# 1045. *Fungal Metabolites. Part II.*[1] *The Structure of Byssochlamic Acid: X-Ray Analysis of Byssochlamic Acid Bis-*p*-bromophenylhydrazide.*[2]

By I. C. PAUL, G. A. SIM, T. A. HAMOR, and J. MONTEATH ROBERTSON.

The molecular structure of byssochlamic acid, $C_{18}H_{20}O_6$, the characteristic metabolite of *Byssochlamys fulva*, has been determined by an *X*-ray study of the heavy-atom derivative, byssochlamic acid bis-*p*-bromophenylhydrazide. The crystals are tetragonal, of space group $P4_12_12$, with eight molecules of $C_{30}H_{30}Br_2N_4O_4$ in the unit cell of dimensions $a = b = 10{\cdot}07$, $c = 57{\cdot}61$ Å. Phase determination was based initially on the bromine atoms, and Fourier and least-squares refinement methods were employed in the determination of the atomic co-ordinates. Final results define the constitution and relative stereochemistry of byssochlamic acid bis-p-bromophenylhydrazide to be as in (I); byssochlamic acid is the corresponding dianhydride.

IN 1933 Raistrick and Smith[3] isolated from *Byssochlamys fulva* Olliver and Smith a metabolite, byssochlamic acid, $C_{18}H_{20}O_6$, which contains two anhydride rings. Byssochlamic acid and the biogenetically related glaucanic acid, $C_{18}H_{20}O_6$, and glauconic acid, $C_{18}H_{20}O_7$,[4] have since been investigated intensively by Professor D. H. R. Barton, F.R.S., Dr. J. K. Sutherland, and their collaborators.[5] In their studies they prepared a beautifully crystalline bis-*p*-bromophenylhydrazide by treating byssochlamic acid with *p*-bromophenylhydrazine in chloroform. We have determined the crystal structure of this derivative in order to elucidate the molecular structure of byssochlamic acid.

When we began our *X*-ray study the chemical information was very limited. We knew from earlier inconclusive chemical investigations[3,6] that the molecular formula was $C_{18}H_{20}O_6$ and that the " acid " contained two five-membered anhydride rings.

A direct determination of the crystal structure of the bis-*p*-bromophenylhydrazide was effected by means of the usual phase-determining heavy-atom method.[7] A number of three-dimensional electron-density distributions were evaluated, followed by a cycle of least-squares refinement of the atomic parameters, and at the end of the analysis the value of *R*, the average discrepancy between measured and calculated structure amplitudes, is 16·8% over 1190 independent reflexions.

Our final results establish the constitution and relative stereochemistry of the

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[1] Part I, Brown and Sim, *J.*, 1963, 1050.

[2] For a preliminary report see Hamor, Paul, Robertson, and Sim, *Experientia*, 1962, **18**, 352.

[3] Raistrick and Smith, *Biochem. J.*, 1933, **27**, 1814.

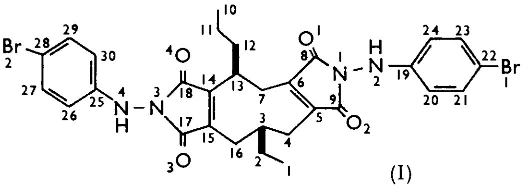
[4] Wijkman, *Annalen*, 1931, **485**, 61.

[5] Barton, Bloomer, Jackman, Rodriguez-Hahn, and Sutherland, *Experientia*, 1962, **18**, 345.

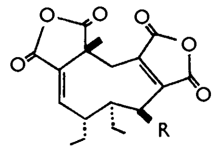
[6] See Paton, Ph.D. Thesis, 1954, University of Glasgow.

[7] Robertson and Woodward, *J.*, 1937, 219; 1940, 36; Sim, in " Computing Methods and the Phase Problem in *X*-ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon Press, Oxford, 1961, p. 227.

bis-*p*-bromophenylhydrazide to be as in (I); it follows that byssochlamic acid is the corresponding dianhydride.* The independent chemical studies at Imperial College are fully



(I)



(II)

consistent with this structure.[5] Byssochlamic acid is obviously closely related biogenetically to glauconic acid (II; R = OH) [5,8] and to glaucanic acid (II; R = H),[5] and a common biosynthetic route from acetate units *via* an intermediate citric acid has been proposed.[5]

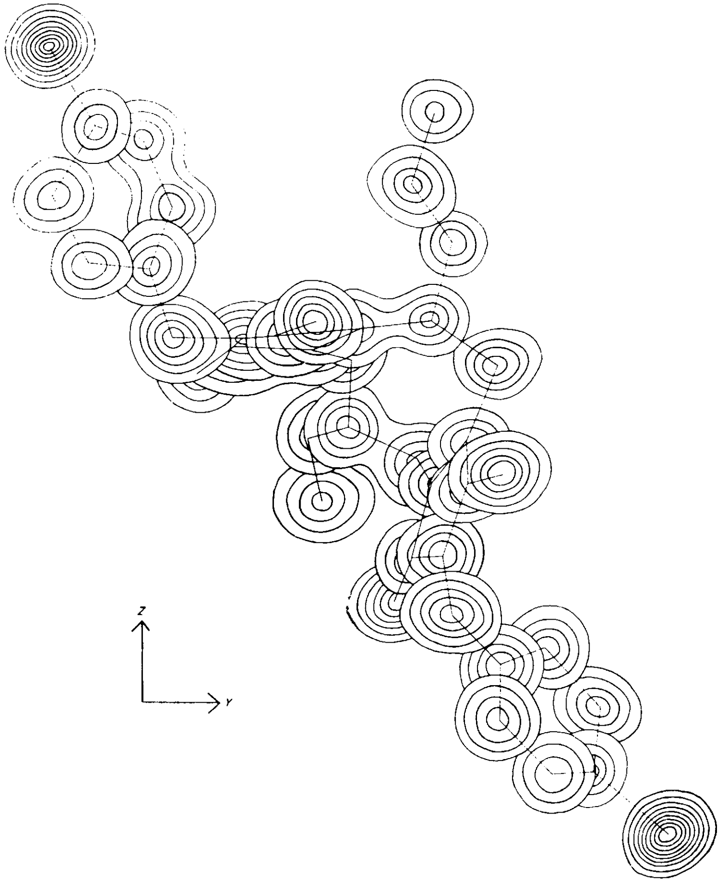


FIG. 1. The final three-dimensional electron-density distribution for byssochlamic acid bis-*p*-bromophenylhydrazide shown by means of superimposed contour sections drawn parallel to (100). Contour interval 1 eÅ$^{-3}$ except around the bromine atoms where it is 3 eÅ$^{-3}$. The lowest contour level is 2 eÅ$^{-3}$.

The final three-dimensional electron-density distribution for byssochlamic acid bis-*p*-bromophenylhydrazide is shown in Fig. 1 as superimposed contour sections drawn parallel

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* It will be apparent that byssochlamic acid is not an acid and that the derivative (I) would not normally be termed a hydrazide. The old nomenclature is, however, retained in this paper. ED.

[8] Ferguson, Sim, and Robertson, *Proc. Chem. Soc.*, 1962, 385.

TABLE 1.

Atomic co-ordinates and temperature factors.

(Origin of co-ordinates as in " International Tables." *)

| Atom | $x/a$ | $y/b$ | $x/c$ | $B$ | Atom | $x/a$ | $y/b$ | $x/c$ | $B$ |
|---|---|---|---|---|---|---|---|---|---|
| C(1) | 0·3966 | −0·1639 | 0·3798 | 6·4 | C(21) | −0·2681 | 0·2407 | 0·2879 | 5·6 |
| C(2) | 0·3299 | −0·1444 | 0·4038 | 6·4 | C(22) | −0·1551 | 0·3307 | 0·2897 | 5·6 |
| C(3) | 0·1836 | −0·1048 | 0·4029 | 4·7 | C(23) | −0·0870 | 0·3518 | 0·3101 | 5·6 |
| C(4) | 0·1689 | 0·0208 | 0·3909 | 4·7 | C(24) | −0·1238 | 0·2617 | 0·3272 | 5·6 |
| C(5) | 0·0220 | 0·0498 | 0·3836 | 5·0 | C(25) | −0·2664 | −0·4664 | 0·4579 | 5·6 |
| C(6) | −0·0842 | 0·1134 | 0·3967 | 4·7 | C(26) | −0·2075 | −0·4227 | 0·4759 | 5·6 |
| C(7) | −0·0686 | 0·1669 | 0·4225 | 4·7 | C(27) | −0·2268 | −0·4681 | 0·4990 | 5·6 |
| C(8) | −0·2037 | 0·1158 | 0·3828 | 5·0 | C(28) | −0·3192 | −0·5643 | 0·5024 | 5·6 |
| C(9) | −0·0458 | 0·0146 | 0·3624 | 4·7 | C(29) | −0·3736 | −0·6310 | 0·4836 | 5·6 |
| C(10) | −0·1862 | 0·0801 | 0·5044 | 6·4 | C(30) | −0·3528 | −0·5723 | 0·4600 | 5·6 |
| C(11) | −0·1998 | 0·0425 | 0·4818 | 6·4 | O(1) | −0·3009 | 0·1754 | 0·3861 | 5·6 |
| C(12) | −0·1234 | 0·1065 | 0·4609 | 6·4 | O(2) | 0·0173 | −0·0433 | 0·3454 | 5·6 |
| C(13) | −0·1559 | 0·0569 | 0·4379 | 4·7 | O(3) | 0·0579 | −0·3792 | 0·4282 | 5·6 |
| C(14) | −0·1362 | −0·0838 | 0·4356 | 5·0 | O(4) | −0·3504 | −0·1554 | 0·4392 | 5·6 |
| C(15) | −0·0193 | −0·1456 | 0·4306 | 4·7 | N(1) | −0·1707 | 0·0541 | 0·3630 | 5·6 |
| C(16) | 0·1285 | −0·1030 | 0·4295 | 4·7 | N(2) | −0·2568 | 0·0612 | 0·3417 | 5·6 |
| C(17) | −0·0452 | −0·2928 | 0·4309 | 4·3 | N(3) | −0·1831 | −0·3018 | 0·4331 | 5·6 |
| C(18) | −0·2441 | −0·1805 | 0·4343 | 4·3 | N(4) | −0·2518 | −0·4256 | 0·4354 | 5·6 |
| C(19) | −0·2048 | 0·1569 | 0·3266 | 5·6 | Br(1) | −0·1131 | 0·4561 | 0·2665 | 6·8 |
| C(20) | −0·2851 | 0·1491 | 0·3063 | 5·6 | Br(2) | −0·3574 | −0·6279 | 0·5312 | 6·8 |

* " International Tables for *X*-Ray Crystallography," The Kynoch Press, Birmingham, 1952, Vol. I.

