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About the Treatment of Weak Reflexions in Direct Procedures

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Abstract

Measurement of weak reflexions is often skipped by computer-controlled diffractometers. Such a practice can cause systematic errors in the scale and thermal factors as calculated by a Wilson plot, with consequent difficulties for the solution of the crystal structure by direct methods. A simple statistical method for estimating unobserved reflexions is described together with applications of the method.

1. Abbreviations

N number of atoms in the unit cell
s $(\sin\theta)/\lambda$
B overall thermal factor
K absolute scale factor
f_j^0 scattering factor of jth atom at rest
f_j scattering factor of jth atom, thermal vibration included
F structure factor

$$\sum\nolimits_o = \sum_{j=1}^{N} f_j^{02}$$

$$\sum = \sum_{j=1}^{N} f_j^2$$

n_o number of different observed reflexions in a shell of the Wilson plot (symmetry equivalents and/or Friedel pairs included)
n_u number of different unobserved reflexions in a shell (symmetry equivalent and/or Friedel pairs included)

$n_t = n_o + n_u$

$I = |F_{\rm obs}|^2$

I_M threshold intensity for unobserved reflexions in a given range of the Wilson plot
z normalized value of I
z_M threshold value of z for unobserved reflexions in a given range of the Wilson plot.

2. Introduction

Reflexions with intensity below a certain threshold are often considered as 'unobserved', *i.e.* their measurement is skipped by computer-controlled diffractometers. There are practical reasons for this: time is saved in data collection and crystal radiation damage is reduced. But there are also some drawbacks:

(*a*) The structural information contained in such weak reflexions is neglected. This may be vitally

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important when a choice has to be made between centrosymmetric and non-centrosymmetric space groups or when refining a superstructure or when accurate electron density information is required (Schwarzenbach, 1989).

(*b*) The application of direct methods for structure solution could be unsuccessful.

We are mostly interested in point (*b*).

The influence of errors due to unobserved reflexions on the statistical tests for assessing centrosymmetry of crystals has been studied by Rogers, Stanley & Wilson (1955). They suggested the inclusion of all unobserved reflexions at $0{\cdot}43z_M$ for acentric and at $0{\cdot}30z_M$ for centric crystals. The treatment of unobserved reflexions in the least-squares adjustment of crystal structures was studied by Hamilton (1955). He suggested an assignment to all the unobserved reflexions of the value $I_M/2$ for non-centric and $I_M/3$ for centric crystals.

Vicković & Viterbo (1979) associated with the amplitude of each unobserved reflexion a random number in the range $(0, I_M)$ distributed according to the proper centric or non-centric distribution.

Further insight into the problem was given by Subramanian & Hall (1982) and by Hall & Subramanian (1982). They showed that a reliable estimate of the overall temperature factor B is important to the calculation of the normalized structure factors and to the application of structure invariant and seminvariant phasing methods. Triplet and quartet structure invariant relationships were shown to be more reliably estimated if the true value of B is used.

The first aim of this paper is to describe a statistical theory providing estimates for unobserved reflexions which, used in a Wilson scaling procedure, cause negligible deviations of the calculated B and K values from the true ones. Some applications will also be shown.

3. A statistical approach for estimating unobserved intensities

According to Wilson (1949) for each shell we have to calculate parameters occurring in the equation

$$\ln\left(\langle I\rangle/\textstyle\sum_o\right) \simeq \ln K - 2Bs^2 \qquad (1)$$

where $\langle I\rangle = (\sum_{p=1}^{n_t} I_p)/n_t$. Relation (1) may be written in a more explicit way as

$$\ln\left\{\frac{(\sum_{\text{obs}} I + \sum_{\text{unobs}} I)}{(n_o + n_u)\sum_o}\right\} \simeq \ln K - 2Bs^2. \qquad (2)$$

If unobserved reflexions are omitted, (2) is replaced by

$$\ln\left\{\frac{\sum_{\text{obs}} I}{n_o \sum_o}\right\} \simeq \ln K - 2Bs^2. \qquad (3)$$

Comparison of (3) with (2) shows that omitting weak data causes the average intensity for each shell to increase, so producing error in the scaling factor. If the proportion of unobserved data increases with s (as usual), the value of B estimated *via* the Wilson plot will be smaller than the true one. A supplemental consequence is that the z distribution will appear acentric if the structure is centric or centric if the structure is hypercentric or suffers from some pseudotranslational symmetry. These problems cannot be overcome by introducing in the data set unobserved reflexions with zero intensity. In this case (2) is replaced by

$$\ln\left\{\frac{\sum_{\text{obs}} I}{(n_o + n_u)\sum_o}\right\} \simeq \ln K - 2Bs^2; \qquad (4)$$

the average intensity for each shell decreases and, in agreement with previous observations, the estimated B value will be too large, the z distribution will appear centric for acentric structures or hypercentric for centric ones.

