Acta Cryst. (1969). A**25**, 427

Pseudo-Merohedral Twinning: The Treatment of Overlapped Data

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(*Received* 10 *October* 1968)

Expressions which enable data to be 'corrected' for twinning are examined. If the volume ratio of the twin individuals is known, structure factors may be calculated and used in the normal way. Particular care must, however, be taken with weighting. For least-squares refinement or for comparing observed and calculated intensities, overlapped data can be used directly in the normal Kennicott procedure which initially requires a knowledge of the volume ratio, but can refine it. Alternatively the data can be modified in such a way that calculations are independent of the volume ratio. All three methods have been successfully applied to overlapped data from Para Red (1-*p*-nitrobenzeneazo-2-naphthol).

1. Introduction

It is well known (*e.g.* Buerger, 1960, Chapter 5) that in some types of twinning, reflexions from one individual of the twin may be superimposed or almost superimposed, on reflexions of the other. In some cases all the reflexions are thus superimposed or 'overlapped' (*i.e.* not resolved), but in others there exist reflexions arising entirely from one or other of the individuals. The latter condition may, for example, be brought about because of systematic absences.

Crystals which are twinned by pseudo-merohedry can belong to either of these groups depending upon the twin obliquity angle; if the obliquity is small enough it is possible that all reflexions are overlapped, but if large all, or most, may be resolved.* The present work was done in connection with Para Red (1-*p*-nitrobenzeneazo-2-naphthol, $C_{16}H_{11}N_3O_3$) which, as described elsewhere (Grainger & McConnell, 1969*a*) is pseudo-merohedrally twinned, exhibits anisotropic thermal contraction, and in addition has a comparatively small obliquity (0°22′) at room temperature. As a result the reflexions at room temperature are effectively overlapped, while at −140 °C, when the obliquity has increased (to 0°48′) many reflexions are single. The latter circumstance enabled the twinning to be detected by X-ray means and also enabled the type of twinning, and the intensity symmetry between the two interleaved reciprocal lattices to be determined. The two reciprocal lattices could equally well be related by a mirror plane or by either of two twofold axes (with a corresponding uncertainty in the direct-space symmetry). For the methods below any of these would suffice. A knowledge of the twinning symmetry in reciprocal space is essential for the application of any correction methods, and must be arrived at by X-ray, optical, or trial-and-error methods.

If normal structure-solving or refinement procedures are to be followed, it is necessary to derive structure factors 'corrected for twinning' from the overlapped data, and this is treated in part A below. If, however, one merely wishes to carry out a least-squares refinement, or to compare observed and calculated intensities, one can use either the normal method of Kennicott (Kennicott, 1963), which uses overlapped data directly, or a modification of it which has the advantage of being independent of the volume ratio of the two individuals of the twin: these are treated in part B.

All these methods rely upon the premise that if the crystal comprises two individuals *A* and *B* in twin relationship, these scatter independently, and the total energy reaching each spot on the film is the arithmetic sum of the contributions at that point due to each of the components separately. Absorption is assumed to be negligible.

One can assign two meanings to the concept of 'correction' of intensities or structure factors in this context. 'Correction' can mean that the contribution to a given spot on the film from the individual *B* of smaller volume is mathematically removed, so that the set of 'corrected' readings refers only to the larger individual *A*. Alternatively, 'correction' can mean that one mathematically reorients *B*, and takes into account the contributions it would have made if correctly oriented, thus obtaining the readings which would have been obtained from an untwinned crystal of the same total volume. The latter connotation was selected and it facilitated the preparation of the data for the different procedures described below, since scale factors were not then sensitive to the volume ratio of the two individuals.

While the methods below are illustrated by reference to the data from Para Red, the principles are readily adaptable to other cases of overlapping.

* The twin obliquity alone does not reliably indicate whether reflexions will be resolved. Other considerations are the strength of the high-angle reflexions (which depends in turn upon the structure, the temperature, and the X-ray wavelength), the spot shape, and the Weissenberg geometry (Grainger, 1969).

A. Structure amplitudes from overlapped twin crystal data

2. *Expressions for correction of intensity readings*

As pointed out elsewhere, in the photographs of twinned Para Red at room temperature, pairs of reflexions of the type hkl and $h,-(6h+k),l$ (related by the (010) plane) called herein 'related reflexions', consist in general of two unresolved components, and each of these reflexions contains a contribution from the part of the crystal in twin orientation which may be regarded as belonging to the related reflexion.

Consider a pair of related reflexions P and Q. Let their observed readings be P_o and Q_o, and their required corrected readings P_c and Q_c respectively, on an arbitrary scale. Let the twinned crystal contain two individuals A and B, A being the larger, the reflexions being correctly indexed for A. Let the twin fraction, *i.e.* the ratio of the volume of the small component B to the total volume, be $f(\leq 0{\cdot}5)$. The contributions from the two individuals A and B will be proportional to their volumes which are in the ratio $(1-f):f$. Of the total 'intensity' P_c, a fraction $(1-f)$, due to A, will be located at the 'proper' reflexion P and the remaining fraction f (due to B) will be superimposed on reflexion Q. The reverse is true for the corrected intensity Q_c. We therefore have the following expressions for the reflexions P and Q:

$$P_o=(1-f)P_c+fQ_c\,, \tag{1}$$

$$Q_o=fP_c+(1-f)Q_c\,. \tag{2}$$

Solving,

$$P_c=P_o+\alpha(P_o-Q_o)\,, \tag{3}$$

$$Q_c=Q_o+\alpha(Q_o-P_o)\,, \tag{4}$$

where

$$\alpha=\frac{f}{1-2f}\,. \tag{5}$$

Thus if the twin fraction f is known for the crystal, one may correct a reading I_o by use of the following relation which expresses both (3) and (4):

$$\text{Corrected intensity reading}=I_c=I_o+\alpha(I_o-I_o') \tag{6}$$

where I_o' is the observed intensity of the related reflexion. This relation has been employed by Zachariasen & Plettinger (1965). Since the difference (I_o-I_o') is positive or negative according to whether the reflexion I_o is the stronger or weaker of the pair, this correction has the effect of increasing the stronger and decreasing the weaker. In effect, one finds α(stronger–weaker) for a related pair, adds it to the stronger and subtracts it from the weaker, the fraction α being a constant for a given crystal. The correction of intensities can be done equally well before or after making Lorentz and polarization corrections, since they are the same for related reflexions. It was found more convenient to do so before making Lp corrections. For a crystal having $f=0{\cdot}50$ this correction procedure would fail, as related reflexions would be of equal intensity and α would be infinite.

3. *Finding the twin fraction for a crystal of Para Red*

In order to apply the correction equation (6), one requires f, the twin fraction. It may be found experimentally by cooling the crystal so that the twin components of each spot are resolved (or split) as much as possible. Their intensities can then be measured separately.

Consider the split components of two related reflexions P and Q (Fig. 1). Components P_A and Q_A are those belonging to the larger individual A of the twin crystal, while Q_B and P_B respectively may be regarded as the mirror images of these, belonging to the smaller individual B.

The intensity relationships are

$$\frac{P_A}{Q_B}=\frac{Q_A}{P_B}=\frac{\text{volume of } A}{\text{volume of } B}=\frac{1-f}{f} \tag{7}$$

where P_A, *etc.* represent the intensity readings.

Thus in principle one can obtain two values of f for each pair of related reflexions on a low temperature photograph using (7). This method was used to determine experimentally the twin fraction of the crystal used for collecting the Para Red data at room temperature, so that these data could be corrected for twinning. From a low-temperature $hk0$ photograph taken with this crystal forty values of the ratio $(1-f)/f$ were obtained, with the following results:

$$\left.\begin{aligned}(1-f)/f&=2{\cdot}50\pm 0{\cdot}50\\ f&=0{\cdot}29\pm 0{\cdot}04\\ \alpha&=0{\cdot}67\pm 0{\cdot}22\end{aligned}\right\} \tag{8}$$

(The errors are the standard deviations.)

It was thus revealed that this crystal contained approximately 29% in twin orientation.

4. *The accuracy of corrected intensity readings*

The expression for the corrected intensity, equation (6), may be written

$$I_c=(1+\alpha)I_o-\alpha I_o'\,. \tag{9}$$

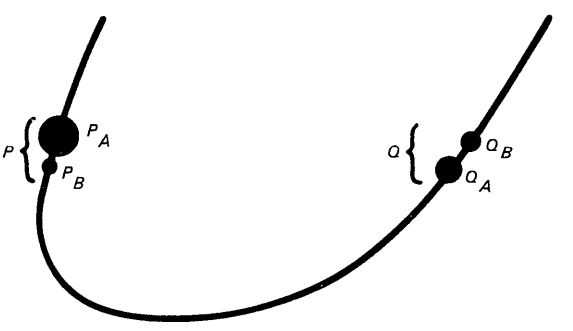

Fig. 1. Split related reflexions of Para Red as they appear on a Weissenberg festoon. (Diagrammatic only).

The theory of propagation of errors (*e.g.* Worthing & Geffner, 1943, p.209) gives for the standard deviation of I_c,

$$\sigma_c^2 = \left(\frac{\partial I_c}{\partial I_o}\right)^2 \sigma_o^2 + \left(\frac{\partial I_c}{\partial I_o'}\right)^2 (\sigma_o')^2 + \left(\frac{\partial I_c}{\partial \alpha}\right)^2 \sigma_\alpha^2 , \quad (10)$$

where σ_c, σ_o, σ_o' and σ_α are the standard deviations of I_c, I_o, I_o' and α respectively. Hence

$$\sigma_c^2 = (1+\alpha)^2\sigma_o^2 + \alpha^2(\sigma_o')^2 + (I_o - I_o')^2\sigma_\alpha^2 \quad (11)$$

gives the variance of I_c in terms of the variances of I_o, I_o' and α.

For Para Red typical values employed were

$\sigma_o = 0{\cdot}15 I_o$ if $I_o > 4I_{\min}$,
$\sigma_o = 0{\cdot}15 \times 4I_{\min}$ if $I_o \le 4I_{\min}$
(and similarly for σ_o') .
$\alpha = 0{\cdot}67$ [equation (8)]
$\sigma_\alpha = 0{\cdot}22$ [equation (8)] .

