 10 min before the data were collected.

In order to avoid having to obtain hundreds of spectra, we prepared seven stock solutions containing approximately equal amounts of several of our entries, chosen in such a way as to prevent intermolecular interactions and possible ambiguities in assignment. Solution 1: acetone, *tert*-butyl methyl ether, dimethylformamide, ethanol, toluene. Solution 2: benzene, dimethyl sulfoxide, ethyl acetate, methanol. Solution 3: acetic acid, chloroform, diethyl ether, 2-propanol, tetrahydrofuran. Solution 4: acetonitrile, dichloromethane, dioxane, *n*-hexane, HMPA. Solution 5: 1,2-dichloroethane, ethyl methyl ketone, *n*-pentane, pyridine. Solution 6: *tert*-butyl alcohol, BHT, cyclohexane, 1,2-dimethoxyethane, nitromethane, silicone grease, triethylamine. Solution 7: diglyme, dimethylacetamide, ethylene glycol, "grease" (engine oil). For  $\text{D}_2\text{O}$ . Solution 1: acetone, *tert*-butyl methyl ether, dimethylformamide, ethanol, 2-propanol. Solution 2: dimethyl sulfoxide, ethyl acetate, ethylene glycol, methanol. Solution 3: acetonitrile, diglyme, dioxane, HMPA, pyridine. Solution 4: 1,2-dimethoxyethane, dimethylacetamide, ethyl methyl ketone, triethylamine. Solution 5: acetic acid, *tert*-butyl alcohol, diethyl ether, tetrahydrofuran. In  $\text{D}_2\text{O}$  and  $\text{CD}_3\text{OD}$  nitromethane was run separately, as the protons exchanged with deuterium in presence of triethylamine.

### Results

**Proton Spectra** (Table 1). A sample of 0.6 mL of the solvent, containing 1  $\mu\text{L}$  of  $\text{TMS}^1$  was first run on its own. From this spectrum we determined the chemical shifts of the solvent residual peak<sup>2</sup> and the water peak. It should be noted that the latter is quite temperature-



**Figure 1.** Chemical shift of *HDO* as a function of temperature.

dependent (*vide infra*). Also, any potential hydrogen-bond acceptor will tend to shift the water signal downfield; this is particularly true for nonpolar solvents. In contrast, in *e.g.* DMSO the water is already strongly hydrogen-bonded to the solvent, and solutes have only a negligible effect on its chemical shift. This is also true for  $\text{D}_2\text{O}$ ; the chemical shift of the residual HDO is very temperature-dependent (*vide infra*) but, maybe counter-intuitively, remarkably solute (and pH) independent.

We then added 3  $\mu\text{L}$  of one of our stock solutions to the NMR tube. The chemical shifts were read and are presented in Table 1. Except where indicated, the coupling constants, and therefore the peak shapes, are essentially solvent-independent and are presented only once.

For  $\text{D}_2\text{O}$  as a solvent, the accepted reference peak ( $\delta = 0$ ) is the methyl signal of the sodium salt of 3-(trimethylsilyl)propanesulfonic acid; one crystal of this was added to each NMR tube. This material has several disadvantages, however: it is not volatile, so it cannot be readily eliminated if the sample has to be recovered. In addition, unless one purchases it in the relatively expensive deuterated form, it adds three more signals to the spectrum (methylenes 1, 2, and 3 appear at 2.91, 1.76, and 0.63 ppm, respectively). We suggest that the residual HDO peak be used as a secondary reference; we find that if the effects of temperature are taken into account (*vide infra*), this is very reproducible. For  $\text{D}_2\text{O}$ , we used a different set of stock solutions, since many of the less polar substrates are not significantly water-soluble (see Table 1). We also ran sodium acetate and sodium formate (chemical shifts: 1.90 and 8.44 ppm, respectively).

**Carbon Spectra** (Table 2). To each tube, 50  $\mu\text{L}$  of the stock solution and 3  $\mu\text{L}$  of  $\text{TMS}^1$  were added. The solvent chemical shifts<sup>3</sup> were obtained from the spectra containing the solutes, and the ranges of chemical shifts

(2) *I.e.*, the signal of the proton for the isotopomer with one less deuterium than the perdeuterated material, *e.g.*,  $\text{CHCl}_3$  in  $\text{CDCl}_3$  or  $\text{C}_6\text{D}_5\text{H}$  in  $\text{C}_6\text{D}_6$ . Except for  $\text{CHCl}_3$ , the splitting due to  $J_{\text{HD}}$  is typically observed (to a good approximation, it is 1/6.5 of the value of the corresponding  $J_{\text{HH}}$ ). For  $\text{CHD}_2$  groups (deuterated acetone, DMSO, acetonitrile), this signal is a 1:2:3:2:1 quintet with a splitting of *ca.* 2 Hz.

(3) In contrast to what was said in note 2, in the  $^{13}\text{C}$  spectra the solvent signal is due to the perdeuterated isotopomer, and the one-bond couplings to deuterium are always observable (*ca.* 20–30 Hz).

(1) For recommendations on the publication of NMR data, see: IUPAC Commission on Molecular Structure and Spectroscopy. *Pure Appl. Chem.* 1972, 29, 627; 1976, 45, 217.

Table 1. <sup>1</sup>H NMR Data

	proton	mult	CDCl <sub>3</sub>	(CD <sub>3</sub> ) <sub>2</sub> CO	(CD <sub>3</sub> ) <sub>2</sub> SO	C <sub>6</sub> D <sub>6</sub>	CD <sub>3</sub> CN	CD <sub>3</sub> OD	D <sub>2</sub> O
solvent residual peak			7.26	2.05	2.50	7.16	1.94	3.31	4.79
H <sub>2</sub> O			1.56	2.84 <sup>a</sup>	3.33 <sup>a</sup>	0.40	2.13	4.87	
acetic acid			2.10	1.96	1.91	1.55	1.96	1.99	2.08
acetone			2.17	2.09	2.09	1.55	2.08	2.15	2.22
acetonitrile			2.10	2.05	2.07	1.55	1.96	2.03	2.06
benzene			7.36	7.36	7.37	7.15	7.37	7.33	
<i>tert</i> -butyl alcohol			1.28	1.18	1.11	1.05	1.16	1.40	1.24
			OH <sup>c</sup>		4.19	1.55	2.18		
<i>tert</i> -butyl methyl ether			1.19	1.13	1.11	1.07	1.14	1.15	1.21
			OCH <sub>3</sub>	3.22	3.13	3.08	3.13	3.20	3.22
BHT <sup>b</sup>			6.98	6.96	6.87	7.05	6.97	6.92	
			OH <sup>c</sup>	5.01	6.65	4.79	5.20		
			ArCH <sub>3</sub>	2.27	2.22	2.18	2.24	2.21	
			ArC(CH <sub>3</sub> ) <sub>3</sub>	1.43	1.41	1.36	1.38	1.39	1.40
chloroform			7.26	8.02	8.32	6.15	7.58	7.90	
cyclohexane			1.43	1.43	1.40	1.40	1.44	1.45	
1,2-dichloroethane			3.73	3.87	3.90	2.90	3.81	3.78	
dichloromethane			5.30	5.63	5.76	4.27	5.44	5.49	
diethyl ether			1.21	1.11	1.09	1.11	1.12	1.18	1.17
			CH <sub>2</sub>	3.48	3.41	3.38	3.26	3.42	3.49
			q, 7						
diglyme			3.65	3.56	3.51	3.46	3.53	3.61	3.67
			CH <sub>2</sub>	3.57	3.47	3.38	3.34	3.45	3.58
			m						
			OCH <sub>3</sub>	3.39	3.28	3.24	3.11	3.29	3.35
1,2-dimethoxyethane			3.40	3.28	3.24	3.12	3.28	3.35	3.37
			CH <sub>3</sub>	3.55	3.46	3.43	3.33	3.45	3.52
			s						
dimethylacetamide			2.09	1.97	1.96	1.60	1.97	2.07	2.08
			CH <sub>3</sub> CO	3.02	3.00	2.94	2.57	2.96	3.31
			NCH <sub>3</sub>	2.94	2.83	2.78	2.05	2.83	2.92
			s						
dimethylformamide			8.02	7.96	7.95	7.63	7.92	7.97	7.92
			CH	2.96	2.94	2.89	2.36	2.89	2.99
			CH <sub>3</sub>	2.88	2.78	2.73	1.86	2.77	2.86
			s						
dimethyl sulfoxide			2.62	2.52	2.54	1.68	2.50	2.65	2.71
			CH <sub>3</sub>	3.71	3.59	3.57	3.35	3.60	3.66
dioxane			3.71	3.59	3.57	3.35	3.60	3.66	3.75
ethanol			1.25	1.12	1.06	0.96	1.12	1.19	1.17
			CH <sub>3</sub>	3.72	3.57	3.44	3.34	3.54	3.60
			q, 7 <sup>d</sup>						
			OH	1.32	3.39	4.63	2.47		
			s <sup>c,d</sup>						
ethyl acetate			2.05	1.97	1.99	1.65	1.97	2.01	2.07
			CH <sub>3</sub> CO	4.12	4.05	4.03	3.89	4.06	4.09
			q, 7						
			CH <sub>2</sub> CH <sub>3</sub>	1.26	1.20	1.17	0.92	1.20	1.24
			t, 7						
ethyl methyl ketone			2.14	2.07	2.07	1.58	2.06	2.12	2.19
			CH <sub>3</sub> CO	2.46	2.45	2.43	1.81	2.43	2.50
			q, 7						
			CH <sub>2</sub> CH <sub>3</sub>	1.06	0.96	0.91	0.85	0.96	1.01
			t, 7						
ethylene glycol			3.76	3.28	3.34	3.41	3.51	3.59	3.65
			CH	0.86	0.87	0.92	0.86	0.88	
			m						
			CH <sub>3</sub>	1.26	1.29	1.36	1.27	1.29	
			br s						
<i>n</i> -hexane			0.88	0.88	0.86	0.89	0.89	0.90	
			CH <sub>3</sub>	1.26	1.28	1.25	1.24	1.28	
			m						
HMPA <sup>g</sup>			2.65	2.59	2.53	2.40	2.57	2.64	2.61
			CH <sub>3</sub>	3.49	3.31	3.16	3.07	3.28	3.34
			s <sup>h</sup>						
methanol			1.09	3.12	4.01	2.16			
			OH	4.33	4.43	4.42	2.94	4.31	4.34
			s						
nitromethane			4.33	4.43	4.42	2.94	4.31	4.34	4.40
<i>n</i> -pentane			0.88	0.88	0.86	0.87	0.89	0.90	
			CH <sub>3</sub>	1.27	1.27	1.27	1.23	1.29	
			m						
2-propanol			1.22	1.10	1.04	0.95	1.09	1.50	1.17
			CH <sub>3</sub>	4.04	3.90	3.78	3.67	3.87	3.92
			sep, 6						
pyridine			8.62	8.58	8.58	8.53	8.57	8.53	8.52
			CH(2)	7.29	7.35	7.39	6.66	7.33	7.44
			m						
			CH(3)	7.68	7.76	7.79	6.98	7.73	7.85
			m						
silicone grease <sup>i</sup>			0.07	0.13		0.29	0.08	0.10	
			CH <sub>3</sub>	1.85	1.79	1.76	1.40	1.80	1.87
			m						
tetrahydrofuran			3.76	3.63	3.60	3.57	3.64	3.71	3.74
			CH <sub>2</sub> O	2.36	2.32	2.30	2.11	2.33	2.32
			s						
toluene			7.17	7.1–7.2	7.18	7.02	7.1–7.3	7.16	
			CH( <i>o/p</i> )						
			m						
			CH( <i>m</i> )	7.25	7.1–7.2	7.25	7.13	7.1–7.3	7.16
			m						
triethylamine			1.03	0.96	0.93	0.96	0.96	1.05	0.99
			CH <sub>3</sub>	2.53	2.45	2.43	2.40	2.45	2.58
			q, 7						

<sup>a</sup> In these solvents the intermolecular rate of exchange is slow enough that a peak due to HDO is usually also observed; it appears at 2.81 and 3.30 ppm in acetone and DMSO, respectively. In the former solvent, it is often seen as a 1:1:1 triplet, with <sup>2</sup>J<sub>H,D</sub> = 1 Hz. <sup>b</sup> 2,6-Dimethyl-4-*tert*-butylphenol. <sup>c</sup> The signals from exchangeable protons were not always identified. <sup>d</sup> In some cases (see note *a*), the coupling interaction between the CH<sub>2</sub> and the OH protons may be observed (*J* = 5 Hz). <sup>e</sup> In CD<sub>3</sub>CN, the OH proton was seen as a multiplet at δ 2.69, and extra coupling was also apparent on the methylene peak. <sup>f</sup> Long-chain, linear aliphatic hydrocarbons. Their solubility in DMSO was too low to give visible peaks. <sup>g</sup> Hexamethylphosphoramide. <sup>h</sup> In some cases (see notes *a*, *d*), the coupling interaction between the CH<sub>3</sub> and the OH protons may be observed (*J* = 5.5 Hz). <sup>i</sup> Poly(dimethylsiloxane). Its solubility in DMSO was too low to give visible peaks.

show their degree of variability. Occasionally, in order to distinguish between peaks whose assignment was

ambiguous, a further 1–2 μL of a specific substrate were added and the spectra run again.

**Table 2.**  $^{13}\text{C}$  NMR Data<sup>a</sup>

		$\text{CDCl}_3$	$(\text{CD}_3)_2\text{CO}$	$(\text{CD}_3)_2\text{SO}$	$\text{C}_6\text{D}_6$	$\text{CD}_3\text{CN}$	$\text{CD}_3\text{OD}$	$\text{D}_2\text{O}$
solvent signals		$77.16 \pm 0.06$	$29.84 \pm 0.01$ $206.26 \pm 0.13$	$39.52 \pm 0.06$	$128.06 \pm 0.02$	$1.32 \pm 0.02$ $118.26 \pm 0.02$	$49.00 \pm 0.01$	
acetic acid	CO	175.99	172.31	171.93	175.82	173.21	175.11	177.21
	CH <sub>3</sub>	20.81	20.51	20.95	20.37	20.73	20.56	21.03
acetone	CO	207.07	205.87	206.31	204.43	207.43	209.67	215.94
	CH <sub>3</sub>	30.92	30.60	30.56	30.14	30.91	30.67	30.89
acetonitrile	CN	116.43	117.60	117.91	116.02	118.26	118.06	119.68
	CH <sub>3</sub>	1.89	1.12	1.03	0.20	1.79	0.85	1.47
benzene	CH	128.37	129.15	128.30	128.62	129.32	129.34	
<i>tert</i> -butyl alcohol	C	69.15	68.13	66.88	68.19	68.74	69.40	70.36
	CH <sub>3</sub>	31.25	30.72	30.38	30.47	30.68	30.91	30.29
<i>tert</i> -butyl methyl ether	OCH <sub>3</sub>	49.45	49.35	48.70	49.19	49.52	49.66	49.37
	C	72.87	72.81	72.04	72.40	73.17	74.32	75.62
BHT	CCH <sub>3</sub>	26.99	27.24	26.79	27.09	27.28	27.22	26.60
	C(1)	151.55	152.51	151.47	152.05	152.42	152.85	
	C(2)	135.87	138.19	139.12	136.08	138.13	139.09	
	CH(3)	125.55	129.05	127.97	128.52	129.61	129.49	
	C(4)	128.27	126.03	124.85	125.83	126.38	126.11	
	CH <sub>3</sub> Ar	21.20	21.31	20.97	21.40	21.23	21.38	
	CH <sub>3</sub> C	30.33	31.61	31.25	31.34	31.50	31.15	
	C	34.25	35.00	34.33	34.35	35.05	35.36	
chloroform	CH	77.36	79.19	79.16	77.79	79.17	79.44	
cyclohexane	CH <sub>2</sub>	26.94	27.51	26.33	27.23	27.63	27.96	
1,2-dichloroethane	CH <sub>2</sub>	43.50	45.25	45.02	43.59	45.54	45.11	
dichloromethane	CH <sub>2</sub>	53.52	54.95	54.84	53.46	55.32	54.78	
diethyl ether	CH <sub>3</sub>	15.20	15.78	15.12	15.46	15.63	15.46	14.77
	CH <sub>2</sub>	65.91	66.12	62.05	65.94	66.32	66.88	66.42
diglyme	CH <sub>3</sub>	59.01	58.77	57.98	58.66	58.90	59.06	58.67
	CH <sub>2</sub>	70.51	71.03	69.54	70.87	70.99	71.33	70.05
1,2-dimethoxyethane	CH <sub>2</sub>	71.90	72.63	71.25	72.35	72.63	72.92	71.63
	CH <sub>3</sub>	59.08	58.45	58.01	58.68	58.89	59.06	58.67
dimethylacetamide	CH <sub>2</sub>	71.84	72.47	71.07	72.21	72.47	72.72	71.49
	CH <sub>3</sub>	21.53	21.51	21.29	21.16	21.76	21.32	21.09
	CO	171.07	170.61	169.54	169.95	171.31	173.32	174.57
	NCH <sub>3</sub>	35.28	34.89	37.38	34.67	35.17	35.50	35.03
dimethylformamide	NCH <sub>3</sub>	38.13	37.92	34.42	37.03	38.26	38.43	38.76
	CH	162.62	162.79	162.29	162.13	163.31	164.73	165.53
	CH <sub>3</sub>	36.50	36.15	35.73	35.25	36.57	36.89	37.54
	CH <sub>3</sub>	31.45	31.03	30.73	30.72	31.32	31.61	32.03
dimethyl sulfoxide	CH <sub>3</sub>	40.76	41.23	40.45	40.03	41.31	40.45	39.39
dioxane	CH <sub>2</sub>	67.14	67.60	66.36	67.16	67.72	68.11	67.19
ethanol	CH <sub>3</sub>	18.41	18.89	18.51	18.72	18.80	18.40	17.47
	CH <sub>2</sub>	58.28	57.72	56.07	57.86	57.96	58.26	58.05
ethyl acetate	CH <sub>3</sub> CO	21.04	20.83	20.68	20.56	21.16	20.88	21.15
	CO	171.36	170.96	170.31	170.44	171.68	172.89	175.26
	CH <sub>2</sub>	60.49	60.56	59.74	60.21	60.98	61.50	62.32
	CH <sub>3</sub>	14.19	14.50	14.40	14.19	14.54	14.49	13.92
ethyl methyl ketone	CH <sub>3</sub> CO	29.49	29.30	29.26	28.56	29.60	29.39	29.49
	CO	209.56	208.30	208.72	206.55	209.88	212.16	218.43
	CH <sub>2</sub> CH <sub>3</sub>	36.89	36.75	35.83	36.36	37.09	37.34	37.27
	CH <sub>2</sub> CH <sub>3</sub>	7.86	8.03	7.61	7.91	8.14	8.09	7.87
ethylene glycol	CH <sub>2</sub>	63.79	64.26	62.76	64.34	64.22	64.30	63.17
"grease"	CH <sub>2</sub>	29.76	30.73	29.20	30.21	30.86	31.29	
<i>n</i> -hexane	CH <sub>3</sub>	14.14	14.34	13.88	14.32	14.43	14.45	
	CH <sub>2</sub> (2)	22.70	23.28	22.05	23.04	23.40	23.68	
	CH <sub>2</sub> (3)	31.64	32.30	30.95	31.96	32.36	32.73	
HMPA <sup>b</sup>	CH <sub>3</sub>	36.87	37.04	36.42	36.88	37.10	37.00	36.46
methanol	CH <sub>3</sub>	50.41	49.77	48.59	49.97	49.90	49.86	49.50 <sup>c</sup>
nitromethane	CH <sub>3</sub>	62.50	63.21	63.28	61.16	63.66	63.08	63.22
<i>n</i> -pentane	CH <sub>3</sub>	14.08	14.29	13.28	14.25	14.37	14.39	
	CH <sub>2</sub> (2)	22.38	22.98	21.70	22.72	23.08	23.38	
	CH <sub>2</sub> (3)	34.16	34.83	33.48	34.45	34.89	35.30	
2-propanol	CH <sub>3</sub>	25.14	25.67	25.43	25.18	25.55	25.27	24.38
	CH	64.50	63.85	64.92	64.23	64.30	64.71	64.88
pyridine	CH(2)	149.90	150.67	149.58	150.27	150.76	150.07	149.18
	CH(3)	123.75	124.57	123.84	123.58	127.76	125.53	125.12
	CH(4)	135.96	136.56	136.05	135.28	136.89	138.35	138.27
silicone grease	CH <sub>3</sub>	1.04	1.40		1.38		2.10	
	CH <sub>2</sub>	25.62	26.15	25.14	25.72	26.27	26.48	25.67
tetrahydrofuran	CH <sub>2</sub> O	67.97	68.07	67.03	67.80	68.33	68.83	68.68
toluene	CH <sub>3</sub>	21.46	21.46	20.99	21.10	21.50	21.50	
	C( <i>h</i> )	137.89	138.48	137.35	137.91	138.90	138.85	
	CH( <i>o</i> )	129.07	129.76	128.88	129.33	129.94	129.91	
	CH( <i>m</i> )	128.26	129.03	128.18	128.56	129.23	129.20	
	CH( <i>p</i> )	125.33	126.12	125.29	125.68	126.28	126.29	
triethylamine	CH <sub>3</sub>	11.61	12.49	11.74	12.35	12.38	11.09	9.07
	CH <sub>2</sub>	46.25	47.07	45.74	46.77	47.10	46.96	47.19

<sup>a</sup> See footnotes for Table 1. <sup>b</sup>  $^2J_{\text{PC}} = 3$  Hz. <sup>c</sup> Reference material; see text.

For D<sub>2</sub>O solutions there is no accepted reference for carbon chemical shifts. We suggest the addition of a drop of methanol, and the position of its signal to be defined as 49.50 ppm; on this basis, the entries in Table 2 were recorded. The chemical shifts thus obtained are, on the whole, very similar to those for the other solvents. Alternatively, we suggest the use of dioxane when the methanol peak is expected to fall in a crowded area of the spectrum. We also report the chemical shifts of sodium formate (171.67 ppm), sodium acetate (182.02 and 23.97 ppm), sodium carbonate (168.88 ppm), sodium bicarbonate (161.08 ppm), and sodium 3-(trimethylsilyl)propanesulfonate [54.90, 19.66, 15.56 (methylenes 1, 2, and 3, respectively), and -2.04 ppm (methyls)], in D<sub>2</sub>O.

**Temperature Dependence of HDO Chemical Shifts.** We recorded the <sup>1</sup>H spectrum of a sample of D<sub>2</sub>O, containing a crystal of sodium 3-(trimethylsilyl)propanesulfonate as reference, as a function of temperature. The

data are shown in Figure 1. The solid line connecting the experimental points corresponds to the equation

$$\delta = 5.060 - 0.0122T + (2.11 \times 10^{-5})T^2 \quad (1)$$

which reproduces the measured values to better than 1 ppb. For the 0 - 50°C range, the simpler

$$\delta = 5.051 - 0.0111T \quad (2)$$

gives values correct to 10 ppb. For both equations, *T* is the temperature in °C.

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## Supporting Information

# NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist

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<b>Corrections and Comments</b> .....	<b>S2</b>
<b><sup>1</sup>H NMR Data (Table S1)</b> .....	<b>S3</b>
<b><sup>13</sup>C NMR Data (Table S2)</b> .....	<b>S5</b>
<b>Individual Solvent Tables – NMR Data Sorted by Chemical Shift (Tables S3–S26)</b> .....	<b>S7</b>
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## **Corrections and Comments**

In the preparation of this manuscript, several errors were discovered in the original paper<sup>1</sup> and are reported herein. While comparing the <sup>1</sup>H NMR spectral data obtained in toluene-*d*<sub>8</sub> to that in C<sub>6</sub>D<sub>6</sub>, it was discovered that the <sup>1</sup>H NMR chemical shifts for acetic acid (CH<sub>3</sub>), acetonitrile (CH<sub>3</sub>) and *tert*-butyl alcohol (OH) in C<sub>6</sub>D<sub>6</sub> had each been misreported at 1.55 ppm in the original paper; the values have now been correctly listed as 1.52, 0.58, and 0.63 ppm, respectively. The original paper's assignments for BHT's C(3,5) and C(4) in C<sub>6</sub>D<sub>6</sub>, (CD<sub>3</sub>)<sub>2</sub>CO, (CD<sub>3</sub>)<sub>2</sub>SO, CD<sub>3</sub>CN, and CD<sub>3</sub>OD were reversed and are now corrected. The resonances for 1,2-dimethoxyethane (CH<sub>2</sub>) in (CD<sub>3</sub>)<sub>2</sub>CO, silicone grease (CH<sub>3</sub>) in CDCl<sub>3</sub>, and 2-propanol (CH<sub>3</sub>) in CD<sub>3</sub>OD have been corrected and are reported as 72.47, 1.04, and 1.15 ppm, respectively. No other significant differences were discovered when comparing our data to that which had been previously reported; however, we have additionally provided the OH resonance for ethanol in C<sub>6</sub>D<sub>6</sub> (0.50 ppm), the CH<sub>3</sub> resonance for silicone grease in (CD<sub>3</sub>)<sub>2</sub>SO (−0.06 ppm), and replaced the “grease” entry (formerly motor oil<sup>1</sup>) with VWR vacuum pump oil #19, which is now reported in each deuterated solvent.

