CHEHNA, M., PRADERE, J. P., VICENS, J., TOUPET, L. & QUINIOU, H. (1988). *Bull. Soc. Chim. Fr.* **5**, 897–900.

DECORET, C., BAYARD, F., BAERT, F., CHEHNA, M., PRADERE, J. P. & VICENS, J. (1990). *Mol. Cryst. Liq. Cryst.* **187**, 109–114.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

METZGER, J. V. (1979). Editor. *The Chemistry of Heterocyclic Compounds*, Vol. 34, *Thiazole and Its Derivatives*. New York: Wiley.

MORIN, R. B. & GORMAN, M. (1982). Editors. *Chemistry and Biology of β-Lactam Antibiotics*. New York: Academic Press.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SCHROTH, W., SPITZNER, R., FREITAG, S., RICHTER, M. & DOBNER, B. (1986). *Synthesis*, **11**, 916–920.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TEA, G. C., CHEHNA, M., PRADERE, J. P., DUGUAY, G. & TOUPET, L. (1986). *Phosphorus Sulfur*, **27**, 327–339.

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Acta Cryst. (1992). C**48**, 731–733

Structure of Potassium Strontium Cuprate, $K_{0.95}Sr_{0.05}CuO_2$

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Abstract. $K_{0.95}Sr_{0.05}CuO_2$, $M_r = 137.07$, orthorhombic, *Cmcm*, $a = 4.3727\,(1)$, $b = 11.6794\,(4)$, $c = 5.4112\,(1)$ Å, $V = 276.35\,(2)$ Å^3, $Z = 4$, $D_x = 3.296$ g cm^{-3}, $\lambda(\text{Cu}\ K\alpha_1) = 1.54056$ Å, $\mu = 231.85$ cm^{-1}, $F(000) = 259.8$, $T = 298$ K, $R_{wp} = 14.57\%$, $R_{ex} = 10.48\%$, $\chi^2 = 1.93$ for 22 basic variables with 4900 observations corresponding to 115 reflections. The structure of the title compound has been refined by Rietveld analysis of X-ray powder diffraction data. Cu is surrounded by four O atoms in a rectangular configuration. These CuO_4 units share edges to form infinite chains, which are separated from each other by K^+/Sr^{2+} ions in trigonal prismatic sites formed by the oxide ions. $K_{0.95}Sr_{0.05}CuO_2$ is a solid solution based on the stoichiometric end member $KCuO_2$ and represents a mixed-valence compound rich in copper(III).

Experimental. $K_{0.95}Sr_{0.05}CuO_2$ was prepared as a polycrystalline powder by solid-state reaction between KO_2, SrO_2 and CuO. Because of the air-sensitive nature of the materials all manipulations were carried out in a Miller–Howe high-integrity argon-filled glove box. Appropriate quantities of the starting materials were ground together in an agate mortar and pestle for 15 min. The resulting mixture was then formed into pellets and placed in a Pt boat which was located within a silica tube fitted with two gas breakthroughs and taps. The tube was removed from the glove box, connected to an oxygen supply, and placed within a cylindrical furnace. The sample was heated under flowing oxygen at 673 K for 6 d. Weight-loss measurements confirmed the loss of only O_2 from the reaction mixture. Phase purity was determined by X-ray powder diffraction photographs using a Stoe Guinier camera with Cu $K\alpha$ radiation. Solid-solution formation was confirmed by shifts in lattice parameters away from those of stoichiometric $KCuO_2$ ($a = 4.3857$, $b = 11.6878$, $c = 5.4135$ Å) (Pienkowski, Bruce, Abrahams, Janes &

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Table 1. *Refined atomic parameters for* $K_{0.95}Sr_{0.05}CuO_2$ *with e.s.d.'s in parentheses*

	Site	x	y	z	B_{iso}(Å^2)	Occupancy
K	4(*c*)	0.0	0.3007 (3)	0.75	0.6 (1)	0.955 (5)
Sr	4(*c*)	0.0	0.3007 (3)	0.75	0.6 (1)	0.045 (5)
Cu	4(*c*)	0.0	−0.0018 (2)	0.25	0.68 (5)	1.0
O	8(*f*)	0.0	0.1017 (4)	0.003 (1)	0.5 (1)	1.0

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *in* $K_{0.95}Sr_{0.05}CuO_2$ *with e.s.d.'s in parentheses*

		Symmetry code
Cu—O	1.800 (6)	x, y, z
Cu—O′	1.800 (6)	$x, -y, -z$
K—O	2.699 (6)	x, y, z
K—O″	2.804 (4)	$x+\frac{1}{2}, \frac{1}{2}-y, -z$
O—Cu—O′	83.5 (2)	

Edwards, 1992). Oxygen contents were determined by thermogravimetric decomposition in air.

