**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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## Corrections and Comments

In the preparation of this manuscript, several errors were discovered in the original paper[1] and are reported herein. While comparing the $^1$H NMR spectral data obtained in toluene-$d_8$ to that in $C_6D_6$, it was discovered that the $^1$H NMR chemical shifts for acetic acid (C*H*$_3$), acetonitrile (C*H*$_3$) and *tert*-butyl alcohol (O*H*) in $C_6D_6$ 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 $C_6D_6$, $(CD_3)_2CO$, $(CD_3)_2SO$, $CD_3CN$, and $CD_3OD$ were reversed and are now corrected. The resonances for 1,2-dimethoxyethane (*C*H$_2$) in $(CD_3)_2CO$, silicone grease (*C*H$_3$) in $CDCl_3$, and 2-propanol (C*H*$_3$) in $CD_3OD$ 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 O*H* resonance for ethanol in $C_6D_6$ (0.50 ppm), the C*H*$_3$ resonance for silicone grease in $(CD_3)_2SO$ (–0.06 ppm), and replaced the “grease” entry (formerly motor oil[1]) with VWR vacuum pump oil #19, which is now reported in each deuterated solvent.

**Table S1.** [1]H NMR Data[2]

| | proton | mult | THF-$d_8$ | $CD_2Cl_2$ | $CDCl_3$ | toluene-$d_8$ | $C_6D_6$ | $C_6D_5Cl$ | $(CD_3)_2CO$ | $(CD_3)_2SO$ | $CD_3CN$ | TFE-$d_3$ | $CD_3OD$ | $D_2O$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| solvent residual signals | | | 1.72<br>3.58 | 5.32 | 7.26 | 2.08<br>6.97<br>7.01<br>7.09 | 7.16 | 6.96<br>6.99<br>7.14 | 2.05 | 2.50 | 1.94 | 5.02<br>3.88 | 3.31 | 4.79 |
| water | OH | s | 2.46 | 1.52 | 1.56 | 0.43 | 0.40 | 1.03 | 2.84[3] | 3.33[3] | 2.13 | 3.66 | 4.87 | - |
| acetic acid | $CH_3$ | 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 | $CH_3$ | 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 | $CH_3$ | 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 | C***H***$CH_2$ | ddt | 5.90 | 5.92 | 5.93 | 5.67[4] | 5.68[4] | 5.77 | 5.92 | 5.91 | 5.93 | 5.93 | 5.94 | 5.99 |
| | CHC***H***$_2$(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 |
| | CHC***H***$_2$(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 |
| | $CH_2$ | 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 |
| | $CH_3$ | 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 | - |
| *tert*-butyl alcohol | $CH_3$ | 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[5] | 3.16 | - | - | 0.58 | 0.63 | 1.30 | - | 4.19 | 2.18 | 2.20 | - | - |
| BHA | ArH | | | | | | | | | | | | | |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| *n* -hexane | $CH_3$ | t, 7 | 0.89 | 0.89 | 0.88 | 0.88 | 0.89 | 0.85 | 0.88 | 0.86 | 0.89 | 0.91 | 0.90 | - |
| | $CH_2$ | m | 1.29 | 1.27 | 1.26 | 1.22 | 1.24 | 1.19 | 1.28 | 1.25 | 1.28 | 1.31 | 1.29 | - |
| HMPA | $CH_3$ | 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 | $H_2$ | 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 | $CH_4$ | 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 | $CH_3$ | s[9] | 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[5,9] | 3.02 | 1.09 | 1.09 | - | - | 1.30 | 3.12 | 4.01 | 2.16 | - | - | - |
| nitromethane | $CH_3$ | 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 |
| *n* -pentane | $CH_3$ | t, 7 | 0.89 | 0.89 | 0.88 | 0.87 | 0.87 | 0.84 | 0.88 | 0.86 | 0.89 | 0.90 | 0.90 | - |
| | $CH_2$ | m | 1.31 | 1.30 | 1.27 | 1.25 | 1.23 | 1.23 | 1.27 | 1.27 | 1.29 | 1.33 | 1.29 | - |
| propane | $CH_3$ | 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 |
| | $CH_2$ | 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 | $CH_3$ | 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 | $CH_3$ | 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 |
| | $CH_2$(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 |
| | $CH_2$(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 | $CH_3$ | 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 | - |
| | $CH_2$ | 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[10] | $CH_2$(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 |
| | $CH_2$(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 | $CH_3$ | s | 0.11 | 0.09 | 0.07 | 0.26 | 0.29 | 0.14 | 0.13 | –0.06 | 0.08 | 0.16 | 0.10 | - |
| tetrahydrofuran | $CH_2$(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 |
| | $CH_2$(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 | $CH_3$ | 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 | $CH_3$ | 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 |
| | $CH_2$ | 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.** $^{13}C\{^{1}H\}$ NMR Data[2]

| | carbon | THF-$d_8$ | $CD_2Cl_2$ | $CDCl_3$ | toluene-$d_8$ | $C_6D_6$ | $C_6D_5Cl$ | $(CD_3)_2CO$ | $(CD_3)_2SO$ | $CD_3CN$ | TFE-$d_3$ | $CD_3OD$ | $D_2O$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| solvent signals | | 67.21<br>25.31 | 53.84 | 77.16 | 137.48<br>128.87<br>127.96<br>125.13<br>20.43 | 128.06 | 134.19<br>129.26<br>128.25<br>125.96 | 29.84<br>206.26 | 39.52 | 1.32<br>118.26 | 61.50<br>126.28 | 49.00 | - |
| 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 |
| | $CH_3$ | 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 |
| | $CH_3$ | 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 |
| | $CH_3$ | 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 |
| | $\boldsymbol{C}HCH_2$ | 133.90 | 132.94 | 132.33 | 132.98 | 132.90 | 132.69 | 133.76 | 132.71 | 133.83 | 133.33 | 133.71 | 132.48 |
| | $CH\boldsymbol{C}H_2$ | 117.58 | 118.00 | 118.34 | 117.49 | 117.64 | 117.63 | 117.81 | 117.64 | 118.06 | 119.39 | 118.22 | 119.03 |
| | $CH_2$ | 65.31 | 65.36 | 65.28 | 64.87 | 64.92 | 64.86 | 65.28 | 64.32 | 65.55 | 67.61 | 66.14 | 66.52 |
| | $CH_3$ | 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 | - |
| *tert* -butyl alcohol | $(CH_3)_3\boldsymbol{C}$ | 67.50 | 69.11 | 69.15 | 68.12 | 68.19 | 68.19 | 68.13 | 66.88 | 68.74 | 72.35 | 69.40 | 70.36 |
| | $(\boldsymbol{C}H_3)_3C$ | 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 | - |
| | $CH_3O$ | 55.39 | 55.88 | 55.70 | 55.04 | 55.27 | 55.08 | 55.51 | 54.89 | 55.94 | 57.55 | 55.96 | - |
| | $(\boldsymbol{C}H_3)_3C$ | 30.65 | 30.37 | 30.32 | 30.30 | 30.35 | 30.21 | 30.64 | 30.30 | 30.55 | 30.80 | 30.82 | - |
| | $(CH_3)_3\boldsymbol{C}$ | 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 | - |
| | $CH_3Ar$ | 21.21 | 21.27 | 21.20 | 21.42 | 21.40 | 21.10 | 21.31 | 20.97 | 21.23 | 21.34 | 21.38 | - |
| | $(\boldsymbol{C}H_3)_3C$ | 31.55 | 30.54 | 30.33 | 31.39 | 31.34 | 30.19 | 31.61 | 31.25 | 31.50 | 31.01 | 31.15 | - |
| | $(CH_3)_3\boldsymbol{C}$ | 34.91 | 34.56 | 34.25 | 34.39 | 34.35 | 34.11 | 35.00 | 34.33 | 35.05 | 35.69 | 35.36 | - |
| carbon dioxide | $CO_2$ | 125.69 | 125.26 | 124.99 | 124.86 | 124.76 | 126.08 | 125.81 | 124.21 | 125.89 | 126.92 | 126.31 | - |
| carbon disulfide | $CS_2$ | 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 | $CCl_4$ | 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 | $CH_2$ | 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 | $CH_2$ | 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 |
| | $CH_2$(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 |
| | $CH_2$(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 |
| | $CH_2$(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 |
| | $\boldsymbol{C}HCH_2$ | 133.08 | 132.24 | 131.58 | 132.30 | 132.18 | 131.93 | 133.16 | 132.18 | 133.20 | 132.72 | 133.25 | 132.76 |
| | $CH\boldsymbol{C}H_2$ | 117.70 | 118.75 | 118.96 | 118.04 | 118.22 | 118.22 | 118.53 | 118.32 | 118.86 | 120.15 | 118.74 | 118.75 |
| | $CH_2$ | 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 | $CH_2$ | 44.64 | 44.35 | 43.50 | 43.40 | 43.59 | 43.60 | 45.25 | 45.02 | 45.54 | 45.28 | 45.11 | - |
| dichloromethane | $CH_2$ | 54.67 | 54.24 | 53.52 | 53.47 | 53.46 | 53.54 | 54.95 | 54.84 | 55.32 | 54.46 | 54.78 | - |
| diethyl ether | $CH_3$ | 15.49 | 15.44 | 15.20 | 15.47 | 15.46 | 15.35 | 15.78 | 15.12 | 15.63 | 15.33 | 15.46 | 14.77 |
| | $CH_2$ | 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 | $CH_3$ | 58.72 | 58.95 | 59.01 | 58.62 | 58.66 | 58.42 | 58.77 | 57.98 | 58.90 | 59.40 | 59.06 | 58.67 |
| | $CH_2$ | 71.17 | 70.70 | 70.51 | 70.92 | 70.87 | 70.56 | 71.03 | 69.54 | 70.99 | 73.05 | 71.33 | 70.05 |
| | $CH_2$ | 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 | $CH_3$ | 58.72 | 59.02 | 59.08 | 58.63 | 58.68 | 58.31 | 58.45 | 58.03 | 58.89 | 59.52 | 59.06 | 58.67 |
| | $CH_2$ | 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 | $CH_3$ | 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 |
| | $NCH_3$ | 34.60 | 35.23 | 35.28 | 34.58 | 34.67 | 34.59 | 34.89 | 34.42 | 35.17 | 36.28 | 35.50 | 35.03 |
| | $NCH_3$ | 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 |
| | $CH_3$ | 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 | $CO_2$ | 167.14 | 167.32 | 167.18 | 166.49 | 166.66 | 166.51 | 167.58 | 166.91 | 168.07 | 170.88 | 168.70 | 170.12 |
| | $CH_3$ | 52.07 | 52.75 | 52.57 | 51.76 | 51.86 | 51.89 | 52.47 | 52.08 | 52.95 | 54.00 | 52.83 | 53.65 |
| | $CH_2$ | 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 | 163.17 | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 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 |
| | $CH_2$***C***$H_3$ | 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 | | |

**Table S3.** THF-$d_8$ ($^1$H NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.07 | s | $CH_3$ | hexamethyldisiloxane | 2.21 | s | ArCH | | | | | |

