LEONARD, B. F., DESBOROUGH, G. A., AND N. J. PAGE (1969). Ore microscopy and chemical composition of some laurites. *Amer. Mineral.* **54,** 1330–1346.

MANDARINO, J. A. (1964) New data, osmium-iridium alloys. [Discussion of HEY (1963)]. *Amer. Mineral.* **49,** 818–819.

PALACHE, C., BERMAN, H., AND C. FRONDEL (1944). *The System of Mineralogy . . . of Dana Vol. 1.* John Wiley, New York.

STUMPFL, E. F., AND A. M. CLARK (1965). Electron-probe microanalysis of gold-platinoid concentrates from southeast Borneo. *Bull. Inst. Mining. Met.* (*London*) **708,** 933–946.

WITTRY, D. B. (1964). Methods of quantitative electron microprobe analysis. *Proc. Twelfth Ann. Conf. Applied X-Ray Analysis, Denver, 1963.* Plenum Press, New York, pp. 395–418.

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# SYNTHESIS OF SCANDIUM PSEUDOBROOKITE, $Sc_2TiO_5$[1]

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ABSTRACT

Synthesis of small crystals (1×1×2 mm) of $Sc_2TiO_5$, scandium pseudobrookite, has been effected by slow cooling from 1,460°C–1,000°C using sodium tungstate, $Na_2WO_4$, as a flux. Complete solid-solution series were obtained between pseudobrookite, $Fe_2TiO_5$, and scandium pseudobrookite, $Sc_2TiO_5$, by heating at 1,150°C gels in air.

Isostructural compounds $R_2^{3+}$ with ortho-rhombic space group *Bbmm* include $Al_2TiO_5$ (Yamaguchi, 1944; Lang, Filmore, and Maxwell, 1952; Kim and Hummel, 1960; Goldberg, 1968), $Ga_2TiO_5$ (Goldberg, 1968), and $Fe^3TiO_5$, pseudobrookite (Akimoto, Nagata and Katsura, 1957; Karkhanavala, 1959; McChesney and Muan, 1959; Goldberg, 1968; Buddington and Lindsley, 1964).

Synthesis of small crystals of $Sc_2TiO_5$, scandium pseudobrookite, has been effected by slow cooling (2°C/hr.) from 1,460–1,000°C in Pt crucible of $Sc_2O_3$ and $TiO_2$ in sodium tungstate, $Na_2WO_4$, as a solvent. Optimum charge ratio of the oxides and flux was found to be in the vicinity of $Sc_2O_3 = 22.0$. $TiO_2 = 22.0$ and $Na_2WO_4 = 56.0$ (in mole per cent).

The tabular to prismatic crystals are faintly yellow due to $Fe_2O_3$ (0.17 percent). They are elongated on [001], measure approx. 1×1×2 mm and often show terminal faces. At the end of the runs, only $Sc_2TiO_5$ remained after dissolving the flux in hot water. Previously reported

[1] Mineralogical Contribution No. 480.

TABLE 1. X-RAY POWDER DATA FOR SYNTHETIC SC-PSEUDOBROOKITE
$Sc_2TiO_5$, *BBMM*, *a* 10.127(1), *b* 10.274(1), *c* 3.8509(5).
Cu radiation (Ni filtered) $K\alpha_1 = 1.54051$. All *d*-values calculated with computer program (IBM 7094) and are given in Angstrom. $Sc_2TiO_5$ contains 0.17% of $Fe_2O_3$; specific gravity is $3.62 \pm 0.01$.

