SHORT COMMUNICATION

The existence of metrically similar unit cells based on the same lattice: a precautionary note. By Wilson H.

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Lattices with metric pseudosymmetry may have two different unit cells which appear to be based on the same three lattice translations. The similar dimensions of the unit cells may lead to incorrect conclusions concerning their identity. An example of such a case is condelphine hydroiodide, which forms triclinic crystals with a metrically pseudohexagonal unit cell. A non-reduced unit cell exists with dimensions which are almost equal to the reduced cell. The least-squares refinement and reduction of both cells are presented in detail.

Computer-controlled diffractometers generally include programs in their software which search for, center and index reflections, thus establishing unit-cell dimensions with minimal user intervention. The user may not always be advised of the problems associated with this procedure. In particular, the possibility of the existence of *more than one unit cell, apparently based on the same set of three shortest translations* in reciprocal space, does not seem to be well documented. Lattices may exist for which two or more dimensionally similar unit cells may be found. However, they differ in orientation, and thus cannot be identical. An example of such a lattice is presented here as a precaution against the uncritical acceptance of the results of autoindexing programs.

Condelphine (Pelletier, Keith & Parthasarathy, 1967) forms a hydroiodide which crystallizes in the triclinic space group *P*1. Two sets of integrated intensity data were collected independently, both using Cu *Kα* radiation, but selecting crystals from different samples (Pelletier & Herald, 1970; Pelletier & Grimm, 1972). Comparison of corresponding reflections showed that the relative intensities differed by amounts far exceeding any reasonable experimental error Table 1 lists the unit-cell dimensions, as established by the diffractometer software, for both crystals.* The values reported for cell (I) were reduced using a locally written Delaunay reduction program (Pelletier & Herald, 1970). Cell (II) was reduced by the diffractometer software.

Table 1. *Unit-cell dimensions for condelphine hydroiodide, as established by diffractometer centering and autoindexing programs*

	Cell (I)	Cell (II)	Cell (I′)
a	9·34 Å	9·32 Å	9·34 Å
b	17·39	17·45	17·39
c	9·10	9·09	9·10
α	94·85°	94·84°	94·85°
β	119·15	118·83	119·15
γ	88·57	86·50	86·71

The minor differences in the dimensions of the two cells were initially assumed to result from experimental errors, especially absorption. The unit cell of a third crystal was

* Both cells were measured on an Enraf–Nonius diffractometer. However, cell (I) was found using the CAD-3 system, while cell (II) was found using the CAD-4 system.

Table 2. *Least-squares refinement of cell dimensions for condelphine hydroiodide* ($\lambda = 1\cdot5418$ Å)

| Observed | | Cell (I) | | Cell (II) |
2θ	hkl	Calculated 2θ	hkl	Calculated 2θ
21·0904	1$\bar2$1	21·0767	22$\bar1$	21·0757
24·7420	2$\bar3$1	24·7587	131	24·7593
32·7218	$\bar2\bar3$3	32·7311	13$\bar3$	32·7310
37·9826	1$\bar5$2	37·9869	35$\bar2$	37·9860
40·9416	$\bar1\bar5$2	40·9447	$\bar3$52	40·9399
41·6332	2$\bar7$2	41·6364	07$\bar2$	41·6355
41·8312	2$\bar6$1	41·8287	36$\bar1$	41·8285
42·8022	43$\bar3$	42·8121	1$\bar3$3	42·8119
43·0840	$\bar3\bar3$4	43·0811	13$\bar4$	43·0812
43·9998	$\bar2$6$\bar1$	44·0164	$\bar3$61	44·0114
44·1622	08$\bar1$	44·1705	$\bar1$81	44·1683
44·2648	2$\bar7$2	44·2601	072	44·2598
45·4536	$\bar3\bar3$4	45·4491	$\bar1$34	45·4489
46·5520	2$\bar8$1	46·5511	181	46·5513
47·3880	$\bar3\bar5$4	47·3900	15$\bar4$	47·3899
49·1210	3$\bar7$0	49·1271	370	49·1275
50·2318	370	50·2344	3$\bar7$0	50·2295
51·0246	3$\bar5$4	51·0288	$\bar1$54	51·0283
51·1774	46$\bar1$	51·1719	3$\bar6$1	51·1679
51·2304	4$\bar6$1	51·2263	361	51·2270
51·3490	401	51·3528	50$\bar1$	51·3481
54·0182	53$\bar4$	54·0144	1$\bar3$4	54·0146
55·4982	55$\bar2$	55·4883	35$\bar2$	55·4854
55·7484	5$\bar3\bar4$	55·7402	134	55·7416
56·3120	5$\bar5\bar2$	56·3020	352	56·3029
57·7966	55$\bar4$	57·8055	1$\bar5$4	57·8051
60·5330	55$\bar4$	60·5452	154	60·5467
63·3186	60$\bar1$	63·3431	501	63·3399
71·6130	006	71·6199	60$\bar6$	71·6129

