

example, a systematically extinct reflexion, then it may be possible to observe a net intensity at $h_2k_2l_2$. From this criterion, the above rule may be written as $2(h_1 + h_3) + (k_1 + k_3) = 4n + 2$, on noting that $k_1 + k_3$ must always be even. With a list of the ten strongest reflexions (some of which were not recorded by Trojer) and their symmetry equivalents to define \mathbf{d}_1^* and \mathbf{d}_3^* , computation has shown that no combinations of $\mathbf{d}_1^* + \mathbf{d}_3^* = \mathbf{d}_2^*$ satisfy the above rule for any orientation of the crystal or any number of reciprocal lattice points on the sphere at the same time. Therefore multiple reflexion effects can be eliminated as a cause of the observable intensities and hence the true space group may be taken as $P2_1/a$.

As a corollary to the above analysis, it was of interest to see whether or not Tolliday's data could equally well have been described by $P2_1/a$ symmetry, a possibility which she did not apparently consider. In order to verify this, a Patterson map was computed in sections of interval $\frac{1}{30}$ with her data (Tolliday, 1959). The peaks that she found equivalent in height to the origin peak at $\pm(\frac{1}{2}, \frac{1}{4}, 0)^*$ were in fact just over half the height in this computation. Moreover, if the interatomic vectors between symmetry-related atoms due to a -glide symmetry, which take the form $\pm(\frac{1}{2}, \frac{1}{2} + 2y, 0)$, are considered, then the y coordinates would have values $\frac{1}{8}, \frac{3}{8}, \frac{5}{8}$ and $\frac{7}{8}$ if the vector peaks are exactly located at $\pm(\frac{1}{2}, \frac{1}{4}, 0)$. Since the densest part of the peaks on the Patterson map at these positions were spread over four intervals from $\pm\frac{6}{30}$ to $\pm\frac{9}{30}$ in the V direction, the expected deviations of the y coordinates from the ideal values of $\frac{1}{8}, \frac{3}{8}, \frac{5}{8}$ and $\frac{7}{8}$ would be within ± 0.07 which, from an analysis of Table 1 for both Tolliday's and Trojer's fractional coordinates, is the case for all atoms. A structure factor calculation based on Trojer's atomic

* The additional peak at $(\frac{1}{2}, \frac{3}{4}, 0)$ is invoked by the symmetry of the Patterson synthesis.

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Phase Extension and Refinement

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Abstract

Two procedures are described to extend and refine phases starting from a medium-sized set of known phases. From tests with 376 and 400 atom structures it was found that for extension purposes the tangent formula is suitable; for refinement purposes the tangent

positions and isotropic thermal parameters was made with Tolliday's data. R was 30% after adjustments were made to the scale factor. Although at the time it was not feasible to pursue this analysis to least-squares refinement, the above results do clearly indicate a fit between Tolliday's data and space group $P2_1/a$.

Conclusions

The striking resemblance between Trojer's and Tolliday's structures of parawollastonite lies in their ability to satisfy the conditions for which the two space groups $C'2_1$ and $P2_1/a$ are equivalent. It is therefore not surprising that Tolliday found the structure refined successfully in $C'2_1$ and not in $C'2_1/a$, its centrosymmetric counterpart. Furthermore, in view of the conclusions of the previous section, structural descriptions based on Tolliday's space group, such as the one given by Bragg & Claringbull (1965), would seemingly have to be revised.

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formula was adapted in order to maintain the enantiomorph.

Introduction

In protein crystallography, direct methods have been used to extend and refine phases by employing: (1) the

Sayre equation as such, and (2) the angular part of the Sayre equation. The first method (Sayre, 1972) refines phases by minimizing

$$R_1(\varphi_H, \varphi_H', \dots) = \sum_H W_H \left| \theta(|H|) F_H - \sum_K F_K F_{H-K} \right|^2. \quad (1)$$

Here $\theta(|H|)$ is a known function of $|H|$. As in each cycle of this iterative method the equation system

$$\theta(|H|) F_H = \sum_K F_K F_{H-K} \quad (2)$$

is processed, the computations are lengthy, even by today's standards. Further research along this line concentrates therefore on convolutional expressions other than (2), requiring fewer computational operations (Sayre, 1978).

