*Acta Cryst.* (1958). **11**, 315

# The Crystal Structure of Sodium Triphosphate, $Na_5P_3O_{10}$, Phase II

By David R. Davies* and D. E. C. Corbridge†

*Research Department, Albright and Wilson (Mfg.) Limited, Oldbury, Birmingham, England*

(*Received* 26 *July* 1957)

The crystal structure of the low-temperature form of sodium triphosphate, $Na_5P_3O_{10}$, Phase II, has been determined by Fourier methods. The unit cell is monoclinic with $[a] = 16{\cdot}00$, $[b] = 5{\cdot}24$, $[c] = 11{\cdot}25$ Å, $\beta = 93°$. The space group is $C2/c$ and the unit cell contains 4 units of $Na_5P_3O_{10}$. The triphosphate ions have twofold axial symmetry with the central phosphorus atoms lying on twofold axes of the unit cell. Bond lengths are P–O (chain) = $1{\cdot}61 \pm 0{\cdot}03$ Å (inner), $1{\cdot}68 \pm 0{\cdot}03$ Å (outer), P–O (terminal) = $1{\cdot}50 \pm 0{\cdot}03$ Å. The sodium ions are co-ordinated by oxygen in distorted octahedral arrangements, which form channels parallel to the $[b]$ axis and sheets parallel to the $(\bar{1}01)$ plane.

## Introduction

Sodium triphosphate crystallizes as a hexahydrate $Na_5P_3O_{10}.6\,H_2O$, and in two anhydrous forms Phase I and Phase II. Phase I hydrates rapidly in the atmosphere and is the stable form above about 450° C. Phase II, an important constituent of modern detergent powders, hydrates much less readily and is the stable form at ordinary temperatures.

The structure of sodium triphosphate Phase II has been investigated as part of a programme to determine the configuration of the triphosphate ion and the structural differences between the two anhydrous phases.

Small crystals of Phase II were obtained by the method of Dymon & King (1951). A melt of composition 45% sodium pyrophosphate ($Na_4P_2O_7$) and 55% sodium trimetaphosphate ($Na_3P_3O_9$) was cooled in a small furnace at the rate of 4° C. per hour from 640 to about 450° C. The resulting mass was quenched in a bath of acetone and dry ice, and when dried and broken up it yielded some small brick-like crystals of the low-temperature form. These crystals, partially freed from adhering fragments, were used for the investigation.

## Unit cell and space group

Rotation and Weissenberg photographs showed the crystals to be monoclinic with

$$[a]=16{\cdot}00\pm0{\cdot}05,\ [b]=5{\cdot}24\pm0{\cdot}02,\ [c]=11{\cdot}25\pm0{\cdot}03\ \text{Å},\ \beta = 93{\cdot}0\pm0{\cdot}5° .$$

Systematically absent reflexions were $hkl$ for $h+k$ odd, and $h0l$ for $l$ odd, which indicated space group $C2/c$ or $Cc$. These data were found to be in agreement with those reported by Dymon & King (1951).

The statistical method of Howells, Phillips & Rogers (1950), when applied to the $\{h0l\}$ data, indicated the presence of a centre of symmetry. The crystals were found to be non-pyroelectric and space group $C2/c$ was adopted. The density, determined by flotation, was found to be $2{\cdot}57$ g.cm.$^{-3}$, while that calculated assuming 4 units of $Na_5P_3O_{10}$ per cell was $2{\cdot}59$ g.cm.$^{-3}$.

## Intensity data

Intensity data were obtained by visual estimation of $[a]$-, $[b]$- and $[c]$-axis zero-layer Weissenberg photographs taken with Cu $K\alpha$ radiation. A multiple-film technique was used and the various photographs were related and corrected for geometrical and polarization factors. The final set of data consisted of 106 $\{h0l\}$, 48 $\{hk0\}$ and 25 $\{0kl\}$ terms of measurable amplitude. Absorption corrections were not made, but errors were minimized by selecting, for each axis, small and as well formed crystals as were available.

