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

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Tables of $^{1}H$ and $^{13}C$ NMR chemical shifts have been compiled for common organic compounds often used as reagents or found as products or contaminants in deuterated organic solvents. Building upon the work of Gottlieb, Kotlyar, and Nudelman in the *Journal of Organic Chemistry,* signals for common impurities are now reported in additional NMR solvents (tetrahydrofuran-$d_8$, toluene-$d_8$, dichloromethane-$d_2$, chlorobenzene-$d_5$, and 2,2,2-trifluoroethanol-$d_3$) which are frequently used in organometallic laboratories. Chemical shifts for other organics which are often used as reagents or internal standards or are found as products in organometallic chemistry are also reported for all the listed solvents.

Hanging above the desk of most every chemist whose work relies heavily on using NMR spectroscopy[1] is *NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities* by Gottlieb, Kotlyar, and Nudelman.[2] By compiling the chemical shifts of a large number of contaminants commonly encountered in synthetic chemistry, the publication has become an essential reference, allowing for easy identification of known impurities in a variety of deuterated organic solvents. However, despite the utility of Gottlieb et al.'s work,[3] the chemical shifts of impurities in a number of NMR solvents often used by organometallic chemists were not included. Tetrahydrofuran-$d_8$ (THF-$d_8$), toluene-$d_8$, dichloromethane-$d_2$ ($CD_2Cl_2$), chlorobenzene-$d_5$ ($C_6D_5Cl$), and 2,2,2-trifluoroethanol-$d_3$ (TFE-$d_3$) are commonplace in laboratories practicing inorganic syntheses. Therefore, we have expanded the spectral data compilation with the inclusion of chemical shifts of common impurities recorded in the deuterated solvents heavily employed in our organometallic laboratories. The chemical shifts of various gases (hydrogen, methane, ethane, propane, ethylene, propylene, and carbon dioxide) often encountered as reagents or products in organometallic reactions, along with organic compounds relevant to organometallic chemists (allyl acetate, benzaldehyde, carbon disulfide, carbon tetrachloride, 18-crown-6, cyclohexanone, diallyl carbonate, dimethyl carbonate, dimethyl malonate, furan, Apiezon H grease, hexamethylbenzene, hexamethyldisiloxane, imidazole, pyrrole, and pyrrolidine), have also been added to this expanded list.

## Experimental Section

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

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(1) For general information on $^{1}H$ and $^{13}C\{^{1}H\}$ NMR spectroscopy, see: Balcí, M. *Basic $^{1}H$- and $^{13}C$-NMR Spectroscopy*; Elsevier: Amsterdam, 2005.

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

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

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**Table 1. $^1$H NMR Data[a]**

| | 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 | | | 1.72 | 5.32 | 7.26 | 2.08 | 7.16 | 6.96 | 2.05 | 2.50 | 1.94 | 5.02 | 3.31 | 4.79 |
| signals | | | 3.58 | | | 6.97 | | 6.99 | | | | 3.88 | | |
| | | | | | | 7.01 | | 7.14 | | | | | | |
| | | | | | | 7.09 | | | | | | | | |
| water | OH | s | 2.46 | 1.52 | 1.56 | 0.43 | 0.40 | 1.03 | 2.84[b] | 3.33[b] | 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 |
| 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[c] | 3.16 | | | 0.58 | 0.63 | 1.30 | | 4.19 | 2.18 | 2.20 | | |
| chloroform | CH | s | 7.89 | 7.32 | 7.26 | 6.10 | 6.15 | 6.74 | 8.02 | 8.32 | 7.58 | 7.33 | 7.90 | |
| 18-crown-6 | $CH_2$ | s | 3.57 | 3.59 | 3.67 | 3.36 | 3.39 | 3.41 | 3.59 | 3.51 | 3.51 | 3.64 | 3.64 | 3.80 |
| cyclohexane | $CH_2$ | s | 1.44 | 1.44 | 1.43 | 1.40 | 1.40 | 1.37 | 1.43 | 1.40 | 1.44 | 1.47 | 1.45 | |
| 1,2-dichloroethane | $CH_2$ | s | 3.77 | 3.76 | 3.73 | 2.91 | 2.90 | 3.26 | 3.87 | 3.90 | 3.81 | 3.71 | 3.78 | |
| dichloromethane | $CH_2$ | s | 5.51 | 5.33 | 5.30 | 4.32 | 4.27 | 4.77 | 5.63 | 5.76 | 5.44 | 5.24 | 5.49 | |
| diethyl ether | $CH_3$ | t, 7 | 1.12 | 1.15 | 1.21 | 1.10 | 1.11 | 1.10 | 1.11 | 1.09 | 1.12 | 1.20 | 1.18 | 1.17 |
| | $CH_2$ | q, 7 | 3.38 | 3.43 | 3.48 | 3.25 | 3.26 | 3.31 | 3.41 | 3.38 | 3.42 | 3.58 | 3.49 | 3.56 |
| diglyme | $CH_2$ | m | 3.43 | 3.57 | 3.65 | 3.43 | 3.46 | 3.49 | 3.56 | 3.51 | 3.53 | 3.67 | 3.61 | 3.67 |
| | $CH_2$ | m | 3.53 | 3.50 | 3.57 | 3.31 | 3.34 | 3.37 | 3.47 | 3.38 | 3.45 | 3.62 | 3.58 | 3.61 |
| | $OCH_3$ | s | 3.28 | 3.33 | 3.39 | 3.12 | 3.11 | 3.16 | 3.28 | 3.24 | 3.29 | 3.41 | 3.35 | 3.37 |
| dimethylformamide | CH | s | 7.91 | 7.96 | 8.02 | 7.57 | 7.63 | 7.73 | 7.96 | 7.95 | 7.92 | 7.86 | 7.97 | 7.92 |
| | $CH_3$ | s | 2.88 | 2.91 | 2.96 | 2.37 | 2.36 | 2.51 | 2.94 | 2.89 | 2.89 | 2.98 | 2.99 | 3.01 |
| | $CH_3$ | s | 2.76 | 2.82 | 2.88 | 1.96 | 1.86 | 2.30 | 2.78 | 2.73 | 2.77 | 2.88 | 2.86 | 2.85 |
| 1,4-dioxane | $CH_2$ | s | 3.56 | 3.65 | 3.71 | 3.33 | 3.35 | 3.45 | 3.59 | 3.57 | 3.60 | 3.76 | 3.66 | 3.75 |
| DME | $CH_3$ | s | 3.28 | 3.34 | 3.40 | 3.12 | 3.12 | 3.17 | 3.28 | 3.24 | 3.28 | 3.40 | 3.35 | 3.37 |
| | $CH_2$ | s | 3.43 | 3.49 | 3.55 | 3.31 | 3.33 | 3.37 | 3.46 | 3.43 | 3.45 | 3.61 | 3.52 | 3.60 |
| ethane | $CH_3$ | s | 0.85 | 0.85 | 0.87 | 0.81 | 0.80 | 0.79 | 0.83 | 0.82 | 0.85 | 0.85 | 0.85 | 0.82 |
| ethanol | $CH_3$ | t, 7 | 1.10 | 1.19 | 1.25 | 0.97 | 0.96 | 1.06 | 1.12 | 1.06 | 1.12 | 1.22 | 1.19 | 1.17 |
| | $CH_2$ | q, 7[d] | 3.51 | 3.66 | 3.72 | 3.36 | 3.34 | 3.51 | 3.57 | 3.44 | 3.54 | 3.71 | 3.60 | 3.65 |
| | OH | s[c,d] | 3.30 | 1.33 | 1.32 | 0.83 | 0.50 | 1.39 | 3.39 | 4.63 | 2.47 | | | |
| ethyl acetate | $CH_3CO$ | s | 1.94 | 2.00 | 2.05 | 1.69 | 1.65 | 1.78 | 1.97 | 1.99 | 1.97 | 2.03 | 2.01 | 2.07 |
| | $CH_2CH_3$ | q, 7 | 4.04 | 4.08 | 4.12 | 3.87 | 3.89 | 3.96 | 4.05 | 4.03 | 4.06 | 4.14 | 4.09 | 4.14 |
| | $CH_2CH_3$ | t, 7 | 1.19 | 1.23 | 1.26 | 0.94 | 0.92 | 1.04 | 1.20 | 1.17 | 1.20 | 1.26 | 1.24 | 1.24 |
| ethylene | $CH_2$ | s | 5.36 | 5.40 | 5.40 | 5.25 | 5.25 | 5.29 | 5.38 | 5.41 | 5.41 | 5.40 | 5.39 | 5.44 |
| ethylene glycol | $CH_2$ | s[e] | 3.48 | 3.66 | 3.76 | 3.36 | 3.41 | 3.58 | 3.28 | 3.34 | 3.51 | 3.72 | 3.59 | 3.65 |
| H grease[f] | $CH_3$ | m | 0.85−0.91 | 0.84−0.90 | 0.84−0.87 | 0.89−0.96 | 0.90−0.98 | 0.86−0.92 | 0.90 | 0.82−0.88 | | 0.88−0.94 | 0.86−0.93 | |
| | $CH_2$ | br s | 1.29 | 1.27 | 1.25 | 1.33 | 1.32 | 1.30 | 1.29 | 1.24 | | 1.33 | 1.29 | |
| hexamethylbenzene | $CH_3$ | s | 2.18 | 2.20 | 2.24 | 2.10 | 2.13 | 2.10 | 2.17 | 2.14 | 2.19 | 2.24 | 2.19 | |
| *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 | |
| HMDSO | $CH_3$ | s | 0.07 | 0.07 | 0.07 | 0.10 | 0.12 | 0.10 | 0.07 | 0.06 | 0.07 | 0.08 | 0.07 | 0.28 |
| HMPA | $CH_3$ | 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[g] | 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[c,g] | 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 | 1.50 | 1.17 |
| | CH | sept, 6 | 3.82 | 3.97 | 4.04 | 3.65 | 3.67 | 3.82 | 3.90 | 3.78 | 3.87 | 4.05 | 3.92 | 4.02 |
| propylene | $CH_3$ | 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 |
| 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[h] | $CH_2$(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 |

[a] Except for the compounds in solutions 8−10, as well as the gas samples, hexamethylbenzene, and the corrected values mentioned in the Supporting Information, all data for the solvents $CDCl_3$, $C_6D_6$, $(CD_3)_2CO$, $(CD_3)_2SO$, $CD_3CN$, $CD_3OD$, and $D_2O$ were previously reported in ref 2. [b] 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). [c] Not all OH signals were observable. [d] In some solvents, the coupling interaction between the $CH_2$ and the OH protons may be observed ($J$ = 5 Hz). [e] In $CD_3CN$, the OH proton was seen as a multiplet at 2.69 ppm, as well as extra coupling to the $CH_2$ resonance. [f] Apiezon brand H grease. [g] In some solvents, a coupling interaction between the $CH_3$ and the OH protons may be observed ($J$ = 5.5 Hz). [h] Pyrrolidine was observed to react with $(CD_3)_2CO$.