| *h k l* | $I/I_0$ | *d* (obs.) | *d* (calc.) |
|---|---|---|---|
| 0 2 0 | 20 | 5.13 | 5.14 |
| 2 0 0 | 50 | 5.06 | 5.06 |
| 1 0 1 | 100 | 3.60 | 3.60 |
| 1 1 1 | 8 | 3.397 | 3.397 |
| 1 2 1 | 5 | 2.946 | 2.948 |
| 2 3 0 | 100 | 2.838 | 2.837 |
| 3 0 1 | 30 | 2.537 | 2.538 |
| 1 3 1 | 25 | 2.480 | 2.481 |
| 2 4 0 | 20 | 2.290 | 2.290 |
| 4 2 0 | 25 | 2.271 | 2.271 |
| 4 3 0 | 30 | 2.037 | 2.036 |
| 0 0 2 | 20 | 1.926 | 1.925 |
| 2 5 0 | 10 | 1.905 | 1.904 |
| 3 4 1 | 5 | 1.806 | 1.805 |
| 2 0 2 | 5 | 1.801 | 1.800 |
| 0 6 0 | 15 | 1.713 | 1.712 |
| 5 2 1 | 20 | 1.693 | 1.692 |
| 6 1 0 | 5 | 1.666 | 1.666 |
| 2 6 0 | 5 | 1.623 | 1.622 |
| 3 5 1 | 5 | 1.597 | 1.597 |
| 2 3 2 | 25 | 1.593 | 1.593 |
| 5 3 1 | 20 | 1.588 | 1.588 |
| 1 6 1 | 5 | 1.547 | 1.546 |
| 2 4 2 | 5 | 1.474 | 1.474 |
| 5 4 1 | 10 | 1.469 | 1.470 |
| 6 4 0 | 3 | 1.410 | 1.410 |
| 4 3 2 | 8 | 1.399 | 1.399 |
| 1 7 1 | 5 | 1.359 | 1.360 |
| 7 0 1 | 8 | 1.354 | 1.354 |
| 7 1 1 | 3 | 1.343 | 1.343 |
| 7 2 1 | 15 | 1.309 | 1.310 |
| 0 6 2 | 5 | 1.280 | 1.280 |
| 1 3 3 | 5 | 1.194 | 1.194 |

$2Sc_2O_3 \cdot 3TiO_2$ and $3Sc_2O_3 \cdot 2TiO_2$ have not been found as products (Kommissarova and Pokrovskii, 1966).

Single-crystal study using the precession method with $MoK\alpha_1$ radiation indicates that $Sc_2TiO_5$ has the same space group as natural pseudobrookite, *Bbmm*, given by Pauling (1930), and the other $R^{3+}{}_2TiO_5$ compounds mentioned above. Unambiguously indexed computer refined

TABLE 2. UNIT-CELL DIMENSIONS OF THE SOLID-SOLUTION SERIES OF Sc-PSEUDOBROOKITE—PSEUDOBROOKITE (ORTHORHOMBIC *BBMM*).

| Composition | Temperature (°C) | $a$(Å) | $b$(Å) | $c$(Å) | $V$(Å$^3$) |
|---|---|---|---|---|---|
| $Sc_2TiO_5$[1,2]($Fe_2O_3$ 0.17%) | Grown in flux 1,300–1,000°C | 10.127 (1) | 10.274 (1) | 3.8509 (5) | 400.66 |
| $(Sc_{75}Fe_{25})_2TiO_5$ | 1,150 | 10.07 | 10.23 | 3.810 | 392.4 |
| $(Sc_{50}Fe_{50})_2TiO_5$ | 1,150 | 9.979 | 10.14 | 3.776 | 382.1 |
| $(Sc_{25}Fe_{75})_2TiO_5$ | 1,150 | 9.904 | 10.06 | 3.757 | 374.8 |
| $Fe_2TiO_5$ | 1,150 | 9.813 | 9.975 | 3.729 | 365.0 |

[1] Spectographic analysis of the flux grown crystals of $Sc_2TiO_5$ gave: Si, Mg, Na, Al≅0.01%; Ni, Cu, Ca, Sn≅0.001% and Be, Yb≅0.0001%.

[2] This compound has been synthesized also by heating the gel at 1,150°C in air, but the flux-grown crystals were chosen for the unit-cell refinement.