## Structure determination

Space group $C2/c$ contains eightfold general point positions. Since the unit cell contains only four molecules of $Na_5P_3O_{10}$, the $P_3O_{10}^{5-}$ ion must lie either on a twofold symmetry axis or on a centre of symmetry. The latter can be excluded since a tetrahedral configuration of oxygen atoms about the central phosphorus atom is to be expected. The central phosphorus atom ($P_1$) must therefore lie on a twofold axis with the remaining atoms occupying sets of eightfold general positions. Similarly, one set of sodium ions ($Na_1$) will lie on centres of symmetry or twofold symmetry axes, while the remaining sodium ions ($Na_2$ and $Na_3$) are expected to occupy two sets of general positions and to occur in pairs related by twofold axial symmetry.

---

[*] Now at the National Institute of Mental Health, Bethesda, Maryland, U.S.A.

[†] Now at Research Department, Unilever Limited, Port Sunlight, Cheshire, England.

The Patterson function was computed for the [$b$]-axis projection and this enabled a preliminary set of $(x, z)$ co-ordinates to be obtained for the phosphorus atom $P_2$. Trial-and-error studies with models showed that with suitable $y$ co-ordinates for the triphosphate ion, a sodium atom located at $(\frac{1}{4}, \frac{3}{4}, \frac{1}{2})$ could be surrounded by a roughly octahedral arrangement of oxygen atoms and this was assumed to be the position of the unique sodium atom ($Na_1$). Structure factors for the $\{h0l\}$ zone were then calculated, using only the contributions of the atoms $P_1$, $P_2$ and $Na_1$. A Fourier synthesis was computed and this led to the assignment of $(x, z)$ co-ordinates to the remaining sodium ions and then to the oxygen atoms. The structure was then refined by successive electron-density and difference syntheses until no further reduction in the agreement factor, $R_{h0l}$, could be obtained.