The values for σ_o were somewhat arbitrarily selected, but seemed reasonable in view of the photographic visual comparison technique employed in measuring the intensities.

The corrected intensities (before applying the Lorentz and polarization factors) are then given by (6), and their variances by (11).

5. *Corrected 'observed' structure factors and their standard deviations*

The 'observed' structure amplitude is given by

$$|F_o| = S\sqrt{\frac{I_c}{\mathrm{Lp}}} , \quad (12)$$

where 1/Lp is the Lorentz and polarization correction factor and S a scale factor placing the structure factors on approximately the absolute scale.

The estimated standard deviation (e.s.d.) of $|F_o|$ on conventional error theory is σ_F given by

$$\frac{\sigma_F}{|F_o|} = \tfrac{1}{2} \cdot \frac{\sigma_c}{I_c} , \quad (13)$$

where σ_c is the e.s.d. of I_c.

For the majority of reflexions, expressions (12) and (13) are quite satisfactory. However, an obvious deficiency is revealed in the few cases where I_c is negative, when both equations fail. (Owing to errors in I_o, I_o' and α, I_c sometimes turns out to be negative when correcting the weaker of a related pair.) Nor can conventional small-error theory [equation (13)] be expected to apply when σ_c is greater than I_c or comparable with it, which can occur for the weaker of a related pair if I_o and I_o' are large and, therefore, have large absolute error estimates.

At first, such 'awkward' reflexions were omitted from least-squares calculations. Later they were included, $|F_o|$ and σ_F being calculated according to the value of σ_c as follows.

Group (i) If $\sigma_c < 0{\cdot}3I_c$, the conventional equations (12) and (13) were employed.

Group (ii) If $I_c \ge \sigma_c \ge 0{\cdot}3I_c$,

$$|F_o| = \tfrac{1}{2}S[\mathrm{Lp}]^{-1/2}[(I_c+\sigma_c)^{1/2} + (I_c-\sigma_c)^{1/2}] , \quad (14a)$$

$$\sigma_F = \tfrac{1}{2}S[\mathrm{Lp}]^{-1/2}[(I_c+\sigma_c)^{1/2} - (I_c-\sigma_c)^{1/2}] . \quad (14b)$$

Group (iii) If $\sigma_c > |I_c|$ and $I_c + \sigma_c > 0$,

$$|F_o| = \tfrac{1}{2}S[\mathrm{Lp}]^{-1/2}(I_c+\sigma_c)^{1/2} , \quad (15a)$$

$$\sigma_F = |F_o| . \quad (15b)$$

This group includes some instances where I_c is negative.

Group (iv) If $I_c + \sigma_c \le 0$,

$$|F_o| = 0 , \quad (16a)$$

$$\sigma_F = 0{\cdot}5 . \quad (16b)$$

This group contains only negative values of I_c.

The following assumptions were made in arriving at these relations, for the respective groups above:

(i) Small-error theory must apply if σ_c is small compared with I_c. The selection of the $0{\cdot}3I_c$ limit is explained below.

(ii) If $I_c = 9 \pm 7$, small error theory would give $\sqrt{I_c} = 3 \pm 1{\cdot}2$. However, if one assumes (see below) that the true value if I_c lies between $9-7$ and $9+7$, then $\sqrt{I_c}$ will lie between 1·4 and 4·0, so the answer will be $\sqrt{I_c} = \frac{1}{2}(4{\cdot}0+1{\cdot}4) \pm \frac{1}{2}(4{\cdot}0-1{\cdot}4) = 2{\cdot}7 \pm 1{\cdot}3$. This is the basis of equations (14).

The assumption that $I_c \pm \sigma_c$ defines the limits of the 'likely' or 'possible' values of I_c is unusual, since for a normal distribution the probability that I_c lies within this range is only 0·68. However, if the limits are generalized to $I_c \pm n\sigma_c$, this gives $n\sigma_c$ in place of σ_c in both equations (14). One then finds that for small errors ($\sigma_c \ll I_c$), equation (14*b*) reduces to (13) only if $n=1$, while (14*a*) reduces to (12) for any small value of n. Thus, by taking $n=1$ continuity is assured: in fact the conventional equations are preferred for Group (i) reflexions only because of the ease and accuracy of the calculation, not as a matter of principle. When $\sigma_c = 0{\cdot}3I_c$ both sets of equations give essentially the same result.

(iii) The true value of I_c is known to be at least zero, algebraically. Thus, if it is calculated from the data that $I_c = -2 \pm 11$ or $I_c = 2 \pm 7$, this is taken in both cases to mean that I_c lies between 0 and 9, so that $\sqrt{I_c}$ is $\frac{1}{2}(\sqrt{9}+0)$ and the s.d. of $\sqrt{I_c}$ is $\frac{1}{2}(\sqrt{9}-0)$, in accordance with equations (15).

(iv) A few cases were encountered where I_c was negative, and even $I_c + \sigma_c$ did not exceed zero. Consider, for example, the case where the 'corrected' intensity is $I_c = -3 \pm 1$. This is taken to indicate that $I_c = 0$ with some certainty, so that $\sqrt{I_c} = 0$. The value of the s.d. has to be set at some arbitrary figure for the least-squares program, as the above principles can-

Table 1. *Structure factor table for Para Red*

The column headings FOBS, ESD, FCAL and ANG indicate respectively $|F_o|$, the e.s.d. of $|F_o|$, $|F_c|$, and the phase angle of F_c. T represents the group code number (see text).