**Table S1.** <sup>1</sup>H NMR Data<sup>2</sup>

	proton	mult	THF- <i>d</i> <sub>8</sub>	CD <sub>2</sub> Cl <sub>2</sub>	CDCl <sub>3</sub>	toluene- <i>d</i> <sub>8</sub>	C <sub>6</sub> D <sub>6</sub>	C <sub>6</sub> D <sub>5</sub> Cl	(CD <sub>3</sub> ) <sub>2</sub> CO	(CD <sub>3</sub> ) <sub>2</sub> SO	CD <sub>3</sub> CN	TFE- <i>d</i> <sub>3</sub>	CD <sub>3</sub> OD	D <sub>2</sub> O
solvent residual signals			1.72	5.32	7.26	2.08	7.16	6.96	2.05	2.50	1.94	5.02	3.31	4.79
			3.58			6.97		6.99				3.88		
						7.01		7.14						
						7.09								
water	OH	s	2.46	1.52	1.56	0.43	0.40	1.03	2.84 <sup>3</sup>	3.33 <sup>3</sup>	2.13	3.66	4.87	-
acetic acid	CH <sub>3</sub>	s	1.89	2.06	2.10	1.57	1.52	1.76	1.96	1.91	1.96	2.06	1.99	2.08
acetone	CH <sub>3</sub>	s	2.05	2.12	2.17	1.57	1.55	1.77	2.09	2.09	2.08	2.19	2.15	2.22
acetonitrile	CH <sub>3</sub>	s	1.95	1.97	2.10	0.69	0.58	1.21	2.05	2.07	1.96	1.95	2.03	2.06
allyl acetate	CHCH <sub>2</sub>	ddt	5.90	5.92	5.93	5.67 <sup>4</sup>	5.68 <sup>4</sup>	5.77	5.92	5.91	5.93	5.93	5.94	5.99
	CHCH <sub>2</sub> (1)	ddt	5.27	5.31	5.32	5.05	5.06	5.15	5.29	5.29	5.29	5.32	5.30	5.37
	CHCH <sub>2</sub> (2)	ddt	5.15	5.22	5.24	4.94	4.94	5.04	5.18	5.20	5.21	5.25	5.21	5.30
	CH <sub>2</sub>	ddd	4.50	4.55	4.57	4.34	4.38	4.44	4.53	4.52	4.53	4.58	4.56	4.62
	CH <sub>3</sub>	s	1.98	2.05	2.09	1.63	1.63	1.80	2.02	2.03	2.02	2.07	2.05	2.13
benzaldehyde	HCO	s	9.98	10.01	10.03	9.57	9.64	9.77	10.05	10.02	10.01	9.88	10.00	9.96
	CH(2,6)	m	7.86–7.88	7.87–7.89	7.88–7.91	7.45–7.47	7.49–7.53	7.59–7.61	7.92–7.94	7.91–7.93	7.89–7.91	7.90–7.92	7.90–7.93	7.97–7.99
	CH(3,5)	m	7.51–7.55	7.53–7.57	7.51–7.57	6.95–6.99	6.93–6.99	7.15–7.19	7.59–7.63	7.61–7.67	7.57–7.61	7.56–7.59	7.56–7.60	7.57–7.66
	CH(4)	m	7.60–7.64	7.63–7.67	7.61–7.65	7.03–7.07	7.01–7.07	7.24–7.28	7.69–7.73	7.69–7.75	7.67–7.71	7.68–7.72	7.66–7.70	7.76–7.80
benzene	CH	s	7.31	7.35	7.36	7.12	7.15	7.20	7.36	7.37	7.37	7.36	7.33	-
<i>tert</i> -butyl alcohol	CH <sub>3</sub>	s	1.15	1.24	1.28	1.03	1.05	1.12	1.18	1.11	1.16	1.28	1.40	1.24
	OH	s <sup>5</sup>	3.16	-	-	0.58	0.63	1.30	-	4.19	2.18	2.20	-	-
BHA	ArH	s	6.68	6.73	6.76	6.83	6.93	6.83	6.72	6.62	6.73	6.87	6.71	-
	OH	s <sup>5</sup>	5.64	4.76	4.76	4.45	4.53	4.62	5.65	6.52	4.98	-	4.85	-
	ArOCH <sub>3</sub>	s	3.68	3.73	3.77	3.48	3.48	3.61	3.72	3.66	3.72	3.79	3.72	-
	ArC(CH <sub>3</sub> ) <sub>3</sub>	s	1.40	1.42	1.44	1.34	1.41	1.37	1.41	1.36	1.40	1.44	1.41	-
BHT	ArH	s	6.92	6.97	6.98	6.99	7.05	6.97	6.96	6.87	6.97	7.06	6.92	-
	OH	s <sup>5</sup>	5.81	5.00	5.01	4.72	4.79	5.50	-	6.65	5.20	-	-	-
	ArCH <sub>3</sub>	s	2.21	2.25	2.27	2.23	2.24	2.20	2.22	2.18	2.22	2.24	2.21	-
	ArC(CH <sub>3</sub> ) <sub>3</sub>	s	1.40	1.42	1.43	1.36	1.38	1.37	1.41	1.36	1.39	1.43	1.40	-
chloroform	CH	s	7.89	7.32	7.26	6.10	6.15	6.74	8.02	8.32	7.58	7.33	7.90	-
18-crown-6	CH <sub>2</sub>	s	3.57	3.59	3.67	3.36	3.39	3.41	3.59	3.51	3.51	3.64	3.64	3.80
cyclohexane	CH <sub>2</sub>	s	1.44	1.44	1.43	1.40	1.40	1.37	1.43	1.40	1.44	1.47	1.45	-
cyclohexanone	CH <sub>2</sub> (2,6)	t	2.24	2.29	2.33	1.95	1.98	2.08	2.27	2.25	2.27	2.38	2.34	2.40
	CH <sub>2</sub> (3,5)	m	1.77–1.82	1.81–1.87	1.84–1.86	1.33–1.39	1.28–1.37	1.48–1.53	1.79–1.83	1.74–1.78	1.79–1.84	1.87–1.92	1.85–1.87	1.85–1.90
	CH <sub>2</sub> (4)	m	1.68–1.71	1.69–1.72	1.71–1.73	1.16–1.20	1.08–1.16	1.33–1.37	1.70–1.74	1.64–1.66	1.67–1.72	1.75–1.78	1.74–1.76	1.70–1.75
diallyl carbonate	CHCH <sub>2</sub>	ddt	5.92	5.95	5.94	5.63	5.65	5.75	5.96	5.93	5.96	5.92	5.94	5.99
	CHCH <sub>2</sub> (1)	ddt	5.31	5.35	5.37	5.09	5.09	5.17	5.35	5.33	5.34	5.35	5.34	5.40
	CHCH <sub>2</sub> (2)	ddt	5.19	5.26	5.27	4.92	4.92	5.03	5.23	5.25	5.25	5.28	5.25	5.32
	CH <sub>2</sub>	ddd	4.58	4.61	4.64	4.34	4.38	4.46	4.62	4.61	4.61	4.62	4.61	4.69
1,2-dichloroethane	CH <sub>2</sub>	s	3.77	3.76	3.73	2.91	2.90	3.26	3.87	3.90	3.81	3.71	3.78	-
dichloromethane	CH <sub>2</sub>	s	5.51	5.33	5.30	4.32	4.27	4.77	5.63	5.76	5.44	5.24	5.49	-
diethyl ether	CH <sub>3</sub>	t, 7	1.12	1.15	1.21	1.10	1.11	1.10	1.11	1.09	1.12	1.20	1.18	1.17
	CH <sub>2</sub>	q, 7	3.38	3.43	3.48	3.25	3.26	3.31	3.41	3.38	3.42	3.58	3.49	3.56
diglyme	CH <sub>2</sub>	m	3.43	3.57	3.65	3.43	3.46	3.49	3.56	3.51	3.53	3.67	3.61	3.67
	CH <sub>2</sub>	m	3.53	3.50	3.57	3.31	3.34	3.37	3.47	3.38	3.45	3.62	3.58	3.61
	OCH <sub>3</sub>	s	3.28	3.33	3.39	3.12	3.11	3.16	3.28	3.24	3.29	3.41	3.35	3.37
1,2-dimethoxyethane	CH <sub>3</sub>	s	3.28	3.34	3.40	3.12	3.12	3.17	3.28	3.24	3.28	3.40	3.35	3.37
	CH <sub>2</sub>	s	3.43	3.49	3.55	3.31	3.33	3.37	3.46	3.43	3.45	3.61	3.52	3.60
dimethylacetamide	CH <sub>3</sub> CO	s	1.94	2.02	2.09	1.59	1.60	1.74	1.97	1.96	1.97	2.09	2.07	2.08
	NCH <sub>3</sub>	s	2.95	2.97	3.02	2.56	2.57	2.65	3.00	2.94	2.96	3.05	3.31	3.06
	NCH <sub>3</sub>	s	2.82	2.87	2.94	2.11	2.05	2.42	2.83	2.78	2.83	2.94	2.92	2.90
dimethyl carbonate	CH <sub>3</sub>	s	3.69	3.75	3.79	3.31	3.30	3.48	3.72	3.69	3.72	3.77	3.74	3.69
dimethyl malonate	CH <sub>3</sub>	s	3.65	3.72	3.75	3.24	3.23	3.41	3.68	3.65	3.68	3.76	3.72	3.78
	CH <sub>2</sub>	s	3.35	3.37	3.40	2.92	2.97	3.15	3.42	3.53	3.38	3.41	3.44	3.60
dimethylformamide	CH	s	7.91	7.96	8.02	7.57	7.63	7.73	7.96	7.95	7.92	7.86	7.97	7.92
	CH <sub>3</sub>	s	2.88	2.91	2.96	2.37	2.36	2.51	2.94	2.89	2.89	2.98	2.99	3.01
	CH <sub>3</sub>	s	2.76	2.82	2.88	1.96	1.86	2.30	2.78	2.73	2.77	2.88	2.86	2.85
dimethyl sulfoxide	CH <sub>3</sub>	s	2.45	2.55	2.62	1.64	1.68	2.03	2.52	2.54	2.50	2.63	2.65	2.71
1,4-dioxane	CH <sub>2</sub>	s	3.56	3.65	3.71	3.33	3.35	3.45	3.59	3.57	3.60	3.76	3.66	3.75
ethane	CH <sub>3</sub>	s	0.85	0.85	0.87	0.81	0.80	0.79	0.83	0.82	0.85	0.85	0.85	0.82
ethanol	CH <sub>3</sub>	t, 7	1.10	1.19	1.25	0.97	0.96	1.06	1.12	1.06	1.12	1.22	1.19	1.17
	CH <sub>2</sub>	q, 7 <sup>6</sup>	3.51	3.66	3.72	3.36	3.34	3.51	3.57	3.44	3.54	3.71	3.60	3.65
	OH	s <sup>5,6</sup>	3.30	1.33	1.32	0.83	0.50	1.39	3.39	4.63	2.47	-	-	-
ethyl acetate	CH <sub>3</sub> CO	s	1.94	2.00	2.05	1.69	1.65	1.78	1.97	1.99	1.97	2.03	2.01	2.07
	CH <sub>2</sub> CH <sub>3</sub>	q, 7	4.04	4.08	4.12	3.87	3.89	3.96	4.05	4.03	4.06	4.14	4.09	4.14
	CH <sub>3</sub> CH <sub>3</sub>	t, 7	1.19	1.23	1.26	0.94	0.92	1.04	1.20	1.17	1.20	1.26	1.24	1.24
ethyl methyl ketone	CH <sub>3</sub> CO	s	2.03	2.09	2.14	1.59	1.58	1.78	2.07	2.07	2.06	2.16	2.12	2.19
	CH <sub>2</sub> CH <sub>3</sub>	q, 7	2.39	2.43	2.46	1.82	1.81	2.06	2.45	2.43	2.43	2.49	2.50	3.18
	CH <sub>3</sub> CH <sub>3</sub>	t, 7	0.96	1.00	1.06	0.84	0.85	0.89	0.96	0.91	0.96	1.05	1.01	1.26
ethylene	CH <sub>2</sub>	s	5.36	5.40	5.40	5.25	5.25	5.29	5.38	5.41	5.41	5.40	5.39	5.44
ethylene glycol	CH <sub>2</sub>	s <sup>7</sup>	3.48	3.66	3.76	3.36	3.41	3.58	3.28	3.34	3.51	3.72	3.59	3.65
furan	CH(2,5)	dd	7.48	7.46	7.45	7.10	7.13	7.24	7.56	7.67	7.52	7.44	7.49	7.57
	CH(3,4)	dd	6.37	6.41	6.40	6.07	6.08	6.19	6.43	6.47	6.44	6.42	6.40	6.51
H grease <sup>8</sup>	CH <sub>3</sub>	m	0.85–0.91	0.84–0.90	0.84–0.87	0.89–0.96	0.90–0.98	0.86–0.92	0.90	0.82–0.88	-	0.88–0.94	0.86–0.93	-
	CH <sub>2</sub>	br s	1.29	1.27	1.25	1.33	1.32	1.30	1.29	1.24	-	1.33	1.29	-
hexamethylbenzene	CH <sub>3</sub>	s	2.18	2.20	2.24	2.10	2.13	2.10	2.17	2.14	2.19	2.24	2.19	-
hexamethyldisiloxane	CH <sub>3</sub>	s	0.07	0.07	0.07	0.10	0.12	0.10	0.07	0.06	0.07	0.08	0.07	0.28

<i>n</i> -hexane	CH <sub>3</sub>	t, 7	0.89	0.89	0.88	0.88	0.89	0.85	0.88	0.86	0.89	0.91	0.90	-
	CH <sub>2</sub>	m	1.29	1.27	1.26	1.22	1.24	1.19	1.28	1.25	1.28	1.31	1.29	-
HMPA	CH <sub>3</sub>	d, 9.5	2.58	2.60	2.65	2.42	2.40	2.47	2.59	2.53	2.57	2.63	2.64	2.61
hydrogen	H <sub>2</sub>	s	4.55	4.59	4.62	4.50	4.47	4.49	4.54	4.61	4.57	4.53	4.56	-
imidazole	CH(2)	s	7.48	7.63	7.67	7.30	7.33	7.53	7.62	7.63	7.57	7.61	7.67	7.78
	CH(4,5)	s	6.94	7.07	7.10	6.86	6.90	7.01	7.04	7.01	7.01	7.03	7.05	7.14
methane	CH <sub>4</sub>	s	0.19	0.21	0.22	0.17	0.16	0.15	0.17	0.20	0.20	0.18	0.20	0.18
methanol	CH <sub>3</sub>	s <sup>9</sup>	3.27	3.42	3.49	3.03	3.07	3.25	3.31	3.16	3.28	3.44	3.34	3.34
	OH	s <sup>5,9</sup>	3.02	1.09	1.09	-	-	1.30	3.12	4.01	2.16	-	-	-
nitromethane	CH <sub>3</sub>	s	4.31	4.31	4.33	3.01	2.94	3.59	4.43	4.42	4.31	4.28	4.34	4.40
<i>n</i> -pentane	CH <sub>3</sub>	t, 7	0.89	0.89	0.88	0.87	0.87	0.84	0.88	0.86	0.89	0.90	0.90	-
	CH <sub>2</sub>	m	1.31	1.30	1.27	1.25	1.23	1.23	1.27	1.27	1.29	1.33	1.29	-
propane	CH <sub>3</sub>	t, 7.3	0.90	0.90	0.90	0.89	0.86	0.84	0.88	0.87	0.90	0.90	0.91	0.88
	CH <sub>2</sub>	sept, 7.3	1.33	1.32	1.32	1.32	1.26	1.26	1.31	1.29	1.33	1.33	1.34	1.30
2-propanol	CH <sub>3</sub>	d, 6	1.08	1.17	1.22	0.95	0.95	1.04	1.10	1.04	1.09	1.20	1.15	1.17
	CH	sept, 6	3.82	3.97	4.04	3.65	3.67	3.82	3.90	3.78	3.87	4.05	3.92	4.02
propylene	CH <sub>3</sub>	dt, 6.4, 1.5	1.69	1.71	1.73	1.55	1.55	1.58	1.68	1.68	1.70	1.70	1.70	1.70
	CH <sub>2</sub> (1)	dm, 10	4.89	4.93	4.94	4.92	4.95	4.91	4.90	4.94	4.93	4.93	4.91	4.95
	CH <sub>2</sub> (2)	dm, 17	4.99	5.03	5.03	4.98	5.01	4.98	5.00	5.03	5.04	5.03	5.01	5.06
	CH	m	5.79	5.84	5.83	5.70	5.72	5.72	5.81	5.80	5.85	5.87	5.82	5.90
pump oil	CH <sub>3</sub>	m	0.86–0.90	0.84–0.89	0.83–0.89	0.88–0.96	0.91–0.97	0.88–0.91	0.87	0.74	0.85	0.99	0.86–0.91	-
	CH <sub>2</sub>	br s	1.29	1.27	1.26	1.30	1.37	1.31	1.29	1.15	1.27	1.41	1.29	-
pyridine	CH(2,6)	m	8.54	8.59	8.62	8.47	8.53	8.51	8.58	8.58	8.57	8.45	8.53	8.52
	CH(3,5)	m	7.25	7.28	7.29	6.67	6.66	6.90	7.35	7.39	7.33	7.40	7.44	7.45
	CH(4)	m	7.65	7.68	7.68	6.99	6.98	7.25	7.76	7.79	7.73	7.82	7.85	7.87
pyrrole	NH	br t	9.96	8.69	8.40	7.71	7.80	8.61	10.02	10.75	9.27	-	-	-
	CH(2,5)	m	6.66	6.79	6.83	6.43	6.48	6.62	6.77	6.73	6.75	6.84	6.72	6.93
	CH(3,4)	m	6.02	6.19	6.26	6.27	6.37	6.27	6.07	6.01	6.10	6.24	6.08	6.26
pyrrolidine <sup>10</sup>	CH <sub>2</sub> (2,5)	m	2.75	2.82	2.87	2.54	2.54	2.64	-	2.67	2.75	3.11	2.80	3.07
	CH <sub>2</sub> (3,4)	m	1.59	1.67	1.68	1.36	1.33	1.43	-	1.55	1.61	1.93	1.72	1.87
silicone grease	CH <sub>3</sub>	s	0.11	0.09	0.07	0.26	0.29	0.14	0.13	-0.06	0.08	0.16	0.10	-
tetrahydrofuran	CH <sub>2</sub> (2,5)	m	3.62	3.69	3.76	3.54	3.57	3.59	3.63	3.60	3.64	3.78	3.71	3.74
	CH <sub>2</sub> (3,4)	m	1.79	1.82	1.85	1.43	1.40	1.55	1.79	1.76	1.80	1.91	1.87	1.88
toluene	CH <sub>3</sub>	s	2.31	2.34	2.36	2.11	2.11	2.16	2.32	2.30	2.33	2.33	2.32	-
	CH(2,4,6)	m	7.10	7.15	7.17	6.96–7.01	7.02	7.01–7.08	7.10–7.20	7.18	7.10–7.30	7.10–7.30	7.16	-
	CH(3,5)	m	7.19	7.24	7.25	7.09	7.13	7.10–7.17	7.10–7.20	7.25	7.10–7.30	7.10–7.30	7.16	-
triethylamine	CH <sub>3</sub>	t, 7	0.97	0.99	1.03	0.95	0.96	0.93	0.96	0.93	0.96	1.31	1.05	0.99
	CH <sub>2</sub>	q, 7	2.46	2.48	2.53	2.39	2.40	2.39	2.45	2.43	2.45	3.12	2.58	2.57

**Table S2.**  $^{13}\text{C}\{^1\text{H}\}$  NMR Data<sup>2</sup>

	carbon	THF- <i>d</i> <sub>8</sub>	CD <sub>2</sub> Cl <sub>2</sub>	CDCl <sub>3</sub>	toluene- <i>d</i> <sub>8</sub>	C <sub>6</sub> D <sub>6</sub>	C <sub>6</sub> D <sub>5</sub> Cl	(CD <sub>2</sub> ) <sub>2</sub> CO	(CD <sub>3</sub> ) <sub>2</sub> SO	CD <sub>3</sub> CN	TFE- <i>d</i> <sub>3</sub>	CD <sub>3</sub> OD	D <sub>2</sub> O
solvent signals		67.21	53.84	77.16	137.48	128.06	134.19	29.84	39.52	1.32	61.50	49.00	-
		25.31			128.87		129.26	206.26		118.26	126.28		
					127.96		128.25						
					125.13		125.96						
acetic acid	CO	171.69	175.85	175.99	175.30	175.82	175.67	172.31	171.93	173.21	177.96	175.11	177.21
	CH <sub>3</sub>	20.13	20.91	20.81	20.27	20.37	20.40	20.51	20.95	20.73	20.91	20.56	21.03
acetone	CO	204.19	206.78	207.07	204.00	204.43	204.83	205.87	206.31	207.43	32.35	209.67	215.94
	CH <sub>3</sub>	30.17	31.00	30.92	30.03	30.14	30.12	30.60	30.56	30.91	214.98	30.67	30.89
acetonitrile	CN	116.79	116.92	116.43	115.76	116.02	115.93	117.60	117.91	118.26	118.95	118.06	119.68
	CH <sub>3</sub>	0.45	2.03	1.89	0.03	0.20	0.63	1.12	1.03	1.79	1.00	0.85	1.47
allyl acetate	CO	170.14	170.83	170.81	169.44	169.67	169.59	170.61	169.97	171.32	175.98	172.41	174.78
	C HCH <sub>2</sub>	133.90	132.94	132.33	132.98	132.90	132.69	133.76	132.71	133.83	133.33	133.71	132.48
	CHCH <sub>2</sub>	117.58	118.00	118.34	117.49	117.64	117.63	117.81	117.64	118.06	119.39	118.22	119.03
	CH <sub>2</sub>	65.31	65.36	65.28	64.87	64.92	64.86	65.28	64.32	65.55	67.61	66.14	66.52
	CH <sub>3</sub>	20.45	21.06	21.02	20.21	20.37	20.40	20.68	20.54	21.02	21.10	20.71	21.00
benzaldehyde	HCO	191.95	192.61	192.67	191.09	191.43	191.24	192.95	193.08	193.64	197.63	194.11	191.67
	C(1)	137.78	136.98	136.58	137.12	137.05	136.78	137.66	136.20	137.62	137.84	137.96	136.11
	CH(2,6)	129.98	129.98	129.91	129.61	129.65	129.49	130.23	129.45	130.42	131.78	130.64	130.09
	CH(3,5)	129.56	129.42	129.16	128.68	128.95	128.87	129.90	129.10	130.07	130.82	130.12	129.48
	CH(4)	134.67	134.79	134.64	133.88	133.95	134.02	135.14	134.52	135.40	137.17	135.60	134.70
benzene	CH	128.84	128.68	128.37	128.57	128.62	128.38	129.15	128.30	129.32	129.84	129.34	-
<i>tert</i> -butyl alcohol	(CH <sub>3</sub> ) <sub>3</sub> C	67.50	69.11	69.15	68.12	68.19	68.19	68.13	66.88	68.74	72.35	69.40	70.36
	(CH <sub>3</sub> ) <sub>2</sub> C	30.57	31.46	31.25	30.49	30.47	31.13	30.72	30.38	30.68	31.07	30.91	30.29
BHA	C(1)	154.07	153.05	152.57	153.50	153.62	153.19	153.97	152.53	154.02	153.74	154.34	-
	C(2,6)	148.62	148.06	147.85	148.06	148.13	147.87	148.48	147.44	148.39	150.52	149.04	-
	CH(3,5)	110.94	110.93	110.69	110.99	111.15	110.84	111.00	109.80	111.35	112.90	111.30	-
	C(4)	140.07	137.77	137.36	137.34	137.50	137.29	140.32	141.16	140.20	140.23	141.36	-
	CH <sub>2</sub> O	55.39	55.88	55.70	55.04	55.27	55.08	55.51	54.89	55.94	57.55	55.96	-
	(C H <sub>2</sub> ) <sub>3</sub> C	30.65	30.37	30.32	30.30	30.35	30.21	30.64	30.30	30.55	30.80	30.82	-
	(CH <sub>3</sub> ) <sub>3</sub> C	35.51	34.91	34.72	34.69	34.72	34.56	35.45	34.76	35.48	36.07	35.83	-
BHT	C(1)	152.48	151.92	151.55	152.06	152.05	151.69	152.51	151.47	152.42	153.46	152.85	-
	C(2,6)	137.93	136.32	135.87	136.12	136.08	135.92	138.19	139.12	138.13	138.59	139.09	-
	CH(3,5)	125.71	125.84	125.55	125.79	125.83	125.58	126.03	124.85	126.38	127.11	126.11	-
	C(4)	128.64	128.73	128.27	128.44	128.52	128.26	129.05	127.97	129.61	130.62	129.49	-
	CH <sub>2</sub> Ar	21.21	21.27	21.20	21.42	21.40	21.10	21.31	20.97	21.23	21.34	21.38	-
	(C H <sub>2</sub> ) <sub>3</sub> C	31.55	30.54	30.33	31.39	31.34	30.19	31.61	31.25	31.50	31.01	31.15	-
	(CH <sub>3</sub> ) <sub>3</sub> C	34.91	34.56	34.25	34.39	34.35	34.11	35.00	34.33	35.05	35.69	35.36	-
carbon dioxide	CO <sub>2</sub>	125.69	125.26	124.99	124.86	124.76	126.08	125.81	124.21	125.89	126.92	126.31	-
carbon disulfide	CS <sub>2</sub>	193.37	192.95	192.83	192.71	192.69	192.49	193.58	192.63	193.60	196.26	193.82	197.25
carbon tetrachloride	CCl <sub>4</sub>	96.89	96.52	96.34	96.57	96.44	96.38	96.65	95.44	96.68	97.74	97.21	96.73
chloroform	CH	79.24	77.99	77.36	77.89	77.79	77.67	79.19	79.16	79.17	78.83	79.44	-
18-crown-6	CH <sub>2</sub>	71.34	70.47	70.55	70.86	70.59	70.55	71.25	69.85	71.22	70.80	71.47	70.14
cyclohexane	CH <sub>2</sub>	27.58	27.38	26.94	27.31	27.23	26.99	27.51	26.33	27.63	28.34	27.96	-
cyclohexanone	CO	208.79	211.82	212.57	208.60	209.10	209.30	210.36	210.63	211.99	221.30	214.69	221.22
	CH <sub>2</sub> (2,6)	42.17	42.31	41.97	41.78	41.83	41.79	42.24	41.32	42.44	43.16	42.61	42.02
	CH <sub>2</sub> (3,5)	27.69	27.47	27.00	27.05	27.00	27.02	27.68	26.46	27.80	28.56	28.16	27.50
	CH <sub>2</sub> (4)	25.76	25.42	24.97	25.15	25.03	25.07	25.59	24.32	25.62	26.00	25.86	24.77
diallyl carbonate	CO	155.36	155.15	154.88	155.15	155.24	154.87	155.48	154.16	155.66	157.39	156.28	157.78
	C HCH <sub>2</sub>	133.08	132.24	131.58	132.30	132.18	131.93	133.16	132.18	133.20	132.72	133.25	132.76
	CHCH <sub>2</sub>	117.70	118.75	118.96	118.04	118.22	118.22	118.53	118.32	118.86	120.15	118.74	118.75
	CH <sub>2</sub>	68.58	68.76	68.55	68.20	68.28	68.19	68.78	67.86	69.09	70.69	69.35	68.81
1,2-dichloroethane	CH <sub>2</sub>	44.64	44.35	43.50	43.40	43.59	43.60	45.25	45.02	45.54	45.28	45.11	-
dichloromethane	CH <sub>2</sub>	54.67	54.24	53.52	53.47	53.46	53.54	54.95	54.84	55.32	54.46	54.78	-
diethyl ether	CH <sub>3</sub>	15.49	15.44	15.20	15.47	15.46	15.35	15.78	15.12	15.63	15.33	15.46	14.77
	CH <sub>2</sub>	66.14	66.11	65.91	65.94	65.94	65.79	66.12	62.05	66.32	67.55	66.88	66.42
diglyme	CH <sub>3</sub>	58.72	58.95	59.01	58.62	58.66	58.42	58.77	57.98	58.90	59.40	59.06	58.67
	CH <sub>2</sub>	71.17	70.70	70.51	70.92	70.87	70.56	71.03	69.54	70.99	73.05	71.33	70.05
	CH <sub>2</sub>	72.72	72.25	71.90	72.39	72.35	72.07	72.63	71.25	72.63	71.33	72.92	71.63
1,2-dimethoxyethane	CH <sub>3</sub>	58.72	59.02	59.08	58.63	58.68	58.31	58.45	58.03	58.89	59.52	59.06	58.67
	CH <sub>2</sub>	72.58	72.24	71.84	72.25	72.21	71.81	72.47	71.17	72.47	72.87	72.72	71.49
dimethylacetamide	CH <sub>3</sub>	21.15	21.64	21.53	21.05	21.16	21.03	21.51	21.29	21.76	21.40	21.32	21.09
	CO	169.77	171.05	171.07	169.65	169.95	169.79	170.61	169.54	171.31	175.74	173.32	174.57
	NCH <sub>3</sub>	34.60	35.23	35.28	34.58	34.67	34.59	34.89	34.42	35.17	36.28	35.50	35.03
	NCH <sub>3</sub>	37.56	38.22	38.13	36.98	37.03	37.13	37.92	37.38	38.26	39.06	38.43	38.76
dimethyl carbonate	CO	156.91	156.73	156.45	156.61	156.71	156.36	157.04	155.76	157.26	159.04	157.91	163.96
	CH <sub>3</sub>	54.58	55.09	54.89	54.13	54.30	54.23	54.95	54.63	55.39	56.17	55.25	55.81
dimethyl malonate	CO <sub>2</sub>	167.14	167.32	167.18	166.49	166.66	166.51	167.58	166.91	168.07	170.88	168.70	170.12
	CH <sub>3</sub>	52.07	52.75	52.57	51.76	51.86	51.89	52.47	52.08	52.95	54.00	52.83	53.65
	CH <sub>2</sub>	41.15	41.48	41.11	40.88	41.04	40.93	41.43	40.72	41.77	42.13	41.60	42.13
dimethylformamide	CH	161.96	162.57	162.62	161.93	162.13	162.01	162.79	162.29	163.31	166.01	164.73	165.53
	CH <sub>3</sub>	35.65	36.56	36.50	35.22	35.25	35.45	36.15	35.73	36.57	37.76	36.89	37.54
	CH <sub>2</sub>	30.70	31.39	31.45	30.64	30.72	30.71	31.03	30.73	31.32	30.96	31.61	32.03
dimethyl sulfoxide	CH <sub>3</sub>	41.21	41.33	40.76	40.41	40.03	40.27	41.23	40.45	41.31	40.06	40.45	39.39
1,4-dioxane	CH <sub>2</sub>	67.65	67.47	67.14	67.17	67.16	66.95	67.60	66.36	67.72	68.52	68.11	67.19
ethane	CH <sub>3</sub>	6.79	6.91	6.89	6.94	6.96	6.91	6.88	6.61	6.99	7.01	6.98	-
ethanol	CH <sub>3</sub>	18.90	18.69	18.41	18.78	18.72	18.55	18.89	18.51	18.80	18.11	18.40	17.47
	CH <sub>2</sub>	57.60	58.57	58.28	57.81	57.86	57.63	57.72	56.07				