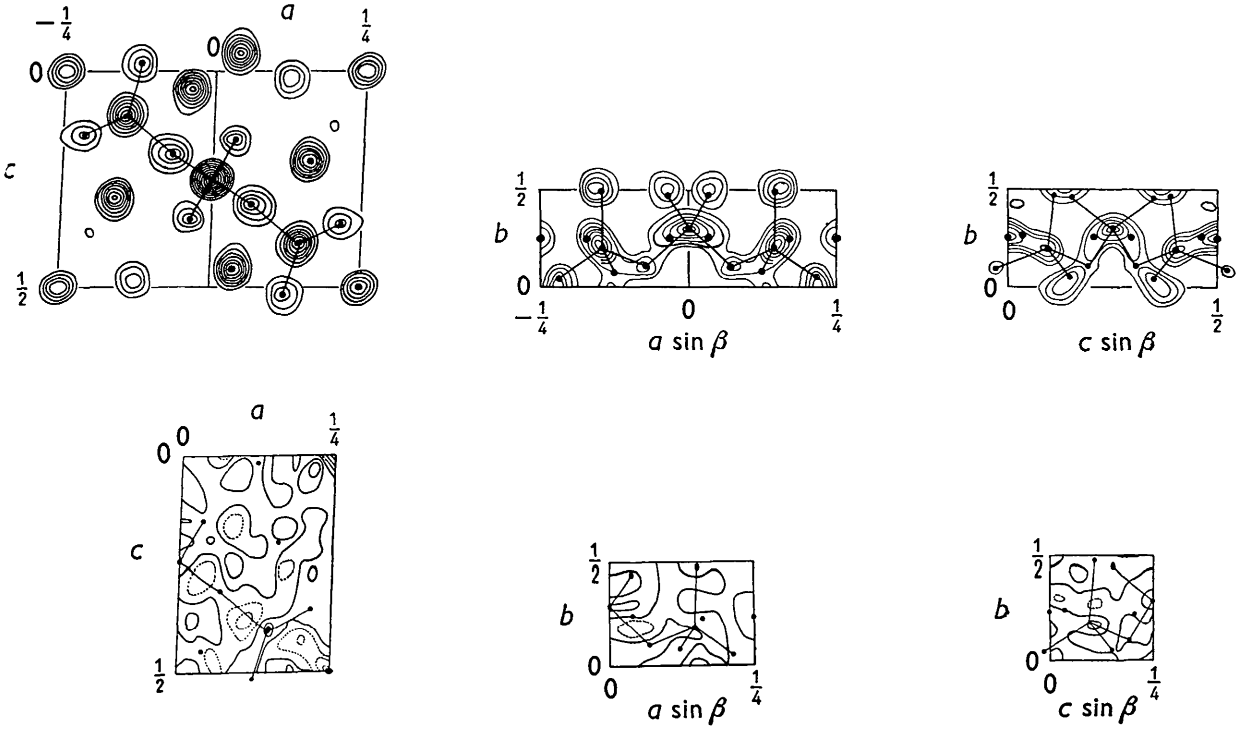
*The y co-ordinates*

Reasonable bond lengths and bond angles were assigned to the triphosphate ion, and with these the approximate $y$ co-ordinates of the atoms relative to the central phosphorus atom ($P_1$) could be deduced. The absolute $y$ co-ordinates of the triphosphate ion were then obtained by moving the ion along the two-fold axis until the sodium ion $Na_1$ at $(\frac{1}{4}, \frac{3}{4}, \frac{1}{2})$ was surrounded by six oxygen atoms at roughly equal distances. The $y$ co-ordinates of the remaining sodium ions $Na_2$ and $Na_3$ were then obtained from packing considerations. A preliminary calculation of the $\{hk0\}$ structure factors, using these $y$ co-ordinates and the $x$ co-ordinates from the [$b$]-axis projection, gave an agreement factor of $R = 28\%$. Refinement was then carried out by successive electron-density and difference syntheses. Using the co-ordinates obtained from the [$b$]- and [$c$]-axis projections, an electron-density and two difference syntheses were computed for the $\{0kl\}$ zone, after which $R_{0kl}$ could not be reduced further.

From the results of the three projections, a mean

Table 1. *Final atomic co-ordinates*

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| $P_1$ | 0·000 | 0·305 | 0·250 |
| $P_2$ | 0·146 | 0·195 | 0·400 |
| $O_1$ | 0·038 | 0·456 | 0·155 |
| $O_2$ | 0·067 | 0·103 | 0·306 |
| $O_3$ | 0·150 | 0·481 | 0·393 |
| $O_4$ | 0·216 | 0·053 | 0·348 |
| $O_5$ | 0·123 | 0·085 | 0·517 |
| $Na_1$ | 0·250 | 0·750 | 0·500 |
| $Na_2$ | 0·040 | 0·756 | 0·460 |
| $Na_3$ | 0·160 | 0·740 | 0·208 |



(a) (b) (c)

Fig. 1. Projections ($a$) along [010], ($b$) along [001], ($c$) along [100].
Electron-density and difference syntheses, with contours drawn at arbitrary intervals. Contours on difference maps (lower figures) drawn at half the intervals of the corresponding electron-density maps (upper figures). Negative contours drawn in broken lines.

set of parameters was chosen which gave the lowest values of the agreement factors. The final set of parameters is given in Table 1, for which values of $R_{h0l} = 13{\cdot}8\%$, $R_{hk0} = 11{\cdot}8\%$ and $R_{0kl} = 13{\cdot}4\%$ were obtained. Final electron-density projections are shown in Fig. 1.

A temperature factor with $B = 3{\cdot}3$ Å$^2$ was used for the preliminary stages of refinement, this value having been obtained from the $\{h0l\}$ intensities by the Wilson (1942) method. Subsequent comparison of observed and calculated structure factors indicated $B = 3{\cdot}0$ Å$^2$ as a more satisfactory value for the mean isotropic temperature factor of the three equatorial zones.

## Discussion of the structure

### *The configuration of the triphosphate ion*

A diagram of the structure in [*b*] projection is shown in Fig. 2. The triphosphate ion has a diad axis and the central phosphorus atom lies on a twofold axis of the cell. The ion is slightly distorted from a symmetrical *trans* configuration (Fig. 3) and the direction of the chain axis lies almost parallel to [101]. Table 2 lists the interatomic distances and angles calculated from the data in Table 1.

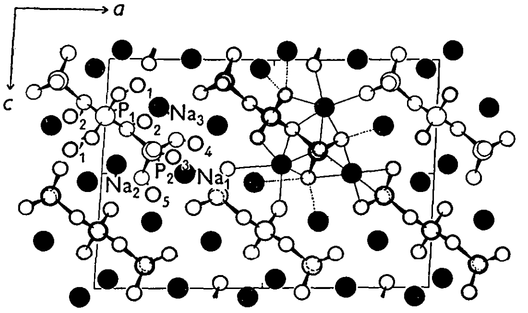


Fig. 2. Sodium triphosphate structure in *b* projection. Co-ordination of sodium ions shown by full lines and co-ordination of oxygen atoms completed by broken lines.