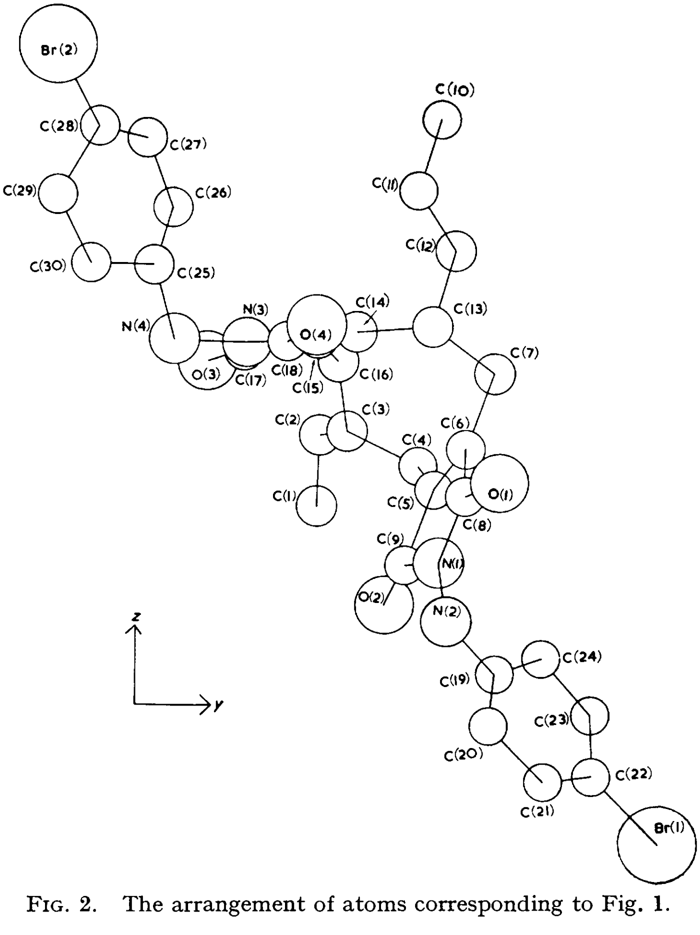

FIG. 2. The arrangement of atoms corresponding to Fig. 1.

to (100) and covering the region of one molecule; the corresponding atomic arrangement is explained in Fig. 2. The arrangement of the molecules in the crystal as viewed in projection along the *a*-axis is illustrated in Fig. 3. The conformation of the nine-membered carbocyclic ring can be studied in Fig. 4.

The interatomic distances and valency angles calculated from the final atomic co-ordinates (see Table 1) are listed in Table 2. The standard deviations of the final atomic

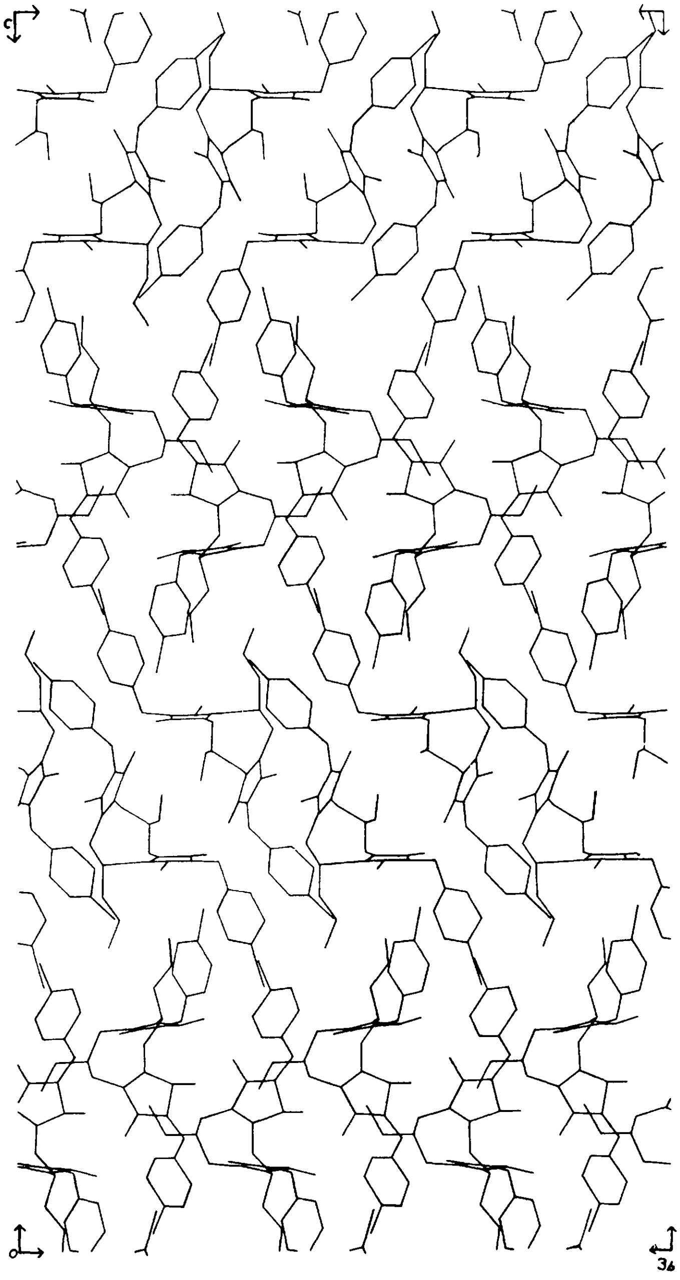

FIG. 3. Line drawings of the molecular frameworks of three unit cells as seen in projection along the *a*-axis.

TABLE 2.

Interatomic distances (Å) and angles.

*Intramolecular bonded distances.*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C(1)–C(2) | 1·55 | C(8)–N(1) | 1·34 | C(17)–O(3) | 1·36 | C(23)–C(24) | 1·39 |
| C(2)–C(3) | 1·53 | C(9)–O(2) | 1·31 | C(17)–N(3) | 1·40 | C(25)–N(4) | 1·37 |
| C(3)–C(4) | 1·45 | C(9)–N(1) | 1·32 | C(18)–O(4) | 1·14 | C(25)–C(26) | 1·27 |
| C(3)–C(16) | 1·63 | C(10)–C(11) | 1·36 | C(18)–N(3) | 1·37 | C(25)–C(30) | 1·38 |
| C(4)–C(5) | 1·57 | C(11)–C(12) | 1·57 | C(19)–N(2) | 1·40 | C(26)–C(27) | 1·42 |
| C(5)–C(6) | 1·46 | C(12)–C(13) | 1·45 | C(19)–C(20) | 1·43 | C(27)–C(28) | 1·36 |
| C(5)–C(9) | 1·44 | C(13)–C(14) | 1·44 | C(19)–C(24) | 1·33 | C(28)–C(29) | 1·39 |
| C(6)–C(7) | 1·59 | C(14)–C(15) | 1·36 | C(20)–C(21) | 1·41 | C(28)–Br(2) | 1·82 |
| C(6)–C(8) | 1·45 | C(14)–C(18) | 1·46 | C(21)–C(22) | 1·46 | C(29)–C(30) | 1·50 |
| C(7)–C(13) | 1·67 | C(15)–C(16) | 1·55 | C(22)–C(23) | 1·38 | N(1)–N(2) | 1·50 |
| C(8)–O(1) | 1·16 | C(15)–C(17) | 1·51 | C(22)–Br(1) | 1·89 | N(3)–N(4) | 1·43 |

*Intramolecular non-bonded distances.*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C(1) ··· C(4) | 3·02 | C(5) ··· C(15) | 3·38 | C(8) ··· C(13) | 3·26 | C(18) ··· C(26) | 3·44 |
| C(3) ··· C(6) | 3·50 | C(5) ··· C(16) | 3·24 | C(8) ··· C(14) | 3·71 | C(19) ··· O(1) | 3·57 |
| C(3) ··· C(7) | 3·90 | C(6) ··· C(14) | 3·04 | C(8) ··· C(19) | 3·27 | C(19) ··· O(2) | 3·20 |
| C(3) ··· C(13) | 4·29 | C(6) ··· C(15) | 3·33 | C(8) ··· C(24) | 3·62 | C(20) ··· N(1) | 3·59 |
| C(3) ··· C(14) | 3·74 | C(6) ··· C(16) | 3·59 | C(9) ··· C(19) | 2·98 | C(24) ··· O(1) | 3·93 |
| C(3) ··· C(17) | 3·39 | C(7) ··· C(11) | 3·87 | C(9) ··· C(24) | 3·31 | C(24) ··· O(2) | 3·54 |
| C(3) ··· O(3) | 3·37 | C(7) ··· C(15) | 3·22 | C(11) ··· O(4) | 3·51 | C(24) ··· N(1) | 2·97 |
| C(4) ··· C(7) | 3·35 | C(7) ··· C(16) | 3·39 | C(13) ··· C(16) | 3·32 | C(25) ··· O(3) | 3·79 |
| C(4) ··· C(13) | 4·26 | C(7) ··· C(18) | 3·98 | C(13) ··· O(1) | 3·53 | C(25) ··· O(4) | 3·42 |
| C(4) ··· C(14) | 4·14 | C(7) ··· O(1) | 3·15 | C(13) ··· O(4) | 2·90 | C(26) ··· O(3) | 3·86 |
| C(4) ··· C(15) | 3·41 | O(1) ··· N(2) | 2·84 | C(16) ··· O(3) | 2·87 | C(26) ··· O(4) | 3·71 |
| C(4) ··· O(2) | 3·10 | O(2) ··· N(2) | 2·96 | C(17) ··· C(25) | 3·23 | C(26) ··· N(3) | 2·76 |
| C(5) ··· C(13) | 3·61 | O(3) ··· N(4) | 3·18 | C(17) ··· C(26) | 3·33 | C(30) ··· N(3) | 3·57 |
| C(5) ··· C(14) | 3·65 | O(4) ··· N(4) | 2·91 | C(18) ··· C(25) | 3·19 | | |

*Intermolecular distances* (<4 Å).