In order to provide a sensitive intensity estimate for unobserved reflexions we write (2) in the form

$$\ln\left\{\frac{\sum_{\text{obs}} I}{n_t \sum_o}\left[1 + \frac{\sum_{\text{unobs}} I}{\sum_{\text{obs}} I}\right]\right\} \simeq \ln K - 2Bs^2.$$

No important errors in K and B estimates will be produced if the ratio $(\sum_{\text{unobs}} I)/(\sum_{\text{obs}} I)$ is correctly estimated. Since

$$\frac{\sum_{\text{unobs}} I}{\sum_{\text{obs}} I} \simeq \frac{\sum_{\text{unobs}} z}{\sum_{\text{obs}} z}, \qquad (5)$$

the well known distribution of z variables can be exploited for estimating the ratio (5). Indeed,

$$\sum_{\text{unobs}} z = n_u \int_0^{z_M} zP(z)\,dz \Big/ \int_0^{z_M} P(z)\,dz \qquad (6a)$$

$$\sum_{\text{obs}} z = n_o \int_{z_M}^{\infty} zP(z)\,dz \Big/ \int_{z_M}^{\infty} P(z)\,dz$$

$$= n_o\left\{1 - \int_0^{z_M} zP(z)\,dz\right\} \Big/ \left\{1 - \int_0^{z_M} P(z)\,dz\right\}. \qquad (6b)$$

According to the actual space group, $P(z)$ will coincide with a centric or acentric Wilson distribution.

The main obstacle to the use of (6) is that I_M is known but z_M is unknown: indeed this last quantity depends on the thermal factor, which is still unknown at this state of the process. However, a reliable estimate of z_M may be obtained from the property of the cumulative distribution function $N(z)$ (Howells, Phillips & Rogers, 1950). Since $N(z_M) \simeq n_u/n_t$, the $N(z)$ function may be inverted to provide z_M.

For acentric crystals $N(z) = 1 - \exp(-z)$, from which

$$\exp(z_M) = n_t/n_o$$

or

$$z_M = \ln(n_t/n_o). \quad (7)$$

For centric crystals $N(z) = \mathrm{erf}(z/2)^{1/2}$: thus z_M is defined by the equation

$$n_u/n_t = (2/\pi)^{1/2} \int_0^{z_M^{1/2}} \exp(-z^2/2)\,dz. \quad (8)$$

Once z_M has been calculated from (7) or (8), the ratio (5) may be estimated *via* (6). We obtain, for acentric crystals,

$$\sum_{\text{unobs}} z = n_u \gamma(2, z_M)/[\gamma(1, z_M)] \quad (9a)$$

$$\sum_{\text{obs}} z = n_o[1-\gamma(2, z_M)]/[1-\gamma(1, z_M)] \quad (9b)$$

where

$$\gamma(a, u) = \int_0^u z^{a-1}e^{-z}\,dz$$

is the incomplete gamma function.

For centric crystals we obtain

$$\sum_{\text{unobs}} z \simeq 2n_u \gamma(\tfrac{3}{2}, z_M)/\gamma(\tfrac{1}{2}, z_M) \quad (10a)$$

$$\sum_{\text{obs}} z \simeq [1-(2/\pi^{1/2})\gamma(\tfrac{3}{2}, z_M)]/[1-\pi^{-1/2}\gamma(\tfrac{1}{2}, z_M)] \quad (10b)$$