H	K	L	FOBS	ESD	FCAL	ANG	T
0	2	0	18.2	1.4	18.4	177	21
0	4	0	20.1	1.5	21.4	179	21
0	6	0	27.0	2.0	29.5	163	21
0	8	0	13.6	1.0	12.3	68	21
0	10	0	19.1	1.4	20.4	18	21
0	12	0	7.6	0.6	7.9	160	21
0	14	0	16.4	1.2	17.1	193	21
0	16	0	8.8	0.7	9.0	181	21
0	18	0	6.4	0.5	6.2	140	21
0	20	0	5.3	0.4	5.3	81	21
0	22	0	4.5	0.3	4.1	133	21
0	24	0	4.8	0.4	4.4	82	21
0	26	0	0.9	0.9	1.5	316	23
0	28	0	0.8	0.8	0.7	354	23
0	30	0	3.6	0.3	3.4	266	21
1	0	0	137.3	12.2	120.5	351	1
1	2	0	68.9	6.7	63.3	245	1
1	4	0	4.5	1.8	4.1	300	12
1	6	0	13.2	1.4	9.8	205	1
1	8	0	6.7	1.0	7.6	40	11
1	10	0	35.4	3.5	31.4	335	1
1	12	0	12.5	1.7	9.6	128	31
1	14	0	7.3	1.7	6.8	181	12
1	16	0	7.8	0.7	7.1	138	1
1	18	0	6.1	1.6	3.7	260	12
1	20	0	7.2	1.3	5.3	99	12
1	22	0	3.2	0.5	3.3	290	1
1	24	0	4.4	0.6	3.9	34	31
1	26	0	0.7	0.7	0.9	92	33
2	0	0	20.5	1.8	18.6	0	1
2	2	0	21.8	2.0	19.5	228	1
2	4	0	5.2	0.5	4.3	168	1
2	6	0	7.7	0.9	7.1	183	1
2	8	0	5.3	1.9	4.9	185	12
2	10	0	18.5	1.8	15.6	329	1
2	12	0	10.4	5.1	7.3	187	12
2	14	0	2.9	1.3	2.3	350	12
2	16	0	2.9	0.5	2.3	94	2
2	18	0	1.1	1.1	0.7	357	13
2	20	0	6.8	0.6	6.0	138	1
2	22	0	4.2	0.5	3.3	289	1
3	0	0	8.9	0.8	7.9	336	1
3	2	0	1.2	1.2	1.0	241	13
3	4	0	0.6	0.6	0.8	79	13
3	6	0	0.5	0.5	2.2	141	13
3	8	0	1.2	1.2	2.5	118	13
3	10	0	4.2	0.4	4.1	349	1
3	12	0	4.0	0.5	4.5	192	1
3	14	0	0.7	0.7	0.5	335	33
4	0	0	5.6	1.0	3.0	307	12
4	2	0	0.0	0.5	0.3	216	14
4	4	0	0.0	0.5	0.8	231	14
4	6	0	0.4	0.4	0.6	359	33
-1	2	0	37.4	7.1	31.8	137	12
-1	4	0	53.8	6.0	53.6	206	1
-1	6	0	16.5	16.5	22.4	227	13
-1	8	0	25.8	11.7	22.4	213	12
-1	10	0	11.0	1.1	11.3	80	1
-1	12	0	8.6	1.5	13.7	208	12
-1	14	0	7.2	0.9	5.7	258	1
-1	16	0	13.1	6.0	14.3	201	12
-1	18	0	12.5	1.7	13.0	156	31
-1	20	0	12.4	1.3	12.2	224	1
-1	22	0	1.5	1.5	2.6	313	13
-1	24	0	11.5	1.2	11.7	74	1
-1	26	0	9.6	1.1	10.9	326	1
-1	28	0	1.7	0.9	2.2	6	12
-1	30	0	4.5	0.6	4.9	291	31
-1	32	0	0.8	0.8	0.6	187	33
-2	2	0	12.8	1.5	12.3	262	1
-2	4	0	4.5	4.5	6.8	247	13
-2	6	0	10.5	0.8	11.2	296	21
-2	8	0	19.0	1.8	16.3	214	1
-2	10	0	10.5	1.7	10.4	250	12
-2	12	0	2.4	2.4	3.2	225	13
-2	14	0	3.1	3.1	6.3	3	13
-2	16	0	3.0	0.7	3.7	233	12
-2	18	0	6.3	1.0	5.4	241	12
-2	20	0	12.4	1.2	12.2	265	1
-2	22	0	7.5	3.0	7.5	344	12
-2	24	0	28.8	2.8	30.0	78	1
-2	26	0	7.7	0.8	7.4	332	1
-2	28	0	0.7	0.7	1.1	54	13
-2	30	0	4.4	0.4	3.7	272	1
-2	32	0	1.4	1.4	2.3	134	13
-2	34	0	3.9	0.6	4.0	80	11
-3	2	0	5.6	0.5	5.2	240	1
-3	4	0	4.8	2.0	5.8	21	12
-3	6	0	4.0	1.0	1.9	173	12
-3	8	0	5.3	1.6	4.9	235	12
-3	10	0	10.7	1.1	9.0	269	1
-3	12	0	7.3	0.8	6.5	352	1
-3	14	0	12.3	1.2	10.7	89	1
-3	16	0	2.1	1.0	2.2	35	12
-3	18	0	0.9	0.9	1.6	340	13
-3	20	0	4.2	0.4	3.8	303	1
-3	22	0	5.4	0.5	4.0	170	1
-3	24	0	28.0	2.4	23.3	95	1
-3	26	0	6.0	0.6	4.8	257	1
-3	28	0	2.5	0.6	3.3	273	12
-3	30	0	3.1	0.5	2.6	270	12
-3	32	0	0.7	0.7	1.2	175	33
-4	2	0	7.3	0.8	4.2	186	1
-4	4	0	4.3	0.5	3.5	318	1
-4	6	0	0.9	0.9	0.7	140	13
-4	8	0	0.9	0.9	0.7	329	13
-4	10	0	4.1	1.5	1.2	309	12
-4	12	0	2.4	0.4	1.9	321	21
-4	14	0	9.6	1.0	8.4	110	1
-4	16	0	1.7	0.9	1.4	216	2
-4	18	0	1.7	0.9	0.8	288	2
-4	20	0	2.5	0.6	2.1	282	12
-4	22	0	4.7	1.0	5.3	201	12
-4	24	0	7.5	0.9	7.5	103	1
-4	26	0	5.8	0.5	4.7	289	1
-4	28	0	2.7	0.2	2.0	308	1
-4	30	0	0.5	0.5	0.3	345	33
0	2	1	43.1	3.2	53.2	113	21
0	3	1	44.1	3.3	50.4	107	21
0	4	1	31.9	2.4	34.6	66	21
0	5	1	17.4	1.3	17.4	272	21
0	6	1	22.2	1.7	22.7	68	21
0	7	1	17.2	1.3	16.6	169	21
0	8	1	15.4	1.2	13.3	3	21
0	9	1	8.1	0.6	7.9	218	21
0	10	1	8.7	0.6	8.2	5	21
0	11	1	22.6	1.7	22.5	185	21
0	12	1	28.3	2.1	26.2	121	21
0	13	1	19.7	1.5	20.4	29	21
0	14	1	6.2	0.5	5.8	7	21
0	15	1	3.4	0.3	3.3	38	21
0	16	1	3.5	0.3	4.0	61	21
0	17	1	5.2	0.4	6.3	44	21
0	18	1	0.7	0.7	1.4	157	23
0	19	1	10.0	0.8	10.9	172	21
0	20	1	1.3	0.4	1.2	132	22
0	21	1	4.2	0.3	4.5	211	21
0	22	1	16.8	1.3	17.8	281	21
0	23	1	3.5	0.3	3.4	61	21
0	24	1	7.6	0.6	8.0	147	21
0	25	1	5.0	0.4	5.5	278	21
0	26	1	0.7	0.7	0.8	161	23
0	27	1	2.1	0.2	1.9	329	21
0	28	1	0.6	0.6	1.0	145	23
1	1	1	20.8	2.0	18.9	342	1
1	2	1	20.6	2.2	18.4	72	1
1	3	1	6.4	4.9	7.8	85	12
1	4	1	7.9	1.4	7.5	19	12
1	5	1	2.8	2.8	4.4	103	13
1	6	1	2.8	2.8	5.0	85	13
1	7	1	4.4	4.4	5.8	112	13
1	8	1	8.3	1.1	9.6	297	31
1	9	1	2.8	2.8	3.3	38	13
1	10	1	9.3	1.0	9.1	353	1
1	11	1	7.5	0.7	6.8	204	1
1	12	1	10.3	1.0	9.6	106	1
1	13	1	10.1	1.3	10.8	65	1
1	14	1	10.2	1.0	9.3	2	1
1	15	1	6.6	1.5	4.1	250	12
1	16	1	5.9	5.9	1.5	74	13
1	17	1	7.1	2.3	3.1	29	12
1	18	1	1.3	1.3	1.3	104	13
1	19	1	1.0	1.0	1.1	301	13
1	20	1	1.6	0.7	1.2	83	32
1	21	1	4.8	0.4	4.2	67	1
1	22	1	9.7	0.8	8.6	286	1
1	23	1	2.4	0.3	2.1	125	1
1	24	1	4.3	0.4	4.7	146	1
1	25	1	0.5	0.5	0.6	270	13
1	26	1	0.3	0.3	0.5	63	13
2	1	1	12.0	2.3	13.0	211	12
2	2	1	4.2	0.4	4.3	81	1
2	3	1	5.5	0.7	4.9	264	1
2	4	1	2.2	0.4	1.7	269	12
2	5	1	4.9	0.5	3.7	165	1
2	6	1	2.6	0.3	2.3	135	31
2	7	1	1.1	1.1	1.5	55	13
2	8	1	3.9	1.5	4.2	217	12
2	9	1	6.7	1.0	6.8	288	11
2	10	1	6.5	2.6	3.8	327	12
2	11	1	2.3	2.3	1.9	313	13
2	12	1	1.5	1.5	2.3	98	13
2	13	1	4.6	0.4	4.5	74	1
2	14	1	2.8	0.4	3.0	348	31
2	15	1	0.9	0.9	0.2	82	13
2	16	1	0.6	0.6	0.5	145	13
2	17	1	0.9	0.9	1.7	206	33
2	18	1	0.5	0.5	0.5	73	13
2	19	1	1.9	0.3	1.8	331	32
2	20	1	0.6	0.6	1.1	90	13
2	21	1	0.5	0.5	0.5	69	33
3	1	1	5.8	0.5	5.3	181	1
3	2	1	1.9	0.6	1.3	33	12
3	3	1	1.4	1.4	0.6	131	13
3	4	1	0.9	0.9	0.4	311	13
3	5	1	2.7	0.4	2.8	124	1
3	6	1	0.7	0.7	1.0	50	13
3	7	1	1.2	1.2	2.1	140	13
3	8	1	4.7	0.7	4.5	187	11
3	9	1	1.7	0.5	2.4	276	32
3	10	1	3.2	0.3	3.1	342	1
3	11	1	1.5	0.4	2.2	192	32
3	12	1	0.7	0.7	1.5	84	13
3	13	1	0.4	0.4	0.9	323	13
3	14	1	0.3	0.3	0.5	283	13
3	15	1	0.7	0.3	0.7	10	12
4	1	1	0.8	0.8	0.7	216	13
4	2	1	0.4	0.4	0.8	333	13
4	3	1	0.3	0.3	0.5	15	13
4	4	1	0.5	0.5	0.6	347	33
-1	0	1	21.1	2.8	17.9	25	31
-1	1	1	70.5	6.6	72.5	239	1
-1	2	1	66.4	6.8	79.7	151	1
-1	3	1	36.5	2.7	37.6	48	21
-1	4	1	34.2	9.4	22.6	91	12
-1	5	1	15.3	15.3	19.9	330	13
-1	6	1	21.1	2.8	19.3	92	31
-1	7	1	5.8	4.5	4.7	62	12
-1	8	1	13.2	2.7	12.1	58	12
-1	9	1	22.7	2.2	23.5	218	1
-1	10	1	10.6	1.2	12.3	231	1
-1	11	1	20.0	1.8	19.8	192	1
-1	12	1	12.8	1.2	11.2	162	1
-1	13	1	21.6	2.0	20.2	51	1
-1	14	1	8.2	1.1	7.0	230	31
-1	15	1	12.1	1.1	11.1	47	1
-1	16	1	4.7	1.3	4.4	19	12
-1	17	1	1.4	1.4	2.3	274	13
-1	18	1	4.1	1.7	1.9	281	12
-1	19	1	9.1	1.3	9.8	221	11
-1	20	1	2.8	2.2	5.1	326	12