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| O(3) ··· N(2)$^{\mathrm{I}}$ | 2·86 | O(2) ··· C(30)$^{\mathrm{I}}$ | 3·64 | C(2) ··· O(4)$^{\mathrm{VII}}$ | 3·81 | C(26) ··· C(10)$^{\mathrm{IV}}$ | 3·92 |
| O(2) ··· N(4)$^{\mathrm{I}}$ | 2·94 | C(2) ··· O(2)$^{\mathrm{I}}$ | 3·67 | C(10) ··· C(28)$^{\mathrm{VI}}$ | 3·82 | C(16) ··· C(10)$^{\mathrm{IV}}$ | 3·93 |
| Br(1) ··· C(27)$^{\mathrm{II}}$ | 3·25 | C(10) ··· C(29)$^{\mathrm{VI}}$ | 3·67 | C(27) ··· N(4)$^{\mathrm{IV}}$ | 3·82 | C(16) ··· C(22)$^{\mathrm{III}}$ | 3·93 |
| O(3) ··· C(20)$^{\mathrm{I}}$ | 3·27 | O(3) ··· C(19)$^{\mathrm{I}}$ | 3·69 | C(23) ··· C(30)$^{\mathrm{I}}$ | 3·85 | C(24) ··· O(1)$^{\mathrm{III}}$ | 3·93 |
| C(4) ··· O(1)$^{\mathrm{III}}$ | 3·44 | C(2) ··· C(23)$^{\mathrm{III}}$ | 3·70 | C(25) ··· Br(2)$^{\mathrm{IV}}$ | 3·85 | C(26) ··· C(28)$^{\mathrm{IV}}$ | 3·94 |
| C(27) ··· C(27)$^{\mathrm{IV}}$ | 3·44 | C(22) ··· C(29)$^{\mathrm{I}}$ | 3·72 | C(29) ··· Br(2)$^{\mathrm{IV}}$ | 3·86 | O(3) ··· Br(2)$^{\mathrm{IX}}$ | 3·94 |
| C(5) ··· O(1)$^{\mathrm{III}}$ | 3·48 | C(27) ··· C(28)$^{\mathrm{IV}}$ | 3·72 | O(4) ··· Br(2)$^{\mathrm{IV}}$ | 3·86 | Br(1) ··· C(26)$^{\mathrm{II}}$ | 3·94 |
| C(1) ··· O(2)$^{\mathrm{I}}$ | 3·49 | C(1) ··· N(3)$^{\mathrm{I}}$ | 3·73 | C(16) ··· C(23)$^{\mathrm{III}}$ | 3·87 | C(4) ··· C(19)$^{\mathrm{III}}$ | 3·96 |
| C(28) ··· C(28)$^{\mathrm{IV}}$ | 3·50 | C(11) ··· C(29)$^{\mathrm{VI}}$ | 3·73 | O(2) ··· O(4)$^{\mathrm{I}}$ | 3·87 | C(4) ··· C(23)$^{\mathrm{III}}$ | 3·96 |
| C(4) ··· C(24)$^{\mathrm{III}}$ | 3·54 | C(1) ··· O(3)$^{\mathrm{I}}$ | 3·74 | C(12) ··· C(29)$^{\mathrm{VI}}$ | 3·88 | C(24) ··· C(30)$^{\mathrm{I}}$ | 3·96 |
| C(30) ··· Br(2)$^{\mathrm{IV}}$ | 3·55 | C(10) ··· Br(2)$^{\mathrm{VI}}$ | 3·74 | C(23) ··· O(4)$^{\mathrm{III}}$ | 3·88 | C(28) ··· C(30)$^{\mathrm{IV}}$ | 3·97 |
| C(1) ··· C(17)$^{\mathrm{I}}$ | 3·58 | C(28) ··· C(29)$^{\mathrm{IV}}$ | 3·77 | C(7) ··· C(21)$^{\mathrm{III}}$ | 3·90 | C(9) ··· O(1)$^{\mathrm{III}}$ | 3·98 |
| C(26) ··· C(27)$^{\mathrm{IV}}$ | 3·59 | O(2) ··· C(25)$^{\mathrm{I}}$ | 3·77 | C(12) ··· C(21)$^{\mathrm{III}}$ | 3·90 | C(12) ··· C(30)$^{\mathrm{VI}}$ | 3·98 |
| C(1) ··· C(9)$^{\mathrm{I}}$ | 3·61 | O(2) ··· N(3)$^{\mathrm{I}}$ | 3·77 | C(15) ··· C(10)$^{\mathrm{IV}}$ | 3·90 | C(27) ··· C(11)$^{\mathrm{IV}}$ | 3·98 |
| C(7) ··· C(20)$^{\mathrm{III}}$ | 3·62 | Br(1) ··· C(28)$^{\mathrm{II}}$ | 3·78 | C(10) ··· Br(1)$^{\mathrm{VIII}}$ | 3·92 | C(23) ··· C(29)$^{\mathrm{I}}$ | 3·99 |
| C(20) ··· Br(2)$^{\mathrm{V}}$ | 3·64 | | | | | | |

The superscripts refer to the following positions:

I $\frac{1}{2} + x,\ -\frac{1}{2} - y,\ \frac{3}{4} - z.$
II $\frac{1}{2} + y,\ \frac{1}{2} - x,\ \frac{3}{4} + z.$
III $\frac{1}{2} + x,\ \frac{1}{2} - y,\ \frac{3}{4} - z.$
IV $y,\ x,\ -z.$
V $-\frac{1}{2} + x,\ \frac{1}{2} - y,\ \frac{3}{4} - z.$
VI $x,\ 1 + y,\ z.$
VII $1 + x,\ y,\ z.$
VIII $\frac{1}{2} - y,\ \frac{1}{2} + x,\ \frac{1}{4} + z.$
IX $1 + y,\ x,\ -z.$

*Valency angles.*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C(1)C(2)C(3) | 115° | C(5)C(9)N(1) | 111° | O(3)C(17)N(3) | 136° | C( N(4) | 112° |
| C(2)C(3)C(4) | 110 | O(2)C(9)N(1) | 128 | C(14)C(18)C(4) | 123 | C( C(27) | 126 |
| C(2)C(3)C(16) | 107 | C(10)C(11)C(12) | 125 | C(14)C(18)N(3) | 105 | C( C(28) | 117 |
| C(4)C(3)C(16) | 114 | C(11)C(12)C(13) | 117 | C(4)C(18)N(3) | 129 | C(27)C(28)Br(2) | 122 |
| C(3)C(4)C(5) | 113 | C(12)C(13)C(7) | 98 | C(20)C(19)C(24) | 114 | C(29)C(28)Br(2) | 117 |
| C(4)C(5)C(6) | 129 | C(12)C(13)C(14) | 113 | C(20)C(19)N(2) | 105 | C(28)C(29)C(30) | 118 |
| C(4)C(5)C(9) | 129 | C(7)C(13)C(14) | 105 | C(24)C(19)N(2) | 139 | C(25)C(30)C(29) | 118 |
| C(6)C(5)C(9) | 101 | C(13)C(14)C(15) | 126 | C(19)C(20)C(21) | 121 | C(8)N(1)C(9) | 113 |
| C(5)C(6)C(7) | 124 | C(13)C(14)C(18) | 124 | C(20)C(21)C(22) | 117 | C(8)N(1)N(2) | 122 |
| C(5)C(6)C(8) | 109 | C(15)C(14)(C18) | 109 | C(21)C(22)C(23) | 123 | C(9)N(1)N(2) | 123 |
| C(7)C(6)C(8) | 126 | C(14)C(15)C(16) | 135 | C(21)C(22)Br(1) | 123 | C(19)N(2)N(1) | 109 |
| C(6)C(7)C(13) | 103 | C(14)C(15)C(17) | 107 | C(23)C(22)Br(1) | 113 | C(17)N(3)C(18) | 113 |
| C(6)C(8)O(1) | 128 | C(16)C(15)C(17) | 116 | C(22)C(23)C(24) | 112 | C(17)N(3)N(4) | 123 |
| C(6)C(8)N(1) | 105 | C(3)C(16)C(15) | 111 | C(19)C(24)C(23) | 132 | C(18)N(3)N(4) | 124 |
| O(1)C(8)N(1) | 126 | C(15)C(17)O(3) | 120 | C(26)C(25)C(30) | 119 | C(25)N(4)N(3) | 114 |
| C(5)C(9)O(2) | 121 | C(15)C(17)N(3) | 104 | C(26)C(25)N(4) | 128 | C(27)C(28)C(29) | 120 |

co-ordinates were derived in the usual manner from the least-squares residuals (see Experimental section); the values obtained are 0·01 Å for the bromine atoms and 0·03—0·07 Å for oxygen, nitrogen, and carbon atoms. From the results the average e.s.d. of a carbon–carbon bond length is about 0·08 Å and the average e.s.d. of a valency angle is about 4·5°.

The average carbon–carbon bond length in the benzene rings, 1·39 Å, and the average *sp*$^3$-carbon–*sp*$^3$-carbon bond length, 1·53 Å, do not differ significantly from the values 1·397 Å and 1·545 Å in benzene and diamond, respectively. The average *sp*$^2$-carbon–*sp*$^2$-carbon single-bond length of 1·47 Å is close to the standard value of 1·48 Å proposed by Dewar and Schmeising[9] and to the values of 1·483 Å in butadiene[10] and 1·477 Å in *p*-benzoquinone.[11]

The average carbon–oxygen double-bond length of 1·24 Å compares favourably with the values of 1·222 Å in *p*-benzoquinone,[11] 1·23 Å in formaldehyde,[12] and 1·227 Å in succinimide.[13] The average *sp*$^2$-carbon–bromine bond length of 1·85 Å is not significantly different from the values of 1·86 Å in 1,3,5-tribromobenzene,[14] 1·89 Å in *o*-bromobenzoic acid,[15] and 1·90 Å in 1,2,4,5-tetrabromobenzene.[16] The average *sp*$^2$-carbon–nitrogen bond length, 1·37 Å, is in good agreement with the values reported for such bonds in *p*-nitroaniline, 1·371 Å,[17] 2-chloro-4-nitroaniline, 1·38 Å,[18] succinimide, 1·385 Å,[13] and *N*-chlorosuccinimide, 1·39 Å.[19] The average nitrogen–nitrogen single-bond length is 1·47 Å, in good agreement with the values of 1·46 and 1·47 Å reported in *X*-ray[20] and electron-diffraction[21] studies of hydrazine.

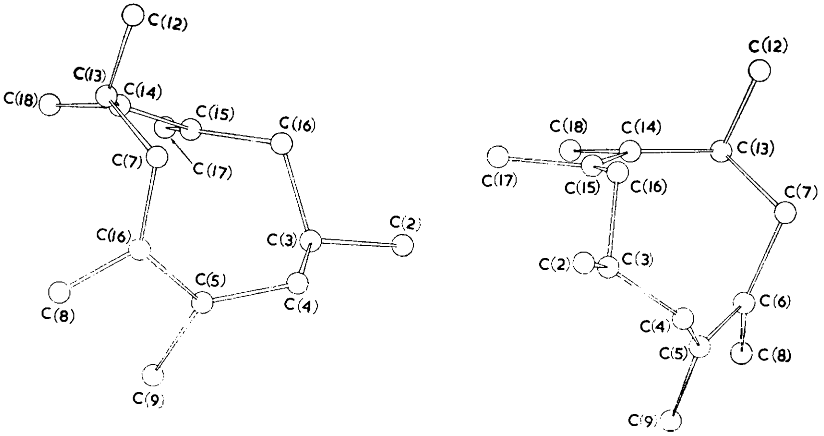

FIG. 4(*a*). FIG. 4(*b*).

FIG. 4. The conformation of the nine-membered ring as seen in projection down (*a*) the *b*-axis, and (*b*) the *a*-axis.

The average valency angle at an *sp*$^3$-hybridized carbon atom is 111°, not significantly different from tetrahedral. The average value of the angles in the benzene rings is 120°.