In the second method, tangent refinement (Coulter & Dewar, 1971) or large determinants (de Rango, Mauguen & Tsoucaris, 1975) are employed. In polar space groups such as $P2_1$ and $R3$, tangent-refinement techniques have been found to lead to centrosymmetric phases in a few cycles. On the other hand, in space groups such as $P2_12_12_1$ successful phase refinements and extensions have been reported (Coulter & Dewar, 1971). The determinant technique starts with an $N \times N$ determinant consisting of approximate phases for most structure factors. It determines and refines phases for the last row of an $(N+1) \times (N+1)$ determinant. Because there is a vast amount of *a priori* phase information not subject to change, the new phases are strongly related to those of the starting set. Hence, even in polar space groups, results are satisfactory, but the computations are evidently very time consuming.

In the present investigation our aim was to adapt the tangent formula such that the phase calculation would be both fast and enantiomorph conserving. From the triplet phase sums $(-\varphi_H + \varphi_K + \varphi_{H-K})$, for a medium-sized set of known phases, the average triplet phase sums Δ_3 are calculated as a function of E_3 . As a next step, new phases are calculated either using a procedure similar to that of Hauptman, Fischer, Hancock & Norton (1969) or by means of the normal tangent formula. The third and last step of the method is an enantiomorph-specific phase refinement based on an adapted tangent formula, in which the average phase sums Δ_3 provide the necessary stability for maintaining the enantiomorph.

Preliminary results were reported at the Tenth International Congress of Crystallography (Sint & Schenk, 1975). The examples given in this paper show that the method is enantiomorph conserving and fast. Our next efforts will include its application to small proteins.

Calculation of the average triplet phase sums

The starting point is a set of known phases, such as, in the case of proteins, those corresponding to a resolution of 2.5 Å. Within this group all triplets for which E_3 exceeds a limit value are generated and their phase sums are calculated:

$$\delta_{HK} = -\varphi_H + \varphi_K + \varphi_{H-K}.$$

The E_3 range is divided into a number of intervals over which δ_{hk} is averaged:

$$\delta(E_3) = \langle |\delta_{HK}| \rangle_{E_3}. \quad (3)$$

From a plot of $\delta(E_3)$ as a function of E_3 , the Δ_3 function is obtained as the curve representing the best fit. Due to the small range of E_3 values in our tests, Δ_3 is a straight line. The $\Delta_3(E_3)$ curve provides an estimate of the absolute value of a triplet phase sum. An example of a Δ_3 curve is given in Fig. 1.

Phase extension

As mentioned in the introduction two methods were developed to extend a set of phases. In the first procedure (to be referred to as graphical procedure) values of

$$\Phi(\varphi_H) = \sum_K E_3 | -\varphi_H + \varphi_K + \varphi_{H-K} - s \Delta_3 | \quad (4)$$

are calculated for φ_H in the range from 0–1 cycle in steps of 0.01 cycle from the phases φ_K and φ_{H-K} for a large number of reflections K . In (4), for each term the sign $s = \pm 1$ is chosen such that the smallest contribution of the term to the summation is obtained. Expression (4) is similar to the expression used by

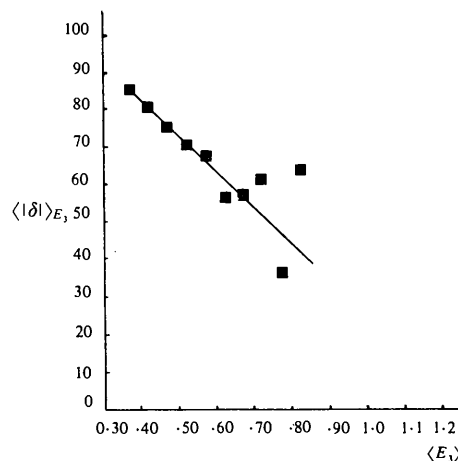


Fig. 1. A plot of $\langle |\delta| \rangle_{E_3}$ (given in mcycles) vs $\langle E_3 \rangle$ for ten groups of triplets. From left to right the points in the graph represent 13 014, 9207, 3828, 2010, 918, 504, 240, 117, 72 and 39 triplets respectively.

