

Table 1. ¹H NMR Data

	proton	mult	CDCl ₃	(CD ₃) ₂ CO	(CD ₃) ₂ SO	C ₆ D ₆	CD ₃ CN	CD ₃ OD	D ₂ O		
solvent residual peak			7.26	2.05	2.50	7.16	1.94	3.31	4.79		
H ₂ O			1.56	2.84 ^a	3.33 ^a	0.40	2.13	4.87			
acetic acid			2.10	1.96	1.91	1.55	1.96	1.99	2.08		
acetone			CH ₃	2.17	2.09	2.09	1.55	2.08	2.15	2.22	
acetonitrile			CH ₃	2.10	2.05	2.07	1.55	1.96	2.03	2.06	
benzene			CH	7.36	7.36	7.37	7.15	7.37	7.33		
<i>tert</i> -butyl alcohol			CH ₃	1.28	1.18	1.11	1.05	1.16	1.40	1.24	
			OH ^c			4.19	1.55	2.18			
<i>tert</i> -butyl methyl ether			CCH ₃	1.19	1.13	1.11	1.07	1.14	1.15	1.21	
			OCH ₃	3.22	3.13	3.08	3.04	3.13	3.20	3.22	
BHT ^b			ArH	6.98	6.96	6.87	7.05	6.97	6.92		
			OH ^c	5.01		6.65	4.79	5.20			
			ArCH ₃	2.27	2.22	2.18	2.24	2.22	2.21		
			ArC(CH ₃) ₃	1.43	1.41	1.36	1.38	1.39	1.40		
chloroform			CH	7.26	8.02	8.32	6.15	7.58	7.90		
cyclohexane			CH ₂	1.43	1.43	1.40	1.40	1.44	1.45		
1,2-dichloroethane			CH ₂	3.73	3.87	3.90	2.90	3.81	3.78		
dichloromethane			CH ₂	5.30	5.63	5.76	4.27	5.44	5.49		
diethyl ether			CH ₃	t, 7	1.21	1.11	1.09	1.11	1.12	1.18	1.17
			CH ₂	q, 7	3.48	3.41	3.38	3.26	3.42	3.49	3.56
diglyme			CH ₂	m	3.65	3.56	3.51	3.46	3.53	3.61	3.67
			CH ₂	m	3.57	3.47	3.38	3.34	3.45	3.58	3.61
1,2-dimethoxyethane			OCH ₃	s	3.39	3.28	3.24	3.11	3.29	3.35	3.37
			CH ₃	s	3.40	3.28	3.24	3.12	3.28	3.35	3.37
			CH ₂	s	3.55	3.46	3.43	3.33	3.45	3.52	3.60
dimethylacetamide			CH ₃ CO	s	2.09	1.97	1.96	1.60	1.97	2.07	2.08
			NCH ₃	s	3.02	3.00	2.94	2.57	2.96	3.31	3.06
			NCH ₃	s	2.94	2.83	2.78	2.05	2.83	2.92	2.90
dimethylformamide			CH	s	8.02	7.96	7.95	7.63	7.92	7.97	7.92
			CH ₃	s	2.96	2.94	2.89	2.36	2.89	2.99	3.01
			CH ₃	s	2.88	2.78	2.73	1.86	2.77	2.86	2.85
dimethyl sulfoxide			CH ₃	s	2.62	2.52	2.54	1.68	2.50	2.65	2.71