-1	21	1	11.1	1.2	11.6	236	1
-1	22	1	25.4	2.4	24.9	285	1
-1	23	1	15.5	1.6	16.1	23	1
-1	24	1	5.8	0.6	6.1	144	1
-1	25	1	4.0	0.4	3.4	338	1
-1	26	1	1.6	0.7	1.6	143	32
-1	27	1	1.0	1.0	2.3	357	13
-1	28	1	0.0	0.5	0.8	122	14
-1	29	1	2.0	0.4	2.3	224	12
-1	30	1	0.0	0.5	0.5	219	14
-1	31	1	1.5	0.4	1.5	133	2
-1	32	1	1.5	0.3	1.4	351	2
-2	0	1	8.7	2.5	10.6	326	12
-2	1	1	21.7	2.6	22.2	233	1
-2	2	1	46.2	4.3	42.7	165	1
-2	3	1	21.8	2.4	22.7	35	1
-2	4	1	2.9	0.7	2.8	306	12
-2	5	1	19.3	1.7	16.2	74	1
-2	6	1	2.5	0.2	2.6	88	21
-2	7	1	3.1	3.1	0.8	156	13
-2	8	1	5.2	0.5	4.9	34	1
-2	9	1	13.6	2.9	12.6	210	12
-2	10	1	9.1	9.1	6.4	250	13
-2	11	1	17.2	2.8	16.1	235	12
-2	12	1	17.5	1.8	14.9	306	1
-2	13	1	17.2	1.9	17.3	68	1
-2	14	1	0.8	0.8	2.0	283	13
-2	15	1	5.2	0.7	6.1	37	11
-2	16	1	2.9	0.3	3.3	41	1
-2	17	1	2.7	0.7	3.4	10	12
-2	18	1	2.6	0.3	2.7	12	31
-2	19	1	5.8	0.5	5.9	308	1
-2	20	1	9.2	0.9	9.1	353	1
-2	21	1	7.4	0.9	8.0	172	1
-2	22	1	15.9	1.6	16.9	309	1
-2	23	1	12.9	1.2	11.5	36	1
-2	24	1	6.2	0.6	5.9	120	1
-2	25	1	0.8	0.8	1.9	77	13
-2	26	1	2.8	0.4	3.1	165	31
-2	27	1	3.2	0.3	3.2	206	1
-2	28	1	2.8	0.3	2.6	129	1
-2	29	1	0.9	0.9	1.4	248	33
-2	30	1	2.0	0.4	1.7	146	2
-2	31	1	1.9	0.3	1.8	178	32
-2	32	1	2.3	0.2	2.3	64	1
-2	33	1	0.5	0.5	0.7	30	33
-3	0	1	7.1	0.7	7.1	313	1
-3	1	1	4.7	0.5	5.1	166	1
-3	2	1	16.4	1.5	14.1	162	1
-3	3	1	3.9	0.4	3.6	127	1
-3	4	1	3.5	0.4	3.7	335	1
-3	5	1	2.9	1.6	2.8	92	12
-3	6	1	1.7	1.7	2.2	342	13
-3	7	1	2.3	1.8	2.9	249	12
-3	8	1	2.0	0.5	1.7	14	12
-3	9	1	1.9	0.3	1.5	354	21
-3	10	1	2.9	0.3	2.8	50	1
-3	11	1	8.3	0.8	7.9	218	1
-3	12	1	8.8	0.8	8.6	331	1
-3	13	1	8.7	0.8	8.2	65	1
-3	14	1	2.3	0.5	2.9	80	12
-3	15	1	1.0	1.0	1.1	113	13
-3	16	1	3.4	3.4	3.1	128	13
-3	17	1	2.4	0.7	1.3	79	12
-3	18	1	1.5	1.5	1.2	75	13
-3	19	1	0.5	0.5	1.0	274	13
-3	20	1	4.3	0.4	4.3	21	1
-3	21	1	7.5	0.7	7.4	169	1
-3	22	1	6.0	0.5	6.1	348	1
-3	23	1	1.4	0.7	1.6	343	12
-3	24	1	6.8	0.6	6.4	129	1
-3	25	1	5.7	0.5	5.6	312	1
-3	26	1	5.3	0.7	6.0	221	1
-3	27	1	1.7	0.5	2.2	186	32
-3	28	1	1.9	0.4	2.3	153	12
-3	29	1	1.5	0.5	1.4	16	32
-3	30	1	2.4	0.2	2.3	160	1
-3	31	1	1.6	0.3	1.4	165	2
-3	32	1	1.4	0.3	1.6	111	2
-3	33	1	1.3	0.2	1.3	314	1
-4	0	1	0.8	0.8	2.0	312	13
-4	1	1	0.8	0.8	1.3	188	13
-4	2	1	5.0	0.5	5.0	165	1
-4	3	1	0.7	0.7	1.6	181	13
-4	4	1	0.9	0.9	2.0	0	33
-4	5	1	0.9	0.9	1.9	301	33
-4	6	1	0.8	0.8	0.7	331	33
-4	7	1	0.8	0.8	1.2	18	33
-4	8	1	0.8	0.8	0.3	206	33
-4	9	1	0.9	0.9	1.2	45	33
-4	10	1	1.3	0.7	1.8	67	12
-4	11	1	4.4	0.4	4.5	191	1
-4	12	1	1.8	0.3	2.3	352	21
-4	13	1	0.0	0.5	0.7	37	14
-4	14	1	2.5	0.4	2.3	136	1
-4	15	1	0.9	0.9	2.0	318	33
-4	16	1	0.8	0.8	1.9	194	33
-4	17	1	0.8	0.8	0.4	239	33
-4	18	1	0.8	0.8	0.3	181	33
-4	19	1	0.9	0.9	0.9	266	33
-4	20	1	0.9	0.9	0.5	53	33
-4	21	1	1.9	0.4	1.8	191	2
-4	22	1	1.1	1.1	1.6	24	13
-4	23	1	2.4	0.3	2.4	7	1
-4	24	1	3.0	0.3	2.8	134	1
-4	25	1	4.5	0.4	4.8	329	1
-4	26	1	6.3	0.6	6.1	248	1
-4	27	1	2.6	0.2	2.8	123	1
-4	28	1	0.5	0.5	0.3	69	33
0	3	2	1.2	0.1	1.2	51	21
0	4	2	11.9	0.9	11.8	313	21
0	5	2	19.6	1.5	20.5	75	21
0	6	2	18.4	1.4	18.8	338	21
0	7	2	26.4	2.0	26.1	27	21
0	8	2	4.5	0.3	3.8	219	21
0	9	2	9.6	0.7	8.8	106	21
0	10	2	16.9	1.3	18.1	152	21
0	11	2	20.5	1.5	19.6	263	21
0	12	2	10.5	0.8	9.4	88	21
0	13	2	4.0	0.3	4.2	175	21
0	14	2	9.8	0.7	9.3	314	21
0	15	2	7.6	0.6	7.8	215	21
0	16	2	8.3	0.6	8.4	140	21
0	17	2	6.1	0.5	6.9	87	21
0	18	2	5.7	0.4	5.8	204	21
0	19	2	3.7	0.3	4.0	51	21
0	20	2	15.2	1.1	16.2	162	21
0	21	2	16.9	1.3	15.6	223	21
0	22	2	9.5	0.7	10.7	312	21
0	23	2	5.1	0.4	5.3	185	21
0	24	2	4.3	0.3	5.0	49	21
0	25	2	3.1	0.2	3.4	31	21
0	26	2	3.1	0.2	3.1	323	21
0	27	2	0.5	0.5	0.6	303	23
0	28	2	0.5	0.5	0.5	15	23
0	29	2	0.4	0.4	0.2	82	23
0	30	2	0.3	0.3	0.8	288	23
1	0	2	31.9	2.8	32.5	333	1
1	1	2	15.0	1.6	15.0	268	1
1	2	2	5.7	0.7	7.0	61	1
1	3	2	5.6	2.1	10.2	41	12
1	4	2	12.9	5.3	6.3	257	12
1	5	2	8.9	8.0	13.5	37	12
1	6	2	5.6	0.8	4.5	348	1
1	7	2	12.0	2.1	12.1	357	12
1	8	2	4.4	4.4	6.1	221	13
1	9	2	7.9	1.6	7.0	345	12
1	10	2	5.0	0.7	4.5	145	31
1	11	2	3.5	0.6	3.8	281	12
1	12	2	3.7	1.4	1.3	43	12
1	13	2	0.4	0.4	0.6	152	13
1	14	2	4.6	1.2	4.4	317	12
1	15	2	2.1	2.1	1.0	30	13
1	16	2	3.6	1.0	3.3	1	12
1	17	2	5.8	0.6	4.9	59	1
1	18	2	5.4	1.2	3.8	115	12
1	19	2	7.0	0.8	5.9	54	1
1	20	2	11.1	1.0	10.6	149	1
1	21	2	9.5	0.9	8.9	221	1
1	22	2	4.0	0.4	3.9	337	1
1	23	2	6.0	0.5	4.5	123	1
1	24	2	0.6	0.6	0.8	139	33
1	25	2	1.7	0.2	1.4	80	1
1	26	2	1.7	0.1	1.5	308	1
2	0	2	20.2	1.9	20.1	321	1
2	1	2	10.4	2.0	11.5	290	12
2	2	2	0.0	0.5	1.3	288	14
2	3	2	1.9	1.9	0.6	228	13
2	4	2	1.8	0.3	2.3	319	32
2	5	2	3.6	0.6	2.9	23	12
2	6	2	0.7	0.7	0.8	34	33
2	7	2	2.7	0.3	3.2	233	1
2	8	2	1.2	1.2	2.1	186	13
2	9	2	3.0	0.5	3.6	2	12
2	10	2	4.5	0.4	4.0	332	1
2	11	2	6.1	0.8	6.7	247	31
2	12	2	3.7	0.8	3.6	97	12
2	13	2	3.2	0.3	3.4	312	1
2	14	2	3.1	0.3	2.5	214	1
2	15	2	0.7	0.7	0.8	24	33
2	16	2	1.9	0.3	2.6	330	12
2	17	2	1.5	0.6	1.6	30	12
2	18	2	0.4	0.4	1.0	217	13
2	19	2	2.8	0.3	2.6	26	1
2	20	2	2.8	0.2	2.9	119	1
2	21	2	3.4	0.3	3.9	249	1
3	0	2	3.0	0.3	3.1	316	1
3	1	2	2.6	0.3	2.4	315	1
3	2	2	1.6	0.5	1.8	213	32
3	3	2	1.8	0.4	1.9	201	12
3	4	2	0.7	0.7	0.4	167	33
3	5	2	0.4	0.4	0.6	57	13
3	6	2	0.8	0.8	1.2	125	13
3	7	2	2.5	0.3	2.2	139	1
3	8	2	3.3	0.4	3.7	203	1
3	9	2	3.2	0.3	3.3	78	1
3	10	2	6.2	0.6	5.8	324	1
3	11	2	4.8	0.4	5.1	239	1
3	12	2	1.8	0.2	2.1	105	1
3	13	2	0.0	0.5	0.7	300	14
4	0	2	0.9	0.9	0.5	8	13
4	1	2	0.5	0.5	1.0	340	33
4	2	2	0.0	0.5	0.2	180	14
4	3	2	0.9	0.3	0.5	226	32
-1	1	2	10.3	1.0	9.9	107	1
-1	2	2	10.2	2.2	10.7	208	12
-1	3	2	8.7	0.7	9.7	305	21
-1	4	2	16.5	1.8	20.3	2	1
-1	5	2	2.5	2.5	3.2	128	13
-1	6	2	3.1	3.1	3.7	163	13
-1	7	2	8.0	2.1	9.6	37	12
-1	8	2	4.4	0.8	3.5	250	12
-1	9	2	13.1	1.3	12.3	83	1
-1	10	2	32.4	3.2	34.3	170	1
-1	11	2	34.4	3.3	31.7	272	1
-1	12	2	5.5	0.8	7.5	82	11
-1	13	2	16.3	1.9	15.6	338	1
-1	14	2	21.7	2.0	18.9	276	1
-1	15	2	12.1	1.3	11.8	207	1
-1	16	2	5.0	0.7	5.1	156	31
-1	17	2	4.4	0.5	4.1	110	1
-1	18	2	8.7	0.9	8.8	261	1
-1	19	2	1.9	0.3	2.2	5	2
-1	20	2	8.6	0.9	7.9	130	1
-1	21	2	11.3	1.0	11.4	256	1
-1	22	2	7.2	0.7	7.4	319	1
-1	23	2	3.0	0.8	4.1	179	12
-1	24	2	9.0	1.0	10.4	61	1
-1	25	2	5.5	0.9	6.0	17	12
-1	26	2	2.5	2.5	0.9	333	13
-1	27	2	1.6	1.6	0.8	88	13
-1	28	2	0.6	0.6	1.3	354	13
-1	29	2	0.5	0.5	1.9	83	13