**Table S5.** $CD_2Cl_2$ ($^1$H NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.07 | s | $CH_3$ | hexamethyldisiloxane | 2.09 | s | $CH_3CO$ | ethyl methyl ketone | 4.61 | ddd | $CH_2$ | diallyl carbonate |
| 0.09 | s | $CH_3$ | silicone grease | 2.12 | s | $CH_3$ | acetone | 4.76 | s | OH | BHA |
| 0.21 | s | $CH_4$ | methane | 2.20 | s | $CH_3$ | hexamethylbenzene | 4.93 | dm, 10 | $CH_2$(1) | propylene |
| 0.84–0.89 | m | $CH_3$ | pump oil | 2.25 | s | $ArCH_3$ | BHT | 5.00 | s | OH | BHT |
| 0.84–0.90 | m | $CH_3$ | H grease[8] | 2.29 | t | $CH_2$(2,6) | cyclohexanone | 5.03 | dm, 17 | $CH_2$(2) | propylene |
| 0.85 | s | $CH_3$ | ethane | 2.34 | s | $CH_3$ | toluene | 5.22 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.89 | t, 7 | $CH_3$ | n-hexane | 2.43 | q, 7 | C***H***$_2$CH$_3$ | ethyl methyl ketone | 5.26 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 0.89 | t, 7 | $CH_3$ | n-pentane | 2.48 | q, 7 | $CH_2$ | triethylamine | 5.31 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 0.90 | t, 7.3 | $CH_3$ | propane | 2.55 | s | $CH_3$ | dimethyl sulfoxide | 5.32 | t | $CDHCl_2$ | $CD_2Cl_2$ residual |
| 0.99 | t, 7 | $CH_3$ | triethylamine | 2.60 | d, 9.5 | $CH_3$ | HMPA | 5.33 | s | $CH_2$ | dichloromethane |
| 1.00 | t, 7 | $CH_2C\boldsymbol{H}_3$ | ethyl methyl ketone | 2.82 | s | $CH_3$ | dimethylformamide | 5.35 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 1.09 | s[9] | OH | methanol | 2.82 | m | $CH_2$(2,5) | pyrrolidine | 5.40 | s | $CH_2$ | ethylene |
| 1.15 | t, 7 | $CH_3$ | diethyl ether | 2.87 | s | $NCH_3$ | dimethylacetamide | 5.84 | m | CH | propylene |
| 1.17 | d, 6 | $CH_3$ | 2-propanol | 2.91 | s | $CH_3$ | dimethylformamide | 5.92 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.19 | t, 7 | $CH_3$ | ethanol | 2.97 | s | $NCH_3$ | dimethylacetamide | 5.95 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.23 | t, 7 | $CH_2C\boldsymbol{H}_3$ | ethyl acetate | 3.33 | s | $OCH_3$ | diglyme | 6.19 | m | CH(3,4) | pyrrole |
| 1.24 | s | $CH_3$ | tert-butyl alcohol | 3.34 | s | $CH_3$ | 1,2-dimethoxyethane | 6.41 | dd | CH(3,4) | furan |
| 1.27 | br s | $CH_2$ | H grease[8] | 3.37 | s | $CH_2$ | dimethyl malonate | 6.73 | s | ArH | BHA |
| 1.27 | m | $CH_2$ | n-hexane | 3.42 | s[9] | $CH_3$ | methanol | 6.79 | m | CH(2,5) | pyrrole |
| 1.27 | br s | $CH_2$ | pump oil | 3.43 | q, 7 | $CH_2$ | diethyl ether | 6.97 | s | ArH | BHT |
| 1.30 | m | $CH_2$ | n-pentane | 3.49 | s | $CH_2$ | 1,2-dimethoxyethane | 7.07 | s | CH(4,5) | imidazole |
| 1.32 | sept, 7.3 | $CH_2$ | propane | 3.50 | m | $CH_2$ | diglyme | 7.15 | m | CH(2,4,6) | toluene |
| 1.33 | s[6] | OH | ethanol | 3.57 | m | $CH_2$ | diglyme | 7.24 | m | CH(3,5) | toluene |
| 1.42 | s | $ArC(CH_3)_3$ | BHA | 3.59 | s | $CH_2$ | 18-crown-6 | 7.28 | m | CH(3,5) | pyridine |
| 1.42 | s | $ArC(CH_3)_3$ | BHT | 3.65 | s | $CH_2$ | 1,4-dioxane | 7.32 | s | CH | chloroform |
| 1.44 | s | $CH_2$ | cyclohexane | 3.66 | q, 7[6] | $CH_2$ | ethanol | 7.35 | s | CH | benzene |
| 1.52 | s | OH | water | 3.66 | s | $CH_2$ | ethylene glycol | 7.46 | dd | CH(2,5) | furan |
| 1.69–1.72 | m | $CH_2$(4) | cyclohexanone | 3.69 | m | $CH_2$(2,5) | tetrahydrofuran | 7.53–7.57 | m | CH(3,5) | benzaldehyde |
| 1.67 | m | $CH_2$(3,4) | pyrrolidine | 3.72 | s | $CH_3$ | dimethyl malonate | 7.63 | s | CH(2) | imidazole |
| 1.71 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.73 | s | $ArOCH_3$ | BHA | 7.63–7.67 | m | CH(4) | benzaldehyde |
| 1.81–1.87 | m | $CH_2$(3,5) | cyclohexanone | 3.75 | s | $CH_3$ | dimethyl carbonate | 7.68 | m | CH(4) | pyridine |
| 1.82 | m | $CH_2$(3,4) | tetrahydrofuran | 3.76 | s | $CH_2$ | 1,2-dichloroethane | 7.87–7.89 | m | CH(2,6) | benzaldehyde |
| 1.97 | s | $CH_3$ | acetonitrile | 3.97 | sept, 6 | CH | 2-propanol | 7.96 | s | CH | dimethylformamide |
| 2.00 | s | $CH_3CO$ | ethyl acetate | 4.08 | q, 7 | C***H***$_2$CH$_3$ | ethyl acetate | 8.59 | m | CH(2,6) | pyridine |
| 2.02 | s | $CH_3CO$ | dimethylacetamide | 4.31 | s | $CH_3$ | nitromethane | 8.69 | br t | NH | pyrrole |
| 2.05 | s | $CH_3$ | allyl acetate | 4.55 | ddd | $CH_2$ | allyl acetate | 10.01 | s | HCO | benzaldehyde |
| 2.06 | s | $CH_3$ | acetic acid | 4.59 | s | $H_2$ | hydrogen | | | | |

**Table S6.** $CD_2Cl_2$ ($^{13}$C{$^1$H} NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.33 | $CH_4$ | methane | 30.14 | $CH_2$ | H grease[8] | 64.08 | $CH_2$ | ethylene glycol | 129.42 | CH(3,5) | benzaldehyde |
| 1.22 | $CH_3$ | silicone grease | 30.37 | ($\boldsymbol{C}H_3)_3C$ | BHA | 64.67 | CH | 2-propanol | 129.98 | CH(2,6) | benzaldehyde |
| 1.96 | $CH_3$ | hexamethyldisiloxane | 30.54 | ($\boldsymbol{C}H_3)_3C$ | BHT | 65.36 | $CH_2$ | allyl acetate | 132.09 | C | hexamethylbenzene |
| 2.03 | $CH_3$ | acetonitrile | 31.00 | $CH_3$ | acetone | 66.11 | $CH_2$ | diethyl ether | 132.24 | $CHCH_2$ | diallyl carbonate |
| 6.91 | $CH_3$ | ethane | 31.39 | $CH_3$ | dimethylformamide | 67.47 | $CH_2$ | 1,4-dioxane | 132.94 | $CHCH_2$ | allyl acetate |
| 7.94 | $CH_2\boldsymbol{C}H_3$ | ethyl methyl ketone | 31.46 | ($\boldsymbol{C}H_3)_3C$ | tert-butyl alcohol | 68.16 | $CH_2$(2,5) | tetrahydrofuran | 134.21 | CH | propylene |
| 12.12 | $CH_3$ | triethylamine | 32.01 | $CH_2$(3,4) | n-hexane | 68.76 | $CH_2$ | diallyl carbonate | 134.79 | CH (4) | benzaldehyde |
| 14.24 | $CH_3$ | n-pentane | 34.56 | $(CH_3)_3\boldsymbol{C}$ | BHT | 69.11 | $(CH_3)_3\boldsymbol{C}$ | tert-butyl alcohol | 135.76 | CH(2) | imidazole |
| 14.28 | $CH_3$ | n-hexane | 34.57 | $CH_2$(3) | n-pentane | 70.47 | $CH_2$ | 18-crown-6 | 136.16 | CH(4) | pyridine |
| 14.37 | $CH_3$ | ethyl acetate | 34.91 | $(CH_3)_3\boldsymbol{C}$ | BHA | 70.70 | $CH_2$ | diglyme | 136.32 | C(2,6) | BHT |
| 15.44 | $CH_3$ | diethyl ether | 35.23 | $NCH_3$ | dimethylacetamide | 72.24 | $CH_2$ | 1,2-dimethoxyethane | 136.98 | C(1) | benzaldehyde |
| 16.63 | $CH_3$ | propane | 36.56 | $CH_3$ | dimethylformamide | 72.25 | $CH_2$ | diglyme | 137.77 | C(4) | BHA |
| 16.63 | $CH_2$ | propane | 36.99 (d) | $CH_3$ | HMPA[i] | 77.99 | CH | chloroform | 138.36 | C(1) | toluene |
| 16.93 | $CH_3$ | hexamethylbenzene | 37.01 | $CH_2CH_3$ | ethyl methyl ketone | 96.52 | $CCl_4$ | carbon tetrachloride | 142.98 | CH(2,5) | furan |
| 18.69 | $CH_3$ | ethanol | 38.22 | $NCH_3$ | dimethylacetamide | 108.02 | CH(3,4) | pyrrole | 148.06 | C(2,6) | BHA |
| 19.47 | $CH_3$ | propylene | 41.33 | $CH_3$ | dimethyl sulfoxide | 109.86 | CH(3,4) | furan | 150.27 | CH(2,6) | pyridine |
| 20.91 | $CH_3$ | acetic acid | 41.48 | $CH_2$ | dimethyl malonate | 110.93 | CH(3,5) | BHA | 151.92 | C(1) | BHT |
| 21.06 | $CH_3$ | allyl acetate | 42.31 | $CH_2$(2,6) | cyclohexanone | 115.7 | $CH_2$ | propylene | 153.05 | C(1) | BHA |
| 21.15 | $CH_3CO$ | ethyl acetate | 44.35 | $CH_2$ | 1,2-dichloroethane | 116.92 | CN | acetonitrile | 155.15 | CO | diallyl carbonate |
| 21.27 | $CH_3Ar$ | BHT | 46.75 | $CH_2$ | triethylamine | 117.93 | CH(2,5) | pyrrole | 156.73 | CO | dimethyl carbonate |
| 21.53 | $CH_3$ | toluene | 47.02 | $CH_2$(2,5) | pyrrolidine | 118.00 | $CH\boldsymbol{C}H_2$ | allyl acetate | 162.57 | CH | dimethylformamide |
| 21.64 | $CH_3$ | dimethylacetamide | 50.45 | $CH_3$ | methanol | 118.75 | $CH\boldsymbol{C}H_2$ | diallyl carbonate | 167.32 | $CO_2$ | dimethyl malonate |
| 22.77 | $CH_2$(2,4) | n-pentane | 52.75 | $CH_3$ | dimethyl malonate | 122.16 | CH(4,5) | imidazole | 170.83 | CO | allyl acetate |
| 23.07 | $CH_2$(2,5) | n-hexane | 53.84 (p) | $CD_2Cl_2$ | $CD_2Cl_2$ signal | 123.20 | $CH_2$ | ethylene | 171.05 | CO | dimethylacetamide |
| 25.42 | $CH_2$(4) | cyclohexanone | 54.24 | $CH_2$ | dichloromethane | 124.06 | CH(3,5) | pyridine | 171.24 | CO | ethyl acetate |
| 25.43 | $CH_3$ | 2-propanol | 55.09 | $CH_3$ | dimethyl carbonate | 125.26 | $CO_2$ | carbon dioxide | 175.85 | CO | acetic acid |
| 25.83 | $CH_2$(3,4) | pyrrolidine | 55.88 | $CH_3O$ | BHA | 125.62 | CH(4) | toluene | 192.61 | HCO | benzaldehyde |
| 25.98 | $CH_2$(3,4) | tetrahydrofuran | 58.57 | $CH_2$ | ethanol | 125.84 | CH(3,5) | BHT | 192.95 | $CS_2$ | carbon disulfide |
| 27.38 | $CH_2$ | cyclohexane | 58.95 | $CH_3$ | diglyme | 128.54 | CH(3,5) | toluene | 206.78 | CO | acetone |
| 27.47 | $CH_2$(3,5) | cyclohexanone | 59.02 | $CH_3$ | 1,2-dimethoxyethane | 128.68 | CH | benzene | 209.57 | CO | ethyl methyl ketone |
| 29.55 | $CH_3CO$ | ethyl methyl ketone | 60.63 | $CH_2$ | ethyl acetate | 128.73 | C(4) | BHT | 211.82 | CO | cyclohexanone |
| 30.13 | $CH_2$ | pump oil | 63.03 | $CH_3$ | nitromethane | 129.35 | CH(2,6) | toluene | | | |