Hauptman, Fisher, Hancock & Norton (1969) except for the relative weights of the individual terms. The lowest value of $\Phi(\varphi_H)$ gives the most probable value for φ_H . In the case where two pronounced minima are found with a small difference between them the phase is not accepted.

The second procedure is based on the normal tangent formula

$$\tan \varphi_H = \frac{\sum_K E_3 \sin(\varphi_K + \varphi_{H-K})}{\sum_K E_3 \cos(\varphi_K + \varphi_{H-K})}. \quad (5)$$

New phases are accepted only if the number of terms in the summation over K is sufficiently large. It is expected that errors, introduced by taking all $\delta_{HK} = 0$ will cancel out provided that the phases φ_K and φ_{H-K} have random errors only.

From a theoretical point of view the first procedure is to be preferred, because it employs all information about the enantiomorph both in the phases (φ_K, φ_{H-K}) and in the triplet relationships (Δ_3). The tangent formula utilizes only the enantiomorph information contained in the phases but, since it takes only about 2% of the computing time required for the other method, it is to be preferred as long as the new phases are enantiomorph specific.

Phase refinement

Once a set of newly determined phases has been obtained, the new phases together with those of the starting reflections are refined in an enantiomorph specific way by means of the adapted tangent formula

$$\tan \varphi_H = \frac{\sum_K E_3 \sin(\varphi_K + \varphi_{H-K} - s\Delta_3)}{\sum_K E_3 \cos(\varphi_K + \varphi_{H-K} - s\Delta_3)}, \quad (6)$$

in which $E_3 = N^{-1}|E_H E_K E_{H-K}|$ and Δ_3 is the estimated value of $|- \varphi_H + \varphi_K + \varphi_{H-K}|$ mentioned before. Again the sign s is determined such that $- \varphi_H + \varphi_K + \varphi_{H-K} - s\Delta_3$ is closest to zero.

The fact that this procedure is capable of maintaining the enantiomorph can be seen by the following. If Δ_3 in (6) is replaced by $|\delta_{HK}|$ all signs s will be determined such that $s|\delta_{HK}| = -\varphi_H + \varphi_K + \varphi_{H-K}$. As a consequence φ_H will be unchanged under refinement. Use of Δ_3 instead of $|\delta_{HK}|$ introduces random differences $\Delta_3 - |\delta_{HK}|$ and hence refinement based on (6) will be enantiomorph specific.

The refinement criterion of (6)

Schenk (1972) pointed out that instead of the tangent formula (5) the exponential expression

$$\exp(i\varphi_H) = \sum_K E_3 \exp[i(\varphi_K + \varphi_{H-K})] / \sum_K E_3 \quad (7)$$

is commonly employed. He showed that the quantity which is actually minimized is

$$R_T = \sum_H \sum_K E_3 \sin^2 \frac{1}{2}(-\varphi_H + \varphi_K + \varphi_{H-K}). \quad (8)$$

Similarly, instead of (6) the exponential equation

$$\exp(i\varphi_H) = \sum_K E_3 \exp[i(\varphi_K + \varphi_{H-K} - s\Delta_3)] / \sum_K E_3 \quad (9)$$

is used leading to the refinement criterion

$$R_{AT} = \sum_H \sum_K E_3 \sin^2 \frac{1}{2}(-\varphi_H + \varphi_K + \varphi_{H-K} - s\Delta_3). \quad (10)$$

Test of the adapted tangent formula (6)

The adapted tangent refinement procedure has been applied to two large artificial structures.