The accuracy of the structure determination was limited by the degree of overlap occurring in each projection, and the quality of the crystals obtainable. Calculation of the standard deviation of atomic co-ordinates by Cruickshank's method for the (010) projection, gives $\sigma_x = \sigma_z \sim 0{\cdot}025$ Å for the resolved oxygen atoms, and $\sigma_x = \sigma_z \sim 0{\cdot}006$ Å for the phosphorus atom. The values of the standard deviations are of the same order as the maximum differences found between the $x$, $y$ and $z$ co-ordinates obtained independently from each projection. These figures suggest a standard deviation of about $\pm 0{\cdot}03$ Å in the P–O bond length determinations. A significant difference between the inner and outer P–O chain bond lengths is therefore indicated, but a full three-dimensional analysis would be desirable to confirm this.

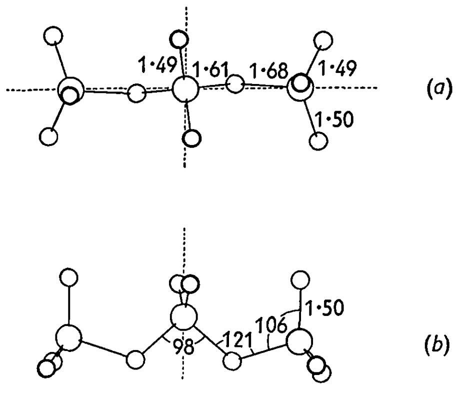


Fig. 3. Diagram of triphosphate ion viewed (*a*) in *b* projection; (*b*) perpendicular to ($\bar{1}01$) plane. Broken lines denote mirror planes of a truly *trans* symmetrical configuration. Distances in Ångström units and angles in degrees.

Table 2. *Interatomic distances and angles in sodium triphosphate*

| | | | | | |
|---|---|---|---|---|---|
| $P_1$–$O_1$ | = 1·485 Å | $O_1$–$O_1'$ | = 2·51 Å | $P_1$–$O_2$–$P_2$ | = 121·5° |
| $P_1$–$O_2$ | = 1·611 | $O_2$–$O_2'$ | = 2·43 | $O_1$–$P_1$–$O_1'$ | = 115·6 |
| $P_2$–$O_2$ | = 1·676 | $O_1'$–$O_2$ | = 2·55 | $O_1$–$P_1$–$O_2$ | = 110·0 |
| $P_2$–$O_3$ | = 1·502 | $O_2$–$O_1$ | = 2·54 | $O_1'$–$P_1$–$O_2$ | = 111·0 |
| $P_2$–$O_4$ | = 1·490 | $O_2$–$O_3$ | = 2·55 | $O_2'$–$P_1$–$O_2$ | = 97·9 |
| $P_2$–$O_5$ | = 1·500 | $O_2$–$O_4$ | = 2·42 | $O_2$–$P_2$–$O_3$ | = 106·7 |
| $P_1$–$P_2$ | = 2·87 | $O_2$–$O_5$ | = 2·49 | $O_2$–$P_2$–$O_5$ | = 103·4 |
| | | $O_3$–$O_4$ | = 2·54 | $O_2$–$P_2$–$O_4$ | = 99·4 |
| | | $O_3$–$O_5$ | = 2·55 | $O_3$–$P_2$–$O_4$ | = 116·3 |
| | | $O_4$–$O_5$ | = 2·48 | $O_3$–$P_2$–$O_5$ | = 116·3 |
| | | | | $O_4$–$P_2$–$O_5$ | = 112·1 |

### *Ionic co-ordination scheme*

The sodium ions are surrounded by oxygen atoms in distorted octahedral arrangements (Fig. 4), which link together to form a complex three-dimensional ionic structure. A comparatively large variation of the Na–O distance is found within the octahedra (Table 3) but the mean values in each case are not greatly different from those found in simpler structures, e.g. sodium phosphoramidate (2·39 and 2·43 Å), sodium sulphate (2·41 Å) and sodium bicarbonate (2·47 Å). A similar variation of co-ordination distances (2·36–2·78 Å) has been found in sodium pyrophosphate decahydrate (MacArthur & Beevers, 1957).

