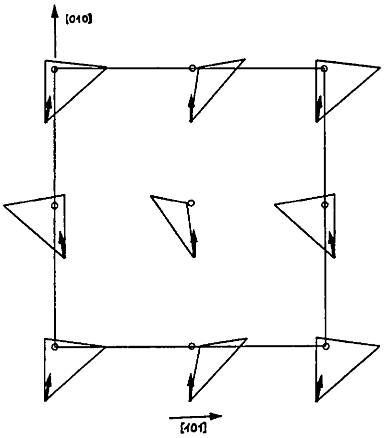

Fig. 5. A graphical illustration of the transformation mechanism. The arrows represent the transformation vectors. Only one layer of the IO_3 pyramids is shown.

the proposed structure for the high temperature form will be about 1·78 Å for the shorter and 2·83 Å for the longer bond. The important transformation vectors can be seen in Fig. 5. This mechanism will lead to smaller and more regular IO_3 pyramids compared to the room-temperature form. As the contracting I–O bond is nearly parallel to **b**, it must cause a small shrinkage parallel to **b**.

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The Application of Direct Methods to Centrosymmetric Structures containing Heavy Atoms. II

BY R. O. GOULD

Edinburgh University, Department of Chemistry, West Mains Road, Edinburgh, Scotland

AND TH. E. M. VAN DEN HARK AND PAUL T. BEURSKENS*

Crystallography Laboratory, Toernooiveld, Nijmegen, The Netherlands

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Direct methods are applied to the difference structure factors for a structure containing one or more heavy atoms in known positions. For those reflexions whose sign is determined by the heavy atoms, the known heavy-atom contribution is subtracted from the observed structure factor to obtain the magnitude and the sign of the light-atom contribution. The signs of the reflexions that do not have an appreciable heavy-atom contribution are found using the $\sum_2$ sign relationship. For reflexions with an intermediate heavy-atom contribution the sign and magnitude ambiguity is also solved by the $\sum_2$ formula. Thus it is possible to maximize the number of correct signs, and correct some of the amplitudes, before a difference Fourier map is calculated.

Introduction

In paper I (Beurskens & Noodik, 1971) direct methods were used to solve the phase problem for those special cases where the heavy atoms are on special positions, such that the heavy atoms do not contribute to several reflexion parity groups. In those cases the crystal structure is not determined by the positions of the heavy atoms only: one or two phases have to be chosen in order to specify the origin fully.†

The present paper deals with the general case: the positions of the heavy atoms completely determine the structure; the phase problem is solved in principle: the positions of the remaining 'light' atoms can be found by standard Patterson and Fourier techniques. The time and effort necessary for finding the light-atom structure is reduced by the present procedure, especially when the heavy atoms are marginally sufficient to solve the phase problem.

Define:

$\lvert F_{\rm obs}\rvert$	is the observed structure factor.
F_H	is the calculated contribution of the known part of the structure ('heavy' atoms).
F_L	is the contribution of the remaining part of the structure ('light' atoms).
$S(F)$	is the sign of F.

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* Author for correspondence.

† A Fortran program for the execution of this procedure, *DIRDIF.A*, is now available on request.

In the normal procedure, difference coefficients,

$$\Delta F = S(F_H) \cdot (|F_{\rm obs}| - |F_H|)$$

are calculated and accepted as an estimate for F_L. Only in very favorable circumstances can the complete light-atom structure be unambiguously deduced from the Fourier synthesis based on these coefficients. In the present procedure, a $\sum_2$ refinement procedure is used to convert input ΔF values to correct F_L values.

Two categories of reflexions may be distinguished (see Fig. 1).

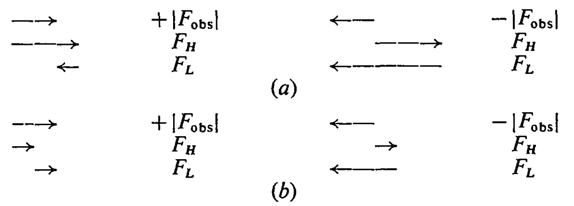

Fig. 1. Two categories of reflexions. Drawings for a given $|F_{\rm obs}|$ value, and $S(F_H) = +$. See text.