The first structure was generated in space group $P2_1$ with one heavy atom (relative weight 13) and 187 light atoms (relative weight 1) in the asymmetric unit. The heavy atom was situated at $y = 0.250$ with randomly chosen x and z coordinates, in order to have an easy check on the centrosymmetry of the phases. The 187 light atoms were situated at randomly chosen x, y and z coordinates. For this 376 atom structure 5000 E values were calculated, the 1000 strongest of which show a phase distribution given in Table 1, column 1. The presence of the heavy atom causes the phases to be pseudo-centrosymmetric. 10000 unique triplets with $E_e \geq 0.35$ among the 1000 strongest reflections were used. The first experiment was a normal tangent refinement. At the start of the refinement all reflections were given their correct phases. After only one iteration it was obvious that the phases tended to become centrosymmetric (see Table 1). After three iterations this process was nearly completed and most of the phases were within 5 mcycles ($\approx 2^\circ$) of 0 or π . The average phase error was 62 mcycles. Next the adapted tangent procedure was applied and as can be seen from Table 1, columns 4 and 5, the distribution of the phases after 4 and 20 cycles was far from centrosymmetric, although there are slight differences with respect to the distribution of the true phases. The average phase error after four iterations was 33 mcycles and after 20 iterations 38 mcycles. In the set of 1000 reflections, six phases were in error (phase difference larger than 250

mcycles) and for eight reflections the phase of the enantiomorph was found rather than the correct phase.

To provide another test a structure was generated in space group $P2$ with one moderately heavy atom (relative weight five) and 199 light atoms (relative weight one) in the asymmetric unit. In this case the heavy atom was situated at $x = 0.2150$, $y = 0$ and $z = 0.2675$, and the light atoms were randomly positioned. A total of 8921 E values was calculated, of which only the 990 strongest were used for the test. The distribution of the true phases is given in Table 1, column 6. 9146 unique triplets were generated with $E_3 \geq 0.29$. Again the first test was a normal tangent refinement. In this case only the phase distribution after 11 cycles of tangent refinement is given in Table 1, column 7. As can be seen there is again a tendency for the phase to be 0 or 500 mcycles, although, due to the fact that the heavy atom now has a smaller relative weight, this

effect is not nearly as prominent as for the first structure. The average phase error is 134 mcycles. The second experiment was again the application of (6). The resulting phase distribution given in Table 1, column 8 is in good agreement with that determined from the true phases. Now the average phase error is 51 mcycles, only two phases are in error and for 18 reflections the phases corresponding to the enantiomorph have been found instead of the correct phases. From an E map calculated with this set of phases, 191 atoms could be identified among the 227 strongest unique peaks. An E map calculated with the 990 correct phases revealed 194 atoms among the 226 strongest unique peaks. From this it is clear that hardly any information about the structure is lost during the refinement procedure based on (6).

From the above two structures we conclude that the adapted tangent refinement maintains the enantiomorph when refining a set of phases related to one single enantiomorph, whereas the normal tangent refinement leads to centrosymmetric sets of phases.

Table 1. Phase distributions and average phase errors $\langle |\Delta\phi| \rangle$ for both test structures

Columns 1 and 6 give the true phase set distributions for the 376 atom and 400 atom structures, respectively. The figures represent the number of phases $|\phi|$ in the respective intervals. Note that the intervals are not equal. Results of the normal tangent refinement (5) are presented in columns 2 and 3 for 376 atom structure and in column 7 for the 400 atom structure. Columns 4 and 5 and column 8 give the results obtained with the adapted tangent procedure (6) for the two structures, respectively.

Column number	1	2	3	4	5	6	7	8
Number of iterations	0	1	3	4	20	0	11	10
$ \phi $ in mcycles								
0-10	86	342	488	59	48	66	240	57
10-20	43	112	4	51	52	25	89	26
20-30	39	25	3	50	50	25	39	20
30-40	45	9	0	50	44	31	30	28
40-50	33	3	0	44	42	26	16	19
50-60	39	1	0	56	64	21	16	20
60-70	29	2	0	38	48	16	12	19
70-80	31	0	0	40	37	16	4	17
80-90	13	0	0	39	37	29	4	13
90-100	21	0	0	26	25	14	7	29
100-150	83	2	2	38	42	94	26	99
150-200	19	1	1	2	4	66	10	91
200-300	25	5	1	12	11	149	17	136
300-350	22	0	1	7	8	61	18	87
350-400	60	1	0	25	28	74	20	106
400-410	16	1	0	17	14	31	6	15
410-420	22	1	1	26	32	18	7	23
420-430	28	0	0	33	38	24	9	16
430-440	32	0	0	42	43	29	7	14
440-450	30	1	1	53	54	30	18	13
450-460	49	0	0	49	49	27	27	22
460-470	45	11	3	61	57	19	37	22
470-480	35	37	1	53	39	23	61	26
480-490	49	111	3	63	59	24	96	24
490-500	106	335	491	67	76	52	179	52
$\langle \Delta\phi \rangle$ in mcycles	0	—	62	33	38	0	134	51