A sample of the title compound was sealed in a 0.5 mm capillary and an X-ray powder diffraction profile collected at room temperature on a Stoe STADI/P diffractometer in capillary mode using Ge-monochromated Cu $K\alpha_1$ radiation. Data were collected in the 2θ range 10–108° in steps of 0.02°. Calibration was achieved using an external Si standard. Intensity data were corrected for cylindrical absorption by linear interpolation of tabulated literature values (*International Tables for X-ray Crystallography*, 1972, Vol. II, pp. 295–298). A value for μR of 3.5 was derived from measurements of the sample transmission factor at $\theta = 0°$, and was used in all subsequent calculations. The structure was refined by Rietveld analysis using a program for multiphase profile refinement (*MPROF* vX14.4; Bendall, Fitch & Fender, 1983), with peak shapes modeled by a variable Lorentzian function. Scattering factor curves for neutral atoms were used and corrections for real and imaginary parts of the anomalous dispersion applied (*International Tables for X-ray Crystallography*, 1974, Vol. IV, pp. 99–101, 149–150). A simultaneous polynomial background fit was carried out with five variable terms. A correction for the monochromator polarization fraction was also applied. Profile plots were produced using *GENIE* (David *et al.*, 1986) and structural projections generated using *PLUTO* (Motherwell, 1979).

The previously determined structure of $KCuO_2$ (Hestermann & Hoppe, 1969) was used as a starting model in the Rietveld refinement with Sr sharing the K site. Initially scale and background parameters were refined, followed in subsequent iterations by cell, peak shape, atomic and isotropic thermal parameters. The location of the Sr was confirmed by allowing the K/Sr occupancy ratio to vary and by comparison with a model where the site was fixed with full occupancy by K. The K/Sr occupancies refined to within one e.s.d. of that expected from the formula and gave a final R factor of $R_{wp} = 14.57\%$; this compares with a value of 14.66% for the refinement with K only and represents a significant improvement at a 0.005 confidence level (Hamilton, 1965). $\Delta/\sigma = 0.015$. Attempts to locate Sr in the other available trigonal prismatic site resulted in a negative occupancy on that site.

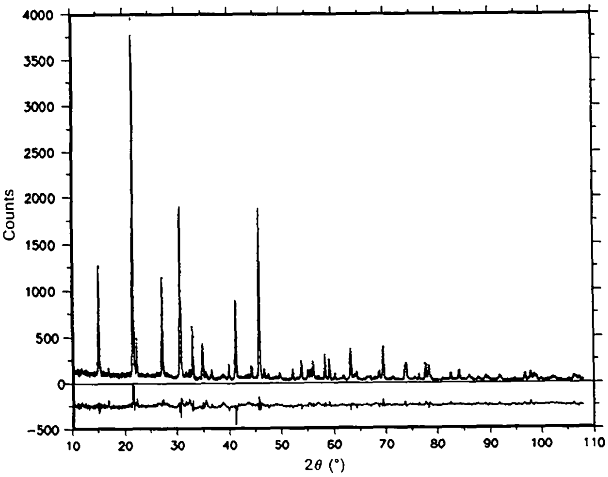

Fig. 1. Final profile plot for $K_{0.95}Sr_{0.05}CuO_2$ showing observed (points), calculated (line) and difference (lower) profiles.

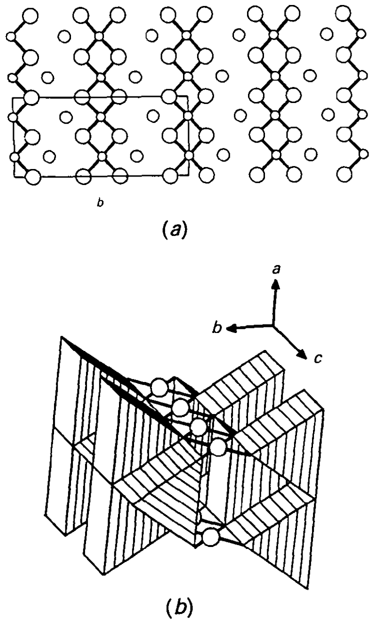

Fig. 2. (*a*) Projection down the *a* axis of $K_{0.95}Sr_{0.05}CuO_2$, where open circles represent O, K/Sr and Cu ions in order of decreasing size. (*b*) View of the structure of $K_{0.95}Sr_{0.05}CuO_2$. K/Sr ions are located within the trigonal prismatic sites, open circles represent Cu ions.