**Table 2. $^{13}C\{^1H\}$ NMR Data[a]**

| | 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 |
| 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 | $(CH_3)_3C$ | 67.50 | 69.11 | 69.15 | 68.12 | 68.19 | 68.19 | 68.13 | 66.88 | 68.74 | 72.35 | 69.40 | 70.36 |
| | $(CH_3)_3C$ | 30.57 | 31.46 | 31.25 | 30.49 | 30.47 | 31.13 | 30.72 | 30.38 | 30.68 | 31.07 | 30.91 | 30.29 |
| 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 | |
| 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 |
| dimethylformamide | CH | 161.96 | 162.57 | 162.62 | 161.93 | 162.13 | 162.01 | 162.79 | 162.29 | 163.31 | 166.01 | 164.73 | 165.53 |
| | $CH_3$ | 35.65 | 36.56 | 36.50 | 35.22 | 35.25 | 35.45 | 36.15 | 35.73 | 36.57 | 37.76 | 36.89 | 37.54 |
| | $CH_3$ | 30.70 | 31.39 | 31.45 | 30.64 | 30.72 | 30.71 | 31.03 | 30.73 | 31.32 | 30.96 | 31.61 | 32.03 |
| 1,4-dioxane | $CH_2$ | 67.65 | 67.47 | 67.14 | 67.17 | 67.16 | 66.95 | 67.60 | 66.36 | 67.72 | 68.52 | 68.11 | 67.19 |
| DME | $CH_3$ | 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 |
| ethane | $CH_3$ | 6.79 | 6.91 | 6.89 | 6.94 | 6.96 | 6.91 | 6.88 | 6.61 | 6.99 | 7.01 | 6.98 | |
| ethanol | $CH_3$ | 18.90 | 18.69 | 18.41 | 18.78 | 18.72 | 18.55 | 18.89 | 18.51 | 18.80 | 18.11 | 18.40 | 17.47 |
| | $CH_2$ | 57.60 | 58.57 | 58.28 | 57.81 | 57.86 | 57.63 | 57.72 | 56.07 | 57.96 | 59.68 | 58.26 | 58.05 |
| ethyl acetate | $CH_3CO$ | 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 |
| ethylene | $CH_2$ | 123.09 | 123.20 | 123.13 | 122.92 | 122.96 | 122.95 | 123.47 | 123.52 | 123.69 | 124.08 | 123.46 | |
| ethylene glycol | $CH_2$ | 64.35 | 64.08 | 63.79 | 64.29 | 64.34 | 64.03 | 64.26 | 62.76 | 64.22 | 64.87 | 64.30 | 63.17 |
| H grease[b] | $CH_2$ | 30.45 | 30.14 | 29.71 | 30.31 | 30.22 | 30.11 | | | | | | |
| hexamethylbenzene | C | 131.88 | 132.09 | 132.21 | 131.72 | 131.79 | 131.54 | 132.22 | 131.10 | 132.61 | 134.04 | 132.53 | |
| | $CH_3$ | 16.71 | 16.93 | 16.98 | 16.84 | 16.95 | 16.68 | 16.86 | 16.60 | 16.94 | 17.04 | 16.90 | |
| *n*-hexane | $CH_3$ | 14.22 | 14.28 | 14.14 | 14.34 | 14.32 | 14.18 | 14.34 | 13.88 | 14.43 | 14.63 | 14.45 | |
| | $CH_2$(2,5) | 23.33 | 23.07 | 22.70 | 23.12 | 23.04 | 22.86 | 23.28 | 22.05 | 23.40 | 24.06 | 23.68 | |
| | $CH_2$(3,4) | 32.34 | 32.01 | 31.64 | 32.06 | 31.96 | 31.77 | 32.30 | 30.95 | 32.36 | 33.17 | 32.73 | |
| HMDSO | $CH_3$ | 1.83 | 1.96 | 1.97 | 1.99 | 2.05 | 1.92 | 2.01 | 1.96 | 2.07 | 2.09 | 1.99 | 2.31 |
| HMPA[c] | $CH_3$ | 36.89 | 36.99 | 36.87 | 36.80 | 36.88 | 36.64 | 37.04 | 36.42 | 37.10 | 37.21 | 37.00 | 36.46 |
| imidazole | CH(2) | 135.72 | 135.76 | 135.38 | 135.57 | 135.76 | 135.50 | 135.89 | 135.15 | 136.33 | 136.58 | 136.31 | 136.65 |
| | CH(4,5) | 122.20 | 122.16 | 122.00 | 122.13 | 122.16 | 121.96 | 122.31 | 121.55 | 122.78 | 122.93 | 122.60 | 122.43 |
| methane | $CH_4$ | −4.90 | −4.33 | −4.63 | −4.34 | −4.29 | −4.33 | −5.33 | −4.01 | −4.61 | −5.88 | −4.90 | |
| methanol | $CH_3$ | 49.64 | 50.45 | 50.41 | 49.90 | 49.97 | 49.66 | 49.77 | 48.59 | 49.90 | 50.67 | 49.86 | 49.50[d] |
| nitromethane | $CH_3$ | 62.49 | 63.03 | 62.50 | 61.14 | 61.16 | 61.68 | 63.21 | 63.28 | 63.66 | 63.17 | 63.08 | 63.22 |
| *n*-pentane | $CH_3$ | 14.18 | 14.24 | 14.08 | 14.27 | 14.25 | 14.10 | 14.29 | 13.28 | 14.37 | 14.54 | 14.39 | |
| | $CH_2$(2,4) | 23.00 | 22.77 | 22.38 | 22.79 | 22.72 | 22.54 | 22.98 | 21.70 | 23.08 | 23.75 | 23.38 | |
| | $CH_2$(3) | 34.87 | 34.57 | 34.16 | 34.54 | 34.45 | 34.26 | 34.83 | 33.48 | 34.89 | 35.76 | 35.30 | |
| propane | $CH_3$ | 16.60 | 16.63 | 16.63 | 16.65 | 16.66 | 16.56 | 16.68 | 16.34 | 16.73 | 16.93 | 16.80 | |
| | $CH_2$ | 16.82 | 16.63 | 16.37 | 16.63 | 16.60 | 16.48 | 16.78 | 15.67 | 16.91 | 17.46 | 17.19 | |
| 2-propanol | $CH_3$ | 25.70 | 25.43 | 25.14 | 25.24 | 25.18 | 25.14 | 25.67 | 25.43 | 25.55 | 25.21 | 25.27 | 24.38 |
| | CH | 66.14 | 64.67 | 64.50 | 64.12 | 64.23 | 64.18 | 63.85 | 64.92 | 64.30 | 66.69 | 64.71 | 64.88 |
| propylene | $CH_3$ | 19.27 | 19.47 | 19.50 | 19.32 | 19.38 | 19.32 | 19.42 | 19.20 | 19.48 | 19.63 | 19.50 | |
| | $CH_2$ | 115.74 | 115.70 | 115.74 | 115.89 | 115.92 | 115.86 | 116.03 | 116.07 | 116.12 | 116.38 | 116.04 | |
| | CH | 134.02 | 134.21 | 133.91 | 133.61 | 133.69 | 133.57 | 134.34 | 133.55 | 134.78 | 136.00 | 134.61 | |
| pyridine | CH(2,6) | 150.57 | 150.27 | 149.90 | 150.25 | 150.27 | 149.93 | 150.67 | 149.58 | 150.76 | 149.76 | 150.07 | 149.18 |
| | CH(3,5) | 124.08 | 124.06 | 123.75 | 123.46 | 123.58 | 123.49 | 124.57 | 123.84 | 127.76 | 126.27 | 125.53 | 125.12 |
| | CH(4) | 135.99 | 136.16 | 135.96 | 135.17 | 135.28 | 135.32 | 136.56 | 136.05 | 136.89 | 139.62 | 138.35 | 138.27 |
| pyrrole | CH(2,5) | 118.03 | 117.93 | 117.77 | 117.61 | 117.78 | 117.65 | 117.98 | 117.32 | 118.47 | 119.61 | 118.28 | 119.06 |
| | CH(3,4) | 107.74 | 108.02 | 107.98 | 108.15 | 108.21 | 108.03 | 108.04 | 107.07 | 108.31 | 108.85 | 108.11 | 107.83 |
| pyrrolidine[e] | $CH_2$(2,5) | 45.82 | 47.02 | 46.93 | 47.12 | 46.86 | 46.75 | | 46.51 | 47.57 | 47.43 | 47.23 | 46.83 |
| | $CH_2$(3,4) | 26.17 | 25.83 | 25.56 | 25.75 | 25.65 | 25.59 | | 25.26 | 26.34 | 25.73 | 26.29 | 25.86 |
| silicone grease | $CH_3$ | 1.20 | 1.22 | 1.19 | 1.37 | 1.38 | 1.09 | 1.40 | | | 2.87 | 2.10 | |
| tetrahydrofuran | $CH_2$(2,5) | 68.03 | 68.16 | 67.97 | 67.75 | 67.80 | 67.64 | 68.07 | 67.03 | 68.33 | 69.53 | 68.83 | 68.68 |
| | $CH_2$(3,4) | 26.19 | 25.98 | 25.62 | 25.79 | 25.72 | 25.68 | 26.15 | 25.14 | 26.27 | 26.69 | 26.48 | 25.67 |
| toluene | $CH_3$ | 21.29 | 21.53 | 21.46 | 21.37 | 21.10 | 21.23 | 21.46 | 20.99 | 21.50 | 21.62 | 21.50 | |
| | C(1) | 138.24 | 138.36 | 137.89 | 137.84 | 137.91 | 137.65 | 138.48 | 137.35 | 138.90 | 139.92 | 138.85 | |
| | CH(2,6) | 129.47 | 129.35 | 129.07 | 129.33 | 129.33 | 129.12 | 129.76 | 128.88 | 129.94 | 130.58 | 129.91 | |
| | CH(3,5) | 128.71 | 128.54 | 128.26 | 128.51 | 128.56 | 128.31 | 129.03 | 128.18 | 129.23 | 129.79 | 129.20 | |
| | CH(4) | 125.84 | 125.62 | 125.33 | 125.66 | 125.68 | 125.43 | 126.12 | 125.29 | 126.28 | 126.82 | 126.29 | |
| triethylamine | $CH_3$ | 12.51 | 12.12 | 11.61 | 12.39 | 12.35 | 11.87 | 12.49 | 11.74 | 12.38 | 9.51 | 11.09 | 9.07 |
| | $CH_2$ | 47.18 | 46.75 | 46.25 | 46.82 | 46.77 | 46.36 | 47.07 | 45.74 | 47.10 | 48.45 | 46.96 | 47.19 |

[a] Except for the compounds in solutions 8−10, as well as the gas samples, hexamethylbenzene, and the corrected values mentioned in the Supporting Information, all data for the solvents $CDCl_3$, $C_6D_6$, $(CD_3)_2CO$, $(CD_3)_2SO$, $CD_3CN$, $CD_3OD$, and $D_2O$ were previously reported in ref 2. [b] Apiezon brand H grease. [c] Phosphorus coupling was observed ($^2J_{PC}$ = 3 Hz). [d] Internal reference; see text. [e] Pyrrolidine was observed to react with $(CD_3)_2CO$.

1,2-dimethoxyethane (DME), nitromethane, poly(dimethylsiloxane) (silicone grease), triethylamine; (solution 7) diglyme, dimethylacetamide, ethylene glycol, ethyl methyl ketone; (solution 8) allyl acetate, 2,6-di-*tert*-butyl-4-methoxyphenol (BHA), long-chain, linear aliphatic hydrocarbons from pump oil;[4] (solution 9) benzaldehyde, carbon disulfide, carbon tetrachloride, cyclohexanone, dimethyl malonate, furan, Apiezon H grease (H grease); (solution 10) 18-crown-6, diallyl carbonate, dimethyl carbonate, hexamethyldisiloxane (HMDSO), imidazole, pyrrole, pyrrolidine.[5] In the case of TFE-$d_3$, nitromethane was omitted from solution 6 and run separately, since the protons of nitromethane exchange with deuterium from TFE-$d_3$ in the presence of triethylamine. In the case of $(CD_3)_2CO$, pyrrolidine was omitted from solution 10, since the two compounds were observed to react with each other. The gases used in this study included hydrogen, methane, ethane, propane, ethylene, propylene, and carbon dioxide.

Before examining the various standard contaminant solutions by $^1$H NMR spectroscopy, solvent residual signals[6] and chemical shifts for $H_2O$[7] for each NMR solvent were referenced against tetramethylsilane (TMS, $\delta$ 0 ppm) and reported. Before collecting $^{13}C\{^1H\}$ NMR spectral data, solvent signals[6] were recorded with reference to the signal of a TMS internal standard. For $D_2O$, $^1$H NMR spectra were referenced to the methyl signal ($\delta$ 0 ppm) of sodium 3-(trimethylsilyl)propanesulfonate,[8,9] and $^{13}C\{^1H\}$ NMR spectra were referenced to the signal for the methyl group of methanol (one drop, added as an internal standard), which was set to 49.50 ppm.[2]

In a typical experiment for collecting $^1$H NMR spectral data, a 3 $\mu$L sample of a standard contaminant solution was added to an NMR tube containing approximately 0.4 mL of a deuterated solvent. For $^{13}C\{^1H\}$ NMR spectral data collection, an approximately 50 $\mu$L sample of the standard contaminant solution was added. When there was any uncertainty in the assignment of a resonance, the solution was spiked with an additional 1−2 $\mu$L of the impurity in question to accurately identify its chemical shift. In cases where the chemical shifts of resonances were highly dependent on the concentration of the impurities present, ambiguous resonances were instead resolved via gradient-selected heteronuclear single-quantum coherence (gs-HSQC) and gradient-selected heteronuclear multiple-quantum coherence (gs-HMQC) NMR spectroscopies. For the experiments involving gases, a J. Young NMR tube containing approximately 0.4 mL of NMR solvent was first degassed with three freeze−pump−thaw cycles. Using a vacuum line equipped with a gas manifold, 1 atm of the desired gas was added to the tube. Each gas was run separately, degassing between each gas sample.

## Results and Discussion

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

It was noted that the amount of gas dissolved in solution gave $^1$H NMR signal integrations that were qualitatively comparable to those for the solutions made with the 3 $\mu$L additions of the liquid or solid contaminants. However, typically in order to observe signals for the gas samples by $^{13}C\{^1H\}$ NMR spectroscopy, additional time for data collection was required. The solubility of each gas in $D_2O$ was extremely limited, making $^{13}$C detection impractical. Of all the gases, methane required the most number of transients in order to obtain an observable signal by $^{13}C\{^1H\}$ NMR spectroscopy. In most cases, the $^{13}$C chemical shift of methane was acquired through the use of gs-HMQC NMR spectroscopy to provide enhanced sensitivity. In order to reflect what would be observed in typical NMR-scale experiments, $^{13}$C detection was not pursued with isotopically enriched gases. A number of misreported values were discovered in the years since the original publication[10] and in the preparation of this paper. These are detailed in the Supporting Information, and the values are now correctly listed in Tables 1 and 2.

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

(4) VWR brand vacuum pump oil #19.

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

(6) For $^1$H NMR spectra, the solvent residual signals arise from the proton of isotopomers containing one less deuterium atom than the perdeuterated solvent: e.g., $CDHCl_2$ in $CD_2Cl_2$. For $^{13}$C NMR spectra, the solvent signals arise from the $^{13}$C atoms at natural abundance in the perdeuterated solvent.

(7) The chemical shift for $H_2O$ can vary depending on the temperature, [$H_2O$], and the solutes present: e.g., a downfield shift may be observed in the presence of any hydrogen bond acceptors. For more information see page 75 of ref 1.

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

(9) For information on the temperature dependence of HDO chemical shifts in $D_2O$, see ref 2.

(10) The misreported value for acetonitrile in $C_6D_6$ from the original paper[2] was also pointed out by Dr. Jongwook Choi, to whom we are grateful.

