

A Specialist Periodical Report

Spectroscopic Properties of Inorganic and Organometallic Compounds

Volume 17

A Review of the Recent Literature Published
up to Late 1983

Senior Reporters

G. Davidson,

E. A. V. Ebsworth,

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Foreword

This volume follows the form of its recent predecessors; there are no new sections, and the general coverage remains the same. Despite the steady increase in the amount of published work, the reviewers have done wonders in controlling the size of their contributions, and as ever I am extremely grateful to them for their prompt and careful work. The next volume will be produced from camera-ready copy; I hope that this will lead to a reduced price and to quicker publication.

November 1984

E. A. V. EBSWORTH

Conversion Factors

1 kJ mol⁻¹

$2.3901 \times 10^{-1} \text{ kcal mol}^{-1}$
 $1.0364 \times 10^{-2} \text{ eV atom}^{-1}$
 $8.3593 \times 10 \text{ cm}^{-1}$
 $2.5061 \times 10^6 \text{ MHz}$

1 kcal mol⁻¹

$4.1840 \text{ kJ mol}^{-1}$
 $4.3364 \times 10^{-2} \text{ eV atom}^{-1}$
 $3.4976 \times 10^2 \text{ cm}^{-1}$
 $1.0486 \times 10^7 \text{ MHz}$

1 cm⁻¹

$1.1963 \times 10^{-2} \text{ kJ mol}^{-1}$
 $2.8592 \times 10^{-3} \text{ kcal mol}^{-1}$
 $1.2399 \times 10^{-4} \text{ eV atom}^{-1}$
 $2.9979 \times 10^4 \text{ MHz}$

1 MHz

$3.9903 \times 10^{-7} \text{ kJ mol}^{-1}$
 $9.5370 \times 10^{-8} \text{ kcal mol}^{-1}$
 $4.1357 \times 10^{-9} \text{ eV atom}^{-1}$
 $3.3356 \times 10^{-5} \text{ cm}^{-1}$

1 eV atom⁻¹

$9.6485 \times 10 \text{ kJ mol}^{-1}$
 $2.3060 \times 10 \text{ kcal mol}^{-1}$
 $8.0655 \times 10^3 \text{ cm}^{-1}$
 $2.4180 \times 10^8 \text{ MHz}$

Mössbauer Spectra: $E_\nu(^{57}\text{Fe}) = 14.413 \text{ keV}$

1 mm s⁻¹

$4.639 \times 10^{-6} \text{ kJ mol}^{-1}$
 $1.109 \times 10^{-6} \text{ kcal mol}^{-1}$
 $4.808 \times 10^{-8} \text{ eV atom}^{-1}$
 $3.878 \times 10^{-4} \text{ cm}^{-1}$
 $1.162 \times 10 \text{ MHz}$

For other Mössbauer nuclides, multiply the above conversion factors by $E_\nu(\text{keV})/14.413$

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Nuclear Magnetic Resonance Spectroscopy

BY B. E. MANN

1 Introduction

Following the criteria established in earlier volumes, only books and reviews directly relevant to this chapter are included, and the reader who requires a complete list is referred to the Specialist Periodical Reports 'Nuclear Magnetic Resonance',¹ where a complete list of books and reviews is given. Reviews which are of direct relevance to a section of this Report are included in the beginning of that section rather than here. Papers where only ^1H n.m.r. spectroscopy is used are only included when the ^1H n.m.r. spectra make a non-routine contribution, but complete coverage of relevant papers is still attempted where nuclei other than the proton are involved.

Several reviews have appeared, including 'Physical methods and techniques. Part (ii). N.m.r. spectroscopy',² 'High resolution multinuclear magnetic resonance: instrumentation requirements and detection procedures',³ 'Transition metal n.m.r. spectroscopy',⁴ 'Nuclear magnetic resonance studies in cluster chemistry',⁵ 'Magnetic resonance of oxidised metalloporphyrins',⁶ 'Methods to analyze metal-protein binding. Multinuclear n.m.r. studies of metalloproteins',⁷ 'Nuclear magnetic resonance of calcium-binding proteins',⁸ 'N.m.r. and e.p.r. investigations of bimetalloenzymes',⁹ and 'N.m.r. and e.p.r. studies of chromium and cobalt nucleotides and their interactions with enzymes'.¹⁰

A number of papers have been published which are too broadly based to fit into a later section and are included here. The nuclear magnetic shielding function for H_2 has been extracted from spin-rotation and isotope-shift data.¹¹ $^1J(^{13}\text{C}, ^{13}\text{C})$

¹ 'Nuclear Magnetic Resonance', ed. R. J. Abraham (Specialist Periodical Reports), The Royal Society of Chemistry, London, 1983, Vol. 12; 1984, Vol. 13.