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[9] Dewar and Schmeising, *Tetrahedron*, 1959, **5**, 166.
[10] Almenningen, Bastiansen, and Traetteberg, *Acta Chem. Scand.*, 1958, **12**, 1221.
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[13] Mason, *Acta Cryst.*, 1961, **14**, 720.
[14] Milledge and Pant, *Acta Cryst.*, 1960, **13**, 285.
[15] Ferguson and Sim., *Acta Cryst.*, 1962, **15**, 346.
[16] Gafner and Herbstein, *Acta Cryst.*, 1960, **13**, 706.
[17] Trueblood, Goldish, and Donohue, *Acta Cryst.*, 1961, **14**, 1009.
[18] McPhail and Sim, unpublished results.
[19] Brown, *Acta Cryst.*, 1961, **14**, 711.
[20] Collin and Lipscomb, *Acta Cryst.*, 1951, **4**, 10.
[21] Giguère and Schomaker, *J. Amer. Chem. Soc.*, 1943, **65**, 2025.

In the maleimide rings the CNC angles (113°) are distinctly larger than the NCC and CCC angles; a similar pattern exists in succinimide[13] and *N*-chlorosuccinimide.[19] Since the sum of the valency angles at N(1) is 358° and at N(3) is 360°, it is clear that the state of hybridization of these atoms is $sp^2$ and not $sp^3$.

There are two hydrogen-bonded contacts of less than 3Å between molecules in the crystal, O(3)⋯N(2)$^{\text{I}}$ of 2·86 Å and O(2)⋯N(4)$^{\text{I}}$ of 2·94 Å, where the superscript I refers to the position $\frac{1}{2} + x$, $-\frac{1}{2} - y$, $\frac{3}{4} - z$. These distances are comparable with NH⋯O hydrogen-bonded distances in other crystals, *e.g.*, 2·79, 2·83, 2·96 Å in L-serine phosphate,[22] 2·86, 2·92, 2·94 Å in L-glutamic acid.[23] The other intermolecular contacts (see Table 2) are all greater than 3 Å and correspond to normal van der Waals interactions.

The final isotropic temperature factors assigned to the atoms of the byssochlamic acid bis-*p*-bromophenylhydrazide molecule are listed in Table 1. The carbon atoms of the central part of the molecule (the nine-membered ring and the maleimide rings) have smaller temperature factors than the atoms of the peripheral groups. The central carbon atoms have $B \approx 4{\cdot}8$ Å$^2$, the benzene-carbon atoms, the nitrogen atoms, and the oxygen atoms have $B = 5{\cdot}6$ Å$^2$, the ethyl and n-propyl side-chains have $B = 6{\cdot}4$ Å$^2$, and the bromine atoms have $B = 6{\cdot}8$ Å$^2$. These variations are reminiscent of those observed in echitamine bromide[24] and are clearly physically acceptable.

## EXPERIMENTAL

*Crystal Data.*—Byssochlamic acid bis-*p*-bromophenylhydrazide, $C_{30}H_{30}Br_2N_4O_4$; *M*, 669·8; m. p. 165—166°. Tetragonal, $a = b = 10{\cdot}07$, $c = 57{\cdot}61$ Å, $U = 5842$ Å$^3$, $D_m = 1{\cdot}505$ g. cm.$^{-3}$ (by flotation), $Z = 8$, $D_c = 1{\cdot}524$ g. cm.$^{-3}$, space group $P4_12_12 - D_4^4$ (or the enantiomorphous $P4_32_12 - D_4^8$). Absorption coefficient for *X*-rays ($\lambda = 1{\cdot}542$ Å) $\mu = 40{\cdot}3$ cm.$^{-1}$. Total number of electrons in the unit cell $= F(000) = 2720$.

*Measurements.*—Rotation, oscillation, and Weissenberg photographs were taken with copper-$K_\alpha$ ($\lambda = 1{\cdot}542$ Å) radiation; precession photographs were taken with molybdenum-$K_\alpha$ ($\lambda = 0{\cdot}7107$ Å) radiation. The cell dimensions were obtained from the precession and rotation photographs. The space group was determined from the systematic halvings in the reflexions. For the intensity survey a small crystal was employed, completely bathed in a uniform *X*-ray beam, and no corrections for absorption were applied. The intensities were estimated visually from multiple-film equatorial and equi-inclination upper-layer Weissenberg photographs obtained from a crystal rotated about the *a*-axis. The usual correction factors (Lorentz, polarization, and rotation) were applied and 1190 independent structure amplitudes derived. The various sets of structure amplitudes were placed on the same relative scale by comparison of equivalent reflexions *hkl* and *khl*; the absolute scale was obtained at a later stage by correlation with the final calculated structure amplitudes, $|F_c|$.

The crystal density was determined by flotation in aqueous zinc chloride.

*Analysis of the Structure.*—In view of the lengths of the unit-cell edges it was evident that there would be considerable overlap of atoms in projection along the crystallographic axes. Three-dimensional methods were therefore used throughout for the elucidation of the structure and for the refinement of the atomic parameters.

In order to determine the positions of the two bromine atoms in the asymmetric crystal unit (one molecule of byssochlamic acid bis-*p*-bromophenylhydrazide) we calculated a sharpened three-dimensional Patterson function $P(UVW)$. Sections through this function at $W = \frac{1}{4}$, $W = \frac{1}{2}$, and $V = \frac{1}{2}$, which contain peaks representing vectors between symmetry-related bromine atoms, are shown in Fig. 5. A detailed consideration of these sections and of the general Patterson peaks correspondingly to vectors between non-related bromine atoms enabled us to assign initial co-ordinates to the bromine atoms.[25]

The Fourier programme[26] for the DEUCE computer cannot be used in a straightforward

[22] McCallum, Robertson, and Sim, *Nature*, 1959, **184**, 1863.

[23] Hirokawa, *Acta Cryst.*, 1955, **8**, 637.

[24] Hamilton, Hamor, Robertson, and Sim., *J.*, 1962, 5061.

[25] Paul, Ph.D. Thesis, 1962, University of Glasgow.

[26] Rollett, in " Computing Methods and the Phase Problem in *X*-ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon Press, Oxford, 1961, p. 87.

way when the space group is $P4_12_12$. We overcame this difficulty by treating the unit cell in the subsequent structure-factor and Fourier calculations as if it belonged to the orthorhombic space group $P2_12_12_1$, with two molecules in the asymmetric crystal unit.

The first set of calculated structure amplitudes and phase angles was based on the bromine atoms alone; the value of $R$ was 48%. The first Fourier synthesis was then evaluated with the measured values of the structure amplitudes and the phase constants appropriate to the bromine atoms. The resulting three-dimensional electron-density distribution was displayed as contoured sections drawn on sheets of glass and stacked in a frame.

On examination of this distribution several chemical features were identified. A five-membered ring with a substituent on each atom indicated that the *p*-bromophenylhydrazide derivative preserves the five-membered ring structure of byssochlamic acid, *i.e.*, that the derivative can be formulated as (III). One of the bromine atoms, Br(1), was seen attached to a benzene ring which had a substituent in the *para*-position. The point of attachment of the other bromine atom could not be discerned.

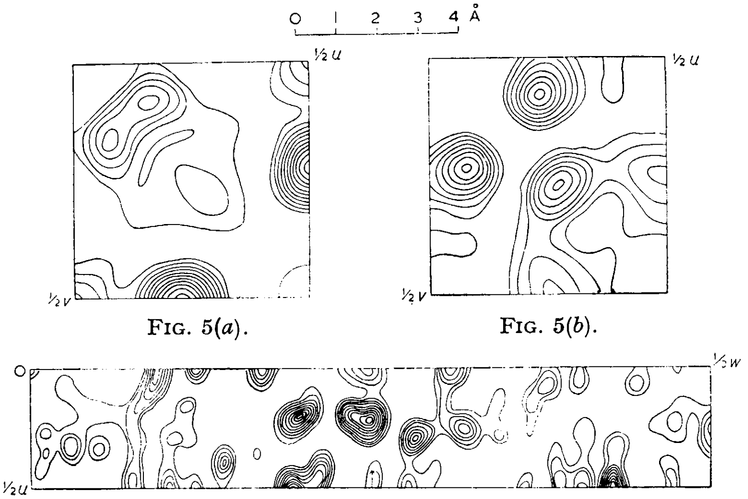
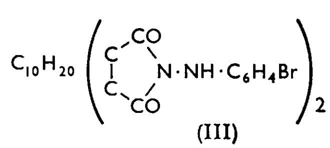
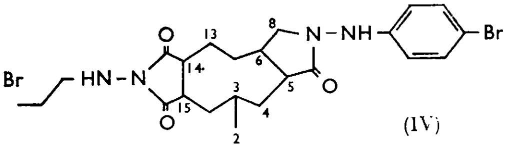


Fig. 5(*a*). Fig. 5(*b*).

Fig. 5. Sections through the three-dimensional sharpened Patterson function $P(UVW)$, (*a*) at $W = \frac{1}{4}$, (*b*) at $W = \frac{1}{2}$, and (*c*) at $V = \frac{1}{2}$. Contour scale arbitrary.



(III)



(IV)

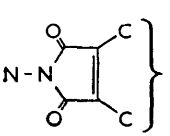
Although the complete molecular structure was not apparent at this stage, we were able to select sixteen well-defined peaks (C-14—21; C-24; N-1—4; O-3; O-4) for inclusion with the bromine atoms in the next round of structure-factor calculations. The light atoms were all weighted as carbon and an overall isotropic temperature factor $B$ of 4 Å$^2$ was adopted. The value of $R$ was reduced to 39%.

In the three-dimensional electron-density distribution derived from the improved phases all the atoms which had been included in the phasing calculations had satisfactory peak heights. Two of the atoms of the five-membered ring which had been discerned in the first electron-density distribution were now seen to participate also in a nine-membered ring which was fused to a further five-membered ring. The second five-membered ring was attached *via* an intermediate atom (presumably nitrogen) to the *para*-position of the bromobenzene ring located in the first electron-density distribution. A number of other apparently genuine peaks could be seen and when these were considered the atomic arrangement in (IV) was indicated at this stage.

The thirty light atoms (hydrogen being neglected) shown in formula (IV) were included in the third structure-factor calculation as carbon atoms and the overall isotropic temperature factor $B$ was increased to 5 Å$^2$. The value of $R$ was 31%.

In the subsequent electron-density distribution the last three carbon atoms of the benzene ring and the oxygen substituent at C(8) were clearly distinguished. The remaining four carbon atoms in the molecule, however, proved more difficult to locate unambiguously. Close to C(2) a peak [C(1)] of height 2·4 eÅ$^{-3}$ indicated that C(3) carried an ethyl group. There was a fairly substantial peak [A] of height 2·2 eÅ$^{-3}$ at a distance of about 2·3 Å from C(4) and this, with a smaller maximum of 1·4 eÅ$^{-3}$ nearer C(4), suggested that C(4) might also carry an ethyl group. At about 1·5 Å from C(13) a peak [C(12)] of height 2·8 eÅ$^{-3}$ appeared to be a genuine atom while a further 1·5 Å away from this peak there was another peak [C(11)] of height 2·2 eÅ$^{-3}$.