**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 | s | 6.68 | 6.73 | 6.76 | 6.83 | 6.93 | 6.83 | 6.72 | 6.62 | 6.73 | 6.87 | 6.71 | - |
| | OH | s[5] | 5.64 | 4.76 | 4.76 | 4.45 | 4.53 | 4.62 | 5.65 | 6.52 | 4.98 | - | 4.85 | - |
| | $ArOCH_3$ | s | 3.68 | 3.73 | 3.77 | 3.48 | 3.48 | 3.61 | 3.72 | 3.66 | 3.72 | 3.79 | 3.72 | - |
| | $ArC(CH_3)_3$ | s | 1.40 | 1.42 | 1.44 | 1.34 | 1.41 | 1.37 | 1.41 | 1.36 | 1.40 | 1.44 | 1.41 | - |
| BHT | ArH | s | 6.92 | 6.97 | 6.98 | 6.99 | 7.05 | 6.97 | 6.96 | 6.87 | 6.97 | 7.06 | 6.92 | - |
| | OH | s[5] | 5.81 | 5.00 | 5.01 | 4.72 | 4.79 | 5.50 | - | 6.65 | 5.20 | - | - | - |
| | $ArCH_3$ | s | 2.21 | 2.25 | 2.27 | 2.23 | 2.24 | 2.20 | 2.22 | 2.18 | 2.22 | 2.24 | 2.21 | - |
| | $ArC(CH_3)_3$ | s | 1.40 | 1.42 | 1.43 | 1.36 | 1.38 | 1.37 | 1.41 | 1.36 | 1.39 | 1.43 | 1.40 | - |
| chloroform | CH | s | 7.89 | 7.32 | 7.26 | 6.10 | 6.15 | 6.74 | 8.02 | 8.32 | 7.58 | 7.33 | 7.90 | - |
| 18-crown-6 | $CH_2$ | s | 3.57 | 3.59 | 3.67 | 3.36 | 3.39 | 3.41 | 3.59 | 3.51 | 3.51 | 3.64 | 3.64 | 3.80 |
| cyclohexane | $CH_2$ | s | 1.44 | 1.44 | 1.43 | 1.40 | 1.40 | 1.37 | 1.43 | 1.40 | 1.44 | 1.47 | 1.45 | - |
| cyclohexanone | $CH_2$(2,6) | t | 2.24 | 2.29 | 2.33 | 1.95 | 1.98 | 2.08 | 2.27 | 2.25 | 2.27 | 2.38 | 2.34 | 2.40 |
| | $CH_2$(3,5) | m | 1.77–1.82 | 1.81–1.87 | 1.84–1.86 | 1.33–1.39 | 1.28–1.37 | 1.48–1.53 | 1.79–1.83 | 1.74–1.78 | 1.79–1.84 | 1.87–1.92 | 1.85–1.87 | 1.85–1.90 |
| | $CH_2$(4) | m | 1.68–1.71 | 1.69–1.72 | 1.71–1.73 | 1.16–1.20 | 1.08–1.16 | 1.33–1.37 | 1.70–1.74 | 1.64–1.66 | 1.67–1.72 | 1.75–1.78 | 1.74–1.76 | 1.70–1.75 |
| diallyl carbonate | C***H***$CH_2$ | ddt | 5.92 | 5.95 | 5.94 | 5.63 | 5.65 | 5.75 | 5.96 | 5.93 | 5.96 | 5.92 | 5.94 | 5.99 |
| | CHC***H***$_2$(1) | ddt | 5.31 | 5.35 | 5.37 | 5.09 | 5.09 | 5.17 | 5.35 | 5.33 | 5.34 | 5.35 | 5.34 | 5.40 |
| | CHC***H***$_2$(2) | ddt | 5.19 | 5.26 | 5.27 | 4.92 | 4.92 | 5.03 | 5.23 | 5.25 | 5.25 | 5.28 | 5.25 | 5.32 |
| | $CH_2$ | ddd | 4.58 | 4.61 | 4.64 | 4.34 | 4.38 | 4.46 | 4.62 | 4.61 | 4.61 | 4.62 | 4.61 | 4.69 |
| 1,2-dichloroethane | $CH_2$ | s | 3.77 | 3.76 | 3.73 | 2.91 | 2.90 | 3.26 | 3.87 | 3.90 | 3.81 | 3.71 | 3.78 | - |
| dichloromethane | $CH_2$ | s | 5.51 | 5.33 | 5.30 | 4.32 | 4.27 | 4.77 | 5.63 | 5.76 | 5.44 | 5.24 | 5.49 | - |
| diethyl ether | $CH_3$ | t, 7 | 1.12 | 1.15 | 1.21 | 1.10 | 1.11 | 1.10 | 1.11 | 1.09 | 1.12 | 1.20 | 1.18 | 1.17 |
| | $CH_2$ | q, 7 | 3.38 | 3.43 | 3.48 | 3.25 | 3.26 | 3.31 | 3.41 | 3.38 | 3.42 | 3.58 | 3.49 | 3.56 |
| diglyme | $CH_2$ | m | 3.43 | 3.57 | 3.65 | 3.43 | 3.46 | 3.49 | 3.56 | 3.51 | 3.53 | 3.67 | 3.61 | 3.67 |
| | $CH_2$ | m | 3.53 | 3.50 | 3.57 | 3.31 | 3.34 | 3.37 | 3.47 | 3.38 | 3.45 | 3.62 | 3.58 | 3.61 |
| | $OCH_3$ | s | 3.28 | 3.33 | 3.39 | 3.12 | 3.11 | 3.16 | 3.28 | 3.24 | 3.29 | 3.41 | 3.35 | 3.37 |
| 1,2-dimethoxyethane | $CH_3$ | s | 3.28 | 3.34 | 3.40 | 3.12 | 3.12 | 3.17 | 3.28 | 3.24 | 3.28 | 3.40 | 3.35 | 3.37 |
| | $CH_2$ | s | 3.43 | 3.49 | 3.55 | 3.31 | 3.33 | 3.37 | 3.46 | 3.43 | 3.45 | 3.61 | 3.52 | 3.60 |
| dimethylacetamide | $CH_3CO$ | s | 1.94 | 2.02 | 2.09 | 1.59 | 1.60 | 1.74 | 1.97 | 1.96 | 1.97 | 2.09 | 2.07 | 2.08 |
| | $NCH_3$ | s | 2.95 | 2.97 | 3.02 | 2.56 | 2.57 | 2.65 | 3.00 | 2.94 | 2.96 | 3.05 | 3.31 | 3.06 |
| | $NCH_3$ | s | 2.82 | 2.87 | 2.94 | 2.11 | 2.05 | 2.42 | 2.83 | 2.78 | 2.83 | 2.94 | 2.92 | 2.90 |
| dimethyl carbonate | $CH_3$ | s | 3.69 | 3.75 | 3.79 | 3.31 | 3.30 | 3.48 | 3.72 | 3.69 | 3.72 | 3.77 | 3.74 | 3.69 |
| dimethyl malonate | $CH_3$ | s | 3.65 | 3.72 | 3.75 | 3.24 | 3.23 | 3.41 | 3.68 | 3.65 | 3.68 | 3.76 | 3.72 | 3.78 |
| | $CH_2$ | s | 3.35 | 3.37 | 3.40 | 2.92 | 2.97 | 3.15 | 3.42 | 3.53 | 3.38 | 3.41 | 3.44 | 3.60 |
| dimethylformamide | CH | s | 7.91 | 7.96 | 8.02 | 7.57 | 7.63 | 7.73 | 7.96 | 7.95 | 7.92 | 7.86 | 7.97 | 7.92 |
| | $CH_3$ | s | 2.88 | 2.91 | 2.96 | 2.37 | 2.36 | 2.51 | 2.94 | 2.89 | 2.89 | 2.98 | 2.99 | 3.01 |
| | $CH_3$ | s | 2.76 | 2.82 | 2.88 | 1.96 | 1.86 | 2.30 | 2.78 | 2.73 | 2.77 | 2.88 | 2.86 | 2.85 |
| dimethyl sulfoxide | $CH_3$ | s | 2.45 | 2.55 | 2.62 | 1.64 | 1.68 | 2.03 | 2.52 | 2.54 | 2.50 | 2.63 | 2.65 | 2.71 |
| 1,4-dioxane | $CH_2$ | s | 3.56 | 3.65 | 3.71 | 3.33 | 3.35 | 3.45 | 3.59 | 3.57 | 3.60 | 3.76 | 3.66 | 3.75 |
| ethane | $CH_3$ | s | 0.85 | 0.85 | 0.87 | 0.81 | 0.80 | 0.79 | 0.83 | 0.82 | 0.85 | 0.85 | 0.85 | 0.82 |
| ethanol | $CH_3$ | t, 7 | 1.10 | 1.19 | 1.25 | 0.97 | 0.96 | 1.06 | 1.12 | 1.06 | 1.12 | 1.22 | 1.19 | 1.17 |
| | $CH_2$ | q, 7[6] | 3.51 | 3.66 | 3.72 | 3.36 | 3.34 | 3.51 | 3.57 | 3.44 | 3.54 | 3.71 | 3.60 | 3.65 |
| | OH | s[5,6] | 3.30 | 1.33 | 1.32 | 0.83 | 0.50 | 1.39 | 3.39 | 4.63 | 2.47 | - | - | - |
| ethyl acetate | $CH_3CO$ | s | 1.94 | 2.00 | 2.05 | 1.69 | 1.65 | 1.78 | 1.97 | 1.99 | 1.97 | 2.03 | 2.01 | 2.07 |
| | C***H***$_2CH_3$ | q, 7 | 4.04 | 4.08 | 4.12 | 3.87 | 3.89 | 3.96 | 4.05 | 4.03 | 4.06 | 4.14 | 4.09 | 4.14 |
| | $CH_2C$***H***$_3$ | t, 7 | 1.19 | 1.23 | 1.26 | 0.94 | 0.92 | 1.04 | 1.20 | 1.17 | 1.20 | 1.26 | 1.24 | 1.24 |
| ethyl methyl ketone | $CH_3CO$ | s | 2.03 | 2.09 | 2.14 | 1.59 | 1.58 | 1.78 | 2.07 | 2.07 | 2.06 | 2.16 | 2.12 | 2.19 |
| | C***H***$_2CH_3$ | q, 7 | 2.39 | 2.43 | 2.46 | 1.82 | 1.81 | 2.06 | 2.45 | 2.43 | 2.43 | 2.49 | 2.50 | 3.18 |
| | $CH_2C$***H***$_3$ | t, 7 | 0.96 | 1.00 | 1.06 | 0.84 | 0.85 | 0.89 | 0.96 | 0.91 | 0.96 | 1.05 | 1.01 | 1.26 |
| ethylene | $CH_2$ | s | 5.36 | 5.40 | 5.40 | 5.25 | 5.25 | 5.29 | 5.38 | 5.41 | 5.41 | 5.40 | 5.39 | 5.44 |
| ethylene glycol | $CH_2$ | s[7] | 3.48 | 3.66 | 3.76 | 3.36 | 3.41 | 3.58 | 3.28 | 3.34 | 3.51 | 3.72 | 3.59 | 3.65 |
| furan | CH(2,5) | dd | 7.48 | 7.46 | 7.45 | 7.10 | 7.13 | 7.24 | 7.56 | 7.67 | 7.52 | 7.44 | 7.49 | 7.57 |
| | CH(3,4) | dd | 6.37 | 6.41 | 6.40 | 6.07 | 6.08 | 6.19 | 6.43 | 6.47 | 6.44 | 6.42 | 6.40 | 6.51 |
| H grease[8] | $CH_3$ | m | 0.85–0.91 | 0.84–0.90 | 0.84–0.87 | 0.89–0.96 | 0.90–0.98 | 0.86–0.92 | 0.90 | 0.82–0.88 | - | 0.88–0.94 | 0.86–0.93 | - |
| | $CH_2$ | br s | 1.29 | 1.27 | 1.25 | 1.33 | 1.32 | 1.30 | 1.29 | 1.24 | - | 1.33 | 1.29 | - |
| hexamethylbenzene | $CH_3$ | s | 2.18 | 2.20 | 2.24 | 2.10 | 2.13 | 2.10 | 2.17 | 2.14 | 2.19 | 2.24 | 2.19 | - |
| hexamethyldisiloxane | $CH_3$ | s | 0.07 | 0.07 | 0.07 | 0.10 | 0.12 | 0.10 | 0.07 | 0.06 | 0.07 | 0.08 | 0.07 | 0.28 |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| *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 | 163.31 | 166.01 | 164.73 | 165.53 |
| | $CH_3$ | 35.65 | 36.56 | 36.50 | 35.22 | 35.25 | 35.45 | 36.15 | 35.73 | 36.57 | 37.76 | 36.89 | 37.54 |
| | $CH_3$ | 30.70 | 31.39 | 31.45 | 30.64 | 30.72 | 30.71 | 31.03 | 30.73 | 31.32 | 30.96 | 31.61 | 32.03 |
| dimethyl sulfoxide | $CH_3$ | 41.21 | 41.33 | 40.76 | 40.41 | 40.03 | 40.27 | 41.23 | 40.45 | 41.31 | 40.06 | 40.45 | 39.39 |
| 1,4-dioxane | $CH_2$ | 67.65 | 67.47 | 67.14 | 67.17 | 67.16 | 66.95 | 67.60 | 66.36 | 67.72 | 68.52 | 68.11 | 67.19 |
| ethane | $CH_3$ | 6.79 | 6.91 | 6.89 | 6.94 | 6.96 | 6.91 | 6.88 | 6.61 | 6.99 | 7.01 | 6.98 | - |
| ethanol | $CH_3$ | 18.90 | 18.69 | 18.41 | 18.78 | 18.72 | 18.55 | 18.89 | 18.51 | 18.80 | 18.11 | 18.40 | 17.47 |
| | $CH_2$ | 57.60 | 58.57 | 58.28 | 57.81 | 57.86 | 57.63 | 57.72 | 56.07 | 57.96 | 59.68 | 58.26 | 58.05 |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ethyl acetate | ***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 | 64.30 | 63.17 |
| furan | CH(2,5) | 143.26 | 142.98 | 142.71 | 142.65 | 142.73 | 142.49 | 143.49 | 142.82 | 143.74 | 144.22 | 143.68 | 143.57 |
| | CH(3,4) | 109.88 | 109.86 | 109.57 | 109.63 | 109.67 | 109.64 | 110.24 | 109.62 | 110.49 | 111.06 | 110.33 | 110.23 |
| H grease[8] | $CH_2$ | 30.45 | 30.14 | 29.71 | 30.31 | 30.22 | 30.11 | - | - | - | - | - | - |
| hexamethylbenzene | C | 131.88 | 132.09 | 132.21 | 131.72 | 131.79 | 131.54 | 132.22 | 131.10 | 132.61 | 134.04 | 132.53 | - |
| | $CH_3$ | 16.71 | 16.93 | 16.98 | 16.84 | 16.95 | 16.68 | 16.86 | 16.60 | 16.94 | 17.04 | 16.90 | - |
| hexamethyldisiloxane | $CH_3$ | 1.83 | 1.96 | 1.97 | 1.99 | 2.05 | 1.92 | 2.01 | 1.96 | 2.07 | 2.09 | 1.99 | 2.31 |
| *n* -hexane | $CH_3$ | 14.22 | 14.28 | 14.14 | 14.34 | 14.32 | 14.18 | 14.34 | 13.88 | 14.43 | 14.63 | 14.45 | - |
| | $CH_2$(2,5) | 23.33 | 23.07 | 22.70 | 23.12 | 23.04 | 22.86 | 23.28 | 22.05 | 23.40 | 24.06 | 23.68 | - |
| | $CH_2$(3,4) | 32.34 | 32.01 | 31.64 | 32.06 | 31.96 | 31.77 | 32.30 | 30.95 | 32.36 | 33.17 | 32.73 | - |
| HMPA[11] | $CH_3$ | 36.89 | 36.99 | 36.87 | 36.80 | 36.88 | 36.64 | 37.04 | 36.42 | 37.10 | 37.21 | 37.00 | 36.46 |
| imidazole | CH(2) | 135.72 | 135.76 | 135.38 | 135.57 | 135.76 | 135.50 | 135.89 | 135.15 | 136.33 | 136.58 | 136.31 | 136.65 |
| | CH(4,5) | 122.20 | 122.16 | 122.00 | 122.13 | 122.16 | 121.96 | 122.31 | 121.55 | 122.78 | 122.93 | 122.60 | 122.43 |
| methane | $CH_4$ | –4.90 | –4.33 | –4.63 | –4.34 | –4.29 | –4.33 | –5.33 | –4.01 | –4.61 | –5.88 | –4.90 | - |
| methanol | $CH_3$ | 49.64 | 50.45 | 50.41 | 49.90 | 49.97 | 49.66 | 49.77 | 48.59 | 49.90 | 50.67 | 49.86 | 49.50[12] |
| nitromethane | $CH_3$ | 62.49 | 63.03 | 62.50 | 61.14 | 61.16 | 61.68 | 63.21 | 63.28 | 63.66 | 63.17 | 63.08 | 63.22 |
| *n* -pentane | $CH_3$ | 14.18 | 14.24 | 14.08 | 14.27 | 14.25 | 14.10 | 14.29 | 13.28 | 14.37 | 14.54 | 14.39 | - |
| | $CH_2$(2,4) | 23.00 | 22.77 | 22.38 | 22.79 | 22.72 | 22.54 | 22.98 | 21.70 | 23.08 | 23.75 | 23.38 | - |
| | $CH_2$(3) | 34.87 | 34.57 | 34.16 | 34.54 | 34.45 | 34.26 | 34.83 | 33.48 | 34.89 | 35.76 | 35.30 | - |
| propane | $CH_3$ | 16.60 | 16.63 | 16.63 | 16.65 | 16.66 | 16.56 | 16.68 | 16.34 | 16.73 | 16.93 | 16.80 | - |
| | $CH_2$ | 16.82 | 16.63 | 16.37 | 16.63 | 16.60 | 16.48 | 16.78 | 15.67 | 16.91 | 17.46 | 17.19 | - |
| 2-propanol | $CH_3$ | 25.70 | 25.43 | 25.14 | 25.24 | 25.18 | 25.14 | 25.67 | 25.43 | 25.55 | 25.21 | 25.27 | 24.38 |
| | CH | 66.14 | 64.67 | 64.50 | 64.12 | 64.23 | 64.18 | 63.85 | 64.92 | 64.30 | 66.69 | 64.71 | 64.88 |
| propylene | $CH_3$ | 19.27 | 19.47 | 19.50 | 19.32 | 19.38 | 19.32 | 19.42 | 19.20 | 19.48 | 19.63 | 19.50 | - |
| | $CH_2$ | 115.74 | 115.70 | 115.74 | 115.89 | 115.92 | 115.86 | 116.03 | 116.07 | 116.12 | 116.38 | 116.04 | - |
| | CH | 134.02 | 134.21 | 133.91 | 133.61 | 133.69 | 133.57 | 134.34 | 133.55 | 134.78 | 136.00 | 134.61 | - |
| pump oil | $CH_2$ | 30.63 | 30.13 | 29.84 | 30.33 | 30.24 | 30.11 | 30.36 | 29.33 | 30.86 | 31.85 | 31.35 | - |
| pyridine | CH(2,6) | 150.57 | 150.27 | 149.90 | 150.25 | 150.27 | 149.93 | 150.67 | 149.58 | 150.76 | 149.76 | 150.07 | 149.18 |
| | CH(3,5) | 124.08 | 124.06 | 123.75 | 123.46 | 123.58 | 123.49 | 124.57 | 123.84 | 127.76 | 126.27 | 125.53 | 125.12 |
| | CH(4) | 135.99 | 136.16 | 135.96 | 135.17 | 135.28 | 135.32 | 136.56 | 136.05 | 136.89 | 139.62 | 138.35 | 138.27 |
| pyrrole | CH(2,5) | 118.03 | 117.93 | 117.77 | 117.61 | 117.78 | 117.65 | 117.98 | 117.32 | 118.47 | 119.61 | 118.28 | 119.06 |
| | CH(3,4) | 107.74 | 108.02 | 107.98 | 108.15 | 108.21 | 108.03 | 108.04 | 107.07 | 108.31 | 108.85 | 108.11 | 107.83 |
| pyrrolidine[10] | $CH_2$(2,5) | 45.82 | 47.02 | 46.93 | 47.12 | 46.86 | 46.75 | - | 46.51 | 47.57 | 47.43 | 47.23 | 46.83 |
| | $CH_2$(3,4) | 26.17 | 25.83 | 25.56 | 25.75 | 25.65 | 25.59 | - | 25.26 | 26.34 | 25.73 | 26.29 | 25.86 |
| silicone grease | $CH_3$ | 1.20 | 1.22 | 1.19 | 1.37 | 1.38 | 1.09 | 1.40 | - | - | 2.87 | 2.10 | - |
| tetrahydrofuran | $CH_2$(2,5) | 68.03 | 68.16 | 67.97 | 67.75 | 67.80 | 67.64 | 68.07 | 67.03 | 68.33 | 69.53 | 68.83 | 68.68 |
| | $CH_2$(3,4) | 26.19 | 25.98 | 25.62 | 25.79 | 25.72 | 25.68 | 26.15 | 25.14 | 26.27 | 26.69 | 26.48 | 25.67 |
| toluene | $CH_3$ | 21.29 | 21.53 | 21.46 | 21.37 | 21.10 | 21.23 | 21.46 | 20.99 | 21.50 | 21.62 | 21.50 | - |
| | C(1) | 138.24 | 138.36 | 137.89 | 137.84 | 137.91 | 137.65 | 138.48 | 137.35 | 138.90 | 139.92 | 138.85 | - |
| | CH(2,6) | 129.47 | 129.35 | 129.07 | 129.33 | 129.33 | 129.12 | 129.76 | 128.88 | 129.94 | 130.58 | 129.91 | - |
| | CH(3,5) | 128.71 | 128.54 | 128.26 | 128.51 | 128.56 | 128.31 | 129.03 | 128.18 | 129.23 | 129.79 | 129.20 | - |
| | CH(4) | 125.84 | 125.62 | 125.33 | 125.66 | 125.68 | 125.43 | 126.12 | 125.29 | 126.28 | 126.82 | 126.29 | - |
| triethylamine | $CH_3$ | 12.51 | 12.12 | 11.61 | 12.39 | 12.35 | 11.87 | 12.49 | 11.74 | 12.38 | 9.51 | 11.09 | 9.07 |
| | $CH_2$ | 47.18 | 46.75 | 46.25 | 46.82 | 46.77 | 46.36 | 47.07 | 45.74 | 47.10 | 48.45 | 46.96 | 47.19 |