(*a*). $|F_H| > |F_{\rm obs}|$. In this case [Fig. 1(*a*)] the sign of F_L is known to be opposite to the sign of F_H. Nevertheless, the absolute value of F_L is not known for certain as the sign of $F_{\rm obs}$ is not known. The more probable value of $|F_L|$ is the smaller of the two possibilities. If this is correct, then $F_L = \Delta F$. There will in general be very few reflexions in this category for which the larger magnitude is correct *and* significantly different from the smaller; no attempt is made to find these reflexions.

(*b*). $|F_{\rm obs}| > |F_H|$. In this case [Fig. 1(*b*)] neither sign nor absolute value of F_L is known, but sign and absolute value are correlated. Again the smaller of the two possible $|F_L|$ values is the more probable one, corresponding to $F_L = \Delta F$. If ΔF is sufficiently large (see below) then this reflexion will take part in our $\sum_2$ refinement procedure, and when it is found that the sign of F_L is wrong, we not only change the sign but also the absolute value of F_L. In the special case where the heavy-atom contribution is very weak, if not zero, ΔF cannot be calculated; nevertheless the absolute value of F_L is known, and the sign of F_L may be calculated by the $\sum_2$ formula.

Description of the procedure

The procedure consists of the following steps: calculation of a 'two-dimensional' Wilson plot, the calculation of 'dual' E values for the light-atom structure, the determination and refinement of signs by the $\sum_2$ formula, and the selection of Fourier coefficients.

Symbols used:

$I = K \cdot |F_{\rm obs}|^2$, observed intensity on a relative scale K.
F_H^0 calculated structure factor on absolute scale for the known heavy atoms with a temperature factor of $B_H = 0{\cdot}0$.
f_H, f_L, f: scattering factors for a heavy, light, or any atom.
$B_H, B_L, B_{\rm ov}$: overall temperature factors for the heavy atoms, the unknown part of the structure, or the whole structure.
E_H, E_L: normalized structure factors for a structure consisting of heavy or light atoms only.
$\sum^H, \sum^L, \sum$: summation over the heavy, light, or all atoms of a unit cell.
$S = \sin\theta/\lambda$.

Wilson plot

We have stressed (Paper I) the importance of a well designed Wilson-plot routine to obtain the best possible estimates for the $|F_L|$ values and to avoid disasters during the automatic execution of the procedure. The 'local' intensity average for a partially known structure is [equation (2) in Paper I; Parthasarathy (1966)]:

$$\langle I \rangle_{\mathbf h} = K[\langle \textstyle\sum^L f_L^2 \exp(-2B_L S^2)\rangle_{\mathbf h} + \langle |F_H^0|^2 \exp(-2B_H S^2)\rangle_h] . \quad (1)$$

The average is taken over reflections $\mathbf{h}$ within a given interval.

Initially, setting $B_L = B_H = B_{\rm ov}$, and applying (1) in ranges of S, a one-dimensional Wilson plot is calculated. In Paper I the value of B_L was found by a Wilson plot on those reflexions that had no heavy-atom contributions; thereafter B_H was found by a 'difference' Wilson plot.

It is often possible to refine simultaneously the temperature factors of the heavy and light atoms separately. The main restriction is that the heavy atom must not lie in a position (such as a centre of symmetry in space group $P\overline{1}$), where it contributes uniformly to all reflexions. Less accurate results may be expected if there are a number of moderately heavy atoms (*e.g.* S or P) grouped around the known atoms, but this should normally give little trouble. The procedure, adopted in our *DIRDIF.B* program, is a non-linear iterative one, and depends on the reflexions being distributed over a two-dimensional array in ranges of S and of $|E_H|$, where

$$|E_H|^2 = |F_H^0|^2 / \textstyle\sum^H f_H^2 .$$

Define:

$$G_{\rm obs} = \langle I / \textstyle\sum f^2 \rangle_{\mathbf h}$$
$$G_H = K\langle |F_H^0|^2 \exp(-2B_H S^2) / \textstyle\sum f^2 \rangle_{\mathbf h}$$
$$G_L = K\langle \textstyle\sum^L f_L^2 \cdot \exp(-2B_L S^2) / \sum f^2 \rangle_{\mathbf h} ,$$

where the averaging is done over the appropriate range of S and $|E_H|$. The quantity to be minimized is then

$$\sum_{\rm ranges} (G_{\rm obs} - G_H - G_L)^2$$

and the parameters to be refined are K, B_H and B_L. Refinement normally takes three cycles.