ethyl acetate	$C H_3CO$	20.45	21.15	21.04	20.46	20.56	20.50	20.83	20.68	21.16	21.18	20.88	21.15
	CO	170.32	171.24	171.36	170.02	170.44	170.20	170.96	170.31	171.68	175.55	172.89	175.26
	$CH_2$	60.30	60.63	60.49	60.08	60.21	60.06	60.56	59.74	60.98	62.70	61.50	62.32
	$CH_3$	14.37	14.37	14.19	14.23	14.19	14.07	14.50	14.40	14.54	14.36	14.49	13.92
ethyl methyl ketone	$C H_3CO$	28.92	29.55	29.49	28.74	28.56	28.82	29.30	29.26	29.60	29.64	29.39	29.49
	CO	207.05	209.57	209.56	206.31	206.55	206.87	208.30	208.72	209.88	218.31	212.16	218.43
	$C H_2CH_3$	36.59	37.01	36.89	36.32	36.36	36.39	36.75	35.83	37.09	38.23	37.34	37.27
	$CH_2C H_3$	7.87	7.94	7.86	7.89	7.91	7.79	8.03	7.61	8.14	8.29	8.09	7.87
ethylene	$CH_2$	123.09	123.20	123.13	122.92	122.96	122.95	123.47	123.52	123.69	124.08	123.46	-
ethylene glycol	$CH_2$	64.35	64.08	63.79	64.29	64.34	64.03	64.26	62.76	64.22	64.87	64.30	63.17
furan	$CH(2,5)$	143.26	142.98	142.71	142.65	142.73	142.49	143.49	142.82	143.74	144.22	143.68	143.57
	$CH(3,4)$	109.88	109.86	109.57	109.63	109.67	109.64	110.24	109.62	110.49	111.06	110.33	110.23
H grease <sup>8</sup>	$CH_2$	30.45	30.14	29.71	30.31	30.22	30.11	-	-	-	-	-	-
hexamethylbenzene	C	131.88	132.09	132.21	131.72	131.79	131.54	132.22	131.10	132.61	134.04	132.53	-
	$CH_3$	16.71	16.93	16.98	16.84	16.95	16.68	16.86	16.60	16.94	17.04	16.90	-
hexamethyldisiloxane	$CH_3$	1.83	1.96	1.97	1.99	2.05	1.92	2.01	1.96	2.07	2.09	1.99	2.31
<i>n</i> -hexane	$CH_3$	14.22	14.28	14.14	14.34	14.32	14.18	14.34	13.88	14.43	14.63	14.45	-
	$CH_2(2,5)$	23.33	23.07	22.70	23.12	23.04	22.86	23.28	22.05	23.40	24.06	23.68	-
	$CH_2(3,4)$	32.34	32.01	31.64	32.06	31.96	31.77	32.30	30.95	32.36	33.17	32.73	-
HMPA <sup>11</sup>	$CH_3$	36.89	36.99	36.87	36.80	36.88	36.64	37.04	36.42	37.10	37.21	37.00	36.46
imidazole	$CH(2)$	135.72	135.76	135.38	135.57	135.76	135.50	135.89	135.15	136.33	136.58	136.31	136.65
	$CH(4,5)$	122.20	122.16	122.00	122.13	122.16	121.96	122.31	121.55	122.78	122.93	122.60	122.43
methane	$CH_4$	-4.90	-4.33	-4.63	-4.34	-4.29	-4.33	-5.33	-4.01	-4.61	-5.88	-4.90	-
methanol	$CH_3$	49.64	50.45	50.41	49.90	49.97	49.66	49.77	48.59	49.90	50.67	49.86	49.50 <sup>12</sup>
nitromethane	$CH_3$	62.49	63.03	62.50	61.14	61.16	61.68	63.21	63.28	63.66	63.17	63.08	63.22
<i>n</i> -pentane	$CH_3$	14.18	14.24	14.08	14.27	14.25	14.10	14.29	13.28	14.37	14.54	14.39	-
	$CH_2(2,4)$	23.00	22.77	22.38	22.79	22.72	22.54	22.98	21.70	23.08	23.75	23.38	-
	$CH_2(3)$	34.87	34.57	34.16	34.54	34.45	34.26	34.83	33.48	34.89	35.76	35.30	-
propane	$CH_3$	16.60	16.63	16.63	16.65	16.66	16.56	16.68	16.34	16.73	16.93	16.80	-
	$CH_2$	16.82	16.63	16.37	16.63	16.60	16.48	16.78	15.67	16.91	17.46	17.19	-
2-propanol	$CH_3$	25.70	25.43	25.14	25.24	25.18	25.14	25.67	25.43	25.55	25.21	25.27	24.38
	CH	66.14	64.67	64.50	64.12	64.23	64.18	63.85	64.92	64.30	66.69	64.71	64.88
propylene	$CH_3$	19.27	19.47	19.50	19.32	19.38	19.32	19.42	19.20	19.48	19.63	19.50	-
	$CH_2$	115.74	115.70	115.74	115.89	115.92	115.86	116.03	116.07	116.12	116.38	116.04	-
	CH	134.02	134.21	133.91	133.61	133.69	133.57	134.34	133.55	134.78	136.00	134.61	-
pump oil	$CH_2$	30.63	30.13	29.84	30.33	30.24	30.11	30.36	29.33	30.86	31.85	31.35	-
pyridine	$CH(2,6)$	150.57	150.27	149.90	150.25	150.27	149.93	150.67	149.58	150.76	149.76	150.07	149.18
	$CH(3,5)$	124.08	124.06	123.75	123.46	123.58	123.49	124.57	123.84	127.76	126.27	125.53	125.12
	$CH(4)$	135.99	136.16	135.96	135.17	135.28	135.32	136.56	136.05	136.89	139.62	138.35	138.27
pyrrole	$CH(2,5)$	118.03	117.93	117.77	117.61	117.78	117.65	117.98	117.32	118.47	119.61	118.28	119.06
	$CH(3,4)$	107.74	108.02	107.98	108.15	108.21	108.03	108.04	107.07	108.31	108.85	108.11	107.83
pyrrolidine <sup>10</sup>	$CH_2(2,5)$	45.82	47.02	46.93	47.12	46.86	46.75	-	46.51	47.57	47.43	47.23	46.83
	$CH_2(3,4)$	26.17	25.83	25.56	25.75	25.65	25.59	-	25.26	26.34	25.73	26.29	25.86
silicone grease	$CH_3$	1.20	1.22	1.19	1.37	1.38	1.09	1.40	-	-	2.87	2.10	-
tetrahydrofuran	$CH_2(2,5)$	68.03	68.16	67.97	67.75	67.80	67.64	68.07	67.03	68.33	69.53	68.83	68.68
	$CH_2(3,4)$	26.19	25.98	25.62	25.79	25.72	25.68	26.15	25.14	26.27	26.69	26.48	25.67
toluene	$CH_3$	21.29	21.53	21.46	21.37	21.10	21.23	21.46	20.99	21.50	21.62	21.50	-
	C(1)	138.24	138.36	137.89	137.84	137.91	137.65	138.48	137.35	138.90	139.92	138.85	-
	$CH(2,6)$	129.47	129.35	129.07	129.33	129.33	129.12	129.76	128.88	129.94	130.58	129.91	-
	$CH(3,5)$	128.71	128.54	128.26	128.51	128.56	128.31	129.03	128.18	129.23	129.79	129.20	-
triethylamine	$CH(4)$	125.84	125.62	125.33	125.66	125.68	125.43	126.12	125.29	126.28	126.82	126.29	-
	$CH_3$	12.51	12.12	11.61	12.39	12.35	11.87	12.49	11.74	12.38	9.51	11.09	9.07
	$CH_2$	47.18	46.75	46.25	46.82	46.77	46.36	47.07	45.74	47.10	48.45	46.96	47.19

**Table S3. THF-*d*<sub>8</sub> (<sup>1</sup>H NMR data by chemical shift in ppm)**

<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>	<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>	<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.21	s	ArCH <sub>3</sub>	BHT	4.50	ddd	CH <sub>2</sub>	allyl acetate
0.11	s	CH <sub>3</sub>	silicone grease	2.24	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.55	s	H <sub>2</sub>	hydrogen
0.19	s	CH <sub>4</sub>	methane	2.31	s	CH <sub>3</sub>	toluene	4.58	ddd	CH <sub>2</sub>	diallyl carbonate
0.85	s	CH <sub>3</sub>	ethane	2.39	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.89	dm, 10	CH <sub>2</sub> (1)	propylene
0.85–0.91	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.45	s	CH <sub>3</sub>	dimethyl sulfoxide	4.99	dm, 17	CH <sub>2</sub> (2)	propylene
0.86–0.90	m	CH <sub>3</sub>	pump oil	2.46	s	OH	water	5.15	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.89	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.46	q, 7	CH <sub>2</sub>	triethylamine	5.19	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.89	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.58	d, 9.5	CH <sub>3</sub>	HMPA	5.27	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.90	t, 7.3	CH <sub>3</sub>	propane	2.75	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.31	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.96	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.76	s	CH <sub>3</sub>	dimethylformamide	5.36	s	CH <sub>2</sub>	ethylene
0.97	t, 7	CH <sub>3</sub>	triethylamine	2.82	s	NCH <sub>3</sub>	dimethylacetamide	5.51	s	CH <sub>2</sub>	dichloromethane
1.08	d, 6	CH <sub>3</sub>	2-propanol	2.88	s	CH <sub>3</sub>	dimethylformamide	5.64	s	OH	BHA
1.10	t, 7	CH <sub>3</sub>	ethanol	2.95	s	NCH <sub>3</sub>	dimethylacetamide	5.79	m	CH	propylene
1.12	t, 7	CH <sub>3</sub>	diethyl ether	3.02	s <sup>o</sup>	OH	methanol	5.81	s	OH	BHT
1.15	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.16	s	OH	<i>tert</i> -butyl alcohol	5.90	ddt	CHCH <sub>2</sub>	allyl acetate
1.19	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.27	s <sup>o</sup>	CH <sub>3</sub>	methanol	5.92	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.29	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.28	s	OCH <sub>3</sub>	diglyme	6.02	m	CH(3,4)	pyrrole
1.29	m	CH <sub>2</sub>	<i>n</i> -hexane	3.28	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.37	dd	CH(3,4)	furan
1.29	br s	CH <sub>2</sub>	pump oil	3.30	s <sup>o</sup>	OH	ethanol	6.66	m	CH(2,5)	pyrrole
1.31	m	CH <sub>2</sub>	<i>n</i> -pentane	3.35	s	CH <sub>2</sub>	dimethyl malonate	6.68	s	ArH	BHA
1.33	sept, 7.3	CH <sub>2</sub>	propane	3.38	q, 7	CH <sub>2</sub>	diethyl ether	6.92	s	ArH	BHT
1.40	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.43	m	CH <sub>2</sub>	diglyme	6.94	s	CH(4,5)	imidazole
1.40	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.43	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.10	m	CH(2,4,6)	toluene
1.44	s	CH <sub>2</sub>	cyclohexane	3.48	s	CH <sub>2</sub>	ethylene glycol	7.19	m	CH(3,5)	toluene
1.59	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.51	q, 7 <sup>o</sup>	CH <sub>2</sub>	ethanol	7.25	m	CH(3,5)	pyridine
1.68–1.71	m	CH <sub>2</sub> (4)	cyclohexanone	3.53	m	CH <sub>2</sub>	diglyme	7.31	s	CH	benzene
1.69	dt, 6.4, 1.5	CH <sub>3</sub>	propylene	3.56	s	CH <sub>2</sub>	1,4-dioxane	7.48	dd	CH(2,5)	furan
1.72	m	CHD(3,4)	THF- <i>d</i> <sub>8</sub> residual	3.57	s	CH <sub>2</sub>	18-crown-6	7.48	s	CH(2)	imidazole
1.77–1.82	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.58	m	CHD(2,5)	THF- <i>d</i> <sub>8</sub> residual	7.51–7.55	m	CH(3,5)	benzaldehyde
1.79	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.62	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.60–7.64	m	CH(4)	benzaldehyde
1.89	s	CH <sub>3</sub>	acetic acid	3.65	s	CH <sub>3</sub>	dimethyl malonate	7.65	m	CH(4)	pyridine
1.94	s	CH <sub>2</sub> CO	dimethylacetamide	3.68	s	ArOCH <sub>3</sub>	BHA	7.86–7.88	m	CH(2,6)	benzaldehyde
1.94	s	CH <sub>2</sub> CO	ethyl acetate	3.69	s	CH <sub>3</sub>	dimethyl carbonate	7.89	s	CH	chloroform
1.95	s	CH <sub>3</sub>	acetonitrile	3.77	s	CH <sub>2</sub>	1,2-dichloroethane	7.91	s	CH	dimethylformamide
1.98	s	CH <sub>3</sub>	allyl acetate	3.82	sept, 6	CH	2-propanol	8.54	m	CH(2,6)	pyridine
2.03	s	CH <sub>2</sub> CO	ethyl methyl ketone	4.04	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	9.96	br t	NH	pyrrole
2.05	s	CH <sub>3</sub>	acetone	4.31	s	CH <sub>3</sub>	nitromethane	9.98	s	HCO	benzaldehyde
2.18	s	CH <sub>3</sub>	hexamethylbenzene								

**Table S4. THF-*d*<sub>8</sub> (<sup>13</sup>C{<sup>1</sup>H}) NMR data by chemical shift in ppm)**

<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>
-4.90	CH <sub>4</sub>	methane	30.17	CH <sub>3</sub>	acetone	64.35	CH <sub>2</sub>	ethylene glycol	129.47	CH(2,6)	toluene
0.45	CH <sub>3</sub>	acetonitrile	30.45	CH <sub>2</sub>	H grease <sup>8</sup>	65.31	CH <sub>2</sub>	allyl acetate	129.56	CH(3,5)	benzaldehyde
1.20	CH <sub>2</sub>	silicone grease	30.57	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	66.14	CH <sub>2</sub>	diethyl ether	129.98	CH(2,6)	benzaldehyde
1.83	CH <sub>3</sub>	hexamethyldisiloxane	30.63	CH <sub>2</sub>	pump oil	66.14	CH	2-propanol	131.88	C	hexamethylbenzene
6.79	CH <sub>3</sub>	ethane	30.65	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	67.21 (p)	CD <sub>2</sub> (2,5)	THF- <i>d</i> <sub>8</sub> signal	133.08	CHCH <sub>2</sub>	diallyl carbonate
7.87	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	30.70	CH <sub>3</sub>	dimethylformamide	67.50	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	133.90	CHCH <sub>2</sub>	allyl acetate
12.51	CH <sub>3</sub>	triethylamine	31.55	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	67.65	CH <sub>2</sub>	1,4-dioxane	134.02	CH	propylene
14.18	CH <sub>3</sub>	<i>n</i> -pentane	32.34	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	68.03	CH <sub>2</sub> (2,5)	tetrahydrofuran	134.67	CH(4)	benzaldehyde
14.22	CH <sub>3</sub>	<i>n</i> -hexane	34.60	NCH <sub>3</sub>	dimethylacetamide	68.58	CH <sub>2</sub>	diallyl carbonate	135.72	CH(2)	imidazole
14.37	CH <sub>2</sub>	ethyl acetate	34.87	CH <sub>2</sub> (3)	<i>n</i> -pentane	71.17	CH <sub>2</sub>	diglyme	135.99	CH(4)	pyridine
15.49	CH <sub>3</sub>	diethyl ether	34.91	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	71.34	CH <sub>2</sub>	18-crown-6	137.78	C(1)	benzaldehyde
16.60	CH <sub>3</sub>	propane	35.51	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	72.58	CH <sub>2</sub>	1,2-dimethoxyethane	137.93	C(2,6)	BHT
16.71	CH <sub>3</sub>	hexamethylbenzene	35.65	CH <sub>3</sub>	dimethylformamide	72.72	CH <sub>2</sub>	diglyme	138.24	C(1)	toluene
16.82	CH <sub>2</sub>	propane	36.59	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	79.24	CH	chloroform	140.07	C(4)	BHA
18.90	CH <sub>3</sub>	ethanol	36.89 (d)	CH <sub>3</sub>	HMPA <sup>11</sup>	96.89	CCl <sub>4</sub>	carbon tetrachloride	143.26	CH(2,5)	furan
19.27	CH <sub>3</sub>	propylene	37.56	NCH <sub>3</sub>	dimethylacetamide	107.74	CH(3,4)	pyrrole	148.62	C(2,6)	BHA
20.13	CH <sub>2</sub>	acetic acid	41.15	CH <sub>2</sub>	dimethyl malonate	109.88	CH(3,4)	furan	150.57	CH(2,6)	pyridine
20.45	CH <sub>3</sub>	allyl acetate	41.21	CH <sub>3</sub>	dimethyl sulfoxide	110.94	CH(3,5)	BHA	152.48	C(1)	BHT
20.45	CH <sub>2</sub> CO	ethyl acetate	42.17	CH <sub>2</sub> (2,6)	cyclohexanone	115.74	CH <sub>2</sub>	propylene	154.07	C(1)	BHA
21.15	CH <sub>3</sub>	dimethylacetamide	44.64	CH <sub>2</sub>	1,2-dichloroethane	116.79	CN	acetonitrile	155.36	CO	diallyl carbonate
21.21	CH <sub>2</sub> Ar	BHT	45.82	CH <sub>2</sub> (2,5)	pyrrolidine	117.58	CHCH <sub>2</sub>	allyl acetate	156.91	CO	dimethyl carbonate
21.29	CH <sub>3</sub>	toluene	47.18	CH <sub>2</sub>	triethylamine	117.70	CHCH <sub>2</sub>	diallyl carbonate	161.96	CH	dimethylformamide
23.00	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	49.64	CH <sub>3</sub>	methanol	118.03	CH(2,5)	pyrrole	167.14	CO <sub>2</sub>	dimethyl malonate
23.33	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	52.07	CH <sub>3</sub>	dimethyl malonate	122.20	CH(4,5)	imidazole	169.77	CO	dimethylacetamide
25.31 (p)	CD <sub>2</sub> (3,4)	THF- <i>d</i> <sub>8</sub> signal	54.58	CH <sub>3</sub>	dimethyl carbonate	123.09	CH <sub>2</sub>	ethylene	170.14	CO	allyl acetate
25.70	CH <sub>3</sub>	2-propanol	54.67	CH <sub>2</sub>	dichloromethane	124.08	CH(3,5)	pyridine	170.32	CO	ethyl acetate
25.76	CH <sub>2</sub> (4)	cyclohexanone	55.39	CH <sub>2</sub> O	BHA	125.69	CO <sub>2</sub>	carbon dioxide	171.69	CO	acetic acid
26.17	CH <sub>2</sub> (3,4)	pyrrolidine	57.60	CH <sub>2</sub>	ethanol	125.71	CH(3,5)	BHT	191.95	HCO	benzaldehyde
26.19	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.72	CH <sub>3</sub>	diglyme	125.84	CH(4)	toluene	193.37	CS <sub>2</sub>	carbon disulfide
27.58	CH <sub>2</sub>	cyclohexane	58.72	CH <sub>3</sub>	1,2-dimethoxyethane	128.64	C(4)	BHT	204.19	CO	acetone
27.69	CH <sub>2</sub> (3,5)	cyclohexanone	60.30	CH <sub>2</sub>	ethyl acetate	128.71	CH(3,5)	toluene	207.05	CO	ethyl methyl ketone
28.92	CH <sub>2</sub> CO	ethyl methyl ketone	62.49	CH <sub>3</sub>	nitromethane	128.84	CH	benzene	208.79	CO	cyclohexanone