## **Table S7.** $CDCl_3$ ($^1$H NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.07 | s | $CH_3$ | hexamethyldisiloxane | 2.14 | s | $CH_3CO$ | ethyl methyl ketone | 4.64 | ddd | $CH_2$ | diallyl carbonate |
| 0.07 | s | $CH_3$ | silicone grease | 2.17 | s | $CH_3$ | acetone | 4.76 | s | OH[d] | BHA |
| 0.22 | s | $CH_4$ | methane | 2.24 | s | $CH_3$ | hexamethylbenzene | 4.94 | dm, 10 | $CH_2$(1) | propylene |
| 0.83–0.89 | m | $CH_3$ | pump oil | 2.27 | s | $ArCH_3$ | BHT | 5.01 | s | OH[d] | BHT |
| 0.84–0.87 | m | $CH_3$ | H grease[8] | 2.33 | t | $CH_2$(2,6) | cyclohexanone | 5.03 | dm, 17 | $CH_2$(2) | propylene |
| 0.87 | s | $CH_3$ | ethane | 2.36 | s | $CH_3$ | toluene | 5.24 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.88 | t, 7 | $CH_3$ | *n*-hexane | 2.46 | q, 7 | C***H***$_2$CH$_3$ | ethyl methyl ketone | 5.27 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 0.88 | t, 7 | $CH_3$ | *n*-pentane | 2.53 | q, 7 | $CH_2$ | triethylamine | 5.30 | s | $CH_2$ | dichloromethane |
| 0.90 | t, 7.3 | $CH_3$ | propane | 2.62 | s | $CH_3$ | dimethyl sulfoxide | 5.32 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 1.03 | t, 7 | $CH_3$ | triethylamine | 2.65 | d, 9.5 | $CH_3$ | HMPA | 5.37 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 1.06 | t, 7 | $CH_2C$***H***$_3$ | ethyl methyl ketone | 2.87 | m | $CH_2$(2,5) | pyrrolidine | 5.40 | s | $CH_2$ | ethylene |
| 1.09 | s[9] | OH | methanol | 2.88 | s | $CH_3$ | dimethylformamide | 5.83 | m | CH | propylene |
| 1.21 | t, 7 | $CH_3$ | diethyl ether | 2.94 | s | $NCH_3$ | dimethylacetamide | 5.93 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.22 | d, 6 | $CH_3$ | 2-propanol | 2.96 | s | $CH_3$ | dimethylformamide | 5.94 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.25 | t, 7 | $CH_3$ | ethanol | 3.02 | s | $NCH_3$ | dimethylacetamide | 6.26 | m | CH(3,4) | pyrrole |
| 1.25 | br s | $CH_2$ | H grease[8] | 3.39 | s | $OCH_3$ | diglyme | 6.40 | dd | CH(3,4) | furan |
| 1.26 | t, 7 | $CH_2C$***H***$_3$ | ethyl acetate | 3.40 | s | $CH_3$ | 1,2-dimethoxyethane | 6.76 | s | ArH | BHA |
| 1.26 | m | $CH_2$ | *n*-hexane | 3.40 | s | $CH_2$ | dimethyl malonate | 6.83 | m | CH(2,5) | pyrrole |
| 1.26 | br s | $CH_2$ | pump oil | 3.48 | q, 7 | $CH_2$ | diethyl ether | 6.98 | s | ArH | BHT |
| 1.27 | m | $CH_2$ | *n*-pentane | 3.49 | s[9] | $CH_3$ | methanol | 7.10 | s | CH(4,5) | imidazole |
| 1.28 | s | $CH_3$ | *tert*-butyl alcohol | 3.55 | s | $CH_2$ | 1,2-dimethoxyethane | 7.17 | m | CH(2,4,6) | toluene |
| 1.32 | s[6] | OH | ethanol | 3.57 | m | $CH_2$ | diglyme | 7.25 | m | CH(3,5) | toluene |
| 1.32 | sept, 7.3 | $CH_2$ | propane | 3.65 | m | $CH_2$ | diglyme | 7.26 | s | CH | $CDCl_3$ residual |
| 1.43 | s | $ArC(CH_3)_3$ | BHT | 3.67 | s | $CH_2$ | 18-crown-6 | 7.26 | s | CH | chloroform |
| 1.43 | s | $CH_2$ | cyclohexane | 3.71 | s | $CH_2$ | 1,4-dioxane | 7.29 | m | CH(3,5) | pyridine |
| 1.44 | s | $ArC(CH_3)_3$ | BHA | 3.72 | q, 7[9] | $CH_2$ | ethanol | 7.36 | s | CH | benzene |
| 1.56 | s | OH | water | 3.73 | s | $CH_2$ | 1,2-dichloroethane | 7.45 | dd | CH(2,5) | furan |
| 1.68 | m | $CH_2$(3,4) | pyrrolidine | 3.75 | s | $CH_3$ | dimethyl malonate | 7.51–7.57 | m | CH(3,5) | benzaldehyde |
| 1.71–1.73 | m | $CH_2$(4) | cyclohexanone | 3.76 | s | $CH_2$ | ethylene glycol | 7.61–7.65 | m | CH(4) | benzaldehyde |
| 1.73 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.76 | m | $CH_2$(2,5) | tetrahydrofuran | 7.67 | s | CH(2) | imidazole |
| 1.84–1.86 | m | $CH_2$(3,5) | cyclohexanone | 3.77 | s | $ArOCH_3$ | BHA | 7.68 | m | CH(4) | pyridine |
| 1.85 | m | $CH_2$(3,4) | tetrahydrofuran | 3.79 | s | $CH_3$ | dimethyl carbonate | 7.88–7.91 | m | CH(2,6) | benzaldehyde |
| 2.05 | s | $CH_3CO$ | ethyl acetate | 4.04 | sept, 6 | CH | 2-propanol | 8.02 | s | CH | dimethylformamide |
| 2.09 | s | $CH_3$ | allyl acetate | 4.12 | q, 7 | C***H***$_2$CH$_3$ | ethyl acetate | 8.40 | br t | NH | pyrrole |
| 2.09 | s | $CH_3CO$ | dimethylacetamide | 4.33 | s | $CH_3$ | nitromethane | 8.62 | m | CH(2,6) | pyridine |
| 2.10 | s | $CH_3$ | acetic acid | 4.57 | ddd | $CH_2$ | allyl acetate | 10.03 | s | HCO | benzaldehyde |
| 2.10 | s | $CH_3$ | acetonitrile | 4.62 | s | $H_2$ | hydrogen | | | | |

## **Table S8.** $CDCl_3$ ($^{13}$C{$^1$H} NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.63 | $CH_4$ | methane | 29.84 | $CH_2$ | pump oil | 64.50 | CH | 2-propanol | 129.16 | CH(3,5) | benzaldehyde |
| 1.19 | $CH_3$ | silicone grease | 30.32 | ($C$H$_3$)$_3$C | BHA | 65.28 | $CH_2$ | allyl acetate | 129.91 | CH(2,6) | benzaldehyde |
| 1.89 | $CH_3$ | acetonitrile | 30.33 | ($C$H$_3$)$_3$C | BHT | 65.91 | $CH_2$ | diethyl ether | 131.58 | ***C***H$CH_2$ | diallyl carbonate |
| 1.97 | $CH_3$ | hexamethyldisiloxane | 30.92 | $CH_3$ | acetone | 67.14 | $CH_2$ | 1,4-dioxane | 132.21 | C | hexamethylbenzene |
| 6.89 | $CH_3$ | ethane | 31.25 | ($C$H$_3$)$_3$C | *tert*-butyl alcohol | 67.97 | $CH_2$(2,5) | tetrahydrofuran | 132.33 | ***C***H$CH_2$ | allyl acetate |
| 7.86 | $CH_2C$H$_3$ | ethyl methyl ketone | 31.45 | $CH_3$ | dimethyl malonate | 68.55 | $CH_2$ | diallyl carbonate | 133.91 | CH | propylene |
| 11.61 | $CH_3$ | triethylamine | 31.64 | $CH_2$(3,4) | *n*-hexane | 69.15 | (CH$_3$)$_3$***C*** | *tert*-butyl alcohol | 134.64 | CH (4) | benzaldehyde |
| 14.08 | $CH_3$ | *n*-pentane | 34.16 | $CH_2$(3) | *n*-pentane | 70.51 | $CH_2$ | diglyme | 135.38 | CH(2) | imidazole |
| 14.14 | $CH_3$ | *n*-hexane | 34.25 | (CH$_3$)$_3$***C*** | BHT | 70.55 | $CH_2$ | 18-crown-6 | 135.87 | C(2,6) | BHT |
| 14.19 | $CH_3$ | ethyl acetate | 34.72 | (CH$_3$)$_3$***C*** | BHA | 71.84 | $CH_2$ | 1,2-dimethoxyethane | 135.96 | CH(4) | pyridine |
| 15.20 | $CH_3$ | diethyl ether | 35.28 | $NCH_3$ | dimethylacetamide | 71.90 | $CH_2$ | diglyme | 136.58 | C(1) | benzaldehyde |
| 16.37 | $CH_2$ | propane | 36.50 | $CH_3$ | dimethyl malonate | 77.16 (t) | $CDCl_3$ | $CDCl_3$ signal | 137.36 | C(4) | BHA |
| 16.63 | $CH_3$ | propane | 36.87 (d) | $CH_3$ | HMPA[l] | 77.36 | CH | chloroform | 137.89 | C(1) | toluene |
| 16.98 | $CH_3$ | hexamethylbenzene | 36.89 | ***C***H$_2$CH$_3$ | ethyl methyl ketone | 96.34 | $CCl_4$ | carbon tetrachloride | 142.71 | CH(2,5) | furan |
| 18.41 | $CH_3$ | ethanol | 38.13 | $NCH_3$ | | | | | | | |

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

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.10 | s | $CH_3$ | hexamethyldisiloxane | 1.64 | s | $CH_3$ | dimethyl sulfoxide | 4.45 | s | $OH$[5] | BHA |
| 0.17 | s | $CH_4$ | methane | 1.69 | s | $CH_3CO$ | ethyl acetate | 4.50 | s | $H_2$ | hydrogen |
| 0.26 | s | $CH_3$ | silicone grease | 1.82 | q, 7 | $CH_2CH_3$ | ethyl methyl ketone | 4.72 | s | $OH$[5] | BHT |
| 0.43 | s | OH | water | 1.95 | t | $CH_2$(2,6) | cyclohexanone | 4.92 | ddt | $CHCH_2$(2) | diallyl carbonate |
| 0.58 | s | OH | *tert*-butyl alcohol | 1.96 | s | $CH_3$ | dimethylformamide | 4.92 | dm, 10 | $CH_2$(1) | propylene |
| 0.69 | s | $CH_3$ | acetonitrile | 2.08 | p | $CH_3$ | Toluene-$d_8$ residual | 4.94 | ddt | $CHCH_2$(2) | allyl acetate |
| 0.81 | s | $CH_3$ | ethane | 2.10 | s | $CH_3$ | hexamethylbenzene | 4.98 | dm, 17 | $CH_2$(2) | propylene |
| 0.83 | s[6] | OH | ethanol | 2.11 | s | $NCH_3$ | dimethylacetamide | 5.05 | ddt | $CHCH_2$(1) | allyl acetate |
| 0.84 | t, 7 | $CH_2CH_3$ | ethyl methyl ketone | 2.11 | s | $CH_3$ | toluene | 5.09 | ddt | $CHCH_2$(1) | diallyl carbonate |
| 0.87 | t, 7 | $CH_3$ | *n*-pentane | 2.23 | s | $ArCH_3$ | BHT | 5.25 | s | $CH_2$ | ethylene |
| 0.88 | t, 7 | $CH_3$ | *n*-hexane | 2.37 | s | $CH_3$ | dimethylformamide | 5.63 | ddt | $CHCH_2$ | diallyl carbonate |
| 0.88–0.96 | m | $CH_3$ | pump oil | 2.39 | q, 7 | $CH_2$ | triethylamine | 5.67[4] | t(nfo ABX) | $CHCH_2$ | allyl acetate |
| 0.89 | t, 7.3 | $CH_3$ | propane | 2.42 | d, 9.5 | $CH_3$ | HMPA | 5.70 | m | CH | propylene |
| 0.89–0.96 | m | $CH_3$ | H grease[8] | 2.54 | m | $CH_2$(2,5) | pyrrolidine | 6.07 | dd | CH(3,4) | furan |
| 0.94 | t, 7 | $CH_2CH_3$ | ethyl acetate | 2.56 | s | $NCH_3$ | dimethylacetamide | 6.10 | s | CH | chloroform |
| 0.95 | d, 6 | $CH_3$ | 2-propanol | 2.91 | s | $CH_2$ | 1,2-dichloroethane | 6.27 | m | CH(3,4) | pyrrole |
| 0.95 | t, 7 | $CH_3$ | triethylamine | 2.92 | s | $CH_2$ | dimethyl malonate | 6.43 | m | CH(2,5) | pyrrole |
| 0.97 | t, 7 | $CH_3$ | ethanol | 3.01 | s | $CH_3$ | nitromethane | 6.67 | m | CH(3,5) | pyridine |
| 1.03 | s | $CH_3$ | *tert*-butyl alcohol | 3.03 | s[9] | $CH_3$ | methanol | 6.83 | s | ArH | BHA |
| 1.10 | t, 7 | $CH_3$ | diethyl ether | 3.12 | s | $OCH_3$ | diglyme | 6.86 | s | CH(4,5) | imidazole |
| 1.16–1.20 | m | $CH_2$(4) | cyclohexanone | 3.12 | s | $CH_3$ | 1,2-dimethoxyethane | 6.95–6.99 | m | CH(3,5) | benzaldehyde |
| 1.22 | m | $CH_2$ | *n*-hexane | 3.24 | s | $CH_3$ | dimethyl malonate | 6.96–7.01 | m | CH(2,4,6) | toluene |
| 1.25 | m | $CH_2$ | *n*-pentane | 3.25 | q, 7 | CH | | | | | |