² R. F. M. White, *Annu. Rep. Prog. Chem., Sect. B* 1982, 1981, 78, 15.

³ C. Brevard, *NATO ASI Ser., Ser. C*, 1983, 103, 1 (*Chem. Abstr.*, 1983, 99, 132 383).

⁴ R. G. Kidd, *NATO ASI Ser., Ser. C*, 1983, 103, 445 (*Chem. Abstr.*, 1983, 99, 132 402).

⁵ B. T. Heaton, *Philos. Trans. R. Soc. London, Ser. A*, 1982, 308, 95.

⁶ H. M. Goff, M. A. Phillippi, A. D. Boersma, and A. P. Hansen, *Adv. Chem. Ser.*, 1982, 201, 357 (*Chem. Abstr.*, 1983, 98, 67 206).

⁷ Y. Arata and T. Sawatari, *Tanpakushitsu Kakusan Koso, Bessatsu*, 1983, 74 (*Chem. Abstr.*, 1983, 99, 172 101).

⁸ Y. Shibata and T. Miyazawa, *Tanpakushitsu Kakusan Koso*, 1982, 27, 2226 (*Chem. Abstr.*, 1983, 98, 29 719).

⁹ J. J. Villafranca and F. M. Raushel, *Adv. Inorg. Biochem.*, 1982, 4, 289 (*Chem. Abstr.*, 1983, 98, 175 332).

¹⁰ J. J. Villafranca, *Methods Enzymol.*, 1982, 87, 180 (*Chem. Abstr.*, 1983, 98, 13 374).

¹¹ W. T. Raynes and N. Panteli, *Mol. Phys.*, 1983, 48, 439.

and $^1J(M, ^{13}C)$ have been reported for 27 organometallic derivatives of alkanes, alkenes, benzene, and alkynes ($M = B, Sn, Pb, Hg,$ or Bi).¹² The spin-lattice relaxation times of ^{17}O at 54.25 MHz have been found to range from 8 to 50 ms for several metalloboranes, and a stereochemical dependence has been found.¹³ Spin-lattice relaxation times of ^{35}S , ^{51}V , ^{53}Cr , and ^{55}Mn in tetrahedral oxoanions have been determined in aqueous solution.¹⁴ The temperature dependence of T_1 of $[MO_4]^{n-}$ ($M = Cl, Br, Mn, Cr, V, Mo, Tc, Re,$ or Ru) has been determined, and for $[MnO_4]^-$, $[VO_4]^{3-}$, RuO_4 , and $[TcO_4]^-$ a minimum was found.¹⁵ ^{15}N n.m.r. spectra of transition-metal nitrosyl complexes have been reported, and for MNO angles near 120° the nitrogen atom is strongly deshielded relative to linear MNO complexes.^{16,17} Deshieldings of 350–700 p.p.m. have been observed in the ^{15}N n.m.r. spectra of strongly bent nitrosyl groups.¹⁸ A model of the interaction of metal ions with the phosphate group, based on ^{31}P n.m.r. studies, has been described.¹⁹ The interaction of metals with N,N,N',N' -tetra-butyl-3,6-dioxaoctanedithioamide has been investigated using ^{13}C , ^{113}Cd , ^{195}Pt , and ^{199}Hg n.m.r. spectroscopy.²⁰ A sensitive ^{13}C n.m.r. shift thermometer using Dy^{3+} in acetate buffer has been described.²¹ The effect of cation on $[MF_6]^{2-}$ ($M = Si, Ge,$ or Sn) T_1 has been examined by ^{19}F n.m.r. spectroscopy.²²

2 Stereochemistry

This section is subdivided into ten parts which contain n.m.r. information about Groups IA and IIA and transition-metal complexes presented by Groups according to the Periodic Table. Within each Group, classification is by ligand type.