**Table S5. CD<sub>2</sub>Cl<sub>2</sub> (<sup>1</sup>H NMR data by chemical shift in ppm)**

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.09	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.61	ddd	CH <sub>2</sub>	diallyl carbonate
0.09	s	CH <sub>3</sub>	silicone grease	2.12	s	CH <sub>3</sub>	acetone	4.76	s	OH	BHA
0.21	s	CH <sub>4</sub>	methane	2.20	s	CH <sub>3</sub>	hexamethylbenzene	4.93	dm, 10	CH <sub>2</sub> (1)	propylene
0.84-0.89	m	CH <sub>3</sub>	pump oil	2.25	s	ArCH <sub>3</sub>	BHT	5.00	s	OH	BHT
0.84-0.90	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.29	t	CH <sub>2</sub> (2,6)	cyclohexanone	5.03	dm, 17	CH <sub>2</sub> (2)	propylene
0.85	s	CH <sub>3</sub>	ethane	2.34	s	CH <sub>3</sub>	toluene	5.22	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.89	t, 7	CH <sub>3</sub>	n-hexane	2.43	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.26	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.89	t, 7	CH <sub>3</sub>	n-pentane	2.48	q, 7	CH <sub>2</sub>	triethylamine	5.31	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.90	t, 7,3	CH <sub>3</sub>	propane	2.55	s	CH <sub>3</sub>	dimethyl sulfoxide	5.32	t	CDHCl <sub>2</sub>	CD <sub>2</sub> Cl <sub>2</sub> residual
0.99	t, 7	CH <sub>3</sub>	triethylamine	2.60	d, 9,5	CH <sub>3</sub>	HMPA	5.33	s	CH <sub>2</sub>	dichloromethane
1.00	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.82	s	CH <sub>3</sub>	dimethylformamide	5.35	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.09	s <sup>9</sup>	OH	methanol	2.82	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.40	s	CH <sub>2</sub>	ethylene
1.15	t, 7	CH <sub>3</sub>	diethyl ether	2.87	s	NCH <sub>3</sub>	dimethylacetamide	5.84	m	CH	propylene
1.17	d, 6	CH <sub>3</sub>	2-propanol	2.91	s	CH <sub>3</sub>	dimethylformamide	5.92	ddt	CHCH <sub>2</sub>	allyl acetate
1.19	t, 7	CH <sub>3</sub>	ethanol	2.97	s	NCH <sub>3</sub>	dimethylacetamide	5.95	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.23	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.33	s	OCH <sub>3</sub>	diglyme	6.19	m	CH(3,4)	pyrrole
1.24	s	CH <sub>3</sub>	tert-butyl alcohol	3.34	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.41	dd	CH(3,4)	furane
1.27	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.37	s	CH <sub>2</sub>	dimethyl malonate	6.73	s	ArH	BHA
1.27	m	CH <sub>2</sub>	n-hexane	3.42	s <sup>9</sup>	CH <sub>3</sub>	methanol	6.79	m	CH(2,5)	pyrrole
1.27	br s	CH <sub>2</sub>	pump oil	3.43	q, 7	CH <sub>2</sub>	diethyl ether	6.97	s	ArH	BHT
1.30	m	CH <sub>2</sub>	n-pentane	3.49	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.07	s	CH(4,5)	imidazole
1.32	sept, 7,3	CH <sub>2</sub>	propane	3.50	m	CH <sub>2</sub>	diglyme	7.15	m	CH(2,4,6)	toluene
1.33	s <sup>9</sup>	OH	ethanol	3.57	m	CH <sub>2</sub>	diglyme	7.24	m	CH(3,5)	toluene
1.42	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.59	s	CH <sub>2</sub>	18-crown-6	7.28	m	CH(3,5)	pyridine
1.42	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.65	s	CH <sub>2</sub>	1,4-dioxane	7.32	s	CH	chloroform
1.44	s	CH <sub>2</sub>	cyclohexane	3.66	q, 7 <sup>9</sup>	CH <sub>2</sub>	ethanol	7.35	s	CH	benzene
1.52	s	OH	water	3.66	s	CH <sub>2</sub>	ethylene glycol	7.46	dd	CH(2,5)	furane
1.69-1.72	m	CH <sub>2</sub> (4)	cyclohexanone	3.69	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.53-7.57	m	CH(3,5)	benzaldehyde
1.67	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.72	s	CH <sub>3</sub>	dimethyl malonate	7.63	s	CH(2)	imidazole
1.71	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.73	s	ArOCH <sub>3</sub>	BHA	7.63-7.67	m	CH(4)	benzaldehyde
1.81-1.87	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.75	s	CH <sub>3</sub>	dimethyl carbonate	7.68	m	CH(4)	pyridine
1.82	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.76	s	CH <sub>2</sub>	1,2-dichloroethane	7.87-7.89	m	CH(2,6)	benzaldehyde
1.97	s	CH <sub>3</sub>	acetonitrile	3.97	sept, 6	CH	2-propanol	7.96	s	CH	dimethylformamide
2.00	s	CH <sub>2</sub> CO	ethyl acetate	4.08	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	8.59	m	CH(2,6)	pyridine
2.02	s	CH <sub>2</sub> CO	dimethylacetamide	4.31	s	CH <sub>3</sub>	nitromethane	8.69	br t	NH	pyrrole
2.05	s	CH <sub>3</sub>	allyl acetate	4.55	ddd	CH <sub>2</sub>	allyl acetate	10.01	s	HCO	benzaldehyde
2.06	s	CH <sub>3</sub>	acetic acid	4.59	s	H <sub>2</sub>	hydrogen				

**Table S6. CD<sub>2</sub>Cl<sub>2</sub> (<sup>13</sup>C{<sup>1</sup>H}) NMR data by chemical shift in ppm)**

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.33	CH <sub>4</sub>	methane	30.14	CH <sub>2</sub>	H grease <sup>8</sup>	64.08	CH <sub>2</sub>	ethylene glycol	129.42	CH(3,5)	benzaldehyde
1.22	CH <sub>3</sub>	silicone grease	30.37	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	64.67	CH	2-propanol	129.98	CH(2,6)	benzaldehyde
1.96	CH <sub>3</sub>	hexamethyldisiloxane	30.54	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	65.36	CH <sub>2</sub>	allyl acetate	132.09	C	hexamethylbenzene
2.03	CH <sub>3</sub>	acetonitrile	31.00	CH <sub>3</sub>	acetone	66.11	CH <sub>2</sub>	diethyl ether	132.24	CHCH <sub>2</sub>	diallyl carbonate
6.91	CH <sub>3</sub>	ethane	31.39	CH <sub>3</sub>	dimethylformamide	67.47	CH <sub>2</sub>	1,4-dioxane	132.94	CHCH <sub>2</sub>	allyl acetate
7.94	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.46	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	68.16	CH <sub>2</sub> (2,5)	tetrahydrofuran	134.21	CH	propylene
12.12	CH <sub>3</sub>	triethylamine	32.01	CH <sub>2</sub> (3,4)	n-hexane	68.76	CH <sub>2</sub>	diallyl carbonate	134.79	CH(4)	benzaldehyde
14.24	CH <sub>3</sub>	n-pentane	34.56	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	69.11	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	135.76	CH(2)	imidazole
14.28	CH <sub>3</sub>	n-hexane	34.57	CH <sub>2</sub> (3)	n-pentane	70.47	CH <sub>2</sub>	18-crown-6	136.16	CH(4)	pyridine
14.37	CH <sub>3</sub>	ethyl acetate	34.91	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	70.70	CH <sub>2</sub>	diglyme	136.32	C(2,6)	BHT
15.44	CH <sub>3</sub>	diethyl ether	35.23	NCH <sub>3</sub>	dimethylacetamide	72.24	CH <sub>2</sub>	1,2-dimethoxyethane	136.98	C(1)	benzaldehyde
16.63	CH <sub>3</sub>	propane	36.56	CH <sub>3</sub>	dimethylformamide	72.25	CH <sub>2</sub>	diglyme	137.77	C(4)	BHA
16.63	CH <sub>2</sub>	propane	36.99 (d)	CH <sub>3</sub>	HMPA'	77.99	CH	chloroform	138.36	C(1)	toluene
16.93	CH <sub>3</sub>	hexamethylbenzene	37.01	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	96.52	CCl <sub>4</sub>	carbon tetrachloride	142.98	CH(2,5)	furane
18.69	CH <sub>3</sub>	ethanol	38.22	NCH <sub>3</sub>	dimethylacetamide	108.02	CH(3,4)	pyrrole	148.06	C(2,6)	BHA
19.47	CH <sub>3</sub>	propylene	41.33	CH <sub>3</sub>	dimethyl sulfoxide	109.86	CH(3,4)	furane	150.27	CH(2,6)	pyridine
20.91	CH <sub>3</sub>	acetic acid	41.48	CH <sub>2</sub>	dimethyl malonate	110.93	CH(3,5)	BHA	151.92	C(1)	BHT
21.06	CH <sub>3</sub>	allyl acetate	42.31	CH <sub>2</sub> (2,6)	cyclohexanone	115.7	CH <sub>2</sub>	propylene	153.05	C(1)	BHA
21.15	CH <sub>3</sub> CO	ethyl acetate	44.35	CH <sub>2</sub>	1,2-dichloroethane	116.92	CN	acetonitrile	155.15	CO	diallyl carbonate
21.27	CH <sub>2</sub> Ar	BHT	46.75	CH <sub>2</sub>	triethylamine	117.93	CH(2,5)	pyrrole	156.73	CO	dimethyl carbonate
21.53	CH <sub>3</sub>	toluene	47.02	CH <sub>2</sub> (2,5)	pyrrolidine	118.00	CHCH <sub>2</sub>	allyl acetate	162.57	CH	dimethylformamide
21.64	CH <sub>3</sub>	dimethylacetamide	50.45	CH <sub>3</sub>	methanol	118.75	CHCH <sub>2</sub>	diallyl carbonate	167.32	CO <sub>2</sub>	dimethyl malonate
22.77	CH <sub>2</sub> (2,4)	n-pentane	52.75	CH <sub>3</sub>	dimethyl malonate	122.16	CH(4,5)	imidazole	170.83	CO	allyl acetate
23.07	CH <sub>2</sub> (2,5)	n-hexane	53.84 (p)	CD <sub>2</sub> Cl <sub>2</sub>	CD <sub>2</sub> Cl <sub>2</sub> signal	123.20	CH <sub>2</sub>	ethylene	171.05	CO	dimethylacetamide
25.42	CH <sub>2</sub> (4)	cyclohexanone	54.24	CH <sub>2</sub>	dichloromethane	124.06	CH(3,5)	pyridine	171.24	CO	ethyl acetate
25.43	CH <sub>3</sub>	2-propanol	55.09	CH <sub>3</sub>	dimethyl carbonate	125.26	CO <sub>2</sub>	carbon dioxide	175.85	CO	acetic acid
25.83	CH <sub>2</sub> (3,4)	pyrrolidine	55.88	CH <sub>3</sub> O	BHA	125.62	CH(4)	toluene	192.61	HCO	benzaldehyde
25.98	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.57	CH <sub>2</sub>	ethanol	125.84	CH(3,5)	BHT	192.95	CS <sub>2</sub>	carbon disulfide
27.38	CH <sub>2</sub>	cyclohexane	58.95	CH <sub>3</sub>	diglyme	128.54	CH(3,5)	toluene	206.78	CO	acetone
27.47	CH <sub>2</sub> (3,5)	cyclohexanone	59.02	CH <sub>3</sub>	1,2-dimethoxyethane	128.68	CH	benzene	209.57	CO	ethyl methyl ketone
29.55	CH <sub>2</sub> CO	ethyl methyl ketone	60.63	CH <sub>2</sub>	ethyl acetate	128.73	C(4)	BHT	211.82	CO	cyclohexanone
30.13	CH <sub>2</sub>	pump oil	63.03	CH <sub>3</sub>	nitromethane	129.35	CH(2,6)	toluene			



**Table S7.** CDCl<sub>3</sub> (<sup>1</sup>H NMR data by chemical shift in ppm)

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.14	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.64	ddd	CH <sub>2</sub>	diallyl carbonate
0.07	s	CH <sub>3</sub>	silicone grease	2.17	s	CH <sub>3</sub>	acetone	4.76	s	OH <sup>d</sup>	BHA
0.22	s	CH <sub>4</sub>	methane	2.24	s	CH <sub>3</sub>	hexamethylbenzene	4.94	dm, 10	CH <sub>2</sub> (1)	propylene
0.83–0.89	m	CH <sub>3</sub>	pump oil	2.27	s	ArCH <sub>3</sub>	BHT	5.01	s	OH <sup>d</sup>	BHT
0.84–0.87	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.33	t	CH <sub>2</sub> (2,6)	cyclohexanone	5.03	dm, 17	CH <sub>2</sub> (2)	propylene
0.87	s	CH <sub>3</sub>	ethane	2.36	s	CH <sub>3</sub>	toluene	5.24	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.88	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.46	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.27	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.88	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.53	q, 7	CH <sub>2</sub>	triethylamine	5.30	s	CH <sub>2</sub>	dichloromethane
0.90	t, 7,3	CH <sub>3</sub>	propane	2.62	s	CH <sub>3</sub>	dimethyl sulfoxide	5.32	ddt	CHCH <sub>2</sub> (1)	allyl acetate
1.03	t, 7	CH <sub>3</sub>	triethylamine	2.65	d, 9.5	CH <sub>3</sub>	HMPA	5.37	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.06	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.87	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.40	s	CH <sub>2</sub>	ethylene
1.09	s <sup>9</sup>	OH	methanol	2.88	s	CH <sub>3</sub>	dimethylformamide	5.83	m	CH	propylene
1.21	t, 7	CH <sub>3</sub>	diethyl ether	2.94	s	NCH <sub>3</sub>	dimethylacetamide	5.93	ddt	CHCH <sub>2</sub>	allyl acetate
1.22	d, 6	CH <sub>3</sub>	2-propanol	2.96	s	CH <sub>3</sub>	dimethylformamide	5.94	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.25	t, 7	CH <sub>3</sub>	ethanol	3.02	s	NCH <sub>3</sub>	dimethylacetamide	6.26	m	CH(3,4)	pyrrole
1.25	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.39	s	OCH <sub>3</sub>	diglyme	6.40	dd	CH(3,4)	furan
1.26	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.40	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.76	s	ArH	BHA
1.26	m	CH <sub>2</sub>	<i>n</i> -hexane	3.40	s	CH <sub>2</sub>	dimethyl malonate	6.83	m	CH(2,5)	pyrrole
1.26	br s	CH <sub>2</sub>	pump oil	3.48	q, 7	CH <sub>2</sub>	diethyl ether	6.98	s	ArH	BHT
1.27	m	CH <sub>2</sub>	<i>n</i> -pentane	3.49	s <sup>9</sup>	CH <sub>3</sub>	methanol	7.10	s	CH(4,5)	imidazole
1.28	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.55	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.17	m	CH(2,4,6)	toluene
1.32	s <sup>6</sup>	OH	ethanol	3.57	m	CH <sub>2</sub>	diglyme	7.25	m	CH(3,5)	toluene
1.32	sept, 7.3	CH <sub>2</sub>	propane	3.65	m	CH <sub>2</sub>	diglyme	7.26	s	CH	CDCl <sub>3</sub> residual
1.43	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.67	s	CH <sub>2</sub>	18-crown-6	7.26	s	CH	chloroform
1.43	s	CH <sub>2</sub>	cyclohexane	3.71	s	CH <sub>2</sub>	1,4-dioxane	7.29	m	CH(3,5)	pyridine
1.44	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.72	q, 7 <sup>9</sup>	CH <sub>2</sub>	ethanol	7.36	s	CH	benzene
1.56	s	OH	water	3.73	s	CH <sub>2</sub>	1,2-dichloroethane	7.45	dd	CH(2,5)	furan
1.68	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.75	s	CH <sub>3</sub>	dimethyl malonate	7.51–7.57	m	CH(3,5)	benzaldehyde
1.71–1.73	m	CH <sub>2</sub> (4)	cyclohexanone	3.76	s	CH <sub>2</sub>	ethylene glycol	7.61–7.65	m	CH(4)	benzaldehyde
1.73	dt, 6.4, 1.5	CH <sub>3</sub>	propylene	3.76	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.67	s	CH(2)	imidazole
1.84–1.86	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.77	s	ArOCH <sub>3</sub>	BHA	7.68	m	CH(4)	pyridine
1.85	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.79	s	CH <sub>3</sub>	dimethyl carbonate	7.88–7.91	m	CH(2,6)	benzaldehyde
2.05	s	CH <sub>3</sub> CO	ethyl acetate	4.04	sept, 6	CH	2-propanol	8.02	s	CH	dimethylformamide
2.09	s	CH <sub>3</sub>	allyl acetate	4.12	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	8.40	br t	NH	pyrrole
2.09	s	CH <sub>3</sub> CO	dimethylacetamide	4.33	s	CH <sub>3</sub>	nitromethane	8.62	m	CH(2,6)	pyridine
2.10	s	CH <sub>3</sub>	acetic acid	4.57	ddd	CH <sub>2</sub>	allyl acetate	10.03	s	HCO	benzaldehyde
2.10	s	CH <sub>3</sub>	acetonitrile	4.62	s	H <sub>2</sub>	hydrogen				

**Table S8.** CDCl<sub>3</sub> (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.63	CH <sub>4</sub>	methane	29.84	CH <sub>2</sub>	pump oil	64.50	CH	2-propanol	129.16	CH(3,5)	benzaldehyde
1.19	CH <sub>3</sub>	silicone grease	30.32	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	65.28	CH <sub>2</sub>	allyl acetate	129.91	CH(2,6)	benzaldehyde
1.89	CH <sub>3</sub>	acetonitrile	30.33	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	65.91	CH <sub>2</sub>	diethyl ether	131.58	CHCH <sub>2</sub>	diallyl carbonate
1.97	CH <sub>3</sub>	hexamethyldisiloxane	30.92	CH <sub>3</sub>	acetone	67.14	CH <sub>2</sub>	1,4-dioxane	132.21	C	hexamethylbenzene
6.89	CH <sub>3</sub>	ethane	31.25	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	67.97	CH <sub>2</sub> (2,5)	tetrahydrofuran	132.33	CHCH <sub>2</sub>	allyl acetate
7.86	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.45	CH <sub>3</sub>	dimethyl malonate	68.55	CH <sub>2</sub>	diallyl carbonate	133.91	CH	propylene
11.61	CH <sub>3</sub>	triethylamine	31.64	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	69.15	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	134.64	CH(4)	benzaldehyde
14.08	CH <sub>3</sub>	<i>n</i> -pentane	34.16	CH <sub>2</sub> (3)	<i>n</i> -pentane	70.51	CH <sub>2</sub>	diglyme	135.38	CH(2)	imidazole
14.14	CH <sub>3</sub>	<i>n</i> -hexane	34.25	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	70.55	CH <sub>2</sub>	18-crown-6	135.87	C(2,6)	BHT
14.19	CH <sub>3</sub>	ethyl acetate	34.72	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	71.84	CH <sub>2</sub>	1,2-dimethoxyethane	135.96	CH(4)	pyridine
15.20	CH <sub>3</sub>	diethyl ether	35.28	NCH <sub>3</sub>	dimethylacetamide	71.90	CH <sub>2</sub>	diglyme	136.58	C(1)	benzaldehyde
16.37	CH <sub>2</sub>	propane	36.50	CH <sub>3</sub>	dimethyl malonate	77.16 (t)	CDCl <sub>3</sub>	CDCl <sub>3</sub> signal	137.36	C(4)	BHA
16.63	CH <sub>3</sub>	propane	36.87 (d)	CH <sub>3</sub>	HMPA <sup>1</sup>	77.36	CH	chloroform	137.89	C(1)	toluene
16.98	CH <sub>3</sub>	hexamethylbenzene	36.89	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	96.34	CCl <sub>4</sub>	carbon tetrachloride	142.71	CH(2,5)	furan
18.41	CH <sub>3</sub>	ethanol	38.13	NCH <sub>3</sub>	dimethylacetamide	107.98	CH(3,4)	pyrrole	147.85	C(2,6)	BHA
19.50	CH <sub>3</sub>	propylene	40.76	CH <sub>3</sub>	dimethyl sulfoxide	109.57	CH(3,4)	furan	149.90	CH(2,6)	pyridine
20.81	CH <sub>3</sub>	acetic acid	41.11	CH <sub>2</sub>	dimethyl malonate	110.69	CH(3,5)	BHA	151.55	C(1)	BHT
21.02	CH <sub>3</sub>	allyl acetate	41.97	CH <sub>2</sub> (2,6)	cyclohexanone	115.74	CH <sub>2</sub>	propylene	152.57	C(1)	BHA
21.04	CH <sub>3</sub> CO	ethyl acetate	43.50	CH <sub>2</sub>	1,2-dichloroethane	116.43	CN	acetonitrile	154.88	CO	diallyl carbonate
21.20	CH <sub>3</sub> Ar	BHT	46.25	CH <sub>2</sub>	triethylamine	117.77	CH(2,5)	pyrrole	156.45	CO	dimethyl carbonate
21.46	CH <sub>3</sub>	toluene	46.93	CH <sub>2</sub> (2,5)	pyrrolidine	118.34	CHCH <sub>2</sub>	allyl acetate	162.62	CH	dimethylformamide
21.53	CH <sub>3</sub>	dimethylacetamide	50.41	CH <sub>3</sub>	methanol	118.96	CHCH <sub>2</sub>	diallyl carbonate	167.18	CO <sub>2</sub>	dimethyl malonate
22.38	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	52.57	CH <sub>3</sub>	dimethyl malonate	122.00	CH(4,5)	imidazole	170.81	CO	allyl acetate
22.70	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	53.52	CH <sub>2</sub>	dichloromethane	123.13	CH <sub>2</sub>	ethylene	171.07	CO	dimethylacetamide
24.97	CH <sub>2</sub> (4)	cyclohexanone	54.89	CH <sub>3</sub>	dimethyl carbonate	123.75	CH(3,5)	pyridine	171.36	CO	ethyl acetate
25.14	CH <sub>3</sub>	2-propanol	55.70	CH <sub>3</sub> O	BHA	124.99	CO <sub>2</sub>	carbon dioxide	175.99	CO	acetic acid
25.56	CH <sub>3</sub> (3,4)	pyrrolidine	58.28	CH <sub>2</sub>	ethanol	125.33	CH(4)	toluene	192.67	HCO	benzaldehyde
25.62	CH <sub>2</sub> (3,4)	tetrahydrofuran	59.01	CH <sub>3</sub>	diglyme	125.55	CH(3,5)	BHT	192.83	CS <sub>2</sub>	carbon disulfide
26.94	CH <sub>2</sub>	cyclohexane	59.08	CH <sub>3</sub>	1,2-dimethoxyethane	128.26	CH(3,5)	toluene	207.07	CO	acetone
27.00	CH <sub>2</sub> (3,5)	cyclohexanone	60.49	CH <sub>2</sub>	ethyl acetate	128.27	C(4)	BHT	209.56	CO	ethyl methyl ketone
29.49	CH <sub>3</sub> CO	ethyl methyl ketone	62.50	CH <sub>3</sub>	nitromethane	128.37	CH	benzene	212.57	CO	cyclohexanone
29.71	CH <sub>2</sub>	H grease <sup>8</sup>	63.79	CH <sub>2</sub>	ethylene glycol	129.07	CH(2,6)	toluene			

**Table S9.** Toluene- $d_8$  ( $^1\text{H}$  NMR data by chemical shift in ppm)

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.10	s	CH <sub>3</sub>	hexamethyldisiloxane	1.64	s	CH <sub>3</sub>	dimethyl sulfoxide	4.45	s	OH <sup>δ</sup>	BHA
0.17	s	CH <sub>4</sub>	methane	1.69	s	CH <sub>3</sub> CO	ethyl acetate	4.50	s	H <sub>2</sub>	hydrogen
0.26	s	CH <sub>3</sub>	silicone grease	1.82	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.72	s	OH <sup>δ</sup>	BHT
0.43	s	OH	water	1.95	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.92	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.58	s	OH	tert-butyl alcohol	1.96	s	CH <sub>3</sub>	dimethylformamide	4.92	dm, 10	CH <sub>2</sub> (1)	propylene
0.69	s	CH <sub>3</sub>	acetonitrile	2.08	p	CH <sub>3</sub>	Toluene- $d_8$ residual	4.94	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.81	s	CH <sub>3</sub>	ethane	2.10	s	CH <sub>3</sub>	hexamethylbenzene	4.98	dm, 17	CH <sub>2</sub> (2)	propylene
0.83	s <sup>δ</sup>	OH	ethanol	2.11	s	NCH <sub>3</sub>	dimethylacetamide	5.05	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.84	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.11	s	CH <sub>3</sub>	toluene	5.09	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.87	t, 7	CH <sub>3</sub>	n-pentane	2.23	s	ArCH <sub>3</sub>	BHT	5.25	s	CH <sub>2</sub>	ethylene
0.88	t, 7	CH <sub>3</sub>	n-hexane	2.37	s	CH <sub>3</sub>	dimethylformamide	5.63	ddt	CHCH <sub>2</sub>	diallyl carbonate
0.88-0.96	m	CH <sub>3</sub>	pump oil	2.39	q, 7	CH <sub>2</sub>	triethylamine	5.67 <sup>†</sup>	(nfo ABX)	CHCH <sub>2</sub>	allyl acetate
0.89	t, 7,3	CH <sub>3</sub>	propane	2.42	d, 9,5	CH <sub>3</sub>	HMPA	5.70	m	CH	propylene
0.89-0.96	m	CH <sub>3</sub>	H grease <sup>δ</sup>	2.54	m	CH <sub>2</sub> (2,5)	pyrrolidine	6.07	dd	CH(3,4)	furan
0.94	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	2.56	s	NCH <sub>3</sub>	dimethylacetamide	6.10	s	CH	chloroform
0.95	d, 6	CH <sub>3</sub>	2-propanol	2.91	s	CH <sub>2</sub>	1,2-dichloroethane	6.27	m	CH(3,4)	pyrrole
0.95	t, 7	CH <sub>3</sub>	triethylamine	2.92	s	CH <sub>2</sub>	dimethyl malonate	6.43	m	CH(2,5)	pyrrole
0.97	t, 7	CH <sub>3</sub>	ethanol	3.01	s	CH <sub>3</sub>	nitromethane	6.67	m	CH(3,5)	pyridine
1.03	s	CH <sub>3</sub>	tert-butyl alcohol	3.03	s <sup>δ</sup>	CH <sub>3</sub>	methanol	6.83	s	ArH	BHA
1.10	t, 7	CH <sub>3</sub>	diethyl ether	3.12	s	OCH <sub>3</sub>	diglyme	6.86	s	CH(4,5)	imidazole
1.16-1.20	m	CH <sub>2</sub> (4)	cyclohexanone	3.12	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.95-6.99	m	CH(3,5)	benzaldehyde
1.22	m	CH <sub>2</sub>	n-hexane	3.24	s	CH <sub>3</sub>	dimethyl malonate	6.96-7.01	m	CH(2,4,6)	toluene
1.25	m	CH <sub>2</sub>	n-pentane	3.25	q, 7	CH <sub>2</sub>	diethyl ether	6.97	p	CH(4)	Toluene- $d_8$ residual
1.30	br s	CH <sub>2</sub>	pump oil	3.31	m	CH <sub>2</sub>	diglyme	6.99	s	ArH	BHT
1.32	sept, 7,3	CH <sub>2</sub>	propane	3.31	s	CH <sub>2</sub>	1,2-dimethoxyethane	6.99	m	CH(4)	pyridine
1.33	br s	CH <sub>2</sub>	H grease <sup>δ</sup>	3.31	s	CH <sub>3</sub>	dimethyl carbonate	7.01	s	CH(2,6)	Toluene- $d_8$ residual
1.33-1.39	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.33	s	CH <sub>2</sub>	1,4-dioxane	7.03-7.07	m	CH(4)	benzaldehyde
1.34	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.36	s	CH <sub>2</sub>	18-crown-6	7.09	m	CH(3,5)	Toluene- $d_8$ residual
1.36	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.36	q, 7 <sup>δ</sup>	CH <sub>2</sub>	ethanol	7.09	m	CH(3,5)	toluene
1.36	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.36	s	CH <sub>2</sub>	ethylene glycol	7.10	dd	CH(2,5)	furan
1.40	s	CH <sub>2</sub>	cyclohexane	3.43	m	CH <sub>2</sub>	diglyme	7.12	s	CH	benzene
1.43	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.48	s	ArOCH <sub>3</sub>	BHA	7.30	s	CH(2)	imidazole
1.55	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.54	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.45-7.47	m	CH(2,6)	benzaldehyde
1.57	s	CH <sub>3</sub>	acetic acid	3.65	sept, 6	CH	2-propanol	7.57	s	CH	dimethylformamide
1.57	s	CH <sub>3</sub>	acetone	3.87	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	7.71	br t	NH	pyrrole
1.59	s	CH <sub>3</sub> CO	dimethylacetamide	4.32	s	CH <sub>2</sub>	dichloromethane	8.47	m	CH(2,6)	pyridine
1.59	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.34	ddd	CH <sub>2</sub>	allyl acetate	9.57	s	HCO	benzaldehyde
1.63	s	CH <sub>3</sub>	allyl acetate	4.34	ddd	CH <sub>2</sub>	diallyl carbonate				