In the final refinement there were no correlations between structural parameters above 35%. Refined atomic parameters are given in Table 1 with selected interatomic contact distances and angles in Table 2. The final difference profile is shown in Fig. 1* with views of the structure shown in Fig. 2.

Related literature. The structure determination of stoichiometric $KCuO_2$ has been reported previously (Hestermann & Hoppe, 1969; Brese, O'Keeffe, von Dreele & Young, 1989). Electrical and magnetic data for the title compound are presented elsewhere (Pienkowski *et al.*, 1992).

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References

BENDALL, P. J., FITCH, A. N. & FENDER, B. E. F. (1983). *J. Appl. Cryst.* **16**, 164–170.

BRESE, N. E., O'KEEFFE, M., VON DREELE, R. B. & YOUNG, V. G. JR (1989). *J. Solid State Chem.* **83**, 1–7.

DAVID, W. I. F., JOHNSON, M. W., KNOWLES, K. J., MORETON-SMITH, C. M., CROSBIE, G. D., CAMPBELL, E. P., GRAHAM, S. P. & LYALL, J. S. (1986). *GENIE. A Language for Spectrum Manipulation and Display.* Rutherford–Appleton Laboratory, Oxon OX11 0QX, England.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.

HESTERMANN, K. & HOPPE, R. (1969). *Z. Anorg. Allg. Chem.* **367**, 249–260.

MOTHERWELL, W. D. S. (1979). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

PIENKOWSKI, M. C., BRUCE, P. G., ABRAHAMS, I., JANES, R. & EDWARDS, P. P. (1992), *J. Mater. Chem.* Submitted.

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* A list of primary diffraction data, *i.e.* the numerical intensity of each measured point on the profile as a function of scattering angle, has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54711 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0076]

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Structure of Orthorhombic $NbPO_5$

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Abstract. Niobium phosphate, $M_r = 203.88$, orthorhombic, $Pnma$, $a = 11.304\,(2)$, $b = 5.316\,(2)$, $c = 6.640\,(2)$ Å, $V = 399.0\,(3)$ Å^3, $Z = 4$, $D_x = 3.39$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.71069$ Å, $\mu = 31.8$ cm^{-1}, $F(000) = 384$, $T = 296$ K, $R = 0.030$, $wR = 0.043$ for 462 unique reflections ($R_{\text{int}} = 0.017$) with $F_o^2 > 3\sigma(F_o^2)$. The framework is built up from NbO_6 octahedra and PO_4 tetrahedra. The NbO_6 octahedra share opposite corners to form infinite zigzag chains along the *b* axis. These parallel octahedral chains are interconnected to each other *via* corner-sharing PO_4 tetrahedra.

Experimental. Gem-shaped crystals of $NbPO_5$ were obtained from a reaction in the K–Nb–P–O system. The single crystals were grown at 1375 K from a reaction with the target composition $KNb_{12}P_6O_{33}$. A crystal, with average dimensions $0.15 \times 0.1 \times 0.05$ mm, was selected for indexing and intensity data collection. Diffraction data were collected by an ω scan ($4°\omega$ min^{-1}, $\pm 0.45°$ in ω) at room temperature on a Rigaku AFC5S four-circle diffractometer equipped with a graphite monochromator. The unit-cell parameters and the orientation matrix for data collection were determined by the least-squares fit of 25 peak maxima with $7 < 2\theta < 27°$. There was no detectable decay during the data collection according to the intensities of three standard reflections ($\bar{1}2\bar{1}$, $\bar{1}1\bar{1}$, $\bar{3}3\bar{1}$) measured every 100 reflections. Total of 1043 reflections ($0 \le h \le 14$, $-6 \le k \le 6$, $0 \le l \le 8$; $2\theta_{\text{max}} = 55°$) were measured, of which 839 reflections were observed. The *TEXSAN* (Molecular Structure Corporation, 1989) software package was used for the crystal structure solution and refinement. The systematic extinction conditions, $(0kl)$: $k + l \ne 2n$ and $(hk0)$: $h \ne 2n$, led to two possible space groups, $Pn2_1a$ (No. 33) and $Pnma$ (No. 62). The final space group, $Pnma$, was determined by successful structure refinement using the program *PROCESS*. Lorentz–polarization and empirical absorption corrections (transmission range 0.94–1.00) based on three azimuthal scans ($2\theta = 16.37$, 23.09, 23.90°) were

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