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# The Structure of the Centrosymmetric Isomer of 1:2:3:4-Tetraphenylcyclobutane

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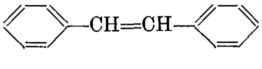
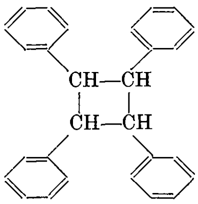
The crystal and molecular structure of the centrosymmetric isomer of 1:2:3:4-tetraphenylcyclobutane has been established by a comprehensive X-ray analysis. The intramolecular bond lengths and angles have been determined with an accuracy of about 0·02 A. and 2° respectively. It is found that the benzene rings are regular hexagons of side 1·39 A. and that the bonds adjacent to them are shortened from 1·54 to 1·50 A. An increase from 1·54 A. appears to occur for the bonds in the cyclobutane ring, the measured values being 1·585 and 1·555 A. This increase may be associated with a change in the hybridization ratios of the orbitals in the cyclobutane ring. The molecules are held together by a system of van der Waals bonds between the phenyl groups and achieve a very compact packing in the crystal.

## Introduction

A preliminary X-ray analysis (Fulton & Dunitz, 1947) of the dimer of stilbene (I), m.p. 163° C., has shown it to be the centrosymmetric isomer of 1:2:3:4-tetraphenylcyclobutane (II).



(I)



(II)

One well-resolved Fourier projection, on (010), of the structure was obtained and from this it was clear (see Fig. 3) that the formula (II) must be assigned to the molecule of the dimer. As this molecule is non-planar, a knowledge of the $y$ parameters, normal to the plane of the projection obtained, is necessary in order to describe the structure completely. Other projections gave very poor resolution of the atoms, but it was found possible to assign $y$ parameters which gave good agreement between the calculated and observed $F$'s for the $(0kl)$ zone of reflexions. From the parameters thus obtained the dimensions of the molecule could be calculated to a first approximation; it was found that although most of the interatomic distances were in accordance with expectation, those in the central ring were not so. One side of this ring measured 1·63 A. (an increase of 0·09 A. over the normal single-bonded C–C distance), the other 1·49 A. Since these distances were regarded as reliable to about 0·05 A., it seemed desirable to carry the analysis further, in order to establish with greater precision the dimensions of the 4-membered ring. A more detailed investigation, making use of comprehensive three-dimensional data, has now been carried out, and is described below, together with a fuller account of the preliminary analysis than has been given previously.

## Preparation of crystals

Crystals of a dimer of stilbene, $C_{28}H_{24}$, m.p. 163° C., were obtained from Dr J. D. Fulton, of the National Institute for Medical Research, Hampstead, London. The substance had been prepared:

(*a*) By irradiation of solutions of stilbene in benzene with sunlight or ultra-violet light.

(*b*) By irradiation of solutions of stilbamidine (4:4′-diamidinostilbene $\beta$-hydroxy ethane sulphonate) in water with sunlight, followed by hydrolysis and subsequent decarboxylation of the product, as described by Fulton (1948).

Samples prepared by the two methods gave identical X-ray photographs and the crystals used in the analysis were obtained by crystallizing both samples together from benzene or amyl acetate. The colourless, monoclinic crystals are generally in the form of needles, elongated in the direction of the $b$ axis. The end faces were not identified but in the main zone the faces usually developed were (001), (100) and $(20\bar{1})$.

Other crystals of the same empirical formula, m.p. 149° C., were also obtained by preparation (*b*). It is believed that these consist of the isomer of 1:2:3:4-tetraphenylcyclobutane with a fourfold alternating axis of symmetry ($S_4$). The preliminary X-ray measurements (Fulton & Dunitz, 1947) show, however, that no molecular symmetry is imposed crystallographically, so that a full structure analysis would be necessary to settle this point.

## Unit cell and space group

Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54$ A.) was used throughout the analysis. The unit-cell dimensions were found from rotation and oscillation photographs about [100], [010], [001] and [102] to be: $a = 17{\cdot}02 \pm 0{\cdot}05$, $b = 5{\cdot}775 \pm 0{\cdot}02$, $c = 12{\cdot}35 \pm 0{\cdot}05$ A., $\beta = 127 \pm 1°$.

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No $(h0l)$ reflexions with $h$ odd, nor $(0k0)$ reflexions with $k$ odd were observed; the space group is therefore $P2_1/a$. The observed density, 1·23 g.cm.$^{-3}$, is consistent with the presence of four asymmetric units, $C_{14}H_{12}$, in the unit cell. If these are grouped in pairs to form two molecules of the dimer, $C_{28}H_{24}$, the resulting molecules must each possess a centre of symmetry.

### Intensity measurements

For the preliminary analysis the $(h0l)$ and $(0kl)$ reflexions were recorded on Weissenberg photographs. At a later stage it was decided to record all $(hkl)$ reflexions within the sphere of reflexion for Cu $K\alpha$ radiation. Using the Buerger equi-inclination camera the $(h1l)$–$(h4l)$ layers were photographed; for reflexions with $k>4$ a series of photographs of the $(1kl)$–$(12{\cdot}k{\cdot}l)$ layers was made. A series of oscillation photographs about [102] practically completed the exploration of the sphere of reflexions, only 4 reflexions, all close to the limit, being outside the range of the photographs.

In all cases care was taken to record the reflexions from crystals for which absorption errors would be small, since it was impracticable to carry out even approximate absorption corrections for the 1260 reflexions whose intensities were measured. The actual crystals used varied between $0{\cdot}004\times0{\cdot}006$ cm. and $0{\cdot}04\times0{\cdot}06$ cm. in cross-section, but showed no significant variation in the relative intensities recorded.

The multiple-film technique was employed throughout to ensure that a large range of intensities could be measured from each exposure. The estimation of the intensities was carried out visually with the aid of calibrated scales, prepared by recording timed exposures of a crystal reflexion. The usual Lorentz and polarization corrections were made and the further correction to the Lorentz factor necessary for non-equatorial reflexions was carried out by the method of Tunell (1939) for equi-inclination photographs and by the method of Cox & Shaw (1930) for oscillation photographs.

The set of relative $F^2$ values for the $(h0l)$ zone was brought to an approximately absolute scale after a trial structure had been found, by multiplication by a factor to make $\Sigma|F_{\text{obs.}}|=\Sigma|F_{\text{calc.}}|$. The sets of relative $F^2$ values for the other series of photographs were made approximately absolute by correlation with the $(h0l)$ reflexions. Many of the reflexions appeared on more than one series of photographs and intensive cross-checking was carried out to ensure internal consistency.

### Calculation of structure factors

Throughout the analysis the structure factors were calculated in the following manner. The normalized geometric structure amplitude

$$\frac{1}{14}\sum_{i=1}^{14}\cos 2\pi(hx_i+lz_i)\cos 2\pi ky_i,\quad \text{when } h+k=2n,$$

and

$$-\frac{1}{14}\sum_{i=1}^{14}\sin 2\pi(hx_i+lz_i)\sin 2\pi ky_i,\quad \text{when } h+k=2n+1,$$

summed over the 14 carbon atoms in the asymmetric unit, was multiplied by the angle-dependent scattering factor $f$, obtaining by scaling the anthracene $f$ curve (Robertson, 1935) so as to make its maximum 384 $(=F(000))$ equal to the total number of electrons in the unit cell. This procedure ensured that the calculated $F$'s were on an approximately absolute scale. The observed $F$'s were brought to the same scale as described in the previous section.

### Structure analysis

In the absence of any definite chemical evidence concerning the molecular structure of the stilbene dimer, it was assumed in the first place that the substance was the centrosymmetric isomer of 1:2:3:4-tetraphenylcyclobutane. This original assumption has clearly been justified by the success of the subsequent analysis.

The preliminary trial analysis was based on the presence of certain outstandingly strong $(h0l)$ reflexions.

It has been shown by Robertson & White (1945, 1947) that consideration of the outstandingly strong high-order reflexions may lead to the establishment of a reasonably accurate trial structure, especially for a fairly rigid molecule. Since these reflexions are sensitive to small shifts in the atomic positions, it may be assumed to a first approximation that the contributions of all the atoms to these reflexions are in phase and have a maximum value. For reflexions whose geometric structure factor is of the type $\cos 2\pi(hx+lz)$ this method is especially powerful, since all the atoms must lie on the traces, either of the planes or of the 'half-planes' $(h0l)$, where their contributions have a maximum positive or negative value. The traces of the strong planes (and half-planes) may be drawn and a model of the molecule adjusted until the above conditions are satisfied as nearly as possible.

Examination of the $(h0l)$ reflexions showed that the $F$ values for the low-order reflexions $(20\bar{3})$ and $(40\bar{1})$, and for the high-order reflexions $(20{\cdot}0{\cdot}\overline{13})$ $(18{\cdot}0{\cdot}\overline{14})$, $(605)$ and $(10{\cdot}0{\cdot}4)$ were outstandingly high. A rough molecular model was constructed and various positions and orientations of the phenyl groups were tested in an attempt to account for these observations. The strength of $(20\bar{3})$ and $(40\bar{1})$ could be explained by assuming that the traces of these planes on (010) passed approximately through the mid-points of the projected phenyl groups. The phenyl groups were then oriented so as to account for the strength of the high-order reflexions. The final adjustment, to make $(20{\cdot}0{\cdot}\overline{13})$ strongly positive and $(18{\cdot}0{\cdot}\overline{14})$, $(605)$ and $(10{\cdot}0{\cdot}4)$ strongly negative, is shown in Fig. 1. That this trial structure is reasonably accurate may be seen by comparison of Fig. 1 with Fig. 2.

The calculated $F$'s for this structure agreed sufficiently well with the observed $F$'s for the ($h0l$) reflexions, to allow refinement by a series of successive Fourier projections, which were calculated, as were all subsequent Fourier summations, with the aid of Beevers-Lipson strips. After each projection the $F$'s were recalculated and the discrepancy, expressed in the usual way as $\Sigma\,|\,F_{\text{obs.}}-F_{\text{calc.}}\,|\div\Sigma\,|\,F_{\text{obs.}}\,|$, was found to decrease steadily as follows:

| | Discrepancy |
|---|---|
| Trial parameters | 0·37 |
| Parameters from 1st projection | 0·26 |
| Parameters from 2nd projection | 0·19 |
| Parameters from 3rd projection | 0·16 |

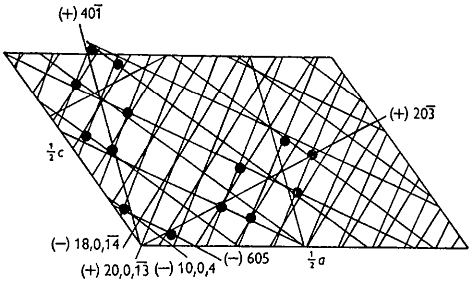

Fig. 1. Trial structure based on outstandingly strong ($h0l$) reflexions.

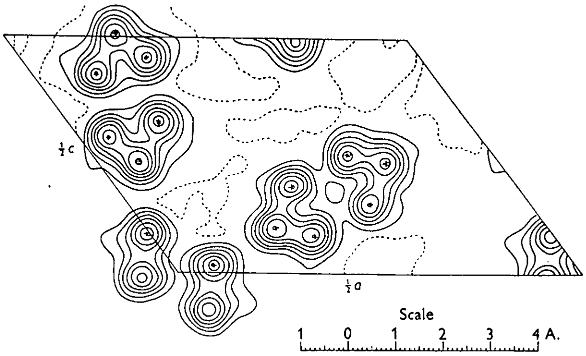

Fig. 2. Final projection on (010) showing the asymmetric crystal unit. Contours are drawn at electron-density increments of approximately one electron per A.$^2$, the one-electron line being dotted. The crosses and circles represent the atomic positions of Tables 1 and 2 respectively.

Of the 161 ($h0l$) reflexions observed, all except two doubtful traces were included in the final synthesis, which thus represents effectively the limit of refinement possible using the ($h0l$) reflexions alone. A plot of the asymmetric unit for the final projection is shown in Fig. 2 and of the complete unit cell in Fig. 3.

The $y$ parameters were obtained by trial and error but it was not possible to obtain much refinement by Fourier projections, owing to the overlapping of atoms in projections normal to (010). Fig. 4 shows the Fourier projection on (100) with an explanatory diagram. After some adjustment of the $y$ parameters, the discrepancy for the ($0kl$) reflexions was reduced to 0·16 so that serious errors were not likely to be present.

## Results of the two-dimensional analysis

It is perhaps useful to review here the results of the analysis up to this stage; first, as they were considered to justify the somewhat laborious three-dimensional calculations which followed, and secondly, as it may be interesting to compare them with the final results.

As a consequence of the preceding analysis it could be said with certainty that the chemical individual in question was the centrosymmetric isomer of 1:2:3:4-tetraphenylcyclobutane. Furthermore, the low values obtained for the discrepancies of the ($h0l$) and ($0kl$) reflexions indicated that the atomic parameters which had been assigned could not be in error by very large amounts. The atomic positions projected on (010) were regarded as accurate to about 0·03 A. while it was hoped that the $y$ parameters were accurate to 0·05 A. The parameters are given in Table 1.

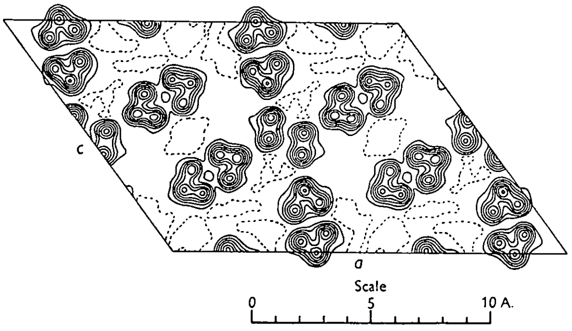

Fig. 3. The unit cell projected on (010), showing a complete molecule of 1:2:3:4-tetraphenylcyclobutane.

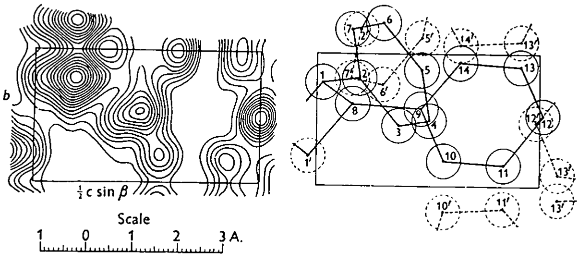

Fig. 4. Projection on (100), showing the asymmetric unit. Contours represent density increments of one electron per A.$^2$, the lowest contour drawn being the three-electron line. A drawing of the crystal structure projected on (100) is shown alongside.

The dimensions of the non-planar tetraphenylcyclobutane molecule, calculated from the above coordinates, are shown in Fig. 5. It is seen that the dimensions of the benzene rings are consistent, within the estimated limit of accuracy of 0·06 A., with their being regular hexagons of side 1·39 A. The dimensions found for the four-membered ring are, on the other hand, somewhat surprising, the bond 8–1 measuring 1·63 A., an increase of 0·09 A. over the normal single-bond distance for carbon atoms. This bond measures 1·57 A. in projection (a minimum distance for the bond) so that the deviation could not be entirely ascribed to faulty assignment of the $y$ parameters. Furthermore, in the original trial structure the projected length of this bond was taken as 1·4 A. so that the apparent increase was a result of the observed $F$'s included in the Fourier syntheses, and not of the initial assumptions. The evidence seemed to suggest that the bond 8–1 was definitely longer than 1·54 A.

Table 1. *Atomic parameters obtained by two-dimensional analysis, with corresponding $x'$, $y$, $z'$ co-ordinates (with respect to the $a$, $b$, and $c'$ crystal axes, where $c'$ is perpendicular to $a$ and $b$)*

| Atom | $x/a$ | $y/b$ | $z/c$ | $x'$ (A.) | $y$ (A.) | $z'$ (A.) |
|---|---|---|---|---|---|---|
| 1 | 0·051 | 0·138 | 0·015 | 0·76 | 0·80 | 0·14 |
| 2 | 0·163 | 0·156 | 0·096 | 2·05 | 0·90 | 0·95 |
| 3 | 0·221 | −0·023 | 0·185 | 2·38 | −0·13 | 1·82 |
| 4 | 0·319 | −0·003 | 0·254 | 3·54 | −0·02 | 2·50 |
| 5 | 0·360 | 0·187 | 0·235 | 4·37 | 1·08 | 2·32 |
| 6 | 0·303 | 0·366 | 0·148 | 4·05 | 2·11 | 1·46 |
| 7 | 0·204 | 0·343 | 0·078 | 2·88 | 1·98 | 0·77 |
| 8 | −0·004 | 0·060 | 0·081 | −0·67 | 0·35 | 0·80 |
| 9 | 0·054 | 0·043 | 0·236 | −0·84 | 0·25 | 2·32 |
| 10 | 0·037 | −0·151 | 0·286 | −1·49 | −0·87 | 2·82 |
| 11 | 0·081 | −0·168 | 0·420 | −1·75 | −0·97 | 4·14 |
| 12 | 0·142 | 0·009 | 0·508 | −1·36 | 0·05 | 5·00 |
| 13 | 0·159 | 0·203 | 0·458 | −0·69 | 1·17 | 4·50 |
| 14 | 0·115 | 0·220 | 0·322 | −0·43 | 1·27 | 3·17 |

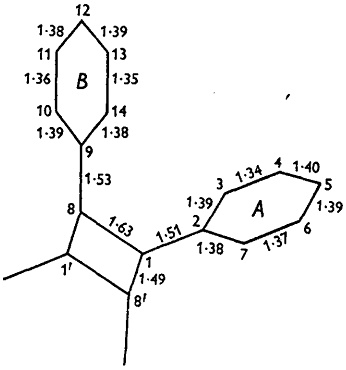

Fig. 5. Bond lengths calculated from the co-ordinates of Table 1.