**Table S3.** THF-$d_8$ ($^1$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 | $ArCH_3$ | BHT | 4.50 | ddd | $CH_2$ | allyl acetate |
| 0.11 | s | $CH_3$ | silicone grease | 2.24 | t | $CH_2$(2,6) | cyclohexanone | 4.55 | s | $H_2$ | hydrogen |
| 0.19 | s | $CH_4$ | methane | 2.31 | s | $CH_3$ | toluene | 4.58 | ddd | $CH_2$ | diallyl carbonate |
| 0.85 | s | $CH_3$ | ethane | 2.39 | q, 7 | C***H***$_2$$CH_3$ | ethyl methyl ketone | 4.89 | dm, 10 | $CH_2$(1) | propylene |
| 0.85–0.91 | m | $CH_3$ | H grease[8] | 2.45 | s | $CH_3$ | dimethyl sulfoxide | 4.99 | dm, 17 | $CH_2$(2) | propylene |
| 0.86–0.90 | m | $CH_3$ | pump oil | 2.46 | s | OH | water | 5.15 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.89 | t, 7 | $CH_3$ | *n*-hexane | 2.46 | q, 7 | $CH_2$ | triethylamine | 5.19 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 0.89 | t, 7 | $CH_3$ | *n*-pentane | 2.58 | d, 9.5 | $CH_3$ | HMPA | 5.27 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 0.90 | t, 7.3 | $CH_3$ | propane | 2.75 | m | $CH_2$(2,5) | pyrrolidine | 5.31 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 0.96 | t, 7 | $CH_2$C***H***$_3$ | ethyl methyl ketone | 2.76 | s | $CH_3$ | dimethylformamide | 5.36 | s | $CH_2$ | ethylene |
| 0.97 | t, 7 | $CH_3$ | triethylamine | 2.82 | s | $NCH_3$ | dimethylacetamide | 5.51 | s | $CH_2$ | dichloromethane |
| 1.08 | d, 6 | $CH_3$ | 2-propanol | 2.88 | s | $CH_3$ | dimethylformamide | 5.64 | s | OH | BHA |
| 1.10 | t, 7 | $CH_3$ | ethanol | 2.95 | s | $NCH_3$ | dimethylacetamide | 5.79 | m | CH | propylene |
| 1.12 | t, 7 | $CH_3$ | diethyl ether | 3.02 | s[9] | OH | methanol | 5.81 | s | OH | BHT |
| 1.15 | s | $CH_3$ | *tert*-butyl alcohol | 3.16 | s | OH | *tert*-butyl alcohol | 5.90 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.19 | t, 7 | $CH_2$C***H***$_3$ | ethyl acetate | 3.27 | s[9] | $CH_3$ | methanol | 5.92 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.29 | br s | $CH_2$ | H grease[8] | 3.28 | s | $OCH_3$ | diglyme | 6.02 | m | CH(3,4) | pyrrole |
| 1.29 | m | $CH_2$ | *n*-hexane | 3.28 | s | $CH_3$ | 1,2-dimethoxyethane | 6.37 | dd | CH(3,4) | furan |
| 1.29 | br s | $CH_2$ | pump oil | 3.30 | s[6] | OH | ethanol | 6.66 | m | CH(2,5) | pyrrole |
| 1.31 | m | $CH_2$ | *n*-pentane | 3.35 | s | $CH_2$ | dimethyl malonate | 6.68 | s | ArH | BHA |
| 1.33 | sept, 7.3 | $CH_2$ | propane | 3.38 | q, 7 | $CH_2$ | diethyl ether | 6.92 | s | ArH | BHT |
| 1.40 | s | $ArC(CH_3)_3$ | BHA | 3.43 | m | $CH_2$ | diglyme | 6.94 | s | CH(4,5) | imidazole |
| 1.40 | s | $ArC(CH_3)_3$ | BHT | 3.43 | s | $CH_2$ | 1,2-dimethoxyethane | 7.10 | m | CH(2,4,6) | toluene |
| 1.44 | s | $CH_2$ | cyclohexane | 3.48 | s | $CH_2$ | ethylene glycol | 7.19 | m | CH(3,5) | toluene |
| 1.59 | m | $CH_2$(3,4) | pyrrolidine | 3.51 | q, 7[6] | $CH_2$ | ethanol | 7.25 | m | CH(3,5) | pyridine |
| 1.68–1.71 | m | $CH_2$(4) | cyclohexanone | 3.53 | m | $CH_2$ | diglyme | 7.31 | s | CH | benzene |
| 1.69 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.56 | s | $CH_2$ | 1,4-dioxane | 7.48 | dd | CH(2,5) | furan |
| 1.72 | m | CHD(3,4) | THF-$d_8$ residual | 3.57 | s | $CH_2$ | 18-crown-6 | 7.48 | s | CH(2) | imidazole |
| 1.77–1.82 | m | $CH_2$(3,5) | cyclohexanone | 3.58 | m | CHD(2,5) | THF-$d_8$ residual | 7.51–7.55 | m | CH(3,5) | benzaldehyde |
| 1.79 | m | $CH_2$(3,4) | tetrahydrofuran | 3.62 | m | $CH_2$(2,5) | tetrahydrofuran | 7.60–7.64 | m | CH(4) | benzaldehyde |
| 1.89 | s | $CH_3$ | acetic acid | 3.65 | s | $CH_3$ | dimethyl malonate | 7.65 | m | CH(4) | pyridine |
| 1.94 | s | $CH_3CO$ | dimethylacetamide | 3.68 | s | $ArOCH_3$ | BHA | 7.86–7.88 | m | CH(2,6) | benzaldehyde |
| 1.94 | s | $CH_3CO$ | ethyl acetate | 3.69 | s | $CH_3$ | dimethyl carbonate | 7.89 | s | CH | chloroform |
| 1.95 | s | $CH_3$ | acetonitrile | 3.77 | s | $CH_2$ | 1,2-dichloroethane | 7.91 | s | CH | dimethylformamide |
| 1.98 | s | $CH_3$ | allyl acetate | 3.82 | sept, 6 | CH | 2-propanol | 8.54 | m | CH(2,6) | pyridine |
| 2.03 | s | $CH_3CO$ | ethyl methyl ketone | 4.04 | q, 7 | C***H***$_2$$CH_3$ | ethyl acetate | 9.96 | br t | NH | pyrrole |
| 2.05 | s | $CH_3$ | acetone | 4.31 | s | $CH_3$ | nitromethane | 9.98 | s | HCO | benzaldehyde |
| 2.18 | s | $CH_3$ | hexamethylbenzene | | | | | | | | |