Test of the complete procedure

The complete extension and refinement procedure was first applied to the 376 atom structure described in the previous section. The starting point was the inner sphere of 1500 reflections and the purpose was to phase as many reflections as possible within the sphere of 3500 reflections. Again, the strongest reflections only (20%) were used in the process and the area to be phased was gradually enlarged from 322 reflections to 701 reflections in four steps. In each cycle first the Δ_3 curve was calculated, next new phases were determined using (4) and finally all old and new phases were refined with (6) in three or four iterations.

The results are summarized in Table 2. The final set consisted of 501 phased reflections out of 701 with an average phase error of 38 mcycles; four phases are in error, 16 correspond to the other enantiomorph and 481 are correct. Although the quality of the phases is good, the number of new phases is not completely satisfactory. Another feature of Table 2 is the fact that $\langle |\delta| \rangle_{0.37}$ gradually drops from 84 mcycles before the first step (which equals $\langle |\delta| \rangle_{0.37}$ calculated with all phases correct) to 71 mcycles after the third step. This drift suggests that in spite of the use of the adapted tangent refinement the phases still have a tendency to become centrosymmetric. Since the recalculation of the Δ_3 curve strengthens this effect, a remedy might be to omit it.

Next, the procedure was tested for the 400 atom $P2$ structure. The starting point was a set of 214 reflections belonging to the 990 strongest ones. The area to be phased was again gradually enlarged in four steps, but

one extra extension/refinement stage was added. The final set of phases consisted of 450 out of 990 phases (see Table 2) with an average phase error of 80 mcycles; 45 phases were clearly in error and 18 phases were found to correspond to the other enantiomorph. An E map calculated with these 450 phases showed 120 atoms among the 241 strongest unique peaks, to be compared with 90 atoms which could be identified among the 227 highest unique peaks in an E map, calculated with the 214 correct starting phases. The number of newly determined phases is small again (236), owing to the fact that strict acceptance criteria have been used in the extension stage in order to avoid phasing errors. These results have led us to try the following alternative procedure; phase extension by the normal tangent formula (5) followed by phase refinement based on the adapted formula (6), until all phases in a phasing area have been determined. A test was carried out for the 400 atom structure and, as can be

seen from the upper half of Table 3, after the fourth stage all 990 phases were determined, and therefore stage five consisted merely of three refinement cycles applying (6) to all 990 reflections and 27 438 redundant triplets. The average phase error was 105 mcycles compared with the correct structure. With respect to the enantiomorph, there is a phase difference of 189 mcycles. A total of 114 phases had an error of more than 250 mcycles, while 67 phases were found to correspond to the enantiomorph. An E map calculated with the above set of phases revealed 157 atoms among the 245 strongest unique peaks.

Critical examination of the phases determined in each cycle showed that phases calculated with only a few contributing triplets had higher average error than those determined by many contributing triplets. Therefore the test was repeated but now a phase was accepted only if there were at least NMIN contributing triplets. In Table 3 results are given for NMIN equal to

Table 2. Results of the procedure using the graphical determination of new phases for the 376 atom structure (columns 1–5) and the 400 atom structure (columns 6–10)

Cycle number	0	1	2	3	4	0	1	2	3	4	5
Total number of reflections in sphere	322	390	490	600	701	214	346	481	804	990	990
Number of newly determined phases		33	46	56	44		28	36	71	70	31
Total number of known phases	322	355	401	457	501	214	242	278	349	419	450
Total number of unique triplets among the known reflections	997	1254	1602	2140	2510	397	551	627	929	1154	1325
$\langle \delta \rangle_{0-37}$ in mcycles	84	80	77	71	—	—	—	—	—	—	—
$\langle \delta \rangle_{0-29}$ in mcycles	—	—	—	—	—	166	165	166	162	160	—

Table 3. Data on the phase extension and refinement procedure using expressions (5) and (6), respectively, for the 400 atom structure

In rows 3, 4, 5, 6, 7 and 12, data are given for $A_3 = \langle |\delta_{hk}| \rangle$; in rows 3, 8, 9, 10, 11 and 12 results are given for the case $A_3 = 0$.