Normalized structure factors

For every reflexion two possible values for E_L, the normalized structure factor of the light-atom structure are calculated:

$$E_1=[S(F_H)\,.\,|F_{\rm obs}|-F_H]/(\varepsilon \textstyle\sum {}^Lf_L^2)^{1/2}\exp(-B_LS^2)$$
$$E_2=[-S(F_H)\,.\,|F_{\rm obs}|-F_H]/(\varepsilon \textstyle\sum {}^Lf_L^2)^{1/2}\exp(-B_LS^2) \qquad (2)$$

where $|F_{\rm obs}|=(I/K)^{1/2}$, $F_H=F_H^0\exp(-B_HS^2)$, and ε is the well-known correction for symmetry enhancement (Hauptman & Karle, 1953). Always $|E_1|\le|E_2|$; for most reflexions $|E_1|<|E_2|$, and E_1 is the most probable value. It is the aim of our procedure to find reflexions where the less probable value, E_2, is the correct value.

Let us define P_1 as the probability that the *sign* of E_1 is correct. For reflexions in category *b*, the probability that the *magnitude* of E_1 is correct is also given by P_1. This probability can be calculated from the magnitudes of E_1 and E_2, using the distribution of E values in centrosymmetric space groups: $P(E)=(2\pi)^{-1/2}\exp[-\frac{1}{2}E^2]$ (Hauptman & Karle, 1953). This gives (Woolfson, 1956):

$$P_1=\exp(-\tfrac{1}{2}E_1^2)/[\exp(-\tfrac{1}{2}E_1^2)+\exp(-\tfrac{1}{2}E_2^2)] \qquad (3)$$

(see Table 1) and the weight to be assigned to the value E_1 is taken as:

$$W_1=(2P_1-1)^2 \qquad (4)$$

(Note: $|E_1|\le|E_2|\,.\,0{\cdot}5\le P_1\le 1$, thus: $0\le W_1\le 1$). The special case $F_H\simeq 0$ leads to $E_1\simeq -E_2$ with $P_1\simeq P_2\simeq 0{\cdot}5$, which does not need special treatment.

For reflexions in category *a*, E_1 and E_2 have the same sign, and this sign is known and will be used to determine other signs. As explained above, we do not try to find the correct absolute value, and for safety reasons we have to accept the lower $|E|$ values. Therefore E_1 is accepted, with P_1 equal to 1·0:

$$\text{for}\quad |F_H|>|F_{\rm obs}|:\quad P_1=1{\cdot}0\,,\quad W_1=1{\cdot}0\,. \qquad (5)$$

The special case $F_{\rm obs}\simeq 0$, however, may easily lead to severe practical problems, especially for high-order reflexions. These are the so-called 'less-than' or 'unobserved' reflexions. The $|F_{\rm obs}|$ value is very badly determined and, to avoid overestimating the E_L values, the E_1 and E_2 values are decreased by one or two standard deviations. It should be noted that the unobserved reflexions may play an important role in this procedure.

Σ_2 formula

When a sufficient number of signs is known, the $\sum_2$ formula (Hauptman & Karle, 1953) will easily lead to refinement of probable signs, and to a calculation of new signs. The weighted $\sum_2$ formula may be given as:

$$S(E_{\mathbf{h}})\simeq S(\textstyle\sum_{\mathbf{K}} W_{\mathbf{k}}W_{\mathbf{h}-\mathbf{k}}E_{\mathbf{k}}E_{\mathbf{h}-\mathbf{k}}) \qquad (6)$$

where $E_{\mathbf{k}}$ is E_L for the reflexion **k**, and $W_{\mathbf{k}}$ is the weight for $E_{\mathbf{k}}$. The summation is over all available terms.