According to (9) and (10), the expected value of an unobserved reflexion is

$$\langle z\rangle_{\text{unobs}} = \gamma(2, z_M)/\gamma(1, z_M)$$

for acentric crystals,

$$\langle z\rangle_{\text{unobs}} = 2\gamma(\tfrac{3}{2}, z_M)/\gamma(\tfrac{1}{2}, z_M)$$

for centric ones. The accuracy of each estimate depends on z_M: the smaller z_M, the smaller the associated variance $\sigma^2(z)$. Indeed

$$\sigma^2(z) = \frac{\int_0^{z_M} z^2 \exp(-z)\,dz}{\int_0^{z_M} \exp(-z)\,dz} - \langle z\rangle^2$$

$$= \gamma(3, z_M)/\gamma(1, z_M) - [\gamma(2, z_M)/\gamma(1, z_M)]^2$$

for acentric crystals and

$$\sigma^2(z) = \frac{(2\pi)^{-1/2}\int_0^{z_M} z^{3/2}\exp(-z/2)\,dz}{(2\pi)^{-1/2}\int_0^{z_M} z^{-1/2}\exp(-z/2)\,dz} - \langle z\rangle^2$$

$$= 4\{\gamma(\tfrac{5}{2}, z_M)/\gamma(\tfrac{1}{2}, z_M) - [\gamma(\tfrac{3}{2}, z_M)/\gamma(\tfrac{1}{2}, z_M)]^2\}$$

for centric structures.

4. Applications

The efficiency of our approach has been tested on the structures quoted in Table 1. The last two structures (BOBBY and POCRO) suffer from strong pseudotranslational symmetry: the pseudotranslational vectors are $\mathbf{u} = (\mathbf{a}+\mathbf{b}+\mathbf{c})/2$ and $\mathbf{u} = \mathbf{a}/3$ respectively. The estimated fractional scattering powers of the substructural parts as estimated by *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) are 57% for BOBBY and 70% for POCRO. Owing to the strong pseudosymmetry, BOBBY shows a centric and POCRO a hypercentric z distribution. Their inclusion in the set of test structures aims at checking if their anomalous z distributions invalidate the robustness of our approach.

Table 1. *Code name, space group, crystallochemical formula and* $(\sin\theta/\lambda)_{max}$ *data for test structures*

Structure code	Space group	Molecular formula	Z	s_{max} (Å^{-1})
LOGANIN	$P2_12_12_1$	$C_{17}H_{26}O_{10}$	4	0·67
CEPHAL	$C2$	$C_{18}H_{21}NO_3$	8	0·58
INOS	$P2_1/n$	$C_6H_{12}O_6.H_2O$	8	0·60
DIAM	$P4_2/n$	$C_{14}H_{20}O$	8	0·56
BOBBY	$P2_13$	$Na^+Ca^{2+}N(CH_2CO_2)_3^{3-}$	4	0·59
POCRO*	$B112/m$	Cr_5KSe_8	2	0·70

Complete references for the structures are not given for the sake of brevity. The reader is referred to a magnetic tape distributed by the crystallographic group in Göttingen.

* Nguyen-Huy Dung, Vo-Van Tien, Behm & Beurskens (1987).

For each structure in Table 1 the code name, the space group, the molecular formula and the number of molecules in the unit cell (Z) are given; in the last column s_{max} is the maximum value of $(\sin\theta)/\lambda$ for the observed reflexions.

Weak reflexions are not always contained in the experimental data sets (for LOGANIN, CEPHAL, DIAM, INOS); on the contrary, weak intensities are set to zero for POCRO. In order to have homogeneous starting conditions, we decided to use for our tests the calculated instead of the observed structure factors. They were generated up to the appropriate limit s_{max} by using the same temperature factor for all the atoms.

Tests were made according to the following protocols:

(*a*) *Protocol A*: we calculated the Wilson plot for all the generated reflexions. Let B and K be the overall temperature and the scale factors in these conditions.

(*b*) *Protocol B*: we eliminated the reflexions with the smallest $|F|$ values and we calculated again the Wilson plot. Let B' and K' be the new estimated parameters: $(B', K')_p$ are the values of B and K obtained when about p% of the reflexions have been eliminated.

(*c*) *Protocol C*: we associated with the weakest reflexions the value $|F| = 0$. The Wilson plot gives in these conditions the parameters $(B'', K'')_p$.

(*d*) *Protocol D*: weak reflexion intensities were estimated according to our approach described in § 3 and were used for the calculation of the Wilson plot. $(B''', K''')_p$ are the calculated parameters.