**Table S10.** Toluene- $d_8$  ( $^{13}\text{C}\{^1\text{H}\}$  NMR data by chemical shift in ppm)

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.34	CH <sub>4</sub>	methane	30.30	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	64.87	CH <sub>2</sub>	allyl acetate	129.33	CH(2,6)	toluene
0.03	CH <sub>3</sub>	acetonitrile	30.31	CH <sub>2</sub>	H grease <sup>δ</sup>	65.94	CH <sub>2</sub>	diethyl ether	129.61	CH(2,6)	benzaldehyde
1.37	CH <sub>3</sub>	silicone grease	30.33	CH <sub>2</sub>	pump oil	67.17	CH <sub>2</sub>	1,4-dioxane	131.72	C	hexamethylbenzene
1.99	CH <sub>3</sub>	hexamethyldisiloxane	30.49	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	67.75	CH <sub>2</sub> (2,5)	tetrahydrofuran	132.30	CHCH <sub>2</sub>	diallyl carbonate
6.94	CH <sub>3</sub>	ethane	30.64	CH <sub>3</sub>	dimethylformamide	68.12	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	132.98	CHCH <sub>2</sub>	allyl acetate
7.89	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.39	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	68.20	CH <sub>2</sub>	diallyl carbonate	133.61	CH	propylene
12.39	CH <sub>3</sub>	triethylamine	32.06	CH <sub>2</sub> (3,4)	n-hexane	70.86	CH <sub>2</sub>	18-crown-6	133.88	CH(4)	benzaldehyde
14.23	CH <sub>3</sub>	ethyl acetate	34.39	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	70.92	CH <sub>2</sub>	diglyme	135.17	CH(4)	pyridine
14.27	CH <sub>3</sub>	n-pentane	34.54	CH <sub>2</sub> (3)	n-pentane	72.25	CH <sub>2</sub>	1,2-dimethoxyethane	135.57	CH(2)	imidazole
14.34	CH <sub>3</sub>	n-hexane	34.58	NCH <sub>3</sub>	dimethylacetamide	72.39	CH <sub>2</sub>	diglyme	136.12	C(2,6)	BHT
15.47	CH <sub>3</sub>	diethyl ether	34.69	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	77.89	CH	chloroform	137.12	C(1)	benzaldehyde
16.63	CH <sub>2</sub>	propane	35.22	CH <sub>3</sub>	dimethylformamide	96.57	CCl <sub>4</sub>	carbon tetrachloride	137.34	C(4)	BHA
16.65	CH <sub>3</sub>	propane	36.32	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	108.15	CH(3,4)	pyrrole	137.48	C	toluene- $d_8$ signal
16.84	CH <sub>3</sub>	hexamethylbenzene	36.8 (d)	CH <sub>3</sub>	HMPA <sup>††</sup>	109.63	CH(3,4)	furan	137.84	C(1)	toluene
18.78	CH <sub>3</sub>	ethanol	36.98	NCH <sub>3</sub>	dimethylacetamide	110.99	CH(3,5)	BHA	142.65	CH(2,5)	furan
19.32	CH <sub>3</sub>	propylene	40.41	CH <sub>3</sub>	dimethyl sulfoxide	115.76	CN	acetonitrile	148.06	C(2,6)	BHA
20.21	CH <sub>3</sub>	allyl acetate	40.88	CH <sub>2</sub>	dimethyl malonate	115.89	CH <sub>2</sub>	propylene	150.25	CH(2,6)	pyridine
20.27	CH <sub>3</sub>	acetic acid	41.78	CH <sub>2</sub> (2,6)	cyclohexanone	117.49	CHCH <sub>2</sub>	allyl acetate	152.06	C(1)	BHT
20.43 (sept)	CD <sub>3</sub>	toluene- $d_8$ signal	43.40	CH <sub>2</sub>	1,2-dichloroethane	117.61	CH(2,5)	pyrrole	153.50	C(1)	BHA
20.46	CH <sub>2</sub> CO	ethyl acetate	46.82	CH <sub>2</sub>		118.04	CHCH <sub>2</sub>	diallyl carbonate	155.15	CO	diallyl carbonate
21.05	CH <sub>3</sub>	dimethylacetamide	47.12	CH <sub>2</sub> (2,5)	pyrrolidine	122.13	CH(4,5)	imidazole	156.61	CO	dimethyl carbonate
21.37	CH <sub>3</sub>	toluene	49.90	CH <sub>3</sub>	methanol	122.92	CH <sub>2</sub>	ethylene	161.93	CH	dimethylformamide
21.42	CH <sub>3</sub> Ar	BHT	51.76	CH <sub>3</sub>	dimethyl malonate	123.46	CH(3,5)	pyridine	166.49	CO <sub>2</sub>	dimethyl malonate
22.79	CH <sub>2</sub> (2,4)	n-pentane	53.47	CH <sub>2</sub>	dichloromethane	124.86	CO <sub>2</sub>	carbon dioxide	169.44	CO	allyl acetate
23.12	CH <sub>2</sub> (2,5)	n-hexane	54.13	CH <sub>3</sub>	dimethyl carbonate	125.13 (t)	CD(4)	toluene- $d_8$ signal	169.65	CO	dimethylacetamide
25.15	CH <sub>2</sub> (4)	cyclohexanone	55.04	CH <sub>2</sub> O	BHA	125.66	CH(4)	toluene	170.02	CO	ethyl acetate
25.24	CH <sub>3</sub>	2-propanol	57.81	CH <sub>2</sub>	ethanol	125.79	CH(3,5)	BHT	175.30	CO	acetic acid
25.75	CH <sub>2</sub> (3,4)	pyrrolidine	58.62	CH <sub>3</sub>	diglyme	127.96 (t)	CD(3,5)	toluene- $d_8$ signal	191.09	HCO	benzaldehyde
25.79	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.63	CH <sub>3</sub>	1,2-dimethoxyethane	128.44	C(4)	BHT	192.71	CS <sub>2</sub>	carbon disulfide
27.05	CH <sub>2</sub> (3,5)	cyclohexanone	60.08	CH <sub>2</sub>	ethyl acetate	128.51	CH(3,5)	toluene	204.00	CO	acetone
27.31	CH <sub>2</sub>	cyclohexane	61.14	CH <sub>3</sub>	nitromethane	128.57	CH	benzene	206.31	CO	ethyl methyl ketone
28.74	CH <sub>3</sub> CO	ethyl methyl ketone	64.12	CH	2-propanol	128.68	CH(3,5)	benzaldehyde	208.60	CO	cyclohexanone
30.03	CH <sub>3</sub>	acetone	64.29	CH <sub>2</sub>	ethylene glycol	128.87 (t)	CD(2,6)	toluene- $d_8$ signal			

**Table S11. C<sub>6</sub>D<sub>6</sub> (<sup>1</sup>H NMR data by chemical shift in ppm)**

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.12	s	CH <sub>3</sub>	hexamethyldisiloxane	1.63	s	CH <sub>3</sub>	allyl acetate	4.38	ddd	CH <sub>2</sub>	diallyl carbonate
0.16	s	CH <sub>4</sub>	methane	1.65	s	CH <sub>3</sub> CO	ethyl acetate	4.47	s	H <sub>2</sub>	hydrogen
0.29	s	CH <sub>3</sub>	silicone grease	1.68	s	CH <sub>3</sub>	dimethyl sulfoxide	4.53	s	OH <sup>δ</sup>	BHA
0.40	s	OH	water	1.81	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.79	s	OH <sup>δ</sup>	BHT
0.50	s <sup>δ</sup>	OH	ethanol	1.86	s	CH <sub>3</sub>	dimethylformamide	4.92	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.58	s	CH <sub>3</sub>	acetonitrile	1.98	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.94	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.63	s	OH	<i>tert</i> -butyl alcohol	2.05	s	NCH <sub>3</sub>	dimethylacetamide	4.95	dm, 10	CH <sub>2</sub> (1)	propylene
0.80	s	CH <sub>3</sub>	ethane	2.11	s	CH <sub>3</sub>	toluene	5.01	dm, 17	CH <sub>2</sub> (2)	propylene
0.85	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.13	s	CH <sub>3</sub>	hexamethylbenzene	5.06	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.86	t, 7, 3	CH <sub>3</sub>	propane	2.24	s	ArCH <sub>3</sub>	BHT	5.09	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.87	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.36	s	CH <sub>3</sub>	dimethylformamide	5.25	s	CH <sub>2</sub>	ethylene
0.89	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.40	d, 9, 5	CH <sub>3</sub>	HMPA	5.65	ddt	CHCH <sub>2</sub>	diallyl carbonate
0.90–0.98	m	CH <sub>3</sub>	H grease <sup>δ</sup>	2.40	q, 7	CH <sub>2</sub>	triethylamine	5.68 <sup>δ</sup>	t(nfo ABX)	CHCH <sub>2</sub>	allyl acetate
0.91–0.97	m	CH <sub>3</sub>	pump oil	2.54	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.72	m	CH	propylene
0.92	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	2.57	s	NCH <sub>3</sub>	dimethylacetamide	6.08	dd	CH(3,4)	uran
0.95	d, 6	CH <sub>3</sub>	2-propanol	2.90	s	CH <sub>2</sub>	1,2-dichloroethane	6.15	s	CH	chloroform
0.96	t, 7	CH <sub>3</sub>	ethanol	2.94	s	CH <sub>3</sub>	nitromethane	6.37	m	CH(3,4)	pyrrole
0.96	t, 7	CH <sub>3</sub>	triethylamine	2.97	s	CH <sub>2</sub>	dimethyl malonate	6.48	m	CH(2,5)	pyrrole
1.05	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.07	s <sup>δ</sup>	CH <sub>3</sub>	methanol	6.66	m	CH(3,5)	pyridine
1.08–1.16	m	CH <sub>2</sub> (4)	cyclohexanone	3.11	s	OCH <sub>3</sub>	diglyme	6.90	s	CH(4,5)	imidazole
1.11	t, 7	CH <sub>3</sub>	diethyl ether	3.12	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.93	s	ArH	BHA
1.23	m	CH <sub>2</sub>	<i>n</i> -pentane	3.23	s	CH <sub>3</sub>	dimethyl malonate	6.93–6.99	m	CH(3,5)	benzaldehyde
1.24	m	CH <sub>2</sub>	<i>n</i> -hexane	3.26	q, 7	CH <sub>2</sub>	diethyl ether	6.98	m	CH(4)	pyridine
1.26	sept, 7, 3	CH <sub>2</sub>	propane	3.30	s	CH <sub>3</sub>	dimethyl carbonate	7.01–7.07	m	CH(4)	benzaldehyde
1.28–1.37	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.33	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.02	m	CH(2,4,6)	toluene
1.32	br s	CH <sub>2</sub>	H grease <sup>δ</sup>	3.34	m	CH <sub>2</sub>	diglyme	7.05	s	ArH	BHT
1.33	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.34	q, 7 <sup>δ</sup>	CH <sub>2</sub>	ethanol	7.13	dd	CH(2,5)	furan
1.37	br s	CH <sub>2</sub>	pump oil	3.35	s	CH <sub>2</sub>	1,4-dioxane	7.13	m	CH(3,5)	toluene
1.38	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.39	s	CH <sub>2</sub>	18-crown-6	7.15	s	CH	benzene
1.40	s	CH <sub>2</sub>	cyclohexane	3.41	s	CH <sub>2</sub>	ethylene glycol	7.16	s	CH	C <sub>6</sub> D <sub>6</sub> residual
1.40	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.46	m	CH <sub>2</sub>	diglyme	7.33	s	CH(2)	imidazole
1.41	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.48	s	ArOCH <sub>3</sub>	BHA	7.49–7.53	m	CH(2,6)	benzaldehyde
1.52	s	CH <sub>3</sub>	acetic acid	3.57	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.63	s	CH	dimethylformamide
1.55	s	CH <sub>3</sub>	acetone	3.67	sept, 6	CH	2-propanol	7.80	br t	NH	pyrrole
1.55	dt, 6, 4, 1, 5	CH <sub>3</sub>	propylene	3.89	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	8.53	m	CH(2,6)	pyridine
1.58	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.27	s	CH <sub>2</sub>	dichloromethane	9.64	s	HCO	benzaldehyde
1.60	s	CH <sub>2</sub> CO	dimethylacetamide	4.38	ddd	CH <sub>2</sub>	allyl acetate				

**Table S12. C<sub>6</sub>D<sub>6</sub> (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)**

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.29	CH <sub>4</sub>	methane	30.22	CH <sub>2</sub>	H grease <sup>δ</sup>	64.34	CH <sub>2</sub>	ethylene glycol	129.33	CH(2,6)	toluene
0.20	CH <sub>3</sub>	acetonitrile	30.24	CH <sub>2</sub>	pump oil	64.92	CH <sub>2</sub>	allyl acetate	129.65	CH(2,6)	benzaldehyde
1.38	CH <sub>3</sub>	silicone grease	30.35	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	65.94	CH <sub>2</sub>	diethyl ether	131.79	C	hexamethylbenzene
2.05	CH <sub>3</sub>	hexamethyldisiloxane	30.47	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	67.16	CH <sub>2</sub>	1,4-dioxane	132.18	CHCH <sub>2</sub>	diallyl carbonate
6.96	CH <sub>3</sub>	ethane	30.72	CH <sub>3</sub>	dimethylformamide	67.80	CH <sub>2</sub> (2,5)	tetrahydrofuran	132.90	CHCH <sub>2</sub>	allyl acetate
7.91	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.34	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	68.19	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	133.69	CH	propylene
12.35	CH <sub>3</sub>	triethylamine	31.96	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	68.28	CH <sub>2</sub>	diallyl carbonate	133.95	CH(4)	benzaldehyde
14.19	CH <sub>3</sub>	ethyl acetate	34.35	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	70.59	CH <sub>2</sub>	18-crown-6	135.28	CH(4)	pyridine
14.25	CH <sub>3</sub>	<i>n</i> -pentane	34.45	CH <sub>2</sub> (3)	<i>n</i> -pentane	70.87	CH <sub>2</sub>	diglyme	135.76	CH(2)	imidazole
14.32	CH <sub>3</sub>	<i>n</i> -hexane	34.67	NCH <sub>3</sub>	dimethylacetamide	72.21	CH <sub>2</sub>	1,2-dimethoxyethane	136.08	C(2,6)	BHT
15.46	CH <sub>3</sub>	diethyl ether	34.72	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	72.35	CH <sub>2</sub>	diglyme	137.05	C(1)	benzaldehyde
16.60	CH <sub>2</sub>	propane	35.25	CH <sub>3</sub>	dimethylformamide	77.79	CH	chloroform	137.50	C(4)	BHA
16.66	CH <sub>3</sub>	propane	36.36	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	96.44	CCl <sub>4</sub>	carbon tetrachloride	137.91	C(1)	toluene
16.95	CH <sub>3</sub>	hexamethylbenzene	36.88 (d)	CH <sub>3</sub>	HMPA <sup>11</sup>	108.21	CH(3,4)	pyrrole	142.73	CH(2,5)	furan
18.72	CH <sub>3</sub>	ethanol	37.03	NCH <sub>3</sub>	dimethylacetamide	109.67	CH(3,4)	furan	148.13	C(2,6)	BHA
19.38	CH <sub>3</sub>	propylene	40.03	CH <sub>3</sub>	dimethyl sulfoxide	111.15	CH(3,5)	BHA	150.27	CH(2,6)	pyridine
20.37	CH <sub>3</sub>	acetic acid	41.04	CH <sub>2</sub>	dimethyl malonate	115.92	CH <sub>2</sub>	propylene	152.05	C(1)	BHT
20.37	CH <sub>3</sub>	allyl acetate	41.83	CH <sub>2</sub> (2,6)	cyclohexanone	116.02	CN	acetonitrile	153.62	C(1)	BHA
20.56	CH <sub>3</sub> CO	ethyl acetate	43.59	CH <sub>2</sub>	1,2-dichloroethane	117.64	CHCH <sub>2</sub>	allyl acetate	155.24	CO	diallyl carbonate
21.10	CH <sub>3</sub>	toluene	46.77	CH <sub>2</sub>	triethylamine	117.78	CH(2,5)	pyrrole	156.71	CO	dimethyl carbonate
21.16	CH <sub>3</sub>	dimethylacetamide	46.86	CH <sub>2</sub> (2,5)	pyrrolidine	118.22	CHCH <sub>2</sub>	diallyl carbonate	162.13	CH	dimethylformamide
21.40	CH <sub>2</sub> Ar	BHT	49.97	CH <sub>3</sub>	methanol	122.16	CH(4,5)	imidazole	166.66	CO <sub>2</sub>	dimethyl malonate
22.72	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	51.86	CH <sub>3</sub>	dimethyl malonate	122.96	CH <sub>2</sub>	ethylene	169.67	CO	allyl acetate
23.04	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	53.46	CH <sub>2</sub>	dichloromethane	123.58	CH(3,5)	pyridine	169.95	CO	dimethylacetamide
25.03	CH <sub>2</sub> (4)	cyclohexanone	54.30	CH <sub>3</sub>	dimethyl carbonate	124.76	CO <sub>2</sub>	carbon dioxide	170.44	CO	ethyl acetate
25.18	CH <sub>3</sub>	2-propanol	55.27	CH <sub>3</sub> O	BHA	125.68	CH(4)	toluene	175.82	CO	acetic acid
25.65	CH <sub>2</sub> (3,4)	pyrrolidine	57.86	CH <sub>2</sub>	ethanol	125.83	CH(3,5)	BHT	191.43	HCO	benzaldehyde
25.72	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.66	CH <sub>3</sub>	diglyme	128.06 (t)	CD	C <sub>6</sub> D <sub>6</sub> signal	192.69	CS <sub>2</sub>	carbon disulfide
27.00	CH <sub>2</sub> (3,5)	cyclohexanone	58.68	CH <sub>3</sub>	1,2-dimethoxyethane	128.52	C(4)	BHT	204.43	CO	acetone
27.23	CH <sub>2</sub>	cyclohexane	60.21	CH <sub>2</sub>	ethyl acetate	128.56	CH(3,5)	toluene	206.55	CO	ethyl methyl ketone
28.56	CH <sub>3</sub> CO	ethyl methyl ketone	61.16	CH <sub>3</sub>	nitromethane	128.62	CH	benzene	209.10	CO	cyclohexanone
30.14	CH <sub>3</sub>	acetone	64.23	CH	2-propanol	128.95	CH(3,5)	benzaldehyde			

**Table S13.** C<sub>6</sub>D<sub>5</sub>Cl (<sup>1</sup>H NMR data by chemical shift in ppm)

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.10	s	CH <sub>3</sub>	hexamethyldisiloxane	1.78	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.62	s	OH <sup>δ</sup>	BHA
0.14	s	CH <sub>3</sub>	silicone grease	1.80	s	CH <sub>3</sub>	allyl acetate	4.77	s	CH <sub>2</sub>	dichloromethane
0.15	s	CH <sub>4</sub>	methane	2.03	s	CH <sub>3</sub>	dimethyl sulfoxide	4.91	dm, 10	CH <sub>2</sub> (1)	propylene
0.79	s	CH <sub>3</sub>	ethane	2.06	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.98	dm, 17	CH <sub>2</sub> (2)	propylene
0.84	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.08	t	CH <sub>2</sub> (2,6)	cyclohexanone	5.03	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.84	t, 7,3	CH <sub>3</sub>	propane	2.10	s	CH <sub>3</sub>	hexamethylbenzene	5.04	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.85	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.16	s	CH <sub>3</sub>	toluene	5.15	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.86-0.92	m	CH <sub>3</sub>	H grease <sup>δ</sup>	2.20	s	ArCH <sub>3</sub>	BHT	5.17	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.88-0.91	m	CH <sub>3</sub>	pump oil	2.30	s	CH <sub>3</sub>	dimethylformamide	5.29	s	CH <sub>2</sub>	ethylene
0.89	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.39	q, 7	CH <sub>2</sub>	triethylamine	5.50	s	OH <sup>δ</sup>	BHT
0.93	t, 7	CH <sub>3</sub>	triethylamine	2.42	s	NCH <sub>3</sub>	dimethylacetamide	5.72	m	CH	propylene
1.03	s	OH	water	2.47	d, 9,5	CH <sub>3</sub>	HMPA	5.75	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.04	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	2.51	s	CH <sub>3</sub>	dimethylformamide	5.77	ddt	CHCH <sub>2</sub>	allyl acetate
1.04	d, 6	CH <sub>3</sub>	2-propanol	2.64	m	CH <sub>2</sub> (2,5)	pyrrolidine	6.19	dd	CH(3,4)	furan
1.06	t, 7	CH <sub>3</sub>	ethanol	2.65	s	NCH <sub>3</sub>	dimethylacetamide	6.27	m	CH(3,4)	pyrrole
1.10	t, 7	CH <sub>3</sub>	diethyl ether	3.15	s	CH <sub>2</sub>	dimethyl malonate	6.62	m	CH(2,5)	pyrrole
1.12	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.16	s	OCH <sub>3</sub>	diglyme	6.74	s	CH	chloroform
1.19	m	CH <sub>2</sub>	<i>n</i> -hexane	3.17	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.83	s	ArH	BHA
1.21	s	CH <sub>3</sub>	acetonitrile	3.25	s <sup>δ</sup>	CH <sub>3</sub>	methanol	6.90	m	CH(3,5)	pyridine
1.23	m	CH <sub>2</sub>	<i>n</i> -pentane	3.26	s	CH <sub>2</sub>	1,2-dichloroethane	6.96	br. s	CH(4)	C <sub>6</sub> D <sub>5</sub> Cl residual
1.26	sept, 7,3	CH <sub>2</sub>	propane	3.31	q, 7	CH <sub>2</sub>	diethyl ether	6.97	s	ArH	BHT
1.30	s	OH	<i>tert</i> -butyl alcohol	3.37	m	CH <sub>2</sub>	diglyme	6.99	br. s	CH(3,5)	C <sub>6</sub> D <sub>5</sub> Cl residual
1.30	br s	CH <sub>2</sub>	H grease <sup>δ</sup>	3.37	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.01	s	CH(4,5)	imidazole
1.30	s <sup>δ</sup>	OH	methanol	3.41	s	CH <sub>2</sub>	18-crown-6	7.01-7.08	m	CH(2,4,6)	toluene
1.31	br s	CH <sub>2</sub>	pump oil	3.41	s	CH <sub>3</sub>	dimethyl malonate	7.10-7.17	m	CH(3,5)	toluene
1.33-1.37	m	CH <sub>2</sub> (4)	cyclohexanone	3.45	s	CH <sub>2</sub>	1,4-dioxane	7.14	br. s	CH(2,6)	C <sub>6</sub> D <sub>5</sub> Cl residual
1.37	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.48	s	CH <sub>3</sub>	dimethyl carbonate	7.15-7.19	m	CH(3,5)	benzaldehyde
1.37	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.49	m	CH <sub>2</sub>	diglyme	7.20	s	CH	benzene
1.37	s	CH <sub>2</sub>	cyclohexane	3.51	q, 7 <sup>δ</sup>	CH <sub>2</sub>	ethanol	7.24	dd	CH(2,5)	furan
1.39	s <sup>δ</sup>	OH	ethanol	3.58	s	CH <sub>2</sub>	ethylene glycol	7.24-7.28	m	CH(4)	benzaldehyde
1.43	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.59	s	CH <sub>3</sub>	nitromethane	7.25	m	CH(4)	pyridine
1.48-1.53	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.59	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.53	s	CH(2)	imidazole
1.55	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.61	s	ArOCH <sub>3</sub>	BHA	7.59-7.61	m	CH(2,6)	benzaldehyde
1.58	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.82	sept, 6	CH	2-propanol	7.73	s	CH	dimethylformamide
1.74	s	CH <sub>3</sub> CO	dimethylacetamide	3.96	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	8.51	m	CH(2,6)	pyridine
1.76	s	CH <sub>3</sub>	acetic acid	4.44	ddd	CH <sub>2</sub>	allyl acetate	8.61	br t	NH	pyrrole
1.77	s	CH <sub>3</sub>	acetone	4.46	ddd	CH <sub>2</sub>	diallyl carbonate	9.77	s	HCO	benzaldehyde
1.78	s	CH <sub>3</sub> CO	ethyl acetate	4.49	s	H <sub>2</sub>	hydrogen				

