

Table 3. Comparison of the observed and simulated lattice parameters for the $Ti_{1-x}Zr_xO_2$ system

Values in parentheses are the degree of expansion (%).

	Amount of replacement, x				
	0	0.05	0.0625	0.10	0.125
Observed					
a (Å)	4.594	4.605 (0.24)	–	4.619 (0.53)	–
c (Å)	2.959	2.974 (0.55)	–	2.991 (1.09)	–
Simulated					
a (Å)	4.585	–	4.596 (0.26)	–	4.608 (0.53)
c (Å)	2.964	–	2.982 (0.59)	–	2.999 (1.17)

The authors would like to thank Professor A. Miyamoto of Tohoku University for his kind support during this research. We are also grateful to A. Mitani for his help with the experiment.

References

- ABRAHAMS, S. C. & BERNSTEIN, J. L. (1971). *J. Chem. Phys.* **55**, 3206–3211.
- BROWN, F. H. JR & DUWEZ, P. (1954). *J. Am. Ceram. Soc.* **37**, 129–132.
- EGERTON, L. & THOMSON, J. JR (1971). *Am. Ceram. Soc. Bull.* **50**, 924–928.
- GALASSO, F. S. (1970). *Structure and Properties of Inorganic Solids*, International Series of Monographs in Solid State Physics, Vol. 7. New York: Pergamon Press.
- GILBERT, T. L. (1968). *J. Chem. Phys.* **49**, 2640–2642.
- KAWAMURA, K. (1992). *Molecular Dynamics Simulation*, Springer Series in Solid-State Science, Vol. 103, edited by F. YONEZAWA. Heidelberg: Springer-Verlag.
- MATSUI, M. (1988). *Phys. Chem. Miner.* **16**, 234–238.
- MATSUI, M. (1990). *Tech. Rep. ISEI Ser. C*, (6), 32–36.
- RAO, K. V. K., NAIDU, S. V. N. & IYENGAR, L. (1970). *J. Am. Ceram. Soc.* **53**, 124–126.

Acta Cryst. (1993). **B49**, 783

Refinement of atomic positions in bixbyite oxides using perturbed angular correlation spectroscopy.

Erratum. By A. BARTOS, K. P. LIEB, M. UHRMACHER and D. WIARDA, *II. Physikalisches Institut Universität Göttingen, D-3400 Göttingen, Germany*

(Received 21 May 1993)

Abstract

The name of R. W. G. Wyckoff is incorrectly given as Wyckhoff on pages 165 and 169 of the paper by Bartos,

Lieb, Uhrmacher & Wiarda [*Acta Cryst.* (1993), **B49**, 165–169].

All relevant information is given in the *Abstract*.

FAST COMMUNICATIONS

Contributions intended for this section should be submitted to any of the Co-editors of Acta Crystallographica or Journal of Applied Crystallography. In the letter accompanying the submission authors should state why rapid publication is essential. The paper should not exceed two printed pages (about 2000 words or eight pages of double-spaced typescript including tables and figures) and figures should be clearly lettered. If the paper is available on 5.25" IBM PC compatible or 3.5" Apple/Macintosh diskettes it would be helpful if these could be sent with the manuscript together with details of the word-processing package used. Papers not judged suitable for this section will be considered for publication in the appropriate section of Acta Crystallographica or in Journal of Applied Crystallography.

Acta Cryst. (1993). B49, 784–786

1-Carbonyl- μ -carboxylato-1 κ C:2 κ O:2 κ O'-1- η^5 -indenyl-2,2,2-triphenyl-1-(triphenylphosphine)iron tin

BY DOROTHY H. GIBSON,* JOHN F. RICHARDSON* AND OKEY P. MBADIKE

Department of Chemistry, University of Louisville, Louisville, KY 40292, USA

(Received 26 March 1993; accepted 24 May 1993)

Abstract. [FeSn(C₆H₅)₃(C₉H₇)(CO)(CO₂)₂{P(C₆H₅)₃}], $M_r = 855.33$, monoclinic, $P2_1/n$, $a = 17.715(5)$, $b = 13.156(4)$, $c = 17.749(5)$ Å, $\beta = 112.22(3)^\circ$, $V = 3829.2$ Å³, $Z = 4$, $D_x = 1.48$ g cm⁻³, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 11.1$ cm⁻¹, $F(000) = 1736$, $T = 296$ K, 7057 unique reflections, $R = 0.027$, $wR = 0.031$ for 5480 observed reflections with $I > 3\sigma(I)$. The coordination environment about the Fe atom consists of a triphenylphosphine, the five-membered ring of the indenyl ligand, a carbonyl and a C-bound carboxylate. A triphenyltin unit is bound to the O atoms of the carboxylate group giving a bimetallic complex. The Sn—O bond lengths differ by 0.467 Å which is considerably more than the difference observed for the related cyclopentadienyl analog; the steric demands of the bulky indenyl ligand are responsible for this difference. The geometry about the Sn atom is best described as a distorted trigonal bipyramid.

Introduction. The possibility for thermal activation of CO₂ through bifunctional systems having a highly basic metal center to bind carbon and an acidic center to bind one or both O atoms was suggested previously (Gambarotta, Arena, Floriani & Zanuzzi, 1982). As part of a general effort to synthesize and characterize bimetallic CO₂-bridged compounds (Gibson, Richardson & Ong, 1991; Gibson, Ye & Richardson, 1992) we have prepared the title compound (see Fig. 1); its structure determination and comparisons with two other closely related compounds are presented herein.

Experimental. Under nitrogen, (η^5 -C₉H₇)Fe(CO)₂-(PPh₃)⁺I⁻ (2.00 g, 3.25 mmol) and Ph₃SnCl (1.25 g,

3.25 mmol) were dissolved in 15 ml of THF and cooled to 273 K. A solution of KOH (0.73 g, 13.0 mmol) in 2 ml of H₂O was added, with stirring, to this mixture. The mixture was allowed to stir for 10 min and became dark red during this time. Cold (273 K) water, 15 ml, was added and the mixture was then transferred to a separatory funnel to separate the organic layer. This layer was then dried over MgSO₄, filtered, and concentrated on a rotary evaporator. Cold ether (30 ml) was then added to precipitate the product as a red-orange powder (1.50 g, 54% yield). A sample of the product was dissolved in CH₂Cl₂ and the solution was carefully layered with pentane and then chilled to 243 K; orange crystals were obtained after 1 week.

X-ray diffraction data were obtained with an Enraf-Nonius CAD-4H diffractometer, Mo $K\alpha$ radiation, incident-beam graphite monochromator, from an orange pyramidal crystal having approximate dimensions 0.23 × 0.32 × 0.40 mm mounted on a glass fiber in a random orientation. The cell dimensions were taken from a least-squares refinement of the setting angles of 25 reflections whose θ angles ranged from 13 to 16°. An orthorhombic C -centered cell ($a = 19.775$, $b = 29.439$, $c = 13.156$ Å, $\alpha = \beta = 90$, $\gamma = 89.88^\circ$) was considered but eliminated due to the lack of mmm symmetry [axial photos; $R_{int}(F^2) = 0.54$ for observed equivalent reflections]. Data were

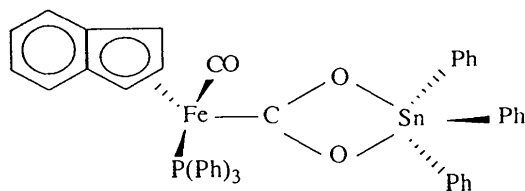


Fig. 1. Sketch of the title complex (Ph = phenyl ring).

* Authors to whom correspondence should be addressed.