Table 1 (*cont.*)

H	K	L	FOBS	ESD	FCAL	ANG	T
-1	30	2	0.6	0.6	0.4	317	33
-1	31	2	0.0	0.5	0.8	5	14
-1	32	2	0.0	0.5	0.9	255	14
-2	1	2	11.1	1.1	10.8	26	1
-2	2	2	21.0	2.6	21.9	151	1
-2	3	2	18.6	1.9	18.4	32	1
-2	4	2	14.3	1.9	15.7	290	1
-2	5	2	4.1	0.4	5.0	225	1
-2	6	2	8.0	0.6	8.9	198	21
-2	7	2	1.9	0.6	2.5	168	12
-2	8	2	13.6	1.9	13.1	287	11
-2	9	2	8.8	2.7	10.3	49	12
-2	10	2	17.7	2.8	16.5	165	12
-2	11	2	5.8	1.6	7.6	295	12
-2	12	2	4.5	4.5	6.5	7	13
-2	13	2	15.1	1.7	14.1	0	1
-2	14	2	14.8	1.3	12.9	292	1
-2	15	2	7.8	0.7	7.0	172	1
-2	16	2	1.8	0.3	2.0	72	32
-2	17	2	4.6	0.5	4.2	200	1
-2	18	2	0.7	0.7	1.0	73	33
-2	19	2	2.0	0.3	1.7	113	12
-2	20	2	5.3	0.5	4.7	107	1
-2	21	2	3.8	0.4	4.3	284	1
-2	22	2	1.0	1.0	1.1	184	13
-2	23	2	6.1	0.8	6.0	118	31
-2	24	2	5.9	0.6	5.3	84	1
-2	25	2	1.9	0.4	1.7	9	12
-2	26	2	1.9	0.4	1.8	326	12
-2	27	2	0.7	0.7	0.9	156	33
-2	28	2	2.6	0.3	2.3	352	1
-2	29	2	3.5	0.3	3.0	116	1
-2	30	2	1.6	0.3	1.6	46	2
-2	31	2	2.5	0.4	2.6	102	11
-2	32	2	0.3	0.3	0.7	258	13
-2	33	2	0.0	0.5	1.3	123	14
-3	1	2	4.5	0.5	3.8	84	1
-3	2	2	9.5	0.9	8.8	152	1
-3	3	2	11.8	1.2	12.6	48	1
-3	4	2	12.1	1.3	13.5	287	1
-3	5	2	7.7	0.7	6.7	198	1
-3	6	2	2.2	0.4	2.8	333	12
-3	7	2	4.1	0.3	4.0	195	1
-3	8	2	2.7	0.7	3.1	313	12
-3	9	2	2.3	0.2	2.1	59	21
-3	10	2	4.9	0.5	5.6	132	1
-3	11	2	0.0	0.5	0.4	76	14
-3	12	2	3.4	0.4	3.3	49	1
-3	13	2	1.4	1.4	2.0	43	13
-3	14	2	7.7	1.6	4.5	301	12
-3	15	2	5.7	1.7	3.7	205	12
-3	16	2	1.9	1.9	1.8	144	13
-3	17	2	2.6	0.6	3.2	239	12
-3	18	2	1.4	0.6	1.7	112	12
-3	19	2	1.8	0.4	1.7	180	12
-3	20	2	1.6	0.5	1.2	228	32
-3	21	2	2.6	0.3	2.4	237	1
-3	22	2	0.7	0.7	0.8	351	33
-3	23	2	2.0	0.4	2.0	109	2
-3	24	2	4.0	0.4	4.0	104	1
-3	25	2	0.7	0.7	1.1	250	13
-3	26	2	2.3	0.4	2.0	335	12
-3	27	2	0.6	0.6	0.9	281	13
-3	28	2	1.2	1.2	1.9	67	13
-3	29	2	1.0	1.0	1.1	124	13
-3	30	2	0.0	0.5	1.0	96	14
-3	31	2	1.9	0.2	1.6	134	1
-4	1	2	3.0	0.3	2.6	153	1
-4	2	2	2.3	0.2	1.9	123	1
-4	3	2	4.2	0.4	3.8	31	1
-4	4	2	4.7	0.4	4.3	312	1
-4	5	2	3.9	0.3	3.5	219	1
-4	6	2	1.0	0.5	1.0	49	2
-4	7	2	2.2	0.3	1.8	208	1
-4	8	2	0.8	0.8	1.6	145	33
-4	9	2	0.5	0.5	0.6	237	13
-4	10	2	0.8	0.8	1.7	202	33
-4	11	2	1.4	0.4	1.9	295	32
-4	12	2	2.1	0.2	2.2	27	21
-4	13	2	1.4	0.4	1.6	28	32
-4	14	2	0.8	0.8	0.2	293	33
-4	15	2	2.2	0.3	2.2	241	1
-4	16	2	0.8	0.8	0.6	198	33
-4	17	2	0.0	0.5	1.0	325	14
-4	18	2	0.6	0.6	0.4	64	13
-4	19	2	0.5	0.5	0.4	209	13
-4	20	2	0.8	0.8	1.0	306	13
-4	21	2	0.5	0.5	1.1	263	13
-4	22	2	1.1	0.4	1.4	49	12
-4	23	2	1.3	0.5	1.9	341	12
-4	24	2	4.4	0.4	4.1	125	1
-4	25	2	0.5	0.5	0.7	50	33
-4	26	2	2.4	0.2	2.3	243	1
-4	27	2	0.9	0.3	1.2	102	32
0	4	3	5.4	0.4	5.8	319	21
0	5	3	3.3	0.2	3.4	52	21
0	6	3	7.4	0.6	6.9	163	21
0	7	3	10.4	0.8	9.6	148	21
0	10	3	13.9	1.0	12.7	207	21
0	11	3	9.8	0.7	10.0	164	21
0	12	3	4.6	0.3	4.7	175	21
0	13	3	3.3	0.2	3.7	71	21
0	14	3	3.6	0.3	3.7	302	21
0	15	3	3.4	0.3	3.8	103	21
0	16	3	0.5	0.5	1.5	267	23
0	17	3	3.7	0.3	3.5	210	21
0	18	3	0.5	0.5	0.7	43	23
0	19	3	4.4	0.3	4.7	78	21
0	20	3	2.6	0.2	2.4	219	21
0	21	3	2.3	0.2	2.3	223	21
0	22	3	2.6	0.2	2.6	281	21
0	23	3	2.8	0.2	3.0	323	21
0	24	3	2.7	0.2	2.7	314	21
0	25	3	2.0	0.2	1.9	268	21
0	26	3	1.5	0.2	1.4	268	21
0	27	3	1.7	0.2	1.7	284	21

