SHORT COMMUNICATIONS

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Acta Cryst. (1984). C**40**, 570

Redetermination of the structure of potassium hexafluorosilicate, K_2SiF_6.

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(*Received* 11 *October* 1983; *accepted* 23 *November* 1983)

Abstract

$M_r = 220{\cdot}27$, cubic, $Fm3m$, $a_o = 8{\cdot}134$ (1) Å, $V = 538{\cdot}2$ (2) Å^3, $Z = 4$, $D_m = 2{\cdot}71$ (1), $D_x = 2{\cdot}719$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 19{\cdot}9$ cm^{-1}, $F(000) = 424$, $T = 295$ K, $R = 0{\cdot}015$ for 89 unique observed reflections. The Si–F bond length, 1·683 (2) Å, is consistent with those of other hexafluorosilicates.

Introduction

K_2SiF_6 was chosen for an undergraduate physical chemistry laboratory experiment (Loehlin & Norton, 1983) designed to use both diffraction angle and intensity information to illustrate how molecular parameters are obtained crystallographically. This redetermination was made to provide accurate data for the experiment and to check the suspiciously long (1·76 Å) bond reported by Ketelaar (1935).

Experimental

D_m from suspension in tribromomethane–tetrachloromethane. Aqueous recrystallization gave 0·2 mm cube from commerical salt (Pfaltz & Bauer, Inc.). Syntex $P2_1$ diffractometer with graphite monochromator. Lattice parameter from 16 reflections with $2\theta = 41$ to 46°. Empirical absorption correction (transmission factors 0·80 to 1·00). $(\sin\theta/\lambda)_{max} = 0{\cdot}807$ Å^{-1}. hkl range: 0 to 13. Three standard reflections, maximum deviation = 2σ from mean. 358 reflections measured, 89 unique reflections, $R_{int} = 0{\cdot}018$, no unobserved reflections, $[I/\sigma(I)]_{min} = 3{\cdot}0$. Ketelaar (1935) structure refined by full-matrix least squares based on F. Parameters refined and results: scale factor; K: $U = 0{\cdot}0238$ (4) Å^2; Si: $U = 0{\cdot}0169$ (4) Å^2; F: $x = 0{\cdot}2069$ (2), $U_{11} = 0{\cdot}0162$ (6) Å^2, $U_{22} = 0{\cdot}0352$ (5) Å^2. Secondary extinction $r^* = 2{\cdot}6$ (2) $\times 10^{-3}$, where $F_c^* = k|F_c|(1 + 2r^*|F_c|^2\delta)^{-1/4}$ (Larson, 1970).† $R = 0{\cdot}015$, $wR = 0{\cdot}041$, $S = 1{\cdot}26$; $1/w = \sigma(F)^2 + (0{\cdot}025|F_o|)^2$, $\sigma(F)$ from counting statistics. $(\Delta/\sigma)_{max} = 0{\cdot}020$. $\Delta\rho = -0{\cdot}41$ to 0·28 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970). Independent refinements by Syntex (1976) and *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) gave parameters within reported uncertainties. *XRAY*76 values given.

Discussion

The 8·134 (1) Å value for the lattice constant is consistent with the 8·13 Å determination of Cox & Sharpe (1953) and the 8·133 Å one at 299 K of the National Bureau of Standards (1955) but differs significantly from 8·184 (7) Å as given by Ketelaar (1935) and the 8·18 Å measurement of Cipriani (1954). The 1·683 (2) Å Si–F bond length is consistent with the range of reported values in other hexafluorosilicates, 1·643 (5)–1·706 (9) Å (Ray, Zalkin & Templeton; 1973; Lynton & Siew, 1973). The given value is calculated assuming independent thermal motion of the atoms and would be expected to be somewhat longer (~1%) if the more probable model, assuming librational motion of the entire SiF_6^{2-} ion, had been used.

I wish to thank Professor Bruce Foxman of Brandeis University for the use of his diffractometer and for many helpful discussions related to this study.

References

CIPRIANI, C. (1954). *Rend. Soc. Mineral. Ital.* **10**, 253–268.

COX, B. & SHARPE, A. G. (1953). *J. Chem. Soc.* pp. 1783–1784.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **5**, 1891–1898.

International Tables for X-ray Crystallography (1968). Vol. III, 2nd ed., pp. 202–203. Birmingham: Kynoch Press.

KETELAAR, J. A. A. (1935). *Z. Kristallogr.* **92**, 155–156.

LARSON, A. C. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 209–213. Copenhagen: Munksgaard.

LOEHLIN, J. H. & NORTON, A. P. (1983). *Crystallographic Determination of Molecular Parameters–A Physical Chemistry Laboratory Experiment.* 185th Natl Meet., Am. Chem. Soc., Div. Chem. Ed. No. 69, Seattle, WA. In preparation.

LYNTON, H. & SIEW, P-Y. (1973). *Can. J. Chem.* **51**, 227–229.

National Bureau of Standards (1955). *Circular* 539, Vol. 5, p. 50.

RAY, S., ZALKIN, A. & TEMPLETON, D. H. (1973). *Acta Cryst.* B**29**, 2741–2747, 2748–2751.

STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Syntex (1976). *Syntex XTL Operations Manual*, 2nd ed. Syntex Analytical Instruments, Cupertino, California.

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39044 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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