For weights, defined according to (4), it is justified to generalize the formula for the probability that (6) gives the correct result (Cochran & Woolfson, 1955):

$$P_S=\tfrac{1}{2}+\tfrac{1}{2}\tanh\left[\frac{\sigma_3}{\sigma_2^{3/2}}\textstyle\sum_{\mathbf{K}}W_{\mathbf{k}}W_{\mathbf{h}-\mathbf{k}}E_{\mathbf{k}}E_{\mathbf{h}}E_{\mathbf{h}-\mathbf{k}}\right] \qquad (7)$$

with $\sigma_n=\sum^L Z^n$, Z being the atomic number of the light atoms. Analogous to (4) we define for the weight of the $\sum_2$ result:

$$W_S=(2P_S-1)^2\,. \qquad (8)$$

These formulae are applied as follows:

(i) Reflexions with $|E_1|$ less than some minimum value (say: $E_{\rm min}=0{\cdot}9$) are not used.

(ii) The reflexions of category *a* and some of the reflexions of category *b* (*i.e.* those with P_1 nearly equal to unity) are not recalculated; their E_1 values serve as input to the $\sum_2$ formula.

(iii) The remaining reflexions serve as input as well as output to equation (6).

Any resulting sign, $S(E_{\mathbf{h}})$ in (6), is compared with the input sign $S(E_1)$ for the reflexion **h**. In case these signs are not equal, and if the new sign is more reliable (*i.e.* if $W_S>W_1$), then E_1 is replaced by E_2 for this reflexion, and W_S is taken as its weight.

Convergence is achieved by repeating these calculations three or four times. Most of the reflexions still have $E_L=E_1$, but some are changed to $E_L=E_2$. Relatively more reflexions have $E_L=E_2$ for structures where the known 'heavy' atoms do not fix the signs of the dif-

Table 1. *Probability P_1 for dual E_L values*

$\lvert E_2\rvert=$	0·2	0·4	0·6	0·8	1·0	1·2	1·5	2·0	2·5	3·0	3·5	4·0	5·0
$\lvert E_1\rvert=0{\cdot}2$	0·50	0·52	0·54	0·57	0·62	0·67	0·75	0·88	0·957	0·989	0·998	1·000	1·000
$\lvert E_1\rvert=0{\cdot}4$		0·50	0·52	0·56	0·60	0·66	0·74	0·87	0·954	0·988	0·998	1·000	1·000
$\lvert E_1\rvert=0{\cdot}6$			0·50	0·54	0·58	0·63	0·72	0·86	0·950	0·987	0·997	1·000	1·000
$\lvert E_1\rvert=0{\cdot}8$				0·50	0·55	0·60	0·69	0·84	0·943	0·985	0·997	1·000	1·000
$\lvert E_1\rvert=1{\cdot}0$					0·50	0·56	0·65	0·82	0·932	0·982	0·996	0·999	1·000
$\lvert E_1\rvert=1{\cdot}2$						0·50	0·60	0·78	0·917	0·978	0·996	0·999	1·000
$\lvert E_1\rvert=1{\cdot}5$							0·50	0·71	0·88	0·967	0·993	0·999	1·000
$\lvert E_1\rvert=2{\cdot}0$								0·50	0·76	0·924	0·984	0·998	1·000
$\lvert E_1\rvert=2{\cdot}5$									0·50	0·80	0·952	0·992	1·000
$\lvert E_1\rvert=3{\cdot}0$										0·50	0·84	0·971	1·000
$\lvert E_1\rvert=3{\cdot}5$											0·50	0·87	0·998
$\lvert E_1\rvert=4{\cdot}0$												0·50	0·989
$\lvert E_1\rvert=5{\cdot}0$													0·50

ference coefficients well, either because they are in pseudo-special positions, or because they are not sufficiently heavy relative to the other atoms.

