tively. It was evident that this is not so. The calculated intensities, listed in Table 1, were corrected with the appropriate *LP* factor and scaled to the observed intensities. No correction for temperature movement was applied. The atom positions used in the calculation are:

Space group $P6_3mmc$

4 S in f $\frac{1}{3}, \frac{2}{3}, z;\ \ldots;\ z=\frac{1}{8}$ (assumed);
2 Nb in d $\frac{1}{3}, \frac{2}{3}, \frac{3}{4};\ \frac{2}{3}, \frac{1}{3}, \frac{1}{4}$;
4/3 Cu in f $\frac{1}{3}, \frac{2}{3}, z;\ \ldots;\ z=0{\cdot}563$ (assumed).

As stated above, for z_{S} the value $\frac{1}{8}$ has been assumed. For z_{Cu} the above-mentioned value is calculated by assuming equal distances from Cu to the corners of the S tetrahedron. A better fit would probably be obtained by slight shifts in these parameters. However, for such a refinement the study of the isomorphous Se compound seems to be more suited. On the basis of the chosen parameters, atomic distances are as follows:

Nb–S	2·5 Å
Cu–S	2·5
Nb–Cu	2·5
Cu–Cu	2·5

Discussion

The structures of NbS_2, MoS_2 and $Cu_{0\cdot65}NbS_2$ are compared in Fig. 1(*a*), (*b*) and (*c*) respectively. The shift of the Nb from its positions in NbS_2 to those in $Cu_{0\cdot65}NbS_2$ is the more remarkable since we were unable to prepare an analogous compound Cu_xMoS_2 ($\sim 0{\cdot}6 \le x \le 0{\cdot}8$). The distance Nb–Cu is comparable to the sum of the metallic atomic radii.

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Crystal symmetry of the dimer of cyclobutene-1,2-dicarboxylic acid dimethyl ester.

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In a review article on small ring compounds, Vogel (1960) mentions several interesting tricyclic dimers of various derivatives of cyclobutene and cyclobutadiene. Although all of these compounds possess the same basic eight-membered 3-ring skeleton, there is considerable uncertainty about the configuration of individual members of this series. Criegee (1958) has shown from chemical evidence and dipole moment measurements that in the dimer of tetramethylcyclobutadiene the two cyclobutene rings are in a *syn* configuration. Kitahara, Caserio, Scardiglia & Roberts (1960) suggest that the dimer of fluorotriphenyl cyclobutadiene is *anti*-1,2-difluoro-3,4,5,6,7,8-hexaphenyltricyclo[4.2.0.$0^{2,5}$]octa-3,7-diene. In an X-ray diffraction investigation of this compound Fritchie & Hughes (1962) confirm this structure. Vogel & Roos (1962) have recently prepared the dimer of cyclobutene-1,2-dicarboxylic acid dimethyl ester. Although the gross structure resembles that of the above compounds there is no conclusive evidence of whether the compound is *syn* or *anti*. We have therefore undertaken an X-ray diffraction study of this compound.

The material was recrystallized from methanol as thin tetragonal blocks. Small single crystals were chosen for X-ray examination. Rotation and Weissenberg photographs around the *a* and *c* axes showed the substance to be tetragonal, with

$$a=7{\cdot}11\pm0{\cdot}01,\ c=33{\cdot}37\pm0{\cdot}03\ \text{Å}$$
$$(\lambda\ \text{Cu}\ K\alpha=1{\cdot}5418\ \text{Å})\ .$$

The density, as measured pycnometrically with t-butanol, is 1·29 g.cm^{-3}; the density calculated for four molecules of the dimer per unit cell is 1·33 g.cm^{-3}. The systematic conditions limiting possible reflections on the Weissenberg photographs, namely $(h00)$ with $h=2n$ and $(00l)$ with $l=4n$, indicate that the space group is the noncentric space group $P4_12_12(D_4^4)$ or the related $P4_32_12(D_4^8)$.

The general equivalent positions of these space groups are eightfold. Placing the atoms in the special fourfold positions would lead to a chemically unrealistic structure; therefore each molecule must contain two halves related by one of the symmetry elements of the space group. The only symmetry element leading to discrete molecules is the diagonal twofold axis. Unfortunately, the presence of a twofold axis in the molecule will not lead to a distinction between the *syn* and *anti* forms, nor does it lead to a choice from packing considerations. No further work is planned on this structure.

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