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The Crystal Structure of $CsYb_3F_{10}$: Refinement in a Higher-Symmetry Space Group*

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The crystal structure of $CsYb_3F_{10}$ has been reported as monoclinic, space group *Pc* (a = 4.2893(2), b = 6.7437(4), c = 16.196(2) Å, $\beta = 90°$); it was refined to an R of 0.031 for 1406 reflections with $F > 3\sigma(F)$ (*1*). The authors noted that Weissenberg photographs indicated an orthorhombic cell, and further noted that the atomic positions are "very close to those corresponding to a description of the structure in the centrosymmetric space group *Pmcm*"; however, they were unable to refine in *Pmcm* below an R of 0.09 and reported that the isotropic temperature factors for some of the F atoms became unacceptably large. Refinements in another orthorhombic space group, $Pmc2_1$, also led to unacceptable B's and an R of 0.077. Accordingly, the authors resorted to the monoclinic space group *Pc*.

I found no problem in refining the structure in *Pmcm* (space group No. 51; the standard setting is *Pmma*). The 1406 reflections obtained as supplementary material were averaged according to Laue group *mmm* to yield 810 independent values of F, and the atomic coordinates were recast and averaged relative to a center of symmetry at the cesium atom ($\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$). An initial least-squares refinement of the coordinates and anisotropic B's quickly converged at R = 0.049; introduction of a secondary extinction parameter led to a final R of 0.031 for 48 parameters—the same value as obtained by the earlier authors (*1*) for 127 parameters in *Pc*. The quantity minimized was $\Sigma w(F_o^2 - F_c^2)^2$; since weights w were not available, they were taken equal to $1/F_o^2$ for $F_o^2 \geq 80$ and $1/80\ F_o$ for $F_o^2 \leq 80$ (*2*). Final parameters are given in Table I. The final value for the extinction coefficient g (*3*) was 0.36(2) $\times 10^{-6}$.

This refinement experience is quite different from that reported by the earlier workers (*1*) in that the *Pmcm* structure now leads to a quite satisfactory R and to no unusual temperature factors. It is impossible to know what might have caused the earlier problems. They may have resulted from computer difficulties; in particular, the large B's for some of the F atoms suggest the possibility that the assignments of site multiplicities were in error (some of the atoms lie in sites of multiplicity 4 and others of 2; see Table I).

Recasting the structure in *Pmcm* has led

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TABLE I

FINAL PARAMETERS, SPACE GROUP *Pmcm*

Atom	No. in cell	x	y	z	B_{eq}[a]
Yb(1,3)	4	0	0.03449(7)	0.11365(4)	0.47(1)
Yb(2)	2	0	0.49218(11)	¼	0.49(1)
Cs	2	½	½	½	2.17(2)
F(1)	2	0	0.1251(17)	¼	1.3(3)
F(2,9)	4	0	0.1796(12)	0.5104(6)	1.7(2)
F(3,7)	4	½	0.0471(17)	0.3893(8)	2.3(2)
F(4,6)	4	0	0.3590(12)	0.3702(6)	1.8(3)
F(5,10)	4	0	0.2516(13)	0.8298(6)	2.9(4)
F(8)	2	½	0.4762(24)	¼	2.6(3)

[a] $(8\pi^2/3)(U_{11} + U_{22} + U_{33})$.

to shifts in the atomic positions by amounts ranging up to about 0.15 Å; and while the general description of the structure remains unchanged, many of the details are different. For example, whereas in the *Pc* structure the cesium atom lay in an asymmetric site with eight F neighbors at varying distances between 2.89 and 3.30 Å, in the *Pmcm* structure it lies on a site of 2/*m* symmetry with four F atoms at 3.05(1) Å and four others at 3.15(1) Å. The YbF_7 polyhedra are similarly, but less dramatically, more regular in the *Pmcm* description.

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