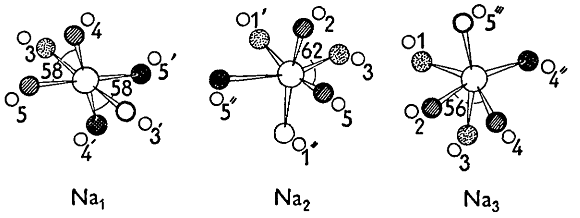


Fig. 4. Co-ordination of sodium ions, viewed as in Fig. 2. Oxygen atoms in each octahedra which belong to the same triphosphate ion are shaded similarly.

Table 3. *Sodium–oxygen distances*

| | | | | | |
|---|---|---|---|---|---|
| $Na_1$–$O_5$ | = 2·70 Å | $Na_2$–$O_2$ | = 2·56 Å | $Na_3$–$O_1$ | = 2·50 Å |
| $Na_1$–$O_3$ | = 2·41 | $Na_2$–$O_3$ | = 2·43 | $Na_3$–$O_5'''$ | = 2·38 |
| $Na_1$–$O_4$ | = 2·38 | $Na_2$–$O_5$ | = 2·25 | $Na_3$–$O_4''$ | = 2·33 |
| $Na_1$–$O_5'$ | = 2·70 | $Na_2$–$O_1''$ | = 2·46 | $Na_3$–$O_4$ | = 2·41 |
| $Na_1$–$O_3'$ | = 2·41 | $Na_2$–$O_5''$ | = 2·76 | $Na_3$–$O_3$ | = 2·50 |
| $Na_1$–$O_4'$ | = 2·38 | $Na_2$–$O_1'$ | = 2·35 | $Na_3$–$O_2$ | = 2·69 |
| $Na_1$–O(mean) | = 2·50 | $Na_2$–O(mean) | = 2·47 | $Na_3$–O(mean) | = 2·47 |

The sodium ions link the triphosphate chains in the [*b*] direction to corresponding chains in the next cell. In addition, the chains are linked 'end on' by the ions $Na_1^+$ and laterally by the ions $Na_2^+$ and $Na_3^+$. The $Na_2^+$ and $Na_3^+$ octahedra form continuous sheets parallel to the $(\bar{1}01)$ plane, and channels parallel to the [*b*] axis are formed by the $Na_1^+$ octahedra (Fig. 5).

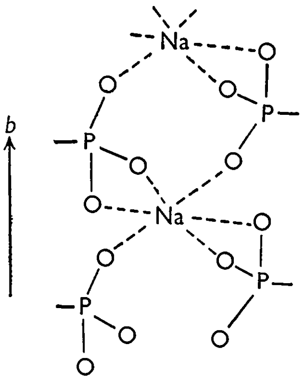


Fig. 5. Co-ordination scheme for $Na_1^+$ forming channels parallel to *b*.

The terminal atoms $O_1$, $O_3$ and $O_4$ are each co-ordinated to three sodium ions, forming a corner common to three octahedra. Terminal atom $O_5$ forms a corner common to four octahedra and $O_2$ is common to two octahedra. Only one case of two octahedra sharing a common edge is found, and this is between $Na_2$ and $Na_2'$, which share the long edge $O_5$–$O_5''$ (Fig. 4).

The edges of some octahedra are spanned by two oxygen atoms attached to the same phosphorus atom, and this may be associated with the distortion of the octahedra. The $Na_1^+$ octahedron has two edges and the $Na_2^+$ and $Na_3^+$ octahedra each have one edge spanned in this way. This type of mutual sharing of an edge between phosphorus tetrahedra and sodium octahedra is not found in sodium pyrophosphate decahydrate (MacArthur & Beevers, 1957).

## Comparison with other structures

In Table 4 the P–O bond parameters in the triphosphate ion are compared with the values found in other structures. The chain bonds in every case are considerably longer than those to terminal oxygen atoms, and the chain distances involving the central phosphorus atom lie close to the calculated 'single'-bond value (sum of covalent radii with Schomaker–Stevenson correction = 1·63 Å).