Stage	0	1	1	1	2	2	2	3	3	3	4	4	4	5	5	5
Minimum number of terms in expressions (5) and (6)	—	1	5	7	1	5	7	1	5	7	1	5	7	1	5	7
Number of reflections phased after this stage	214	346	291	255	481	408	341	804	745	621	990	959	888	990	960	900
Average phase error	0	61	39	21	83	62	43	101	87	73	105	94	86	105	94	87
Average phase difference with respect to incorrect enantiomorph	190	190	190	189	195	191	194	195	188	189	189	184	187	189	185	185
Number of phases in error	0	21	7	3	48	18	9	95	53	37	114	78	74	114	81	76
Number of phases in error with respect to incorrect enantiomorph	72	121	101	85	161	137	120	282	247	187	315	301	275	314	303	279
Average phase error	0	86	55	39	112	94	78	132	119	118	140	133	134	142	137	138
Average phase difference with respect to incorrect enantiomorph	190	178	178	182	167	172	171	156	156	156	154	156	153	154	155	152
Number of phases in error	0	22	8	3	60	32	27	139	104	84	184	159	151	188	170	165
Number of phases in error with respect to incorrect enantiomorph	72	102	88	78	122	113	108	178	174	145	227	225	194	229	222	195
Number of unique triplets known after this stage	397	1121	1054	817	2306	2086	1624	6026	5821	4998	9146	9036	8622	9146	9041	8792

1, 5 and 7. It is clear that the use of a higher NMIN gives better results for the average phase deviation as well as a lower number of phases with an error of over 250 mcycles. An E map calculated from the phases obtained with NMIN equal to 7 revealed 162 atoms among the 240 strongest unique peaks; this number is not much larger than in the case of NMIN = 1, but the peaks were much higher, although the number of contributing reflections was smaller.

The alternative procedure was also tested using $\Delta_3 = 0$ in (6), which is equivalent to the use of the usual formula (5). From the results given in the lower half of Table 3 it can be concluded that: (a) the final phase sets are almost centrosymmetric and (b) the use of a higher number for NMIN only slightly slows down the appearance of the other enantiomorph. For the sake of comparison an E map was calculated from the phases obtained with $\Delta_3 = 0$ and NMIN = 7. Only 93 out of the 243 strongest unique peaks could be identified as atoms.

The total computing time for the tangent extension procedure mentioned above ranged from 78 to 105 c.p.u.s (on a Cyber 73 computer). The procedure based on the graphical determination of new phases took 147 s c.p.u. time for the 400 atom structure despite the fact that only ± 1300 unique triplets were used in the final refinement cycles. Our conclusion is that the procedure based on graphical phase determination is rather expensive and difficult to optimize for accepting new phases. Since the procedure using (5) leads to a maximum number of more or less correct phases in a minimum of time, it is to be preferred. If this procedure

is combined with a refinement procedure based on (6) the results are sufficiently enantiomorph specific to lead to a correct solution, starting from a medium-sized phase set.

The main conclusion of this paper is that with a relatively simple improvement in the estimates used, the centering tendency of the tangent formula is efficiently blocked. However, since it is expected that better estimates will improve the quality of the final map, this will be an important part of our future efforts.

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The Archimedean Truncated Octahedron, and Packing of Geometric Units in Cubic Crystal Structures

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Abstract

Any cubic crystal structure can be divided into small units in the form of congruent semi-regular (Archimedean) truncated octahedra. The centers of these polyhedra can be chosen at invariant equivalent positions for most cubic space groups. The part of a

crystal structure enclosed by an Archimedean polyhedron is called a geometric unit (or unit for short); however, the boundary of the unit may be relaxed to include a whole molecule or ion in case the geometric division is not convenient. Based on the properties and arrangements of such geometric units, there is an interesting relationship among the 36 cubic space