Complexes of Groups IA and IIA.—Two reviews have appeared: 'N.m.r. of the alkali metals'²³ and 'N.m.r. of the alkaline earth metals'.²⁴

The degree of aggregation in solution of organolithium derivatives has been correlated with the multiplicities of the signals due to $^1J(^{13}C, ^6Li)$.²⁵ The structures of monosilylated pentadienyl-lithium and 1,5-disilylated pentadienyl-lithium,

¹² B. Wrackmeyer, *Spectrosc.: Int. J.*, 1982, 1, 201.

¹³ S. Aime, R. Gobetto, D. Osella, L. Milone, G. E. Hawkes, and E. W. Randall, *J. Chem. Soc., Chem. Commun.*, 1983, 794.

¹⁴ E. Haid, D. Köhnlein, G. Kössler, O. Lutz, and W. Schick, *J. Magn. Reson.*, 1983, 55, 145.

¹⁵ V. P. Tarasov, V. I. Privalov, and Yu. A. Buslaev, *Dokl. Akad. Nauk SSSR*, 1983, 269, 640 (*Chem. Abstr.*, 1983, 98, 226 750).

¹⁶ D. H. Evans, D. M. P. Mingos, J. Mason, and A. Richards, *J. Organomet. Chem.*, 1983, 249, 293.

¹⁷ L. K. Bell, J. Mason, D. M. P. Mingos, and D. G. Tew, *Inorg. Chem.*, 1983, 22, 3497.

¹⁸ L. K. Bell, D. M. P. Mingos, D. G. Tew, L. F. Larkworthy, S. Sandell, D. C. Povey, and J. Mason, *J. Chem. Soc., Chem. Commun.*, 1983, 125.

¹⁹ J. Pokorny, *Biologia (Bratislava)*, 1983, 38, 289 (*Chem. Abstr.*, 1983, 98, 156 744).

²⁰ P. Hofstetter, E. Pretsch, and W. Simon, *Helv. Chim. Acta*, 1983, 66, 2103.

²¹ P. J. Smolenaers, M. T. Kelso, and J. K. Beattie, *J. Magn. Reson.*, 1983, 52, 118.

²² Yu. N. Moskvich, A. M. Polyakov, G. I. Dotsenko, and M. L. Afanas'ev, *Zh. Neorg. Khim.*, 1982, 27, 1972 (*Chem. Abstr.*, 1983, 98, 45 626).

²³ P. Laszlo, *NATO ASI Ser., Ser. C*, 1983, 103, 261 (*Chem. Abstr.*, 1983, 99, 132 394).

²⁴ O. Lutz, *NATO ASI Ser., Ser. C*, 1983, 103, 297 (*Chem. Abstr.*, 1983, 99, 132 395).

²⁵ D. Seebach, R. Haessig, and J. Gabriel, *Helv. Chim. Acta*, 1983, 66, 308.