Models of the various possible structures were constructed on the assumption that there are two carbon–carbon double bonds in the nine-membered ring. The only model that simulated the conformation of the ring in the electron-density distribution was one in which there was a double bond between C(5) and C(6) and between C(14) and C(15). These locations were substantiated by the close planarity of the groups (V) in the molecule.



(V)

In the next calculation of structure factors the additional light atoms included were C(12), C(1), C(28), C(29), C(30), O(1), and A. Each atom was assigned its proper chemical type (peak A was treated as carbon) and an overall isotropic temperature factor $B$ of 5·5 Å$^2$ was adopted. The value of $R$ fell to 26%.

The electron-density distribution obtained from the improved phases provided clear evidence that there was no substituent on C(4). While the other carbon atoms in the molecule had peak heights of 4—5 eÅ$^{-3}$, the " atom " A now had a peak height of only 2·1 eÅ$^{-3}$ and was clearly spurious. On the other hand, the peak C(11) came up to a height of 2·6 eÅ$^{-3}$, although it had not been included in the phasing calculations. On calculation of a further set of structure factors with the atom C(11) replacing the atom A, the value of $R$ was 22% and in the electron-density distribution derived from the new phases the last carbon atom [C(10)] was located as the terminal carbon of an n-propyl group attached to C(13). The structure (I) was now established for byssochlamic acid bis-*p*-bromophenylhydrazide.

Two further rounds of structure-factor and Fourier calculations were carried out. Both $F_o$ and $F_c$ syntheses were computed, back-shift corrections for errors due to termination of series were applied to the atomic co-ordinates, and individual isotropic temperature factors were assigned. The average discrepancy between measured and calculated structure amplitudes fell to 18·1%.

The analysis was completed by means of a cycle of least-squares refinement of positional and thermal atomic parameters. The programme employed was that devised by Rollett,[26] and the weighting scheme employed was:

$$\sqrt{w} = |F_o|/43 \quad \text{if} \quad |F_o| < 43;$$
$$\sqrt{w} = 43/|F_o| \quad \text{if} \quad |F_o| > 43.$$

The anisotropic temperature-factor parameters, $b_{ij}$, given by the least-squares programme did not indicate a marked degree of anisotropic thermal motion and we therefore derived from the anisotropic parameters for each atom an isotropic temperature factor $B$. These isotropic temperature factors are listed with the final atomic co-ordinates in Table 1.

The final set of structure factors based on the co-ordinates and temperature factors of Table 1 was then calculated; the value of $R$ was 16·8%. The final values of $|F_o|$, $|F_c|$, and $\alpha$ are shown in Table 3. The theoretical atomic scattering factors used in all the structure-factor calculations were those of Berghuis *et al.*[27] for carbon, nitrogen, and oxygen, and the Thomas-Fermi values [28] for bromine.

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[27] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.

[28] " Internationale Tabellen zur Bestimmung von Kristallstrukturen," Borntraeger, Berlin, 1935, Vol. II, p. 572.

TABLE 3.

Measured and calculated values of the structure factors.