The bond 8′–1 was found to be 1·49 A., but as it is inclined more steeply to (010) it is rather more sensitive to errors in the $y$ parameters. The apparent difference between the lengths of the two sides of the four-membered ring was provisionally attributed to the different configurations of the phenyl groups about these sides (*cis*— about 1–8, *trans*— about 1–8′).

Since no example of a C–C bond definitely longer than 1·54 A. had been previously reported, it seemed desirable that such a surprising result should be confirmed or otherwise by a more detailed investigation. A comprehensive three-dimensional analysis was therefore undertaken with the main object of obtaining more accurate values for the dimensions of the 4-membered ring.

## Three-dimensional calculations

The laborious task of calculating more than 2000 structure factors was undertaken by Mr D. J. Finney and the staff of the Lectureship in the Design and Analysis of Scientific Experiment, in the University of Oxford. Of the 1260 reflexions whose $F$ values could be measured, 47 were omitted owing to ambiguity of sign. 1213 reflexions were thus included in the three-dimensional Fourier syntheses.

Since projections of the structure normal to (010) were most unsatisfactory, it was decided to calculate section-projections (Booth, 1945) with the object of obtaining more accurate $y$ parameters for the atoms in the phenyl groups.

A section-projection down the $c$ axis was calculated for the limits $z=0\rightarrow\frac{1}{4}$, when the Fourier series takes the form

$$
\begin{aligned}
B_0^{\frac{1}{4}}(x,y) &= \int_0^{\frac{1}{4}} \rho(x,y,z)\,dz \\
&= \frac{4}{\pi A} \sum_{\substack{0\\h+k=2n}}^{h}\sum_0^{k}\sum_0^{l} \left\{ \frac{\pi F(hk0)}{4}\cos 2\pi hx \right. \\
&\qquad + (F_{hkl}+F_{hk\bar{l}})\,C_l \cos 2\pi hx \\
&\qquad \left. - (F_{hkl}-F_{hk\bar{l}})\,S_l \sin 2\pi hx \right\} \cos 2\pi ky \\
&\quad - \frac{4}{\pi A} \sum_{\substack{0\\h+k=2n+1}}^{h}\sum_0^{k}\sum_0^{l} \left\{ \frac{\pi F(hk0)}{4}\sin 2\pi hx \right. \\
&\qquad + (F_{hkl}+F_{hk\bar{l}})\,C_l \sin 2\pi hx \\
&\qquad \left. + (F_{hkl}-F_{hk\bar{l}})\,S_l \cos 2\pi hx \right\} \sin 2\pi ky,
\end{aligned}
$$

where

$C_l=0$ when $l=2n$; $S_l=0$ when $l=4n$;

$C_l=1/(2l)$ when $l=4n+1$; $S_l=1/(2l)$ when $l=2n+1$;

$C_l=-1/(2l)$ when $l=4n+3$; $S_l=1/l$ when $l=4n+2$.

A portion of this section-projection is shown in Fig. 6. Atoms 4, 5, 1′, 1 are cut by the limits while 8, 9, 1 are not resolved. Accurate $x$ and $y$ co-ordinates can, however, be obtained for atoms 2, 3, 6, 7.

A section-projection down the $a$ axis was calculated with the limits $x=0\rightarrow\frac{1}{4}$. The Fourier series is of analogous form to that quoted previously. Two portions of this section-projection are shown in Fig. 7. In Fig. 7 (*b*) the phenyl group $B$ is resolved, with the exception of atom 9, which overlaps with 3. In Fig. 7 (*a*) atoms 4 and 5, which were cut by the limits of the first section-projection, are shown, along with atom 6. It should be noted that these atoms are not in the standard asymmetric unit at $x, y, z$ but in the related one at $\frac{1}{2}-x, \frac{1}{2}+y, \bar{z}$.

Since the chief interest lay in the dimensions of the 4-membered ring, lines and sections were now calculated through atoms 1 and 8. Lines parallel to $b$ were calculated through the $x$, $z$ positions previously found.

The maxima corresponding to $y_1$ and $y_8$ were found by differential synthesis (Booth, 1946), as, in the case of a line, the differential formula becomes very simple.

Sections parallel to (010) were now calculated at $y_1$ and $y_8$, and the portions close to the atoms 1 and 8 are shown superimposed in Fig. 8. These sections pass close to the centres of several other atoms and more accurate positions for atoms 2, 6, 7, 9 could also be established.

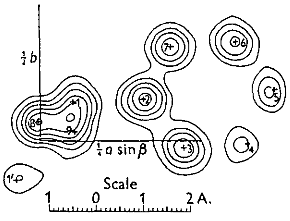

Fig. 6. Portion of section-projection down the $c$ axis, limits $z = 0 \rightarrow \frac{1}{4}$, showing benzene ring $A$. The contours represent density increments of approximately one electron per A.$^2$, the one-electron line being omitted.

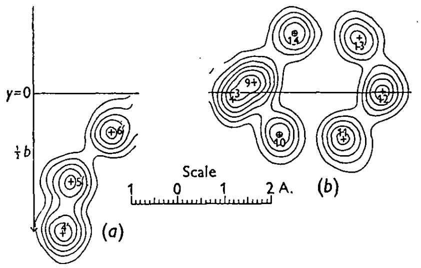

Fig. 7. Portions of section-projection down the $a$ axis, limits $x = 0 \rightarrow \frac{1}{4}$. Contours are drawn as in Fig. 6. ($a$) Portion of benzene ring $A$, cut by the limits in the previous section-projection; ($b$) benzene ring $B$.

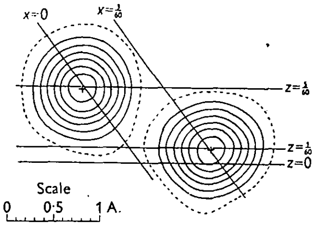

Fig. 8. Sections at $y_1$ and $y_8$ superimposed, showing atoms 1 and 8. The one-electron contour is dotted.

A line parallel to $b$ was calculated through atom 9, since this atom had not been well resolved in the previous section-projections. All of the 42 parameters required to define the structure could now be measured directly from at least one well-resolved projection, section-projection line or section.

### Results of three-dimensional calculations

The parameters finally assigned are given in Table 2, together with the corresponding co-ordinates as in Table 1. Parameters obtained from three-dimensional lines and sections are shown in heavy type; they are probably the most accurate. Parameters from section-projections are shown in ordinary type, while parameters obtained only from the projection on (010) are in italics.

Table 2. *Final parameters assigned to the atoms, together with corresponding $x'$, $y$, $z'$ co-ordinates (see Table* 1)

| | $x/a$ | $y/b$ | $x/c$ | $x'$ (A.) | $y$ (A.) | $z'$ (A.) |
|---|---|---|---|---|---|---|
| 1 | **0·0506** | **0·142** | **0·0150** | 0·750 | 0·820 | 0·148 |
| 2 | **0·1607** | 0·152 | **0·0969** | 2·015 | 0·878 | 0·956 |
| 3 | 0·221 | −0·025 | *0·183* | 2·402 | −0·144 | 1·805 |
| 4 | *0·321* | −0·010 | 0·253 | 3·584 | −0·058 | 2·495 |
| 5 | *0·362* | 0·182 | 0·236 | 4·407 | 1·051 | 2·328 |
| 6 | **0·3031** | 0·360 | **0·1484** | 4·055 | 2·079 | 1·464 |
| 7 | **0·2028** | 0·3425 | **0·0795** | 2·861 | 1·978 | 0·784 |
| 8 | **−0·0024** | **0·0715** | **0·0805** | −0·639 | 0·413 | 0·794 |
| 9 | **0·0537** | **0·038** | **0·2310** | −0·803 | 0·220 | 2·278 |
| 10 | *0·037* | −0·154 | 0·285 | −1·488 | −0·890 | 2·810 |
| 11 | *0·081* | −0·172 | 0·4217 | −1·756 | −0·993 | 4·160 |
| 12 | *0·142* | 0·002 | 0·5067 | −1·349 | 0·012 | 4·998 |
| 13 | *0·158* | 0·198 | 0·4553 | −0·695 | 1·144 | 4·491 |
| 14 | *0·1135* | 0·216 | 0·3175 | −0·428 | 1·248 | 3·132 |

The mean and maximum differences between the co-ordinates given in Tables 1 and 2 are as follows:

| | Mean (A.) | Maximum (A.) |
|---|---|---|
| Difference in $x'$ | 0·017 | 0·045 |
| Difference in $y$ | 0·027 | 0·065 |
| Difference in $z'$ | 0·014 | 0·04 |
| Total movement | 0·040 | 0·07 |

One difficulty is that there is a difference of about 0·03 A. in the position of atom 8 as measured from the three-dimensional section and from the final projection on (010), where the atom is comparatively well resolved (Fig. 2). This discrepancy may be due to the displacement of the peak of atom 8 in the projection, by the electrons in the C–H bond, which may be assumed to be directed towards the 'hump' in the 2-electron contour round atom 8. It is possible, of course, that the hump is purely spurious, but it is perhaps significant that other humps in the 2-electron contour round the phenyl groups are clearly in the directions of the C–H bonds.

From the co-ordinates in Table 2 the bond lengths and bond angles in the molecule may be calculated. They are shown in Fig. 9. The differences in the bond lengths in the phenyl groups do not appear to be systematic and each measurement may be regarded as an independent measurement of the same quantity, the aromatic C–C distance. The same holds for the bond angles in the phenyl groups. If these measurements are averaged, the following results are obtained.

| | Ring $A$ | Ring $B$ | Combined |
|---|---|---|---|
| Average bond length (A.) | 1·385 | 1·390 | 1·388 |
| Mean deviation (A.) | 0·008 | 0·010 | 0·009 |
| Maximum deviation (A.) | 0·014 | 0·020 | 0·021 |
| Average bond angle (°) | 120·0 | 119·8 | 119·9 |
| Mean deviation (°) | 0·9 | 0·7 | 0·8 |
| Maximum deviation (°) | 1·8 | 1·6 | 1·7 |

These results may be regarded as good evidence that the benzene rings in this compound are regular hexagons of side 1·39 ± 0·02 A., identical with the most accurate experimental value for benzene itself (Schomaker & Pauling, 1939). The internal consistency of the above results suggests further that it is unlikely that the observed bond lengths and angles are in error by more than 0·02 A. and 2° respectively.

We are thus led to the conclusion that the observed bond lengths in the central ring, 1·585 and 1·555 A., strongly favour the view that there occurs a small but real lengthening from the C–C single-bond distance of 1·5445 A. in diamond (Lonsdale, 1947). It may be said that 1–8 is definitely longer than 1·54 A. and 1–8′ probably so. The question of whether or not the ring is square, is, however, still open, as the difference between the two bond lengths is hardly greater than the present uncertainty. Further calculations making fuller use of the experimental data may be carried out when improved computing facilities are available, and may give a more definite answer to this question.

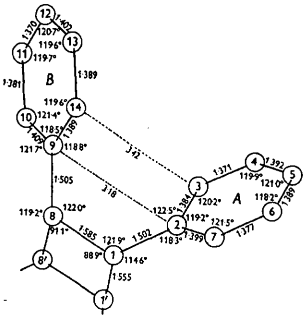

Fig. 9. Bond lengths and bond angles calculated from final co-ordinates.

It is, on the other hand, fairly safe to assume that the distances 1–2 and 8–9, measured as 1·502 and 1·505 A., are actually equal.

### Comparison with the dibenzyl analysis

It is interesting to compare the results of this analysis with those obtained by Jeffrey (1947) for dibenzyl. Jeffrey's experimental data comprised the intensities of 746 reflexions as against more than 1200 in the present analysis. But as there are only 7 atoms in the asymmetric crystal unit of dibenzyl, and 14 in that of tetraphenylcyclobutane, Jeffrey's data may be regarded as more comprehensive. Furthermore, in the dibenzyl analysis the three-dimensional Fourier syntheses were refined to a considerably greater degree than in this analysis. On the other hand, Jeffrey's intensities, measured from oscillation photographs, are probably rather less accurate than the intensities employed here, which were measured almost entirely from Weissenberg photographs. On the whole, Jeffrey's results are likely to be slightly more trustworthy than the present results.

The short central bond of 1·48 A. in dibenzyl has no exact parallel in tetraphenylcyclobutane, where the central bonds form the 4-membered ring. It may be noted that the central bond 1–8, about which the configuration of the phenyl groups is *trans* (as in dibenzyl), is measured shorter than the bond 1–8, about which the configuration is *cis*, but, as has already been pointed out, it is difficult to attach very much significance to the small difference observed.

The bonds adjacent to the phenyl groups are 1·50 A. in both tetraphenylcyclobutane and dibenzyl. We might expect that, as found for dibenzyl, the shortening of these bonds might be reflected by a systematic redistribution of the bond lengths in the benzene rings. In fact, no evidence whatsoever is found for this effect in the present analysis, where the consistency of the bond distances and angles in the benzene rings support the view that the phenyl groups in tetraphenylcyclobutane are quite regular.

### The dimensions of cyclobutane rings

Little detailed information concerning the dimensions of cyclobutane rings is so far available. Cyclobutane itself does not seem to have been examined by diffraction methods, but two independent electron-diffraction studies have been carried out for methylene cyclobutane. The C–C distance in the ring was found by Bauer & Beach (1942) to be 1·56 ± 0·03 A. and by Shand, Schomaker & Fischer (1944) to be 1·55 ± 0·02 A. In addition, the value 1·56 ± 0·05 A. has been given for the side of the cyclobutane ring in dimethyl ketene dimer (Lipscomb & Schomaker, 1946). It is noticeable that, while all these results are compatible with the normal single-bond distance, 1·5445 A., none of them is less than 1·55 A. In the present analysis the 4-membered ring is found to have sides 1·585 and 1·555 A. (both ± 0·02 A.).

It seems possible in 4-membered rings, where the bond angles are constrained to be close to 90°, that the usual $sp^3$ hybridization of the carbon orbitals may be disturbed to some extent. The orbitals involved in the formation of the cyclobutane ring would tend to acquire extra $p$ character at the expense of increased $s$ character in the orbitals directed towards the hydrogen atoms. This tendency, although slight for cyclobutane itself, would be accentuated in tetraphenylcyclobutane by hyperconjugation between the orbitals in the aromatic rings and those in the central ring; the orbitals directed towards the phenyl groups would tend to become $sp^2$ (with consequent decrease in bond length, 1·54 → 1·50 A.), while the orbitals involved in the formation of the 4-membered ring would acquire additional $p$ character (with consequent increase in bond length). A preliminary X-ray analysis of dinaphthylene cyclobutane (Dunitz & Weissman, 1949) indicates that the sides of the central ring in this compound may be somewhat longer than 1·54 A.

### Distortions in the molecule

It might be expected that the molecule of centrosymmetric 1:2:3:4-tetraphenylcyclobutane would exist in a configuration with the phenyl groups *A* and *B* related to one another by an additional plane of symmetry as shown in Fig. 10 (*a*) to give the molecule the symmetry $2/m$. In the crystal the molecules do not attain this symmetry and have only the symmetry $\bar{1}$ imposed by the space-group restrictions. The actual configuration is shown diagrammatically in Fig. 10 (*b*); the plane of phenyl group *A* is nearly normal (92°) and the plane of phenyl group *B* is inclined at only 39° to the plane of the central ring.

The spokes 1–2 and 8–9 are inclined at 48° and 43° respectively to the plane of the central ring, but they are neither quite collinear with 2–5 and 9–12 nor are they co-planar with the benzene rings. For 1–2 the deviation is only about 2°, resulting in atom 1 being 0·05 A. out of the plane of ring *A*, but for 8–9 the deviation is much more serious, almost 7°, with atom 8 situated at 0·18 A. from the plane of ring *B*. It is seen from Fig. 9 that other small distortions occur in the angles round atoms 1, 2, 8, 9.

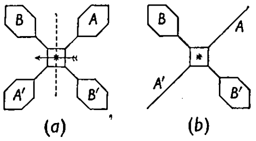

Fig. 10. Diagrammatic representation of molecular configurations. (*a*) Molecule with symmetry $C_{2h}(2/m)$; (*b*) actual symmetry $C_i(\bar{1})$.

Three possible causes may be postulated for the observed distortions:

(*a*) They may be due to experimental errors in the observations, incompleteness of the Fourier series and computational errors. The consistency of the bond lengths and bond angles observed in the phenyl groups suggests that errors due to these factors are probably less than 0·02 A. and 2°. It may be regarded as highly improbable that these factors are significant in explaining distortions of up to 7°.

(*b*) The interactions between the *cis*-related phenyl groups may account for some of the distortions. The observed angles around atoms 1 and 2 suggest that the phenyl group *A* is being pushed away from phenyl group *B*. One might expect that similar distortions should occur in the angles round atoms 8 and 9, but these do not occur. On the other hand, the deviation of atom 8 from the plane of ring *B* is considerably greater than the deviation of atom 1 from the plane of ring *A*. The different distortion effects for the phenyl groups *A* and *B* may be associated with the different orientations of the benzene rings to the 4-membered ring.

The distances between the non-bonded atoms 2 and 9, and 3 and 14 are 3·18 and 3·42 A. respectively. The latter distance is almost identical with the interlaminar spacing in graphite, while both distances are close to the distances between non-bonded atoms in dibenzyl, 3·18 and 3·48 A. The only other molecule, containing phenyl groups in a *cis*-configuration, whose structure has been determined is *cis*-azobenzene where the non-bonded *o*-atoms are separated by 3·34 A. (Hampson & Robertson, 1941). The closest approaches between atoms in the *trans*-related phenyl groups are 3·62 A. for 2–9′ and 4·00 A. for 7–10′.