**Table S14.** C<sub>6</sub>D<sub>5</sub>Cl (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.33	CH <sub>4</sub>	methane	30.12	CH <sub>3</sub>	acetone	65.79	CH <sub>2</sub>	diethyl ether	129.26 (t)	CD(2,6)	C <sub>6</sub> D <sub>5</sub> Cl signal
0.63	CH <sub>3</sub>	acetonitrile	30.19	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	66.95	CH <sub>2</sub>	1,4-dioxane	129.49	CH(2,6)	benzaldehyde
1.09	CH <sub>3</sub>	silicone grease	30.21	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	67.64	CH <sub>2</sub> (2,5)	tetrahydrofuran	131.54	C	hexamethylbenzene
1.92	CH <sub>3</sub>	hexamethyldisiloxane	30.71	CH <sub>3</sub>	dimethylformamide	68.19	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	131.93	CHCH <sub>2</sub>	diallyl carbonate
6.91	CH <sub>3</sub>	ethane	31.13	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	68.19	CH <sub>2</sub>	diallyl carbonate	132.69	CHCH <sub>2</sub>	allyl acetate
7.79	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.77	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	70.55	CH <sub>2</sub>	18-crown-6	133.57	CH	propylene
11.87	CH <sub>3</sub>	triethylamine	34.11	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	70.56	CH <sub>2</sub>	diglyme	134.02	CH(4)	benzaldehyde
14.07	CH <sub>3</sub>	ethyl acetate	34.26	CH <sub>2</sub> (3)	<i>n</i> -pentane	71.81	CH <sub>2</sub>	1,2-dimethoxyethane	134.19	CCl	C <sub>6</sub> D <sub>5</sub> Cl signal
14.10	CH <sub>3</sub>	<i>n</i> -pentane	34.56	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	72.07	CH <sub>2</sub>	diglyme	135.32	CH(4)	pyridine
14.18	CH <sub>3</sub>	<i>n</i> -hexane	34.59	NCH <sub>3</sub>	dimethylacetamide	77.67	CH	chloroform	135.50	CH(2)	imidazole
15.35	CH <sub>3</sub>	diethyl ether	35.45	CH <sub>3</sub>	dimethylformamide	96.38	CCl <sub>4</sub>	carbon tetrachloride	135.92	C(2,6)	BHT
16.48	CH <sub>2</sub>	propane	36.39	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	108.03	CH(3,4)	pyrrole	136.78	C(1)	benzaldehyde
16.56	CH <sub>3</sub>	propane	36.64 (d)	CH <sub>3</sub>	HMPA <sup>17</sup>	109.64	CH(3,4)	furan	137.29	C(4)	BHA
16.68	CH <sub>3</sub>	hexamethylbenzene	37.13	NCH <sub>3</sub>	dimethylacetamide	110.84	CH(3,5)	BHA	137.65	C(1)	toluene
18.55	CH <sub>3</sub>	ethanol	40.27	CH <sub>3</sub>	dimethyl sulfoxide	115.86	CH <sub>2</sub>	propylene	142.49	CH(2,5)	furan
19.32	CH <sub>3</sub>	propylene	40.93	CH <sub>2</sub>	dimethyl malonate	115.93	CN	acetonitrile	147.87	C(2,6)	BHA
20.40	CH <sub>3</sub>	acetic acid	41.79	CH <sub>2</sub> (2,6)	cyclohexanone	117.63	CHCH <sub>2</sub>	allyl acetate	149.93	CH(2,6)	pyridine
20.40	CH <sub>3</sub>	allyl acetate	43.60	CH <sub>2</sub>	1,2-dichloroethane	117.65	CH(2,5)	pyrrole	151.69	C(1)	BHT
20.50	CH <sub>3</sub> CO	ethyl acetate	46.36	CH <sub>2</sub>	triethylamine	118.22	CHCH <sub>2</sub>	diallyl carbonate	153.19	C(1)	BHA
21.03	CH <sub>3</sub>	dimethylacetamide	46.75	CH <sub>2</sub> (2,5)	pyrrolidine	121.96	CH(4,5)	imidazole	154.87	CO	diallyl carbonate
21.10	CH <sub>2</sub> Ar	BHT	49.66	CH <sub>3</sub>	methanol	122.95	CH <sub>2</sub>	ethylene	156.36	CO	dimethyl carbonate
21.23	CH <sub>3</sub>	toluene	51.89	CH <sub>3</sub>	dimethyl malonate	123.49	CH(3,5)	pyridine	162.01	CH	dimethylformamide
22.54	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	53.54	CH <sub>2</sub>	dichloromethane	125.43	CH(4)	toluene	166.51	CO <sub>2</sub>	dimethyl malonate
22.86	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	54.23	CH <sub>3</sub>	dimethyl carbonate	125.58	CH(3,5)	BHT	169.59	CO	allyl acetate
25.07	CH <sub>2</sub> (4)	cyclohexanone	55.08	CH <sub>3</sub> O	BHA	125.96 (t)	CD(4)	C <sub>6</sub> D <sub>5</sub> Cl signal	169.79	CO	dimethylacetamide
25.14	CH <sub>3</sub>	2-propanol	57.63	CH <sub>2</sub>	ethanol	126.08	CO <sub>2</sub>	carbon dioxide	170.20	CO	ethyl acetate
25.59	CH <sub>2</sub> (3,4)	pyrrolidine	58.31	CH <sub>3</sub>	1,2-dimethoxyethane	128.25 (t)	CD(3,5)	C <sub>6</sub> D <sub>5</sub> Cl signal	175.67	CO	acetic acid
25.68	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.42	CH <sub>3</sub>	diglyme	128.26	C(4)	BHT	191.24	HCO	benzaldehyde
26.99	CH <sub>2</sub>	cyclohexane	60.06	CH <sub>2</sub>	ethyl acetate	128.31	CH(3,5)	toluene	192.49	CS <sub>2</sub>	carbon disulfide
27.02	CH <sub>2</sub> (3,5)	cyclohexanone	61.68	CH <sub>3</sub>	nitromethane	128.38	CH	benzene	204.83	CO	acetone
28.82	CH <sub>3</sub> CO	ethyl methyl ketone	64.03	CH <sub>2</sub>	ethylene glycol	128.87	CH(3,5)	benzaldehyde	206.87	CO	ethyl methyl ketone
30.11	CH <sub>2</sub>	H grease <sup>δ</sup>	64.18	CH	2-propanol	129.12	CH(2,6)	toluene	209.30	CO	cyclohexanone
30.11	CH <sub>2</sub>	pump oil	64.86	CH <sub>2</sub>	allyl acetate						

**Table S15. (CD<sub>3</sub>)<sub>2</sub>CO (<sup>1</sup>H NMR data by chemical shift in ppm)**

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.17	s	CH <sub>3</sub>	hexamethylbenzene	4.54	s	H <sub>2</sub>	hydrogen
0.13	s	CH <sub>3</sub>	silicone grease	2.22	s	ArCH <sub>3</sub>	BHT	4.62	ddd	CH <sub>2</sub>	diallyl carbonate
0.17	s	CH <sub>4</sub>	methane	2.27	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.90	dm, 10	CH <sub>2</sub> (1)	propylene
0.83	s	CH <sub>3</sub>	ethane	2.32	s	CH <sub>3</sub>	toluene	5.00	dm, 17	CH <sub>2</sub> (2)	propylene
0.87	m	CH <sub>3</sub>	pump oil	2.45	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.18	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.88	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.45	q, 7	CH <sub>2</sub>	triethylamine	5.23	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.88	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.52	s	CH <sub>3</sub>	dimethyl sulfoxide	5.29	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.88	t, 7.3	CH <sub>3</sub>	propane	2.59	d, 9.5	CH <sub>3</sub>	HMPA	5.35	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.90	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.78	s	CH <sub>3</sub>	dimethylformamide	5.38	s	CH <sub>2</sub>	ethylene
0.96	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.83	s	NCH <sub>3</sub>	dimethylacetamide	5.63	s	CH <sub>2</sub>	dichloromethane
0.96	t, 7	CH <sub>3</sub>	triethylamine	2.84 <sup>3</sup>	s	OH	water	5.65	s	OH <sup>5</sup>	BHA
1.10	d, 6	CH <sub>3</sub>	2-propanol	2.94	s	CH <sub>3</sub>	dimethylformamide	5.81	m	CH	propylene
1.11	t, 7	CH <sub>3</sub>	diethyl ether	3.00	s	NCH <sub>3</sub>	dimethylacetamide	5.92	ddt	CHCH <sub>2</sub>	allyl acetate
1.12	t, 7	CH <sub>3</sub>	ethanol	3.12	s <sup>9</sup>	OH	methanol	5.96	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.18	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.28	s	OCH <sub>3</sub>	diglyme	6.07	m	CH(3,4)	pyrrole
1.20	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.28	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.43	dd	CH(3,4)	furan
1.27	m	CH <sub>2</sub>	<i>n</i> -pentane	3.28	s	CH <sub>2</sub>	ethylene glycol	6.72	s	ArH	BHA
1.28	m	CH <sub>2</sub>	<i>n</i> -hexane	3.31	s <sup>9</sup>	CH <sub>3</sub>	methanol	6.77	m	CH(2,5)	pyrrole
1.29	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.39	s <sup>6</sup>	OH	ethanol	6.96	s	ArH	BHT
1.29	br s	CH <sub>2</sub>	pump oil	3.41	q, 7	CH <sub>2</sub>	diethyl ether	7.04	s	CH(4,5)	imidazole
1.31	sept, 7.3	CH <sub>2</sub>	propane	3.42	s	CH <sub>2</sub>	dimethyl malonate	7.10–7.20	m	CH(2,4,6)	toluene
1.41	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.46	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.10–7.20	m	CH(3,5)	toluene
1.41	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.47	m	CH <sub>2</sub>	diglyme	7.35	m	CH(3,5)	pyridine
1.43	s	CH <sub>2</sub>	cyclohexane	3.56	m	CH <sub>2</sub>	diglyme	7.36	s	CH	benzene
1.68	dt, 6.4, 1.5	CH <sub>3</sub>	propylene	3.57	q, 7 <sup>6</sup>	CH <sub>2</sub>	ethanol	7.56	dd	CH(2,5)	furan
1.70–1.74	m	CH <sub>2</sub> (4)	cyclohexanone	3.59	s	CH <sub>2</sub>	18-crown-6	7.59–7.63	m	CH(3,5)	benzaldehyde
1.79	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.59	s	CH <sub>2</sub>	1,4-dioxane	7.62	s	CH(2)	imidazole
1.79–1.83	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.63	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.69–7.73	m	CH(4)	benzaldehyde
1.96	s	CH <sub>3</sub>	acetic acid	3.68	s	CH <sub>3</sub>	dimethyl malonate	7.76	m	CH(4)	pyridine
1.97	s	CH <sub>2</sub> CO	dimethylacetamide	3.72	s	ArOCH <sub>3</sub>	BHA	7.92–7.94	m	CH(2,6)	benzaldehyde
1.97	s	CH <sub>2</sub> CO	ethyl acetate	3.72	s	CH <sub>3</sub>	dimethyl carbonate	7.96	s	CH	dimethylformamide
2.02	s	CH <sub>3</sub>	allyl acetate	3.87	s	CH <sub>2</sub>	1,2-dichloroethane	8.02	s	CH	chloroform
2.05	s	CH <sub>3</sub>	acetonitrile	3.90	sept, 6	CH	2-propanol	8.58	m	CH(2,6)	pyridine
2.05	p	CHD <sub>2</sub>	(CD <sub>3</sub> ) <sub>2</sub> CO residual	4.05	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	10.02	br t	NH	pyrrole
2.07	s	CH <sub>2</sub> CO	ethyl methyl ketone	4.43	s	CH <sub>3</sub>	nitromethane	10.05	s	HCO	benzaldehyde
2.09	s	CH <sub>3</sub>	acetone	4.53	ddd	CH <sub>2</sub>	allyl acetate				

**Table S16. (CD<sub>3</sub>)<sub>2</sub>CO (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)**

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-5.33	CH <sub>4</sub>	methane	30.60	CH <sub>3</sub>	acetone	65.28	CH <sub>2</sub>	allyl acetate	130.23	CH(2,6)	benzaldehyde
1.12	CH <sub>3</sub>	acetonitrile	30.64	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	66.12	CH <sub>2</sub>	diethyl ether	132.22	C	hexamethylbenzene
1.40	CH <sub>3</sub>	silicone grease	30.72	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	67.60	CH <sub>2</sub>	1,4-dioxane	133.16	CHCH <sub>2</sub>	diallyl carbonate
2.01	CH <sub>3</sub>	hexamethyldisiloxane	31.03	CH <sub>3</sub>	dimethylformamide	68.07	CH <sub>2</sub> (2,5)	tetrahydrofuran	133.76	CHCH <sub>2</sub>	allyl acetate
6.88	CH <sub>3</sub>	ethane	31.61	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	68.13	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	134.34	CH	propylene
8.03	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	32.30	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	68.78	CH <sub>2</sub>	diallyl carbonate	135.14	CH(4)	benzaldehyde
12.49	CH <sub>3</sub>	triethylamine	34.83	CH <sub>2</sub> (3)	<i>n</i> -pentane	71.03	CH <sub>2</sub>	diglyme	135.89	CH(2)	imidazole
14.29	CH <sub>3</sub>	<i>n</i> -pentane	34.89	NCH <sub>3</sub>	dimethylacetamide	71.25	CH <sub>2</sub>	18-crown-6	136.56	CH(4)	pyridine
14.34	CH <sub>3</sub>	<i>n</i> -hexane	35.00	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	72.47	CH <sub>2</sub>	1,2-dimethoxyethane	137.66	C(1)	benzaldehyde
14.50	CH <sub>3</sub>	ethyl acetate	35.45	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	72.63	CH <sub>2</sub>	diglyme	138.19	C(2,6)	BHT
15.78	CH <sub>3</sub>	diethyl ether	36.15	CH <sub>3</sub>	dimethylformamide	79.19	CH	chloroform	138.48	C(1)	toluene
16.68	CH <sub>3</sub>	propane	36.75	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	96.65	CCl <sub>4</sub>	carbon tetrachloride	140.32	CH(4)	BHA
16.78	CH <sub>2</sub>	propane	37.04 (d)	CH <sub>3</sub>	HMPA <sup>11</sup>	108.04	CH(3,4)	pyrrole	143.49	CH(2,5)	furan
16.86	CH <sub>3</sub>	hexamethylbenzene	37.92	NCH <sub>3</sub>	dimethylacetamide	110.24	CH(3,4)	furan	148.48	C(2,6)	BHA
18.89	CH <sub>3</sub>	ethanol	41.23	CH <sub>3</sub>	dimethyl sulfoxide	111.00	CH(3,5)	BHA	150.67	CH(2,6)	pyridine
19.42	CH <sub>3</sub>	propylene	41.43	CH <sub>2</sub>	dimethyl malonate	116.03	CH <sub>2</sub>	propylene	152.51	C(1)	BHT
20.51	CH <sub>3</sub>	acetic acid	42.24	CH <sub>2</sub> (2,6)	cyclohexanone	117.60	CN	acetonitrile	153.97	C(1)	BHA
20.68	CH <sub>3</sub>	allyl acetate	45.25	CH <sub>2</sub>	1,2-dichloroethane	117.81	CHCH <sub>2</sub>	allyl acetate	155.48	CO	diallyl carbonate
20.83	CH <sub>2</sub> CO	ethyl acetate	47.07	CH <sub>2</sub>	triethylamine	117.98	CH(2,5)	pyrrole	157.04	CO	dimethyl carbonate
21.31	CH <sub>3</sub> Ar	BHT	49.77	CH <sub>3</sub>	methanol	118.53	CHCH <sub>2</sub>	diallyl carbonate	162.79	CH	dimethylformamide
21.46	CH <sub>3</sub>	toluene	52.47	CH <sub>3</sub>	dimethyl malonate	122.31	CH(4,5)	imidazole	167.58	CO <sub>2</sub>	dimethyl malonate
21.51	CH <sub>3</sub>	dimethylacetamide	54.95	CH <sub>2</sub>	dichloromethane	123.47	CH <sub>2</sub>	ethylene	170.61	CO	allyl acetate
22.98	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	54.95	CH <sub>3</sub>	dimethyl carbonate	124.57	CH(3,5)	pyridine	170.61	CO	dimethylacetamide
23.28	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	55.51	CH <sub>3</sub> O	BHA	125.81	CO <sub>2</sub>	carbon dioxide	170.96	CO	ethyl acetate
25.59	CH <sub>2</sub> (4)	cyclohexanone	57.72	CH <sub>2</sub>	ethanol	126.03	CH(3,5)	BHT	172.31	CO	acetic acid
25.67	CH <sub>3</sub>	2-propanol	58.45	CH <sub>3</sub>	1,2-dimethoxyethane	126.12	CH(4)	toluene	192.95	HCO	benzaldehyde
26.15	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.77	CH <sub>3</sub>	diglyme	129.03	CH(3,5)	toluene	193.58	CS <sub>2</sub>	carbon disulfide
27.51	CH <sub>2</sub>	cyclohexane	60.56	CH <sub>2</sub>	ethyl acetate	129.05	C(4)	BHT	205.87	CO	acetone
27.68	CH <sub>2</sub> (3,5)	cyclohexanone	63.21	CH <sub>3</sub>	nitromethane	129.15	CH	benzene	206.26	CO	(CD <sub>3</sub> ) <sub>2</sub> CO signal
29.30	CH <sub>2</sub> CO	ethyl methyl ketone	63.85	CH	2-propanol	129.76	CH(2,6)	toluene	208.30	CO	ethyl methyl ketone
29.84 (sept)	CD <sub>3</sub>	(CD <sub>3</sub> ) <sub>2</sub> CO signal	64.26	CH <sub>2</sub>	ethylene glycol	129.90	CH(3,5)	benzaldehyde	210.36	CO	cyclohexanone
30.36	CH <sub>2</sub>	pump oil									

**Table S17. (CD<sub>3</sub>)<sub>2</sub>SO (<sup>1</sup>H NMR data by chemical shift in ppm)**

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
-0.06	s	CH <sub>3</sub>	silicone grease	2.18	s	ArCH <sub>3</sub>	BHT	4.61	ddd	CH <sub>2</sub>	diallyl carbonate
0.06	s	CH <sub>3</sub>	hexamethyldisiloxane	2.25	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.61	s	H <sub>2</sub>	hydrogen
0.20	s	CH <sub>4</sub>	methane	2.30	s	CH <sub>3</sub>	toluene	4.63	s <sup>5</sup>	OH	ethanol
0.74	m	CH <sub>3</sub>	pump oil	2.43	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.94	dm, 10	CH <sub>2</sub> (1)	propylene
0.82	s	CH <sub>3</sub>	ethane	2.43	q, 7	CH <sub>2</sub>	triethylamine	5.03	dm, 17	CH <sub>2</sub> (2)	propylene
0.82-0.88	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.50	p	CHD <sub>2</sub>	(CD <sub>3</sub> ) <sub>2</sub> SO residual	5.20	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.86	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.53	d, 9,5	CH <sub>3</sub>	HMPA	5.25	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.86	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.54	s	CH <sub>3</sub>	dimethyl sulfoxide	5.29	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.87	t, 7,3	CH <sub>3</sub>	propane	2.67	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.33	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
0.91	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.73	s	CH <sub>3</sub>	dimethylformamide	5.41	s	CH <sub>2</sub>	ethylene
0.93	t, 7	CH <sub>3</sub>	triethylamine	2.78	s	NCH <sub>3</sub>	dimethylacetamide	5.76	s	CH <sub>2</sub>	dichloromethane
1.04	d, 6	CH <sub>3</sub>	2-propanol	2.89	s	CH <sub>3</sub>	dimethylformamide	5.80	m	CH	propylene
1.06	t, 7	CH <sub>3</sub>	ethanol	2.94	s	NCH <sub>3</sub>	dimethylacetamide	5.91	ddt	CHCH <sub>2</sub>	allyl acetate
1.09	t, 7	CH <sub>3</sub>	diethyl ether	3.16	s <sup>9</sup>	CH <sub>3</sub>	methanol	5.93	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.11	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.24	s	OCH <sub>3</sub>	diglyme	6.01	m	CH(3,4)	pyrrole
1.15	br s	CH <sub>2</sub>	pump oil	3.24	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.47	dd	CH(3,4)	furan
1.17	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.33 <sup>2</sup>	s	OH	water	6.52	s	OH <sup>5</sup>	BHA
1.24	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.34	s	CH <sub>2</sub>	ethylene glycol	6.62	s	ArH	BHA
1.25	m	CH <sub>2</sub>	<i>n</i> -hexane	3.38	q, 7	CH <sub>2</sub>	diethyl ether	6.65	s	OH <sup>5</sup>	BHT
1.27	m	CH <sub>2</sub>	<i>n</i> -pentane	3.38	m	CH <sub>2</sub>	diglyme	6.73	m	CH(2,5)	pyrrole
1.29	sept, 7,3	CH <sub>2</sub>	propane	3.43	s	CH <sub>2</sub>	1,2-dimethoxyethane	6.87	s	ArH	BHT
1.36	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.44	q, 7 <sup>9</sup>	CH <sub>2</sub>	ethanol	7.01	s	CH(4,5)	imidazole
1.36	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.51	s	CH <sub>2</sub>	18-crown-6	7.18	m	CH(2,4,6)	toluene
1.40	s	CH <sub>2</sub>	cyclohexane	3.51	m	CH <sub>2</sub>	diglyme	7.25	m	CH(3,5)	toluene
1.55	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.53	s	CH <sub>2</sub>	dimethyl malonate	7.37	s	CH	benzene
1.64-1.66	m	CH <sub>2</sub> (4)	cyclohexanone	3.57	s	CH <sub>2</sub>	1,4-dioxane	7.39	m	CH(3,5)	pyridine
1.68	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.60	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.61-7.67	m	CH(3,5)	benzaldehyde
1.74-1.78	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.65	s	CH <sub>3</sub>	dimethyl malonate	7.63	s	CH(2)	imidazole
1.76	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.66	s	ArOCH <sub>3</sub>	BHA	7.67	dd	CH(2,5)	furan
1.91	s	CH <sub>3</sub>	acetic acid	3.69	s	CH <sub>3</sub>	dimethyl carbonate	7.69-7.75	m	CH(4)	benzaldehyde
1.96	s	CH <sub>2</sub> CO	dimethylacetamide	3.78	sept, 6	CH	2-propanol	7.79	m	CH(4)	pyridine
1.99	s	CH <sub>3</sub> CO	ethyl acetate	3.90	s	CH <sub>2</sub>	1,2-dichloroethane	7.91-7.93	m	CH(2,6)	benzaldehyde
2.03	s	CH <sub>3</sub>	allyl acetate	4.01	s <sup>9</sup>	OH	methanol	8.32	s	CH	chloroform
2.07	s	CH <sub>3</sub>	acetonitrile	4.03	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	8.58	m	CH(2,6)	pyridine
2.07	s	CH <sub>2</sub> CO	ethyl methyl ketone	4.19	s	OH	<i>tert</i> -butyl alcohol	10.02	s	HCO	benzaldehyde
2.09	s	CH <sub>3</sub>	acetone	4.42	s	CH <sub>3</sub>	nitromethane	10.75	br t	NH	pyrrole
2.14	s	CH <sub>3</sub>	hexamethylbenzene	4.52	ddd	CH <sub>2</sub>	allyl acetate				