**Table S4.** THF-$d_8$ ($^{13}$C{$^1$H} NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.90 | $CH_4$ | methane | 30.17 | $CH_3$ | acetone | 64.35 | $CH_2$ | ethylene glycol | 129.47 | CH(2,6) | toluene |
| 0.45 | $CH_3$ | acetonitrile | 30.45 | $CH_2$ | H grease[8] | 65.31 | $CH_2$ | allyl acetate | 129.56 | CH(3,5) | benzaldehyde |
| 1.20 | $CH_3$ | silicone grease | 30.57 | ($C$H$_3$)$_3$C | *tert*-butyl alcohol | 66.14 | $CH_2$ | diethyl ether | 129.98 | CH(2,6) | benzaldehyde |
| 1.83 | $CH_3$ | hexamethyldisiloxane | 30.63 | $CH_2$ | pump oil | 66.14 | CH | 2-propanol | 131.88 | C | hexamethylbenzene |
| 6.79 | $CH_3$ | ethane | 30.65 | ($C$H$_3$)$_3$C | BHA | 67.21 (p) | $CD_2$(2,5) | THF-$d_8$ signal | 133.08 | $C$HCH$_2$ | diallyl carbonate |
| 7.87 | $CH_2CH_3$ | ethyl methyl ketone | 30.70 | $CH_3$ | dimethylformamide | 67.50 | (CH$_3$)$_3$$C$ | *tert*-butyl alcohol | 133.90 | $C$HCH$_2$ | allyl acetate |
| 12.51 | $CH_3$ | triethylamine | 31.55 | ($C$H$_3$)$_3$C | BHT | 67.65 | $CH_2$ | 1,4-dioxane | 134.02 | CH | propylene |
| 14.18 | $CH_3$ | *n*-pentane | 32.34 | $CH_2$(3,4) | *n*-hexane | 68.03 | $CH_2$(2,5) | tetrahydrofuran | 134.67 | CH (4) | benzaldehyde |
| 14.22 | $CH_3$ | *n*-hexane | 34.60 | $NCH_3$ | dimethylacetamide | 68.58 | $CH_2$ | diallyl carbonate | 135.72 | CH(2) | imidazole |
| 14.37 | $CH_3$ | ethyl acetate | 34.87 | $CH_2$(3) | *n*-pentane | 71.17 | $CH_2$ | diglyme | 135.99 | CH(4) | pyridine |
| 15.49 | $CH_3$ | diethyl ether | 34.91 | (CH$_3$)$_3$$C$ | BHT | 71.34 | $CH_2$ | 18-crown-6 | 137.78 | C(1) | benzaldehyde |
| 16.60 | $CH_3$ | propane | 35.51 | (CH$_3$)$_3$$C$ | BHA | 72.58 | $CH_2$ | 1,2-dimethoxyethane | 137.93 | C(2,6) | BHT |
| 16.71 | $CH_3$ | hexamethylbenzene | 35.65 | $CH_3$ | dimethylformamide | 72.72 | $CH_2$ | diglyme | 138.24 | C(1) | toluene |
| 16.82 | $CH_2$ | propane | 36.59 | $C$H$_2$CH$_3$ | ethyl methyl ketone | 79.24 | CH | chloroform | 140.07 | C(4) | BHA |
| 18.90 | $CH_3$ | ethanol | 36.89 (d) | $CH_3$ | HMPA[11] | 96.89 | $CCl_4$ | carbon tetrachloride | 143.26 | CH(2,5) | furan |
| 19.27 | $CH_3$ | propylene | 37.56 | $NCH_3$ | dimethylacetamide | 107.74 | CH(3,4) | pyrrole | 148.62 | C(2,6) | BHA |
| 20.13 | $CH_3$ | acetic acid | 41.15 | $CH_2$ | dimethyl malonate | 109.88 | CH(3,4) | furan | 150.57 | CH(2,6) | pyridine |
| 20.45 | $CH_3$ | allyl acetate | 41.21 | $CH_3$ | dimethyl sulfoxide | 110.94 | CH(3,5) | BHA | 152.48 | C(1) | BHT |
| 20.45 | $C$H$_3$CO | ethyl acetate | 42.17 | $CH_2$(2,6) | cyclohexanone | 115.74 | $CH_2$ | propylene | 154.07 | C(1) | BHA |
| 21.15 | $CH_3$ | dimethylacetamide | 44.64 | $CH_2$ | 1,2-dichloroethane | 116.79 | CN | acetonitrile | 155.36 | CO | diallyl carbonate |
| 21.21 | $CH_3$Ar | BHT | 45.82 | $CH_2$(2,5) | pyrrolidine | 117.58 | CH$C$H$_2$ | allyl acetate | 156.91 | CO | dimethyl carbonate |
| 21.29 | $CH_3$ | toluene | 47.18 | $CH_2$ | triethylamine | 117.70 | CH$C$H$_2$ | diallyl carbonate | 161.96 | CH | dimethylformamide |
| 23.00 | $CH_2$(2,4) | *n*-pentane | 49.64 | $CH_3$ | methanol | 118.03 | CH(2,5) | pyrrole | 167.14 | $CO_2$ | dimethyl malonate |
| 23.33 | $CH_2$(2,5) | *n*-hexane | 52.07 | $CH_3$ | dimethyl malonate | 122.20 | CH(4,5) | imidazole | 169.77 | CO | dimethylacetamide |
| 25.31 (p) | $CD_2$(3,4) | THF-$d_8$ signal | 54.58 | $CH_3$ | dimethyl carbonate | 123.09 | $CH_2$ | ethylene | 170.14 | CO | allyl acetate |
| 25.70 | $CH_3$ | 2-propanol | 54.67 | $CH_2$ | dichloromethane | 124.08 | CH(3,5) | pyridine | 170.32 | CO | ethyl acetate |
| 25.76 | $CH_2$(4) | cyclohexanone | 55.39 | $CH_3O$ | BHA | 125.69 | $CO_2$ | carbon dioxide | 171.69 | CO | acetic acid |
| 26.17 | $CH_2$(3,4) | pyrrolidine | 57.60 | $CH_2$ | ethanol | 125.71 | CH(3,5) | BHT | 191.95 | HCO | benzaldehyde |
| 26.19 | $CH_2$(3,4) | tetrahydrofuran | 58.72 | $CH_3$ | diglyme | 125.84 | CH(4) | toluene | 193.37 | $CS_2$ | carbon disulfide |
| 27.58 | $CH_2$ | cyclohexane | 58.72 | $CH_3$ | 1,2-dimethoxyethane | 128.64 | C(4) | BHT | 204.19 | CO | acetone |
| 27.69 | $CH_2$(3,5) | cyclohexanone | 60.30 | $CH_2$ | ethyl acetate | 128.71 | CH(3,5) | toluene | 207.05 | CO | ethyl methyl ketone |
| 28.92 | $C$H$_3$CO | ethyl methyl ketone | 62.49 | $CH_3$ | nitromethane | 128.84 | CH | benzene | 208.79 | CO | cyclohexanone |

**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$_2$C***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$_2$C***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***HCH$_2$ | 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***HCH$_2$ | allyl acetate |
| 7.86 | $CH_2CH_3$ | 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 | $NCH_3$ | dimethylacetamide | 107.98 | CH(3,4) | pyrrole | 147.85 | C(2,6) | BHA |
| 19.50 | $CH_3$ | propylene | 40.76 | $CH_3$ | dimethyl sulfoxide | 109.57 | CH(3,4) | furan | 149.90 | CH(2,6) | pyridine |
| 20.81 | $CH_3$ | acetic acid | 41.11 | $CH_2$ | dimethyl malonate | 110.69 | CH(3,5) | BHA | 151.55 | C(1) | BHT |
| 21.02 | $CH_3$ | allyl acetate | 41.97 | $CH_2$(2,6) | cyclohexanone | 115.74 | $CH_2$ | propylene | 152.57 | C(1) | BHA |
| 21.04 | ***C***H$_3$CO | ethyl acetate | 43.50 | $CH_2$ | 1,2-dichloroethane | 116.43 | CN | acetonitrile | 154.88 | CO | diallyl carbonate |
| 21.20 | $CH_3Ar$ | BHT | 46.25 | $CH_2$ | triethylamine | 117.77 | CH(2,5) | pyrrole | 156.45 | CO | dimethyl carbonate |
| 21.46 | $CH_3$ | toluene | 46.93 | $CH_2$(2,5) | pyrrolidine | 118.34 | CH***C***H$_2$ | allyl acetate | 162.62 | CH | dimethylformamide |
| 21.53 | $CH_3$ | dimethylacetamide | 50.41 | $CH_3$ | methanol | 118.96 | CH***C***H$_2$ | diallyl carbonate | 167.18 | $CO_2$ | dimethyl malonate |
| 22.38 | $CH_2$(2,4) | *n*-pentane | 52.57 | $CH_3$ | dimethyl malonate | 122.00 | CH(4,5) | imidazole | 170.81 | CO | allyl acetate |
| 22.70 | $CH_2$(2,5) | *n*-hexane | 53.52 | $CH_2$ | dichloromethane | 123.13 | $CH_2$ | ethylene | 171.07 | CO | dimethylacetamide |
| 24.97 | $CH_2$(4) | cyclohexanone | 54.89 | $CH_3$ | dimethyl carbonate | 123.75 | CH(3,5) | pyridine | 171.36 | CO | ethyl acetate |
| 25.14 | $CH_3$ | 2-propanol | 55.70 | $CH_3O$ | BHA | 124.99 | $CO_2$ | carbon dioxide | 175.99 | CO | acetic acid |
| 25.56 | $CH_2$(3,4) | pyrrolidine | 58.28 | $CH_2$ | ethanol | 125.33 | CH(4) | toluene | 192.67 | HCO | benzaldehyde |
| 25.62 | $CH_2$(3,4) | tetrahydrofuran | 59.01 | $CH_3$ | diglyme | 125.55 | CH(3,5) | BHT | 192.83 | $CS_2$ | carbon disulfide |
| 26.94 | $CH_2$ | cyclohexane | 59.08 | $CH_3$ | 1,2-dimethoxyethane | 128.26 | CH(3,5) | toluene | 207.07 | CO | acetone |
| 27.00 | $CH_2$(3,5) | cyclohexanone | 60.49 | $CH_2$ | ethyl acetate | 128.27 | C(4) | BHT | 209.56 | CO | ethyl methyl ketone |
| 29.49 | ***C***H$_3$CO | ethyl methyl ketone | 62.50 | $CH_3$ | nitromethane | 128.37 | CH | benzene | 212.57 | CO | cyclohexanone |
| 29.71 | $CH_2$ | H grease[8] | 63.79 | $CH_2$ | ethylene glycol | 129.07 | CH(2,6) | toluene | | | |

**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 | C***H***$_2$CH$_3$ | 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 | CHC***H***$_2$(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 | CHC***H***$_2$(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 | CHC***H***$_2$(1) | allyl acetate |
| 0.84 | t, 7 | $CH_2$C***H***$_3$ | ethyl methyl ketone | 2.11 | s | $CH_3$ | toluene | 5.09 | ddt | CHC***H***$_2$(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 | C***H***$CH_2$ | diallyl carbonate |
| 0.88–0.96 | m | $CH_3$ | pump oil | 2.39 | q, 7 | $CH_2$ | triethylamine | 5.67[4] | t(nfo ABX) | C***H***$CH_2$ | 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 | $CH_2$C***H***$_3$ | 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 | $CH_2$ | diethyl ether | 6.97 | p | CH(4) | Toluene-$d_8$ residual |
| 1.30 | br s | $CH_2$ | pump oil | 3.31 | m | $CH_2$ | diglyme | 6.99 | s | ArH | BHT |
| 1.32 | sept, 7.3 | $CH_2$ | propane | 3.31 | s | $CH_2$ | 1,2-dimethoxyethane | 6.99 | m | CH(4) | pyridine |
| 1.33 | br s | $CH_2$ | H grease[8] | 3.31 | s | $CH_3$ | dimethyl carbonate | 7.01 | s | CH(2,6) | Toluene-$d_8$ residual |
| 1.33–1.39 | m | $CH_2$(3,5) | cyclohexanone | 3.33 | s | $CH_2$ | 1,4-dioxane | 7.03–7.07 | m | CH(4) | benzaldehyde |
| 1.34 | s | $ArC(CH_3)_3$ | BHA | 3.36 | s | $CH_2$ | 18-crown-6 | 7.09 | m | CH(3,5) | Toluene-$d_8$ residual |
| 1.36 | s | $ArC(CH_3)_3$ | BHT | 3.36 | q, 7[6] | $CH_2$ | ethanol | 7.09 | m | CH(3,5) | toluene |
| 1.36 | m | $CH_2$(3,4) | pyrrolidine | 3.36 | s | $CH_2$ | ethylene glycol | 7.10 | dd | CH(2,5) | furan |
| 1.40 | s | $CH_2$ | cyclohexane | 3.43 | m | $CH_2$ | diglyme | 7.12 | s | CH | benzene |
| 1.43 | m | $CH_2$(3,4) | tetrahydrofuran | 3.48 | s | $ArOCH_3$ | BHA | 7.30 | s | CH(2) | imidazole |
| 1.55 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.54 | m | $CH_2$(2,5) | tetrahydrofuran | 7.45–7.47 | m | CH(2,6) | benzaldehyde |
| 1.57 | s | $CH_3$ | acetic acid | 3.65 | sept, 6 | CH | 2-propanol | 7.57 | s | CH | dimethylformamide |
| 1.57 | s | $CH_3$ | acetone | 3.87 | q, 7 | C***H***$_2$CH$_3$ | ethyl acetate | 7.71 | br t | NH | pyrrole |
| 1.59 | s | $CH_3CO$ | dimethylacetamide | 4.32 | s | $CH_2$ | dichloromethane | 8.47 | m | CH(2,6) | pyridine |
| 1.59 | s | $CH_3CO$ | ethyl methyl ketone | 4.34 | ddd | $CH_2$ | allyl acetate | 9.57 | s | HCO | benzaldehyde |
| 1.63 | s | $CH_3$ | allyl acetate | 4.34 | ddd | $CH_2$ | diallyl carbonate | | | | |