(*c*) Distortions may be produced by the molecules attaining a configuration which permits the most compact packing in the crystal. The closest intermolecular approaches are shown in Fig. 11. It is seen that the molecules are held in the crystal by a system of van der Waals bonds between the phenyl groups. Similar close approaches of about 3·80 A. (not shown in Fig. 11) occur between atoms 10 and 14*b*, 11 and 13*b*, 3 and 7*b*, 4 and 6*b* (where the suffix *b* indicates an atom in a molecule one translation removed along the *b* axis). Every atom in the phenyl groups has at least one neighbour in an adjacent phenyl group, at a distance of less than 4 A. Such an arrangement must be very stable with respect to the van der Waals energy, and the stabilization may compensate for any increase in potential energy due to the distortions. The compactness of the molecular packing is reflected by the comparatively high density of the crystals (1·23 g.cm.$^{-3}$), compared with stilbene (1·16) and dibenzyl (1·11). A similar situation has been noted by Dunitz & Robertson (1947) for diacetylene dicarboxylic acid dihydrate, where a very stable system of intermolecular hydrogen bonds is associated with appreciable molecular distortion.

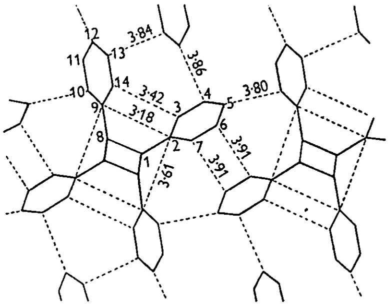

Fig. 11. Close intermolecular approaches.

## Agreement between calculated and observed *F*'s

The *F* values calculated from the parameters in Table 2 are given in Table 3 along with the observed *F*'s. The discrepancy for all observed reflexions is 0·208.

The procedure followed for the calculation of the *F*'s, described in an earlier section, cannot be regarded as entirely satisfactory. The only justification for the use of the anthracene *f* curve for other hydrocarbons is the fact that good agreement between observed and calculated *F*'s can often be obtained, and although the curve is found to be generally applicable it is not suitable in some cases, e.g. pyrene (Robertson & White, 1947), presumably owing to differences in the temperature factor.

In the present analysis the falling off of the relative $F$ values of the $(h0l)$ reflexions with increasing $(\sin\theta)/\lambda$ corresponds fairly closely to the falling off indicated by the anthracene curve; the scale of the observed $F$'s was therefore fixed using these reflexions. For $(hkl)$ reflexions, however, the observed $F$'s (correlated with the $(h0l)$ $F$'s) are in general lower than the calculated $F$'s. The effect is small for low orders of $k$, but is quite striking for high orders; it would suggest that the temperature factor may be anisotropic and that the use of any spherically symmetrical $f$ curve may not be warranted.

Since the calculated $F$'s have been employed only in order to fix the signs of the observed $F$'s, the structure determination itself is independent of the $f$ curve used. The discrepancy between observed and calculated $F$ values, commonly adopted as a criterion for the assessment of the accuracy of a structure analysis, is however artificially great for reflexions of high order of $k$.

The author takes this opportunity of thanking Mrs D. M. Hodgkin (D. Crowfoot) for her continued interest and encouragement. Thanks are also due to Mr D. J. Finney for the calculation of the three-dimensional structure factors, and to the Carnegie Trust for the Universities of Scotland for the award of a Senior Research Scholarship.

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Table 3. *List of observed and calculated structure factors*

| $hkl$ | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ |
|---|---|---|
| 001 | 27 | +29 |
| 002 | 14 | −12 |
| 003 | 25 | −26 |
| 004 | 37 | +32 |
| 005 | 22 | −20 |
| 006 | 20 | −19 |
| 007 | 4 | −2 |
| 008 | <3 | −1 |
| 009 | 4 | +4 |
| 0,0,10 | 5 | +2 |
| 0,0,11 | 4 | +2 |
| 0,0,12 | 8 | +7 |
| 011 | 40 | −47 |
| 012 | 39 | −41 |
| 013 | 23 | −23 |
| 014 | 22 | −17 |
| 015 | 8 | −8 |
| 016 | 3 | +2 |
| 017 | <3 | −3 |
| 018 | 14 | +16 |
| 019 | <3 | −4 |
| 0,1,10 | 4 | −2 |
| 0,1,11 | <3 | +2 |
| 0,1,12 | 2 | +4 |
| 020 | 4 | +1 |
| 021 | 10 | +9 |
| 022 | 11 | −12 |
| 023 | 20 | −25 |
| 024 | 13 | +16 |
| 025 | 6 | +5 |
| 026 | 3 | +2 |
| 027 | 12 | −15 |
| 028 | 11 | +10 |
| 029 | 7 | −8 |
| 0,2,10 | <3 | +1 |
| 0,2,11 | <3 | 0 |
| 0,2,12 | 2 | +4 |
| 031 | 1 | +2 |
| 032 | 19 | −20 |
| 033 | 11 | −14 |
| 034 | 5 | −5 |
| 035 | 4 | −2 |
| 036 | 5 | −5 |
| 037 | 8 | +7 |
| 038 | <3 | +1 |
| 039 | <3 | +1 |
| 0,3,10 | <3 | 0 |
| 0,3,11 | <3 | +1 |
| 040 | 6 | −2 |
| 041 | 17 | −21 |
| 042 | 9 | −13 |
| 043 | 8 | −9 |
| 044 | 9 | +14 |
| 045 | 4 | +2 |
| 046 | 5 | +5 |
| 047 | 7 | −8 |
| 048 | <3 | +1 |
| 049 | <3 | −1 |
| 0,4,10 | <3 | −2 |
| 051 | <3 | +1 |
| 052 | 12 | +15 |
| 053 | 6 | +6 |
| 054 | <3 | −1 |
| 055 | 3 | −4 |
| 056 | <3 | +2 |
| 057 | <3 | +1 |
| 058 | <3 | −1 |
| 059 | 2 | +3 |
| 060 | 13 | +20 |
| 061 | <3 | −1 |
| 062 | <3 | −1 |
| 063 | 3 | −5 |
| 064 | 3 | +6 |
| 065 | <3 | −1 |
| 066 | <3 | −3 |
| 067 | <3 | +2 |
| 071 | <3 | 0 |
| 072 | <3 | −2 |
| 073 | <3 | 0 |
| 074 | <3 | −1 |
| 1,1,12 | <2 | −2 |
| 1,1,11 | <4 | +1 |
| 1,1,10 | 5 | +3 |
| 119 | <4 | +2 |
| 118 | <4 | 0 |
| 117 | <4 | +2 |
| 116 | 6 | −5 |
| 115 | 16 | −18 |
| 114 | 13 | +17 |
| 113 | 18 | +17 |
| 112 | 20 | +17 |
| 111 | 30 | −32 |
| 110 | 65 | +77 |
| $11\bar{1}$ | 45 | +50 |
| $11\bar{2}$ | 16 | +16 |
| $11\bar{3}$ | 39 | −31 |
| $11\bar{4}$ | 4 | −2 |
| $11\bar{5}$ | 11 | +8 |
| $11\bar{6}$ | 4 | +5 |
| $11\bar{7}$ | 7 | −6 |
| $11\bar{8}$ | 4 | −3 |
| $11\bar{9}$ | <4 | 0 |
| $1,1,\overline{10}$ | 8 | +7 |
| $1,1,\overline{11}$ | <4 | −1 |
| $1,1,\overline{12}$ | 7 | +7 |
| $1,1,\overline{13}$ | 4 | +5 |
| 1,2,11 | <2 | −1 |
| 1,2,10 | 2 | −2 |
| 129 | 10 | −10 |
| 128 | <3 | +2 |
| 127 | <3 | 0 |
| 126 | 3 | −0 |
| 125 | 3 | +3 |
| 124 | 18 | −22 |
| 123 | 20 | −24 |
| 122 | 25 | −22 |
| 121 | 10 | +10 |
| 120 | 9 | +2 |
| $12\bar{1}$ | 9 | +6 |
| $12\bar{2}$ | 17 | +22 |
| $12\bar{3}$ | 11 | +10 |
| $12\bar{4}$ | 4 | −7 |
| $12\bar{5}$ | <3 | −3 |
| $12\bar{6}$ | 8 | +6 |
| $12\bar{7}$ | 10 | +10 |
| $12\bar{8}$ | 12 | −10 |
| $12\bar{9}$ | 4 | −6 |
| $1,2,\overline{10}$ | <3 | 0 |
| $1,2,\overline{11}$ | <3 | +1 |
| $1,2,\overline{12}$ | <2 | +2 |
| 1,3,11 | <4 | +1 |
| 1,3,10 | <4 | +3 |
| 139 | <4 | −1 |
| 138 | <4 | −1 |
| 137 | 12 | −15 |
| 136 | 7 | −7 |
| 135 | 8 | +9 |
| 134 | 6 | +5 |
| 133 | 5 | −7 |
| 132 | 24 | −26 |
| 131 | 5 | −5 |
| 130 | 14 | −19 |
| $13\bar{1}$ | 4 | +9 |
| $13\bar{2}$ | 12 | +15 |
| $13\bar{3}$ | 24 | −26 |
| $13\bar{4}$ | 2 | +3 |
| $13\bar{5}$ | 7 | −7 |
| $13\bar{6}$ | 5 | +7 |
| $13\bar{7}$ | 15 | −21 |
| $13\bar{8}$ | 8 | +9 |
| $13\bar{9}$ | <4 | −1 |
| $1,3,\overline{10}$ | 5 | −7 |
| $1,3,\overline{11}$ | 6 | −9 |
| $1,3,\overline{12}$ | <3 | +1 |
| 1,4,10 | <3 | 0 |
| 149 | 5 | +6 |
| 148 | <4 | 0 |
| 147 | 7 | +7 |
| 146 | <4 | −1 |
| 145 | 7 | −9 |
| 144 | 5 | +6 |
| 143 | 5 | +1 |
| 142 | 5 | −7 |
| 141 | 10 | −14 |
| 140 | 6 | +10 |
| $14\bar{1}$ | 7 | +9 |
| $14\bar{2}$ | <2 | −2 |
| $14\bar{3}$ | 4 | −6 |
| $14\bar{4}$ | 5 | +7 |
| $14\bar{5}$ | 9 | +11 |
| $14\bar{6}$ | 4 | +6 |
| $14\bar{7}$ | 8 | −10 |
| $14\bar{8}$ | <4 | −1 |
| $14\bar{9}$ | <4 | +1 |
| $1,4,\overline{10}$ | <4 | −1 |
| $1,4,\overline{11}$ | <4 | −2 |
| 159 | <2 | 0 |
| 158 | <4 | −2 |
| 157 | <5 | +1 |
| 156 | 5 | +5 |
| 155 | <6 | −5 |
| 154 | <6 | +6 |
| 153 | <6 | +2 |
| 152 | <6 | +3 |
| 151 | 17 | −24 |
| 150 | <6 | +4 |
| $15\bar{1}$ | <6 | +2 |
| $15\bar{2}$ | <6 | +1 |
| $15\bar{3}$ | 6 | −7 |
| $15\bar{4}$ | 5 | −4 |
| $15\bar{5}$ | <6 | +2 |
| $15\bar{6}$ | <6 | +3 |
| $15\bar{7}$ | <5 | +2 |
| $15\bar{8}$ | <5 | −3 |
| $15\bar{9}$ | <4 | +2 |
| 167 | <3 | −1 |
| 166 | <4 | +2 |
| 165 | <5 | 0 |
| 164 | <6 | −2 |
| 163 | <6 | +3 |
| 162 | <6 | +3 |
| 161 | <6 | −1 |
| 160 | <6 | −4 |
| $16\bar{1}$ | 7 | +6 |
| $16\bar{2}$ | <6 | −2 |
| $16\bar{3}$ | <6 | −2 |

Table 3 (*cont.*)