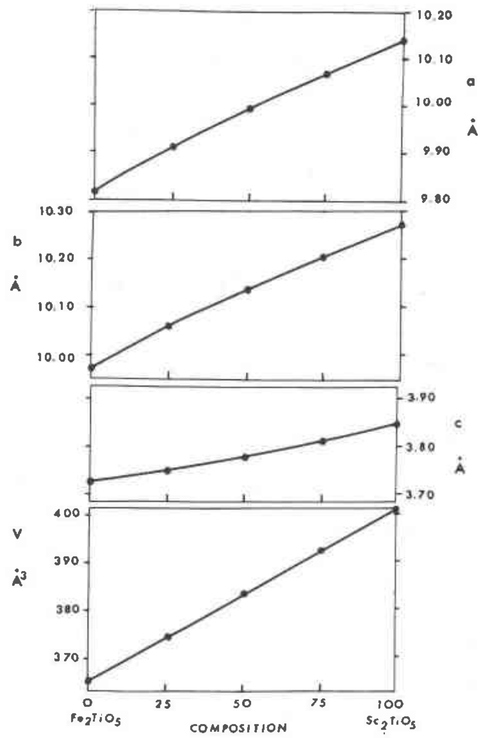


FIG. 1. Unit-cell dimensions of the solid-solution series of Sc-pseudobrookite———pseudobrookite.
$Sc_2TiO_5$ $Fe_2TiO_5$

powder data confirmed by the intensities observations on the precession photographs are given in Table 1. Specific gravity determined by floating crystals in Clerici solution gave 3.62 (calculated Sp. Gr. 3.61).

Natural pseudobrookite, $Fe_2^{3+}TiO_5$, containing 0.74 percent of $Sc_2O_3$ was reported from the Thomas Mountains, Utah (Frondel, 1970). Complete solid-solution series were obtained between pseudobrookite, $Fe_2^{3+}TiO_5$, and scandium pseudobrookite, $Sc_2TiO_5$, by heating at 1,150°C gels containing the exact amount of components precipitated by ammonia.

Unit-cell dimensions of two synthetic end-members and three intermediate compounds were calculated from the powder diffraction data, and are given in Table 2 and Figure 1.

Efforts to synthesize the indium analogue of pseudobrookite failed, $In_2Ti_2O_7$ and $In_2O_3$ being formed instead under the present experimental conditions.

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## References

Akimoto, S., T. Nagata, and T. Katsura (1957): $TiFe_2O_5$—$Ti_2FeO_5$ solid solution series. *Nature* **179,** 37–39.

Buddington, A. F., and D. H. Lindsley (1964): Iron titanium oxide minerals and synthetic equivalents. *J. Petrology* **5,** 310–57.

Frondel, C. (1970): Scandium-rich minerals from rhyolite in the Thomas Mountains, Utah. *Amer. Mineral.* **55,** 1058–1060.

Goldberg, D. (1968): Contribution a l'etude des systems formes par l'alumine avec quelques oxides de metaux trivalents se tetravalents, en particulier l oxide de titane. *Rev. Int. Hautes Temp.* **5,** 181–94.

Karkhanavara, M. D., and A. C. Momin (1959): Subsolidus reactions in the system $Fe_2O_3$-$TiO_2$. *J. Amer. Ceram. Soc.* **42,** 399–402.

Kim, J. H., and F. A. Hummel (1960): Studies in Lithium oxide systems: IX, $Li_2O$-$A_2Ol^3$-$TiO_2$. *J. Amer. Ceram. Soc.* **43,** 611–614.

Komissarova, L. N., and B. I. Pokrovskii (1966): Reaction of scandium with titanium dioxide. *Dokl. Akad. Nauk SSSR* **168,** 1076–79

Lang, S. M., C. L. Filmore, and L. H. Maxwell (1952): The system beryllia-alumina-titania: Phase relations and general physical properties of three components porcelains. *J. Res. U.S. Nat. Bur. Stand.* **48,** 298–312.

McChesney, J. B., and A. Muan (1959): Studies in the system iron oxide-titanium oxide at low oxygen pressures. *Amer. Mineral.* **46,** 572–82.

Pauling, L. (1930): The crystal structure of pseudobrookite. *Z. Kristallogr.* **73,** 97–112.

Yamaguchi, G. (1944): Studies on tieilite. *J. Jap. Ceram. Assoc.* **52,** 6–7.