The various B and K values are shown in Table 2 for each structure. The following may be observed:

(1) Severe errors in K and B estimates arise for increasing values of p, both when weak reflexions are

Table 2. *Overall thermal and scale parameters according to various protocols*

	LOGANIN	CEPHAL	INOS	DIAM	BOBBY	POCRO
(B/K)	2·78	3·33	2·36	4·27	1·96	0·71
	0·98	1·14	1·01	0·95	0·91	1·20
$(B'/K')_{15}$	2·30	2·64	2·04	3·65	1·54	0·68
	1·09	1·28	0·97	1·01	0·94	1·02
$(B'/K')_{30}$	1·76	1·84	1·59	2·92	1·16	0·51
	1·18	1·44	0·96	1·08	0·91	0·93
$(B'/K')_{60}$	0·86	0·16	0·59	0·88	0·32	0·16
	1·26	1·71	0·92	1·21	0·82	0·68
$(B''/K'')_{15}$	2·84	3·45	2·37	4·29	2·01	0·71
	0·96	1·11	1·00	0·94	0·90	1·20
$(B''/K'')_{30}$	3·13	3·93	2·47	4·42	2·09	0·73
	0·88	0·99	0·98	0·91	0·89	1·19
$(B''/K'')_{60}$	4·72	6·98	3·29	5·14	3·20	0·89
	0·57	0·50	0·82	0·81	0·72	1·18
$(B'''/K''')_{15}$	2·76	3·33	2·35	4·25	1·95	0·70
	0·99	1·14	1·01	0·95	0·91	1·20
$(B'''/K''')_{30}$	2·72	3·33	2·31	4·18	1·83	0·69
	1·00	1·14	1·01	0·96	0·93	1·20
$(B'''/K''')_{60}$	2·64	3·20	2·37	3·78	1·77	0·63
	1·01	1·17	0·96	1·02	0·91	1·13

omitted and when a vanishing intensity is associated with them.

(2) Our statistical approach provides reliable estimates of K and B even for the limiting case of $p = 60$. The presence of pseudosymmetry does not invalidate the approach. Indeed, good results are also obtained for BOBBY and POCRO.

In spite of the above achievements caution is advised for the possible subsequent use of the estimated z's in the phasing process. Take CEPHAL as an example: when 60% of the reflexions are statistically estimated, the distributions in the various ranges of the Wilson plot of n_o, n_u, z_M, $\langle z \rangle$ and $\sigma(z)$ are those given in Table 3. The σ column clearly shows the wide range of accuracy for the statistical estimates of z.

A question now arises: if the z's of the unobserved reflexions are statistically evaluated, can the uncertainty on their values compromise the success of the phasing process? Furthermore, can such an uncertainly be fatal for those methods [*i.e.* representation theory (Giacovazzo, 1977) and neighbourhood concept (Hauptman, 1975)] which make full use of the reciprocal space for estimating single phase relationships?

In order to answer the above question we have made a statistical analysis of the triplet reliability for two centrosymmetric structures, INOS and DIAM (see Tables 4 and 5). Calculations were performed according to protocols *A*, *B* (60% of reflexions eliminated from the data set) and *D* (the same 60% regenerated by statistical estimates).

In each table we give for various values of ARG the number of triplet relationships (nr) which have the argument of the hyperbolic tangent (the reliability parameter of the triplet) larger than ARG. According to the Cochran & Woolfsoon (1955) formula, ARG = $|E_{\mathbf{h}}E_{\mathbf{k}}E_{\mathbf{h}-\mathbf{k}}|/N^{1/2}$: thus all the triplets are always estimated positive. According to the P_{10} formula (Cas-

Table 3. *Cephal parameters for each Wilson shell*

Shell	n_o	n_u	z_M	$\langle z \rangle$	$\sigma(z)$
1	68	7	0·098	0·048	0·028
2	117	10	0·082	0·040	0·024
3	145	22	0·141	0·069	0·041
4	169	37	0·198	0·096	0·057
5	179	55	0·268	0·128	0·077
6	181	81	0·370	0·174	0·106
7	151	110	0·547	0·249	0·157
8	169	150	0·635	0·284	0·182
9	174	127	0·548	0·249	0·157
10	184	156	0·614	0·276	0·176
11	160	206	0·827	0·357	0·235
12	141	218	0·935	0·396	0·264
13	92	285	1·410	0·545	0·388
14	59	360	1·960	0·679	0·517
15	20	356	2·934	0·835	0·699
16	15	404	3·330	0·876	0·757
17	5	352	4·268	0·939	0·859