| *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ | *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ | *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ | *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ | *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ | *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 164̅ | <6 | +1 | 223 | 13 | +15 | 255̅ | <5 | +1 | 327̅ | 4 | +3 | 364 | <4 | −2 | 428 | <3 | −1 |
| 165̅ | 4 | +5 | 222 | 9 | −7 | 256̅ | <5 | −2 | 328̅ | 10 | −12 | 363 | <5 | +2 | 427 | 5 | +6 |
| 166̅ | <4 | −2 | 221 | 19 | +20 | 257̅ | 6 | −7 | 329̅ | 6 | −7 | 362 | <5 | +1 | 426 | <3 | −3 |
| 167̅ | <4 | −1 | 220 | 8 | −3 | 258̅ | <4 | −2 | 3,2,1̅0̅ | <3 | +3 | 361 | <5 | −1 | 425 | 9 | −8 |
| 168̅ | <2 | −2 | 221̅ | 5 | +1 | 259̅ | <4 | −4 | 3,2,1̅1̅ | 3 | −3 | 360 | <5 | −4 | 424 | 8 | −6 |
|  |  |  | 222̅ | 33 | +34 | 2,5,1̅0̅ | <3 | 0 | 3,2,1̅2̅ | 4 | −4 | 361̅ | 6 | +7 | 423 | 4 | +6 |
| 174 | <2 | +2 | 223̅ | 20 | −20 |  |  |  | 3,2,1̅3̅ | 3 | −3 | 362̅ | 9 | +14 | 422 | 16 | −15 |
| 173 | <3 | +2 | 224̅ | 15 | −18 | 266 | <3 | +3 |  |  |  | 363̅ | <5 | −1 | 421 | 4 | +1 |
| 172 | <4 | +1 | 225̅ | 15 | −17 | 265 | <4 | +2 | 3,3,10 | 2 | −6 | 364̅ | <5 | −4 | 420 | 18 | +25 |
| 171 | <4 | +1 | 226̅ | 6 | +6 | 264 | <5 | +2 | 339 | 2 | +2 | 365̅ | <5 | −2 | 421̅ | 15 | +15 |
| 170 | <4 | +2 | 227̅ | 11 | −17 | 263 | <5 | −3 | 338 | 3 | +4 | 366̅ | <4 | +2 | 422̅ | 14 | −13 |
| 171̅ | <4 | 0 | 228̅ | <3 | +1 | 262 | <5 | −2 | 337 | 5 | +7 | 367̅ | <4 | −1 | 423̅ | 3 | −4 |
| 172̅ | <4 | +2 | 229̅ | 7 | +8 | 261 | 6 | −5 | 336 | 7 | −9 | 368̅ | <3 | +2 | 424̅ | 6 | −6 |
| 173̅ | <4 | −3 | 2,2,1̅0̅ | <3 | 0 | 260 | 6 | −6 | 335 | <4 | +1 |  |  |  | 425̅ | 22 | +29 |
| 174̅ | <3 | +3 | 2,2,1̅1̅ | <3 | −2 | 261̅ | 6 | −5 | 334 | 6 | −10 | 372 | <3 | +3 | 426̅ | 8 | −9 |
|  |  |  | 2,2,1̅2̅ | <3 | −2 | 262̅ | <5 | 0 | 333 | 3 | +4 | 371 | <3 | −5 | 427̅ | 12 | −13 |
| 2,0,11 | 5 | −5 |  |  |  | 263̅ | 9 | +13 | 332 | 3 | −4 | 370 | <4 | −3 | 428̅ | 21 | −28 |
| 2,0,10 | <4 | +2 | 2,3,10 | <4 | +1 | 264̅ | 5 | +4 | 331 | 20 | +25 | 371̅ | <4 | 0 | 429̅ | 16 | +19 |
| 209 | 13 | +16 | 239 | <4 | +1 | 265̅ | 5 | +5 | 330 | 2 | +1 | 372̅ | <4 | −3 | 4,2,1̅0̅ | <3 | −3 |
| 208 | 6 | +4 | 238 | <4 | −2 | 266̅ | <5 | +2 | 331̅ | 17 | −25 | 373̅ | 6 | +8 | 4,2,1̅1̅ | <3 | −1 |
| 207 | 4 | −4 | 237 | 4 | +3 | 267̅ | <4 | +6 | 332̅ | 6 | −7 | 374̅ | 5 | +5 | 4,2,1̅2̅ | <3 | −2 |
| 206 | <4 | +1 | 236 | <4 | +2 | 268̅ | <3 | +1 | 333̅ | 13 | −16 | 375̅ | <3 | +3 | 4,2,1̅3̅ | 5 | +6 |
| 205 | 5 | −3 | 235 | 10 | −12 |  |  |  | 334̅ | 12 | −14 |  |  |  |  |  |  |
| 204 | 8 | +5 | 234 | 4 | −4 | 273 | <3 | +4 | 335̅ | 5 | −8 | 4,0,10 | 6 | −7 | 439 | 2 | −0 |
| 203 | 7 | +2 | 233 | <4 | −2 | 272 | <3 | −3 | 336̅ | 3 | −3 | 409 | 11 | −14 | 438 | <4 | +2 |
| 202 | 20 | +20 | 232 | 7 | −5 | 271 | <4 | −2 | 337̅ | <4 | 0 | 408 | 13 | −14 | 437 | <4 | −2 |
| 201 | 20 | −30 | 231 | 13 | −16 | 270 | <4 | 0 | 338̅ | 6 | −7 | 407 | 5 | +5 | 436 | <4 | −1 |
| 200 | 3 | −5 | 230 | <2 | +3 | 271̅ | <4 | 0 | 339̅ | 11 | +12 | 406 | <4 | −1 | 435 | 8 | −10 |
| 201̅ | 24 | +25 | 231̅ | <2 | −2 | 272̅ | <4 | 0 | 3,3,1̅0̅ | 5 | −7 | 405 | <4 | −2 | 434 | 7 | −8 |
| 202̅ | 9 | −10 | 232̅ | 6 | +6 | 273̅ | <3 | −3 | 3,3,1̅1̅ | <4 | +2 | 404 | <4 | −6 | 433 | <4 | +2 |
| 203̅ | 85 | +106 | 233̅ | 4 | +3 | 274̅ | <3 | −3 | 3,3,1̅2̅ | 2 | −3 | 403 | 29 | +35 | 432 | <4 | −2 |
| 204̅ | 35 | +37 | 234̅ | 8 | −7 | 275̅ | <2 | +1 |  |  |  | 402 | 20 | +13 | 431 | 9 | −11 |
| 205̅ | 18 | +14 | 235̅ | 5 | +3 |  |  |  | 349 | 3 | +4 | 401 | 13 | +9 | 430 | 6 | −9 |
| 206̅ | 24 | −20 | 236̅ | 12 | +16 | 3,1,11 | <2 | +2 | 348 | 3 | +3 | 400 | 23 | −20 | 431̅ | 7 | +9 |
| 207̅ | 22 | +20 | 237̅ | 5 | +4 | 3,1,10 | 8 | −8 | 347 | 3 | −5 | 401̅ | 81 | +106 | 432̅ | <2 | −3 |
| 208̅ | 7 | −5 | 238̅ | 10 | −11 | 319 | <4 | +1 | 346 | 6 | +9 | 402̅ | 34 | −34 | 433̅ | 13 | +13 |
| 209̅ | 11 | −14 | 239̅ | <4 | −2 | 318 | <4 | +1 | 345 | 5 | −5 | 403̅ | 44 | −45 | 434̅ | 4 | +6 |
| 2,0,1̅0̅ | 8 | +8 | 2,3,1̅0̅ | <4 | −2 | 317 | 6 | +3 | 344 | <3 | −1 | 404̅ | 34 | −31 | 435̅ | 4 | −4 |
| 2,0,1̅1̅ | 4 | +1 | 2,3,1̅1̅ | <4 | −1 | 316 | 15 | −19 | 343 | 3 | −4 | 405̅ | 30 | +28 | 436̅ | 4 | −8 |
| 2,0,1̅2̅ | 5 | +4 | 2,3,1̅2̅ | <4 | −1 | 315 | 8 | −7 | 342 | 2 | +2 | 406̅ | 7 | −3 | 437̅ | <4 | −2 |
| 2,0,1̅3̅ | 3 | +1 |  |  |  | 314 | 9 | +8 | 341 | 3 | −1 | 407̅ | 13 | −8 | 438̅ | 7 | −7 |
|  |  |  | 249 | <3 | +2 | 313 | 25 | +22 | 340 | <2 | −1 | 408̅ | 15 | +15 | 439̅ | 4 | −3 |
| 2,1,11 | 4 | −6 | 248 | <3 | 0 | 312 | 16 | −18 | 341̅ | 11 | +16 | 409̅ | 9 | +6 | 4,3,1̅0̅ | 4 | +4 |
| 2,1,10 | 6 | −5 | 247 | 6 | −8 | 311 | 6 | +6 | 342̅ | 5 | +5 | 4,0,1̅0̅ | 19 | +18 | 4,3,1̅1̅ | <4 | −2 |
| 219 | 8 | −7 | 246 | 3 | −3 | 310 | 14 | +15 | 343̅ | 13 | +16 | 4,0,1̅1̅ | 9 | +8 | 4,3,1̅2̅ | <3 | −3 |
| 218 | 4 | +6 | 245 | 4 | +4 | 311̅ | 44 | +48 | 344̅ | <3 | 0 | 4,0,1̅2̅ | 7 | −7 | 4,3,1̅3̅ | <2 | 0 |
| 217 | <4 | +1 | 244 | 4 | −4 | 312̅ | 32 | −35 | 345̅ | 7 | −7 | 4,0,1̅3̅ | <3 | +1 |  |  |  |
| 216 | 14 | −17 | 243 | <3 | 0 | 313̅ | 41 | +41 | 346̅ | 4 | −5 | 4,0,1̅4̅ | 5 | +4 | 448 | 5 | +6 |
| 215 | 15 | +18 | 242 | 4 | +2 | 314̅ | 35 | +31 | 347̅ | <3 | +2 |  |  |  | 447 | 5 | +7 |
| 214 | <3 | 0 | 241 | 4 | +4 | 315̅ | 14 | +20 | 348̅ | 3 | −1 | 4,1,10 | <3 | +3 | 446 | <3 | −4 |
| 213 | 4 | −2 | 240 | 9 | −7 | 316̅ | 34 | −36 | 349̅ | <3 | −2 | 419 | <4 | 0 | 445 | <3 | +1 |
| 212 | 24 | −23 | 241̅ | 4 | +4 | 317̅ | 10 | −11 | 3,4,1̅0̅ | 3 | −4 | 418 | 9 | −9 | 444 | 7 | −10 |
| 211 | 19 | +13 | 242̅ | 2 | −1 | 318̅ | 4 | +3 | 3,4,1̅1̅ | <3 | +1 | 417 | <4 | +1 | 443 | <3 | 0 |
| 210 | 19 | −19 | 243̅ | 11 | −11 | 319̅ | 7 | +5 |  |  |  | 416 | 16 | −21 | 442 | 6 | −5 |
| 211̅ | 29 | −36 | 244̅ | 14 | −18 | 3,1,1̅0̅ | 4 | +4 | 358 | <2 | +5 | 415 | 11 | −9 | 441 | 2 | +4 |
| 212̅ | 29 | −32 | 245̅ | 14 | −15 | 3,1,1̅1̅ | <4 | +1 | 357 | <4 | +3 | 414 | 3 | −3 | 440 | 7 | −10 |
| 213̅ | 31 | −31 | 246̅ | <4 | −3 | 3,1,1̅2̅ | <4 | 0 | 356 | <5 | −4 | 413 | 14 | −17 | 441̅ | 4 | +8 |
| 214̅ | 6 | +7 | 247̅ | <4 | +2 | 3,1,1̅3̅ | 4 | +3 | 355 | <5 | +1 | 412 | 7 | +2 | 442̅ | 4 | −3 |
| 215̅ | 19 | +19 | 248̅ | <4 | +1 |  |  |  | 354 | <5 | 0 | 411 | 7 | +7 | 443̅ | 7 | −8 |
| 216̅ | 8 | +8 | 249̅ | <4 | +2 | 3,2,10 | <2 | 0 | 353 | <5 | +3 | 410 | 23 | +17 | 444̅ | 4 | +1 |
| 217̅ | 11 | +11 | 2,4,1̅0̅ | <3 | 0 | 329 | 3 | −2 | 352 | 5 | −7 | 411̅ | 47 | −50 | 445̅ | 15 | +20 |
| 218̅ | 6 | +6 | 2,4,1̅1̅ | 4 | −5 | 328 | <3 | +1 | 351 | <5 | +1 | 412̅ | 31 | +34 | 446̅ | 3 | −3 |
| 219̅ | 6 | −5 |  |  |  | 327 | 6 | +7 | 350 | 6 | −4 | 413̅ | 25 | +21 | 447̅ | 11 | −12 |
| 2,1,1̅0̅ | <4 | −1 | 258 | <3 | +1 | 326 | 18 | −20 | 351̅ | 4 | +10 | 414̅ | 10 | −15 | 448̅ | 13 | −14 |
| 2,1,1̅1̅ | 4 | −4 | 257 | <4 | 0 | 325 | 10 | −11 | 352̅ | 11 | −19 | 415̅ | 37 | −27 | 449̅ | <3 | +2 |
| 2,1,1̅2̅ | <4 | +1 | 256 | <5 | +3 | 324 | 7 | −9 | 353̅ | <5 | +3 | 416̅ | 16 | −19 | 4,4,1̅0̅ | 3 | −2 |
| 2,1,1̅3̅ | <3 | +1 | 255 | 5 | −4 | 323 | 4 | +4 | 354̅ | 5 | +4 | 417̅ | 7 | −8 | 4,4,1̅1̅ | <3 | 0 |
|  |  |  | 254 | <5 | −4 | 322 | 7 | −7 | 355̅ | 5 | +9 | 418̅ | 7 | −6 | 4,4,1̅2̅ | <3 | −3 |
| 2,2,11 | <2 | −1 | 253 | <5 | −1 | 321 | 16 | −22 | 356̅ | 5 | −9 | 419̅ | 3 | −2 |  |  |  |
| 2,2,10 | 3 | −3 | 252 | <5 | +2 | 320 | 21 | −24 | 357̅ | <5 | 0 | 4,1,1̅0̅ | <4 | +1 | 457 | 6 | +8 |
| 229 | 3 | +3 | 251 | 6 | −7 | 321̅ | 34 | −36 | 358̅ | <5 | +4 | 4,1,1̅1̅ | 6 | +5 | 456 | 8 | +8 |
| 228 | <3 | 0 | 250 | <5 | −4 | 322̅ | 12 | +11 | 359̅ | <4 | −2 | 4,1,1̅2̅ | <4 | +1 | 455 | <5 | 0 |
| 227 | 3 | −2 | 251̅ | 21 | +25 | 323̅ | 5 | −1 | 3,5,1̅0̅ | <3 | 0 | 4,1,1̅3̅ | 3 | −3 | 454 | <5 | −2 |
| 226 | 18 | −22 | 252̅ | 15 | +19 | 324̅ | 2 | +0 |  |  |  |  |  |  | 453 | <5 | +2 |
| 225 | 2 | +0 | 253̅ | 5 | +3 | 325̅ | 13 | +12 | 366 | <2 | −1 | 4,2,10 | <2 | 0 | 452 | <5 | +1 |
| 224 | <3 | +3 | 254̅ | <5 | −2 | 326̅ | 14 | +17 | 365 | <4 | +2 | 429 | <3 | 0 | 451 | <5 | 0 |

Table 3 (*cont.*)