| *h* | *k* | *l* | $|F_o|$ | $|F_c|$ | α° |
|---|---|---|---|---|---|
| 0 | 0 | 4 | 247 | 211 | 0 |
| | | 8 | 39 | 50 | 180 |
| | | 16 | 48 | 69 | 0 |
| | | 20 | 265 | 270 | 180 |
| | | 24 | 239 | 244 | 180 |
| | | 28 | 162 | 156 | 0 |
| | | 32 | 49 | 75 | 0 |
| | | 36 | 104 | 87 | 180 |
| | | 40 | 109 | 98 | 180 |
| | | 44 | 38 | 33 | 0 |
| | | 48 | 40 | 26 | 180 |
| | | 52 | 66 | 56 | 0 |
| | | 56 | 30 | 11 | 0 |
| | | 60 | 36 | 38 | 0 |
| | | 64 | 21 | 29 | 0 |
| | | 68 | 18 | 11 | 0 |
| 0 | 1 | 2 | 82 | 118 | 180 |
| | | 3 | 54 | 54 | 315 |
| | | 4 | 54 | 47 | 270 |
| | | 5 | 82 | 90 | 45 |
| | | 6 | 150 | 160 | 180 |
| | | 7 | 93 | 139 | 315 |
| | | 8 | 154 | 158 | 270 |
| | | 9 | 97 | 98 | 225 |
| | | 10 | 16 | 15 | 0 |
| | | 11 | 105 | 104 | 315 |
| | | 12 | 204 | 247 | 270 |
| | | 13 | 200 | 199 | 45 |
| | | 14 | 213 | 213 | 0 |
| | | 15 | 56 | 51 | 135 |
| | | 16 | 68 | 80 | 270 |
| | | 17 | 120 | 128 | 45 |
| | | 18 | 118 | 143 | 0 |
| | | 19 | 64 | 80 | 135 |
| | | 20 | 154 | 157 | 270 |
| | | 21 | 102 | 86 | 225 |
| | | 22 | 136 | 95 | 180 |
| | | 23 | 33 | 10 | 315 |
| | | 24 | 114 | 107 | 270 |
| | | 25 | 59 | 81 | 225 |
| | | 26 | 16 | 33 | 180 |
| | | 27 | 101 | 101 | 135 |
| | | 28 | 14 | 25 | 270 |
| | | 29 | 41 | 28 | 45 |
| | | 30 | 41 | 45 | 180 |
| | | 31 | 121 | 118 | 135 |
| | | 32 | 58 | 56 | 90 |
| | | 33 | 122 | 121 | 45 |
| | | 34 | 99 | 95 | 180 |
| | | 35 | 39 | 28 | 315 |
| | | 36 | 12 | 14 | 90 |
| | | 37 | 15 | 2 | 45 |
| | | 38 | 35 | 34 | 180 |
| | | 39 | 22 | 20 | 315 |
| | | 40 | 32 | 33 | 90 |
| | | 41 | 58 | 58 | 45 |
| | | 42 | 19 | 10 | 0 |
| | | 43 | 13 | 16 | 315 |
| | | 44 | 67 | 60 | 90 |
| | | 45 | 89 | 77 | 45 |
| 0 | 1 | 46 | 29 | 28 | 0 |
| | | 47 | 17 | 6 | 135 |
| | | 48 | 66 | 62 | 90 |
| | | 50 | 29 | 22 | 0 |
| | | 53 | 73 | 63 | 225 |
| | | 54 | 27 | 11 | 180 |
| | | 55 | 14 | 1 | 315 |
| | | 58 | 20 | 13 | 0 |
| | | 60 | 16 | 12 | 90 |
| 0 | 2 | 1 | 137 | 175 | 135 |
| | | 2 | 107 | 138 | 270 |
| | | 3 | 149 | 172 | 225 |
| | | 4 | 12 | 9 | 0 |
| | | 5 | 157 | 206 | 135 |
| | | 6 | 54 | 45 | 270 |
| | | 7 | 221 | 253 | 225 |
| | | 8 | 152 | 195 | 0 |
| | | 9 | 203 | 257 | 135 |
| | | 10 | 175 | 171 | 270 |
| | | 11 | 186 | 213 | 225 |
| | | 12 | 72 | 72 | 0 |
| | | 13 | 180 | 207 | 135 |
| | | 14 | 63 | 56 | 270 |
| | | 15 | 84 | 73 | 225 |
| | | 16 | 30 | 29 | 180 |
| | | 17 | 70 | 73 | 135 |
| | | 19 | 61 | 65 | 225 |
| | | 21 | 71 | 57 | 315 |
| | | 22 | 112 | 104 | 270 |
| | | 23 | 108 | 88 | 45 |
| | | 24 | 68 | 65 | 180 |
| | | 25 | 143 | 136 | 315 |
| | | 27 | 14 | 13 | 45 |
| | | 28 | 64 | 77 | 180 |
| | | 29 | 27 | 34 | 315 |
| | | 30 | 63 | 33 | 90 |
| | | 31 | 11 | 6 | 45 |
| | | 32 | 88 | 77 | 180 |
| | | 34 | 46 | 24 | 90 |
| | | 35 | 34 | 17 | 225 |
| | | 36 | 33 | 39 | 180 |
| | | 38 | 22 | 7 | 90 |
| | | 39 | 36 | 30 | 225 |
| | | 40 | 21 | 4 | 180 |
| | | 41 | 16 | 8 | 135 |
| | | 42 | 85 | 73 | 90 |
| | | 43 | 19 | 6 | 225 |
| | | 44 | 22 | 11 | 0 |
| | | 45 | 38 | 18 | 315 |
| | | 46 | 19 | 23 | 90 |
| | | 47 | 51 | 22 | 45 |
| | | 48 | 14 | 4 | 180 |
| | | 49 | 25 | 15 | 315 |
| | | 51 | 18 | 20 | 45 |
| | | 53 | 23 | 14 | 315 |
| | | 55 | 27 | 20 | 45 |
| | | 57 | 22 | 21 | 315 |
| | | 59 | 17 | 19 | 45 |
| 0 | 3 | 1 | 118 | 153 | 225 |
| | | 2 | 163 | 206 | 0 |
| 0 | 3 | 3 | 133 | 185 | 315 |
| | | 4 | 68 | 72 | 90 |
| | | 5 | 29 | 33 | 225 |
| | | 6 | 177 | 207 | 0 |
| | | 7 | 233 | 264 | 315 |
| | | 8 | 76 | 74 | 270 |
| | | 9 | 70 | 69 | 225 |
| | | 10 | 53 | 56 | 180 |
| | | 11 | 22 | 35 | 315 |
| | | 13 | 35 | 31 | 45 |
| | | 14 | 52 | 56 | 180 |
| | | 15 | 71 | 68 | 315 |
| | | 16 | 64 | 84 | 270 |
| | | 17 | 35 | 18 | 225 |
| | | 18 | 179 | 192 | 180 |
| | | 19 | 42 | 21 | 315 |
| | | 20 | 17 | 8 | 270 |
| | | 21 | 24 | 53 | 45 |
| | | 22 | 136 | 126 | 180 |
| | | 23 | 127 | 152 | 135 |
| | | 24 | 32 | 42 | 270 |
| | | 25 | 59 | 51 | 225 |
| | | 26 | 21 | 6 | 0 |
| | | 27 | 61 | 70 | 135 |
| | | 28 | 77 | 60 | 90 |
| | | 29 | 38 | 41 | 45 |
| | | 30 | 35 | 27 | 180 |
| | | 31 | 70 | 69 | 135 |
| | | 32 | 24 | 5 | 90 |
| | | 33 | 43 | 43 | 45 |
| | | 34 | 32 | 19 | 180 |
| | | 35 | 23 | 24 | 315 |
| | | 36 | 31 | 26 | 90 |
| | | 37 | 35 | 34 | 45 |
| | | 38 | 26 | 12 | 0 |
| | | 39 | 48 | 29 | 135 |
| | | 41 | 55 | 37 | 45 |
| | | 42 | 32 | 35 | 180 |
| | | 43 | 19 | 29 | 315 |
| | | 46 | 48 | 39 | 180 |
| | | 47 | 23 | 25 | 315 |
| | | 50 | 29 | 25 | 180 |
| | | 54 | 22 | 8 | 0 |
| | | 55 | 10 | 2 | 315 |
| | | 56 | 10 | 12 | 90 |
| | | 57 | 17 | 21 | 225 |
| | | 58 | 40 | 26 | 0 |
| 0 | 4 | 0 | 232 | 287 | 180 |
| | | 1 | 24 | 23 | 315 |
| | | 2 | 56 | 55 | 90 |
| | | 3 | 95 | 81 | 225 |
| | | 4 | 130 | 145 | 180 |
| | | 5 | 55 | 40 | 315 |
| | | 6 | 36 | 56 | 270 |
| | | 7 | 64 | 64 | 225 |
| | | 8 | 42 | 45 | 0 |
| | | 10 | 139 | 146 | 270 |
| | | 11 | 69 | 74 | 225 |
| | | 12 | 75 | 90 | 0 |
| | | 13 | 22 | 15 | 315 |
| 0 | 4 | 14 | 86 | 96 | 270 |
| | | 15 | 63 | 62 | 225 |
| | | 16 | 67 | 82 | 0 |
| | | 17 | 31 | 28 | 135 |
| | | 18 | 36 | 55 | 270 |
| | | 19 | 27 | 45 | 225 |
| | | 20 | 96 | 117 | 0 |
| | | 21 | 123 | 104 | 135 |
| | | 22 | 34 | 43 | 270 |
| | | 23 | 28 | 53 | 225 |
| | | 24 | 48 | 59 | 0 |
| | | 25 | 114 | 91 | 135 |
| | | 26 | 39 | 41 | 270 |
| | | 27 | 62 | 37 | 45 |
| | | 28 | 53 | 39 | 180 |
| | | 29 | 47 | 33 | 135 |
| | | 30 | 15 | 27 | 270 |
| | | 31 | 27 | 20 | 45 |
| | | 32 | 59 | 54 | 180 |
| | | 34 | 44 | 40 | 90 |
| | | 35 | 41 | 42 | 45 |
| | | 38 | 13 | 7 | 90 |
| | | 39 | 17 | 22 | 45 |
| | | 40 | 17 | 4 | 0 |
| | | 41 | 30 | 24 | 315 |
| | | 42 | 37 | 25 | 90 |
| | | 44 | 71 | 54 | 0 |
| | | 45 | 35 | 34 | 315 |
| | | 46 | 48 | 40 | 90 |
| | | 47 | 23 | 18 | 45 |
| | | 48 | 20 | 17 | 0 |
| | | 49 | 25 | 25 | 315 |
| | | 60 | 17 | 10 | 180 |
| 0 | 5 | 1 | 84 | 94 | 225 |
| | | 2 | 130 | 143 | 0 |
| | | 3 | 27 | 45 | 135 |
| | | 4 | 55 | 61 | 90 |
| | | 5 | 93 | 110 | 225 |
| | | 6 | 85 | 116 | 0 |
| | | 7 | 42 | 45 | 135 |
| | | 8 | 36 | 69 | 90 |
| | | 9 | 90 | 105 | 225 |
| | | 10 | 19 | 6 | 180 |
| | | 11 | 115 | 124 | 135 |
| | | 12 | 23 | 37 | 90 |
| | | 13 | 110 | 97 | 225 |
| | | 14 | 15 | 19 | 180 |
| | | 15 | 55 | 57 | 135 |
| | | 16 | 35 | 37 | 90 |
| | | 17 | 37 | 52 | 225 |
| | | 18 | 73 | 63 | 180 |
| | | 19 | 32 | 24 | 135 |
| | | 20 | 33 | 43 | 90 |
| | | 21 | 29 | 35 | 45 |
| | | 22 | 95 | 80 | 180 |
| | | 23 | 16 | 19 | 315 |
| | | 25 | 54 | 60 | 45 |
| | | 26 | 51 | 43 | 180 |
| | | 27 | 23 | 20 | 315 |
| | | 28 | 36 | 30 | 270 |
| 0 | 5 | 29 | 18 | 27 | 45 |
| | | 30 | 37 | 40 | 180 |
| | | 32 | 33 | 6 | 270 |
| | | 33 | 21 | 20 | 225 |
| | | 34 | 17 | 12 | 0 |
| | | 36 | 28 | 10 | 90 |
| | | 37 | 17 | 10 | 45 |
| | | 39 | 48 | 24 | 135 |
| | | 40 | 20 | 19 | 270 |
| | | 41 | 29 | 20 | 45 |
| | | 48 | 27 | 12 | 270 |
| | | 49 | 20 | 10 | 45 |
| | | 51 | 24 | 20 | 315 |
| | | 52 | 14 | 5 | 270 |
| | | 53 | 21 | 16 | 45 |
| | | 55 | 29 | 25 | 315 |
| | | 56 | 25 | 14 | 270 |
| | | 58 | 15 | 10 | 0 |
| | | 59 | 16 | 11 | 315 |
| 0 | 6 | 0 | 29 | 39 | 0 |
| | | 1 | 88 | 102 | 315 |
| | | 3 | 49 | 56 | 225 |
| | | 4 | 42 | 35 | 180 |
| | | 5 | 45 | 60 | 315 |
| | | 6 | 32 | 38 | 270 |
| | | 9 | 70 | 87 | 315 |
| | | 10 | 16 | 19 | 270 |
| | | 11 | 18 | 38 | 45 |
| | | 12 | 53 | 47 | 180 |
| | | 13 | 24 | 26 | 315 |
| | | 15 | 19 | 11 | 225 |
| | | 17 | 63 | 67 | 135 |
| | | 18 | 19 | 35 | 270 |
| | | 19 | 58 | 50 | 225 |
| | | 21 | 72 | 75 | 135 |
| | | 22 | 18 | 7 | 90 |
| | | 23 | 29 | 20 | 225 |
| | | 24 | 13 | 3 | 180 |
| | | 25 | 63 | 75 | 135 |
| | | 26 | 13 | 5 | 270 |
| | | 27 | 34 | 29 | 45 |
| | | 28 | 29 | 28 | 0 |
| | | 29 | 36 | 44 | 135 |
| | | 31 | 10 | 11 | 45 |
| | | 32 | 17 | 20 | 0 |
| | | 35 | 53 | 45 | 45 |
| | | 36 | 29 | 29 | 0 |
| | | 38 | 20 | 14 | 270 |
| | | 39 | 65 | 53 | 45 |
| | | 41 | 29 | 20 | 315 |
| | | 43 | 27 | 24 | 45 |
| | | 44 | 20 | 2 | 180 |
| | | 46 | 20 | 3 | 90 |
| 0 | 7 | 2 | 61 | 90 | 180 |
| | | 3 | 32 | 60 | 135 |
| | | 4 | 27 | 40 | 270 |
| | | 5 | 9 | 10 | 225 |
| | | 6 | 60 | 62 | 180 |
| | | 7 | 35 | 43 | 135 |
| | | 9 | 59 | 53 | 225 |
| 0 | 7 | 11 | 32 | 31 | 135 |
| | | 13 | 46 | 38 | 225 |
| | | 14 | 42 | 43 | 0 |
| | | 15 | 50 | 54 | 135 |
| | | 17 | 63 | 70 | 225 |
| | | 18 | 46 | 65 | 0 |
| | | 19 | 43 | 26 | 135 |
| | | 20 | 17 | 9 | 90 |
| | | 23 | 37 | 17 | 135 |
| | | 24 | 14 | 10 | 270 |
| | | 25 | 22 | 6 | 45 |
| | | 26 | 17 | 3 | 0 |
| | | 27 | 17 | 3 | 315 |
| | | 28 | 25 | 13 | 90 |
| | | 29 | 20 | 14 | 45 |
| | | 31 | 18 | 11 | 135 |
| | | 33 | 41 | 36 | 45 |
| | | 35 | 14 | 8 | 315 |
| | | 39 | 17 | 8 | 315 |
| | | 41 | 10 | 17 | 45 |
| | | 42 | 28 | 11 | 0 |
| | | 43 | 20 | 11 | 315 |
| | | 46 | 34 | 25 | 0 |
| | | 47 | 16 | 8 | 315 |
| | | 49 | 20 | 16 | 45 |
| | | 51 | 19 | 19 | 315 |
| | | 52 | 16 | 4 | 270 |
| 0 | 8 | 1 | 43 | 40 | 315 |
| | | 3 | 32 | 39 | 225 |
| | | 6 | 59 | 55 | 90 |
| | | 7 | 35 | 58 | 225 |
| | | 11 | 40 | 32 | 225 |
| | | 16 | 51 | 51 | 180 |
| 1 | 1 | 0 | 247 | 224 | 0 |
| | | 2 | 52 | 52 | 180 |
| | | 3 | 93 | 83 | 270 |
| | | 4 | 199 | 200 | 180 |
| | | 5 | 199 | 174 | 270 |
| | | 6 | 168 | 160 | 0 |
| | | 7 | 116 | 101 | 270 |
| | | 9 | 74 | 69 | 270 |
| | | 10 | 184 | 181 | 0 |
| | | 11 | 72 | 74 | 270 |
| | | 12 | 99 | 122 | 180 |
| | | 13 | 62 | 47 | 90 |
| | | 14 | 257 | 244 | 180 |
| | | 15 | 210 | 210 | 90 |
| | | 16 | 97 | 100 | 180 |
| | | 17 | 104 | 76 | 270 |
| | | 18 | 69 | 69 | 180 |
| | | 19 | 114 | 100 | 90 |
| | | 20 | 58 | 50 | 0 |
| | | 21 | 195 | 175 | 270 |
| | | 22 | 36 | 23 | 0 |
| | | 23 | 37 | 54 | 90 |
| | | 24 | 57 | 69 | 0 |
| | | 25 | 77 | 56 | 270 |
| | | 26 | 86 | 80 | 0 |
| | | 27 | 80 | 86 | 90 |
| | | 28 | 188 | 167 | 0 |
| 1 | 1 | 29 | 45 | 36 | 270 |
| | | 30 | 29 | 19 | 0 |
| | | 31 | 63 | 61 | 90 |
| | | 32 | 99 | 99 | 0 |
| | | 33 | 39 | 22 | 270 |
| | | 34 | 62 | 53 | 0 |
| | | 35 | 20 | 23 | 270 |
| | | 36 | 60 | 68 | 0 |
| | | 37 | 99 | 86 | 90 |
| | | 38 | 45 | 36 | 0 |
| | | 39 | 93 | 100 | 270 |
| | | 41 | 64 | 63 | 90 |
| | | 42 | 24 | 23 | 0 |
| | | 43 | 24 | 39 | 270 |
| | | 45 | 29 | 40 | 90 |