Refinement results

	Cell (I)	Cell (II)
a	9·3743 (7) Å	9·3404 (9) Å
b	17·442 (2)	17·442 (2)
c	9·0953 (9)	9·0953 (8)
α	94·891 (9)°	94·891 (9)°
β	119·264 (7)	118·891 (7)
γ	88·652 (7)	86·594 (9)
Goodness of fit	1·017	0·964
Maximum (shift/e.s.d.)	0·684	0·268
Maximum $|2\theta_{\text{obs}}-2\theta_{\text{calc}}|$	0·0245	0·0213
a/b	0·5374	0·5355
c/b	0·5215	0·5215

established, agreeing with cell (II) both in dimensions and relative intensities. The *DETCELL* program from the CAD-4 software was used to make precise measurements of 2θ for 29 reflections. The values reported for cell (II) in Table 2 result from a least-squares refinement of these measurements, using the *PARAM* link of the X-RAY System (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

In order to establish whether cells (I) and (II) referred to the same lattice, the two sets of cell dimensions were sent to Dr Alan D. Mighell for comparison, based on the criteria established by Donnay & Ondik (1972). Only cell (II) was a reduced cell, with cell (I) being transformed to it by the matrix $(101/0\bar{1}0/00\bar{1})$, giving cell (I′) of Table 1.

The precision with which both cells can be determined is shown in Table 2. The Miller indices of the reflections used in the refinement of cell (II) were transformed to cell (I) using the above matrix, and refined to convergence with *PARAM*. Cells (I) and (II) are clearly different on the basis of the e.s.d.'s, although the magnitude of the differences is sufficiently small that, except for γ, they might reasonably be attributed to experimental error. Furthermore, on the basis of the determinative numbers a/b and c/b (Donnay & Ondik, 1972) the cells could be considered identical.

It is well established that the Delaunay algorithm does not necessarily lead to the reduced cell as defined by Niggli (Santoro & Mighell, 1970). The present case represents satisfaction *within experimental error* of their special condition 3(e), i.e. $a \cdot b = \frac{1}{2}a \cdot a$. If the cell dimensions are considered (incorrectly) to be geometrically exact values, then the small difference in γ between cells (I) and (II) alters the sense of the inequality between $a \cdot b$ and $\frac{1}{2}a \cdot a$, and leads to the ambiguous cell reduction. Metrically pseudo-hexagonal cells, for which there must exist three, nearly equal, coplanar lattice translations, are particularly subject to this problem. Such an ambiguity was discussed by Patterson & Love (1957), with respect to the Delaunay reduction, but not generalized to cells with metric pseudosymmetry.

The hazards of uncritical acceptance of cell dimensions produced by diffractometer software should be evident. In particular, it is essential to consider the experimental error of the matrix scalars when testing the Niggli matrix for the special conditions listed by Santoro & Mighell (1970).

References

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International Union of Crystallography

Report of the Tenth General Assembly and International Congress of Crystallography

The Report of the Tenth General Assembly and Congress has been published in *Acta Crystallographica*, Section A [*Acta Cryst.* (1976), A**32**, 691–745]. It includes the minutes of the General Assembly; the triennial reports of the Executive Committee, the Commissions and the Union representatives on bodies not belonging to the Union; the Statutes and By-Laws as amended by the Tenth Assembly; membership of the Executive Committee and the Commissions; names and addresses of Union representatives on other bodies; a list of Adhering Bodies and the membership of National Committees for Crystallography, with names and addresses of the Secretaries. Reprints of the report have been sent to Secretaries of National Committees.

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