H	K	L	FOBS	ESD	FCAL	ANG	T
0	28	3	0.3	0.3	0.5	207	23
1	0	3	17.9	1.7	18.5	31	1
1	2	3	19.3	1.9	19.7	175	1
1	3	3	1.6	1.6	1.6	226	13
1	4	3	6.6	0.6	5.4	78	1
1	5	3	8.3	1.0	8.5	284	1
1	6	3	4.6	0.5	4.4	349	1
1	7	3	6.0	0.6	5.6	298	1
1	8	3	18.7	1.7	16.9	29	1
1	9	3	10.7	1.1	10.8	87	1
1	10	3	9.5	0.9	8.7	246	1
1	11	3	5.4	1.2	6.5	212	12
1	12	3	15.6	1.5	15.6	127	1
1	13	3	11.2	1.0	10.7	19	1
1	14	3	3.9	0.4	3.3	256	1
1	15	3	4.5	0.4	4.6	34	1
1	16	3	1.4	1.4	2.5	18	13
1	17	3	1.2	0.8	0.8	217	12
1	18	3	4.6	0.4	4.0	9	1
1	19	3	10.5	1.0	9.6	91	1
1	20	3	5.3	0.5	5.6	160	1
1	21	3	3.0	0.3	3.1	221	1
1	22	3	4.0	0.3	3.6	203	1
1	23	3	1.5	0.4	1.1	327	12
1	24	3	1.8	0.2	1.5	315	31
1	25	3	0.5	0.5	1.2	228	33
2	0	3	2.7	0.4	2.2	301	31
2	1	3	7.1	0.9	7.4	221	1
2	2	3	4.1	0.7	3.3	181	12
2	3	3	4.4	1.2	3.3	183	12
2	4	3	2.1	0.4	2.2	99	32
2	5	3	1.5	1.5	2.2	284	13
2	6	3	3.0	0.3	3.1	336	1
2	7	3	0.8	0.8	0.9	261	13
2	8	3	8.5	0.8	6.6	337	1
2	9	3	4.7	0.4	4.0	119	1
2	10	3	6.3	0.6	6.3	299	1
2	11	3	8.8	0.8	8.8	226	1
2	12	3	12.1	1.1	10.7	124	1
2	13	3	5.2	0.6	5.4	15	1
2	14	3	1.5	1.5	2.7	313	13
2	15	3	3.6	0.3	3.6	291	1
2	16	3	1.4	0.5	1.8	5	2
2	17	3	2.9	0.3	2.8	324	1
2	18	3	5.3	0.5	4.9	357	1
2	19	3	5.6	0.5	5.7	81	1
2	20	3	2.8	0.5	3.6	158	12
3	0	3	3.9	0.3	3.6	241	1
3	1	3	2.5	0.4	2.1	204	1
3	2	3	0.8	0.8	1.2	190	33
3	3	3	0.8	0.8	0.5	164	13
3	4	3	0.4	0.4	0.3	138	13
3	5	3	0.0	0.5	0.6	339	14
3	6	3	0.9	0.9	1.2	124	13
3	7	3	1.2	0.6	1.0	62	12
3	8	3	2.4	0.3	2.6	297	31
3	9	3	1.1	0.5	1.7	122	12
3	10	3	2.7	0.3	2.9	278	1
3	11	3	3.0	0.3	3.1	207	1
3	12	3	3.4	0.3	3.6	139	1
-1	1	3	6.4	0.8	7.5	215	1
-1	2	3	12.6	1.2	14.9	95	1
-1	3	3	6.0	0.4	5.5	105	21
-1	4	3	3.5	2.7	5.5	338	12
-1	5	3	5.2	0.8	5.2	107	12
-1	6	3	6.7	3.0	6.0	174	12
-1	8	3	7.2	3.3	5.5	343	12
-1	9	3	12.8	1.1	12.0	129	1
-1	10	3	2.3	1.2	4.5	99	12
-1	11	3	6.4	1.1	5.1	216	12
-1	12	3	2.1	0.7	1.6	237	12
-1	13	3	1.4	1.4	2.3	38	13
-1	14	3	3.9	3.9	8.6	350	13
-1	15	3	5.2	1.6	1.4	117	12
-1	16	3	4.0	1.5	3.4	196	12
-1	17	3	9.1	1.0	7.5	26	1
-1	18	3	3.7	3.7	0.6	231	13
-1	19	3	0.4	0.4	1.5	69	13
-1	20	3	0.8	0.8	0.4	342	13
-1	21	3	0.5	0.5	0.8	117	13
-1	22	3	7.1	0.6	6.3	1	1
-1	23	3	1.9	0.5	2.0	336	2
-1	24	3	0.9	0.9	2.0	105	13
-1	25	3	1.9	1.9	4.3	303	13
-1	26	3	2.9	0.7	3.8	254	12
-1	27	3	1.9	0.4	2.2	284	12
-1	28	3	0.3	0.3	1.1	141	13
-1	29	3	2.2	0.2	2.1	340	1
-1	30	3	1.8	0.2	2.1	87	31
-1	31	3	0.5	0.5	0.4	36	33
-2	1	3	3.7	0.5	4.0	275	1
-2	2	3	5.7	0.5	6.1	104	1
-2	3	3	4.2	0.5	3.7	118	1
-2	4	3	3.5	0.4	3.1	69	1
-2	5	3	2.3	0.3	2.2	283	1
-2	6	3	1.3	0.3	1.8	163	22
-2	7	3	1.6	0.4	2.4	240	12
-2	8	3	2.1	0.5	2.2	201	12
-2	9	3	2.6	0.6	3.2	122	12
-2	10	3	1.8	1.1	2.7	89	12
-2	11	3	3.1	0.5	3.3	3	12
-2	12	3	2.7	0.4	2.7	143	31
-2	13	3	6.5	0.9	6.1	74	11
-2	14	3	5.1	0.6	4.7	291	1
-2	15	3	8.5	0.9	9.0	145	1
-2	16	3	2.1	0.4	2.1	90	32
-2	17	3	7.0	0.6	7.0	37	1
-2	18	3	2.3	0.4	1.9	240	12
-2	19	3	2.0	0.4	2.7	191	2
-2	20	3	1.6	1.6	2.4	122	13
-2	21	3	0.9	0.9	2.3	252	13
-2	22	3	2.3	1.1	3.8	333	12
-2	23	3	1.7	1.7	3.5	17	13
-2	24	3	1.6	1.6	4.2	104	13
-2	25	3	4.7	0.7	5.3	314	11
-2	26	3	6.2	0.6	6.0	249	1

H	K	L	FOBS	ESD	FCAL	ANG	T
-2	27	3	0.8	0.8	1.0	99	13
-2	28	3	0.7	0.7	0.9	123	13
-2	29	3	0.8	0.8	1.4	33	13
-2	30	3	0.4	0.4	1.6	132	13
-2	31	3	0.6	0.6	0.8	57	13
-2	32	3	3.9	0.5	4.2	108	1
-3	1	3	2.8	0.3	2.6	322	1
-3	2	3	5.1	0.7	5.4	210	1
-3	3	3	5.0	0.8	5.1	68	12
-3	4	3	4.1	0.4	3.6	303	1
-3	5	3	3.9	0.5	4.5	174	31
-3	6	3	1.5	1.1	2.2	83	12
-3	7	3	3.8	0.4	3.1	84	1
-3	8	3	1.9	0.5	1.9	154	12
-3	9	3	5.5	0.4	5.5	169	21
-3	10	3	2.8	0.3	2.8	221	1
-3	11	3	2.9	0.5	3.7	274	12
-3	12	3	5.2	0.5	5.3	314	1
-3	13	3	3.9	0.5	3.4	75	31
-3	14	3	1.5	0.7	3.0	242	12
-3	15	3	6.3	0.8	6.9	174	1
-3	16	3	4.9	0.7	4.2	56	11
-3	17	3	1.9	0.5	2.6	346	12
-3	18	3	0.5	0.5	0.9	175	13
-3	19	3	1.3	0.7	2.5	266	12
-3	20	3	0.8	0.8	0.2	202	33
-3	21	3	1.6	0.5	1.4	275	2
-3	22	3	3.7	0.3	3.4	290	1
-3	23	3	6.0	0.5	5.0	35	1
-3	24	3	3.4	0.3	3.1	62	1
-3	25	3	2.2	0.3	2.2	301	1
-3	26	3	2.5	0.3	2.9	243	31
-3	27	3	2.0	0.3	2.2	154	1
-3	28	3	0.7	0.7	0.7	79	13
-3	29	3	0.4	0.4	0.7	143	13
-3	30	3	0.7	0.7	0.9	187	13
-4	1	3	0.6	0.6	0.8	300	13
-4	2	3	2.2	0.2	2.3	253	1
-4	3	3	2.3	0.2	2.3	70	1
-4	4	3	3.5	0.3	3.7	306	1
-4	5	3	4.8	0.4	5.0	185	1
-4	6	3	3.3	0.3	3.4	73	1
-4	7	3	0.8	0.8	0.9	305	33
-4	8	3	0.7	0.7	1.5	131	13
-4	9	3	2.2	0.3	1.7	227	31
-4	10	3	1.7	0.4	1.3	220	2
-4	11	3	3.5	0.5	4.1	274	31
-4	12	3	5.8	0.4	5.8	325	21
-4	13	3	3.5	0.5	3.9	36	31
-4	14	3	0.6	0.6	1.5	127	13
-4	15	3	2.2	0.3	2.4	173	31
-4	16	3	2.5	0.2	2.9	95	1
-4	17	3	0.8	0.8	1.6	342	33
-4	18	3	0.6	0.6	0.8	157	13
-4	19	3	0.6	0.6	1.2	292	13
-4	20	3	0.3	0.3	0.9	316	13
-4	21	3	0.3	0.3	0.4	143	13
-4	22	3	0.3	0.3	1.1	324	13
-4	23	3	2.4	0.2	2.7	41	1
0	5	4	7.1	0.5	6.4	3	21
0	6	4	7.8	0.6	7.3	199	21
0	7	4	4.4	0.3	4.3	248	21
0	8	4	3.1	0.2	3.1	227	21
0	9	4	1.9	0.1	2.0	356	21
0	10	4	7.7	0.6	6.9	163	21
0	11	4	2.4	0.2	2.3	148	21
0	12	4	9.3	0.7	8.7	104	21
0	13	4	20.2	1.5	19.1	358	21
0	14	4	15.7	1.2	15.8	276	21
0	15	4	11.4	0.9	10.3	194	21
0	16	4	3.9	0.3	3.9	352	21
0	17	4	2.4	0.2	2.5	157	21
0	18	4	1.7	0.3	1.5	274	21
0	19	4	2.5	0.2	2.3	127	21
0	20	4	1.7	0.3	1.9	131	21
0	21	4	3.0	0.2	3.1	242	21
0	22	4	2.0	0.2	2.1	338	21
0	23	4	3.1	0.2	2.9	88	21
0	24	4	0.6	0.6	0.8	254	23
0	25	4	3.0	0.2	3.0	203	21
0	26	4	0.5	0.5	0.3	235	23
0	27	4	1.1	0.2	1.1	226	21
1	0	4	2.2	0.6	2.5	74	12
1	1	4	4.9	0.6	4.2	60	1
1	2	4	3.5	0.3	3.5	83	1
1	3	4	7.3	0.7	6.7	316	1
1	4	4	5.3	0.7	5.0	3	31
1	5	4	3.3	0.3	3.3	60	1
1	6	4	2.7	2.7	6.2	235	13
1	7	4	8.5	1.1	8.6	299	31
1	8	4	5.7	0.9	5.6	32	12
1	9	4	4.0	1.3	4.1	133	12
1	10	4	9.2	1.0	9.4	90	1
1	11	4	2.7	0.6	2.6	221	12
1	12	4	10.9	1.0	9.6	114	1
1	13	4	14.6	1.3	12.8	359	1
1	14	4	9.6	0.9	8.2	280	1
1	15	4	7.1	1.0	6.8	213	11
1	16	4	4.5	0.5	4.1	157	1
1	17	4	2.1	0.4	1.8	68	32
1	18	4	0.7	0.7	1.4	268	13
1	19	4	1.7	0.4	1.8	121	12
1	20	4	3.0	0.3	3.2	129	1
1	21	4	1.5	0.3	0.9	154	2
1	23	4	0.5	0.5	1.1	32	3
1	24	4	0.6	0.6	1.9	232	13
2	0	4	0.0	0.5	0.7	46	14
2	1	4	4.1	0.4	4.2	123	1
2	2	4	1.4	0.5	1.1	94	12
2	3	4	0.8	0.8	0.7	197	13
2	4	4	2.0	0.4	2.2	266	32
2	5	4	1.3	0.6	2.3	175	12
2	6	4	5.7	0.5	5.4	245	1
2	7	4	6.8	0.6	7.2	316	1
2	8	4	5.2	0.5	4.6	359	1
2	9	4	2.0	2.0	3.1	165	13