dioxane			CH ₂	s	3.71	3.59	3.57	3.35	3.60	3.66	3.75
ethanol			CH ₃	t, 7	1.25	1.12	1.06	0.96	1.12	1.19	1.17
			CH ₂	q, 7 ^d	3.72	3.57	3.44	3.34	3.54	3.60	3.65
			OH	s ^{c,d}	1.32	3.39	4.63		2.47		
ethyl acetate			CH ₃ CO	s	2.05	1.97	1.99	1.65	1.97	2.01	2.07
			CH ₂ CH ₃	q, 7	4.12	4.05	4.03	3.89	4.06	4.09	4.14
			CH ₂ CH ₃	t, 7	1.26	1.20	1.17	0.92	1.20	1.24	1.24
ethyl methyl ketone			CH ₃ CO	s	2.14	2.07	2.07	1.58	2.06	2.12	2.19
			CH ₂ CH ₃	q, 7	2.46	2.45	2.43	1.81	2.43	2.50	3.18
			CH ₂ CH ₃	t, 7	1.06	0.96	0.91	0.85	0.96	1.01	1.26
ethylene glycol "grease" ^f			CH	s ^e	3.76	3.28	3.34	3.41	3.51	3.59	3.65
			CH ₃	m	0.86	0.87		0.92	0.86	0.88	
			CH ₂	br s	1.26	1.29		1.36	1.27	1.29	
<i>n</i> -hexane			CH ₃	t	0.88	0.88	0.86	0.89	0.89	0.90	
			CH ₂	m	1.26	1.28	1.25	1.24	1.28	1.29	
HMPA ^g			CH ₃	d, 9.5	2.65	2.59	2.53	2.40	2.57	2.61	
methanol			CH ₃	s ^h	3.49	3.31	3.16	3.07	3.28	3.34	3.34
			OH	s ^{c,h}	1.09	3.12	4.01		2.16		
nitromethane			CH ₃	s	4.33	4.43	4.42	2.94	4.31	4.34	4.40
<i>n</i> -pentane			CH ₃	t, 7	0.88	0.88	0.86	0.87	0.89	0.90	
			CH ₂	m	1.27	1.27	1.27	1.23	1.29	1.29	
2-propanol			CH ₃	d, 6	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	3.92	4.02
pyridine			CH(2)	m	8.62	8.58	8.58	8.53	8.57	8.53	8.52
			CH(3)	m	7.29	7.35	7.39	6.66	7.33	7.44	7.45
			CH(4)	m	7.68	7.76	7.79	6.98	7.73	7.85	7.87
silicone grease ⁱ			CH ₃	s	0.07	0.13		0.29	0.08	0.10	
tetrahydrofuran			CH ₂	m	1.85	1.79	1.76	1.40	1.80	1.87	1.88
			CH ₂ O	m	3.76	3.63	3.60	3.57	3.64	3.71	3.74
toluene			CH ₃	s	2.36	2.32	2.30	2.11	2.33	2.32	
			CH(<i>o/p</i>)	m	7.17	7.1–7.2	7.18	7.02	7.1–7.3	7.16	
			CH(<i>m</i>)	m	7.25	7.1–7.2	7.25	7.13	7.1–7.3	7.16	
triethylamine			CH ₃	t, 7	1.03	0.96	0.93	0.96	0.96	1.05	0.99
			CH ₂	q, 7	2.53	2.45	2.43	2.40	2.45	2.58	2.57