Fourier coefficients

Practically all reflexions used in the $\sum_2$ calculations, have acceptable E_L values; some reflexions with probabilities (P_1 and P_S) less than some minimum value (say: $P_1 < 0{\cdot}8$ and $P_S < 0{\cdot}97$) are rejected.

In addition to these results we carefully select the weaker E_L values, that is the reflexions with $|E_1| < E_{\min}$. In category *a* all such reflexions have known signs and can be used as Fourier coefficients. In category *b* many such reflexions have E_1 values with acceptable probabilities (say: $P_1 > 0{\cdot}8$) and can therefore be used.

For all accepted reflexions the E_L value is multiplied by a weighting factor ($\sqrt{W_1}$ or $\sqrt{W_S}$) and – if wanted – the E_L values are converted to F_L values. A Fourier synthesis based on these E_L or F_L values should give the complete light-atom structure.

Numerical results*

The present procedure has been tested on four known structures (1–4, Table 2) and has actually been used in the determination of three crystal structures (5–7, Table 2). In all these cases only one metal atom has been accepted as a 'heavy atom'. Of course, the final results will be better if one can add more atoms to the 'known part' of the structure, say one K or one or two *S* atoms in some of the structures listed in Table 2.

Three of the structures (2, 3 and 5) have been analysed in more detail; the results are listed in Tables 3 through 5. In general, some 10 to 20% of the strong reflexions ($|E_1| > 1{\cdot}0$) will change their signs (*i.e.* $E_L = E_2$), including some reflexions with very different $|E_1|$ and $|E_2|$ values. Moreover, a comparable number of strong reflexions with a weak heavy-atom contribution ($|E_1| > 1{\cdot}0$; $|E_H| < 0{\cdot}5$), often excluded from a difference Fourier synthesis, will now have a calculated sign. In our test cases all of the non-hydrogen atoms were found except, in some cases, some of the strongly vibrating carbon atoms. (For example, for structure 5 a difference Fourier map, signed by the Rb atom only, showed 31 of the 42 atoms of the molecule among the top 42 peaks, while a difference Fourier calculated from the *DIRDIF.B* output gave the complete structure from the top 42 peaks.)

Table 3. *Classification of reflexions according to magnitude* $|E_1|$ *and probability* P_1

Structure no.	2	3	5
Number of reflexions with $\vert E_1\vert < 0{\cdot}1$*	454	197	510
Reflexions with $0{\cdot}1 \le \vert E_1\vert < E_{\min}$			
$P_1 < 0{\cdot}7$*	474	275	1032
$P_1 \ge 0{\cdot}7$†	2444	624	1998
Reflexions with $\vert E_1\vert \ge E_{\min}$			
category *a*	185	124	86
category *b*, $P_1 \ge 0{\cdot}9999$	144	88	140
category *b*, $P_1 < 0{\cdot}9999$	538	350	423
Total number of reflexions	4239	1658	4189
($E_{\min}$)	(1·1)	(0·8)	(1·1)

* Reflexions in these classes will not be used for the calculation of a difference Fourier map.

† These reflexions are analysed in Table 5.

Comment about structure no. 4: the Mo atom is situated on a twofold axis, with a *y* coordinate near to $\frac{1}{8}$ ($y = 0{\cdot}1264$; deviation from $y = \frac{1}{8}$: 0·025 Å). Therefore a quarter of all reflexions have almost no Mo contribution to the structure factor. Nevertheless, the origin is completely fixed by the position of the Mo atoms, and our procedure works without any trouble at all.

Comment about structure no. 5: the *y* coordinate of the Rb atom is near to $\frac{1}{4}$ ($y = 0{\cdot}262$; deviation from $y = \frac{1}{4}$: 0·12 Å). Therefore, one half of all reflexions have almost no Rb contribution to the structure factor. Taking $y = 0{\cdot}25$, the origin is not fixed, and procedure *DIRDIF.A* (Paper I) could have been used, with, of course, an inaccurate Wilson plot for the weak reflexions. The origin is fixed by taking $y = 0{\cdot}26$ or 0·27 and the present procedure gives the structure completely.