| | | 46 | 48 | 51 | 180 |
| | | 48 | 35 | 33 | 180 |
| | | 49 | 25 | 23 | 270 |
| 1 | 2 | 0 | 53 | 27 | 0 |
| | | 1 | 105 | 125 | 86 |
| | | 2 | 97 | 87 | 279 |
| | | 3 | 164 | 186 | 126 |
| | | 4 | 197 | 228 | 166 |
| | | 5 | 149 | 144 | 43 |
| | | 6 | 125 | 128 | 92 |
| | | 7 | 81 | 81 | 62 |
| | | 8 | 87 | 74 | 114 |
| | | 9 | 66 | 71 | 344 |
| | | 10 | 92 | 89 | 199 |
| | | 11 | 104 | 92 | 234 |
| | | 12 | 149 | 138 | 14 |
| | | 13 | 50 | 60 | 60 |
| | | 14 | 44 | 41 | 74 |
| | | 15 | 26 | 26 | 165 |
| | | 16 | 131 | 141 | 33 |
| | | 17 | 86 | 99 | 214 |
| | | 18 | 55 | 49 | 82 |
| | | 19 | 72 | 77 | 193 |
| | | 20 | 79 | 88 | 21 |
| | | 21 | 77 | 101 | 173 |
| | | 22 | 43 | 39 | 55 |
| | | 23 | 80 | 80 | 82 |
| | | 24 | 38 | 43 | 73 |
| | | 25 | 136 | 157 | 196 |
| | | 26 | 70 | 58 | 17 |
| | | 27 | 45 | 34 | 8 |
| | | 28 | 22 | 42 | 149 |
| | | 29 | 93 | 103 | 187 |
| | | 30 | 42 | 37 | 48 |
| | | 31 | 23 | 24 | 38 |
| | | 32 | 78 | 75 | 177 |
| | | 33 | 21 | 24 | 225 |
| | | 34 | 52 | 48 | 346 |
| | | 35 | 73 | 78 | 333 |
| | | 36 | 33 | 38 | 210 |
| | | 38 | 22 | 29 | 343 |
| | | 39 | 83 | 87 | 332 |
| | | 40 | 32 | 32 | 303 |
| | | 41 | 23 | 32 | 27 |
| | | 43 | 50 | 51 | 331 |
| 1 | 2 | 44 | 29 | 30 | 316 |
| | | 45 | 24 | 34 | 46 |
| | | 46 | 42 | 39 | 273 |
| | | 47 | 39 | 30 | 289 |
| | | 48 | 26 | 19 | 344 |
| | | 49 | 18 | 29 | 40 |
| | | 50 | 30 | 8 | 336 |
| | | 51 | 26 | 33 | 77 |
| | | 52 | 35 | 34 | 346 |
| | | 53 | 30 | 11 | 291 |
| 1 | 3 | 0 | 53 | 62 | 180 |
| | | 1 | 98 | 116 | 222 |
| | | 2 | 107 | 121 | 328 |
| | | 3 | 20 | 18 | 104 |
| | | 4 | 163 | 173 | 161 |
| | | 5 | 103 | 114 | 211 |
| | | 6 | 65 | 68 | 115 |
| | | 7 | 69 | 81 | 137 |
| | | 8 | 26 | 38 | 96 |
| | | 9 | 120 | 140 | 189 |
| | | 10 | 104 | 103 | 133 |
| | | 11 | 40 | 34 | 107 |
| | | 12 | 36 | 37 | 55 |
| | | 13 | 41 | 42 | 305 |
| | | 14 | 153 | 156 | 110 |
| | | 15 | 127 | 104 | 35 |
| | | 16 | 79 | 95 | 20 |
| | | 17 | 93 | 94 | 311 |
| | | 18 | 87 | 87 | 146 |
| | | 19 | 92 | 94 | 46 |
| | | 20 | 77 | 69 | 14 |
| | | 21 | 87 | 99 | 346 |
| | | 22 | 38 | 39 | 55 |
| | | 23 | 79 | 71 | 25 |
| | | 24 | 30 | 20 | 165 |
| | | 25 | 53 | 68 | 351 |
| | | 26 | 92 | 97 | 29 |
| | | 27 | 124 | 113 | 12 |
| | | 28 | 67 | 67 | 186 |
| | | 29 | 36 | 41 | 84 |
| | | 30 | 93 | 99 | 354 |
| | | 31 | 21 | 22 | 115 |
| | | 32 | 21 | 16 | 207 |
| | | 33 | 29 | 35 | 84 |
| | | 34 | 26 | 21 | 320 |
| | | 35 | 30 | 28 | 193 |
| | | 36 | 23 | 20 | 159 |
| | | 37 | 45 | 57 | 173 |
| | | 38 | 28 | 30 | 306 |
| | | 39 | 49 | 46 | 175 |
| | | 41 | 37 | 52 | 158 |
| | | 43 | 34 | 35 | 200 |
| | | 44 | 34 | 26 | 356 |
| | | 46 | 35 | 29 | 207 |
| | | 51 | 30 | 28 | 258 |
| 1 | 4 | 0 | 67 | 75 | 0 |
| | | 1 | 32 | 24 | 27 |
| | | 2 | 86 | 83 | 178 |
| | | 3 | 67 | 50 | 90 |
| | | 4 | 55 | 69 | 276 |
| 1 | 4 | 5 | 24 | 36 | 274 |
| | | 6 | 81 | 71 | 345 |
| | | 7 | 29 | 39 | 333 |
| | | 8 | 66 | 69 | 220 |
| | | 9 | 52 | 63 | 324 |
| | | 10 | 91 | 91 | 170 |
| | | 11 | 84 | 91 | 109 |
| | | 13 | 74 | 68 | 81 |
| | | 14 | 107 | 113 | 165 |
| | | 15 | 26 | 17 | 346 |
| | | 16 | 36 | 36 | 213 |
| | | 17 | 37 | 29 | 118 |
| | | 18 | 59 | 67 | 138 |
| | | 19 | 77 | 78 | 254 |
| | | 20 | 44 | 35 | 295 |
| | | 21 | 87 | 88 | 148 |
| | | 22 | 41 | 31 | 68 |
| | | 23 | 84 | 85 | 289 |
| | | 24 | 53 | 57 | 258 |
| | | 26 | 58 | 75 | 16 |
| | | 27 | 82 | 74 | 278 |
| | | 28 | 32 | 41 | 43 |
| | | 29 | 44 | 63 | 167 |
| | | 30 | 47 | 58 | 30 |
| | | 32 | 48 | 48 | 10 |
| | | 33 | 47 | 39 | 261 |
| | | 34 | 87 | 91 | 343 |
| | | 35 | 32 | 44 | 325 |
| | | 36 | 28 | 25 | 324 |
| | | 37 | 40 | 51 | 273 |
| | | 38 | 29 | 41 | 339 |
| | | 39 | 45 | 47 | 78 |
| | | 41 | 24 | 26 | 290 |
| | | 43 | 46 | 47 | 56 |
| | | 45 | 30 | 31 | 287 |
| | | 46 | 25 | 26 | 204 |
| 1 | 5 | 1 | 50 | 53 | 150 |
| | | 2 | 122 | 138 | 157 |
| | | 3 | 48 | 58 | 59 |
| | | 4 | 32 | 23 | 130 |
| | | 5 | 45 | 33 | 243 |
| | | 6 | 35 | 40 | 164 |
| | | 7 | 90 | 112 | 42 |
| | | 8 | 63 | 63 | 0 |
| | | 9 | 33 | 36 | 281 |
| | | 10 | 66 | 53 | 134 |
| | | 11 | 72 | 73 | 56 |
| | | 12 | 91 | 104 | 66 |
| | | 14 | 103 | 114 | 15 |
| | | 15 | 30 | 37 | 281 |
| | | 16 | 35 | 40 | 48 |
| | | 17 | 43 | 35 | 316 |
| | | 18 | 75 | 88 | 39 |
| | | 19 | 31 | 43 | 266 |
| | | 20 | 31 | 26 | 83 |
| | | 22 | 46 | 55 | 48 |
| | | 23 | 55 | 63 | 274 |
| | | 25 | 60 | 42 | 64 |
| | | 27 | 22 | 32 | 217 |
| | | 28 | 60 | 62 | 142 |
| 1 | 5 | 29 | 31 | 16 | 42 |
| | | 30 | 27 | 24 | 180 |
| | | 31 | 48 | 56 | 202 |
| | | 32 | 46 | 47 | 189 |
| | | 33 | 33 | 29 | 321 |
| | | 36 | 29 | 19 | 224 |
| | | 37 | 38 | 26 | 310 |
| | | 38 | 25 | 37 | 301 |
| | | 39 | 25 | 32 | 80 |
| | | 41 | 30 | 21 | 309 |
| 1 | 6 | 1 | 20 | 9 | 240 |
| | | 2 | 28 | 33 | 205 |
| | | 3 | 46 | 38 | 221 |
| | | 4 | 31 | 46 | 62 |
| | | 6 | 28 | 36 | 216 |
| | | 7 | 51 | 51 | 233 |
| | | 10 | 29 | 34 | 95 |
| | | 11 | 48 | 30 | 342 |
| | | 12 | 41 | 43 | 199 |
| | | 13 | 68 | 71 | 72 |
| | | 14 | 51 | 51 | 42 |
| | | 15 | 55 | 51 | 311 |
| | | 16 | 61 | 61 | 216 |
| | | 18 | 26 | 15 | 322 |
| | | 19 | 46 | 51 | 334 |
| | | 21 | 64 | 71 | 45 |
| | | 22 | 27 | 19 | 346 |
| | | 23 | 35 | 39 | 306 |
| | | 24 | 36 | 31 | 314 |
| | | 25 | 46 | 51 | 16 |
| | | 27 | 37 | 47 | 320 |
| | | 28 | 37 | 41 | 325 |
| | | 29 | 29 | 36 | 354 |
| | | 32 | 42 | 47 | 359 |
| | | 36 | 35 | 42 | 45 |
| | | 37 | 30 | 28 | 239 |
| | | 39 | 30 | 37 | 149 |
| 1 | 7 | 0 | 41 | 58 | 0 |
| | | 1 | 23 | 40 | 93 |
| | | 2 | 44 | 45 | 162 |
| | | 3 | 27 | 28 | 30 |
| | | 4 | 47 | 39 | 42 |
| | | 5 | 61 | 70 | 92 |
| | | 7 | 52 | 58 | 2 |
| | | 9 | 20 | 18 | 10 |
| | | 10 | 23 | 24 | 277 |
| | | 11 | 23 | 28 | 4 |
| | | 12 | 24 | 22 | 141 |
| | | 13 | 36 | 11 | 3 |
| | | 14 | 24 | 36 | 291 |
| | | 15 | 28 | 12 | 28 |
| | | 16 | 40 | 34 | 159 |
| | | 17 | 24 | 50 | 129 |
| | | 18 | 33 | 12 | 292 |
| | | 19 | 44 | 38 | 248 |
| | | 20 | 25 | 45 | 145 |
| | | 22 | 34 | 19 | 11 |
| | | 23 | 29 | 33 | 185 |
| | | 24 | 24 | 30 | 110 |
| | | 26 | 34 | 6 | 234 |
| 1 | 7 | 27 | 25 | 38 | 174 |
| | | 30 | 35 | 19 | 231 |
| 1 | 8 | 0 | 34 | 33 | 180 |
| | | 3 | 29 | 32 | 218 |
| | | 5 | 48 | 47 | 81 |
| 2 | 2 | 0 | 14 | 29 | 180 |
| | | 1 | 24 | 18 | 90 |
| | | 2 | 463 | 521 | 0 |
| | | 3 | 53 | 33 | 90 |
| | | 4 | 102 | 96 | 180 |
| | | 5 | 267 | 206 | 90 |
| | | 6 | 113 | 187 | 0 |
| | | 7 | 249 | 175 | 90 |
| | | 9 | 105 | 93 | 90 |
| | | 10 | 109 | 100 | 180 |
| | | 11 | 48 | 64 | 270 |
| | | 12 | 99 | 73 | 180 |
| | | 13 | 45 | 43 | 90 |
| | | 14 | 88 | 108 | 180 |
| | | 15 | 71 | 58 | 270 |
| | | 16 | 40 | 37 | 180 |
| | | 17 | 95 | 113 | 90 |
| | | 18 | 131 | 144 | 180 |
| | | 19 | 70 | 58 | 270 |
| | | 21 | 16 | 40 | 90 |
| | | 22 | 67 | 66 | 180 |
| | | 23 | 212 | 176 | 270 |
| | | 24 | 29 | 24 | 180 |
| | | 25 | 21 | 19 | 90 |
| | | 26 | 30 | 48 | 180 |
| | | 27 | 65 | 60 | 270 |
| | | 29 | 92 | 85 | 270 |
| | | 30 | 88 | 84 | 0 |
| | | 31 | 51 | 45 | 270 |
| | | 33 | 45 | 40 | 270 |
| | | 34 | 14 | 23 | 0 |
| | | 35 | 26 | 19 | 90 |
| | | 37 | 26 | 32 | 270 |
| | | 40 | 25 | 23 | 180 |
| | | 41 | 33 | 50 | 270 |
| | | 42 | 64 | 54 | 180 |
| | | 43 | 26 | 25 | 90 |
| | | 45 | 17 | 1 | 270 |
| | | 46 | 38 | 26 | 180 |
| | | 47 | 48 | 46 | 90 |
| 2 | 3 | 0 | 117 | 107 | 180 |
| | | 1 | 80 | 82 | 179 |
| | | 2 | 100 | 122 | 268 |
| | | 3 | 130 | 135 | 1 |
| | | 4 | 107 | 109 | 204 |
| | | 5 | 95 | 76 | 258 |
| | | 6 | 151 | 144 | 311 |
| | | 7 | 178 | 181 | 22 |
| | | 8 | 63 | 47 | 55 |
| | | 9 | 114 | 115 | 316 |
| | | 10 | 57 | 72 | 272 |
| | | 11 | 91 | 94 | 44 |
| | | 12 | 59 | 57 | 355 |
| | | 13 | 68 | 55 | 358 |
| | | 14 | 47 | 63 | 270 |
| 2 | 3 | 15 | 118 | 102 | 21 |
| | | 16 | 77 | 73 | 27 |
| | | 17 | 67 | 56 | 353 |
| | | 18 | 84 | 104 | 281 |
| | | 19 | 20 | 28 | 291 |
| | | 20 | 44 | 49 | 11 |
| | | 21 | 54 | 55 | 65 |
| | | 22 | 81 | 66 | 275 |
| | | 23 | 56 | 54 | 208 |
| | | 24 | 24 | 32 | 117 |
| | | 25 | 32 | 31 | 58 |
| | | 27 | 53 | 67 | 232 |
| | | 28 | 17 | 26 | 144 |
| | | 29 | 34 | 38 | 90 |
| | | 30 | 30 | 35 | 157 |
| | | 31 | 48 | 31 | 124 |
| | | 32 | 56 | 58 | 185 |
| | | 33 | 15 | 20 | 54 |
| | | 34 | 18 | 10 | 171 |
| | | 35 | 39 | 43 | 114 |
| | | 36 | 46 | 53 | 206 |
| | | 37 | 28 | 28 | 300 |
| | | 38 | 25 | 35 | 154 |
| | | 39 | 23 | 29 | 112 |
| | | 41 | 20 | 24 | 287 |
| | | 42 | 34 | 40 | 81 |
| 2 | 4 | 1 | 18 | 24 | 56 |
| | | 3 | 76 | 104 | 111 |
| | | 4 | 81 | 81 | 184 |
| | | 6 | 75 | 66 | 351 |
| | | 7 | 90 | 103 | 121 |
| | | 8 | 74 | 47 | 271 |
| | | 9 | 119 | 105 | 245 |
| | | 10 | 100 | 100 | 334 |
| | | 11 | 115 | 120 | 166 |
| | | 12 | 45 | 41 | 252 |
| | | 13 | 59 | 68 | 147 |
| | | 14 | 31 | 24 | 277 |
| | | 15 | 29 | 21 | 93 |
| | | 16 | 67 | 49 | 242 |
| | | 17 | 46 | 41 | 6 |
| | | 18 | 56 | 55 | 265 |
| | | 19 | 99 | 114 | 324 |
| | | 20 | 53 | 36 | 314 |
| | | 21 | 56 | 58 | 41 |
| | | 22 | 49 | 54 | 263 |
| | | 23 | 61 | 86 | 302 |
| | | 24 | 46 | 50 | 307 |
| | | 26 | 35 | 39 | 194 |
| | | 27 | 57 | 72 | 306 |
| | | 29 | 21 | 23 | 121 |
| | | 30 | 62 | 68 | 175 |
| | | 31 | 19 | 29 | 305 |
| | | 32 | 56 | 48 | 16 |
| | | 33 | 44 | 41 | 241 |
| | | 34 | 53 | 55 | 164 |
| | | 36 | 38 | 36 | 29 |
| | | 37 | 42 | 38 | 218 |
| | | 39 | 41 | 41 | 115 |
| | | 40 | 48 | 51 | 73 |
| 2 | 4 | 41 | 14 | 50 | 242 |
| | | 42 | 27 | 25 | 105 |
| | | 43 | 21 | 16 | 161 |
| 2 | 5 | 0 | 145 | 178 | 0 |
| | | 1 | 39 | 49 | 317 |
| | | 3 | 45 | 46 | 227 |
| | | 4 | 132 | 132 | 1 |
| | | 5 | 42 | 61 | 326 |
| | | 6 | 73 | 69 | 13 |
| | | 7 | 57 | 51 | 79 |
| | | 8 | 80 | 78 | 18 |
| | | 9 | 52 | 63 | 305 |
| | | 10 | 53 | 69 | 307 |
| | | 11 | 40 | 55 | 92 |
| | | 12 | 85 | 83 | 126 |
| | | 13 | 51 | 42 | 322 |
| | | 14 | 49 | 54 | 289 |
| | | 15 | 36 | 43 | 4 |
| | | 16 | 96 | 104 | 164 |
| | | 17 | 80 | 64 | 280 |
| | | 18 | 46 | 52 | 257 |
| | | 19 | 45 | 53 | 128 |
| | | 20 | 69 | 89 | 140 |
| | | 21 | 41 | 45 | 129 |
| | | 22 | 15 | 16 | 249 |
| | | 23 | 23 | 12 | 105 |
| | | 24 | 37 | 40 | 158 |
| | | 25 | 34 | 30 | 208 |
| | | 26 | 19 | 18 | 193 |
| | | 27 | 38 | 24 | 244 |

