

Table 1. Mössbauer Parameters, Zero Field, for Homoleptic Iron(II)–Thiolate Dimers

complex	temp (K)	i.s (mm s ⁻¹)	q.s (mm s ⁻¹) ^b	hwhm ^a	ref
[Fe(SC ₆ H ₂ ^t Bu ₃ -2,4,6) ₂] ₂ (1)	77	0.77(1)	2.47(1)	0.21(1)	this work
	100	0.77	2.49	0.20	5
[Fe(SC ₆ H ₃ Ph ₃ -2,4,6) ₂] ₂ (2)	4.2	0.77	2.28	0.27	5
[Fe(SC ₆ H ₂ Me ₃ -2,4,6) ₂] ₂	77	0.82(1)	2.80(1)	0.20(1)	this work
[Fe(SC ₆ F ₃) ₂] ₂	77	0.80(1)	2.65(1)	0.30(1)	this work

^a hwhm = half-width at half maxima (mm s⁻¹). ^b Value in parentheses is error in last decimal place.

Table 2. Mössbauer Parameters, Zero Field, for 3-Coordinate Iron(II) Complexes

complex	temp (K)	i.s (mm s ⁻¹)	q.s (mm s ⁻¹) ^b	hwhm ^a	ref
[Fe(SC ₆ H ₂ ^t Bu ₃ -2,4,6) ₃] ⁻	100	0.53	0.81	0.21	5
	4.2	0.57	0.81	0.11	5
[Fe(SC ₆ H ₃ (mes) ₂ -2,6){N(SiMe ₃) ₂ }] (5)	4.2	0.66	0.79	0.18	5
[Fe{N(SiMe ₃) ₂ } ₂ (thf)]	77	0.57(1)	1.97(1)	0.19(1)	this work
[Fe{N(SiMe ₃) ₂ } ₂] ₂	77	0.58(1)	1.02(1)	0.22(1)	this work

^a hwhm = half-width at half maxima (mm s⁻¹). ^b Value in parentheses is error in last decimal place.

of 2.37 and 2.42 Å for one of the terminal iron atoms and 2.58 and 2.69 Å for the other. These “long-range” interactions may influence the electric field gradient experienced by the iron atoms, and by Mössbauer criteria the terminal iron atoms can be considered as 5-coordinate. The large line width may arise from the overlap of quadrupole split doublets from each of the terminal iron atoms together with magnetic relaxation effects.

To summarize, consideration of both crystallographic and Mössbauer data has led to the redefinition of the coordination number of iron in several low-coordinate high-spin iron(II) thiolates. The iron atoms of the dimeric homoleptic iron(II) thiolate complexes [Fe₂(SC₆H₂^tBu₃-2,4,6)₄] (1) and [Fe₂(SC₆H₂-Ph₃-2,4,6)₄] (2) are not 3- but 4-coordinate. The complexes [Fe(SC₆H₃(mes)₂-2,6){N(SiMe₃)₂}] (5) and [Fe{SC₆H₃(mes)₂-

2,6}₂] (4), formerly both described as containing 2-coordinate iron, have 3- and 4-coordinate iron, respectively, and the terminal iron atoms of [Fe₃(SC₆H₂^tPr₃-2,4,6)₄{N(SiMe₃)₂}] (3) are, by Mössbauer criteria, 5- and not 3-coordinate. Trigonal planar high-spin iron(II) thiolates have characteristic Mössbauer parameters, i.s. ca. 0.6 mm s⁻¹, and q.s. ca. 0.8 mm s⁻¹ and can, therefore, be readily distinguished from tetrahedral and other geometries about iron. From the discussion above it can also be concluded that there are no unambiguous examples of 2-coordinate iron(II) thiolates.

Acknowledgment. We thank the Biotechnology and Biological Sciences Research Council, United Kingdom, for funding this work.

IC960929D

Additions and Corrections

1996, Volume 35

Ewan J. M. Hamilton, Glenn T. Jordan IV, Edward A. Meyers, and Sheldon G. Shore*: One-Step Preparation of Dimethyl Sulfide Substituted Icosahedral Boranes: The Crystal and Molecular Structures of 1,7-(SMe₂)₂B₁₂H₁₀, 1,12-(SMe₂)₂B₁₂H₁₀, and [SMe₃][B₁₂H₁₁(SMe₂)]·MeCN.

Page 5335. In column 1, the following addendum belongs at the end of the first paragraph:

However, more recently, Jasper, Jones, Mattern, Huffman, and Todd¹³ reported that from the reaction of Me₂SPdCl₂ with Li₂[B₁₂H₁₂] they obtained a mixture of products from which they separated the 1,7-(Me₂S)₂B₁₂H₁₀ and 1,12-(Me₂S)₂B₁₂H₁₀

(13) Jasper, S. A., Jr.; Jones, R. B.; Mattern, J.; Huffman, J. C.; Todd, L. J. *Inorg. Chem.* **1994**, *33*, 5620.

isomers. Identification was based upon comparison of ¹H NMR and IR spectra with those reported by Wright and Kaczmarczyk⁴ for a mixture of isomers.

IC961472A

Zuwei Xie,* Ka-yue Chiu, Bomu Wu, and Thomas C. W. Mak: Autoionization of SmI₃ in Tetrahydrofuran. X-ray Crystal Structure of the Ionic Complex [SmI₂(THF)₅][SmI₄(THF)₂].

Page 5957. In paragraph 1, the word “tetrahydrofuran” was inadvertently omitted from the sentence beginning on line 12. The correct sentence should read as follows: To date, no crystal structure of any LnI₃ tetrahydrofuran complex has, to our knowledge, been reported. In the reference list, refs 19 and 20 should be interchanged.

IC961411C