CRYSTAL DATA 349

randomly distributed in 2(a): 0 0 0; 0 ½ ½. O in 4(f): x x 0; 
½ x ½ ½ x; ½ ½ x x. Thus, to specify the atomic
positions, one space-group variable, x, must be determined.

Experimental
CrSbO₄ was prepared by heating stoichiometric amounts of
Cr₂O₃ and Sb₂O₃ in air at 1473 K for 3 d. FeSbO₄ was
prepared from a mixture of Fe₂O₃ and Sb₂O₃ of molar ratio
Fe'Sb=1:2 heated at 1173 K for 58 h, crushed and
reannealed at 1273 K for 2 d. Alumina crucibles, analytical
grade reagents and a Heraeus KS-120 furnace were employed.
The temperatures were read with calibrated Pt–Pt 10% Rh
thermocouples and are reliable to ± 10 K. The densities of the
samples were determined pycnometrically.

Powder data were recorded with Cu nickel-filtered
radiation (λ = 1.540598 Å), with W (99.99%) as internal
standard, a Philips PW 1310 diffractometer and PW 1050/25
goniometer provided with a curved LiF-crystal-focusing
monochromator, at a scanning rate of 0.125 ° 20 min–¹.

The setting of the samples, and the determination of
interplanar spacings and lattice constants were made
according to the methods followed by JCPDS
(Powder Diffraction Data, 1976). The unit-cell parameters were refined
from the 20 values of the last ten nonaxial reflexions. The
integrated intensities of 0 0 0 were measured with a
Hewlett-Packard 9830A computer provided with a 9864A
digitizer.

The intensities were calculated for x=0.309, 0.310, ..., 0.323 with the computer program LAZY PULVERIX
of Yvon, Jeitschko & Parthé (1977), the scattering factors for
neutral atoms, Debye-Waller factors (Cr 0.65; Sb 0.30; O
0.80; and Fe 0.60) and including correction for anomalous
dispersion. The discrepancy factor R=100 (Σ|I o – I c|)/ΣI o
was computed after making ΣI o = ΣI c.

Results and discussion
The sample of CrSbO₄ was deep gray yellowish brown, and
that of FeSbO₄ was deep yellowish brown. Tables 1 and 2
include the cell dimensions, the interplanar spacings, d, and
the observed, I o, and calculated, I c, intensities for both
compounds. Systematic extinctions confirmed the space

Table 3. Distances M–O and Sb–O in MSbO₄ (M = Cr, Fe)

<table>
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<tr>
<th>Compound</th>
<th>d₁</th>
<th>d₂</th>
<th>d</th>
<th>d₂</th>
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<td>1.975</td>
<td>1.975</td>
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<td>2.066</td>
<td>1.960</td>
<td>1.995</td>
<td>1.995</td>
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</table>

In these oxides the metallic atoms, as well as those of Sb, are
coordinated with six O atoms, two of them at d₁ = 2 ½ a and
the other four at d₂ = a(2x² - 2x + ½ + 2c² a - 2)².

The average sums (M–O, Sb–O) of ionic radii (Shannon &
Prewitt, 1970), d, are also included for comparison with d.

We express our gratitude to Professor E. Parthé, for making
available to us the program LAZY PULVERIX, to the Junta
de Energia Nuclear of Madrid, for permission to use its
UNIVAC 1100 computer; and to Miss B. Galar for technical
assistance.

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1046–1048.
10, 73–74.


Crystal data for Lu₂Si₂O₇. By F. BRETHEAU-RAYNAL, M. LANCE and P. CHARPIN, Section de Chimie Moléculaire,
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(Received 26 November 1980; accepted 19 March 1981)

Abstract
Lutetium pyrosilicate single crystals Lu₂Si₂O₇ were grown by
a floating-zone technique associated with an arc image
furnace. The compound is monoclinic, space group C2/m,
Z=2, a = 6.7665 (14), b = 8.4047 (18), c = 4.7195 (27) Å
and β = 101.95 (3)°. Indexed X-ray powder data at 294 K are given.

Origin of the samples
Single-crystal growth was achieved with a crucible-free
method involving the floating-zone technique associated with a
biellipsoid image furnace, described in detail in previous
papers (Bretheau-Raynal, Dalbiez, Drifford & Blanzat, 1979;
Bretheau-Raynal, Tercier, Blanzat & Drifford, 1980).
CRYSTAL DATA

Table 1. Crystal data for Lu₂Si₂O₇, compared with literature values

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<td>a (Å)</td>
<td>6.78</td>
<td>6.755 ± 0.5*</td>
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<td>b (Å)</td>
<td>8.84</td>
<td>8.8369 ± 5*</td>
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<td>c (Å)</td>
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<td>4.7121 ± 3</td>
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* Errors in the last digit.

Table 2. Powder data for Lu₂Si₂O₇ at 294 K

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Unit cell and powder data

The samples were characterized by X-ray diffraction (Guinier patterns). Lu₂Si₂O₇ is monoclinic, isostructural with ytterbium pyrosilicate (Smolin & Shepelev, 1970), space group C2/m, with two formula units per unit cell. The lattice constants were obtained from single crystals, with a Nonius CAD-4 diffractometer and graphite-monochromated Mo Kα radiation. They result from a least-squares fit to 25 indexed reflexions (Table 1; estimated standard deviations are given in parentheses). The powder data (Table 2) were determined from Guinier patterns (camera diameter = 114.6 mm, 2(Cu Kα) = 1.5405 Å). The d values were corrected with NaCl as internal standard [a = 5.6402 Å]. Intensities were measured with a 3CS Joyce-Loebl microdensitometer.

Comparison with other results

Different authors have already given values for the unit-cell parameters of Lu₂Si₂O₇, all obtained from powder samples. These values are summarized in Table 1 and compared with ours. The cell dimensions reported in the present study are close to most of them, particularly to Chateau’s (1976) parameters, which are less accurately determined. Felsche’s (1970) values are somewhat divergent from the others, especially the b parameter. This small discrepancy might be caused by traces of impurities in his powders, characteristic of the low solid-state reaction rate.

References