**Table S11.** $C_6D_6$ ($^1$H NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.12 | s | $CH_3$ | hexamethyldisiloxane | 1.63 | s | $CH_3$ | allyl acetate | 4.38 | ddd | $CH_2$ | diallyl carbonate |
| 0.16 | s | $CH_4$ | methane | 1.65 | s | $CH_3CO$ | ethyl acetate | 4.47 | s | $H_2$ | hydrogen |
| 0.29 | s | $CH_3$ | silicone grease | 1.68 | s | $CH_3$ | dimethyl sulfoxide | 4.53 | s | OH[5] | BHA |
| 0.40 | s | OH | water | 1.81 | q, 7 | $CH_2CH_3$ | ethyl methyl ketone | 4.79 | s | OH[5] | BHT |
| 0.50 | s[6] | OH | ethanol | 1.86 | s | $CH_3$ | dimethylformamide | 4.92 | ddt | $CHCH_2$(2) | diallyl carbonate |
| 0.58 | s | $CH_3$ | acetonitrile | 1.98 | t | $CH_2$(2,6) | cyclohexanone | 4.94 | ddt | $CHCH_2$(2) | allyl acetate |
| 0.63 | s | OH | *tert*-butyl alcohol | 2.05 | s | $NCH_3$ | dimethylacetamide | 4.95 | dm, 10 | $CH_2$(1) | propylene |
| 0.80 | s | $CH_3$ | ethane | 2.11 | s | $CH_3$ | toluene | 5.01 | dm, 17 | $CH_2$(2) | propylene |
| 0.85 | t, 7 | $CH_2CH_3$ | ethyl methyl ketone | 2.13 | s | $CH_3$ | hexamethylbenzene | 5.06 | ddt | $CHCH_2$(1) | allyl acetate |
| 0.86 | t, 7.3 | $CH_3$ | propane | 2.24 | s | $ArCH_3$ | BHT | 5.09 | ddt | $CHCH_2$(1) | diallyl carbonate |
| 0.87 | t, 7 | $CH_3$ | *n*-pentane | 2.36 | s | $CH_3$ | dimethylformamide | 5.25 | s | $CH_2$ | ethylene |
| 0.89 | t, 7 | $CH_3$ | *n*-hexane | 2.40 | d, 9.5 | $CH_3$ | HMPA | 5.65 | ddt | $CHCH_2$ | diallyl carbonate |
| 0.90–0.98 | m | $CH_3$ | H grease[8] | 2.40 | q, 7 | $CH_2$ | triethylamine | 5.68[4] | t(nfo ABX) | $CHCH_2$ | allyl acetate |
| 0.91–0.97 | m | $CH_3$ | pump oil | 2.54 | m | $CH_2$(2,5) | pyrrolidine | 5.72 | m | CH | propylene |
| 0.92 | t, 7 | $CH_2CH_3$ | ethyl acetate | 2.57 | s | $NCH_3$ | dimethylacetamide | 6.08 | dd | CH(3,4) | furan |
| 0.95 | d, 6 | $CH_3$ | 2-propanol | 2.90 | s | $CH_2$ | 1,2-dichloroethane | 6.15 | s | CH | chloroform |
| 0.96 | t, 7 | $CH_3$ | ethanol | 2.94 | s | $CH_3$ | nitromethane | 6.37 | m | CH(3,4) | pyrrole |
| 0.96 | t, 7 | $CH_3$ | triethylamine | 2.97 | s | $CH_2$ | dimethyl malonate | 6.48 | m | CH(2,5) | pyrrole |
| 1.05 | s | $CH_3$ | *tert*-butyl alcohol | 3.07 | s[9] | $CH_3$ | methanol | 6.66 | m | CH(3,5) | pyridine |
| 1.08–1.16 | m | $CH_2$(4) | cyclohexanone | 3.11 | s | $OCH_3$ | diglyme | 6.90 | s | CH(4,5) | imidazole |
| 1.11 | t, 7 | $CH_3$ | diethyl ether | 3.12 | s | $CH_3$ | 1,2-dimethoxyethane | 6.93 | s | ArH | BHA |
| 1.23 | m | $CH_2$ | *n*-pentane | 3.23 | s | $CH_3$ | dimethyl malonate | 6.93–6.99 | m | CH(3,5) | benzaldehyde |
| 1.24 | m | $CH_2$ | *n*-hexane | 3.26 | q, 7 | $CH_2$ | diethyl ether | 6.98 | m | CH(4) | pyridine |
| 1.26 | sept, 7.3 | $CH_2$ | propane | 3.30 | s | $CH_3$ | dimethyl carbonate | 7.01–7.07 | m | CH(4) | benzaldehyde |
| 1.28–1.37 | m | $CH_2$(3,5) | cyclohexanone | 3.33 | s | $CH_2$ | 1,2-dimethoxyethane | 7.02 | m | CH(2,4,6) | toluene |
| 1.32 | br s | $CH_2$ | H grease[8] | 3.34 | m | $CH_2$ | diglyme | 7.05 | s | ArH | BHT |
| 1.33 | m | $CH_2$(3,4) | pyrrolidine | 3.34 | q, 7[6] | $CH_2$ | ethanol | 7.13 | dd | CH(2,5) | furan |
| 1.37 | br s | $CH_2$ | pump oil | 3.35 | s | $CH_2$ | 1,4-dioxane | 7.13 | m | CH(3,5) | toluene |
| 1.38 | s | $ArC(CH_3)_3$ | BHT | 3.39 | s | $CH_2$ | 18-crown-6 | 7.15 | s | CH | benzene |
| 1.40 | s | $CH_2$ | cyclohexane | 3.41 | s | $CH_2$ | ethylene glycol | 7.16 | s | CH | $C_6D_6$ residual |
| 1.40 | m | $CH_2$(3,4) | tetrahydrofuran | 3.46 | m | $CH_2$ | diglyme | 7.33 | s | CH(2) | imidazole |
| 1.41 | s | $ArC(CH_3)_3$ | BHA | 3.48 | s | $ArOCH_3$ | BHA | 7.49–7.53 | m | CH(2,6) | benzaldehyde |
| 1.52 | s | $CH_3$ | acetic acid | 3.57 | m | $CH_2$(2,5) | tetrahydrofuran | 7.63 | s | CH | dimethylformamide |
| 1.55 | s | $CH_3$ | acetone | 3.67 | sept, 6 | CH | 2-propanol | 7.80 | br t | NH | pyrrole |
| 1.55 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.89 | q, 7 | $CH_2CH_3$ | ethyl acetate | 8.53 | m | CH(2,6) | pyridine |
| 1.58 | s | $CH_3CO$ | ethyl methyl ketone | 4.27 | s | $CH_2$ | dichloromethane | 9.64 | s | HCO | benzaldehyde |
| 1.60 | s | $CH_3CO$ | dimethylacetamide | 4.38 | ddd | $CH_2$ | allyl acetate | | | | |

**Table S12.** $C_6D_6$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.29 | $CH_4$ | methane | 30.22 | $CH_2$ | H grease[8] | 64.34 | $CH_2$ | ethylene glycol | 129.33 | CH(2,6) | toluene |
| 0.20 | $CH_3$ | acetonitrile | 30.24 | $CH_2$ | pump oil | 64.92 | $CH_2$ | allyl acetate | 129.65 | CH(2,6) | benzaldehyde |
| 1.38 | $CH_3$ | silicone grease | 30.35 | $(CH_3)_3C$ | BHA | 65.94 | $CH_2$ | diethyl ether | 131.79 | C | hexamethylbenzene |
| 2.05 | $CH_3$ | hexamethyldisiloxane | 30.47 | $(CH_3)_3C$ | *tert*-butyl alcohol | 67.16 | $CH_2$ | 1,4-dioxane | 132.18 | $CHCH_2$ | diallyl carbonate |
| 6.96 | $CH_3$ | ethane | 30.72 | $CH_3$ | dimethylformamide | 67.80 | $CH_2$(2,5) | tetrahydrofuran | 132.90 | $CHCH_2$ | allyl acetate |
| 7.91 | $CH_2CH_3$ | ethyl methyl ketone | 31.34 | $(CH_3)_3C$ | BHT | 68.19 | $(CH_3)_3C$ | *tert*-butyl alcohol | 133.69 | CH | propylene |
| 12.35 | $CH_3$ | triethylamine | 31.96 | $CH_2$(3,4) | *n*-hexane | 68.28 | $CH_2$ | diallyl carbonate | 133.95 | CH (4) | benzaldehyde |
| 14.19 | $CH_3$ | ethyl acetate | 34.35 | $(CH_3)_3C$ | BHT | 70.59 | $CH_2$ | 18-crown-6 | 135.28 | CH(4) | pyridine |
| 14.25 | $CH_3$ | *n*-pentane | 34.45 | $CH_2$(3) | *n*-pentane | 70.87 | $CH_2$ | diglyme | 135.76 | CH(2) | imidazole |
| 14.32 | $CH_3$ | *n*-hexane | 34.67 | $NCH_3$ | dimethylacetamide | 72.21 | $CH_2$ | 1,2-dimethoxyethane | 136.08 | C(2,6) | BHT |
| 15.46 | $CH_3$ | diethyl ether | 34.72 | $(CH_3)_3C$ | BHA | 72.35 | $CH_2$ | diglyme | 137.05 | C(1) | benzaldehyde |
| 16.60 | $CH_2$ | propane | 35.25 | $CH_3$ | dimethylformamide | 77.79 | CH | chloroform | 137.50 | C(4) | BHA |
| 16.66 | $CH_3$ | propane | 36.36 | $CH_2CH_3$ | ethyl methyl ketone | 96.44 | $CCl_4$ | carbon tetrachloride | 137.91 | C(1) | toluene |
| 16.95 | $CH_3$ | hexamethylbenzene | 36.88 (d) | $CH_3$ | HMPA[11] | 108.21 | CH(3,4) | pyrrole | 142.73 | CH(2,5) | furan |
| 18.72 | $CH_3$ | ethanol | 37.03 | $NCH_3$ | dimethylacetamide | 109.67 | CH(3,4) | furan | 148.13 | C(2,6) | BHA |
| 19.38 | $CH_3$ | propylene | 40.03 | $CH_3$ | dimethyl sulfoxide | 111.15 | CH(3,5) | BHA | 150.27 | CH(2,6) | pyridine |
| 20.37 | $CH_3$ | acetic acid | 41.04 | $CH_2$ | dimethyl malonate | 115.92 | $CH_2$ | propylene | 152.05 | C(1) | BHT |
| 20.37 | $CH_3$ | allyl acetate | 41.83 | $CH_2$(2,6) | cyclohexanone | 116.02 | CN | acetonitrile | 153.62 | C(1) | BHA |
| 20.56 | $CH_3CO$ | ethyl acetate | 43.59 | $CH_2$ | 1,2-dichloroethane | 117.64 | $CHCH_2$ | allyl acetate | 155.24 | CO | diallyl carbonate |
| 21.10 | $CH_3$ | toluene | 46.77 | $CH_2$ | triethylamine | 117.78 | CH(2,5) | pyrrole | 156.71 | CO | dimethyl carbonate |
| 21.16 | $CH_3$ | dimethylacetamide | 46.86 | $CH_2$(2,5) | pyrrolidine | 118.22 | $CHCH_2$ | diallyl carbonate | 162.13 | CH | dimethylformamide |
| 21.40 | $CH_3Ar$ | BHT | 49.97 | $CH_3$ | methanol | 122.16 | CH(4,5) | imidazole | 166.66 | $CO_2$ | dimethyl malonate |
| 22.72 | $CH_2$(2,4) | *n*-pentane | 51.86 | $CH_3$ | dimethyl malonate | 122.96 | $CH_2$ | ethylene | 169.67 | CO | allyl acetate |
| 23.04 | $CH_2$(2,5) | *n*-hexane | 53.46 | $CH_2$ | dichloromethane | 123.58 | CH(3,5) | pyridine | 169.95 | CO | dimethylacetamide |
| 25.03 | $CH_2$(4) | cyclohexanone | 54.30 | $CH_3$ | dimethyl carbonate | 124.76 | $CO_2$ | carbon dioxide | 170.44 | CO | ethyl acetate |
| 25.18 | $CH_3$ | 2-propanol | 55.27 | $CH_3O$ | BHA | 125.68 | CH(4) | toluene | 175.82 | CO | acetic acid |
| 25.65 | $CH_2$(3,4) | pyrrolidine | 57.86 | $CH_2$ | ethanol | 125.83 | CH(3,5) | BHT | 191.43 | HCO | benzaldehyde |
| 25.72 | $CH_2$(3,4) | tetrahydrofuran | 58.66 | $CH_3$ | diglyme | 128.06 (t) | CD | $C_6D_6$ signal | 192.69 | $CS_2$ | carbon disulfide |
| 27.00 | $CH_2$(3,5) | cyclohexanone | 58.68 | $CH_3$ | 1,2-dimethoxyethane | 128.52 | C(4) | BHT | 204.43 | CO | acetone |
| 27.23 | $CH_2$ | cyclohexane | 60.21 | $CH_2$ | ethyl acetate | 128.56 | CH(3,5) | toluene | 206.55 | CO | ethyl methyl ketone |
| 28.56 | $CH_3CO$ | ethyl methyl ketone | 61.16 | $CH_3$ | nitromethane | 128.62 | CH | benzene | 209.10 | CO | cyclohexanone |
| 30.14 | $CH_3$ | acetone | 64.23 | CH | 2-propanol | 128.95 | CH(3,5) | benzaldehyde | | | |