-potassium, and -caesium are in the W form as confirmed by the ^1H and ^{13}C n.m.r. spectra.²⁶ The effect of HMPA on the ^{13}C chemical shift of the α -carbon atom of benzyl-lithium in THF parallels its effects on the one-electron electrochemical oxidation potential.²⁷ The low-temperature ^{13}C n.m.r. spectrum of 1-Li-3,3-dimethylbut-1-yne in THF has shown a non-fluxional cubic tetramer with $^1J(^{13}\text{C}, ^6\text{Li}) = 6 \text{ Hz}$.²⁸ The ^{13}C , ^{17}O , ^7Li , and ^{23}Na n.m.r. shielding tensors have been computed for M^+-CO ($\text{M} = \text{Li}$ or Na).²⁹ ^7Li n.m.r. measurements, including T_1 and T_2 , have been carried out in halotolerant bacterium B_{a1} .³⁰ Lithioesters obtained from Pr^1_2NLi with cycloalkanecarboxylates at low temperature have been examined by ^{13}C n.m.r. spectroscopy.³¹ ^7Li n.m.r. spectroscopy supports a tight ion pair for $\text{LiBr}(\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMeCH}_2\text{CH}_2\text{NMe})$.³² ^{23}Na n.m.r. signals have been observed from frog skin.³³ The ^{23}Na n.m.r. spectrum of $\text{Na}^+[\text{Ph}_2\text{PCHPh}_2\text{R}]^-$ and the ^{31}P broadening suggest a tight ion pair.³⁴ Cholesteric and nematic lyotropic mesophases from disodium *N*-lauroylaspartate have been investigated using ^2H and ^{23}Na n.m.r. spectroscopy.³⁵ The nuclear g factor of $^{39}\text{K}^+$ and the diamagnetic shielding-constant difference between ^{39}K and $^{39}\text{K}^+$ have been determined.³⁶ Intracellular K^+ concentration has been determined by ^{39}K n.m.r. spectroscopy.³⁷ N.m.r. data have also been reported for $(\text{cyclo-C}_3\text{H}_5\text{Li})_2(\text{LiBr})_2(\text{Et}_2\text{O})_4$ (^{13}C),³⁸ $\text{Li}[\text{C}(\text{SiMe}_2\text{Ph})_3] \cdot \text{THF}$ (^7Li),³⁹ $\text{M}(\text{CH}_2\text{-PPh}_2\text{CHPPh}_2)$, $\text{Ag}(\text{PPh}_2)_2\text{CPh}_2\text{CHPPh}_2$ ($\text{M} = \text{Li}, \text{Na}, \text{or K}$; ^{13}C , ^{23}Na , ^{31}P),⁴⁰ and $\text{Li}_2\text{P}_{16} \cdot 8\text{THF}$ (^{31}P).⁴¹

Di-co-ordination of beryllium in $\text{Be}(\text{NR}_2)_2$ results in a high-frequency shift of the ^9Be n.m.r. signal and a large linewidth relative to tri-co-ordinate beryllium. ^{13}C and ^{14}N n.m.r. spectra were also recorded.⁴² The ring-current model has been applied to determine the geometry of the aggregated species of porphyrins

²⁶ H. Yasuda, T. Nishi, K. Lee, and A. Nakamura, *Organometallics*, 1983, 2, 21.

²⁷ R. Breslow and J. Schwarz, *J. Am. Chem. Soc.*, 1983, 105, 6795.

²⁸ G. Fraenkel and P. Pramanik, *J. Chem. Soc., Chem. Commun.*, 1983, 1527.

²⁹ T. Weller, W. Meiler, H. Pfeifer, H. Lischka, and R. Hoeller, *Chem. Phys. Lett.*, 1983, 95, 599.

³⁰ M. Goldberg, M. Risk, and H. Gilboa, *Biochim. Biophys. Acta*, 1983, 763, 35 (*Chem. Abstr.*, 1983, 99, 119 087).

³¹ L. Gorrichon, P. Maroni, Ch. Zedde, and A. Dobrev, *J. Organomet. Chem.*, 1983, 252, 267.

³² S. R. Hall, C. L. Raston, B. W. Skelton, and A. White, *Inorg. Chem.*, 1983, 22, 4070.

³³ M. M. Civan, H. Degani, Y. Margalit, and M. Shporer, *Am. J. Physiol.*, 1983, 245, C213 (*Chem. Abstr.*, 1983, 99, 190 934).

³⁴ H. Schmidbaur, U. Deschler, and D. Seyferth, *Z. Naturforsch., Teil B*, 1982, 37, 950 (*Chem. Abstr.*, 1983, 98, 143 594).

³⁵ M. R. Alcantara, M. V. Marques, C. De Melo, V. R. Paoli, and J. A. Vanin, *Mol. Cryst. Liq. Cryst.*, 1983, 90, 335 (*Chem. Abstr.*, 1983, 99, 46 364).

³⁶ E. I. Obiajunwa, S. A. Adebisi, E. A. Togun, and A. F. Oluwole, *J. Phys. B*, 1983, 16, 2733 (*Chem. Abstr.*, 1983, 99, 204 909).

³⁷ P. J. Brophy, M. K. Hayer, and F. G. Riddell, *Biochem. J.*, 1983, 210, 961 (*Chem. Abstr.*, 1983, 99, 190 916).

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