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

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.34 | $CH_4$ | methane | 30.30 | ($C$H$_3$)$_3$C | BHA | 64.87 | $CH_2$ | allyl acetate | 129.33 | CH(2,6) | toluene |
| 0.03 | $CH_3$ | acetonitrile | 30.31 | $CH_2$ | H grease[8] | 65.94 | $CH_2$ | diethyl ether | 129.61 | CH(2,6) | benzaldehyde |
| 1.37 | $CH_3$ | silicone grease | 30.33 | $CH_2$ | pump oil | 67.17 | $CH_2$ | 1,4-dioxane | 131.72 | C | hexamethylbenzene |
| 1.99 | $CH_3$ | hexamethyldisiloxane | 30.49 | ($C$H$_3$)$_3$C | *tert*-butyl alcohol | 67.75 | $CH_2$(2,5) | tetrahydrofuran | 132.30 | $C$HCH$_2$ | diallyl carbonate |
| 6.94 | $CH_3$ | ethane | 30.64 | $CH_3$ | dimethylformamide | 68.12 | $(CH_3)_3C$ | *tert*-butyl alcohol | 132.98 | $C$HCH$_2$ | allyl acetate |
| 7.89 | $CH_2CH_3$ | ethyl methyl ketone | 31.39 | ($C$H$_3$)$_3$C | BHT | 68.20 | $CH_2$ | diallyl carbonate | 133.61 | CH | propylene |
| 12.39 | $CH_3$ | triethylamine | 32.06 | $CH_2$(3,4) | *n*-hexane | 70.86 | $CH_2$ | 18-crown-6 | 133.88 | CH (4) | benzaldehyde |
| 14.23 | $CH_3$ | ethyl acetate | 34.39 | $(CH_3)_3C$ | BHT | 70.92 | $CH_2$ | diglyme | 135.17 | CH(4) | pyridine |
| 14.27 | $CH_3$ | *n*-pentane | 34.54 | $CH_2$(3) | *n*-pentane | 72.25 | $CH_2$ | 1,2-dimethoxyethane | 135.57 | CH(2) | imidazole |
| 14.34 | $CH_3$ | *n*-hexane | 34.58 | $NCH_3$ | dimethylacetamide | 72.39 | $CH_2$ | diglyme | 136.12 | C(2,6) | BHT |
| 15.47 | $CH_3$ | diethyl ether | 34.69 | $(CH_3)_3C$ | BHA | 77.89 | CH | chloroform | 137.12 | C(1) | benzaldehyde |
| 16.63 | $CH_2$ | propane | 35.22 | $CH_3$ | dimethylformamide | 96.57 | $CCl_4$ | carbon tetrachloride | 137.34 | C(4) | BHA |
| 16.65 | $CH_3$ | propane | 36.32 | $C$H$_2$CH$_3$ | ethyl methyl ketone | 108.15 | CH(3,4) | pyrrole | 137.48 | C | toluene-$d_8$ signal |
| 16.84 | $CH_3$ | hexamethylbenzene | 36.8 (d) | $CH_3$ | HMPA[11] | 109.63 | CH(3,4) | furan | 137.84 | C(1) | toluene |
| 18.78 | $CH_3$ | ethanol | 36.98 | $NCH_3$ | dimethylacetamide | 110.99 | CH(3,5) | BHA | 142.65 | CH(2,5) | furan |
| 19.32 | $CH_3$ | propylene | 40.41 | $CH_3$ | dimethyl sulfoxide | 115.76 | CN | acetonitrile | 148.06 | C(2,6) | BHA |
| 20.21 | $CH_3$ | allyl acetate | 40.88 | $CH_2$ | dimethyl malonate | 115.89 | $CH_2$ | propylene | 150.25 | CH(2,6) | pyridine |
| 20.27 | $CH_3$ | acetic acid | 41.78 | $CH_2$(2,6) | cyclohexanone | 117.49 | CH$C$H$_2$ | allyl acetate | 152.06 | C(1) | BHT |
| 20.43 (sept) | $CD_3$ | toluene-$d_8$ signal | 43.40 | $CH_2$ | 1,2-dichloroethane | 117.61 | CH(2,5) | pyrrole | 153.50 | C(1) | BHA |
| 20.46 | $C$H$_3$CO | ethyl acetate | 46.82 | $CH_2$ | triethylamine | 118.04 | CH$C$H$_2$ | diallyl carbonate | 155.15 | CO | diallyl carbonate |
| 21.05 | $CH_3$ | dimethylacetamide | 47.12 | $CH_2$(2,5) | pyrrolidine | 122.13 | CH(4,5) | imidazole | 156.61 | CO | dimethyl carbonate |
| 21.37 | $CH_3$ | toluene | 49.90 | $CH_3$ | methanol | 122.92 | $CH_2$ | ethylene | 161.93 | CH | dimethylformamide |
| 21.42 | $CH_3Ar$ | BHT | 51.76 | $CH_3$ | dimethyl malonate | 123.46 | CH(3,5) | pyridine | 166.49 | $CO_2$ | dimethyl malonate |
| 22.79 | $CH_2$(2,4) | *n*-pentane | 53.47 | $CH_2$ | dichloromethane | 124.86 | $CO_2$ | carbon dioxide | 169.44 | CO | allyl acetate |
| 23.12 | $CH_2$(2,5) | *n*-hexane | 54.13 | $CH_3$ | dimethyl carbonate | 125.13 (t) | CD(4) | toluene-$d_8$ signal | 169.65 | CO | dimethylacetamide |
| 25.15 | $CH_2$(4) | cyclohexanone | 55.04 | $CH_3O$ | BHA | 125.66 | CH(4) | toluene | 170.02 | CO | ethyl acetate |
| 25.24 | $CH_3$ | 2-propanol | 57.81 | $CH_2$ | ethanol | 125.79 | CH(3,5) | BHT | 175.30 | CO | acetic acid |
| 25.75 | $CH_2$(3,4) | pyrrolidine | 58.62 | $CH_3$ | diglyme | 127.96 (t) | CD(3,5) | toluene-$d_8$ signal | 191.09 | HCO | benzaldehyde |
| 25.79 | $CH_2$(3,4) | tetrahydrofuran | 58.63 | $CH_3$ | 1,2-dimethoxyethane | 128.44 | C(4) | BHT | 192.71 | $CS_2$ | carbon disulfide |
| 27.05 | $CH_2$(3,5) | cyclohexanone | 60.08 | $CH_2$ | ethyl acetate | 128.51 | CH(3,5) | toluene | 204.00 | CO | acetone |
| 27.31 | $CH_2$ | cyclohexane | 61.14 | $CH_3$ | nitromethane | 128.57 | CH | benzene | 206.31 | CO | ethyl methyl ketone |
| 28.74 | $C$H$_3$CO | ethyl methyl ketone | 64.12 | CH | 2-propanol | 128.68 | CH(3,5) | benzaldehyde | 208.60 | CO | cyclohexanone |
| 30.03 | $CH_3$ | acetone | 64.29 | $CH_2$ | ethylene glycol | 128.87 (t) | CD(2,6) | toluene-$d_8$ signal | | | |

**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 S15.** $(CD_3)_2CO$ ($^1$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 | 2.17 | s | $CH_3$ | hexamethylbenzene | 4.54 | s | $H_2$ | hydrogen |
| 0.13 | s | $CH_3$ | silicone grease | 2.22 | s | $ArCH_3$ | BHT | 4.62 | ddd | $CH_2$ | diallyl carbonate |
| 0.17 | s | $CH_4$ | methane | 2.27 | t | $CH_2$(2,6) | cyclohexanone | 4.90 | dm, 10 | $CH_2$(1) | propylene |
| 0.83 | s | $CH_3$ | ethane | 2.32 | s | $CH_3$ | toluene | 5.00 | dm, 17 | $CH_2$(2) | propylene |
| 0.87 | m | $CH_3$ | pump oil | 2.45 | q, 7 | C$\boldsymbol{H}_2$$CH_3$ | ethyl methyl ketone | 5.18 | ddt | CHC$\boldsymbol{H}_2$(2) | allyl acetate |
| 0.88 | t, 7 | $CH_3$ | *n*-hexane | 2.45 | q, 7 | $CH_2$ | triethylamine | 5.23 | ddt | CHC$\boldsymbol{H}_2$(2) | diallyl carbonate |
| 0.88 | t, 7 | $CH_3$ | *n*-pentane | 2.52 | s | $CH_3$ | dimethyl sulfoxide | 5.29 | ddt | CHC$\boldsymbol{H}_2$(1) | allyl acetate |
| 0.88 | t, 7.3 | $CH_3$ | propane | 2.59 | d, 9.5 | $CH_3$ | HMPA | 5.35 | ddt | CHC$\boldsymbol{H}_2$(1) | diallyl carbonate |
| 0.90 | m | $CH_3$ | H grease[8] | 2.78 | s | $CH_3$ | dimethylformamide | 5.38 | s | $CH_2$ | ethylene |
| 0.96 | t, 7 | $CH_2$C$\boldsymbol{H}_3$ | ethyl methyl ketone | 2.83 | s | $NCH_3$ | dimethylacetamide | 5.63 | s | $CH_2$ | dichloromethane |
| 0.96 | t, 7 | $CH_3$ | triethylamine | 2.84[3] | s | OH | water | 5.65 | s | OH[5] | BHA |
| 1.10 | d, 6 | $CH_3$ | 2-propanol | 2.94 | s | $CH_3$ | dimethylformamide | 5.81 | m | CH | propylene |
| 1.11 | t, 7 | $CH_3$ | diethyl ether | 3.00 | s | $NCH_3$ | dimethylacetamide | 5.92 | ddt | C$\boldsymbol{H}$$CH_2$ | allyl acetate |
| 1.12 | t, 7 | $CH_3$ | ethanol | 3.12 | s[9] | OH | methanol | 5.96 | ddt | C$\boldsymbol{H}$$CH_2$ | diallyl carbonate |
| 1.18 | s | $CH_3$ | *tert*-butyl alcohol | 3.28 | s | $OCH_3$ | diglyme | 6.07 | m | CH(3,4) | pyrrole |
| 1.20 | t, 7 | $CH_2$C$\boldsymbol{H}_3$ | ethyl acetate | 3.28 | s | $CH_3$ | 1,2-dimethoxyethane | 6.43 | dd | CH(3,4) | furan |
| 1.27 | m | $CH_2$ | *n*-pentane | 3.28 | s | $CH_2$ | ethylene glycol | 6.72 | s | ArH | BHA |
| 1.28 | m | $CH_2$ | *n*-hexane | 3.31 | s[9] | $CH_3$ | methanol | 6.77 | m | CH(2,5) | pyrrole |
| 1.29 | br s | $CH_2$ | H grease[8] | 3.39 | s[6] | OH | ethanol | 6.96 | s | ArH | BHT |
| 1.29 | br s | $CH_2$ | pump oil | 3.41 | q, 7 | $CH_2$ | diethyl ether | 7.04 | s | CH(4,5) | imidazole |
| 1.31 | sept, 7.3 | $CH_2$ | propane | 3.42 | s | $CH_2$ | dimethyl malonate | 7.10–7.20 | m | CH(2,4,6) | toluene |
| 1.41 | s | $ArC(CH_3)_3$ | BHA | 3.46 | s | $CH_2$ | 1,2-dimethoxyethane | 7.10–7.20 | m | CH(3,5) | toluene |
| 1.41 | s | $ArC(CH_3)_3$ | BHT | 3.47 | m | $CH_2$ | diglyme | 7.35 | m | CH(3,5) | pyridine |
| 1.43 | s | $CH_2$ | cyclohexane | 3.56 | m | $CH_2$ | diglyme | 7.36 | s | CH | benzene |
| 1.68 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.57 | q, 7[6] | $CH_2$ | ethanol | 7.56 | dd | CH(2,5) | furan |
| 1.70–1.74 | m | $CH_2$(4) | cyclohexanone | 3.59 | s | $CH_2$ | 18-crown-6 | 7.59–7.63 | m | CH(3,5) | benzaldehyde |
| 1.79 | m | $CH_2$(3,4) | tetrahydrofuran | 3.59 | s | $CH_2$ | 1,4-dioxane | 7.62 | s | CH(2) | imidazole |
| 1.79–1.83 | m | $CH_2$(3,5) | cyclohexanone | 3.63 | m | $CH_2$(2,5) | tetrahydrofuran | 7.69–7.73 | m | CH(4) | benzaldehyde |
| 1.96 | s | $CH_3$ | acetic acid | 3.68 | s | $CH_3$ | dimethyl malonate | 7.76 | m | CH(4) | pyridine |
| 1.97 | s | $CH_3CO$ | dimethylacetamide | 3.72 | s | $ArOCH_3$ | BHA | 7.92–7.94 | m | CH(2,6) | benzaldehyde |
| 1.97 | s | $CH_3CO$ | ethyl acetate | 3.72 | s | $CH_3$ | dimethyl carbonate | 7.96 | s | CH | dimethylformamide |
| 2.02 | s | $CH_3$ | allyl acetate | 3.87 | s | $CH_2$ | 1,2-dichloroethane | 8.02 | s | CH | chloroform |
| 2.05 | s | $CH_3$ | acetonitrile | 3.90 | sept, 6 | CH | 2-propanol | 8.58 | m | CH(2,6) | pyridine |
| 2.05 | p | $CHD_2$ | $(CD_3)_2CO$ residual | 4.05 | q, 7 | C$\boldsymbol{H}_2$$CH_3$ | ethyl acetate | 10.02 | br t | NH | pyrrole |
| 2.07 | s | $CH_3CO$ | ethyl methyl ketone | 4.43 | s | $CH_3$ | nitromethane | 10.05 | s | HCO | benzaldehyde |
| 2.09 | s | $CH_3$ | acetone | 4.53 | ddd | $CH_2$ | allyl acetate | | | | |

**Table S16.** $(CD_3)_2CO$ ($^{13}$C{$^1$H} NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –5.33 | $CH_4$ | methane | 30.60 | $CH_3$ | acetone | 65.28 | $CH_2$ | allyl acetate | 130.23 | CH(2,6) | benzaldehyde |
| 1.12 | $CH_3$ | acetonitrile | 30.64 | ($\boldsymbol{C}H_3)_3$C | BHA | 66.12 | $CH_2$ | diethyl ether | 132.22 | C | hexamethylbenzene |
| 1.40 | $CH_3$ | silicone grease | 30.72 | ($\boldsymbol{C}H_3)_3$C | *tert*-butyl alcohol | 67.60 | $CH_2$ | 1,4-dioxane | 133.16 | $\boldsymbol{C}HCH_2$ | diallyl carbonate |
| 2.01 | $CH_3$ | hexamethyldisiloxane | 31.03 | $CH_3$ | dimethylformamide | 68.07 | $CH_2$(2,5) | tetrahydrofuran | 133.76 | $\boldsymbol{C}HCH_2$ | allyl acetate |
| 6.88 | $CH_3$ | ethane | 31.61 | ($\boldsymbol{C}H_3)_3$C | BHT | 68.13 | $(CH_3)_3\boldsymbol{C}$ | *tert*-butyl alcohol | 134.34 | CH | propylene |
| 8.03 | $CH_2\boldsymbol{C}H_3$ | ethyl methyl ketone | 32.30 | $CH_2$(3,4) | *n*-hexane | 68.78 | $CH_2$ | diallyl carbonate | 135.14 | CH (4) | benzaldehyde |
| 12.49 | $CH_3$ | triethylamine | 34.83 | $CH_2$(3) | *n*-pentane | 71.03 | $CH_2$ | diglyme | 135.89 | CH(2) | imidazole |
| 14.29 | $CH_3$ | *n*-pentane | 34.89 | $NCH_3$ | dimethylacetamide | 71.25 | $CH_2$ | 18-crown-6 | 136.56 | CH(4) | pyridine |
| 14.34 | $CH_3$ | *n*-hexane | 35.00 | $(CH_3)_3\boldsymbol{C}$ | BHT | 72.47 | $CH_2$ | 1,2-dimethoxyethane | 137.66 | C(1) | benzaldehyde |
| 14.50 | $CH_3$ | ethyl acetate | 35.45 | $(CH_3)_3\boldsymbol{C}$ | BHA | 72.63 | $CH_2$ | diglyme | 138.19 | C(2,6) | BHT |
| 15.78 | $CH_3$ | diethyl ether | 36.15 | $CH_3$ | dimethylformamide | 79.19 | CH | chloroform | 138.48 | C(1) | toluene |
| 16.68 | $CH_3$ | propane | 36.75 | $\boldsymbol{C}H_2CH_3$ | ethyl methyl ketone | 96.65 | $CCl_4$ | carbon tetrachloride | 140.32 | C(4) | BHA |
| 16.78 | $CH_2$ | propane | 37.04 (d) | $CH_3$ | HMPA[11] | 108.04 | CH(3,4) | pyrrole | 143.49 | CH(2,5) | furan |
| 16.86 | $CH_3$ | hexamethylbenzene | 37.92 | $NCH_3$ | dimethylacetamide | 110.24 | CH(3,4) | furan | 148.48 | C(2,6) | BHA |
| 18.89 | $CH_3$ | ethanol | 41.23 | $CH_3$ | dimethyl sulfoxide | 111.00 | CH(3,5) | BHA | 150.67 | CH(2,6) | pyridine |
| 19.42 | $CH_3$ | propylene | 41.43 | $CH_2$ | dimethyl malonate | 116.03 | $CH_2$ | propylene | 152.51 | C(1) | BHT |
| 20.51 | $CH_3$ | acetic acid | 42.24 | $CH_2$(2,6) | cyclohexanone | 117.60 | CN | acetonitrile | 153.97 | C(1) | BHA |
| 20.68 | $CH_3$ | allyl acetate | 45.25 | $CH_2$ | 1,2-dichloroethane | 117.81 | $CH\boldsymbol{C}H_2$ | allyl acetate | 155.48 | CO | diallyl carbonate |
| 20.83 | $\boldsymbol{C}H_3CO$ | ethyl acetate | 47.07 | $CH_2$ | triethylamine | 117.98 | CH(2,5) | pyrrole | 157.04 | CO | dimethyl carbonate |
| 21.31 | $CH_3Ar$ | BHT | 49.77 | $CH_3$ | methanol | 118.53 | $CH\boldsymbol{C}H_2$ | diallyl carbonate | 162.79 | CH | dimethylformamide |
| 21.46 | $CH_3$ | toluene | 52.47 | $CH_3$ | dimethyl malonate | 122.31 | CH(4,5) | imidazole | 167.58 | $CO_2$ | dimethyl malonate |
| 21.51 | $CH_3$ | dimethylacetamide | 54.95 | $CH_2$ | dichloromethane | 123.47 | $CH_2$ | ethylene | 170.61 | CO | allyl acetate |
| 22.98 | $CH_2$(2,4) | *n*-pentane | 54.95 | $CH_3$ | dimethyl carbonate | 124.57 | CH(3,5) | pyridine | 170.61 | CO | dimethylacetamide |
| 23.28 | $CH_2$(2,5) | *n*-hexane | 55.51 | $CH_3O$ | BHA | 125.81 | $CO_2$ | carbon dioxide | 170.96 | CO | ethyl acetate |
| 25.59 | $CH_2$(4) | cyclohexanone | 57.72 | $CH_2$ | ethanol | 126.03 | CH(3,5) | BHT | 172.31 | CO | acetic acid |
| 25.67 | $CH_3$ | 2-propanol | 58.45 | $CH_3$ | 1,2-dimethoxyethane | 126.12 | CH(4) | toluene | 192.95 | HCO | benzaldehyde |
| 26.15 | $CH_2$(3,4) | tetrahydrofuran | 58.77 | $CH_3$ | diglyme | 129.03 | CH(3,5) | toluene | 193.58 | $CS_2$ | carbon disulfide |
| 27.51 | $CH_2$ | cyclohexane | 60.56 | $CH_2$ | ethyl acetate | 129.05 | C(4) | BHT | 205.87 | CO | acetone |
| 27.68 | $CH_2$(3,5) | cyclohexanone | 63.21 | $CH_3$ | nitromethane | 129.15 | CH | benzene | 206.26 | CO | $(CD_3)_2CO$ signal |
| 29.30 | $\boldsymbol{C}H_3CO$ | ethyl methyl ketone | 63.85 | CH | 2-propanol | 129.76 | CH(2,6) | toluene | 208.30 | CO | ethyl methyl ketone |
| 29.84 (sept) | $CD_3$ | $(CD_3)_2CO$ signal | 64.26 | $CH_2$ | ethylene glycol | 129.90 | CH(3,5) | benzaldehyde | 210.36 | CO | cyclohexanone |
| 30.36 | $CH_2$ | pump oil | | | | | | | | | |