**Table S13.** $C_6D_5Cl$ ($^1$H NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.10 | s | $CH_3$ | hexamethyldisiloxane | 1.78 | s | $CH_3CO$ | ethyl methyl ketone | 4.62 | s | OH[5] | BHA |
| 0.14 | s | $CH_3$ | silicone grease | 1.80 | s | $CH_3$ | allyl acetate | 4.77 | s | $CH_2$ | dichloromethane |
| 0.15 | s | $CH_4$ | methane | 2.03 | s | $CH_3$ | dimethyl sulfoxide | 4.91 | dm, 10 | $CH_2$(1) | propylene |
| 0.79 | s | $CH_3$ | ethane | 2.06 | q, 7 | C***H***$_2$$CH_3$ | ethyl methyl ketone | 4.98 | dm, 17 | $CH_2$(2) | propylene |
| 0.84 | t, 7 | $CH_3$ | *n*-pentane | 2.08 | t | $CH_2$(2,6) | cyclohexanone | 5.03 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 0.84 | t, 7.3 | $CH_3$ | propane | 2.10 | s | $CH_3$ | hexamethylbenzene | 5.04 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.85 | t, 7 | $CH_3$ | *n*-hexane | 2.16 | s | $CH_3$ | toluene | 5.15 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 0.86–0.92 | m | $CH_3$ | H grease[8] | 2.20 | s | $ArCH_3$ | BHT | 5.17 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 0.88–0.91 | m | $CH_3$ | pump oil | 2.30 | s | $CH_3$ | dimethylformamide | 5.29 | s | $CH_2$ | ethylene |
| 0.89 | t, 7 | $CH_2C$***H***$_3$ | ethyl methyl ketone | 2.39 | q, 7 | $CH_2$ | triethylamine | 5.50 | s | OH[5] | BHT |
| 0.93 | t, 7 | $CH_3$ | triethylamine | 2.42 | s | $NCH_3$ | dimethylacetamide | 5.72 | m | CH | propylene |
| 1.03 | s | OH | water | 2.47 | d, 9.5 | $CH_3$ | HMPA | 5.75 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.04 | t, 7 | $CH_2C$***H***$_3$ | ethyl acetate | 2.51 | s | $CH_3$ | dimethylformamide | 5.77 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.04 | d, 6 | $CH_3$ | 2-propanol | 2.64 | m | $CH_2$(2,5) | pyrrolidine | 6.19 | dd | CH(3,4) | furan |
| 1.06 | t, 7 | $CH_3$ | ethanol | 2.65 | s | $NCH_3$ | dimethylacetamide | 6.27 | m | CH(3,4) | pyrrole |
| 1.10 | t, 7 | $CH_3$ | diethyl ether | 3.15 | s | $CH_2$ | dimethyl malonate | 6.62 | m | CH(2,5) | pyrrole |
| 1.12 | s | $CH_3$ | *tert*-butyl alcohol | 3.16 | s | $OCH_3$ | diglyme | 6.74 | s | CH | chloroform |
| 1.19 | m | $CH_2$ | *n*-hexane | 3.17 | s | $CH_3$ | 1,2-dimethoxyethane | 6.83 | s | ArH | BHA |
| 1.21 | s | $CH_3$ | acetonitrile | 3.25 | s[9] | $CH_3$ | methanol | 6.90 | m | CH(3,5) | pyridine |
| 1.23 | m | $CH_2$ | *n*-pentane | 3.26 | s | $CH_2$ | 1,2-dichloroethane | 6.96 | br. s | CH(4) | $C_6D_5Cl$ residual |
| 1.26 | sept, 7.3 | $CH_2$ | propane | 3.31 | q, 7 | $CH_2$ | diethyl ether | 6.97 | s | ArH | BHT |
| 1.30 | s | OH | *tert*-butyl alcohol | 3.37 | m | $CH_2$ | diglyme | 6.99 | br. s | CH(3,5) | $C_6D_5Cl$ residual |
| 1.30 | br s | $CH_2$ | H grease[8] | 3.37 | s | $CH_2$ | 1,2-dimethoxyethane | 7.01 | s | CH(4,5) | imidazole |
| 1.30 | s[9] | OH | methanol | 3.41 | s | $CH_2$ | 18-crown-6 | 7.01–7.08 | m | CH(2,4,6) | toluene |
| 1.31 | br s | $CH_2$ | pump oil | 3.41 | s | $CH_3$ | dimethyl malonate | 7.10–7.17 | m | CH(3,5) | toluene |
| 1.33–1.37 | m | $CH_2$(4) | cyclohexanone | 3.45 | s | $CH_2$ | 1,4-dioxane | 7.14 | br. s | CH(2,6) | $C_6D_5Cl$ residual |
| 1.37 | s | $ArC(CH_3)_3$ | BHA | 3.48 | s | $CH_3$ | dimethyl carbonate | 7.15–7.19 | m | CH(3,5) | benzaldehyde |
| 1.37 | s | $ArC(CH_3)_3$ | BHT | 3.49 | m | $CH_2$ | diglyme | 7.20 | s | CH | benzene |
| 1.37 | s | $CH_2$ | cyclohexane | 3.51 | q, 7[6] | $CH_2$ | ethanol | 7.24 | dd | CH(2,5) | furan |
| 1.39 | s[6] | OH | ethanol | 3.58 | s | $CH_2$ | ethylene glycol | 7.24–7.28 | m | CH(4) | benzaldehyde |
| 1.43 | m | $CH_2$(3,4) | pyrrolidine | 3.59 | s | $CH_3$ | nitromethane | 7.25 | m | CH(4) | pyridine |
| 1.48–1.53 | m | $CH_2$(3,5) | cyclohexanone | 3.59 | m | $CH_2$(2,5) | tetrahydrofuran | 7.53 | s | CH(2) | imidazole |
| 1.55 | m | $CH_2$(3,4) | tetrahydrofuran | 3.61 | s | $ArOCH_3$ | BHA | 7.59–7.61 | m | CH(2,6) | benzaldehyde |
| 1.58 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.82 | sept, 6 | CH | 2-propanol | 7.73 | s | CH | dimethylformamide |
| 1.74 | s | $CH_3CO$ | dimethylacetamide | 3.96 | q, 7 | C***H***$_2$$CH_3$ | ethyl acetate | 8.51 | m | CH(2,6) | pyridine |
| 1.76 | s | $CH_3$ | acetic acid | 4.44 | ddd | $CH_2$ | allyl acetate | 8.61 | br t | NH | pyrrole |
| 1.77 | s | $CH_3$ | acetone | 4.46 | ddd | $CH_2$ | diallyl carbonate | 9.77 | s | HCO | benzaldehyde |
| 1.78 | s | $CH_3CO$ | ethyl acetate | 4.49 | s | $H_2$ | hydrogen | | | | |

**Table S14.** $C_6D_5Cl$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.33 | $CH_4$ | methane | 30.12 | $CH_3$ | acetone | 65.79 | $CH_2$ | diethyl ether | 129.26 (t) | CD(2,6) | $C_6D_5Cl$ signal |
| 0.63 | $CH_3$ | acetonitrile | 30.19 | (***C***$H_3$)$_3$C | BHT | 66.95 | $CH_2$ | 1,4-dioxane | 129.49 | CH(2,6) | benzaldehyde |
| 1.09 | $CH_3$ | silicone grease | 30.21 | (***C***$H_3$)$_3$C | BHA | 67.64 | $CH_2$(2,5) | tetrahydrofuran | 131.54 | C | hexamethylbenzene |
| 1.92 | $CH_3$ | hexamethyldisiloxane | 30.71 | $CH_3$ | dimethylformamide | 68.19 | $(CH_3)_3$***C*** | *tert*-butyl alcohol | 131.93 | ***C***$HCH_2$ | diallyl carbonate |
| 6.91 | $CH_3$ | ethane | 31.13 | (***C***$H_3$)$_3$C | *tert*-butyl alcohol | 68.19 | $CH_2$ | diallyl carbonate | 132.69 | ***C***$HCH_2$ | allyl acetate |
| 7.79 | $CH_2$***C***$H_3$ | ethyl methyl ketone | 31.77 | $CH_2$(3,4) | *n*-hexane | 70.55 | $CH_2$ | 18-crown-6 | 133.57 | CH | propylene |
| 11.87 | $CH_3$ | triethylamine | 34.11 | $(CH_3)_3$***C*** | BHT | 70.56 | $CH_2$ | diglyme | 134.02 | CH (4) | benzaldehyde |
| 14.07 | $CH_3$ | ethyl acetate | 34.26 | $CH_2$(3) | *n*-pentane | 71.81 | $CH_2$ | 1,2-dimethoxyethane | 134.19 | CCl | $C_6D_5Cl$ signal |
| 14.10 | $CH_3$ | *n*-pentane | 34.56 | $(CH_3)_3$***C*** | BHA | 72.07 | $CH_2$ | diglyme | 135.32 | CH(4) | pyridine |
| 14.18 | $CH_3$ | *n*-hexane | 34.59 | $NCH_3$ | dimethylacetamide | 77.67 | CH | chloroform | 135.50 | CH(2) | imidazole |
| 15.35 | $CH_3$ | diethyl ether | 35.45 | $CH_3$ | dimethylformamide | 96.38 | $CCl_4$ | carbon tetrachloride | 135.92 | C(2,6) | BHT |
| 16.48 | $CH_2$ | propane | 36.39 | ***C***$H_2CH_3$ | ethyl methyl ketone | 108.03 | CH(3,4) | pyrrole | 136.78 | C(1) | benzaldehyde |
| 16.56 | $CH_3$ | propane | 36.64 (d) | $CH_3$ | HMPA[11] | 109.64 | CH(3,4) | furan | 137.29 | C(4) | BHA |
| 16.68 | $CH_3$ | hexamethylbenzene | 37.13 | $NCH_3$ | dimethylacetamide | 110.84 | CH(3,5) | BHA | 137.65 | C(1) | toluene |
| 18.55 | $CH_3$ | ethanol | 40.27 | $CH_3$ | dimethyl sulfoxide | 115.86 | $CH_2$ | propylene | 142.49 | CH(2,5) | furan |
| 19.32 | $CH_3$ | propylene | 40.93 | $CH_2$ | dimethyl malonate | 115.93 | CN | acetonitrile | 147.87 | C(2,6) | BHA |
| 20.40 | $CH_3$ | acetic acid | 41.79 | $CH_2$(2,6) | cyclohexanone | 117.63 | CH***C***$H_2$ | allyl acetate | 149.93 | CH(2,6) | pyridine |
| 20.40 | $CH_3$ | allyl acetate | 43.60 | $CH_2$ | 1,2-dichloroethane | 117.65 | CH(2,5) | pyrrole | 151.69 | C(1) | BHT |
| 20.50 | ***C***$H_3CO$ | ethyl acetate | 46.36 | $CH_2$ | triethylamine | 118.22 | CH***C***$H_2$ | diallyl carbonate | 153.19 | C(1) | BHA |
| 21.03 | $CH_3$ | dimethylacetamide | 46.75 | $CH_2$(2,5) | pyrrolidine | 121.96 | CH(4,5) | imidazole | 154.87 | CO | diallyl carbonate |
| 21.10 | $CH_3Ar$ | BHT | 49.66 | $CH_3$ | methanol | 122.95 | $CH_2$ | ethylene | 156.36 | CO | dimethyl carbonate |
| 21.23 | $CH_3$ | toluene | 51.89 | $CH_3$ | dimethyl malonate | 123.49 | CH(3,5) | pyridine | 162.01 | CH | dimethylformamide |
| 22.54 | $CH_2$(2,4) | *n*-pentane | 53.54 | $CH_2$ | dichloromethane | 125.43 | CH(4) | toluene | 166.51 | $CO_2$ | dimethyl malonate |
| 22.86 | $CH_2$(2,5) | *n*-hexane | 54.23 | $CH_3$ | dimethyl carbonate | 125.58 | CH(3,5) | BHT | 169.59 | CO | allyl acetate |
| 25.07 | $CH_2$(4) | cyclohexanone | 55.08 | $CH_3O$ | BHA | 125.96 (t) | CD(4) | $C_6D_5Cl$ signal | 169.79 | CO | dimethylacetamide |
| 25.14 | $CH_3$ | 2-propanol | 57.63 | $CH_2$ | ethanol | 126.08 | $CO_2$ | carbon dioxide | 170.20 | CO | ethyl acetate |
| 25.59 | $CH_2$(3,4) | pyrrolidine | 58.31 | $CH_3$ | 1,2-dimethoxyethane | 128.25 (t) | CD(3,5) | $C_6D_5Cl$ signal | 175.67 | CO | acetic acid |
| 25.68 | $CH_2$(3,4) | tetrahydrofuran | 58.42 | $CH_3$ | diglyme | 128.26 | C(4) | BHT | 191.24 | HCO | benzaldehyde |
| 26.99 | $CH_2$ | cyclohexane | 60.06 | $CH_2$ | ethyl acetate | 128.31 | CH(3,5) | toluene | 192.49 | $CS_2$ | carbon disulfide |
| 27.02 | $CH_2$(3,5) | cyclohexanone | 61.68 | $CH_3$ | nitromethane | 128.38 | CH | benzene | 204.83 | CO | acetone |
| 28.82 | ***C***$H_3CO$ | ethyl methyl ketone | 64.03 | $CH_2$ | ethylene glycol | 128.87 | CH(3,5) | benzaldehyde | 206.87 | CO | ethyl methyl ketone |
| 30.11 | $CH_2$ | H grease[8] | 64.18 | CH | 2-propanol | 129.12 | CH(2,6) | toluene | 209.30 | CO | cyclohexanone |
| 30.11 | $CH_2$ | pump oil | 64.86 | $CH_2$ | allyl acetate | | | | | | |