| *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ |
|---|---|---|
| 450 | 7 | −8 |
| $45\bar{1}$ | 6 | +4 |
| $45\bar{2}$ | 7 | +6 |
| $45\bar{3}$ | <5 | 0 |
| $45\bar{4}$ | 8 | +7 |
| $45\bar{5}$ | 11 | +10 |
| $45\bar{6}$ | 9 | +9 |
| $45\bar{7}$ | <5 | −1 |
| $45\bar{8}$ | <5 | +3 |
| $45\bar{9}$ | <4 | 0 |
| $4.5.\overline{10}$ | 5 | −6 |
| 465 | <3 | −1 |
| 464 | <4 | +1 |
| 463 | 5 | +8 |
| 462 | <5 | 0 |
| 461 | <5 | −2 |
| 460 | 5 | −5 |
| $46\bar{1}$ | <5 | +6 |
| $46\bar{2}$ | 7 | −9 |
| $46\bar{3}$ | <5 | −2 |
| $46\bar{4}$ | <5 | −4 |
| $46\bar{5}$ | 6 | +6 |
| $46\bar{6}$ | 4 | +3 |
| $46\bar{7}$ | 4 | +3 |
| $46\bar{8}$ | 4 | +3 |
| 471 | <3 | +1 |
| 470 | <3 | +3 |
| $47\bar{1}$ | <3 | −4 |
| $47\bar{2}$ | <3 | +3 |
| $47\bar{3}$ | <3 | +4 |
| $47\bar{4}$ | <3 | −1 |
| $47\bar{5}$ | <3 | −4 |
| 5.1.10 | <2 | 0 |
| 519 | <3 | −1 |
| 518 | 7 | −6 |
| 517 | 6 | −5 |
| 516 | <4 | +1 |
| 515 | <4 | 0 |
| 514 | <4 | −3 |
| 513 | 5 | −3 |
| 512 | 4 | −1 |
| 511 | 7 | +10 |
| 510 | 10 | +8 |
| $51\bar{1}$ | 30 | +32 |
| $51\bar{2}$ | 6 | −4 |
| $51\bar{3}$ | 27 | −26 |
| $51\bar{4}$ | 33 | −32 |
| $51\bar{5}$ | 3 | +1 |
| $51\bar{6}$ | 16 | +20 |
| $51\bar{7}$ | 10 | +12 |
| $51\bar{8}$ | <4 | −1 |
| $51\bar{9}$ | 5 | −4 |
| $5.1.\overline{10}$ | 7 | +3 |
| $5.1.\overline{11}$ | 11 | +10 |
| $5.1.\overline{12}$ | 3 | −3 |
| $5.1.\overline{13}$ | 3 | +2 |
| $5.1.\overline{14}$ | <2 | 0 |
| 529 | <3 | −2 |
| 528 | 3 | −5 |
| 527 | <3 | +3 |
| 526 | 6 | −7 |
| 525 | 10 | −9 |
| 524 | 3 | +6 |
| 523 | 13 | −15 |
| 522 | 9 | −14 |
| 521 | 15 | −17 |
| 520 | 18 | +24 |
| $52\bar{1}$ | 3 | −6 |
| $52\bar{2}$ | <3 | −4 |
| $52\bar{3}$ | 2 | +3 |
| $52\bar{4}$ | 20 | −22 |
| $52\bar{5}$ | 16 | −18 |
| $52\bar{6}$ | 18 | −23 |
| $52\bar{7}$ | 7 | −7 |
| $52\bar{8}$ | 3 | −5 |
| $52\bar{9}$ | 3 | +5 |
| $5.2.\overline{10}$ | 4 | +4 |
| $5.2.\overline{11}$ | <3 | −2 |
| $5.2.\overline{12}$ | <3 | −2 |
| $5.2.\overline{13}$ | <3 | 0 |
| 539 | <3 | +1 |
| 538 | 3 | +2 |
| 537 | <4 | −3 |
| 536 | <4 | −2 |
| 535 | <4 | +1 |
| 534 | <4 | 0 |
| 533 | <4 | +3 |
| 532 | <4 | 0 |
| 531 | 7 | −7 |
| 530 | <4 | 0 |
| $53\bar{1}$ | 10 | +11 |
| $53\bar{2}$ | 19 | +23 |
| $53\bar{3}$ | 12 | +10 |
| $53\bar{4}$ | 19 | −24 |
| $53\bar{5}$ | 6 | +7 |
| $53\bar{6}$ | <4 | 0 |
| $53\bar{7}$ | 6 | −6 |
| $53\bar{8}$ | 31 | −37 |
| $53\bar{9}$ | <4 | +2 |
| $5.3.\overline{10}$ | 4 | +6 |
| $5.3.\overline{11}$ | <4 | +1 |
| $5.3.\overline{12}$ | <4 | −2 |
| $5.3.\overline{13}$ | <3 | +1 |
| 548 | 2 | +2 |
| 547 | <3 | 0 |
| 546 | 6 | +8 |
| 545 | <3 | +2 |
| 544 | 12 | −17 |
| 543 | <3 | +1 |
| 542 | <3 | +3 |
| 541 | 3 | +6 |
| 540 | 14 | −19 |
| $54\bar{1}$ | 5 | +9 |
| $54\bar{2}$ | 9 | +10 |
| $54\bar{3}$ | 5 | +5 |
| $54\bar{4}$ | 13 | +13 |
| $54\bar{5}$ | 5 | +7 |
| $54\bar{6}$ | 11 | +12 |
| $54\bar{7}$ | <3 | 0 |
| $54\bar{8}$ | 5 | −5 |
| $54\bar{9}$ | 10 | −10 |
| $5.4.\overline{10}$ | <3 | −2 |
| $5.4.\overline{11}$ | <3 | −3 |
| $5.4.\overline{12}$ | 3 | −2 |
| 556 | <3 | +1 |
| 555 | 6 | +7 |
| 554 | 4 | +2 |
| 553 | 4 | −3 |
| 552 | <5 | −2 |
| 551 | <5 | +3 |
| 550 | 6 | −8 |
| $55\bar{1}$ | 4 | −2 |
| $55\bar{2}$ | <5 | +1 |
| $55\bar{3}$ | 4 | −2 |
| $55\bar{4}$ | <5 | 0 |
| $55\bar{5}$ | <5 | +1 |
| $55\bar{6}$ | 5 | +6 |
| $55\bar{7}$ | <5 | 0 |
| $55\bar{8}$ | <5 | +2 |
| $55\bar{9}$ | <5 | −4 |
| $5.5.\overline{10}$ | <4 | −2 |
| $5.5.\overline{11}$ | <3 | +4 |
| 564 | <3 | 0 |
| 563 | <4 | −1 |
| 562 | 4 | +5 |
| 561 | <5 | +1 |
| 560 | <5 | +3 |
| $56\bar{1}$ | <5 | −4 |
| $56\bar{2}$ | 5 | +3 |
| $56\bar{3}$ | 4 | +1 |
| $56\bar{4}$ | 4 | +3 |
| $56\bar{5}$ | 7 | +9 |
| $56\bar{6}$ | 5 | +6 |
| $56\bar{7}$ | <4 | −1 |
| $56\bar{8}$ | <4 | −4 |
| $56\bar{9}$ | <3 | +2 |
| 571 | <2 | −3 |
| 570 | <3 | −1 |
| $57\bar{1}$ | <3 | +1 |
| $57\bar{2}$ | 4 | −6 |
| $57\bar{3}$ | <3 | −1 |
| $57\bar{4}$ | <3 | −3 |
| $57\bar{5}$ | <2 | +3 |
| 609 | <3 | 0 |
| 608 | 4 | +5 |
| 607 | 5 | −4 |
| 606 | 13 | −16 |
| 605 | 30 | −30 |
| 604 | 5 | +4 |
| 603 | <4 | −3 |
| 602 | <3 | −2 |
| 601 | 7 | −8 |
| 600 | 33 | +32 |
| $60\bar{1}$ | 36 | +36 |
| $60\bar{2}$ | 26 | +27 |
| $60\bar{3}$ | 37 | −41 |
| $60\bar{4}$ | 2 | +5 |
| $60\bar{5}$ | 6 | −9 |
| $60\bar{6}$ | 2 | +2 |
| $60\bar{7}$ | 22 | −23 |
| $60\bar{8}$ | 5 | +7 |
| $60\bar{9}$ | 6 | −5 |
| $6.0.\overline{10}$ | 7 | −10 |
| $6.0.\overline{11}$ | 18 | +14 |
| $6.0.\overline{12}$ | 7 | +6 |
| $6.0.\overline{13}$ | 8 | +7 |
| $6.0.\overline{14}$ | <3 | +1 |
| 619 | <3 | −1 |
| 618 | <4 | −1 |
| 617 | 7 | +9 |
| 616 | 9 | +10 |
| 615 | 13 | −15 |
| 614 | <4 | 0 |
| 613 | 13 | −19 |
| 612 | 15 | −16 |
| 611 | 23 | −28 |
| 610 | 7 | +5 |
| $61\bar{1}$ | 5 | −4 |
| $61\bar{2}$ | 11 | −7 |
| $61\bar{3}$ | 35 | +32 |
| $61\bar{4}$ | 35 | −34 |
| $61\bar{5}$ | 7 | −2 |
| $61\bar{6}$ | 5 | +7 |
| $61\bar{7}$ | 10 | +11 |
| $61\bar{8}$ | 20 | −21 |
| $61\bar{9}$ | 13 | −11 |
| $6.1.\overline{10}$ | <4 | −1 |
| $6.1.\overline{11}$ | 7 | −6 |
| $6.1.\overline{12}$ | 3 | −3 |
| $6.1.\overline{13}$ | <4 | −1 |
| $6.1.\overline{14}$ | <3 | +2 |
| 629 | <3 | −1 |
| 628 | 3 | +3 |
| 627 | 5 | −6 |
| 626 | <3 | −1 |
| 625 | <3 | +1 |
| 624 | 6 | +7 |
| 623 | 6 | +6 |
| 622 | 3 | +3 |
| 621 | 10 | −11 |
| 620 | 4 | +5 |
| $62\bar{1}$ | 5 | +3 |
| $62\bar{2}$ | 6 | +4 |
| $62\bar{3}$ | 9 | +10 |
| $62\bar{4}$ | 7 | −9 |
| $62\bar{5}$ | 19 | −22 |
| $62\bar{6}$ | 3 | −4 |
| $62\bar{7}$ | 15 | +16 |
| $62\bar{8}$ | <3 | −1 |
| $62\bar{9}$ | 11 | −10 |
| $6.2.\overline{10}$ | 3 | +4 |
| $6.2.\overline{11}$ | <3 | −2 |
| $6.2.\overline{12}$ | 3 | +4 |
| $6.2.\overline{13}$ | <3 | −1 |
| $6.2.\overline{14}$ | 2 | +2 |
| 638 | <4 | +1 |
| 637 | <4 | +1 |
| 636 | <4 | 0 |
| 635 | <4 | +4 |
| 634 | 7 | −9 |
| 633 | 9 | −11 |
| 632 | 7 | −9 |
| 631 | <4 | 0 |
| 630 | 4 | −4 |
| $63\bar{1}$ | 3 | +6 |
| $63\bar{2}$ | 7 | +7 |
| $63\bar{3}$ | 3 | −2 |
| $63\bar{4}$ | 5 | +5 |
| $63\bar{5}$ | 4 | −5 |
| $63\bar{6}$ | 2 | −0 |
| $63\bar{7}$ | <4 | +1 |
| $63\bar{8}$ | <4 | −1 |
| $63\bar{9}$ | 13 | −14 |
| $6.3.\overline{10}$ | 3 | −5 |
| $6.3.\overline{11}$ | <4 | +1 |
| $6.3.\overline{12}$ | <4 | −1 |
| $6.3.\overline{13}$ | <4 | −1 |
| 647 | <2 | +1 |
| 646 | <3 | −1 |
| 645 | 6 | +6 |
| 644 | 12 | +16 |
| 643 | <4 | +1 |
| 642 | 3 | −3 |
| 641 | 5 | −8 |
| 640 | <3 | −1 |
| $64\bar{1}$ | 9 | −10 |
| $64\bar{2}$ | 9 | +13 |
| $64\bar{3}$ | <3 | +1 |
| $64\bar{4}$ | 5 | −5 |
| $64\bar{5}$ | 3 | −5 |
| $64\bar{6}$ | <3 | +2 |
| $64\bar{7}$ | 5 | +3 |
| $64\bar{8}$ | <4 | +3 |
| $64\bar{9}$ | <4 | +1 |
| $6.4.\overline{10}$ | 4 | −3 |
| $6.4.\overline{11}$ | 3 | −3 |
| $6.4.\overline{12}$ | 2 | −2 |
| 656 | <3 | 0 |
| 655 | <4 | +3 |
| 654 | <5 | −1 |
| 653 | 4 | +4 |
| 652 | 6 | +5 |
| 651 | 7 | +8 |
| 650 | <5 | +1 |
| $65\bar{1}$ | <5 | 0 |
| $65\bar{2}$ | 10 | +8 |
| $65\bar{3}$ | <5 | +2 |
| $65\bar{4}$ | 5 | +3 |
| $65\bar{5}$ | <5 | +1 |
| $65\bar{6}$ | 6 | +5 |
| $65\bar{7}$ | <5 | 0 |
| $65\bar{8}$ | <5 | −2 |
| $65\bar{9}$ | <5 | +2 |
| $6.5.\overline{10}$ | <4 | −1 |
| $6.5.\overline{11}$ | 3 | +5 |
| 664 | <3 | +1 |
| 663 | <3 | −1 |
| 662 | 3 | −3 |
| 661 | 4 | −5 |
| 660 | <5 | +3 |
| $66\bar{1}$ | 4 | +4 |
| $66\bar{2}$ | 4 | −2 |
| $66\bar{3}$ | 4 | −3 |
| $66\bar{4}$ | 4 | +6 |
| $66\bar{5}$ | <5 | −1 |
| $66\bar{6}$ | <5 | +2 |
| $66\bar{7}$ | <4 | 0 |
| $66\bar{8}$ | 3 | +4 |
| $66\bar{9}$ | <3 | −3 |
| 670 | <3 | −2 |
| $67\bar{1}$ | <3 | 0 |
| $67\bar{2}$ | <3 | +1 |
| $67\bar{3}$ | <3 | +4 |
| $67\bar{4}$ | <3 | −4 |
| $67\bar{5}$ | <3 | +1 |
| 718 | <3 | +1 |
| 717 | <4 | −2 |
| 716 | <4 | −2 |
| 715 | 22 | −24 |
| 714 | 7 | −5 |
| 713 | 8 | +10 |
| 712 | 18 | +23 |
| 711 | 11 | −14 |
| 710 | 5 | −2 |
| $71\bar{1}$ | 6 | +9 |
| $71\bar{2}$ | 33 | +30 |
| $71\bar{3}$ | 21 | −17 |
| $71\bar{4}$ | 2 | +2 |
| $71\bar{5}$ | 18 | −19 |
| $71\bar{6}$ | 9 | −9 |
| $71\bar{7}$ | 33 | −31 |
| $71\bar{8}$ | 3 | −2 |
| $71\bar{9}$ | 3 | +3 |
| $7.1.\overline{10}$ | 9 | +8 |
| $7.1.\overline{11}$ | 7 | +6 |
| $7.1.\overline{12}$ | 7 | +6 |
| $7.1.\overline{13}$ | 4 | +6 |
| $7.1.\overline{14}$ | 5 | +4 |
| 728 | <3 | +2 |
| 727 | <3 | +1 |
| 726 | 3 | +3 |
| 725 | 7 | −9 |
| 724 | 6 | −6 |
| 723 | 12 | −16 |
| 722 | 12 | −14 |
| 721 | 3 | −3 |
| 720 | <3 | +2 |
| $72\bar{1}$ | 3 | +0 |
| $72\bar{2}$ | 15 | −19 |
| $72\bar{3}$ | 26 | +35 |
| $72\bar{4}$ | 9 | +7 |
| $72\bar{5}$ | 6 | −6 |
| $72\bar{6}$ | 5 | −7 |
| $72\bar{7}$ | <3 | +2 |
| $72\bar{8}$ | 13 | −13 |
| $72\bar{9}$ | 27 | −32 |
| $7.2.\overline{10}$ | 11 | −13 |
| $7.2.\overline{11}$ | 5 | −5 |
| $7.2.\overline{12}$ | 3 | +3 |
| $7.2.\overline{13}$ | 2 | +3 |
| $7.2.\overline{14}$ | 2 | +2 |
| 737 | 4 | +6 |
| 736 | <4 | +1 |
| 735 | 3 | +2 |
| 734 | 6 | +7 |
| 733 | 6 | +5 |
| 732 | 4 | +4 |
| 731 | 11 | −15 |
| 730 | <4 | +3 |
| $73\bar{1}$ | <4 | −2 |
| $73\bar{2}$ | 3 | +7 |
| $73\bar{3}$ | 7 | −7 |
| $73\bar{4}$ | 5 | +6 |
| $73\bar{5}$ | 6 | +6 |
| $73\bar{6}$ | 8 | +11 |
| $73\bar{7}$ | <4 | +1 |
| $73\bar{8}$ | 8 | +11 |
| $73\bar{9}$ | <4 | 0 |
| $7.3.\overline{10}$ | 3 | +4 |
| $7.3.\overline{11}$ | 19 | −21 |
| $7.3.\overline{12}$ | 4 | −4 |
| $7.3.\overline{13}$ | <4 | +2 |
| 746 | 3 | −5 |
| 745 | <3 | +4 |
| 744 | <4 | −2 |
| 743 | 3 | +3 |
| 742 | 4 | +3 |
| 741 | <4 | −2 |
| 740 | 5 | −3 |
| $74\bar{1}$ | <4 | 0 |
| $74\bar{2}$ | 17 | +21 |
| $74\bar{3}$ | 6 | −12 |
| $74\bar{4}$ | <4 | +1 |
| $74\bar{5}$ | <4 | 0 |
| $74\bar{6}$ | <4 | +4 |
| $74\bar{7}$ | 3 | +1 |
| $74\bar{8}$ | 5 | +3 |
| $74\bar{9}$ | 5 | +5 |
| $7.4.\overline{10}$ | 3 | −3 |
| $7.4.\overline{11}$ | 3 | +3 |
| $7.4.\overline{12}$ | <4 | −1 |
| 755 | 4 | −8 |
| 754 | <3 | +3 |
| 753 | <4 | +1 |
| 752 | 3 | +5 |
| 751 | 4 | −6 |
| 750 | <4 | −1 |
| $75\bar{1}$ | 4 | −4 |
| $75\bar{2}$ | 5 | +10 |
| $75\bar{3}$ | <4 | −3 |
| $75\bar{4}$ | <4 | −1 |
| $75\bar{5}$ | <4 | −2 |
| $75\bar{6}$ | 4 | +6 |
| $75\bar{7}$ | <4 | −3 |
| $75\bar{8}$ | <4 | +1 |
| $75\bar{9}$ | <3 | +4 |
| $7.5.\overline{10}$ | <3 | −2 |
| $7.5.\overline{11}$ | <3 | −1 |
| 763 | <2 | +1 |
| 762 | <3 | +1 |
| 761 | <3 | +1 |
| 760 | <3 | 0 |
| $76\bar{1}$ | <3 | +2 |
| $76\bar{2}$ | 3 | +1 |
| $76\bar{3}$ | 6 | +9 |
| $76\bar{4}$ | <3 | −2 |
| $76\bar{5}$ | 3 | −4 |
| $76\bar{6}$ | <3 | −1 |
| $76\bar{7}$ | 4 | +6 |
| $76\bar{8}$ | 3 | +4 |
| $76\bar{9}$ | <2 | +1 |
| $77\bar{1}$ | <2 | +2 |
| $77\bar{2}$ | <2 | −1 |

Table 3 (*cont.*)