**Table S17.** $(CD_3)_2SO$ ($^1$H 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[6] | OH | ethanol |
| 0.74 | m | $CH_3$ | pump oil | 2.43 | q, 7 | $CH_2CH_3$ | ethyl methyl ketone | 4.94 | dm, 10 | $CH_2(1)$ | propylene |
| 0.82 | s | $CH_3$ | ethane | 2.43 | q, 7 | $CH_2$ | triethylamine | 5.03 | dm, 17 | $CH_2(2)$ | propylene |
| 0.82–0.88 | m | $CH_3$ | H grease[8] | 2.50 | p | $CHD_2$ | $(CD_3)_2SO$ residual | 5.20 | ddt | $CHCH_2(2)$ | allyl acetate |
| 0.86 | t, 7 | $CH_3$ | *n*-hexane | 2.53 | d, 9.5 | $CH_3$ | HMPA | 5.25 | ddt | $CHCH_2(2)$ | diallyl carbonate |
| 0.86 | t, 7 | $CH_3$ | *n*-pentane | 2.54 | s | $CH_3$ | dimethyl sulfoxide | 5.29 | ddt | $CHCH_2(1)$ | allyl acetate |
| 0.87 | t, 7.3 | $CH_3$ | propane | 2.67 | m | $CH_2(2,5)$ | pyrrolidine | 5.33 | ddt | $CHCH_2(1)$ | diallyl carbonate |
| 0.91 | t, 7 | $CH_2CH_3$ | ethyl methyl ketone | 2.73 | s | $CH_3$ | dimethylformamide | 5.41 | s | $CH_2$ | ethylene |
| 0.93 | t, 7 | $CH_3$ | triethylamine | 2.78 | s | $NCH_3$ | dimethylacetamide | 5.76 | s | $CH_2$ | dichloromethane |
| 1.04 | d, 6 | $CH_3$ | 2-propanol | 2.89 | s | $CH_3$ | dimethylformamide | 5.80 | m | CH | propylene |
| 1.06 | t, 7 | $CH_3$ | ethanol | 2.94 | s | $NCH_3$ | dimethylacetamide | 5.91 | ddt | $CHCH_2$ | allyl acetate |
| 1.09 | t, 7 | $CH_3$ | diethyl ether | 3.16 | s[9] | $CH_3$ | methanol | 5.93 | ddt | $CHCH_2$ | diallyl carbonate |
| 1.11 | s | $CH_3$ | *tert*-butyl alcohol | 3.24 | s | $OCH_3$ | diglyme | 6.01 | m | CH(3,4) | pyrrole |
| 1.15 | br s | $CH_2$ | pump oil | 3.24 | s | $CH_3$ | 1,2-dimethoxyethane | 6.47 | dd | CH(3,4) | furan |
| 1.17 | t, 7 | $CH_2CH_3$ | ethyl acetate | 3.33[3] | s | OH | water | 6.52 | s | OH[5] | BHA |
| 1.24 | br s | $CH_2$ | H grease[8] | 3.34 | s | $CH_2$ | ethylene glycol | 6.62 | s | ArH | BHA |
| 1.25 | m | $CH_2$ | *n*-hexane | 3.38 | q, 7 | $CH_2$ | diethyl ether | 6.65 | s | OH[5] | BHT |
| 1.27 | m | $CH_2$ | *n*-pentane | 3.38 | m | $CH_2$ | diglyme | 6.73 | m | CH(2,5) | pyrrole |
| 1.29 | sept, 7.3 | $CH_2$ | propane | 3.43 | s | $CH_2$ | 1,2-dimethoxyethane | 6.87 | s | ArH | BHT |
| 1.36 | s | $ArC(CH_3)_3$ | BHA | 3.44 | q, 7[6] | $CH_2$ | ethanol | 7.01 | s | CH(4,5) | imidazole |
| 1.36 | s | $ArC(CH_3)_3$ | BHT | 3.51 | s | $CH_2$ | 18-crown-6 | 7.18 | m | CH(2,4,6) | toluene |
| 1.40 | s | $CH_2$ | cyclohexane | 3.51 | m | $CH_2$ | diglyme | 7.25 | m | CH(3,5) | toluene |
| 1.55 | m | $CH_2(3,4)$ | pyrrolidine | 3.53 | s | $CH_2$ | dimethyl malonate | 7.37 | s | CH | benzene |
| 1.64–1.66 | m | $CH_2(4)$ | cyclohexanone | 3.57 | s | $CH_2$ | 1,4-dioxane | 7.39 | m | CH(3,5) | pyridine |
| 1.68 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.60 | m | $CH_2(2,5)$ | tetrahydrofuran | 7.61–7.67 | m | CH(3,5) | benzaldehyde |
| 1.74–1.78 | m | $CH_2(3,5)$ | cyclohexanone | 3.65 | s | $CH_3$ | dimethyl malonate | 7.63 | s | CH(2) | imidazole |
| 1.76 | m | $CH_2(3,4)$ | tetrahydrofuran | 3.66 | s | $ArOCH_3$ | BHA | 7.67 | dd | CH(2,5) | furan |
| 1.91 | s | $CH_3$ | acetic acid | 3.69 | s | $CH_3$ | dimethyl carbonate | 7.69–7.75 | m | CH(4) | benzaldehyde |
| 1.96 | s | $CH_3CO$ | dimethylacetamide | 3.78 | sept, 6 | CH | 2-propanol | 7.79 | m | CH(4) | pyridine |
| 1.99 | s | $CH_3CO$ | ethyl acetate | 3.90 | s | $CH_2$ | 1,2-dichloroethane | 7.91–7.93 | m | CH(2,6) | benzaldehyde |
| 2.03 | s | $CH_3$ | allyl acetate | 4.01 | s[9] | OH | methanol | 8.32 | s | CH | chloroform |
| 2.07 | s | $CH_3$ | acetonitrile | 4.03 | q, 7 | $CH_2CH_3$ | ethyl acetate | 8.58 | m | CH(2,6) | pyridine |
| 2.07 | s | $CH_3CO$ | ethyl methyl ketone | 4.19 | s | OH | *tert*-butyl alcohol | 10.02 | s | HCO | benzaldehyde |
| 2.09 | s | $CH_3$ | acetone | 4.42 | s | $CH_3$ | nitromethane | 10.75 | br t | NH | pyrrole |
| 2.14 | s | $CH_3$ | hexamethylbenzene | 4.52 | ddd | $CH_2$ | allyl acetate | | | | |

**Table S18.** $(CD_3)_2SO$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.01 | $CH_4$ | methane | 30.38 | $(\mathbf{C}H_3)_3C$ | *tert*-butyl alcohol | 63.28 | $CH_3$ | nitromethane | 129.10 | CH(3,5) | benzaldehyde |
| 1.03 | $CH_3$ | acetonitrile | 30.56 | $CH_3$ | acetone | 64.32 | $CH_2$ | allyl acetate | 129.45 | CH(2,6) | benzaldehyde |
| 1.96 | $CH_3$ | hexamethyldisiloxane | 30.73 | $CH_3$ | dimethylformamide | 64.92 | CH | 2-propanol | 131.10 | C | hexamethylbenzene |
| 6.61 | $CH_3$ | ethane | 30.95 | $CH_2(3,4)$ | *n*-hexane | 66.36 | $CH_2$ | 1,4-dioxane | 132.18 | $\mathbf{C}HCH_2$ | diallyl carbonate |
| 7.61 | $CH_2\mathbf{C}H_3$ | ethyl methyl ketone | 31.25 | $(\mathbf{C}H_3)_3C$ | BHT | 66.88 | $(CH_3)_3\mathbf{C}$ | *tert*-butyl alcohol | 132.71 | $\mathbf{C}HCH_2$ | allyl acetate |
| 11.74 | $CH_3$ | triethylamine | 33.48 | $CH_2(3)$ | *n*-pentane | 67.03 | $CH_2(2,5)$ | tetrahydrofuran | 133.55 | CH | propylene |
| 13.28 | $CH_3$ | *n*-pentane | 34.33 | $(CH_3)_3\mathbf{C}$ | BHT | 67.86 | $CH_2$ | diallyl carbonate | 134.52 | CH (4) | benzaldehyde |
| 13.88 | $CH_3$ | *n*-hexane | 34.42 | $NCH_3$ | dimethylacetamide | 69.54 | $CH_2$ | diglyme | 135.15 | CH(2) | imidazole |
| 14.40 | $CH_3$ | ethyl acetate | 34.76 | $(CH_3)_3\mathbf{C}$ | BHA | 69.85 | $CH_2$ | 18-crown-6 | 136.05 | CH(4) | pyridine |
| 15.12 | $CH_3$ | diethyl ether | 35.73 | $CH_3$ | dimethylformamide | 71.17 | $CH_2$ | 1,2-dimethoxyethane | 136.20 | C(1) | benzaldehyde |
| 15.67 | $CH_2$ | propane | 35.83 | $\mathbf{C}H_2CH_3$ | ethyl methyl ketone | 71.25 | $CH_2$ | diglyme | 137.35 | C(1) | toluene |
| 16.34 | $CH_3$ | propane | 36.42 (d) | $CH_3$ | HMPA[11] | 79.16 | CH | chloroform | 139.12 | C(2,6) | BHT |
| 16.60 | $CH_3$ | hexamethylbenzene | 37.38 | $NCH_3$ | dimethylacetamide | 95.44 | $CCl_4$ | carbon tetrachloride | 141.16 | C(4) | BHA |
| 18.51 | $CH_3$ | ethanol | 39.52 (sept) | $CD_3$ | $(CD_3)_2SO$ signal | 107.07 | CH(3,4) | pyrrole | 142.82 | CH(2,5) | furan |
| 19.20 | $CH_3$ | propylene | 40.45 | $CH_3$ | dimethyl sulfoxide | 109.62 | CH(3,4) | furan | 147.44 | C(2,6) | BHA |
| 20.54 | $CH_3$ | allyl acetate | 40.72 | $CH_2$ | dimethyl malonate | 109.80 | CH(3,5) | BHA | 149.58 | CH(2,6) | pyridine |
| 20.68 | $\mathbf{C}H_3CO$ | ethyl acetate | 41.32 | $CH_2(2,6)$ | cyclohexanone | 116.07 | $CH_2$ | propylene | 151.47 | C(1) | BHT |
| 20.95 | $CH_3$ | acetic acid | 45.02 | $CH_2$ | 1,2-dichloroethane | 117.32 | CH(2,5) | pyrrole | 152.53 | C(1) | BHA |
| 20.97 | $CH_3Ar$ | BHT | 45.74 | $CH_2$ | triethylamine | 117.64 | $CH\mathbf{C}H_2$ | allyl acetate | 154.16 | CO | diallyl carbonate |
| 20.99 | $CH_3$ | toluene | 46.51 | $CH_2(2,5)$ | pyrrolidine | 117.91 | CN | acetonitrile | 155.76 | CO | dimethyl carbonate |
| 21.29 | $CH_3$ | dimethylacetamide | 48.59 | $CH_3$ | methanol | 118.32 | $CH\mathbf{C}H_2$ | diallyl carbonate | 162.29 | CH | dimethylformamide |
| 21.70 | $CH_2(2,4)$ | *n*-pentane | 52.08 | $CH_3$ | dimethyl malonate | 121.55 | CH(4,5) | imidazole | 166.91 | $CO_2$ | dimethyl malonate |
| 22.05 | $CH_2(2,5)$ | *n*-hexane | 54.63 | $CH_3$ | dimethyl carbonate | 123.52 | $CH_2$ | ethylene | 169.54 | CO | dimethylacetamide |
| 24.32 | $CH_2(4)$ | cyclohexanone | 54.84 | $CH_2$ | dichloromethane | 123.84 | CH(3,5) | pyridine | 169.97 | CO | allyl acetate |
| 25.14 | $CH_2(3,4)$ | tetrahydrofuran | 54.89 | $CH_3O$ | BHA | 124.21 | $CO_2$ | carbon dioxide | 170.31 | CO | ethyl acetate |
| 25.26 | $CH_2(3,4)$ | pyrrolidine | 56.07 | $CH_2$ | ethanol | 124.85 | CH(3,5) | BHT | 171.93 | CO | acetic acid |
| 25.43 | $CH_3$ | 2-propanol | 57.98 | $CH_3$ | diglyme | 125.29 | CH(4) | toluene | 192.63 | $CS_2$ | carbon disulfide |
| 26.33 | $CH_2$ | cyclohexane | 58.03 | $CH_3$ | 1,2-dimethoxyethane | 127.97 | C(4) | BHT | 193.08 | HCO | benzaldehyde |
| 26.46 | $CH_2(3,5)$ | cyclohexanone | 59.74 | $CH_2$ | ethyl acetate | 128.18 | CH(3,5) | toluene | 206.31 | CO | acetone |
| 29.26 | $\mathbf{C}H_3CO$ | ethyl methyl ketone | 62.05 | $CH_2$ | diethyl ether | 128.30 | CH | benzene | 208.72 | CO | ethyl methyl ketone |
| 29.33 | $CH_2$ | pump oil | 62.76 | $CH_2$ | ethylene glycol | 128.88 | CH(2,6) | toluene | 210.63 | CO | cyclohexanone |
| 30.30 | $(\mathbf{C}H_3)_3C$ | BHA | | | | | | | | | |