Table 4. *INOS: number of triplet relationships (nr) and number of wrongly estimated triplet relationships (nw) according to P_3 and P_{10}*

	ARG	P_3 $nr(nw)$	Positive estimated triplets P_{10} $nr(nw)$	Negative estimated triplets P_{10} $nr(nw)$
Protocol *A*	0·2	3157 (281)	2278 (8)	217 (39)
	0·6	3157 (281)	2278 (8)	80 (2)
	1·2	1008 (30)	1317 (0)	
	2·0	193 (3)	401 (0)	
	3·8	11 (0)	8 (0)	
Protocol *B*	0·2	2273 (242)	497 (5)	
	0·6	374 (6)	497 (5)	
	1·0	58 (2)	108 (0)	
	1·6	8 (0)	14 (0)	
Protocol *D*	0·2	3371 (331)	2036 (6)	131 (17)
	0·6	2549 (174)	2036 (6)	22 (1)
	1·2	537 (11)	878 (0)	
	2·0	81 (2)	195 (0)	
	3·8	1 (0)	3 (0)	

Table 5. *DIAM: number of triplet relationships (nr) and number of wrongly estimated triplet relationships (nw) according to P_3 and P_{10}*

	ARG	P_3 $nr(nw)$	Positive estimated triplets P_{10} $nr(nw)$	Negative estimated triplets P_{10} $nr(nw)$
Protocol *A*	0·2	4000 (550)	2485 (21)	267 (50)
	0·6	3712 (472)	2485 (21)	62 (6)
	1·2	676 (37)	1180 (0)	3 (1)
	2·0	98 (4)	296 (0)	
	3·8	2 (0)	5·(0)	
Protocol *B*	0·2	1350 (178)	273 (7)	
	0·6	219 (14)	273 (7)	
	1·0	33 (1)	52 (0)	
	1·4	2 (0)	6 (0)	
Protocol *D*	0·2	4000 (529)	2029 (17)	102 (15)
	0·6	2144 (203)	2029 (17)	11 (0)
	1·2	254 (11)	599 (0)	
	2·0	27 (0)	80 (0)	
	3·4	0 (0)	1 (0)	

carano, Giacovazzo, Camalli, Spagna, Burla, Nunzi & Polidori, 1984), $\mathrm{ARG} = (|E_{\mathbf{h}}E_{\mathbf{k}}E_{\mathbf{h}-\mathbf{k}}|/N^{1/2}\{1+Q\}$ where Q may be a positive or a negative contribution arising from the full reciprocal space. Thus triplets can be estimated positive or negative. The number of wrongly estimated triplets (*nw*) is given in parentheses for each *nr*. In practice, in Tables 4 and 5 the triplet relationships are ranked in decreasing order of reliability according to P_3 and P_{10}.

Tables 4 and 5 clearly show that omitting weak reflexions from the data set is a bad choice. In particular:

(1) If triplets are estimated according to P_3 the number of triplets with reliability larger than a minimum threshold (active triplets in a direct procedure) is markedly small for protocol *B* and sufficiently large for protocols *A* and *D*. The efficiency of protocol *D* in ranking triplets is comparable with that of protocol *A*.

(2) If triplets are estimated according to P_{10}, the loss of efficiency for protocol *B* is dramatic: both the number of possible active triplets and the reliability of the positive estimated triplets for protocol *B* is enormously reduced. Furthermore, no negative triplets are estimated. Conversely the use of statistical estimates for weakest reflexions (protocol *D*) is able to restore an efficiency comparable with that of protocol *A*.

5. Concluding remarks

Skipping measurement of weak reflexions saves data-collection time but may cause systematic errors in the normalization process of the experimental intensities. Such errors usually reduce the efficiency of direct methods: thus the gain in data-collection time may be nullified by the larger time needed for the structure solution.

A probabilistic procedure is described which is able to compensate for the omission of weak reflexions both in the normalization process and in the probabilistic procedures for the estimation of triplet invariants.

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