| *hkl* | $F_{obs.}$ | $F_{calc.}$ | *hkl* | $F_{obs.}$ | $F_{calc.}$ | *hkl* | $F_{obs.}$ | $F_{calc.}$ | *hkl* | $F_{obs.}$ | $F_{calc.}$ | *hkl* | $F_{obs.}$ | $F_{calc.}$ | *hkl* | $F_{obs.}$ | $F_{calc.}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $77\bar{3}$ | <2 | −4 | 837 | <4 | +2 | 915 | <4 | +2 | $94\bar{4}$ | <4 | +2 | $10.1.\bar{7}$ | 3 | −2 | $10.5.\bar{1}$ | <4 | −3 |
| $77\bar{4}$ | 2 | +6 | 836 | <4 | −2 | 914 | 7 | −7 | $94\bar{5}$ | 7 | +9 | $10.1.\bar{8}$ | 15 | +15 | $10.5.\bar{2}$ | <4 | −1 |
| $77\bar{5}$ | <2 | −1 | 835 | 3 | −3 | 913 | <4 | −3 | $94\bar{6}$ | 7 | −10 | $10.1.\bar{9}$ | 9 | −10 | $10.5.\bar{3}$ | <4 | +1 |
|  |  |  | 834 | <4 | −1 | 912 | 10 | −12 | $94\bar{7}$ | 4 | −4 | $10.1.\overline{10}$ | 12 | −11 | $10.5\ \bar{4}$ | <4 | +2 |
| 808 | 2 | −3 | 833 | 3 | −4 | 911 | 4 | −3 | $94\bar{8}$ | 6 | −7 | $10.1.\overline{11}$ | 5 | +4 | $10.5.\bar{5}$ | 6 | +8 |
| 807 | 4 | −6 | 832 | <4 | +3 | 910 | 4 | −2 | $94\bar{9}$ | 4 | +4 | $10.1.\overline{12}$ | 11 | −11 | $10\ 5.\bar{6}$ | <4 | +1 |
| 806 | 9 | +9 | 831 | <4 | 0 | $91\bar{1}$ | 9 | +8 | $9.4.\overline{10}$ | 6 | −7 | $10.1.\overline{13}$ | <4 | −2 | $10.5.\bar{7}$ | <4 | +1 |
| 805 | 9 | −8 | 830 | 5 | −5 | $91\bar{2}$ | <3 | +1 | $9.4.\overline{11}$ | <4 | −2 | $10.1.\overline{14}$ | 6 | −4 | $10.5.\bar{8}$ | 4 | −4 |
| 804 | 7 | −5 | $83\bar{1}$ | 7 | −12 | $91\bar{3}$ | <3 | −4 | $9.4.\overline{12}$ | 5 | +7 | $10.1.\overline{15}$ | <3 | +1 | $10.5.\bar{9}$ | <4 | −3 |
| 803 | <4 | −2 | $83\bar{2}$ | 6 | +10 | $91\bar{4}$ | <3 | −3 | $9.4.\overline{13}$ | 2 | +3 |  |  |  | $10.5.\overline{10}$ | <4 | +1 |
| 802 | <4 | +1 | $83\bar{3}$ | 7 | +7 | $91\bar{5}$ | 7 | −6 |  |  |  | 10.2.6 | 2 | −4 | $10.5.\overline{11}$ | <3 | +2 |
| 801 | 4 | −6 | $83\bar{4}$ | 3 | +4 | $91\bar{6}$ | <3 | 0 | 954 | <2 | −1 | 10.2.5 | 2 | −2 |  |  |  |
| 800 | 18 | −16 | $83\bar{5}$ | <4 | +1 | $91\bar{7}$ | 3 | −2 | 953 | <3 | +4 | 10.2.4 | 3 | +5 | 10.6.0 | 3 | −5 |
| $80\bar{1}$ | 19 | −19 | $83\bar{6}$ | 6 | −5 | $91\bar{8}$ | 11 | −10 | 952 | 4 | −5 | 10.2.3 | <3 | +2 | $10.6.\bar{1}$ | <3 | +2 |
| $80\bar{2}$ | <3 | +6 | $83\bar{7}$ | <4 | −2 | $91\bar{9}$ | 12 | −12 | 951 | <4 | +1 | 10.2.2 | <3 | −2 | $10.6.\bar{2}$ | <3 | 0 |
| $80\bar{3}$ | 12 | +8 | $83\bar{8}$ | <4 | −4 | $9.1.\overline{10}$ | 6 | −3 | 950 | <5 | 0 | 10.2.1 | <3 | −2 | $10.6.\bar{3}$ | 4 | −5 |
| $80\bar{4}$ | 6 | +6 | $83\bar{9}$ | 5 | −5 | $9.1.\overline{11}$ | 7 | +5 | $95\bar{1}$ | <5 | +2 | 10.2.0 | <3 | −1 | $10.6.\bar{4}$ | <4 | −3 |
| $80\bar{5}$ | 18 | −18 | $8.3.\overline{10}$ | 8 | −10 | $9.1.\overline{12}$ | 3 | +3 | $95\bar{2}$ | 5 | −5 | $10.2.\bar{1}$ | 10 | +13 | $10.6.\bar{5}$ | <4 | +3 |
| $80\bar{6}$ | 7 | −7 | $8.3.\overline{11}$ | <4 | +1 | $9.1.\overline{13}$ | 7 | −6 | $95\bar{3}$ | <5 | +2 | $10.2.\bar{2}$ | 5 | +5 | $10.6.\bar{6}$ | <4 | 0 |
| $80\bar{7}$ | 23 | −24 | $8.3.\overline{12}$ | <4 | 0 | $9.1.\overline{14}$ | 3 | +2 | $95\bar{4}$ | <5 | +2 | $10.2.\bar{3}$ | <3 | 0 | $10.6.\bar{7}$ | <3 | +3 |
| $80\bar{8}$ | 10 | −10 | $8.3.\overline{13}$ | <4 | −1 | $9.1.\overline{15}$ | 5 | +5 | $95\bar{5}$ | 4 | +4 | $10.2.\bar{4}$ | 16 | −17 | $10.6.\bar{8}$ | <3 | 0 |
| $80\bar{9}$ | 9 | +9 | $8.3.\overline{14}$ | <4 | 0 |  |  |  | $95\bar{6}$ | 5 | +6 | $10.2.\bar{5}$ | 9 | +13 | $10.6.\bar{9}$ | <2 | 0 |
| $8.0.\overline{10}$ | 8 | +7 |  |  |  | 927 | <3 | 0 | $95\bar{7}$ | <5 | +3 | $10.2.\bar{6}$ | 4 | −5 |  |  |  |
| $8.0.\overline{11}$ | 5 | +6 | 846 | 2 | +4 | 926 | 2 | +4 | $95\bar{8}$ | <5 | −4 | $10.2.\bar{7}$ | <3 | 0 | 11.1.6 | <2 | −3 |
| $8.0.\overline{12}$ | 5 | +4 | 845 | <3 | −4 | 925 | <3 | +1 | $95\bar{9}$ | <4 | −2 | $10.2.\bar{8}$ | 4 | −2 | 11.1.5 | <3 | +1 |
| $8.0.\overline{13}$ | 6 | −7 | 844 | <4 | −1 | 924 | <3 | 0 | $9.5.\overline{10}$ | <3 | +2 | $10.2.\bar{9}$ | 4 | +4 | 11.1.4 | 7 | −9 |
| $8.0.\overline{14}$ | 7 | +3 | 843 | <4 | −2 | 923 | 6 | +8 | $9.5.\overline{11}$ | <2 | −1 | $10.2.\overline{10}$ | 4 | −6 | 11.1.3 | 6 | +6 |
| $8.0.\overline{15}$ | 3 | +3 | 842 | 8 | +10 | 922 | <3 | +2 |  |  |  | $10.2.\overline{11}$ | 7 | +6 | 11.1.2 | 9 | +9 |
|  |  |  | 841 | 7 | +8 | 921 | <3 | −3 | 961 | 3 | +5 | $10.2.\overline{12}$ | 3 | +3 | 11.1.1 | 7 | +7 |
| 818 | <3 | +1 | 840 | 5 | +8 | 920 | 14 | −19 | 960 | <3 | −1 | $10.2.\overline{13}$ | 3 | +4 | 11.1.0 | 10 | −11 |
| 817 | <3 | −2 | $84\bar{1}$ | 6 | −7 | $92\bar{1}$ | 6 | −8 | $96\bar{1}$ | <3 | +1 | $10.2.\overline{14}$ | 3 | −3 | $11.1.\bar{1}$ | <4 | −3 |
| 816 | <4 | +1 | $84\bar{2}$ | 3 | +5 | $92\bar{2}$ | 11 | −13 | $96\bar{2}$ | <3 | +2 |  |  |  | $11.1.\bar{2}$ | <4 | 0 |
| 815 | 6 | −7 | $84\bar{3}$ | 3 | +4 | $92\bar{3}$ | 5 | +5 | $96\bar{3}$ | 3 | +3 | 10.3.5 | <3 | −2 | $11.1.\bar{3}$ | 4 | −5 |
| 814 | <4 | +2 | $84\bar{4}$ | 5 | −7 | $92\bar{4}$ | 11 | +12 | $96\bar{4}$ | 4 | +4 | 10.3.4 | <4 | 0 | $11.1.\bar{4}$ | 19 | −24 |
| 813 | 11 | +14 | $84\bar{5}$ | <4 | −1 | $92\bar{5}$ | 14 | −15 | $96\bar{5}$ | <3 | −3 | 10.3.3 | 3 | −3 | $11.1.\bar{5}$ | 13 | −12 |
| 812 | 4 | −4 | $84\bar{6}$ | 11 | +16 | $92\bar{6}$ | 2 | +5 | $96\bar{6}$ | <3 | +2 | 10.3.2 | 5 | −5 | $11.1.\bar{6}$ | 9 | −10 |
| 811 | 4 | −4 | $84\bar{7}$ | <4 | +2 | $92\bar{7}$ | 3 | +1 | $96\bar{7}$ | <3 | +1 | 10.3.1 | <4 | +1 | $11.1.\bar{7}$ | 12 | +11 |
| 810 | 8 | −7 | $84\bar{8}$ | 5 | −6 | $92\bar{8}$ | 5 | +5 | $96\bar{8}$ | <3 | +1 | 10.3.0 | 5 | +5 | $11.1.\bar{8}$ | 6 | −5 |
| $81\bar{1}$ | 8 | +10 | $84\bar{9}$ | <4 | −2 | $92\bar{9}$ | 11 | −13 | $96\bar{9}$ | <2 | −4 | $10.3.\bar{1}$ | 3 | +2 | $11.1.\bar{9}$ | 4 | +4 |
| $81\bar{2}$ | 41 | −44 | $8.4.\overline{10}$ | 7 | +8 | $9.2.\overline{10}$ | <3 | +3 |  |  |  | $10.3.\bar{2}$ | <4 | 0 | $11.1.\overline{10}$ | 7 | −6 |
| $81\bar{3}$ | <3 | +6 | $8.4.\overline{11}$ | 5 | −5 | $9.2.\overline{11}$ | <3 | +1 | 10.0.6 | <3 | 0 | $10.3.\bar{3}$ | 7 | +11 | $11.1.\overline{11}$ | 6 | +4 |
| $81\bar{4}$ | 9 | +12 | $8.4.\overline{12}$ | <3 | −1 | $9.2.\overline{12}$ | 13 | −13 | 10.0.5 | <4 | +2 | $10.3.\bar{4}$ | <4 | −1 | $11.1.\overline{12}$ | <4 | 0 |
| $81\bar{5}$ | 9 | −7 | $8.4.\overline{13}$ | 2 | −3 | $9.2.\overline{13}$ | 9 | −9 | 10.0.4 | 16 | −18 | $10.3.\bar{5}$ | <4 | +1 | $11.1.\overline{13}$ | 3 | +3 |
| $81\bar{6}$ | 6 | −3 |  |  |  | $9.2.\overline{14}$ | 5 | −5 | 10.0.3 | 8 | +11 | $10.3.\bar{6}$ | <4 | −3 | $11.1.\overline{14}$ | 5 | −2 |
| $81\bar{7}$ | 10 | −12 | 854 | 5 | −8 |  |  |  | 10.0.2 | <4 | −3 | $10.3.\bar{7}$ | <4 | −2 | $11.1.\overline{15}$ | <3 | −1 |
| $81\bar{8}$ | 5 | −4 | 853 | 4 | −5 | 936 | 2 | −5 | 10.0.1 | <4 | +2 | $10.3.\bar{8}$ | 3 | −5 |  |  |  |
| $81\bar{9}$ | 16 | −17 | 852 | <4 | +1 | 935 | <4 | +3 | 10.0.0 | 11 | −8 | $10.3.\bar{9}$ | 4 | −2 | 11.2.5 | 2 | −3 |
| $8.1.\overline{10}$ | 7 | +7 | 851 | <4 | −1 | 934 | 4 | +4 | $10.0.\bar{1}$ | 19 | +21 | $10.3.\overline{10}$ | 4 | −4 | 11.2.4 | <3 | −3 |
| $8.1.\overline{11}$ | 7 | −7 | 850 | <4 | 0 | 933 | 3 | −4 | $10.0.\bar{2}$ | <3 | −1 | $10.3.\overline{11}$ | 3 | −4 | 11.2.3 | <3 | 0 |
| $8.1.\overline{12}$ | 5 | −3 | $85\bar{1}$ | <4 | +2 | 932 | 7 | +11 | $10.0.\bar{3}$ | 23 | −20 | $10.3.\overline{12}$ | <4 | +1 | 11.2.2 | <3 | −2 |
| $8.1.\overline{13}$ | 3 | +3 | $85\bar{2}$ | 10 | +14 | 931 | 14 | +20 | $10.0.\bar{4}$ | 23 | −27 | $10.3.\overline{13}$ | 2 | −3 | 11.2.1 | <3 | +1 |
| $8.1.\overline{14}$ | <3 | 0 | $85\bar{3}$ | 8 | +9 | 930 | 12 | +16 | $10.0.\bar{5}$ | 20 | −22 | $10.3.\overline{14}$ | <3 | −1 | 11.2.0 | 12 | +14 |
| $8.1.\overline{15}$ | <2 | −3 | $85\bar{4}$ | <4 | +2 | $93\bar{1}$ | 4 | −7 | $10.0.\bar{6}$ | 5 | −5 |  |  |  | $11.2.\bar{1}$ | 15 | +20 |
|  |  |  | $85\bar{5}$ | <4 | −2 | $93\bar{2}$ | 4 | −4 | $10.0.\bar{7}$ | 6 | +11 | 10.4.4 | 2 | +2 | $11.2.\bar{2}$ | 9 | +10 |
| 827 | 2 | +3 | $85\bar{6}$ | <4 | −1 | $93\bar{3}$ | <4 | +4 | $10.0.\bar{8}$ | 7 | −10 | 10.4.3 | 5 | +7 | $11.2.\bar{3}$ | 11 | −14 |
| 826 | <3 | 0 | $85\bar{7}$ | 5 | +6 | $93\bar{4}$ | 4 | −5 | $10.0.\bar{9}$ | 14 | −14 | 10.4.2 | <4 | −3 | $11.2.\bar{4}$ | 3 | +3 |
| 825 | <3 | −2 | $85\bar{8}$ | <4 | +2 | $93\bar{5}$ | 6 | −9 | $10.0.\overline{10}$ | 12 | −14 | 10.4.1 | <4 | −2 | $11.2.\bar{5}$ | 10 | −10 |
| 824 | 9 | −10 | $85\bar{9}$ | 3 | +4 | $93\bar{6}$ | <4 | +1 | $10.0.\overline{11}$ | <4 | 0 | 10.4.0 | <4 | −2 | $11.2.\bar{6}$ | 11 | −10 |
| 823 | <3 | −3 | $8.5.\overline{10}$ | <3 | −3 | $93\bar{7}$ | 4 | +7 | $10.0.\overline{12}$ | 8 | +8 | $10.4.\bar{1}$ | 7 | +8 | $11.2.\bar{7}$ | 7 | −7 |
| 822 | 17 | +23 | $8.5.\overline{11}$ | <3 | −3 | $93\bar{8}$ | 3 | +2 | $10.0.\overline{13}$ | <3 | +3 | $10.4.\bar{2}$ | <4 | −1 | $11.2.\bar{8}$ | <3 | −1 |
| 821 | 5 | +5 |  |  |  | $93\bar{9}$ | 7 | −8 | $10.0.\overline{14}$ | <3 | +1 | $10.4.\bar{3}$ | 8 | +10 | $11.2.\bar{9}$ | <3 | −1 |
| 820 | 2 | −3 | 862 | <2 | 0 | $9.3.\overline{10}$ | 8 | +10 | $10.0.\overline{15}$ | <3 | +2 | $10.4.\bar{4}$ | <4 | +1 | $11.2.\overline{10}$ | 9 | −9 |
| $82\bar{1}$ | <3 | −2 | 861 | 2 | −4 | $9.3.\overline{11}$ | 12 | +14 |  |  |  | $10.4.\bar{5}$ | <4 | +3 | $11.2.\overline{11}$ | 6 | +5 |
| $82\bar{2}$ | 19 | +24 | 860 | 5 | −7 | $9.3.\overline{12}$ | 4 | +6 | 10.1.6 | <3 | 0 | $10.4.\bar{6}$ | 4 | +3 | $11.2.\overline{12}$ | <3 | −3 |
| $82\bar{3}$ | 10 | −11 | $86\bar{1}$ | 4 | −5 | $9.3.\overline{13}$ | 4 | −3 | 10.1.5 | <4 | +4 | $10.4.\bar{7}$ | 5 | +5 | $11.2.\overline{13}$ | 3 | +4 |
| $82\bar{4}$ | 2 | −5 | $86\bar{2}$ | <4 | 0 | $9.3.\overline{14}$ | 4 | −6 | 10.1.4 | <4 | −3 | $10.4.\bar{8}$ | <4 | −2 | $11.2.\overline{14}$ | <3 | +3 |
| $82\bar{5}$ | 8 | −9 | $86\bar{3}$ | <4 | −3 |  |  |  | 10.1.3 | 4 | −2 | $10.4.\bar{9}$ | <4 | +1 | $11.2.\overline{15}$ | <3 | −1 |
| $82\bar{6}$ | 14 | +14 | $86\bar{4}$ | 4 | +6 | 945 | 2 | −2 | 10.1.2 | 5 | −6 | $10.4.\overline{10}$ | 6 | +5 |  |  |  |
| $82\bar{7}$ | 7 | −10 | $86\bar{5}$ | <4 | 0 | 944 | 4 | −3 | 10.1.1 | <4 | +1 | $10.4.\overline{11}$ | <4 | +2 | 11 3.4 | <3 | +1 |
| $82\bar{8}$ | 6 | −7 | $86\bar{6}$ | <4 | +1 | 943 | 9 | −11 | 10.1.0 | <4 | +4 | $10.4.\overline{12}$ | <4 | +1 | 11.3.3 | <4 | −1 |
| $82\bar{9}$ | 11 | −13 | $86\bar{7}$ | <3 | −1 | 942 | 3 | −1 | $10.1.\bar{1}$ | 6 | +9 | $10.4.\overline{13}$ | 2 | +2 | 11.3.2 | <4 | −1 |
| $8.2.\overline{10}$ | 17 | +20 | $86\bar{8}$ | <3 | +3 | 941 | 4 | +4 | $10.1.\bar{2}$ | <4 | 0 |  |  |  | 11.3.1 | 3 | +2 |
| $8.2.\overline{11}$ | 3 | +2 | $86\bar{9}$ | <2 | 0 | 940 | 7 | +9 | $10.1.\bar{3}$ | 8 | −6 | 10.5.3 | 3 | −6 | 11.3.0 | 7 | −11 |
| $8.2.\overline{12}$ | 5 | −5 |  |  |  | $94\bar{1}$ | <4 | −4 | $10.1.\bar{4}$ | 20 | +28 | 10.5.2 | <3 | +2 | $11.3.\bar{1}$ | 6 | +9 |
| $8.2.\overline{13}$ | 5 | −5 | 917 | <3 | −1 | $94\bar{2}$ | 6 | +9 | $10.1.\bar{5}$ | 17 | −21 | 10.5.1 | <4 | +2 | $11.3.\bar{2}$ | 12 | +17 |
| $8.2.\overline{14}$ | 7 | +7 | 916 | <3 | +2 | $94\bar{3}$ | 10 | +11 | $10.1.\bar{6}$ | 14 | −13 | 10.5.0 | <4 | +1 | $11.3.\bar{3}$ | 23 | +35 |

Table 3 (*cont.*)