**Table S17.** $(CD_3)_2SO$ ($^1H$ NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –0.06 | s | $CH_3$ | silicone grease | 2.18 | s | $ArCH_3$ | BHT | 4.61 | ddd | $CH_2$ | diallyl carbonate |
| 0.06 | s | $CH_3$ | hexamethyldisiloxane | 2.25 | t | $CH_2(2,6)$ | cyclohexanone | 4.61 | s | $H_2$ | hydrogen |
| 0.20 | s | $CH_4$ | methane | 2.30 | s | $CH_3$ | toluene | 4.63 | s | | |

**Table S19.** $CD_3CN$ ($^1H$ NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.07 | s | $CH_3$ | hexamethyldisiloxane | 2.18 | s | OH | *tert*-butyl alcohol | 4.57 | s | $H_2$ | hydrogen |
| 0.08 | s | $CH_3$ | silicone grease | 2.19 | s | $CH_3$ | hexamethylbenzene | 4.61 | ddd | $CH_2$ | diallyl carbonate |
| 0.20 | s | $CH_4$ | methane | 2.22 | s | $ArCH_3$ | BHT | 4.93 | dm, 10 | $CH_2(1)$ | propylene |
| 0.85 | s | $CH_3$ | ethane | 2.27 | t | $CH_2(2,6)$ | cyclohexanone | 4.98 | s | OH[5] | BHA |
| 0.85 | m | $CH_3$ | pump oil | 2.33 | s | $CH_3$ | toluene | 5.04 | dm, 17 | $CH_2(2)$ | propylene |
| 0.89 | t, 7 | $CH_3$ | *n*-hexane | 2.43 | q, 7 | $CH_2CH_3$ | ethyl methyl ketone | 5.20 | s | OH[5] | BHT |
| 0.89 | t, 7 | $CH_3$ | *n*-pentane | 2.45 | q, 7 | $CH_2$ | triethylamine | 5.21 | ddt | $CHCH_2(2)$ | allyl acetate |
| 0.90 | t, 7.3 | $CH_3$ | propane | 2.47 | s[6] | OH | ethanol | 5.25 | ddt | $CHCH_2(2)$ | diallyl carbonate |
| 0.96 | t, 7 | $CH_2CH_3$ | ethyl methyl ketone | 2.50 | s | $CH_3$ | dimethyl sulfoxide | 5.29 | ddt | $CHCH_2(1)$ | allyl acetate |
| 0.96 | t, 7 | $CH_3$ | triethylamine | 2.57 | d, 9.5 | $CH_3$ | HMPA | 5.34 | ddt | $CHCH_2(1)$ | diallyl carbonate |
| 1.09 | d, 6 | $CH_3$ | 2-propanol | 2.69[7] | m[7] | OH[7] | ethylene glycol[7] | 5.41 | s | $CH_2$ | ethylene |
| 1.12 | t, 7 | $CH_3$ | diethyl ether | 2.75 | m | $CH_2(2,5)$ | pyrrolidine | 5.44 | s | $CH_2$ | dichloromethane |
| 1.12 | t, 7 | $CH_3$ | ethanol | 2.77 | s | $CH_3$ | dimethylformamide | 5.85 | m | CH | propylene |
| 1.16 | s | $CH_3$ | *tert*-butyl alcohol | 2.83 | s | $NCH_3$ | dimethylacetamide | 5.93 | ddt | $CHCH_2$ | allyl acetate |
| 1.20 | t, 7 | $CH_2CH_3$ | ethyl acetate | 2.89 | s | $CH_3$ | dimethylformamide | 5.96 | ddt | $CHCH_2$ | diallyl carbonate |
| 1.27 | br s | $CH_2$ | pump oil | 2.96 | s | $NCH_3$ | dimethylacetamide | 6.10 | m | CH(3,4) | pyrrole |
| 1.28 | m | $CH_2$ | *n*-hexane | 3.28 | s | $CH_3$ | 1,2-dimethoxyethane | 6.44 | dd | CH(3,4) | furan |
| 1.29 | m | $CH_2$ | *n*-pentane | 3.28 | s[9] | $CH_3$ | methanol | 6.73 | s | ArH | BHA |
| 1.33 | sept, 7.3 | $CH_2$ | propane | 3.29 | s | $OCH_3$ | diglyme | 6.75 | m | CH(2,5) | pyrrole |
| 1.39 | s | $ArC(CH_3)_3$ | BHT | 3.38 | s | $CH_2$ | dimethyl malonate | 6.97 | s | ArH | BHT |
| 1.40 | s | $ArC(CH_3)_3$ | BHA | 3.42 | q, 7 | $CH_2$ | diethyl ether | 7.01 | s | CH(4,5) | imidazole |
| 1.44 | s | $CH_2$ | cyclohexane | 3.45 | m | $CH_2$ | diglyme | 7.10–7.30 | m | CH(2,4,6) | toluene |
| 1.61 | m | $CH_2(3,4)$ | pyrrolidine | 3.45 | s | $CH_2$ | 1,2-dimethoxyethane | 7.10–7.30 | m | CH(3,5) | toluene |
| 1.67–1.72 | m | $CH_2(4)$ | cyclohexanone | 3.51 | s | $CH_2$ | 18-crown-6 | 7.33 | m | CH(3,5) | pyridine |
| 1.70 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.51 | m[7] | $CH_2$ | ethylene glycol | 7.37 | s | CH | benzene |
| 1.79–1.84 | m | $CH_2(3,5)$ | cyclohexanone | 3.53 | m | $CH_2$ | diglyme | 7.52 | dd | CH(2,5) | furan |
| 1.80 | m | $CH_2(3,4)$ | tetrahydrofuran | 3.54 | q, 7[6] | $CH_2$ | ethanol | 7.57 | s | CH(2) | imidazole |
| 1.94 | p | $CHD_2$ | $CD_3CN$ residual | 3.60 | s | $CH_2$ | 1,4-dioxane | 7.57–7.61 | m | CH(3,5) | benzaldehyde |
| 1.96 | s | $CH_3$ | acetic acid | 3.64 | m | $CH_2(2,5)$ | tetrahydrofuran | 7.58 | s | CH | chloroform |
| 1.96 | s | $CH_3$ | acetonitrile | 3.68 | s | $CH_3$ | dimethyl malonate | 7.67–7.71 | m | CH(4) | benzaldehyde |
| 1.97 | s | $CH_3CO$ | dimethylacetamide | 3.72 | s | $ArOCH_3$ | BHA | 7.73 | m | CH(4) | pyridine |
| 1.97 | s | $CH_3CO$ | ethyl acetate | 3.72 | s | $CH_3$ | dimethyl carbonate | 7.89–7.91 | m | CH(2,6) | benzaldehyde |
| 2.02 | s | $CH_3$ | allyl acetate | 3.81 | s | $CH_2$ | 1,2-dichloroethane | 7.92 | s | CH | dimethylformamide |
| 2.06 | s | $CH_3CO$ | ethyl methyl ketone | 3.87 | sept, 6 | CH | 2-propanol | 8.57 | m | CH(2,6) | pyridine |
| 2.08 | s | $CH_3$ | acetone | 4.06 | q, 7 | $CH_2CH_3$ | ethyl acetate | 9.27 | br t | NH | pyrrole |
| 2.13 | s | OH | water | 4.31 | s | $CH_3$ | nitromethane | 10.01 | s | HCO | benzaldehyde |
| 2.16 | s[9] | OH | methanol | 4.53 | ddd | $CH_2$ | allyl acetate | | | | |

**Table S20.** $CD_3CN$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.61 | $CH_4$ | methane | 30.68 | $(CH_3)_3C$ | *tert*-butyl alcohol | 65.55 | $CH_2$ | allyl acetate | 130.07 | CH(3,5) | benzaldehyde |
| 1.32 (sept) | $CD_3$ | $CD_3CN$ signal | 30.86 | $CH_2$ | pump oil | 66.32 | $CH_2$ | diethyl ether | 130.42 | CH(2,6) | benzaldehyde |
| 1.79 | $CH_3$ | acetonitrile | 30.91 | $CH_3$ | acetone | 67.72 | $CH_2$ | 1,4-dioxane | 132.61 | C | hexamethylbenzene |
| 2.07 | $CH_3$ | hexamethyldisiloxane | 31.32 | $CH_3$ | dimethylformamide | 68.33 | $CH_2(2,5)$ | tetrahydrofuran | 133.20 | $CHCH_2$ | diallyl carbonate |
| 6.99 | $CH_3$ | ethane | 31.50 | $(CH_3)_3C$ | BHT | 68.74 | $(CH_3)_3C$ | *tert*-butyl alcohol | 133.83 | $CHCH_2$ | allyl acetate |
| 8.14 | $CH_2CH_3$ | ethyl methyl ketone | 32.36 | $CH_2(3,4)$ | *n*-hexane | 69.09 | $CH_2$ | diallyl carbonate | 134.78 | CH | propylene |
| 12.38 | $CH_3$ | triethylamine | 34.89 | $CH_2(3)$ | *n*-pentane | 70.99 | $CH_2$ | diglyme | 135.40 | CH (4) | benzaldehyde |
| 14.37 | $CH_3$ | *n*-pentane | 35.05 | $(CH_3)_3C$ | BHT | 71.22 | $CH_2$ | 18-crown-6 | 136.33 | CH(2) | imidazole |
| 14.43 | $CH_3$ | *n*-hexane | 35.17 | $NCH_3$ | dimethylacetamide | 72.47 | $CH_2$ | 1,2-dimethoxyethane | 136.89 | CH(4) | pyridine |
| 14.54 | $CH_3$ | ethyl acetate | 35.48 | $(CH_3)_3C$ | BHA | 72.63 | $CH_2$ | diglyme | 137.62 | C(1) | benzaldehyde |
| 15.63 | $CH_3$ | diethyl ether | 36.57 | $CH_3$ | dimethylformamide | 79.17 | CH | chloroform | 138.13 | C(2,6) | BHT |
| 16.73 | $CH_3$ | propane | 37.09 | $CH_2CH_3$ | ethyl methyl ketone | 96.68 | $CCl_4$ | carbon tetrachloride | 138.90 | C(1) | toluene |
| 16.91 | $CH_2$ | propane | 37.10 (d) | $CH_3$ | HMPA[11] | 108.31 | CH(3,4) | pyrrole | 140.20 | C(4) | BHA |
| 16.94 | $CH_3$ | hexamethylbenzene | 38.26 | $NCH_3$ | dimethylacetamide | 110.49 | CH(3,4) | furan | 143.74 | CH(2,5) | furan |
| 18.80 | $CH_3$ | ethanol | 41.31 | $CH_3$ | dimethyl sulfoxide | 111.35 | CH(3,5) | BHA | 148.39 | C(2,6) | BHA |
| 19.48 | $CH_3$ | propylene | 41.77 | $CH_2$ | dimethyl malonate | 116.12 | $CH_2$ | propylene | 150.76 | CH(2,6) | pyridine |
| 20.73 | $CH_3$ | acetic acid | 42.44 | $CH_2(2,6)$ | cyclohexanone | 118.06 | $CHCH_2$ | allyl acetate | 152.42 | C(1) | BHT |
| 21.02 | $CH_3$ | allyl acetate | 45.54 | $CH_2$ | 1,2-dichloroethane | 118.26 | CN | $CD_3CN$ signal | 154.02 | C(1) | BHA |
| 21.16 | $CH_3CO$ | ethyl acetate | 47.10 | $CH_2$ | triethylamine | 118.26 | CN | acetonitrile | 155.66 | CO | diallyl carbonate |
| 21.23 | $CH_3Ar$ | BHT | 47.57 | $CH_2(2,5)$ | pyrrolidine | 118.47 | CH(2,5) | pyrrole | 157.26 | CO | dimethyl carbonate |
| 21.50 | $CH_3$ | toluene | 49.90 | $CH_3$ | methanol | 118.86 | $CHCH_2$ | diallyl carbonate | 163.31 | CH | dimethylformamide |
| 21.76 | $CH_3$ | dimethylacetamide | 52.95 | $CH_3$ | dimethyl malonate | 122.78 | CH(4,5) | imidazole | 168.07 | $CO_2$ | dimethyl malonate |
| 23.08 | $CH_2(2,4)$ | *n*-pentane | 55.32 | $CH_2$ | dichloromethane | 123.69 | $CH_2$ | ethylene | 171.31 | CO | dimethylacetamide |
| 23.40 | $CH_2(2,5)$ | *n*-hexane | 55.39 | $CH_3$ | dimethyl carbonate | 125.89 | $CO_2$ | carbon dioxide | 171.32 | CO | allyl acetate |
| 25.55 | $CH_3$ | 2-propanol | 55.94 | $CH_3O$ | BHA | 126.28 | CH(4) | toluene | 171.68 | CO | ethyl acetate |
| 25.62 | $CH_2(4)$ | cyclohexanone | 57.96 | $CH_2$ | ethanol | 126.38 | CH(3,5) | BHT | 173.21 | CO | acetic acid |
| 26.27 | $CH_2(3,4)$ | tetrahydrofuran | 58.89 | $CH_3$ | 1,2-dimethoxyethane | 127.76 | CH(3,5) | pyridine | 193.60 | $CS_2$ | carbon disulfide |
| 26.34 | $CH_2(3,4)$ | pyrrolidine | 58.90 | $CH_3$ | diglyme | 129.23 | CH(3,5) | toluene | 193.64 | HCO | benzaldehyde |
| 27.63 | $CH_2$ | cyclohexane | 60.98 | $CH_2$ | ethyl acetate | 129.32 | CH | benzene | 207.43 | CO | acetone |
| 27.80 | $CH_2(3,5)$ | cyclohexanone | 63.66 | $CH_3$ | nitromethane | 129.61 | C(4) | BHT | 209.88 | CO | ethyl methyl ketone |
| 29.60 | $CH_3CO$ | ethyl methyl ketone | 64.22 | $CH_2$ | ethylene glycol | 129.94 | CH(2,6) | toluene | 211.99 | CO | cyclohexanone |
| 30.55 | $(CH_3)_3C$ | BHA | 64.30 | CH | 2-propanol | | | | | | |