H	K	L	FOBS	ESD	FCAL	ANG	T
2	10	4	2.4	0.5	3.0	58	12
2	11	4	0.7	0.7	1.3	130	13
2	12	4	1.6	0.5	2.1	58	32
2	13	4	3.9	0.4	3.5	352	1
2	14	4	3.5	0.3	3.6	316	1
2	15	4	3.5	0.3	3.2	236	1
2	16	4	2.5	0.3	2.3	168	1
2	17	4	2.2	0.2	2.1	331	1
2	18	4	3.1	0.2	3.0	13	1
3	0	4	1.6	0.5	2.2	166	32
3	1	4	1.9	0.4	1.6	80	2
3	2	4	1.2	0.6	0.7	181	2
3	3	4	1.6	0.6	2.2	119	12
3	4	4	0.7	0.7	0.8	257	13
3	5	4	1.1	0.6	1.2	168	2
3	6	4	1.7	0.3	2.1	220	2
3	7	4	2.3	0.3	2.6	302	1
3	8	4	0.3	0.3	1.0	45	13
3	9	4	0.6	0.6	0.8	202	13
3	10	4	0.3	0.3	0.9	217	13
3	11	4	1.6	0.1	1.7	119	1
-1	1	4	2.6	0.4	2.7	308	12
-1	2	4	8.0	1.3	8.2	148	12
-1	3	4	21.1	1.6	22.0	34	21
-1	4	4	10.0	1.2	10.0	299	1
-1	5	4	3.1	0.4	3.7	300	1
-1	6	4	4.0	0.4	3.7	353	1
-1	7	4	3.8	0.6	3.9	67	12
-1	8	4	1.3	0.6	1.5	1	12
-1	9	4	3.6	1.1	1.1	88	12
-1	10	4	5.2	0.7	4.8	207	31
-1	11	4	0.7	0.7	1.0	169	13
-1	12	4	12.8	1.2	12.0	41	1
-1	13	4	8.5	1.1	9.2	3	31
-1	14	4	7.2	0.9	7.1	290	1
-1	15	4	8.7	0.9	7.6	209	1
-1	16	4	6.7	1.2	5.2	34	12
-1	17	4	4.4	0.5	3.6	179	1
-1	18	4	2.3	2.3	2.9	100	13
-1	19	4	2.7	2.7	1.4	74	13
-1	20	4	1.8	1.8	3.1	151	13
-1	21	4	7.6	1.0	7.5	225	1
-1	22	4	2.0	0.7	2.8	327	12
-1	23	4	2.1	0.4	2.0	76	32
-1	24	4	1.2	0.6	0.7	168	2
-1	25	4	2.4	0.3	2.2	205	1
-1	26	4	0.8	0.8	0.8	87	13
-1	30	4	2.9	0.3	2.4	338	1
-2	1	4	5.6	1.4	5.5	165	12
-2	2	4	2.5	2.5	3.3	93	13
-2	3	4	10.3	1.0	9.7	23	1
-2	4	4	8.2	0.9	5.0	310	1
-2	5	4	1.0	1.0	1.4	231	13
-2	6	4	3.9	0.3	4.0	37	21
-2	7	4	5.2	0.5	4.8	71	1
-2	8	4	5.8	1.1	7.9	95	12
-2	9	4	4.2	1.6	6.4	112	12
-2	10	4	10.5	1.0	11.6	171	1
-2	11	4	10.3	1.1	10.6	258	1
-2	12	4	7.9	0.7	7.1	20	1
-2	13	4	2.6	0.6	2.8	33	12
-2	14	4	2.9	0.3	3.0	267	1
-2	15	4	4.1	0.4	4.1	239	1
-2	16	4	2.0	0.4	2.5	145	32
-2	17	4	2.3	0.3	2.4	102	1
-2	18	4	1.3	1.3	2.5	190	13
-2	19	4	1.2	1.2	1.0	262	13
-2	20	4	2.2	0.8	2.1	149	12
-2	21	4	8.3	0.8	7.2	228	1
-2	22	4	4.1	0.4	3.9	320	1
-2	23	4	1.9	0.4	1.6	310	2
-2	24	4	1.6	0.5	1.5	165	32
-2	25	4	0.8	0.8	1.3	58	13
-2	26	4	0.6	0.6	1.0	186	13
-2	27	4	0.7	0.7	1.5	60	13
-2	28	4	1.5	0.3	1.6	94	12
-3	1	4	2.5	0.3	2.0	187	1
-3	2	4	2.0	0.4	1.5	97	2
-3	3	4	2.8	0.3	2.2	19	1
-3	4	4	0.9	0.9	2.0	310	13
-3	5	4	2.1	0.4	1.0	228	32
-3	6	4	1.2	1.2	1.4	177	13
-3	7	4	2.1	2.1	2.5	109	13
-3	8	4	1.6	1.6	1.2	168	13
-3	9	4	5.1	0.4	4.3	93	21
-3	10	4	9.7	0.9	9.1	180	1
-3	11	4	10.6	1.0	10.1	261	1
-3	12	4	6.7	0.6	6.1	327	1
-3	13	4	2.1	0.4	2.5	34	32
-3	14	4	4.3	0.4	4.5	325	1
-3	15	4	0.8	0.8	1.5	177	13
-3	16	4	0.7	0.7	1.2	73	13
-3	17	4	0.5	0.5	1.6	35	13
-3	18	4	1.6	0.5	2.2	220	32
-3	19	4	0.7	0.7	1.4	241	13
-3	20	4	0.6	0.6	1.3	96	13
-3	21	4	3.9	0.4	3.2	262	1
-3	22	4	2.2	0.3	2.4	331	1
-3	23	4	0.6	0.6	1.1	337	13
-3	24	4	0.4	0.4	0.8	182	13
-3	25	4	1.8	0.3	1.6	50	12
-3	26	4	1.6	0.3	1.6	238	2
-3	27	4	3.0	0.3	2.6	102	1
-3	28	4	1.9	0.2	1.7	33	1
-3	29	4	0.0	0.5	0.5	94	14
-4	6	4	0.6	0.6	0.8	77	3
-4	8	4	1.6	0.3	1.9	287	12
-4	9	4	1.2	0.3	1.7	60	32
-4	10	4	1.9	0.3	2.5	161	12
-4	11	4	3.6	0.3	4.1	269	1
-4	13	4	0.4	0.4	1.1	72	13
-4	14	4	2.3	0.3	2.3	4	1
-4	15	4	1.2	0.4	1.1	137	32
-4	16	4	2.1	0.2	2.1	81	1

not be extended to cover this case. These considerations led to the use of equations (16).

It was convenient to include all these calculations as well as those correcting the intensity readings, into a single data reduction program written in FORTRAN. Special provision was made for the $0kl$ and $h,-3h,l$ reflexions which are not affected by twinning in Para Red, the former because by symmetry they are equal in intensity to the related reflexion (with or without twinning), and the latter because they are self-overlapping, since they lie on the mirror-plane. Their errors were calculated simply by use of $\sigma_c=0{\cdot}15I_o$, *etc.* instead of (11).

6. *Results for Para Red*

The structure of Para Red is reported in full elsewhere (Grainger & McConnell, 1969*b*) but the final structure factor table is given in Table 1. The $|F_o|$ and their e.s.d.'s are listed together with a two-digit code number in the column headed 'T'. The second digit indicates by 1 to 4 which of the above procedures (i) to (iv) was used in computing the $|F_o|$ and the e.s.d. for that reflexion. The first digit indicates by 0, 1, 2, or 3 to which of the groups labelled S, W, U or E the reflexion belongs: these contain respectively, reflexions which are stronger (S) than the related reflexion, reflexions which are weaker (W) than the related reflexion, reflexions which are unaffected (U) by twinning, in either of the ways described above, and reflexions which are accidentally equal (E) to the related reflexion (of which there are surprisingly many). Thus 'T' code numbers

1,2,3,4 indicate groups S(i) to S(iv),
11,12,13,14 indicate groups W(i) to W(iv),
21,22,23,24 indicate groups U(i) to U(iv),
31,32,33,34 indicate groups E(i) to E(iv).

(i) *Agreement between observed and calculated structure amplitudes*

Table 2 lists several quantities pertaining to the various groups mentioned above. In columns 2 and 3 appear the number of reflexions and the mean $|F_o|$: these are guides to the importance of the group.

Table 2. *Agreement between 'observed' and calculated structure amplitudes*

Group	Number in group	Mean $\|F_o\|$	R	Reliability $=\frac{\Sigma\sigma_F}{\Sigma\|F_o\|}$
All data	926	5·7	0·13	0·17
S	346	8·2	0·09	0·10
W	341	3·1	0·30	0·44
U	133	8·2	0·07	0·08
E	106	2·5	0·18	0·26
(i)	479	8·7	0·08	0·10
(ii)	212	4·0	0·22	0·29
(iii)	218	1·2	0·58	1·00
(iv)	17	0·0	1·00*	0·68*

* Based on $\Sigma\,|F_c|$.

Column 4 gives the R value, $\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|$, which is 0·13 for the whole of the data, but lies in the range 0·07 to 0·09 for the S, U and (i) groups. For other groups it ranges as high as 0·58, excluding one small group.