**Table S18. (CD<sub>3</sub>)<sub>2</sub>SO (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)**

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.01	CH <sub>4</sub>	methane	30.38	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	63.28	CH <sub>3</sub>	nitromethane	129.10	CH(3,5)	benzaldehyde
1.03	CH <sub>3</sub>	acetonitrile	30.56	CH <sub>3</sub>	acetone	64.32	CH <sub>2</sub>	allyl acetate	129.45	CH(2,6)	benzaldehyde
1.96	CH <sub>3</sub>	hexamethyldisiloxane	30.73	CH <sub>3</sub>	dimethylformamide	64.92	CH	2-propanol	131.10	C	hexamethylbenzene
6.61	CH <sub>3</sub>	ethane	30.95	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	66.36	CH <sub>2</sub>	1,4-dioxane	132.18	CHCH <sub>2</sub>	diallyl carbonate
7.61	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	31.25	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	66.88	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	132.71	CHCH <sub>2</sub>	allyl acetate
11.74	CH <sub>3</sub>	triethylamine	33.48	CH <sub>2</sub> (3)	<i>n</i> -pentane	67.03	CH <sub>2</sub> (2,5)	tetrahydrofuran	133.55	CH	propylene
13.28	CH <sub>3</sub>	<i>n</i> -pentane	34.33	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	67.86	CH <sub>2</sub>	diallyl carbonate	134.52	CH(4)	benzaldehyde
13.88	CH <sub>3</sub>	<i>n</i> -hexane	34.42	NCH <sub>3</sub>	dimethylacetamide	69.54	CH <sub>2</sub>	diglyme	135.15	CH(2)	imidazole
14.40	CH <sub>3</sub>	ethyl acetate	34.76	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	69.85	CH <sub>2</sub>	18-crown-6	136.05	CH(4)	pyridine
15.12	CH <sub>3</sub>	diethyl ether	35.73	CH <sub>3</sub>	dimethylformamide	71.17	CH <sub>2</sub>	1,2-dimethoxyethane	136.20	C(1)	benzaldehyde
15.67	CH <sub>2</sub>	propane	35.83	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	71.25	CH <sub>2</sub>	diglyme	137.35	C(1)	toluene
16.34	CH <sub>3</sub>	propane	36.42 (d)	CH <sub>3</sub>	HMPA <sup>11</sup>	79.16	CH	chloroform	139.12	C(2,6)	BHT
16.60	CH <sub>3</sub>	hexamethylbenzene	37.38	NCH <sub>3</sub>	dimethylacetamide	95.44	CCl <sub>4</sub>	carbon tetrachloride	141.16	C(4)	BHA
18.51	CH <sub>3</sub>	ethanol	39.52 (sept)	CD <sub>3</sub>	(CD <sub>3</sub> ) <sub>2</sub> SO signal	107.07	CH(3,4)	pyrrole	142.82	CH(2,5)	furan
19.20	CH <sub>3</sub>	propylene	40.45	CH <sub>3</sub>	dimethyl sulfoxide	109.62	CH(3,4)	furan	147.44	C(2,6)	BHA
20.54	CH <sub>3</sub>	allyl acetate	40.72	CH <sub>2</sub>	dimethyl malonate	109.80	CH(3,5)	BHA	149.58	CH(2,6)	pyridine
20.68	CH <sub>2</sub> CO	ethyl acetate	41.32	CH <sub>2</sub> (2,6)	cyclohexanone	116.07	CH <sub>2</sub>	propylene	151.47	C(1)	BHT
20.95	CH <sub>3</sub>	acetic acid	45.02	CH <sub>2</sub>	1,2-dichloroethane	117.32	CH(2,5)	pyrrole	152.53	C(1)	BHA
20.97	CH <sub>2</sub> Ar	BHT	45.74	CH <sub>2</sub>	triethylamine	117.64	CHCH <sub>2</sub>	allyl acetate	154.16	CO	diallyl carbonate
20.99	CH <sub>3</sub>	toluene	46.51	CH <sub>2</sub> (2,5)	pyrrolidine	117.91	CN	acetonitrile	155.76	CO	dimethyl carbonate
21.29	CH <sub>3</sub>	dimethylacetamide	48.59	CH <sub>3</sub>	methanol	118.32	CHCH <sub>2</sub>	diallyl carbonate	162.29	CH	dimethylformamide
21.70	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	52.08	CH <sub>3</sub>	dimethyl malonate	121.55	CH(4,5)	imidazole	166.91	CO <sub>2</sub>	dimethyl malonate
22.05	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	54.63	CH <sub>3</sub>	dimethyl carbonate	123.52	CH <sub>2</sub>	ethylene	169.54	CO	dimethylacetamide
24.32	CH <sub>2</sub> (4)	cyclohexanone	54.84	CH <sub>2</sub>	dichloromethane	123.84	CH(3,5)	pyridine	169.97	CO	allyl acetate
25.14	CH <sub>2</sub> (3,4)	tetrahydrofuran	54.89	CH <sub>3</sub> O	BHA	124.21	CO <sub>2</sub>	carbon dioxide	170.31	CO	ethyl acetate
25.26	CH <sub>2</sub> (3,4)	pyrrolidine	56.07	CH <sub>2</sub>	ethanol	124.85	CH(3,5)	BHT	171.93	CO	acetic acid
25.43	CH <sub>3</sub>	2-propanol	57.98	CH <sub>3</sub>	diglyme	125.29	CH(4)	toluene	192.63	CS <sub>2</sub>	carbon disulfide
26.33	CH <sub>2</sub>	cyclohexane	58.03	CH <sub>3</sub>	1,2-dimethoxyethane	127.97	C(4)	BHT	193.08	HCO	benzaldehyde
26.46	CH <sub>2</sub> (3,5)	cyclohexanone	59.74	CH <sub>2</sub>	ethyl acetate	128.18	CH(3,5)	toluene	206.31	CO	acetone
29.26	CH <sub>3</sub> CO	ethyl methyl ketone	62.05	CH <sub>2</sub>	diethyl ether	128.30	CH	benzene	208.72	CO	ethyl methyl ketone
29.33	CH <sub>2</sub>	pump oil	62.76	CH <sub>2</sub>	ethylene glycol	128.88	CH(2,6)	toluene	210.63	CO	cyclohexanone
30.30	(CH <sub>3</sub> ) <sub>3</sub> C	BHA									

**Table S19.** CD<sub>3</sub>CN (<sup>1</sup>H NMR data by chemical shift in ppm)

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.18	s	OH	<i>tert</i> -butyl alcohol	4.57	s	H <sub>2</sub>	hydrogen
0.08	s	CH <sub>3</sub>	silicone grease	2.19	s	CH <sub>3</sub>	hexamethylbenzene	4.61	ddd	CH <sub>2</sub>	diallyl carbonate
0.20	s	CH <sub>4</sub>	methane	2.22	s	ArCH <sub>3</sub>	BHT	4.93	dm, 10	CH <sub>2</sub> (1)	propylene
0.85	s	CH <sub>3</sub>	ethane	2.27	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.98	s	OH <sup>5</sup>	BHA
0.85	m	CH <sub>3</sub>	pump oil	2.33	s	CH <sub>3</sub>	toluene	5.04	dm, 17	CH <sub>2</sub> (2)	propylene
0.89	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.43	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.20	s	OH <sup>5</sup>	BHT
0.89	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.45	q, 7	CH <sub>2</sub>	triethylamine	5.21	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.90	t, 7, 3	CH <sub>3</sub>	propane	2.47	s <sup>o</sup>	OH	ethanol	5.25	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.96	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.50	s	CH <sub>3</sub>	dimethyl sulfoxide	5.29	ddt	CHCH <sub>2</sub> (1)	allyl acetate
0.96	t, 7	CH <sub>3</sub>	triethylamine	2.57	d, 9, 5	CH <sub>3</sub>	HMPA	5.34	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.09	d, 6	CH <sub>3</sub>	2-propanol	2.69 <sup>7</sup>	m <sup>7</sup>	OH <sup>7</sup>	ethylene glycol <sup>7</sup>	5.41	s	CH <sub>2</sub>	ethylene
1.12	t, 7	CH <sub>3</sub>	diethyl ether	2.75	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.44	s	CH	dichloromethane
1.12	t, 7	CH <sub>3</sub>	ethanol	2.77	s	CH <sub>3</sub>	dimethylformamide	5.85	m	CH	propylene
1.16	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	2.83	s	NCH <sub>3</sub>	dimethylacetamide	5.93	ddt	CHCH <sub>2</sub>	allyl acetate
1.20	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	2.89	s	CH <sub>3</sub>	dimethylformamide	5.96	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.27	br s	CH <sub>2</sub>	pump oil	2.96	s	NCH <sub>3</sub>	dimethylacetamide	6.10	m	CH(3,4)	pyrrole
1.28	m	CH <sub>2</sub>	<i>n</i> -hexane	3.28	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.44	dd	CH(3,4)	furan
1.29	m	CH <sub>2</sub>	<i>n</i> -pentane	3.28	s <sup>o</sup>	CH <sub>3</sub>	methanol	6.73	s	ArH	BHA
1.33	sept, 7, 3	CH <sub>2</sub>	propane	3.29	s	OCH <sub>3</sub>	diglyme	6.75	m	CH(2,5)	pyrrole
1.39	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.38	s	CH <sub>2</sub>	dimethyl malonate	6.97	s	ArH	BHT
1.40	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.42	q, 7	CH <sub>2</sub>	diethyl ether	7.01	s	CH(4,5)	imidazole
1.44	s	CH <sub>2</sub>	cyclohexane	3.45	m	CH <sub>2</sub>	diglyme	7.10-7.30	m	CH(2,4,6)	toluene
1.61	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.45	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.10-7.30	m	CH(3,5)	toluene
1.67-1.72	m	CH <sub>2</sub> (4)	cyclohexanone	3.51	s	CH <sub>2</sub>	18-crown-6	7.33	m	CH(3,5)	pyridine
1.70	dt, 6, 4, 1, 5	CH <sub>3</sub>	propylene	3.51	m <sup>7</sup>	CH <sub>2</sub>	ethylene glycol	7.37	s	CH	benzene
1.79-1.84	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.53	m	CH <sub>2</sub>	diglyme	7.52	dd	CH(2,5)	furan
1.80	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.54	q, 7 <sup>o</sup>	CH <sub>2</sub>	ethanol	7.57	s	CH(2)	imidazole
1.94	p	CHD <sub>2</sub>	CD <sub>3</sub> CN residual	3.60	s	CH <sub>2</sub>	1,4-dioxane	7.57-7.61	m	CH(3,5)	benzaldehyde
1.96	s	CH <sub>3</sub>	acetic acid	3.64	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.58	s	CH	chloroform
1.96	s	CH <sub>3</sub>	acetonitrile	3.68	s	CH <sub>3</sub>	dimethyl malonate	7.67-7.71	m	CH(4)	benzaldehyde
1.97	s	CH <sub>3</sub> CO	dimethylacetamide	3.72	s	ArOCH <sub>3</sub>	BHA	7.73	m	CH(4)	pyridine
1.97	s	CH <sub>3</sub> CO	ethyl acetate	3.72	s	CH <sub>3</sub>	dimethyl carbonate	7.89-7.91	m	CH(2,6)	benzaldehyde
2.02	s	CH <sub>3</sub>	allyl acetate	3.81	s	CH <sub>2</sub>	1,2-dichloroethane	7.92	s	CH	dimethylformamide
2.06	s	CH <sub>3</sub> CO	ethyl methyl ketone	3.87	sept, 6	CH	2-propanol	8.57	m	CH(2,6)	pyridine
2.08	s	CH <sub>3</sub>	acetone	4.06	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	9.27	br t	NH	pyrrole
2.13	s	OH	water	4.31	s	CH <sub>3</sub>	nitromethane	10.01	s	HCO	benzaldehyde
2.16	s <sup>o</sup>	OH	methanol	4.53	ddd	CH <sub>2</sub>	allyl acetate				

**Table S20.** CD<sub>3</sub>CN (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.61	CH <sub>4</sub>	methane	30.68	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	65.55	CH <sub>2</sub>	allyl acetate	130.07	CH(3,5)	benzaldehyde
1.32 (sept)	CD <sub>3</sub>	CD <sub>3</sub> CN signal	30.86	CH <sub>2</sub>	pump oil	66.32	CH <sub>2</sub>	diethyl ether	130.42	CH(2,6)	benzaldehyde
1.79	CH <sub>3</sub>	acetonitrile	30.91	CH <sub>3</sub>	acetone	67.72	CH <sub>2</sub>	1,4-dioxane	132.61	C	hexamethylbenzene
2.07	CH <sub>3</sub>	hexamethyldisiloxane	31.32	CH <sub>3</sub>	dimethylformamide	68.33	CH <sub>2</sub> (2,5)	tetrahydrofuran	133.20	CHCH <sub>2</sub>	diallyl carbonate
6.99	CH <sub>3</sub>	ethane	31.50	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	68.74	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	133.83	CHCH <sub>2</sub>	allyl acetate
8.14	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	32.36	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	69.09	CH <sub>2</sub>	diallyl carbonate	134.78	CH	propylene
12.38	CH <sub>3</sub>	triethylamine	34.89	CH <sub>2</sub> (3)	<i>n</i> -pentane	70.99	CH <sub>2</sub>	diglyme	135.40	CH(4)	benzaldehyde
14.37	CH <sub>3</sub>	<i>n</i> -pentane	35.05	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	71.22	CH <sub>2</sub>	18-crown-6	136.33	CH(2)	imidazole
14.43	CH <sub>3</sub>	<i>n</i> -hexane	35.17	NCH <sub>3</sub>	dimethylacetamide	72.47	CH <sub>2</sub>	1,2-dimethoxyethane	136.89	CH(4)	pyridine
14.54	CH <sub>3</sub>	ethyl acetate	35.48	(CH <sub>3</sub> ) <sub>2</sub> C	BHA	72.63	CH <sub>2</sub>	diglyme	137.62	C(1)	benzaldehyde
15.63	CH <sub>3</sub>	diethyl ether	36.57	CH <sub>3</sub>	dimethylformamide	79.17	CH	chloroform	138.13	C(2,6)	BHT
16.73	CH <sub>3</sub>	propane	37.09	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	96.68	CCl <sub>4</sub>	carbon tetrachloride	138.90	C(1)	toluene
16.91	CH <sub>2</sub>	propane	37.10 (d)	CH <sub>3</sub>	HMPA <sup>11</sup>	108.31	CH(3,4)	pyrrole	140.20	C(4)	BHA
16.94	CH <sub>3</sub>	hexamethylbenzene	38.26	NCH <sub>3</sub>	dimethylacetamide	110.49	CH(3,4)	furan	143.74	CH(2,5)	furan
18.80	CH <sub>3</sub>	ethanol	41.31	CH <sub>3</sub>	dimethyl sulfoxide	111.35	CH(3,5)	BHA	148.39	C(2,6)	BHA
19.48	CH <sub>3</sub>	propylene	41.77	CH <sub>2</sub>	dimethyl malonate	116.12	CH <sub>2</sub>	propylene	150.76	CH(2,6)	pyridine
20.73	CH <sub>3</sub>	acetic acid	42.44	CH <sub>2</sub> (2,6)	cyclohexanone	118.06	CHCH <sub>2</sub>	allyl acetate	152.42	C(1)	BHT
21.02	CH <sub>3</sub>	allyl acetate	45.54	CH <sub>2</sub>	1,2-dichloroethane	118.26	CN	CD <sub>3</sub> CN signal	154.02	C(1)	BHA
21.16	CH <sub>3</sub> CO	ethyl acetate	47.10	CH <sub>2</sub>	triethylamine	118.26	CN	acetonitrile	155.66	CO	diallyl carbonate
21.23	CH <sub>3</sub> Ar	BHT	47.57	CH <sub>2</sub> (2,5)	pyrrolidine	118.47	CH(2,5)	pyrrole	157.26	CO	dimethyl carbonate
21.50	CH <sub>3</sub>	toluene	49.90	CH <sub>3</sub>	methanol	118.86	CHCH <sub>2</sub>	diallyl carbonate	163.31	CH	dimethylformamide
21.76	CH <sub>3</sub>	dimethylacetamide	52.95	CH <sub>3</sub>	dimethyl malonate	122.78	CH(4,5)	imidazole	168.07	CO <sub>2</sub>	dimethyl malonate
23.08	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	55.32	CH <sub>2</sub>	dichloromethane	123.69	CH <sub>2</sub>	ethylene	171.31	CO	dimethylacetamide
23.40	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	55.39	CH <sub>3</sub>	dimethyl carbonate	125.89	CO <sub>2</sub>	carbon dioxide	171.32	CO	allyl acetate
25.55	CH <sub>3</sub>	2-propanol	55.94	CH <sub>3</sub> O	BHA	126.28	CH(4)	toluene	171.68	CO	ethyl acetate
25.62	CH <sub>2</sub> (4)	cyclohexanone	57.96	CH <sub>2</sub>	ethanol	126.38	CH(3,5)	BHT	173.21	CO	acetic acid
26.27	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.89	CH <sub>3</sub>	1,2-dimethoxyethane	127.76	CH(3,5)	pyridine	193.60	CS <sub>2</sub>	carbon disulfide
26.34	CH <sub>2</sub> (3,4)	pyrrolidine	58.90	CH <sub>3</sub>	diglyme	129.23	CH(3,5)	toluene	193.64	HCO	benzaldehyde
27.63	CH <sub>2</sub>	cyclohexane	60.98	CH <sub>2</sub>	ethyl acetate	129.32	CH	benzene	207.43	CO	acetone
27.80	CH <sub>2</sub> (3,5)	cyclohexanone	63.66	CH <sub>3</sub>	nitromethane	129.61	C(4)	BHT	209.88	CO	ethyl methyl ketone
29.60	CH <sub>3</sub> CO	ethyl methyl ketone	64.22	CH <sub>2</sub>	ethylene glycol	129.94	CH(2,6)	toluene	211.99	CO	cyclohexanone
30.55	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	64.30	CH	2-propanol						

**Table S21.** TFE- $d_3$  ( $^1\text{H}$  NMR data by chemical shift in ppm)

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.08	s	CH <sub>3</sub>	hexamethyldisiloxane	2.20	s	OH	<i>tert</i> -butyl alcohol	4.53	s	H <sub>2</sub>	hydrogen
0.16	s	CH <sub>3</sub>	silicone grease	2.24	s	ArCH <sub>3</sub>	BHT	4.58	ddd	CH <sub>2</sub>	allyl acetate
0.18	s	CH <sub>4</sub>	methane	2.24	s	CH <sub>3</sub>	hexamethylbenzene	4.62	ddd	CH <sub>2</sub>	diallyl carbonate
0.85	s	CH <sub>3</sub>	ethane	2.33	s	CH <sub>3</sub>	toluene	4.93	dm, 10	CH <sub>2</sub> (1)	propylene
0.88-0.94	m	CH <sub>3</sub>	H grease <sup>δ</sup>	2.38	t	CH <sub>2</sub> (2,6)	cyclohexanone	5.02	s	OH	TFE- $d_3$ residual
0.90	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.49	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.03	dm, 17	CH <sub>2</sub> (2)	propylene
0.90	t, 7, 3	CH <sub>3</sub>	propane	2.63	s	CH <sub>3</sub>	dimethyl sulfoxide	5.24	s	CH <sub>2</sub>	dichloromethane
0.91	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.63	d, 9, 5	CH <sub>3</sub>	HMPA	5.25	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.99	m	CH <sub>3</sub>	pump oil	2.88	s	CH <sub>3</sub>	dimethylformamide	5.28	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
1.05	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.94	s	NCH <sub>3</sub>	dimethylacetamide	5.32	ddt	CHCH <sub>2</sub> (1)	allyl acetate
1.20	t, 7	CH <sub>3</sub>	diethyl ether	2.98	s	CH <sub>3</sub>	dimethylformamide	5.35	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.20	d, 6	CH <sub>3</sub>	2-propanol	3.05	s	NCH <sub>3</sub>	dimethylacetamide	5.40	s	CH <sub>2</sub>	ethylene
1.22	t, 7	CH <sub>3</sub>	ethanol	3.11	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.87	m	CH	propylene
1.26	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.12	q, 7	CH <sub>2</sub>	triethylamine	5.92	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.28	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.40	s	CH <sub>3</sub>	1,2-dimethoxyethane	5.93	ddt	CHCH <sub>2</sub>	allyl acetate
1.31	m	CH <sub>2</sub>	<i>n</i> -hexane	3.41	s	OCH <sub>3</sub>	diglyme	6.24	m	CH(3,4)	pyrrole
1.31	t, 7	CH <sub>3</sub>	triethylamine	3.41	s	CH <sub>2</sub>	dimethyl malonate	6.42	dd	CH(3,4)	uran
1.33	br s	CH <sub>2</sub>	H grease <sup>δ</sup>	3.44	s	CH <sub>3</sub>	methanol	6.84	m	CH(2,5)	pyrrole
1.33	m	CH <sub>2</sub>	<i>n</i> -pentane	3.58	q, 7	CH <sub>2</sub>	diethyl ether	6.87	s	ArH	BHA
1.33	sept, 7, 3	CH <sub>2</sub>	propane	3.61	s	CH <sub>2</sub>	1,2-dimethoxyethane	7.03	s	CH(4,5)	imidazole
1.41	br s	CH <sub>2</sub>	pump oil	3.62	m	CH <sub>2</sub>	diglyme	7.06	s	ArH	BHT
1.43	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.64	s	CH <sub>2</sub>	18-crown-6	7.10-7.30	m	CH(2,4,6)	toluene
1.44	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.66	s	OH	water	7.10-7.30	m	CH(3,5)	toluene
1.47	s	CH <sub>2</sub>	cyclohexane	3.67	m	CH <sub>2</sub>	diglyme	7.33	s	CH	chloroform
1.70	dt, 6, 4, 1, 5	CH <sub>3</sub>	propylene	3.71	s	CH <sub>2</sub>	1,2-dichloroethane	7.36	s	CH	benzene
1.75-1.78	m	CH <sub>2</sub> (4)	cyclohexanone	3.71	q, 7	CH <sub>2</sub>	ethanol	7.40	dd	CH(3,5)	pyridine
1.87-1.92	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.72	s	CH <sub>2</sub>	ethylene glycol	7.44	dd	CH(2,5)	uran
1.91	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.76	s	CH <sub>3</sub>	dimethyl malonate	7.56-7.59	m	CH(3,5)	benzaldehyde
1.93	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.76	s	CH <sub>2</sub>	1,4-dioxane	7.61	s	CH(2)	imidazole
1.95	s	CH <sub>3</sub>	acetonitrile	3.77	s	CH <sub>3</sub>	dimethyl carbonate	7.68-7.72	m	CH(4)	benzaldehyde
2.03	s	CH <sub>3</sub> CO	ethyl acetate	3.78	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.82	m	CH(4)	pyridine
2.06	s	CH <sub>3</sub>	acetic acid	3.79	s	ArOCH <sub>3</sub>	BHA	7.86	s	CH	dimethylformamide
2.07	s	CH <sub>3</sub>	allyl acetate	3.88	tq	CDH	TFE- $d_3$ residual	7.90-7.92	m	CH(2,6)	benzaldehyde
2.09	s	CH <sub>3</sub> CO	dimethylacetamide	4.05	sept, 6	CH	2-propanol	8.45	m	CH(2,6)	pyridine
2.16	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.14	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	9.88	s	HCO	benzaldehyde
2.19	s	CH <sub>3</sub>	acetone	4.28	s	CH <sub>3</sub>	nitromethane				

**Table S22.** TFE- $d_3$  ( $^{13}\text{C}\{^1\text{H}\}$  NMR data by chemical shift in ppm)

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-5.88	CH <sub>4</sub>	methane	30.96	CH <sub>3</sub>	dimethylformamide	66.69	CH	2-propanol	130.82	CH(3,5)	benzaldehyde
1.00	CH <sub>3</sub>	acetonitrile	31.01	(CH <sub>3</sub> ) <sub>2</sub> C	BHT	67.55	CH <sub>2</sub>	diethyl ether	131.78	CH(2,6)	benzaldehyde
2.09	CH <sub>3</sub>	hexamethyldisiloxane	31.07	(CH <sub>3</sub> ) <sub>2</sub> C	<i>tert</i> -butyl alcohol	67.61	CH <sub>2</sub>	allyl acetate	132.72	CHCH <sub>2</sub>	diallyl carbonate
2.87	CH <sub>3</sub>	silicone grease	31.85	CH <sub>2</sub>	pump oil	68.52	CH <sub>3</sub>	1,4-dioxane	133.33	CHCH <sub>2</sub>	allyl acetate
7.01	CH <sub>3</sub>	ethane	32.35	CO	acetone	69.53	CH <sub>2</sub> (2,5)	tetrahydrofuran	134.04	C	hexamethylbenzene
8.29	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	33.17	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	70.69	CH <sub>2</sub>	diallyl carbonate	136.00	CH	propylene
9.51	CH <sub>3</sub>	triethylamine	35.69	(CH <sub>3</sub> ) <sub>2</sub> C	BHT	70.80	CH <sub>2</sub>	18-crown-6	136.58	CH(2)	imidazole
14.36	CH <sub>3</sub>	ethyl acetate	35.76	CH <sub>2</sub> (3)	<i>n</i> -pentane	71.33	CH <sub>2</sub>	diglyme	137.17	CH(4)	benzaldehyde
14.54	CH <sub>3</sub>	<i>n</i> -pentane	36.07	(CH <sub>3</sub> ) <sub>2</sub> C	BHA	72.35	(CH <sub>3</sub> ) <sub>2</sub> C	<i>tert</i> -butyl alcohol	137.84	C(1)	benzaldehyde
14.63	CH <sub>3</sub>	<i>n</i> -hexane	36.28	NCH <sub>3</sub>	dimethylacetamide	72.87	CH <sub>2</sub>	1,2-dimethoxyethane	138.59	C(2,6)	BHT
15.33	CH <sub>3</sub>	diethyl ether	37.21 (d)	CH <sub>3</sub>	HMPA	73.05	CH <sub>2</sub>	diglyme	139.62	CH(4)	pyridine
16.93	CH <sub>3</sub>	propane	37.76	CH <sub>3</sub>	dimethylformamide	78.83	CH	chloroform	139.92	C(1)	toluene
17.04	CH <sub>3</sub>	hexamethylbenzene	38.23	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	97.74	CCl <sub>4</sub>	carbon tetrachloride	140.23	C(4)	BHA
17.46	CH <sub>2</sub>	propane	39.06	NCH <sub>3</sub>	dimethylacetamide	108.85	CH(3,4)	pyrrole	144.22	CH(2,5)	uran
18.11	CH <sub>2</sub>	ethanol	40.06	CH <sub>3</sub>	dimethyl sulfoxide	111.06	CH(3,4)	uran	149.76	CH(2,6)	pyridine
19.63	CH <sub>3</sub>	propylene	42.13	CH <sub>2</sub>	dimethyl malonate	112.90	CH(3,5)	BHA	150.52	C(2,6)	BHA
20.91	CH <sub>3</sub>	acetic acid	43.16	CH <sub>2</sub> (2,6)	cyclohexanone	116.38	CH <sub>2</sub>	propylene	153.46	C(1)	BHT
21.10	CH <sub>3</sub>	allyl acetate	45.28	CH <sub>2</sub>	1,2-dichloroethane	118.95	CN	acetonitrile	153.74	C(1)	BHA
21.18	CH <sub>3</sub> CO	ethyl acetate	47.43	CH <sub>2</sub> (2,5)	pyrrolidine	119.39	CHCH <sub>2</sub>	allyl acetate	157.39	CO	diallyl carbonate
21.34	CH <sub>3</sub> Ar	BHT	48.45	CH <sub>2</sub>	triethylamine	119.61	CH(2,5)	pyrrole	159.04	CO	dimethyl carbonate
21.40	CH <sub>3</sub>	dimethylacetamide	50.67	CH <sub>3</sub>	methanol	120.15	CHCH <sub>2</sub>	diallyl carbonate	166.01	CH	dimethylformamide
21.62	CH <sub>3</sub>	toluene	54.00	CH <sub>3</sub>	dimethyl malonate	122.93	CH(4,5)	imidazole	170.88	CO <sub>2</sub>	dimethyl malonate
23.75	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	54.46	CH <sub>2</sub>	dichloromethane	124.08	CH <sub>2</sub>	ethylene	175.55	CO	ethyl acetate
24.06	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	56.17	CH <sub>3</sub>	dimethyl carbonate	126.27	CH(3,5)	pyridine	175.74	CO	dimethylacetamide
25.21	CH <sub>3</sub>	2-propanol	57.55	CH <sub>3</sub> O	BHA	126.28 (q)	CF <sub>3</sub>	TFE- $d_3$ signal	175.98	CO	allyl acetate
25.73	CH <sub>2</sub> (3,4)	pyrrolidine	59.40	CH <sub>3</sub>	diglyme	126.82	CH(4)	toluene	177.96	CO	acetic acid
26.00	CH <sub>2</sub> (4)	cyclohexanone	59.52	CH <sub>3</sub>	1,2-dimethoxyethane	126.92	CO <sub>2</sub>	carbon dioxide	196.26	CS <sub>2</sub>	carbon disulfide
26.69	CH <sub>2</sub> (3,4)	tetrahydrofuran	59.68	CH <sub>2</sub>	ethanol	127.11	CH(3,5)	BHT	197.63	HCO	benzaldehyde
28.34	CH <sub>2</sub>	cyclohexane	61.5 (qp)	CD <sub>2</sub>	TFE- $d_3$ signal	129.79	CH(3,5)	toluene	214.98	CH <sub>3</sub>	acetone
28.56	CH <sub>2</sub> (3,5)	cyclohexanone	62.70	CH <sub>2</sub>	ethyl acetate	129.84	CH	benzene	218.31	CO	ethyl methyl ketone
29.64	CH <sub>3</sub> CO	ethyl methyl ketone	63.17	CH <sub>3</sub>	nitromethane	130.58	CH(2,6)	toluene	221.30	CO	cyclohexanone
30.80	(CH <sub>3</sub> ) <sub>2</sub> C	BHA	64.87	CH <sub>2</sub>	ethylene glycol	130.62	C(4)	BHT			