With the exception of zirconium pyrophosphate, which is reported to contain centrosymmetrical $P_2O_7^{4-}$ ions (Levi & Peyronel, 1935), non linear P–O–P linkages have been found in all condensed phosphates so far studied. Preliminary indications of a linear ion in acid sodium pyrophosphate, $Na_2H_2P_2O_7 . 6\,H_2O$ (Corbridge, 1957), have since been disproved (Corbridge, unpublished work). The chain O–P–O angles of near 100° (Table 4) may be compared with similar values found between other 'single' bonds involving phosphorus, as, for example, in black phosphorus (Hultgren & Warren, 1935), $P_4S_3$ (Van Houten, Vos & Wiegers, 1955), $P_4S_{10}$ (Vos & Wiebenga, 1955). The oxygen configuration around the end phosphorus atoms in the triphosphate ion resembles a flattened tetrahedron similar to that found in sodium pyrophosphate decahydrate, $Na_4P_2O_7 . 10\,H_2O$ (MacArthur & Beevers, 1957).

Table 4. *Phosphorus–oxygen bond parameters*

| | P–O (Å) | P–O–(P) (Å) | P–O–P (°) | O–P–O (°) | Reference |
|---|---|---|---|---|---|
| Phosphorus pentoxide I, $P_4O_{10}$ | 1·39 | 1·62 | 123 | 101 | Hampson & Stosick, 1938 |
| Ammonium tetrametaphosphate, $(NH_4PO_3)_4$ | 1·46 (mean) | 1·61 (mean) | 132 | 105 | Romers *et al.*, 1951 |
| Rubidium metaphosphate, $(RbPO_3)_n$ | 1·46 | 1·62 | 129 | 102 | Corbridge, 1956 |
| Sodium pyrophosphate decahydrate, $Na_4P_2O_7 . 10\,H_2O$ | 1·47 | 1·63 | 134 | — | MacArthur & Beevers, 1957 |
| Sodium triphosphate II | 1·50 | 1·61<br>and<br>1·68 | 121 | 98 | This work |

The trisulphate ion $S_3O_{10}^{--}$ (Eriks & MacGillavry, 1954) has a configuration similar, to, although somewhat less symmetrical than, that of the triphosphate ion. The difference in length between the chain bonds ($S\text{–}O_{outer} = 1{\cdot}77$ Å, $S\text{–}O_{inner} = 1{\cdot}55$ Å) appears to be more marked than in the triphosphate ion, and the terminal S–O bonds are very short (1·40 Å average). A flattening of the end tetrahedral group also occurs in the trisulphate ion, and the chain angles are similar to those in the triphosphate ion (O–S–O = 100°, S–O–S = 122°).

The much greater hydrolytic instability of the triphosphate compared with the pyrophosphate ion, may be associated with a weaker end-chain linkage in the former. The hydrolysis of long-chain phosphates proceeds mainly by repeated scission of the end groups, the rate of scission increasing with chain length (Crowther & Westman, 1954; McCullough, Van Wazer & Griffith, 1956). It is conceivable that all such phosphate chains containing more than two phosphorus atoms may be terminated by somewhat longer chain bonds, as the experimental results suggest for the triphosphate ion. In this connexion the lengths of the chain bonds in the tetraphosphate ion would be of interest.

The authors wish to thank Mrs A. L. Standing for assistance with the computation, and Mr F. R. Tromans for expert help in obtaining the photographs.