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

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.08 | s | $CH_3$ | hexamethyldisiloxane | 2.20 | s | OH | *tert*-butyl alcohol | 4.53 | s | $H_2$ | hydrogen |
| 0.16 | s | $CH_3$ | silicone grease | 2.24 | s | $ArCH_3$ | BHT | 4.58 | ddd | $CH_2$ | allyl acetate |
| 0.18 | s | $CH_4$ | methane | 2.24 | s | $CH_3$ | hexamethylbenzene | 4.62 | ddd | $CH_2$ | diallyl carbonate |
| 0.85 | s | $CH_3$ | ethane | 2.33 | s | $CH_3$ | toluene | 4.93 | dm, 10 | $CH_2$(1) | propylene |
| 0.88–0.94 | m | $CH_3$ | H grease[g] | 2.38 | t | $CH_2$(2,6) | cyclohexanone | 5.02 | s | OH | TFE-$d_3$ residual |
| 0.90 | t, 7 | $CH_3$ | *n*-pentane | 2.49 | q, 7 | C***H***$_2$$CH_3$ | ethyl methyl ketone | 5.03 | dm, 17 | $CH_2$(2) | propylene |
| 0.90 | t, 7.3 | $CH_3$ | propane | 2.63 | s | $CH_3$ | dimethyl sulfoxide | 5.24 | s | $CH_2$ | dichloromethane |
| 0.91 | t, 7 | $CH_3$ | *n*-hexane | 2.63 | d, 9.5 | $CH_3$ | HMPA | 5.25 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.99 | m | $CH_3$ | pump oil | 2.88 | s | $CH_3$ | dimethylformamide | 5.28 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 1.05 | t, 7 | $CH_2$C***H***$_3$ | ethyl methyl ketone | 2.94 | s | $NCH_3$ | dimethylacetamide | 5.32 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 1.20 | t, 7 | $CH_3$ | diethyl ether | 2.98 | s | $CH_3$ | dimethylformamide | 5.35 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 1.20 | d, 6 | $CH_3$ | 2-propanol | 3.05 | s | $NCH_3$ | dimethylacetamide | 5.40 | s | $CH_2$ | ethylene |
| 1.22 | t, 7 | $CH_3$ | ethanol | 3.11 | m | $CH_2$(2,5) | pyrrolidine | 5.87 | m | CH | propylene |
| 1.26 | t, 7 | $CH_2$C***H***$_3$ | ethyl acetate | 3.12 | q, 7 | $CH_2$ | triethylamine | 5.92 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.28 | s | $CH_3$ | *tert*-butyl alcohol | 3.40 | s | $CH_3$ | 1,2-dimethoxyethane | 5.93 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.31 | m | $CH_2$ | *n*-hexane | 3.41 | s | $OCH_3$ | diglyme | 6.24 | m | CH(3,4) | pyrrole |
| 1.31 | t, 7 | $CH_3$ | triethylamine | 3.41 | s | $CH_2$ | dimethyl malonate | 6.42 | dd | CH(3,4) | furan |
| 1.33 | br s | $CH_2$ | H grease[g] | 3.44 | s | $CH_3$ | methanol | 6.84 | m | CH(2,5) | pyrrole |
| 1.33 | m | $CH_2$ | *n*-pentane | 3.58 | q, 7 | $CH_2$ | diethyl ether | 6.87 | s | ArH | BHA |
| 1.33 | sept, 7.3 | $CH_2$ | propane | 3.61 | s | $CH_2$ | 1,2-dimethoxyethane | 7.03 | s | CH(4,5) | imidazole |
| 1.41 | br s | $CH_2$ | pump oil | 3.62 | m | $CH_2$ | diglyme | 7.06 | s | ArH | BHT |
| 1.43 | s | $ArC(CH_3)_3$ | BHT | 3.64 | s | $CH_2$ | 18-crown-6 | 7.10–7.30 | m | CH(2,4,6) | toluene |
| 1.44 | s | $ArC(CH_3)_3$ | BHA | 3.66 | s | OH | water | 7.10–7.30 | m | CH(3,5) | toluene |
| 1.47 | s | $CH_2$ | cyclohexane | 3.67 | m | $CH_2$ | diglyme | 7.33 | s | CH | chloroform |
| 1.70 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.71 | s | $CH_2$ | 1,2-dichloroethane | 7.36 | s | CH | benzene |
| 1.75–1.78 | m | $CH_2$(4) | cyclohexanone | 3.71 | q, 7 | $CH_2$ | ethanol | 7.40 | m | CH(3,5) | pyridine |
| 1.87–1.92 | m | $CH_2$(3,5) | cyclohexanone | 3.72 | s | $CH_2$ | ethylene glycol | 7.44 | dd | CH(2,5) | furan |
| 1.91 | m | $CH_2$(3,4) | tetrahydrofuran | 3.76 | s | $CH_3$ | dimethyl malonate | 7.56–7.59 | m | CH(3,5) | benzaldehyde |
| 1.93 | m | $CH_2$(3,4) | pyrrolidine | 3.76 | s | $CH_2$ | 1,4-dioxane | 7.61 | s | CH(2) | imidazole |
| 1.95 | s | $CH_3$ | acetonitrile | 3.77 | s | $CH_3$ | dimethyl carbonate | 7.68–7.72 | m | CH(4) | benzaldehyde |
| 2.03 | s | $CH_3CO$ | ethyl acetate | 3.78 | m | $CH_2$(2,5) | tetrahydrofuran | 7.82 | m | CH(4) | pyridine |
| 2.06 | s | $CH_3$ | acetic acid | 3.79 | s | $ArOCH_3$ | BHA | 7.86 | s | CH | dimethylformamide |
| 2.07 | s | $CH_3$ | allyl acetate | 3.88 | tq | CDH | TFE-$d_3$ residual | 7.90–7.92 | m | CH(2,6) | benzaldehyde |
| 2.09 | s | $CH_3CO$ | dimethylacetamide | 4.05 | sept, 6 | CH | 2-propanol | 8.45 | m | CH(2,6) | pyridine |
| 2.16 | s | $CH_3CO$ | ethyl methyl ketone | 4.14 | q, 7 | C***H***$_2$$CH_3$ | ethyl acetate | 9.88 | s | HCO | benzaldehyde |
| 2.19 | s | $CH_3$ | acetone | 4.28 | s | $CH_3$ | nitromethane | | | | |

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

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –5.88 | $CH_4$ | methane | 30.96 | $CH_3$ | dimethylformamide | 66.69 | CH | 2-propanol | 130.82 | CH(3,5) | benzaldehyde |
| 1.00 | $CH_3$ | acetonitrile | 31.01 | ($C$H$_3$)$_3$C | BHT | 67.55 | $CH_2$ | diethyl ether | 131.78 | CH(2,6) | benzaldehyde |
| 2.09 | $CH_3$ | hexamethyldisiloxane | 31.07 | ($C$H$_3$)$_3$C | *tert*-butyl alcohol | 67.61 | $CH_2$ | allyl acetate | 132.72 | ***C***H$CH_2$ | diallyl carbonate |
| 2.87 | $CH_3$ | silicone grease | 31.85 | $CH_2$ | pump oil | 68.52 | $CH_2$ | 1,4-dioxane | 133.33 | ***C***H$CH_2$ | allyl acetate |
| 7.01 | $CH_3$ | ethane | 32.35 | CO | acetone | 69.53 | $CH_2$(2,5) | tetrahydrofuran | 134.04 | C | hexamethylbenzene |
| 8.29 | $CH_2$***C***$H_3$ | ethyl methyl ketone | 33.17 | $CH_2$(3,4) | *n*-hexane | 70.69 | $CH_2$ | diallyl carbonate | 136.00 | CH | propylene |
| 9.51 | $CH_3$ | triethylamine | 35.69 | ($CH_3$)$_3$***C*** | BHT | 70.80 | $CH_2$ | 18-crown-6 | 136.58 | CH(2) | imidazole |
| 14.36 | $CH_3$ | ethyl acetate | 35.76 | $CH_2$(3) | *n*-pentane | 71.33 | $CH_2$ | diglyme | 137.17 | CH (4) | benzaldehyde |
| 14.54 | $CH_3$ | *n*-pentane | 36.07 | ($CH_3$)$_3$***C*** | BHA | 72.35 | ($CH_3$)$_3$***C*** | *tert*-butyl alcohol | 137.84 | C(1) | benzaldehyde |
| 14.63 | $CH_3$ | *n*-hexane | 36.28 | $NCH_3$ | dimethylacetamide | 72.87 | $CH_2$ | 1,2-dimethoxyethane | 138.59 | C(2,6) | BHT |
| 15.33 | $CH_3$ | diethyl ether | 37.21 (d) | $CH_3$ | HMPA[i] | 73.05 | $CH_2$ | diglyme | 139.62 | CH(4) | pyridine |
| 16.93 | $CH_3$ | propane | 37.76 | $CH_3$ | dimethylformamide | 78.83 | CH | chloroform | 139.92 | C(1) | toluene |
| 17.04 | $CH_3$ | hexamethylbenzene | 38.23 | ***C***H$_2$$CH_3$ | ethyl methyl ketone | 97.74 | $CCl_4$ | carbon tetrachloride | 140.23 | C(4) | BHA |
| 17.46 | $CH_2$ | propane | 39.06 | $NCH_3$ | dimethylacetamide | 108.85 | CH(3,4) | pyrrole | 144.22 | CH(2,5) | furan |
| 18.11 | $CH_3$ | ethanol | 40.06 | $CH_3$ | dimethyl sulfoxide | 111.06 | CH(3,4) | furan | 149.76 | CH(2,6) | pyridine |
| 19.63 | $CH_3$ | propylene | 42.13 | $CH_2$ | dimethyl malonate | 112.90 | CH(3,5) | BHA | 150.52 | C(2,6) | BHA |
| 20.91 | $CH_3$ | acetic acid | 43.16 | $CH_2$(2,6) | cyclohexanone | 116.38 | $CH_2$ | propylene | 153.46 | C(1) | BHT |
| 21.10 | $CH_3$ | allyl acetate | 45.28 | $CH_2$ | 1,2-dichloroethane | 118.95 | CN | acetonitrile | 153.74 | C(1) | BHA |
| 21.18 | ***C***$H_3CO$ | ethyl acetate | 47.43 | $CH_2$(2,5) | pyrrolidine | 119.39 | CH***C***$H_2$ | allyl acetate | 157.39 | CO | diallyl carbonate |
| 21.34 | $CH_3$Ar | BHT | 48.45 | $CH_2$ | triethylamine | 119.61 | CH(2,5) | pyrrole | 159.04 | CO | dimethyl carbonate |
| 21.40 | $CH_3$ | dimethylacetamide | 50.67 | $CH_3$ | methanol | 120.15 | CH***C***$H_2$ | diallyl carbonate | 166.01 | CH | dimethylformamide |
| 21.62 | $CH_3$ | toluene | 54.00 | $CH_3$ | dimethyl malonate | 122.93 | CH(4,5) | imidazole | 170.88 | $CO_2$ | dimethyl malonate |
| 23.75 | $CH_2$(2,4) | *n*-pentane | 54.46 | $CH_2$ | dichloromethane | 124.08 | $CH_2$ | ethylene | 175.55 | CO | ethyl acetate |
| 24.06 | $CH_2$(2,5) | *n*-hexane | 56.17 | $CH_3$ | dimethyl carbonate | 126.27 | CH(3,5) | pyridine | 175.74 | CO | dimethylacetamide |
| 25.21 | $CH_3$ | 2-propanol | 57.55 | $CH_3O$ | BHA | 126.28 (q) | $CF_3$ | TFE-$d_3$ signal | 175.98 | CO | allyl acetate |
| 25.73 | $CH_2$(3,4) | pyrrolidine | 59.40 | $CH_3$ | diglyme | 126.82 | CH(4) | toluene | 177.96 | CO | acetic acid |
| 26.00 | $CH_2$(4) | cyclohexanone | 59.52 | $CH_3$ | 1,2-dimethoxyethane | 126.92 | $CO_2$ | carbon dioxide | 196.26 | $CS_2$ | carbon disulfide |
| 26.69 | $CH_2$(3,4) | tetrahydrofuran | 59.68 | $CH_2$ | ethanol | 127.11 | CH(3,5) | BHT | 197.63 | HCO | benzaldehyde |
| 28.34 | $CH_2$ | cyclohexane | 61.5 (qp) | $CD_2$ | TFE-$d_3$ signal | 129.79 | CH(3,5) | toluene | 214.98 | $CH_3$ | acetone |
| 28.56 | $CH_2$(3,5) | cyclohexanone | 62.70 | $CH_2$ | ethyl acetate | 129.84 | CH | benzene | 218.31 | CO | ethyl methyl ketone |
| 29.64 | ***C***$H_3CO$ | ethyl methyl ketone | 63.17 | $CH_3$ | nitromethane | 130.58 | CH(2,6) | toluene | 221.30 | CO | cyclohexanone |
| 30.80 | ($C$H$_3$)$_3$C | BHA | 64.87 | $CH_2$ | ethylene glycol | 130.62 | C(4) | BHT | | | |