Table 2. *Test structures for DIRDIF.B*

N' = number of symmetry-independent atoms per unit cell (hydrogens excluded); HA = known part of the structure; perc = percentage of scattering power represented by HA; N_{ref} = number of symmetry-independent reflexions.

No.	Structure	Space group	*Z*	N'	HA	Perc	N_{ref}	References
1*	$AuS_4N_3C_{13}H_{18}$	*Pbca*	8	21	Au	30%	3283	Noordik, Hummelink & van der Linden (1973)
2	$CoK_3O_6N_9C_6H_9.6\frac{2}{3}H_2O$	$P\bar{1}$	2	31	Co	9%	4239	Birker, Smits, Bour & Beurskens (1973)
3	$NiS_4N_3C_{13}H_{18}$	$P2_1/c$	4	21	Ni	13%	1658	Hendriks, Bosman & Beurskens (1974)
4	$MoS_8N_4C_{20}H_{40}$	$C2/c$	4	17	Mo	12%	1297	van der Aalsvoort & Beurskens (1974)
5	$RbO_{10}C_{32}H_{54}$	$C2/c$	8	43	Rb	10%	4189	Mooy, Degens, Noordik & van den Hark (1975)
6	$RhClS_3PN_2C_{24}H_{27}.CHCl_3$	$P2_1/c$	4	36	Rh	12%	2905	Bosman & Gal (1975)
7	$MoS_6N_5C_{13}H_{10}.N(C_4H_9)_4$	$P2_1/c$	4	42	Mo	10%	3570	Bosman & Nieuwpoort (1975)

* The preliminary procedure, applied to this structure (Noordik, 1971) has now been improved considerably.

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* A Fortran program, *DIRDIF.B*, is available on request.

Table 4. *Final results for reflexions signed by the* $\sum_2$ *formula (number of reflexions with* $E_L=E_1$ *and* $E_L=E_2$ *for different ranges of* $|E_2|-|E_1|$, *for all reflexions in category b with* $P_1<0·9999$)

The number of incorrectly signed reflexions is given in parentheses.

Range of ($|E_2|-|E_1|$)	Structure 2 $E_L=E_1$	Structure 2 $E_L=E_2$	Structure 3 $E_L=E_1$	Structure 3 $E_L=E_2$	Structure 5 $E_L=E_1$	Structure 5 $E_L=E_2$
0·0–0·5*	66	37 (1)	70 (1)	53 (3)	102 (1)	56
0·5–1·0	63	20	38	18 (2)	60	10
1·0–1·5	61	7	49	10 (1)	69	4
1·5–2·0	97	2	39	5	41	0
2·0†	184	1	68	0	79	2
Total	471	67 (1)	264 (1)	86 (6)	351 (1)	72
Determined with						
probability ≥0·97	532		334 (2)		396 (1)	
probability <0·97	6 (1)		16 (5)		27	

* Most of these reflexions have probabilities P_1 in the range 0·50–0·75.
† All of these reflexions have $P_1>0·98$.

Table 5. *Analysis of weak reflexions, for which the signs are not refined by the* $\sum_2$ *formula (all reflexions with* $0·1\le|E_1|<E_{\min}$, $P_1\ge0·7$)

Structure no.	2	3	5
Probability range (P_1)			
0·7—0·8	151 (46)	60 (18)	167 (58)
0·8—0·9	143 (22)	47 (9)	165 (41)
0·9—0·95	121 (14)	31 (4)	89 (11)
0·95-0·999	450 (18)	86 (7)	281 (29)
>0·999*	1579 (58)	400 (24)	1296 (66)

* This range includes reflexions of category *a* (with $P_1=1·0$). The majority of reflexions with wrong signs in this range had very small $|E_1|$ values ($|E_1|<0·2$); a wrong sign may then be caused by small errors in scale and temperature factors or by errors in the measured intensity.
The number of incorrect signs is given between parentheses.

Conclusions

The procedure described above proved to be useful in routine structure analysis of centrosymmetric heavy-atom compounds. The calculations are performed by an automatic computer program for the use of which no direct methods experience is required. We expect to be able to apply an analogous procedure to non-centrosymmetric structures as well.

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