^a 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 ²J_{H,D} = 1 Hz. ^b 2,6-Dimethyl-4-*tert*-butylphenol. ^c The signals from exchangeable protons were not always identified. ^d In some cases (see note *a*), the coupling interaction between the CH₂ and the OH protons may be observed (*J* = 5 Hz). ^e In CD₃CN, the OH proton was seen as a multiplet at δ 2.69, and extra coupling was also apparent on the methylene peak. ^f Long-chain, linear aliphatic hydrocarbons. Their solubility in DMSO was too low to give visible peaks. ^g Hexamethylphosphoramide. ^h In some cases (see notes *a*, *d*), the coupling interaction between the CH₃ and the OH protons may be observed (*J* = 5.5 Hz). ⁱ 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}C NMR Data^a

		CDCl_3	$(\text{CD}_3)_2\text{CO}$	$(\text{CD}_3)_2\text{SO}$	C_6D_6	CD_3CN	CD_3OD	D_2O
solvent signals		77.16 ± 0.06	29.84 ± 0.01 206.26 ± 0.13	39.52 ± 0.06	128.06 ± 0.02	1.32 ± 0.02 118.26 ± 0.02	49.00 ± 0.01	
acetic acid	CO	175.99	172.31	171.93	175.82	173.21	175.11	177.21
	CH ₃	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 ₃	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 ₃	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 ₃	31.25	30.72	30.38	30.47	30.68	30.91	30.29
<i>tert</i> -butyl methyl ether	OCH ₃	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 ₃	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 ₃ Ar	21.20	21.31	20.97	21.40	21.23	21.38	
	CH ₃ 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 ₂	26.94	27.51	26.33	27.23	27.63	27.96	
1,2-dichloroethane	CH ₂	43.50	45.25	45.02	43.59	45.54	45.11	
dichloromethane	CH ₂	53.52	54.95	54.84	53.46	55.32	54.78	
diethyl ether	CH ₃	15.20	15.78	15.12	15.46	15.63	15.46	14.77
	CH ₂	65.91	66.12	62.05	65.94	66.32	66.88	66.42
diglyme	CH ₃	59.01	58.77	57.98	58.66	58.90	59.06	58.67
	CH ₂	70.51	71.03	69.54	70.87	70.99	71.33	70.05
	CH ₂	71.90	72.63	71.25	72.35	72.63	72.92	71.63
1,2-dimethoxyethane	CH ₃	59.08	58.45	58.01	58.68	58.89	59.06	58.67
	CH ₂	71.84	72.47	71.07	72.21	72.47	72.72	71.49
dimethylacetamide	CH ₃	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 ₃	35.28	34.89	37.38	34.67	35.17	35.50	35.03
	NCH ₃	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 ₃	36.50	36.15	35.73	35.25	36.57	36.89	37.54
	CH ₃	31.45	31.03	30.73	30.72	31.32	31.61	32.03
dimethyl sulfoxide	CH ₃	40.76	41.23	40.45	40.03	41.31	40.45	39.39
dioxane	CH ₂	67.14	67.60	66.36	67.16	67.72	68.11	67.19
ethanol	CH ₃	18.41	18.89	18.51	18.72	18.80	18.40	17.47
	CH ₂	58.28	57.72	56.07	57.86	57.96	58.26	58.05
ethyl acetate	CH ₃ 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 ₂	60.49	60.56	59.74	60.21	60.98	61.50	62.32
	CH ₃	14.19	14.50	14.40	14.19	14.54	14.49	13.92
ethyl methyl ketone	CH ₃ 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 ₂ CH ₃	36.89	36.75	35.83	36.36	37.09	37.34	37.27
	CH ₂ CH ₃	7.86	8.03	7.61	7.91	8.14	8.09	7.87
ethylene glycol	CH ₂	63.79	64.26	62.76	64.34	64.22	64.30	63.17
	CH ₂	29.76	30.73	29.20	30.21	30.86	31.29	
"grease"	CH ₃	14.14	14.34	13.88	14.32	14.43	14.45	
<i>n</i> -hexane	CH ₂ (2)	22.70	23.28	22.05	23.04	23.40	23.68	
	CH ₂ (3)	31.64	32.30	30.95	31.96	32.36	32.73	
	CH ₃	36.87	37.04	36.42	36.88	37.10	37.00	36.46
methanol	CH ₃	50.41	49.77	48.59	49.97	49.90	49.86	49.50 ^c
nitromethane	CH ₃	62.50	63.21	63.28	61.16	63.66	63.08	63.22
<i>n</i> -pentane	CH ₃	14.08	14.29	13.28	14.25	14.37	14.39	
	CH ₂ (2)	22.38	22.98	21.70	22.72	23.08	23.38	
	CH ₂ (3)	34.16	34.83	33.48	34.45	34.89	35.30	
2-propanol	CH ₃	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 ₃	1.04	1.40		1.38		2.10	
silicone grease	CH ₂	25.62	26.15	25.14	25.72	26.27	26.48	25.67
	CH ₂ O	67.97	68.07	67.03	67.80	68.33	68.83	68.68
tetrahydrofuran	CH ₃	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 ₃	11.61	12.49	11.74	12.35	12.38	11.09	9.07
	CH ₂	46.25	47.07	45.74	46.77	47.10	46.96	47.19

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