**Table S23.** $CD_3OD$ ($^1H$ NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|

**Table S25.** $D_2O$ ($^1H$ NMR data by chemical shift in ppm)

| *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* | *shift* | *mult* | *proton* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.18 | s | $CH_4$ | methane | 2.71 | s | $CH_3$ | dimethyl sulfoxide | 4.69 | ddd | $CH_2$ | diallyl carbonate |
| 0.28 | s | $CH_3$ | hexamethyldisiloxane | 2.85 | s | $CH_3$ | dimethylformamide | 4.79 | s | HOD | $D_2O$ residual |
| 0.82 | s | $CH_3$ | ethane | 2.90 | s | $NCH_3$ | dimethylacetamide | 4.95 | dm, 10 | $CH_2$(1) | propylene |
| 0.88 | t, 7.3 | $CH_3$ | propane | 3.01 | s | $CH_3$ | dimethylformamide | 5.06 | dm, 17 | $CH_2$(2) | propylene |
| 0.99 | t, 7 | $CH_3$ | triethylamine | 3.06 | s | $NCH_3$ | dimethylacetamide | 5.30 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 1.17 | t, 7 | $CH_3$ | diethyl ether | 3.07 | m | $CH_2$(2,5) | pyrrolidine | 5.32 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 1.17 | t, 7 | $CH_3$ | ethanol | 3.18 | q, 7 | C***H***$_2$CH$_3$ | ethyl methyl ketone | 5.37 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 1.17 | d, 6 | $CH_3$ | 2-propanol | 3.34 | s | $CH_3$ | methanol | 5.40 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 1.24 | s | $CH_3$ | *tert*-butyl alcohol | 3.37 | s | $OCH_3$ | diglyme | 5.44 | s | $CH_2$ | ethylene |
| 1.24 | t, 7 | $CH_2$C***H***$_3$ | ethyl acetate | 3.37 | s | $CH_3$ | 1,2-dimethoxyethane | 5.90 | m | CH | propylene |
| 1.26 | t, 7 | $CH_2$C***H***$_3$ | ethyl methyl ketone | 3.56 | q, 7 | $CH_2$ | diethyl ether | 5.99 | ddt | C***H***CH$_2$ | allyl acetate |
| 1.30 | sept, 7.3 | $CH_2$ | propane | 3.60 | s | $CH_2$ | 1,2-dimethoxyethane | 5.99 | ddt | C***H***CH$_2$ | diallyl carbonate |
| 1.70 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.60 | s | $CH_2$ | dimethyl malonate | 6.26 | m | CH(3,4) | pyrrole |
| 1.70–1.75 | m | $CH_2$(4) | cyclohexanone | 3.61 | m | $CH_2$ | diglyme | 6.51 | dd | CH(3,4) | furan |
| 1.85–1.90 | m | $CH_2$(3,5) | cyclohexanone | 3.65 | q, 7 | $CH_2$ | ethanol | 6.93 | m | CH(2,5) | pyrrole |
| 1.87 | m | $CH_2$(3,4) | pyrrolidine | 3.65 | s | $CH_2$ | ethylene glycol | 7.14 | s | CH(4,5) | imidazole |
| 1.88 | m | $CH_2$(3,4) | tetrahydrofuran | 3.67 | m | $CH_2$ | diglyme | 7.45 | m | CH(3,5) | pyridine |
| 2.06 | s | $CH_3$ | acetonitrile | 3.69 | s | $CH_3$ | dimethyl carbonate | 7.57 | dd | CH(2,5) | furan |
| 2.07 | s | $CH_3CO$ | ethyl acetate | 3.74 | m | $CH_2$(2,5) | tetrahydrofuran | 7.57–7.66 | m | CH(3,5) | benzaldehyde |
| 2.08 | s | $CH_3$ | acetic acid | 3.75 | s | $CH_2$ | 1,4-dioxane | 7.76–7.80 | m | CH(4) | benzaldehyde |
| 2.08 | s | $CH_3CO$ | dimethylacetamide | 3.78 | s | $CH_3$ | dimethyl malonate | 7.78 | s | CH(2) | imidazole |
| 2.13 | s | $CH_3$ | allyl acetate | 3.80 | s | $CH_2$ | 18-crown-6 | 7.87 | m | CH(4) | pyridine |
| 2.19 | s | $CH_3CO$ | ethyl methyl ketone | 4.02 | sept, 6 | CH | 2-propanol | 7.92 | s | CH | dimethylformamide |
| 2.22 | s | $CH_3$ | acetone | 4.14 | q, 7 | C***H***$_2$CH$_3$ | ethyl acetate | 7.97–7.99 | m | CH(2,6) | benzaldehyde |
| 2.40 | t | $CH_2$(2,6) | cyclohexanone | 4.40 | s | $CH_3$ | nitromethane | 8.52 | m | CH(2,6) | pyridine |
| 2.57 | q, 7 | $CH_2$ | triethylamine | 4.62 | ddd | $CH_2$ | allyl acetate | 9.96 | s | HCO | benzaldehyde |
| 2.61 | d, 9.5 | $CH_3$ | HMPA | | | | | | | | |

**Table S26.** $D_2O$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1.47 | $CH_3$ | acetonitrile | 36.46 (d) | $CH_3$ | HMPA[i] | 67.19 | $CH_2$ | 1,4-dioxane | 132.76 | ***C***HCH$_2$ | diallyl carbonate |
| 2.31 | $CH_3$ | hexamethyldisiloxane | 37.27 | ***C***H$_2$CH$_3$ | ethyl methyl ketone | 68.68 | $CH_2$(2,5) | tetrahydrofuran | 134.70 | CH (4) | benzaldehyde |
| 7.87 | CH$_2$***C***H$_3$ | ethyl methyl ketone | 37.54 | $CH_3$ | dimethylformamide | 68.81 | $CH_2$ | diallyl carbonate | 136.11 | C(1) | benzaldehyde |
| 9.07 | $CH_3$ | triethylamine | 38.76 | $NCH_3$ | dimethylacetamide | 70.05 | $CH_2$ | diglyme | 136.65 | CH(2) | imidazole |
| 13.92 | $CH_3$ | ethyl acetate | 39.39 | $CH_3$ | dimethyl sulfoxide | 70.14 | $CH_2$ | 18-crown-6 | 138.27 | CH(4) | pyridine |
| 14.77 | $CH_3$ | diethyl ether | 42.02 | $CH_2$(2,6) | cyclohexanone | 70.36 | (CH$_3$)$_3$***C*** | *tert*-butyl alcohol | 143.57 | CH(2,5) | furan |
| 17.47 | $CH_3$ | ethanol | 42.13 | $CH_2$ | dimethyl malonate | 71.49 | $CH_2$ | 1,2-dimethoxyethane | 149.18 | CH(2,6) | pyridine |
| 21.00 | $CH_3$ | allyl acetate | 46.83 | $CH_2$(2,5) | pyrrolidine | 71.63 | $CH_2$ | diglyme | 157.78 | CO | diallyl carbonate |
| 21.03 | $CH_3$ | acetic acid | 47.19 | $CH_2$ | triethylamine | 96.73 | $CCl_4$ | carbon tetrachloride | 163.96 | CO | dimethyl carbonate |
| 21.09 | $CH_3$ | dimethylacetamide | 49.50[12] | $CH_3$ | methanol | 107.83 | CH(3,4) | pyrrole | 165.53 | CH | dimethylformamide |
| 21.15 | ***C***H$_3$CO | ethyl acetate | 53.65 | $CH_3$ | dimethyl malonate | 110.23 | CH(3,4) | furan | 170.12 | $CO_2$ | dimethyl malonate |
| 24.38 | $CH_3$ | 2-propanol | 55.81 | $CH_3$ | dimethyl carbonate | 118.75 | CH***C***H$_2$ | diallyl carbonate | 174.57 | CO | dimethylacetamide |
| 24.77 | $CH_2$(4) | cyclohexanone | 58.05 | $CH_2$ | ethanol | 119.03 | CH***C***H$_2$ | allyl acetate | 174.78 | CO | allyl acetate |
| 25.67 | $CH_2$(3,4) | tetrahydrofuran | 58.67 | $CH_3$ | diglyme | 119.06 | CH(2,5) | pyrrole | 175.26 | CO | ethyl acetate |
| 25.86 | $CH_2$(3,4) | pyrrolidine | 58.67 | $CH_3$ | 1,2-dimethoxyethane | 119.68 | CN | acetonitrile | 177.21 | CO | acetic acid |
| 27.50 | $CH_2$(3,5) | cyclohexanone | 62.32 | $CH_2$ | ethyl acetate | 122.43 | CH(4,5) | imidazole | 191.67 | HCO | benzaldehyde |
| 29.49 | ***C***H$_3$CO | ethyl methyl ketone | 63.17 | $CH_2$ | ethylene glycol | 125.12 | CH(3,5) | pyridine | 197.25 | $CS_2$ | carbon disulfide |
| 30.29 | (***C***H$_3$)$_3$C | *tert*-butyl alcohol | 63.22 | $CH_3$ | nitromethane | 129.48 | CH(3,5) | benzaldehyde | 215.94 | CO | acetone |
| 30.89 | $CH_3$ | acetone | 64.88 | CH | 2-propanol | 130.09 | CH(2,6) | benzaldehyde | 218.43 | CO | ethyl methyl ketone |
| 32.03 | $CH_3$ | dimethylformamide | 66.42 | $CH_2$ | diethyl ether | 132.48 | ***C***HCH$_2$ | allyl acetate | 221.22 | CO | cyclohexanone |
| 35.03 | $NCH_3$ | dimethylacetamide | 66.52 | $CH_2$ | 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 $CDCl_3$, $C_6D_6$, $(CD_3)_2CO$, $(CD_3)_2SO$, $CD_3CN$, $CD_3OD$, and $D_2O$ were previously reported in ref 1.

(3) A signal for HDO is also observed in $(CD_3)_2SO$ (3.30 ppm) and $(CD_3)_2CO$ (2.81 ppm), often seen as a 1:1:1 triplet ($^2J_{H,D}$ = 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 $CH_2$ and the OH protons may be observed ($J$ = 5 Hz).

(7) In $CD_3CN$, the OH proton was seen as a multiplet at 2.69 ppm, as well as extra coupling to the $CH_2$ peak.

(8) Apiezon-brand H grease.

(9) In some solvents, the coupling interaction between the $CH_3$ and the OH protons may be observed ($J$ = 5.5 Hz).

(10) Pyrrolidine was observed to react with the solvent $(CD_3)_2CO$.

(11) Phosphorus coupling was observed ($^2J_{PC}$ = 3 Hz).

(12) Internal reference; see Experimental Section in text.