

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
			CH <sub>3</sub>	t, 7					
diglyme			3.65	3.56	3.51	3.46	3.53	3.61	3.67
			CH <sub>2</sub>	m	3.57	3.47	3.38	3.34	3.45
			OCH <sub>3</sub>	s	3.39	3.28	3.24	3.11	3.29
1,2-dimethoxyethane			3.40	3.28	3.24	3.12	3.28	3.35	3.37
			CH <sub>2</sub>	s	3.55	3.46	3.43	3.33	3.45
dimethylacetamide			2.09	1.97	1.96	1.60	1.97	2.07	2.08
			NCH <sub>3</sub>	s	3.02	3.00	2.94	2.57	2.96
			NCH <sub>3</sub>	s	2.94	2.83	2.78	2.05	2.83
dimethylformamide			8.02	7.96	7.95	7.63	7.92	7.97	7.92
			CH <sub>3</sub>	s	2.96	2.94	2.89	2.36	2.89
			CH <sub>3</sub>	s	2.88	2.78	2.73	1.86	2.77
dimethyl sulfoxide			2.62	2.52	2.54	1.68	2.50	2.65	2.71
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>2</sub>	q, 7 <sup>d</sup>	3.72	3.57	3.44	3.34	3.54
			OH	s <sup>c,d</sup>	1.32	3.39	4.63	2.47	3.60
ethyl acetate			2.05	1.97	1.99	1.65	1.97	2.01	2.07
			CH <sub>2</sub> CH <sub>3</sub>	q, 7	4.12	4.05	4.03	3.89	4.06
			CH <sub>2</sub> CH <sub>3</sub>	t, 7	1.26	1.20	1.17	0.92	1.20
ethyl methyl ketone			2.14	2.07	2.07	1.58	2.06	2.12	2.19
			CH <sub>2</sub> CH <sub>3</sub>	q, 7	2.46	2.45	2.43	1.81	2.43
			CH <sub>2</sub> CH <sub>3</sub>	t, 7	1.06	0.96	0.91	0.85	0.96
ethylene glycol			3.76	3.28	3.34	3.41	3.51	3.59	3.65
"grease" <sup>f</sup>			0.86	0.87		0.92	0.86	0.88	
			CH <sub>3</sub>	m			1.36	1.27	1.29
			CH <sub>2</sub>	br s	1.26	1.29			
<i>n</i> -hexane			0.88	0.88	0.86	0.89	0.89	0.90	
			CH <sub>2</sub>	m	1.26	1.28	1.25	1.24	1.28
HMPA <sup>g</sup>			2.65	2.59	2.53	2.40	2.57	2.64	2.61
methanol			3.49	3.31	3.16	3.07	3.28	3.34	3.34
			OH	s <sup>c,h</sup>	1.09	3.12	4.01	2.16	
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>2</sub>	m	1.27	1.27	1.27	1.23	1.29
2-propanol			1.22	1.10	1.04	0.95	1.09	1.50	1.17
			CH	sep, 6	4.04	3.90	3.78	3.67	3.87
pyridine			8.62	8.58	8.58	8.53	8.57	8.53	8.52
			CH(2)	m	7.29	7.35	7.39	6.66	7.33
			CH(3)	m	7.68	7.76	7.79	6.98	7.73
			CH(4)	m	0.07	0.13	0.29	0.08	0.10
silicone grease <sup>i</sup>			1.85	1.79	1.76	1.40	1.80	1.87	1.88
tetrahydrofuran			3.76	3.63	3.60	3.57	3.64	3.71	3.74
			CH <sub>2</sub>	m	2.36	2.32	2.30	2.11	2.33
toluene			7.17	7.1–7.2	7.18	7.02	7.1–7.3	7.16	
			CH( <i>o/p</i> )	m	7.25	7.1–7.2	7.25	7.13	7.1–7.3
			CH( <i>m</i> )	m	1.03	0.96	0.93	0.96	0.96
triethylamine			2.53	2.45	2.43	2.40	2.45	2.58	2.57
			CH <sub>3</sub>	t, 7					
			CH <sub>2</sub>	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
	CH <sub>2</sub>	71.90	72.63	71.25	72.35	72.63	72.92	71.63
1,2-dimethoxyethane	CH <sub>3</sub>	59.08	58.45	58.01	58.68	58.89	59.06	58.67
	CH <sub>2</sub>	71.84	72.47	71.07	72.21	72.47	72.72	71.49
dimethylacetamide	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
	NCH <sub>3</sub>	38.13	37.92	34.42	37.03	38.26	38.43	38.76
dimethylformamide	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
	CH <sub>2</sub>	29.76	30.73	29.20	30.21	30.86	31.29	
"grease"	CH <sub>3</sub>	14.14	14.34	13.88	14.32	14.43	14.45	
<i>n</i> -hexane	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	
	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
	CH <sub>3</sub>	1.04	1.40		1.38		2.10	
silicone grease	CH <sub>2</sub>	25.62	26.15	25.14	25.72	26.27	26.48	25.67
	CH <sub>2</sub> O	67.97	68.07	67.03	67.80	68.33	68.83	68.68
tetrahydrofuran	CH <sub>3</sub>	21.46	21.46	20.99	21.10	21.50	21.50	
	C( <i>l</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.