**Table S23. CD<sub>3</sub>OD (<sup>1</sup>H NMR data by chemical shift in ppm)**

shift	mult	proton	impurity	shift	mult	proton	impurity	shift	mult	proton	impurity
0.07	s	CH <sub>3</sub>	hexamethyldisiloxane	2.19	s	CH <sub>3</sub>	hexamethylbenzene	4.56	s	H <sub>2</sub>	hydrogen
0.10	s	CH <sub>3</sub>	silicone grease	2.21	s	ArCH <sub>3</sub>	BHT	4.61	ddd	CH <sub>2</sub>	diallyl carbonate
0.20	s	CH <sub>4</sub>	methane	2.32	s	CH <sub>3</sub>	toluene	4.85	s	OH <sup>d</sup>	BHA
0.85	s	CH <sub>3</sub>	ethane	2.34	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.87	s	OH	water
0.86-0.91	m	CH <sub>3</sub>	pump oil	2.50	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	4.91	dm, 10	CH <sub>2</sub> (1)	propylene
0.86-0.93	m	CH <sub>3</sub>	H grease <sup>8</sup>	2.58	q, 7	CH <sub>2</sub>	triethylamine	5.01	dm, 17	CH <sub>2</sub> (2)	propylene
0.90	t, 7	CH <sub>3</sub>	<i>n</i> -hexane	2.64	d, 9,5	CH <sub>3</sub>	HMPA	5.21	ddt	CHCH <sub>2</sub> (2)	allyl acetate
0.90	t, 7	CH <sub>3</sub>	<i>n</i> -pentane	2.65	s	CH <sub>3</sub>	dimethyl sulfoxide	5.25	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
0.91	t, 7,3	CH <sub>3</sub>	propane	2.80	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.30	ddt	CHCH <sub>2</sub> (1)	allyl acetate
1.01	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	2.86	s	CH <sub>3</sub>	dimethylformamide	5.34	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.05	t, 7	CH <sub>3</sub>	triethylamine	2.92	s	NCH <sub>3</sub>	dimethylacetamide	5.39	s	CH <sub>2</sub>	ethylene
1.15	d, 6	CH <sub>3</sub>	2-propanol	2.99	s	CH <sub>3</sub>	dimethylformamide	5.49	s	CH <sub>2</sub>	dichloromethane
1.18	t, 7	CH <sub>3</sub>	diethyl ether	3.31	p	CD <sub>2</sub> H	CD <sub>3</sub> OD residual	5.82	m	CH	propylene
1.19	t, 7	CH <sub>3</sub>	ethanol	3.31	s	NCH <sub>3</sub>	dimethylacetamide	5.94	ddt	CHCH <sub>2</sub>	allyl acetate
1.24	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.34	s	CH <sub>3</sub>	methanol	5.94	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.29	br s	CH <sub>2</sub>	H grease <sup>8</sup>	3.35	s	OCH <sub>3</sub>	diglyme	6.08	m	CH(3,4)	pyrrole
1.29	m	CH <sub>2</sub>	<i>n</i> -hexane	3.35	s	CH <sub>3</sub>	1,2-dimethoxyethane	6.40	dd	CH(3,4)	furan
1.29	br s	CH <sub>2</sub>	<i>n</i> -pentane	3.44	s	CH <sub>2</sub>	dimethyl malonate	6.71	s	ArH	BHA
1.29	1.29	CH <sub>2</sub>	pump oil	3.49	q, 7	CH <sub>2</sub>	diethyl ether	6.72	m	CH(2,5)	pyrrole
1.34	sept, 7,3	CH <sub>2</sub>	propane	3.52	s	CH <sub>2</sub>	1,2-dimethoxyethane	6.92	s	ArH	BHT
1.40	s	CH <sub>3</sub>	<i>tert</i> -butyl alcohol	3.58	m	CH <sub>2</sub>	diglyme	7.05	s	CH(4,5)	imidazole
1.40	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHT	3.59	s	CH <sub>2</sub>	ethylene glycol	7.16	m	CH(2,4,6)	toluene
1.41	s	ArC(CH <sub>3</sub> ) <sub>3</sub>	BHA	3.60	q, 7	CH <sub>2</sub>	ethanol	7.16	m	CH(3,5)	toluene
1.45	s	CH <sub>2</sub>	cyclohexane	3.61	m	CH <sub>2</sub>	diglyme	7.33	s	CH	benzene
1.70	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.64	s	CH <sub>2</sub>	18-crown-6	7.44	m	CH(3,5)	pyridine
1.72	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.66	s	CH <sub>2</sub>	1,4-dioxane	7.49	dd	CH(2,5)	furan
1.74-1.76	m	CH <sub>2</sub> (4)	cyclohexanone	3.71	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.56-7.60	m	CH(3,5)	benzaldehyde
1.85-1.87	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.72	s	ArOCH <sub>3</sub>	BHA	7.66-7.70	m	CH(4)	benzaldehyde
1.87	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.72	s	CH <sub>3</sub>	dimethyl malonate	7.67	s	CH(2)	imidazole
1.99	s	CH <sub>3</sub>	acetic acid	3.74	s	CH <sub>3</sub>	dimethyl carbonate	7.85	m	CH(4)	pyridine
2.01	s	CH <sub>3</sub> CO	ethyl acetate	3.78	s	CH <sub>2</sub>	1,2-dichloroethane	7.90	s	CH	chloroform
2.03	s	CH <sub>3</sub>	acetonitrile	3.92	sept, 6	CH	2-propanol	7.90-7.93	m	CH(2,6)	benzaldehyde
2.05	s	CH <sub>3</sub>	allyl acetate	4.09	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	7.97	s	CH	dimethylformamide
2.07	s	CH <sub>3</sub> CO	dimethylacetamide	4.34	s	CH <sub>3</sub>	nitromethane	8.53	m	CH(2,6)	pyridine
2.12	s	CH <sub>3</sub> CO	ethyl methyl ketone	4.56	ddd	CH <sub>2</sub>	allyl acetate	10.00	s	HCO	benzaldehyde
2.15	s	CH <sub>3</sub>	acetone								

**Table S24. CD<sub>3</sub>OD (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)**

shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity	shift	carbon	impurity
-4.90	CH <sub>4</sub>	methane	30.82	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	64.71	CH	2-propanol	130.12	CH(3,5)	benzaldehyde
0.85	CH <sub>3</sub>	acetonitrile	30.91	(CH <sub>3</sub> ) <sub>3</sub> C	<i>tert</i> -butyl alcohol	66.14	CH <sub>2</sub>	allyl acetate	130.64	CH(2,6)	benzaldehyde
1.99	CH <sub>3</sub>	hexamethyldisiloxane	31.15	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	66.88	CH <sub>2</sub>	diethyl ether	132.53	C	hexamethylbenzene
2.10	CH <sub>3</sub>	silicone grease	31.35	CH <sub>2</sub>	pump oil	68.11	CH <sub>2</sub>	1,4-dioxane	133.25	CHCH <sub>2</sub>	diallyl carbonate
6.98	CH <sub>3</sub>	ethane	31.61	CH <sub>3</sub>	dimethylformamide	68.83	CH <sub>2</sub> (2,5)	tetrahydrofuran	133.71	CHCH <sub>2</sub>	allyl acetate
8.09	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	32.73	CH <sub>2</sub> (3,4)	<i>n</i> -hexane	69.35	CH <sub>2</sub>	diallyl carbonate	134.61	CH	propylene
11.09	CH <sub>3</sub>	triethylamine	35.30	CH <sub>2</sub> (3)	<i>n</i> -pentane	69.40	(CH <sub>3</sub> ) <sub>2</sub> C	<i>tert</i> -butyl alcohol	135.60	CH(4)	benzaldehyde
14.39	CH <sub>3</sub>	<i>n</i> -pentane	35.36	(CH <sub>3</sub> ) <sub>3</sub> C	BHT	71.33	CH <sub>2</sub>	diglyme	136.31	CH(2)	imidazole
14.45	CH <sub>3</sub>	<i>n</i> -hexane	35.50	NCH <sub>3</sub>	dimethylacetamide	71.47	CH <sub>2</sub>	18-crown-6	137.96	C(1)	benzaldehyde
14.49	CH <sub>3</sub>	ethyl acetate	35.83	(CH <sub>3</sub> ) <sub>3</sub> C	BHA	72.72	CH <sub>2</sub>	1,2-dimethoxyethane	138.35	CH(4)	pyridine
15.46	CH <sub>3</sub>	diethyl ether	36.89	CH <sub>3</sub>	dimethylformamide	72.92	CH <sub>2</sub>	diglyme	138.85	C(1)	toluene
16.80	CH <sub>3</sub>	propane	37.00 (d)	CH <sub>3</sub>	HMPA <sup>1</sup>	79.44	CH	chloroform	139.09	C(2,6)	BHT
16.90	CH <sub>3</sub>	hexamethylbenzene	37.34	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	97.21	CCl <sub>4</sub>	carbon tetrachloride	141.36	C(4)	BHA
17.19	CH <sub>2</sub>	propane	38.43	NCH <sub>3</sub>	dimethylacetamide	108.11	CH(3,4)	pyrrole	143.68	CH(2,5)	furan
18.40	CH <sub>3</sub>	ethanol	40.45	CH <sub>3</sub>	dimethyl sulfoxide	110.33	CH(3,4)	furan	149.04	C(2,6)	BHA
19.50	CH <sub>3</sub>	propylene	41.60	CH <sub>2</sub>	dimethyl malonate	111.30	CH(3,5)	BHA	150.07	CH(2,6)	pyridine
20.56	CH <sub>3</sub>	acetic acid	42.61	CH <sub>2</sub> (2,6)	cyclohexanone	116.04	CH <sub>2</sub>	propylene	152.85	C(1)	BHT
20.71	CH <sub>3</sub>	allyl acetate	45.11	CH <sub>2</sub>	1,2-dichloroethane	118.06	CN	acetonitrile	154.34	C(1)	BHA
20.88	CH <sub>3</sub> CO	ethyl acetate	46.96	CH <sub>2</sub>	triethylamine	118.22	CHCH <sub>2</sub>	allyl acetate	156.28	CO	diallyl carbonate
21.32	CH <sub>3</sub>	dimethylacetamide	47.23	CH <sub>2</sub> (2,5)	pyrrolidine	118.28	CH(2,5)	pyrrole	157.91	CO	dimethyl carbonate
21.38	CH <sub>3</sub> Ar	BHT	49.00 (sept)	CD <sub>3</sub>	CD <sub>3</sub> OD signal	118.74	CHCH <sub>2</sub>	diallyl carbonate	164.73	CH	dimethylformamide
21.50	CH <sub>3</sub>	toluene	49.86	CH <sub>3</sub>	methanol	122.60	CH(4,5)	imidazole	168.70	CO <sub>2</sub>	dimethyl malonate
23.38	CH <sub>2</sub> (2,4)	<i>n</i> -pentane	52.83	CH <sub>3</sub>	dimethyl malonate	123.46	CH <sub>2</sub>	ethylene	172.41	CO	allyl acetate
23.68	CH <sub>2</sub> (2,5)	<i>n</i> -hexane	54.78	CH <sub>2</sub>	dichloromethane	125.53	CH(3,5)	pyridine	172.89	CO	ethyl acetate
25.27	CH <sub>3</sub>	2-propanol	55.25	CH <sub>3</sub>	dimethyl carbonate	126.11	CH(3,5)	BHT	173.32	CO	dimethylacetamide
25.86	CH <sub>2</sub> (4)	cyclohexanone	55.96	CH <sub>3</sub> O	BHA	126.29	CH(4)	toluene	175.11	CO	acetic acid
26.29	CH <sub>2</sub> (3,4)	pyrrolidine	58.26	CH <sub>2</sub>	ethanol	126.31	CO <sub>2</sub>	carbon dioxide	193.82	CS <sub>2</sub>	carbon disulfide
26.48	CH <sub>2</sub> (3,4)	tetrahydrofuran	59.06	CH <sub>3</sub>	diglyme	129.20	CH(3,5)	toluene	194.11	HCO	benzaldehyde
27.96	CH <sub>2</sub>	cyclohexane	59.06	CH <sub>3</sub>	1,2-dimethoxyethane	129.34	CH	benzene	209.67	CO	acetone
28.16	CH <sub>2</sub> (3,5)	cyclohexanone	61.50	CH <sub>2</sub>	ethyl acetate	129.49	CH	BHT	212.16	CO	ethyl methyl ketone
29.39	CH <sub>3</sub> CO	ethyl methyl ketone	63.08	CH <sub>3</sub>	nitromethane	129.91	CH(2,6)	toluene	214.69	CO	cyclohexanone
30.67	CH <sub>3</sub>	acetone	64.30	CH <sub>2</sub>	ethylene glycol						

**Table S25. D<sub>2</sub>O (<sup>1</sup>H NMR data by chemical shift in ppm)**

<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>	<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>	<i>shift</i>	<i>mult</i>	<i>proton</i>	<i>impurity</i>
0.18	s	CH <sub>4</sub>	methane	2.71	s	CH <sub>3</sub>	dimethyl sulfoxide	4.69	ddd	CH <sub>2</sub>	diallyl carbonate
0.28	s	CH <sub>3</sub>	hexamethyldisiloxane	2.85	s	CH <sub>3</sub>	dimethylformamide	4.79	s	HOD	D <sub>2</sub> O residual
0.82	s	CH <sub>3</sub>	ethane	2.90	s	NCH <sub>3</sub>	dimethylacetamide	4.95	dm, 10	CH <sub>2</sub> (1)	propylene
0.88	t, 7,3	CH <sub>3</sub>	propane	3.01	s	CH <sub>3</sub>	dimethylformamide	5.06	dm, 17	CH <sub>2</sub> (2)	propylene
0.99	t, 7	CH <sub>3</sub>	triethylamine	3.06	s	NCH <sub>3</sub>	dimethylacetamide	5.30	ddt	CHCH <sub>2</sub> (2)	allyl acetate
1.17	t, 7	CH <sub>3</sub>	diethyl ether	3.07	m	CH <sub>2</sub> (2,5)	pyrrolidine	5.32	ddt	CHCH <sub>2</sub> (2)	diallyl carbonate
1.17	t, 7	CH <sub>3</sub>	ethanol	3.18	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	5.37	ddt	CHCH <sub>2</sub> (1)	allyl acetate
1.17	d, 6	CH <sub>3</sub>	2-propanol	3.34	s	CH <sub>3</sub>	methanol	5.40	ddt	CHCH <sub>2</sub> (1)	diallyl carbonate
1.24	s	CH <sub>3</sub>	tert-butyl alcohol	3.37	s	OCH <sub>3</sub>	diglyme	5.44	s	CH <sub>2</sub>	ethylene
1.24	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	3.37	s	CH <sub>3</sub>	1,2-dimethoxyethane	5.90	m	CH	propylene
1.26	t, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	3.56	q, 7	CH <sub>2</sub>	diethyl ether	5.99	ddt	CHCH <sub>2</sub>	allyl acetate
1.30	sept, 7,3	CH <sub>2</sub>	propane	3.60	s	CH <sub>2</sub>	1,2-dimethoxyethane	5.99	ddt	CHCH <sub>2</sub>	diallyl carbonate
1.70	dt, 6,4, 1,5	CH <sub>3</sub>	propylene	3.60	s	CH <sub>2</sub>	dimethyl malonate	6.26	m	CH(3,4)	pyrrole
1.70-1.75	m	CH <sub>2</sub> (4)	cyclohexanone	3.61	m	CH <sub>2</sub>	diglyme	6.51	dd	CH(3,4)	furan
1.85-1.90	m	CH <sub>2</sub> (3,5)	cyclohexanone	3.65	q, 7	CH <sub>2</sub>	ethanol	6.93	m	CH(2,5)	pyrrole
1.87	m	CH <sub>2</sub> (3,4)	pyrrolidine	3.65	s	CH <sub>2</sub>	ethylene glycol	7.14	s	CH(4,5)	imidazole
1.88	m	CH <sub>2</sub> (3,4)	tetrahydrofuran	3.67	m	CH <sub>2</sub>	diglyme	7.45	m	CH(3,5)	pyridine
2.06	s	CH <sub>3</sub>	acetonitrile	3.69	s	CH <sub>3</sub>	dimethyl carbonate	7.57	dd	CH(2,5)	furan
2.07	s	CH <sub>2</sub> CO	ethyl acetate	3.74	m	CH <sub>2</sub> (2,5)	tetrahydrofuran	7.57-7.66	m	CH(3,5)	benzaldehyde
2.08	s	CH <sub>3</sub>	acetic acid	3.75	s	CH <sub>3</sub>	1,4-dioxane	7.76-7.80	m	CH(4)	benzaldehyde
2.08	s	CH <sub>2</sub> CO	dimethylacetamide	3.78	s	CH <sub>3</sub>	dimethyl malonate	7.78	s	CH(2)	imidazole
2.13	s	CH <sub>3</sub>	allyl acetate	3.80	s	CH <sub>2</sub>	18-crown-6	7.87	m	CH(4)	pyridine
2.19	s	CH <sub>2</sub> CO	ethyl methyl ketone	4.02	sept, 6	CH	2-propanol	7.92	s	CH	dimethylformamide
2.22	s	CH <sub>3</sub>	acetone	4.14	q, 7	CH <sub>2</sub> CH <sub>3</sub>	ethyl acetate	7.97-7.99	m	CH(2,6)	benzaldehyde
2.40	t	CH <sub>2</sub> (2,6)	cyclohexanone	4.40	s	CH <sub>3</sub>	nitromethane	8.52	m	CH(2,6)	pyridine
2.57	q, 7	CH <sub>2</sub>	triethylamine	4.62	ddd	CH <sub>2</sub>	allyl acetate	9.96	s	HCO	benzaldehyde
2.61	d, 9,5	CH <sub>3</sub>	HMPA								

**Table S26. D<sub>2</sub>O (<sup>13</sup>C{<sup>1</sup>H} NMR data by chemical shift in ppm)**

<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>	<i>shift</i>	<i>carbon</i>	<i>impurity</i>
1.47	CH <sub>3</sub>	acetonitrile	36.46 (d)	CH <sub>3</sub>	HMPA'	67.19	CH <sub>2</sub>	1,4-dioxane	132.76	CHCH <sub>2</sub>	diallyl carbonate
2.31	CH <sub>3</sub>	hexamethyldisiloxane	37.27	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	68.68	CH <sub>2</sub> (2,5)	tetrahydrofuran	134.70	CH (4)	benzaldehyde
7.87	CH <sub>2</sub> CH <sub>3</sub>	ethyl methyl ketone	37.54	CH <sub>3</sub>	dimethylformamide	68.81	CH <sub>2</sub>	diallyl carbonate	136.11	C(1)	benzaldehyde
9.07	CH <sub>3</sub>	triethylamine	38.76	NCH <sub>3</sub>	dimethylacetamide	70.05	CH <sub>2</sub>	diglyme	136.65	CH(2)	imidazole
13.92	CH <sub>3</sub>	ethyl acetate	39.39	CH <sub>3</sub>	dimethyl sulfoxide	70.14	CH <sub>2</sub>	18-crown-6	138.27	CH(4)	pyridine
14.77	CH <sub>3</sub>	diethyl ether	42.02	CH <sub>2</sub> (2,6)	cyclohexanone	70.36	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	143.57	CH(2,5)	furan
17.47	CH <sub>3</sub>	ethanol	42.13	CH <sub>2</sub>	dimethyl malonate	71.49	CH <sub>2</sub>	1,2-dimethoxyethane	149.18	CH(2,6)	pyridine
21.00	CH <sub>3</sub>	allyl acetate	46.83	CH <sub>2</sub> (2,5)	pyrrolidine	71.63	CH <sub>2</sub>	diglyme	157.78	CO	diallyl carbonate
21.03	CH <sub>3</sub>	acetic acid	47.19	CH <sub>2</sub>	triethylamine	96.73	CCl <sub>4</sub>	carbon tetrachloride	163.96	CO	dimethyl carbonate
21.09	CH <sub>3</sub>	dimethylacetamide	49.50 <sup>12</sup>	CH <sub>3</sub>	methanol	107.83	CH(3,4)	pyrrole	165.53	CH	dimethylformamide
21.15	CH <sub>2</sub> CO	ethyl acetate	53.65	CH <sub>3</sub>	dimethyl malonate	110.23	CH(3,4)	furan	170.12	CO <sub>2</sub>	dimethyl malonate
24.38	CH <sub>3</sub>	2-propanol	55.81	CH <sub>3</sub>	dimethyl carbonate	118.75	CHCH <sub>2</sub>	diallyl carbonate	174.57	CO	dimethylacetamide
24.77	CH <sub>2</sub> (4)	cyclohexanone	58.05	CH <sub>2</sub>	ethanol	119.03	CHCH <sub>2</sub>	allyl acetate	174.78	CO	allyl acetate
25.67	CH <sub>2</sub> (3,4)	tetrahydrofuran	58.67	CH <sub>3</sub>	diglyme	119.06	CH(2,5)	pyrrole	175.26	CO	ethyl acetate
25.86	CH <sub>2</sub> (3,4)	pyrrolidine	58.67	CH <sub>3</sub>	1,2-dimethoxyethane	119.68	CN	acetonitrile	177.21	CO	acetic acid
27.50	CH <sub>2</sub> (3,5)	cyclohexanone	62.32	CH <sub>2</sub>	ethyl acetate	122.43	CH(4,5)	imidazole	191.67	HCO	benzaldehyde
29.49	CH <sub>2</sub> CO	ethyl methyl ketone	63.17	CH <sub>2</sub>	ethylene glycol	125.12	CH(3,5)	pyridine	197.25	CS <sub>2</sub>	carbon disulfide
30.29	(CH <sub>3</sub> ) <sub>3</sub> C	tert-butyl alcohol	63.22	CH <sub>3</sub>	nitromethane	129.48	CH(3,5)	benzaldehyde	215.94	CO	acetone
30.89	CH <sub>3</sub>	acetone	64.88	CH	2-propanol	130.09	CH(2,6)	benzaldehyde	218.43	CO	ethyl methyl ketone
32.03	CH <sub>3</sub>	dimethylformamide	66.42	CH <sub>2</sub>	diethyl ether	132.48	CHCH <sub>2</sub>	allyl acetate	221.22	CO	cyclohexanone
35.03	NCH <sub>3</sub>	dimethylacetamide	66.52	CH <sub>2</sub>	allyl acetate						

## **References**

- (1) Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. *J. Org. Chem.* **1997**, *62*, 7512.
- (2) Except for the compounds in solutions 8–10, as well as the gas samples, hexamethylbenzene, and the corrected values (*vide supra*), all data for the solvents  $\text{CDCl}_3$ ,  $\text{C}_6\text{D}_6$ ,  $(\text{CD}_3)_2\text{CO}$ ,  $(\text{CD}_3)_2\text{SO}$ ,  $\text{CD}_3\text{CN}$ ,  $\text{CD}_3\text{OD}$ , and  $\text{D}_2\text{O}$  were previously reported in ref 1.
- (3) A signal for HDO is also observed in  $(\text{CD}_3)_2\text{SO}$  (3.30 ppm) and  $(\text{CD}_3)_2\text{CO}$  (2.81 ppm), often seen as a 1:1:1 triplet ( $^2J_{\text{H,D}} = 1$  Hz).
- (4) Splitting pattern observed as a triplet of a non-first-order ABX pattern.
- (5) Not all OH signals were observable.
- (6) In some solvents, the coupling interaction between the  $\text{CH}_2$  and the OH protons may be observed ( $J = 5$  Hz).
- (7) In  $\text{CD}_3\text{CN}$ , the OH proton was seen as a multiplet at 2.69 ppm, as well as extra coupling to the  $\text{CH}_2$  peak.
- (8) Apiezon-brand H grease.
- (9) In some solvents, the coupling interaction between the  $\text{CH}_3$  and the OH protons may be observed ( $J = 5.5$  Hz).
- (10) Pyrrolidine was observed to react with the solvent  $(\text{CD}_3)_2\text{CO}$ .
- (11) Phosphorus coupling was observed ( $^2J_{\text{PC}} = 3$  Hz).
- (12) Internal reference; see Experimental Section in text.