Table 5. *Observed and calculated structure amplitudes*

| $h\,k\,l$ | $F_o$ | $F_c$ |
|---|---|---|
| 2 0 0 | 41 | +43 |
| 4 | 38 | −40 |
| 6 | 111 | +113 |
| 8 | 49 | +50 |
| 10 | 137 | −138 |
| 12 | <6 | −3 |
| 14 | 42 | +34 |
| 16 | 26 | −22 |
| 18 | 5 | −3 |
| 20 | 25 | +23 |
| 0 0 2 | 8 | +6 |
| 4 | <7 | +16 |
| 6 | 15 | +18 |
| 8 | 28 | +29 |
| 10 | 30 | +31 |
| 12 | <10 | +4 |
| 14 | 19 | −14 |
| 0 2 0 | 91 | −100 |
| 4 | 84 | +82 |
| 6 | 16 | −20 |
| 2 0 2 | 80 | +92 |
| 4 | 89 | −85 |
| 6 | 74 | −74 |
| 8 | 64 | +64 |
| 10 | 24 | −30 |
| 12 | 128 | −118 |
| 14 | 35 | −37 |
| 16 | 28 | +31 |
| 18 | 41 | −40 |
| 20 | 6 | −3 |
| 2 0 4 | 137 | +144 |
| 4 | 27 | +40 |
| 6 | 45 | −31 |
| 8 | 183 | +157 |
| 10 | 55 | +42 |
| 12 | 15 | +14 |
| 14 | <10 | −10 |
| 16 | 12 | +29 |
| 18 | 10 | −13 |
| 2 0 6 | 96 | −90 |
| 4 | 177 | +178 |
| 6 | <10 | +2 |
| 8 | 56 | −48 |
| 10 | 15 | +9 |
| 12 | <10 | −1 |
| 14 | 74 | −62 |
| 16 | 8 | −10 |
| 18 | 15 | +8 |
| 2 0 8 | 52 | −50 |
| 4 | 20 | +14 |
| 6 | 74 | +69 |
| 8 | 37 | +37 |
| 10 | <10 | +2 |
| 12 | 34 | +27 |
| 14 | 16 | +14 |
| 16 | 13 | −6 |
| 2 0 10 | 60 | −62 |
| 4 | 69 | −58 |
| 6 | 30 | +29 |
| 8 | 15 | +18 |
| 10 | 41 | −36 |
| 12 | 22 | +22 |
| 14 | 10 | +14 |
| 2 0 12 | 27 | +18 |
| 4 | 16 | +15 |
| 6 | 6 | −6 |
| 8 | 58 | +46 |
| 10 | 5 | +6 |
| 2 0 14 | 9 | −13 |
| 4 | 8 | −14 |
| $\bar{2}$ 0 2 | 148 | −177 |
| $\bar{4}$ | 11 | +18 |
| $\bar{6}$ | 46 | −53 |
| $\bar{8}$ | 28 | +35 |
| $\overline{10}$ | 63 | −70 |
| $\overline{12}$ | 9 | +6 |
| $\overline{14}$ | 48 | −46 |
| $\overline{16}$ | 41 | −29 |
| $\overline{18}$ | 9 | −12 |
| $\bar{2}$ 0 4 | 15 | −32 |
| $\bar{4}$ | 75 | +94 |
| $\bar{6}$ | 19 | −23 |
| $\bar{8}$ | 124 | −107 |
| $\overline{10}$ | 33 | +17 |
| $\overline{12}$ | 46 | +46 |
| $\overline{14}$ | 57 | −57 |
| $\overline{16}$ | 38 | +45 |
| $\overline{18}$ | 35 | +48 |
| $\overline{20}$ | 8 | +9 |
| $\bar{2}$ 0 6 | 52 | +50 |
| $\bar{4}$ | 78 | −83 |
| $\bar{6}$ | 150 | −193 |
| $\bar{8}$ | <10 | −7 |
| $\overline{10}$ | <10 | −4 |
| $\overline{12}$ | 76 | −65 |
| $\overline{14}$ | 15 | −14 |
| $\overline{16}$ | 13 | +21 |
| $\overline{18}$ | 6 | −7 |
| $\bar{2}$ 0 8 | 24 | +13 |
| $\bar{4}$ | 13 | +16 |
| $\bar{6}$ | 38 | −43 |
| $\bar{8}$ | 82 | +82 |
| $\overline{10}$ | 30 | +22 |
| $\overline{12}$ | 15 | −9 |
| $\overline{14}$ | 13 | +6 |
| $\overline{16}$ | 30 | +31 |
| $\bar{2}$ 0 10 | 40 | −40 |
| $\bar{4}$ | 86 | −92 |
| $\bar{6}$ | <11 | −8 |
| $\bar{8}$ | <11 | +6 |
| $\overline{10}$ | 56 | −51 |
| $\overline{12}$ | 13 | +19 |
| $\overline{14}$ | 21 | +17 |
| $\bar{2}$ 0 12 | 24 | −27 |
| $\bar{4}$ | 9 | +12 |
| $\bar{6}$ | 6 | +5 |
| $\bar{8}$ | 8 | −3 |
| $\overline{10}$ | 22 | +16 |
| $\overline{12}$ | 20 | +22 |
| $\bar{2}$ 0 14 | 20 | −21 |
| $\bar{4}$ | 23 | +23 |
| $\bar{6}$ | 6 | −6 |
| 1 1 0 | 6 | +5 |
| 3 | 64 | −70 |
| 5 | 40 | −40 |
| 7 | 58 | −59 |
| 9 | 37 | +37 |
| 11 | 12 | −16 |
| 13 | 6 | +4 |
| 15 | 14 | +19 |
| 17 | <7 | +6 |
| 19 | <6 | +2 |
| 2 2 0 | 13 | −14 |
| 4 | 7 | −13 |
| 6 | 65 | −62 |
| 8 | 31 | −30 |
| 10 | 48 | +57 |
| 12 | 38 | −42 |
| 14 | <6 | +3 |
| 16 | <6 | +4 |
| 18 | <4 | +2 |
| 1 3 0 | 29 | −42 |
| 3 | 55 | +50 |
| 5 | 40 | +34 |
| 7 | 26 | −30 |
| 9 | 37 | +48 |
| 11 | 39 | +44 |
| 13 | 6 | −4 |
| 15 | <6 | +3 |
| 17 | 22 | +22 |
| 2 4 0 | 13 | −10 |
| 4 | 13 | +13 |
| 6 | 44 | +38 |
| 8 | 9 | +7 |
| 10 | 41 | −43 |
| 12 | <5 | −1 |
| 14 | <4 | −10 |
| 16 | 18 | −18 |
| 1 5 0 | 33 | −18 |
| 3 | 31 | −19 |
| 5 | 11 | −7 |
| 7 | 7 | +6 |
| 9 | 8 | −11 |
| 11 | 15 | −16 |
| 13 | <4 | +3 |
| 2 6 0 | <6 | 0 |
| 4 | 10 | −6 |
| 6 | 21 | −11 |
| 8 | 9 | +7 |
| 0 2 1 | 39 | +39 |
| 2 | 19 | −22 |
| 3 | 22 | +22 |
| 4 | 98 | −107 |
| 5 | 23 | −34 |
| 6 | 46 | +54 |
| 7 | <10 | −6 |
| 8 | <10 | +2 |
| 9 | 39 | −42 |
| 10 | 20 | −22 |
| 11 | 17 | +14 |
| 12 | <8 | +7 |
| 0 4 1 | 16 | −6 |
| 2 | 16 | +14 |
| 3 | 60 | −57 |
| 4 | <10 | +2 |
| 5 | <10 | +10 |
| 6 | <10 | −4 |
| 7 | <10 | +14 |
| 8 | <10 | +7 |
| 9 | 18 | +19 |
| 10 | <8 | +2 |
| 11 | 28 | −20 |
| 0 6 1 | <8 | −2 |
| 2 | 9 | −7 |
| 3 | 26 | +15 |
| 4 | 23 | −20 |
| 5 | <7 | −6 |
| 6 | 27 | −21 |