**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* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.07 | s | $CH_3$ | hexamethyldisiloxane | 2.19 | s | $CH_3$ | hexamethylbenzene | 4.56 | s | $H_2$ | hydrogen |
| 0.10 | s | $CH_3$ | silicone grease | 2.21 | s | $ArCH_3$ | BHT | 4.61 | ddd | $CH_2$ | diallyl carbonate |
| 0.20 | s | $CH_4$ | methane | 2.32 | s | $CH_3$ | toluene | 4.85 | s | OH[d] | BHA |
| 0.85 | s | $CH_3$ | ethane | 2.34 | t | $CH_2$(2,6) | cyclohexanone | 4.87 | s | OH | water |
| 0.86–0.91 | m | $CH_3$ | pump oil | 2.50 | q, 7 | C***H***$_2$CH$_3$ | ethyl methyl ketone | 4.91 | dm, 10 | $CH_2$(1) | propylene |
| 0.86–0.93 | m | $CH_3$ | H grease[g] | 2.58 | q, 7 | $CH_2$ | triethylamine | 5.01 | dm, 17 | $CH_2$(2) | propylene |
| 0.90 | t, 7 | $CH_3$ | *n*-hexane | 2.64 | d, 9.5 | $CH_3$ | HMPA | 5.21 | ddt | CHC***H***$_2$(2) | allyl acetate |
| 0.90 | t, 7 | $CH_3$ | *n*-pentane | 2.65 | s | $CH_3$ | dimethyl sulfoxide | 5.25 | ddt | CHC***H***$_2$(2) | diallyl carbonate |
| 0.91 | t, 7.3 | $CH_3$ | propane | 2.80 | m | $CH_2$(2,5) | pyrrolidine | 5.30 | ddt | CHC***H***$_2$(1) | allyl acetate |
| 1.01 | t, 7 | $CH_2CH_3$ | ethyl methyl ketone | 2.86 | s | $CH_3$ | dimethylformamide | 5.34 | ddt | CHC***H***$_2$(1) | diallyl carbonate |
| 1.05 | t, 7 | $CH_3$ | triethylamine | 2.92 | s | $NCH_3$ | dimethylacetamide | 5.39 | s | $CH_2$ | ethylene |
| 1.15 | d, 6 | $CH_3$ | 2-propanol | 2.99 | s | $CH_3$ | dimethylformamide | 5.49 | s | $CH_2$ | dichloromethane |
| 1.18 | t, 7 | $CH_3$ | diethyl ether | 3.31 | p | $CD_2H$ | $CD_3OD$ residual | 5.82 | m | CH | propylene |
| 1.19 | t, 7 | $CH_3$ | ethanol | 3.31 | s | $NCH_3$ | dimethylacetamide | 5.94 | ddt | C***H***$CH_2$ | allyl acetate |
| 1.24 | t, 7 | $CH_2CH_3$ | ethyl acetate | 3.34 | s | $CH_3$ | methanol | 5.94 | ddt | C***H***$CH_2$ | diallyl carbonate |
| 1.29 | br s | $CH_2$ | H grease[g] | 3.35 | s | $OCH_3$ | diglyme | 6.08 | m | CH(3,4) | pyrrole |
| 1.29 | m | $CH_2$ | *n*-hexane | 3.35 | s | $CH_3$ | 1,2-dimethoxyethane | 6.40 | dd | CH(3,4) | furan |
| 1.29 | m | $CH_2$ | *n*-pentane | 3.44 | s | $CH_2$ | dimethyl malonate | 6.71 | s | ArH | BHA |
| 1.29 | br s | $CH_2$ | pump oil | 3.49 | q, 7 | $CH_2$ | diethyl ether | 6.72 | m | CH(2,5) | pyrrole |
| 1.34 | sept, 7.3 | $CH_2$ | propane | 3.52 | s | $CH_2$ | 1,2-dimethoxyethane | 6.92 | s | ArH | BHT |
| 1.40 | s | $CH_3$ | *tert*-butyl alcohol | 3.58 | m | $CH_2$ | diglyme | 7.05 | s | CH(4,5) | imidazole |
| 1.40 | s | $ArC(CH_3)_3$ | BHT | 3.59 | s | $CH_2$ | ethylene glycol | 7.16 | m | CH(2,4,6) | toluene |
| 1.41 | s | $ArC(CH_3)_3$ | BHA | 3.60 | q, 7 | $CH_2$ | ethanol | 7.16 | m | CH(3,5) | toluene |
| 1.45 | s | $CH_2$ | cyclohexane | 3.61 | m | $CH_2$ | diglyme | 7.33 | s | CH | benzene |
| 1.70 | dt, 6.4, 1.5 | $CH_3$ | propylene | 3.64 | s | $CH_2$ | 18-crown-6 | 7.44 | m | CH(3,5) | pyridine |
| 1.72 | m | $CH_2$(3,4) | pyrrolidine | 3.66 | s | $CH_2$ | 1,4-dioxane | 7.49 | dd | CH(2,5) | furan |
| 1.74–1.76 | m | $CH_2$(4) | cyclohexanone | 3.71 | m | $CH_2$(2,5) | tetrahydrofuran | 7.56–7.60 | m | CH(3,5) | benzaldehyde |
| 1.85–1.87 | m | $CH_2$(3,5) | cyclohexanone | 3.72 | s | $ArOCH_3$ | BHA | 7.66–7.70 | m | CH(4) | benzaldehyde |
| 1.87 | m | $CH_2$(3,4) | tetrahydrofuran | 3.72 | s | $CH_3$ | dimethyl malonate | 7.67 | s | CH(2) | imidazole |
| 1.99 | s | $CH_3$ | acetic acid | 3.74 | s | $CH_3$ | dimethyl carbonate | 7.85 | m | CH(4) | pyridine |
| 2.01 | s | $CH_3CO$ | ethyl acetate | 3.78 | s | $CH_2$ | 1,2-dichloroethane | 7.90 | s | CH | chloroform |
| 2.03 | s | $CH_3$ | acetonitrile | 3.92 | sept, 6 | CH | 2-propanol | 7.90–7.93 | m | CH(2,6) | benzaldehyde |
| 2.05 | s | $CH_3$ | allyl acetate | 4.09 | q, 7 | C***H***$_2$CH$_3$ | ethyl acetate | 7.97 | s | CH | dimethylformamide |
| 2.07 | s | $CH_3CO$ | dimethylacetamide | 4.34 | s | $CH_3$ | nitromethane | 8.53 | m | CH(2,6) | pyridine |
| 2.12 | s | $CH_3CO$ | ethyl methyl ketone | 4.56 | ddd | $CH_2$ | allyl acetate | 10.00 | s | HCO | benzaldehyde |
| 2.15 | s | $CH_3$ | acetone | | | | | | | | |

## Table S24. $CD_3OD$ ($^{13}C\{^1H\}$ NMR data by chemical shift in ppm)

| *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* | *shift* | *carbon* | *impurity* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| –4.90 | $CH_4$ | methane | 30.82 | ($\mathbf{C}H_3$)$_3$C | BHA | 64.71 | CH | 2-propanol | 130.12 | CH(3,5) | benzaldehyde |
| 0.85 | $CH_3$ | acetonitrile | 30.91 | ($\mathbf{C}H_3$)$_3$C | *tert*-butyl alcohol | 66.14 | $CH_2$ | allyl acetate | 130.64 | CH(2,6) | benzaldehyde |
| 1.99 | $CH_3$ | hexamethyldisiloxane | 31.15 | ($\mathbf{C}H_3$)$_3$C | BHT | 66.88 | $CH_2$ | diethyl ether | 132.53 | C | hexamethylbenzene |
| 2.10 | $CH_3$ | silicone grease | 31.35 | $CH_2$ | pump oil | 68.11 | $CH_2$ | 1,4-dioxane | 133.25 | ***C***$HCH_2$ | diallyl carbonate |
| 6.98 | $CH_3$ | ethane | 31.61 | $CH_3$ | dimethylformamide | 68.83 | $CH_2$(2,5) | tetrahydrofuran | 133.71 | ***C***$HCH_2$ | allyl acetate |
| 8.09 | $CH_2\mathbf{C}H_3$ | ethyl methyl ketone | 32.73 | $CH_2$(3,4) | *n*-hexane | 69.35 | $CH_2$ | diallyl carbonate | 134.61 | CH | propylene |
| 11.09 | $CH_3$ | triethylamine | 35.30 | $CH_2$(3) | *n*-pentane | 69.40 | ($CH_3$)$_3$***C*** | *tert*-butyl alcohol | 135.60 | CH (4) | benzaldehyde |
| 14.39 | $CH_3$ | *n*-pentane | 35.36 | ($CH_3$)$_3$***C*** | BHT | 71.33 | $CH_2$ | diglyme | 136.31 | CH(2) | imidazole |
| 14.45 | $CH_3$ | *n*-hexane | 35.50 | $NCH_3$ | dimethylacetamide | 71.47 | $CH_2$ | 18-crown-6 | 137.96 | C(1) | benzaldehyde |
| 14.49 | $CH_3$ | ethyl acetate | 35.83 | ($CH_3$)$_3$***C*** | BHA | 72.72 | $CH_2$ | 1,2-dimethoxyethane | 138.35 | CH(4) | pyridine |
| 15.46 | $CH_3$ | diethyl ether | 36.89 | $CH_3$ | dimethylformamide | 72.92 | $CH_2$ | diglyme | 138.85 | C(1) | toluene |
| 16.80 | $CH_3$ | propane | 37.00 (d) | $CH_3$ | HMPA[i] | 79.44 | CH | chloroform | 139.09 | C(2,6) | BHT |
| 16.90 | $CH_3$ | hexamethylbenzene | 37.34 | ***C***$H_2CH_3$ | ethyl methyl ketone | 97.21 | $CCl_4$ | carbon tetrachloride | 141.36 | C(4) | BHA |
| 17.19 | $CH_2$ | propane | 38.43 | $NCH_3$ | dimethylacetamide | 108.11 | CH(3,4) | pyrrole | 143.68 | CH(2,5) | furan |
| 18.40 | $CH_3$ | ethanol | 40.45 | $CH_3$ | dimethyl sulfoxide | 110.33 | CH(3,4) | furan | 149.04 | C(2,6) | BHA |
| 19.50 | $CH_3$ | propylene | 41.60 | $CH_2$ | dimethyl malonate | 111.30 | CH(3,5) | BHA | 150.07 | CH(2,6) | pyridine |
| 20.56 | $CH_3$ | acetic acid | 42.61 | $CH_2$(2,6) | cyclohexanone | 116.04 | $CH_2$ | propylene | 152.85 | C(1) | BHT |
| 20.71 | $CH_3$ | allyl acetate | 45.11 | $CH_2$ | 1,2-dichloroethane | 118.06 | CN | acetonitrile | 154.34 | C(1) | BHA |
| 20.88 | ***C***$H_3CO$ | ethyl acetate | 46.96 | $CH_2$ | triethylamine | 118.22 | CH***C***$H_2$ | allyl acetate | 156.28 | CO | diallyl carbonate |
| 21.32 | $CH_3$ | dimethylacetamide | 47.23 | $CH_2$(2,5) | pyrrolidine | 118.28 | CH(2,5) | pyrrole | 157.91 | CO | dimethyl carbonate |
| 21.38 | $CH_3Ar$ | BHT | 49.00 (sept) | $CD_3$ | $CD_3OD$ signal | 118.74 | CH***C***$H_2$ | diallyl carbonate | 164.73 | CH | dimethylformamide |
| 21.50 | $CH_3$ | toluene | 49.86 | $CH_3$ | methanol | 122.60 | CH(4,5) | imidazole | 168.70 | $CO_2$ | dimethyl malonate |
| 23.38 | $CH_2$(2,4) | *n*-pentane | 52.83 | $CH_3$ | dimethyl malonate | 123.46 | $CH_2$ | ethylene | 172.41 | CO | allyl acetate |
| 23.68 | $CH_2$(2,5) | *n*-hexane | 54.78 | $CH_2$ | dichloromethane | 125.53 | CH(3,5) | pyridine | 172.89 | CO | ethyl acetate |
| 25.27 | $CH_3$ | 2-propanol | 55.25 | $CH_3$ | dimethyl carbonate | 126.11 | CH(3,5) | BHT | 173.32 | CO | dimethylacetamide |
| 25.86 | $CH_2$(4) | cyclohexanone | 55.96 | $CH_3O$ | BHA | 126.29 | CH(4) | toluene | 175.11 | CO | acetic acid |
| 26.29 | $CH_2$(3,4) | pyrrolidine | 58.26 | $CH_2$ | ethanol | 126.31 | $CO_2$ | carbon dioxide | 193.82 | $CS_2$ | carbon disulfide |
| 26.48 | $CH_2$(3,4) | tetrahydrofuran | 59.06 | $CH_3$ | diglyme | 129.20 | CH(3,5) | toluene | 194.11 | HCO | benzaldehyde |
| 27.96 | $CH_2$ | cyclohexane | 59.06 | $CH_3$ | 1,2-dimethoxyethane | 129.34 | CH | benzene | 209.67 | CO | acetone |
| 28.16 | $CH_2$(3,5) | cyclohexanone | 61.50 | $CH_2$ | ethyl acetate | 129.49 | C(4) | BHT | 212.16 | CO | ethyl methyl ketone |
| 29.39 | ***C***$H_3CO$ | ethyl methyl ketone | 63.08 | $CH_3$ | nitromethane | 129.91 | CH(2,6) | toluene | 214.69 | CO | cyclohexanone |
| 30.67 | $CH_3$ | acetone | 64.30 | $CH_2$ | ethylene glycol | | | | | | |

**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.