| *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ |
|---|---|---|
| 11.3.$\bar{4}$ | 8 | −12 |
| 11.3.$\bar{5}$ | 4 | −2 |
| 11.3.$\bar{6}$ | <4 | +2 |
| 11.3.$\bar{7}$ | 5 | +8 |
| 11.3.$\bar{8}$ | 7 | −10 |
| 11.3.$\bar{9}$ | <4 | −2 |
| 11.3.$\overline{10}$ | <4 | +1 |
| 11.3.$\overline{11}$ | 5 | +6 |
| 11.3.$\overline{12}$ | 3 | −5 |
| 11.3.$\overline{13}$ | 4 | +4 |
| 11.3.$\overline{14}$ | 4 | +5 |
| 11.4.3 | 3 | −4 |
| 11.4.2 | 4 | −4 |
| 11.4.1 | <3 | −1 |
| 11.4.0 | <3 | −3 |
| 11.4.$\bar{1}$ | 5 | −5 |
| 11.4.$\bar{2}$ | 4 | −4 |
| 11.4.$\bar{3}$ | 9 | +14 |
| 11.4.$\bar{4}$ | 4 | +0 |
| 11.4.$\bar{5}$ | 3 | +5 |
| 11.4.$\bar{6}$ | 4 | +4 |
| 11.4.$\bar{7}$ | 3 | +4 |
| 11.4.$\bar{8}$ | <4 | 0 |
| 11.4.$\bar{9}$ | 6 | −6 |
| 11.4.$\overline{10}$ | 3 | −2 |
| 11.4.$\overline{11}$ | 7 | −9 |
| 11.4.$\overline{12}$ | 5 | +6 |
| 11.4.$\overline{13}$ | <2 | 0 |
| 11.5.2 | <2 | 0 |
| 11.5.1 | <3 | −1 |
| 11.5.0 | 3 | −4 |
| 11.5.$\bar{1}$ | <4 | 0 |
| 11.5.$\bar{2}$ | <4 | +1 |
| 11.5.$\bar{3}$ | <4 | +1 |
| 11.5.$\bar{4}$ | <4 | +2 |
| 11.5.$\bar{5}$ | <4 | −2 |
| 11.5.$\bar{6}$ | <4 | −1 |
| 11.5.$\bar{7}$ | 5 | +6 |
| 11.5.$\bar{8}$ | <4 | +1 |
| 11.5.$\bar{9}$ | <4 | +3 |
| 11.5.$\overline{10}$ | <3 | −1 |
| 11.5.$\overline{11}$ | <2 | −1 |
| 11.6.$\bar{1}$ | <2 | −2 |
| 11.6.$\bar{2}$ | <3 | +2 |
| 11.6.$\bar{3}$ | <3 | −3 |
| 11.6.$\bar{4}$ | 3 | +4 |
| 11.6.$\bar{5}$ | <3 | +4 |
| 11.6.$\bar{6}$ | <3 | +1 |
| 11.6.$\bar{7}$ | <3 | −1 |
| 11.6.$\bar{8}$ | <2 | 0 |
| 12.0.5 | <2 | 0 |
| 12.0.4 | 8 | −10 |
| 12.0.3 | 3 | −1 |
| 12.0.2 | 11 | +13 |
| 12.0.1 | <4 | −1 |
| 12.0.0 | 6 | −6 |
| 12.0.$\bar{1}$ | 10 | −13 |
| 12.0.$\bar{2}$ | 5 | +4 |
| 12.0.$\bar{3}$ | 8 | −8 |
| 12.0.$\bar{4}$ | <4 | +2 |
| 12.0.$\bar{5}$ | 4 | −5 |
| 12.0.$\bar{6}$ | 22 | −25 |
| 12.0.$\bar{7}$ | <4 | −1 |
| 12.0.$\bar{8}$ | 7 | −10 |
| 12.0.$\bar{9}$ | <4 | −1 |
| 12.0.$\overline{10}$ | 5 | −3 |
| 12.0.$\overline{11}$ | 9 | +10 |
| 12.0.$\overline{12}$ | 6 | −5 |
| 12.0.$\overline{13}$ | 7 | −6 |
| 12.0.$\overline{14}$ | 7 | −6 |
| 12.0.$\overline{15}$ | <3 | +2 |
| 12.1.5 | <2 | 0 |
| 12.1.4 | <3 | −1 |
| 12.1.3 | 4 | −2 |
| 12.1.2 | 3 | +4 |
| 12.1.1 | 6 | +5 |
| 12.1.0 | <4 | +1 |
| 12.1.$\bar{1}$ | <4 | −1 |
| 12.1.$\bar{2}$ | 8 | +9 |
| 12.1.$\bar{3}$ | 3 | −5 |
| 12.1.$\bar{4}$ | <4 | +2 |
| 12.1.$\bar{5}$ | <4 | +2 |
| 12.1.$\bar{6}$ | 6 | −6 |
| 12.1.$\bar{7}$ | 7 | +8 |
| 12.1.$\bar{8}$ | <4 | −1 |
| 12.1.$\bar{9}$ | <4 | −2 |
| 12.1.$\overline{10}$ | 12 | −14 |
| 12.1.$\overline{11}$ | 13 | +13 |
| 12.1.$\overline{12}$ | 6 | +2 |
| 12.1.$\overline{13}$ | 5 | −4 |
| 12.1.$\overline{14}$ | <4 | +2 |
| 12.1.$\overline{15}$ | <3 | −1 |
| 12.2.4 | <2 | +1 |
| 12.2.3 | <3 | −2 |
| 12.2.2 | <3 | −2 |
| 12.2.1 | 10 | +13 |
| 12.2.0 | <3 | +1 |
| 12.2.$\bar{1}$ | 4 | −6 |
| 12.2.$\bar{2}$ | 4 | −8 |
| 12.2.$\bar{3}$ | 21 | +24 |
| 12.2.$\bar{4}$ | <3 | −1 |
| 12.2.$\bar{5}$ | 12 | −12 |
| 12.2.$\bar{6}$ | 10 | −11 |
| 12.2.$\bar{7}$ | 3 | +4 |
| 12.2.$\bar{8}$ | 3 | +4 |
| 12.2.$\bar{9}$ | <3 | 0 |
| 12.2.$\overline{10}$ | 3 | −4 |
| 12.2.$\overline{11}$ | 3 | +5 |
| 12.2.$\overline{12}$ | 5 | +5 |
| 12.2.$\overline{13}$ | <3 | +1 |
| 12.2.$\overline{14}$ | 3 | −2 |
| 12.2.$\overline{15}$ | 6 | +7 |
| 12.3.4 | <3 | −2 |
| 12.3.3 | <4 | +1 |
| 12.3.2 | <4 | −3 |
| 12.3.1 | <4 | 0 |
| 12.3.0 | 3 | +4 |
| 12.3.$\bar{1}$ | <4 | +4 |
| 12.3.$\bar{2}$ | <4 | −1 |
| 12.3.$\bar{3}$ | 6 | +5 |
| 12.3.$\bar{4}$ | 9 | +8 |
| 12.3.$\bar{5}$ | 3 | −2 |
| 12.3.$\bar{6}$ | <4 | 0 |
| 12.3.$\bar{7}$ | 3 | −2 |
| 12.3.$\bar{8}$ | <4 | 0 |
| 12.3.$\bar{9}$ | 5 | −5 |
| 12.3.$\overline{10}$ | 3 | −2 |
| 12.3.$\overline{11}$ | 5 | −6 |
| 12.3.$\overline{12}$ | <4 | +2 |
| 12.3.$\overline{13}$ | 4 | +5 |
| 12.3.$\overline{14}$ | <3 | 0 |
| 12.4.2 | <2 | 0 |
| 12.4.1 | 4 | +4 |
| 12.4.0 | <3 | +1 |
| 12.4.$\bar{1}$ | <3 | 0 |
| 12.4.$\bar{2}$ | <3 | 0 |
| 12.4.$\bar{3}$ | 6 | +9 |
| 12.4.$\bar{4}$ | 6 | +6 |
| 12.4.$\bar{5}$ | 4 | −5 |
| 12.4.$\bar{6}$ | <4 | −3 |
| 12.4.$\bar{7}$ | 8 | +10 |
| 12.4.$\bar{8}$ | 6 | +5 |
| 12.4.$\bar{9}$ | <3 | −2 |
| 12.4.$\overline{10}$ | <3 | −1 |
| 12.4.$\overline{11}$ | 4 | +5 |
| 12.4.$\overline{12}$ | 3 | −3 |
| 12.4.$\overline{13}$ | 2 | +3 |
| 12.5.1 | <3 | −1 |
| 12.5.0 | <4 | −3 |
| 12.5.$\bar{1}$ | <4 | −2 |
| 12.5.$\bar{2}$ | <4 | +3 |
| 12.5.$\bar{3}$ | 4 | +6 |
| 12.5.$\bar{4}$ | <4 | −1 |
| 12.5.$\bar{5}$ | <4 | +1 |
| 12.5.$\bar{6}$ | <4 | −2 |
| 12.5.$\bar{7}$ | <4 | −3 |
| 12.5.$\bar{8}$ | <4 | +2 |
| 12.5.$\bar{9}$ | <4 | +1 |
| 12.5.$\overline{10}$ | <3 | −2 |
| 12.5.$\overline{11}$ | 4 | −6 |
| 12.6.$\bar{3}$ | <3 | 0 |
| 12.6.$\bar{4}$ | <3 | 0 |
| 12.6.$\bar{5}$ | <3 | +3 |
| 12.6.$\bar{6}$ | <3 | −1 |
| 12.6.$\bar{7}$ | <3 | +2 |
| 12.6.$\bar{8}$ | <3 | −2 |
| 13.1.4 | <3 | −3 |
| 13.1.3 | 6 | −6 |
| 13.1.2 | 6 | +6 |
| 13.1.1 | 3 | +1 |
| 13.1.0 | 7 | +6 |
| 13.1.$\bar{1}$ | <4 | −1 |
| 13.1.$\bar{2}$ | 7 | +6 |
| 13.1.$\bar{3}$ | <4 | −1 |
| 13.1.$\bar{4}$ | <4 | +3 |
| 13.1.$\bar{5}$ | 6 | −4 |
| 13.1.$\bar{6}$ | 18 | −20 |
| 13.1.$\bar{7}$ | 15 | −17 |
| 13.1.$\bar{8}$ | 7 | −5 |
| 13.1.$\bar{9}$ | <4 | −1 |
| 13.1.$\overline{10}$ | <4 | −2 |
| 13.1.$\overline{11}$ | 7 | +4 |
| 13.1.$\overline{12}$ | 10 | +7 |
| 13.1.$\overline{13}$ | <4 | −2 |
| 13.1.$\overline{14}$ | 6 | −5 |
| 13.1.$\overline{15}$ | <3 | +1 |
| 13.2.3 | <2 | 0 |
| 13.2.2 | 5 | −4 |
| 13.2.1 | <3 | −1 |
| 13.2.0 | <3 | +2 |
| 13.2.$\bar{1}$ | <3 | −1 |
| 13.2.$\bar{2}$ | <3 | 0 |
| 13.2.$\bar{3}$ | 7 | +9 |
| 13.2.$\bar{4}$ | 19 | +24 |
| 13.2.$\bar{5}$ | 15 | +18 |
| 13.2.$\bar{6}$ | 5 | −6 |
| 13.2.$\bar{7}$ | 5 | −3 |
| 13.2.$\bar{8}$ | 3 | −3 |
| 13.2.$\bar{9}$ | 3 | −3 |
| 13.2.$\overline{10}$ | 13 | −15 |
| 13.2.$\overline{11}$ | <3 | −2 |
| 13.2.$\overline{12}$ | 5 | +5 |
| 13.2.$\overline{13}$ | <3 | −1 |
| 13.2.$\overline{14}$ | 3 | +2 |
| 13.2.$\overline{15}$ | <2 | −1 |
| 13.3.3 | 2 | +5 |
| 13.3.2 | <3 | −3 |
| 13.3.1 | <3 | +3 |
| 13.3.0 | 3 | +4 |
| 13.3.$\bar{1}$ | <4 | 0 |
| 13.3.$\bar{2}$ | 8 | −12 |
| 13.3.$\bar{3}$ | 5 | −5 |
| 13.3.$\bar{4}$ | 6 | +8 |
| 13.3.$\bar{5}$ | 3 | +2 |
| 13.3.$\bar{6}$ | 13 | +15 |
| 13.3.$\bar{7}$ | 3 | +2 |
| 13.3.$\bar{8}$ | 5 | +8 |
| 13.3.$\bar{9}$ | <4 | +2 |
| 13.3.$\overline{10}$ | 4 | +4 |
| 13.3.$\overline{11}$ | <3 | −2 |
| 13.3.$\overline{12}$ | <3 | −1 |
| 13.3.$\overline{13}$ | 2 | +3 |
| 13.3.$\overline{14}$ | <2 | 0 |
| 13.4.2 | <2 | +2 |
| 13.4.1 | <3 | +1 |
| 13.4.0 | <3 | +1 |
| 13.4.$\bar{1}$ | <3 | +1 |
| 13.4.$\bar{2}$ | <3 | 0 |
| 13.4.$\bar{3}$ | <3 | +2 |
| 13.4.$\bar{4}$ | <3 | −1 |
| 13.4.$\bar{5}$ | 6 | −7 |
| 13.4.$\bar{6}$ | 3 | −0 |
| 13.4.$\bar{7}$ | 3 | −3 |
| 13.4.$\bar{8}$ | <3 | +1 |
| 13.4.$\bar{9}$ | <3 | −2 |
| 13.4.$\overline{10}$ | 4 | +5 |
| 13.4.$\overline{11}$ | 3 | −3 |
| 13.4.$\overline{12}$ | 3 | −4 |
| 13.4.$\overline{13}$ | 3 | +4 |
| 13.5.$\bar{3}$ | 1 | −2 |
| 13.5.$\bar{4}$ | 3 | +4 |
| 13.5.$\bar{5}$ | 4 | +4 |
| 13.5.$\bar{6}$ | <2 | −3 |
| 13.5.$\bar{7}$ | 1 | +3 |
| 13.5.$\bar{8}$ | 1 | +2 |
| 13.5.$\bar{9}$ | <2 | 0 |
| 13.5.$\overline{10}$ | <2 | −4 |
| 14.0.3 | 7 | −11 |
| 14.0.2 | 4 | +6 |
| 14.0.1 | <4 | −1 |
| 14.0.0 | 5 | +5 |
| 14.0.$\bar{1}$ | <4 | +3 |
| 14.0.$\bar{2}$ | 9 | +12 |
| 14.0.$\bar{3}$ | 4 | −7 |
| 14.0.$\bar{4}$ | 13 | −18 |
| 14.0.$\bar{5}$ | <4 | 0 |
| 14.0.$\bar{6}$ | 12 | −16 |
| 14.0.$\bar{7}$ | 14 | −15 |
| 14.0.$\bar{8}$ | <4 | −2 |
| 14.0.$\bar{9}$ | <4 | −1 |
| 14.0.$\overline{10}$ | <4 | 0 |
| 14.0.$\overline{11}$ | 3 | −3 |
| 14.0.$\overline{12}$ | 11 | +12 |
| 14.0.$\overline{13}$ | <4 | −1 |
| 14.0.$\overline{14}$ | <3 | +2 |
| 14.0.$\overline{15}$ | 5 | −6 |
| 14.1.3 | 5 | +6 |
| 14.1.2 | <3 | −2 |
| 14.1.1 | <4 | −1 |
| 14.1.0 | <4 | 0 |
| 14.1.$\bar{1}$ | 5 | +4 |
| 14.1.$\bar{2}$ | 6 | +7 |
| 14.1.$\bar{3}$ | 7 | +7 |
| 14.1.$\bar{4}$ | 7 | +6 |
| 14.1.$\bar{5}$ | 11 | +13 |
| 14.1.$\bar{6}$ | <4 | 0 |
| 14.1.$\bar{7}$ | 10 | −8 |
| 14.1.$\bar{8}$ | 8 | −8 |
| 14.1.$\bar{9}$ | <4 | +2 |
| 14.1.$\overline{10}$ | <4 | −1 |
| 14.1.$\overline{11}$ | 6 | −4 |
| 14.1.$\overline{12}$ | 7 | +5 |
| 14.1.$\overline{13}$ | 5 | −5 |
| 14.1.$\overline{14}$ | 6 | +6 |
| 14.1.$\overline{15}$ | 5 | +4 |
| 14.2.3 | <2 | 0 |
| 14.2.2 | 2 | +2 |
| 14.2.1 | <3 | +1 |
| 14.2.0 | <3 | +3 |
| 14.2.$\bar{1}$ | <3 | −3 |
| 14.2.$\bar{2}$ | 3 | +4 |
| 14.2.$\bar{3}$ | 3 | −2 |
| 14.2.$\bar{4}$ | 3 | +4 |
| 14.2.$\bar{5}$ | 11 | −15 |
| 14.2.$\bar{6}$ | 13 | +16 |
| 14.2.$\bar{7}$ | 3 | +1 |
| 14.2.$\bar{8}$ | 3 | −2 |
| 14.2.$\bar{9}$ | 9 | −10 |
| 14.2.$\overline{10}$ | 5 | +6 |
| 14.2.$\overline{11}$ | 3 | +2 |
| 14.2.$\overline{12}$ | 3 | +2 |
| 14.2.$\overline{13}$ | <3 | +1 |
| 14.2.$\overline{14}$ | <3 | −1 |
| 14.2.$\overline{15}$ | <2 | −2 |
| 14.3.2 | <2 | +1 |
| 14.3.1 | <3 | −1 |
| 14.3.0 | <4 | +2 |
| 14.3.$\bar{1}$ | 3 | +2 |
| 14.3.$\bar{2}$ | <4 | +1 |
| 14.3.$\bar{3}$ | <4 | +1 |
| 14.3.$\bar{4}$ | 7 | +12 |
| 14.3.$\bar{5}$ | 4 | +5 |
| 14.3.$\bar{6}$ | 5 | −5 |
| 14.3.$\bar{7}$ | 3 | −3 |
| 14.3.$\bar{8}$ | <4 | −1 |
| 14.3.$\bar{9}$ | <4 | −2 |
| 14.3.$\overline{10}$ | 3 | −2 |
| 14.3.$\overline{11}$ | <3 | +1 |
| 14.3.$\overline{12}$ | 2 | −3 |
| 14.3.$\overline{13}$ | <3 | +2 |
| 14.3.$\overline{14}$ | <2 | +1 |
| 14.4.1 | <2 | +1 |
| 14.4.0 | <2 | 0 |
| 14.4.$\bar{1}$ | 3 | −4 |
| 14.4.$\bar{2}$ | <3 | +1 |
| 14.4.$\bar{3}$ | <3 | −3 |
| 14.4.$\bar{4}$ | 3 | +4 |
| 14.4.$\bar{5}$ | 3 | +3 |
| 14.4.$\bar{6}$ | 7 | +10 |
| 14.4.$\bar{7}$ | 5 | +4 |
| 14.4.$\bar{8}$ | <3 | +2 |
| 14.4.$\bar{9}$ | 4 | −5 |
| 14.4.$\overline{10}$ | 3 | +1 |
| 14.4.$\overline{11}$ | 4 | +4 |
| 14.4.$\overline{12}$ | <2 | +1 |
| 14.4.$\overline{13}$ | <2 | −2 |
| 14.5.$\bar{4}$ | 2 | −3 |
| 14.5.$\bar{5}$ | <2 | −1 |
| 14.5.$\bar{6}$ | <2 | −1 |
| 14.5.$\bar{7}$ | 3 | −4 |
| 15.5.$\bar{8}$ | <2 | 0 |
| 14.5.$\bar{9}$ | 1 | +2 |
| 14.5.$\overline{10}$ | <2 | 0 |
| 15.1.2 | <3 | +1 |
| 15.1.1 | 3 | +1 |
| 15.1.0 | <4 | +1 |
| 15.1.$\bar{1}$ | 3 | −3 |
| 15.1.$\bar{2}$ | 4 | +4 |
| 15.1.$\bar{3}$ | 4 | +3 |
| 15.1.$\bar{4}$ | 4 | −5 |
| 15.1.$\bar{5}$ | 8 | −8 |
| 15.1.$\bar{6}$ | <4 | +1 |
| 15.1.$\bar{7}$ | 4 | +3 |
| 15.1.$\bar{8}$ | 8 | +8 |
| 15.1.$\bar{9}$ | 12 | −11 |
| 15.1.$\overline{10}$ | 8 | −6 |
| 15.1.$\overline{11}$ | 7 | −6 |
| 15.1.$\overline{12}$ | 10 | +9 |
| 15.1.$\overline{13}$ | <4 | −2 |
| 15.1.$\overline{14}$ | <3 | +1 |
| 15.1.$\overline{15}$ | <3 | −2 |
| 15.2.2 | <2 | +2 |
| 15.2.1 | <2 | +1 |
| 15.2.0 | 2 | +3 |
| 15.2.$\bar{1}$ | <3 | +2 |
| 15.2.$\bar{2}$ | <3 | +3 |
| 15.2.$\bar{3}$ | <3 | +1 |
| 15.2.$\bar{4}$ | 3 | +2 |
| 15.2.$\bar{5}$ | <3 | 0 |
| 15.2.$\bar{6}$ | <3 | 0 |
| 15.2.$\bar{7}$ | 6 | +6 |
| 15.2.$\bar{8}$ | 6 | +8 |
| 15.2.$\bar{9}$ | 4 | +4 |
| 15.2.$\overline{10}$ | <3 | +1 |
| 15.2.$\overline{11}$ | 3 | −1 |
| 15.2.$\overline{12}$ | 3 | +3 |
| 15.2.$\overline{13}$ | 3 | −6 |
| 15.2.$\overline{14}$ | <2 | −1 |
| 15.2.$\overline{15}$ | 2 | +4 |
| 15.3.1 | <3 | +2 |
| 15.3.0 | 5 | +8 |
| 15.3.$\bar{1}$ | <4 | −3 |
| 15.3.$\bar{2}$ | <4 | +1 |
| 15.3.$\bar{3}$ | <4 | 0 |
| 15.3.$\bar{4}$ | 7 | +12 |
| 15.3.$\bar{5}$ | 9 | −17 |
| 15.3.$\bar{6}$ | 7 | −7 |
| 15.3.$\bar{7}$ | 4 | +2 |
| 15.3.$\bar{8}$ | 6 | +6 |
| 15.3.$\bar{9}$ | <4 | −3 |
| 15.3.$\overline{10}$ | <4 | 0 |
| 15.3.$\overline{11}$ | 8 | +9 |
| 15.3.$\overline{12}$ | 5 | +6 |
| 15.3.$\overline{13}$ | 2 | +2 |
| 15.3.$\overline{14}$ | <2 | 0 |
| 15.4.$\bar{1}$ | 4 | +5 |
| 15.4.$\bar{2}$ | <3 | +1 |
| 15.4.$\bar{3}$ | <3 | −1 |
| 15.4.$\bar{4}$ | <3 | +3 |
| 15.4.$\bar{5}$ | 5 | +6 |
| 15.4.$\bar{6}$ | <3 | +1 |
| 15.4.$\bar{7}$ | 4 | −6 |
| 15.4.$\bar{8}$ | 6 | −7 |
| 15.4.$\bar{9}$ | 3 | −3 |
| 15.4.$\overline{10}$ | 3 | −3 |
| 15.4.$\overline{11}$ | <3 | 0 |
| 15.4.$\overline{12}$ | 3 | −4 |
| 15.5.$\bar{5}$ | 1 | +1 |
| 15.5.$\bar{6}$ | 3 | +3 |
| 15.5.$\bar{7}$ | 3 | +2 |
| 15.5.$\bar{8}$ | 3 | +4 |
| 15.5.$\bar{9}$ | 2 | −4 |
| 15.5.$\overline{10}$ | <2 | +1 |
| 16.0.1 | 7 | +9 |
| 16.0.0 | <3 | 0 |
| 16.0.$\bar{1}$ | 3 | −2 |
| 16.0.$\bar{2}$ | 5 | −5 |
| 16.0.$\bar{3}$ | 8 | +9 |
| 16.0.$\bar{4}$ | <4 | +1 |
| 16.0.$\bar{5}$ | <4 | +4 |
| 16.0.$\bar{6}$ | <4 | −1 |
| 16.0.$\bar{7}$ | 9 | −10 |
| 16.0.$\bar{8}$ | 9 | +10 |
| 16.0.$\bar{9}$ | 5 | −5 |
| 16.0.$\overline{10}$ | 10 | −11 |
| 16.0.$\overline{11}$ | 12 | −16 |
| 16.0.$\overline{12}$ | 10 | +12 |
| 16.0.$\overline{13}$ | 6 | +5 |
| 16.0.$\overline{14}$ | 4 | −4 |
| 16.0.$\overline{15}$ | <3 | 0 |