## References

CORBRIDGE, D. E. C. (1956). *Acta Cryst.* **9**, 308.

CORBRIDGE, D. E. C. (1957). *Acta Cryst.* **10**, 85.

CROWTHER, J. & WESTMAN, A. E. R. (1954). *Canad. J. Chem.* **32**, 42.

DYMON, J. J. & KING, A. J. (1951). *Acta Cryst.* **4**, 378.

ERIKS, K. & MACGILLAVRY, C. H. (1954). *Acta Cryst.* **7**, 430.

HAMPSON, G. C. & STOSICK, A. J. (1938). *J. Amer. Chem. Soc.* **60**, 1814.

HOUTEN, S. VAN, VOS, A. & WIEGERS, G. A. (1955). *Rec. Trav. Chim. Pays. Bas.* **74**, 1167.

HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210.

HULTGREN, R. & WARREN, B. E. (1935). *Phys. Rev.* **47**, 808.

LEVI, G. R. & PEYRONEL, G. (1935). *Z. Kristallogr.* **92**, 190.

MACARTHUR, D. & BEEVERS, C. A. (1957). *Acta Cryst.* **10**, 428.

MCCULLOUGH, J. F., WAZER, J. R. VAN & GRIFFITH, E. J. (1956). *J. Amer. Chem. Soc.* **78**, 4528.

ROMERS, C., KETELAAR, J. A. A. & MACGILLAVRY, C. H. (1951). *Acta Cryst.* **4**, 114.

VOS, A. & WIEBENGA, E. H. (1955). *Acta Cryst.* **8**, 217.

WILSON, A. J. C. (1942). *Nature, Lond.* **150**, 152.