It is interesting to compare these figures with the e.s.d.'s of the 'observed' $|F_o|$ within each group. Were it not for the twinning, the e.s.d. of most reflexions would have been 7·5% of the structure amplitude, since a general 15% error in intensity readings was assumed except for weak readings. A perusal of the values in Table 1 reveals that for some groups the estimated fractional error is frequently much larger than this. A quantitative index of the expected reliability of the $|F_o|$ for each group has been calculated from the e.s.d.'s and $|F_o|$ of Table 1 and this appears in the last column of Table 2. According to this measure the S and U data are expected to be quite reliable (0·10 and 0·08), the E group rather unreliable (0·26), due partly to a preponderance of weak reflexions, and the W reflexions very unreliable indeed (0·44). These figures show considerable quantitative agreement with the corresponding R values. It is interesting to note that without the W and E reflexions R is 0·083, which gives some idea of the intrinsic accuracy of the raw data.

For the groups (i) to (iv), where the grouping is based on the different mathematical methods described in section (5), a similar situation prevails: groups (ii), (iii) and (iv) are expected to be relatively unreliable and this is reflected also in the R values obtained. The reason these reflexions do not greatly inflate the overall R, although comprising over half the total number, is that they are in general rather weak (column 3).

From all this one draws the conclusion that the agreement between the observed and calculated structure amplitudes is good where expected and poor where expected. Because the data contain many reflexions of low accuracy, the overall residual is comparatively high. It does not necessarily follow that this has affected the accuracy of the final parameters, for if the data are correctly weighted this should not happen.

(ii) *Weighting analysis*

The final results were also analysed to check on the effectiveness of the methods of section (5) in estimating standard deviations for the 'observed' structure amplitudes. The usual means of evaluating the success or otherwise of a weighting scheme is to determine whether the mean $w\Delta^2=(|F_o|-|F_c|)^2/\sigma_F^2$ is the same for all groups of data (Cruickshank, 1961, p.45). It is customary, for normal data, to test groups of data having increasing $|F_o|$ or $(\sin\theta)/\lambda$. In the present case there is little point in testing these: the first bears no simple relationship to the original raw readings except for the U and E groups, and in any case it is much more likely that discrepancies will occur between the S, W, U and E groups or groups (i) to (iv). The mean $w\Delta^2$ has been calculated for all combinations of these groups, and are shown in Table 3. They range from 0·44 to 2·51.

The structure amplitudes for the U group do not involve the combination of two raw readings, and are not affected by the twin fraction or its error. Consequently these will tend to reflect any general over- or under-estimate in the assumed standard deviations of the raw readings. That this has not happened is shown by the value for the whole group which is just 1·00. For the S groups the values are not far from unity, and the same is true for the W groups, except for the small group W(iv). The E group is the worst, mainly due to the E(iii) sub-group.

Examination of the Table to check on the error estimates for groups (i) to (iv) reveals more significant differences. The results for groups (i) and (ii) are very satisfactory. In group (iii) the errors appear to have been slightly over-estimated by equation (15*b*). The arbitrary error given to the 17 reflexions in group (iv) appears to be too low: the mistake, however, is really in the value of $|F_o|$ which was set at zero, but in every case $|F_c|$ has turned out to be non-zero, the mean being 0·74 and the standard deviation of the sample 0·09.

In all, the weighting scheme developed for the twinned data is seen to be fairly successful, judged on internal evidence. It should be mentioned that hydrogen atoms were not included when calculating the F_c.

It is clear that a conventional weighting scheme which was simply a function of $|F_o|$ would be unlikely to succeed: comparing the mean e.s.d.'s 0·9, 1·4, 0·7 and 0·7, or the mean values of $|\Delta|$, 0·7, 0·9, 0·6 and 0·5, with the mean $|F_o|$ values of the S, W, U and E groups respectively (Table 2) shows that the larger errors are not in general associated with the larger $|F_o|$ in these data.

B. The direct use of overlapped data in least-squares refinement

7. *Introduction*

There are two cases in which it is impossible to resolve crystallographically distinct reflexions: that of a twinned single crystal and that of powder diffraction data. Kennicott (1963) has demonstrated that it is still possible to refine such data by the least-squares method. Advantages of this in the case of Para Red are first, that it is unnecessary to correct the raw intensity data for twinning, and secondly, that it is possible to refine the numerical value of the twin fraction *f*. Alternatively, it is shown below that the data can be prepared in such a way that a knowledge of the twin fraction is irrelevant.

8. *The normal Kennicott method*

Kennicott modified the Busing, Martin & Levy (1962) least-squares program to prove that his method worked in practice, and a version of *ORFLS* modified similarly was made available to the author by Dr G. Cox of the Australian Atomic Energy Commission Research Establishment, Sutherland, New South Wales. In effect, one informs the computer of the indices of all the planes contributing to an overlapped reflexion, and by means of separate scale factors (two in this case) of the relative amount of each contribution. The calculated F^2 for each composite reflexion is then compared with that actually observed. Positional and vibrational parameters may be refined in the usual way. Refinement of the twin fraction is done indirectly by refining the scale factors.

The experimental program for doing this is very inefficient in the case of Para Red since all reflexions except 0*kl* are overlapped. Each overlapped reflexion involves two indices, so that computing time is almost doubled. Where separate observations are included for both of a related pair (which is usual), the computer must actually perform twice the calculations for both indices.

9. *The summed-data Kennicott method*

It occurred to the author that the Kennicott program could be used in a different way: in this method each 'observation' is obtained by summing the observed intensities of a related pair of reflexions. An immediate advantage of this is that no wasteful computing occurs, each index appearing once only. A second interesting property is that the summed 'intensity' is independent of the twin fraction, since this merely determines the way the 'intensity' is distributed between the two related spots. The twin fraction thus becomes irrelevant, and this is the chief advantage. It may in some cases

Table 3. *The mean value of* $w\Delta^2$ *for the data-groups* S, W, U, E, (i), (ii), (iii), (iv) *and combinations of these*

The number of reflexions involved is in brackets.

Group	(i)	(ii)	(iii)	(iv)	(i) to (iv)
S	1·18 (317)	0·73 (27)	0·69 (2)	– (0)	1·14 (346)
W	0·87 (13)	1·08 (153)	0·96 (158)	2·51 (17)	1·09 (341)
U	1·01 (117)	1·90 (2)	0·77 (14)	– (0)	1·00 (133)
E	0·89 (32)	1·04 (30)	0·44 (44)	– (0)	0·75 (106)
S+W+U+E	1·11 (479)	1·04 (212)	0·84 (218)	2·51 (17)	1·06 (926)

be considered a disadvantage that the method provides no way of refining, or even of determining, the twin fraction. For this reason both methods have their place.

A numerical comparison of the three different procedures employed in preparing Para Red data may serve to clarify the differences. The raw readings for the related pair of reflexions indexed 1 $\overline{1}$ 0 0 and 1 4 0 were 820 and 450 respectively, on an arbitrary scale. Assuming the twin fraction to be 0·29, the 'corrected' readings are 1070 and 200: $|F_o|$ proportional to the square root of these figures could be used in the customary way, as described in part A. Using the normal Kennicott method as in section 8, however, one would use $|F_o|^2$ proportional to the original raw readings; while for the summed-data Kennicott method of this section a single value proportional to the sum 1270 of the two raw readings would be employed, in association with either one of the two indices (since the computer is informed of the other index in the manner usual to this program). The fact that the sum is independent of the twin ratio is illustrated by noting that the sum of the corrected readings is also 1270.

10. *Discussion*

All three methods were used with the overlapped room-temperature data of Para Red. Further details are given in another paper (Grainger & McConnell, 1969*b*). It is worth pointing out that the summed-data method has the disadvantage that the number of 'observations' is drastically reduced, which increases the e.s.d.'s of the refined parameters. On the other hand, it can be used without knowing the twin fraction, and this could be useful in the early stages of a structure determination.

An advantage of the normal Kennicott method is that it permits the use of reflexions which possess related reflexions whose intensity is for some reason unknown.

A logical sequence of refinement, once the structure has been solved, is to use first the summed-data method to refine the atomic parameters and a scale factor or factors, secondly to use the normal Kennicott method to refine the twin fraction, and thirdly, knowing the latter, to derive structure amplitudes which are then used for the final stages and for producing Fourier syntheses of the electron density.

This sequence was used for the last 11 cycles of refinement of Para Red. Two cycles using summed data were followed by 5 cycles with the normal Kennicott method during which the twin fraction refined from 0·275 to 0·286 $\pm$ 0·005 which agrees closely with the experimental result (section 3). The final four cycles used structure amplitudes corrected for twinning using this new value. It should be noted that Para Red was particularly slow to refine, which was aggravated by the fact that only 140 of the 198 parameters could be refined in any one cycle by use of the existing program, and that fractional shifts were then necessary to give convergence. For this reason the three methods were not used in strictly parallel refinements to obtain three final results. Nevertheless the parameters resulting from the normal Kennicott refinement can reasonably be compared with the final ones using corrected data. The values of x and y coordinates were found to differ by less than one s.d. except for five cases where the difference was less than two standard deviations. For the z coordinates about half the differences were less than one standard deviation, all except two were less than two s.d.'s and these two were about $2\frac{1}{2}$ s.d.'s. Considering the fact that the 'observations' are of greater accuracy, it is probable that the coordinates using the conventional Kennicott method are the more accurate of the two sets.

It is possible that the use of corrected structure factors in Fourier summations could produce serious errors owing to the large fractional errors which can occur, as mentioned in section 5. The use of a Fourier weighting scheme may be necessary, but no investigations along these lines have been made.

The principles outlined in this paper should be applicable to cases involving overlapped data from twins of types other than pseudo-merohedral. However, although it is feasible to deal with overlapped data in the ways described, the use of untwinned crystals is recommended if maximum accuracy is desired.

The author wishes to thank Professor J. F. McConnell for helpful discussions, and for his careful checking of the manuscript.

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