Table 3 (*cont.*)

| *hkl* | $F_{\text{obs.}}$ | $F_{\text{calc.}}$ |
|---|---|---|
| $16,1,1$ | 2 | −3 |
| $16,1,0$ | 7 | +7 |
| $16,1,\bar{1}$ | 4 | +3 |
| $16,1,\bar{2}$ | <4 | −1 |
| $16,1,\bar{3}$ | 4 | −4 |
| $16,1,\bar{4}$ | 6 | +4 |
| $16,1,\bar{5}$ | 4 | +3 |
| $16,1,\bar{6}$ | 4 | +4 |
| $16,1,\bar{7}$ | 8 | +6 |
| $16,1,\bar{8}$ | 10 | +9 |
| $16,1,\bar{9}$ | 9 | +6 |
| $16,1,\overline{10}$ | <4 | 0 |
| $16,1,\overline{11}$ | 9 | −8 |
| $16,1,\overline{12}$ | <4 | +1 |
| $16,1,\overline{13}$ | 5 | +2 |
| $16,1,\overline{14}$ | 3 | −2 |
| $16,1,\overline{15}$ | <2 | +1 |
| $16,2,1$ | <2 | −3 |
| $16,2,0$ | <3 | +3 |
| $16,2,\bar{1}$ | 2 | +3 |
| $16,2,\bar{2}$ | <3 | 0 |
| $16,2,\bar{3}$ | 3 | −4 |
| $16,2,\bar{4}$ | 4 | +6 |
| $16,2,\bar{5}$ | 3 | −4 |
| $16,2,\bar{6}$ | 12 | −12 |
| $16,2,\bar{7}$ | <4 | 0 |
| $16,2,\bar{8}$ | 5 | +4 |
| $16,2,\bar{9}$ | 6 | +6 |
| $16,2,\overline{10}$ | <3 | −1 |
| $16,2,\overline{11}$ | <3 | +1 |
| $16,2,\overline{12}$ | <3 | 0 |
| $16,2,\overline{13}$ | 3 | +4 |
| $16,2,\overline{14}$ | 2 | +3 |
| $16,3,0$ | <3 | 0 |
| $16,3,\bar{1}$ | 4 | +5 |
| $16,3,\bar{2}$ | <3 | +3 |
| $16,3,\bar{3}$ | <3 | +1 |
| $16,3,\bar{4}$ | <3 | −1 |
| $16,3,\bar{5}$ | 3 | +4 |
| $16,3,\bar{6}$ | <3 | 0 |
| $16,3,\bar{7}$ | <3 | +2 |
| $16,3,\bar{8}$ | <3 | +3 |
| $16,3,\bar{9}$ | <3 | −1 |
| $16,3,\overline{10}$ | 4 | −4 |
| $16,3,\overline{11}$ | <3 | 0 |
| $16,3,\overline{12}$ | <3 | 0 |
| $16,3,\overline{13}$ | <3 | 0 |
| $16,4,\bar{2}$ | <2 | −1 |
| $16,4,\bar{3}$ | <3 | +1 |
| $16,4,\bar{4}$ | <3 | +2 |
| $16,4,\bar{5}$ | 4 | −4 |
| $16,4,\bar{6}$ | 4 | −6 |
| $16,4,\bar{7}$ | <3 | −2 |
| $16,4,\bar{8}$ | 5 | +7 |
| $16,4,\bar{9}$ | 5 | +7 |
| $16,4,\overline{10}$ | <3 | +1 |
| $16,4,\overline{11}$ | <3 | −1 |
| $16,4,\overline{12}$ | 2 | +4 |
| $16,5,\bar{6}$ | <2 | −1 |
| $16,5,\bar{7}$ | 5 | −5 |
| $16,5,\bar{8}$ | 5 | −8 |
| $17,1,0$ | <3 | 0 |
| $17,1,\bar{1}$ | <3 | +1 |
| $17,1,\bar{2}$ | 3 | −4 |
| $17,1,\bar{3}$ | <4 | +2 |
| $17,1,\bar{4}$ | <4 | −2 |
| $17,1,\bar{5}$ | <4 | +3 |
| $17,1,\bar{6}$ | 4 | −3 |
| $17,1,\bar{7}$ | 10 | −10 |
| $17,1,\bar{8}$ | 4 | −4 |
| $17,1,\bar{9}$ | 9 | +7 |
| $17,1,\overline{10}$ | 8 | +5 |
| $17,1,\overline{11}$ | <4 | −1 |
| $17,1,\overline{12}$ | <4 | −1 |
| $17,1,\overline{13}$ | 7 | +6 |
| $17,1,\overline{14}$ | 3 | −2 |
| $17,1,\overline{15}$ | <3 | −2 |
| $17,2,\bar{1}$ | 6 | +7 |
| $17,2,\bar{2}$ | 5 | +6 |
| $17,2,\bar{3}$ | <3 | 0 |
| $17,2,\bar{4}$ | <3 | +1 |
| $17,2,\bar{5}$ | 5 | +6 |
| $17,2,\bar{6}$ | <3 | +1 |
| $17,2,\bar{7}$ | <3 | 0 |
| $17,2,\bar{8}$ | 3 | −3 |
| $17,2,\bar{9}$ | <3 | −1 |
| $17,2,\overline{10}$ | <3 | +1 |
| $17,2,\overline{11}$ | <3 | 0 |
| $17,2,\overline{12}$ | 3 | +3 |
| $17,2,\overline{13}$ | 4 | +5 |
| $17,2,\overline{14}$ | 3 | +4 |
| $17,3,\bar{2}$ | <2 | 0 |
| $17,3,\bar{3}$ | 2 | +2 |
| $17,3,\bar{4}$ | <3 | +2 |
| $17,3,\bar{5}$ | <3 | +3 |
| $17,3,\bar{6}$ | 5 | −8 |
| $17,3,\bar{7}$ | 3 | +6 |
| $17,3,\bar{8}$ | <3 | −3 |
| $17,3,\bar{9}$ | 2 | +2 |
| $17,3,\overline{10}$ | 3 | −4 |
| $17,3,\overline{11}$ | 3 | +3 |
| $17,3,\overline{12}$ | <3 | −3 |
| $17,3,\overline{13}$ | <2 | −1 |
| $17,4,\bar{4}$ | 2 | +2 |
| $17,4,\bar{5}$ | <3 | 0 |
| $17,4,\bar{6}$ | 2 | −1 |
| $17,4,\bar{7}$ | 4 | −4 |
| $17,4,\bar{8}$ | <3 | +2 |
| $17,4,\bar{9}$ | <3 | +1 |
| $17,4,\overline{10}$ | 2 | −4 |
| $17,4,\overline{11}$ | 3 | −4 |
| $18,0,\bar{1}$ | <3 | 0 |
| $18,0,\bar{2}$ | <3 | −3 |
| $18,0,\bar{3}$ | 3 | +4 |
| $18,0,\bar{4}$ | <3 | −3 |
| $18,0,\bar{5}$ | 7 | −7 |
| $18,0,\bar{6}$ | <4 | +4 |
| $18,0,\bar{7}$ | <4 | 0 |
| $18,0,\bar{8}$ | 3 | −3 |
| $18,0,\bar{9}$ | 7 | +8 |
| $18,0,\overline{10}$ | 8 | +9 |
| $18,0,\overline{11}$ | 8 | +9 |
| $18,0,\overline{12}$ | 5 | −5 |
| $18,0,\overline{13}$ | <3 | +3 |
| $18,0,\overline{14}$ | 12 | −13 |
| $18,0,\overline{15}$ | 2 | +4 |
| $18,1,\bar{2}$ | <2 | +1 |
| $18,1,\bar{3}$ | 3 | +3 |
| $18,1,\bar{4}$ | 3 | +3 |
| $18,1,\bar{5}$ | 3 | +3 |
| $18,1,\bar{6}$ | 7 | −7 |
| $18,1,\bar{7}$ | <3 | 0 |
| $18,1,\bar{8}$ | <3 | −1 |
| $18,1,\bar{9}$ | <3 | −1 |
| $18,1,\overline{10}$ | <3 | +2 |
| $18,1,\overline{11}$ | 9 | +7 |
| $18,1,\overline{12}$ | 8 | +7 |
| $18,1,\overline{13}$ | 5 | +5 |
| $18,1,\overline{14}$ | <3 | 0 |
| $18,2,\bar{2}$ | 3 | −5 |
| $18,2,\bar{3}$ | <3 | 1 |
| $18,2,\bar{4}$ | <3 | 0 |
| $18,2,\bar{5}$ | 4 | +6 |
| $18,2,\bar{6}$ | 6 | −9 |
| $18,2,\bar{7}$ | <3 | +1 |
| $18,2,\bar{8}$ | <3 | −3 |
| $18,2,\bar{9}$ | <3 | −1 |
| $18,2,\overline{10}$ | 3 | −5 |
| $18,2,\overline{11}$ | 3 | +5 |
| $18,2,\overline{12}$ | 3 | +3 |
| $18,2,\overline{13}$ | <2 | −1 |
| $18,2,\overline{14}$ | 1 | +2 |
| $18,3,\bar{3}$ | 2 | +2 |
| $18,3,\bar{4}$ | 2 | +1 |
| $18,3,\bar{5}$ | 3 | +4 |
| $18,3,\bar{6}$ | <3 | +3 |
| $18,3,\bar{7}$ | 4 | −6 |
| $18,3,\bar{8}$ | 2 | −3 |
| $18,3,\bar{9}$ | <3 | +1 |
| $18,3,\overline{10}$ | <3 | +2 |
| $18,3,\overline{11}$ | <3 | −1 |
| $18,3,\overline{12}$ | <2 | +2 |
| $18,4,\bar{6}$ | <2 | −1 |
| $18,4,\bar{7}$ | 2 | +4 |
| $18,4,\bar{8}$ | 3 | −5 |
| $18,4,\bar{9}$ | 2 | −4 |
| $18,4,\overline{10}$ | 2 | −5 |
| $19,1,\bar{3}$ | <3 | 0 |
| $19,1,\bar{4}$ | <3 | +1 |
| $19,1,\bar{5}$ | 3 | −1 |
| $19,1,\bar{6}$ | 6 | −4 |
| $19,1,\bar{7}$ | 3 | −1 |
| $19,1,\bar{8}$ | <3 | +2 |
| $19,1,\bar{9}$ | 6 | +5 |
| $19,1,\overline{10}$ | 4 | −4 |
| $19,1,\overline{11}$ | <3 | +1 |
| $19,1,\overline{12}$ | 3 | +2 |
| $19,1,\overline{13}$ | 9 | +9 |
| $19,1,\overline{14}$ | <3 | −4 |
| $19,2,\bar{4}$ | 3 | +3 |
| $19,2,\bar{5}$ | 9 | +9 |
| $19,2,\bar{6}$ | <3 | −1 |
| $19,2,\bar{7}$ | 5 | −4 |
| $19,2,\bar{8}$ | <3 | 0 |
| $19,2,\bar{9}$ | <3 | +2 |
| $19,2,\overline{10}$ | 3 | +3 |
| $19,2,\overline{11}$ | <3 | 0 |
| $19,2,\overline{12}$ | 2 | +2 |
| $19,2,\overline{13}$ | <2 | +2 |
| $19,3,\bar{5}$ | 2 | +4 |
| $19,3,\bar{6}$ | 2 | −4 |
| $19,3,\bar{7}$ | <2 | 0 |
| $19,3,\bar{8}$ | <2 | 0 |
| $19,3,\bar{9}$ | <2 | −2 |
| $19,3,\overline{10}$ | <2 | −3 |
| $19,3,\overline{11}$ | <2 | 0 |
| $20,0,\bar{4}$ | <2 | 0 |
| $20,0,\bar{5}$ | <3 | −2 |
| $20,0,\bar{6}$ | <3 | 0 |
| $20,0,\bar{7}$ | 5 | −3 |
| $20,0,\bar{8}$ | 6 | −6 |
| $20,0,\bar{9}$ | 6 | +6 |
| $20,0,\overline{10}$ | 5 | +3 |
| $20,0,\overline{11}$ | <3 | −1 |
| $20,0,\overline{12}$ | <3 | −1 |
| $20,0,\overline{13}$ | 14 | +16 |
| $20,1,\bar{4}$ | <3 | −1 |
| $20,1,\bar{5}$ | 5 | +6 |
| $20,1,\bar{6}$ | 3 | +2 |
| $20,1,\bar{7}$ | <3 | −2 |
| $20,1,\bar{8}$ | <3 | +2 |
| $20,1,\bar{9}$ | 4 | −4 |
| $20,1,\overline{10}$ | <3 | −1 |
| $20,1,\overline{11}$ | <3 | −2 |
| $20,1,\overline{12}$ | <3 | −1 |
| $20,1,\overline{13}$ | <3 | −2 |
| $20,2,\bar{5}$ | <2 | −1 |
| $20,2,\bar{6}$ | <2 | 0 |
| $20,2,\bar{7}$ | 2 | −4 |
| $20,2,\bar{8}$ | 2 | +4 |
| $20,2,\bar{9}$ | 2 | +2 |
| $20,2,\overline{10}$ | <2 | 0 |
| $20,2,\overline{11}$ | 3 | −5 |
| $20,2,\overline{13}$ | 1 | +2 |

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# Untersuchungen an Zink-Sublimationskristallen

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Zinc crystals grown by sublimation in air, in argon or *in vacuo* show stacks of plates parallel to the hexagonal basal plane. By direct microscopic and by interferometric measurement the thickness of these plates is shown to vary from 2$\mu$ down to less than 200 A.

## Einleitung

Zink läßt sich infolge seines relativ hohen Dampfdrucks (*ca.* 0,1 mm. Hg bei 400° C.) bekanntlich im Vakuum leicht sublimieren. Bei geeigneter Versuchsführung erhält man das sublimierte Zink in einzelnen kleinen Kriställchen (Straumanis, 1931, 1932), die ausgeprägt geschichteten Aufbau zeigen (Fig. 1). Die ausgezeichnete Ebene ist die Basisebene des hexagonalen Zinkgitters. Es ist nun von Interesse, die Dicke der Schichten zu bestimmen, um eventuell Schlüsse auf den Wachstumsmechanismus der Kristalle ziehen zu können (Graf, 1942). Straumanis folgerte aus seinen Versuchen, daß die Schichtdicke bei Zink 8000 A. oder ein Vielfaches davon sei. Die vorliegende Arbeit berichtet über Untersuchungen mit anderem Ergebnis. Die Schichtdicke wurde insbesondere mit dem Zeißschen Interferenzmikroskop bestimmt (Räntsch, 1944).