TABLE 3. (*Continued.*)

| $h$ | $k$ | $l$ | $\lvert F_o\rvert$ | $\lvert F_c\rvert$ | $\alpha^\circ$ |
|---|---|---|---|---|---|
| | | 28 | 19 | 26 | 158 |
| | | 29 | 34 | 23 | 215 |
| | | 30 | 41 | 42 | 173 |
| | | 31 | 28 | 25 | 199 |
| 2 | 6 | 2 | 49 | 56 | 172 |
| | | 3 | 20 | 29 | 133 |
| | | 4 | 49 | 56 | 250 |
| | | 5 | 28 | 22 | 322 |
| | | 6 | 32 | 28 | 296 |
| | | 7 | 32 | 31 | 344 |
| | | 8 | 56 | 57 | 275 |
| | | 10 | 37 | 37 | 353 |
| | | 11 | 37 | 38 | 256 |
| | | 14 | 86 | 101 | 346 |
| | | 16 | 68 | 81 | 271 |
| | | 17 | 43 | 38 | 253 |
| | | 18 | 40 | 42 | 348 |
| | | 19 | 35 | 33 | 73 |
| | | 20 | 31 | 29 | 277 |
| | | 21 | 27 | 47 | 246 |
| | | 23 | 53 | 48 | 85 |
| | | 25 | 58 | 9 | 159 |
| | | 26 | 23 | 42 | 189 |
| | | 28 | 17 | 17 | 323 |
| | | 29 | 48 | 26 | 96 |
| | | 30 | 27 | 41 | 170 |
| | | 31 | 24 | 21 | 98 |
| | | 33 | 32 | 4 | 40 |
| | | 34 | 22 | 30 | 189 |
| | | 35 | 33 | 26 | 343 |
| 2 | 7 | 0 | 56 | 57 | 0 |
| | | 3 | 38 | 41 | 227 |
| | | 4 | 63 | 69 | 13 |
| | | 5 | 19 | 22 | 120 |
| | | 6 | 27 | 22 | 218 |
| | | 7 | 59 | 68 | 184 |
| | | 8 | 32 | 27 | 2 |
| | | 9 | 36 | 39 | 130 |
| | | 10 | 30 | 34 | 174 |
| | | 11 | 30 | 32 | 165 |
| | | 13 | 32 | 45 | 130 |
| | | 14 | 28 | 16 | 358 |
| | | 15 | 26 | 23 | 187 |
| | | 16 | 28 | 31 | 213 |
| | | 17 | 44 | 47 | 219 |
| | | 18 | 26 | 13 | 258 |
| | | 19 | 26 | 7 | 159 |
| | | 21 | 29 | 42 | 328 |
| | | 22 | 21 | 14 | 264 |
| | | 24 | 42 | 44 | 204 |
| | | 25 | 42 | 53 | 257 |
| | | 27 | 27 | 41 | 4 |
| | | 28 | 39 | 38 | 177 |
| 3 | 3 | 0 | 69 | 52 | 0 |
| | | 1 | 55 | 57 | 270 |
| | | 2 | 33 | 22 | 0 |
| | | 3 | 79 | 68 | 270 |
| | | 4 | 132 | 127 | 0 |
| | | 5 | 130 | 128 | 270 |
| | | 6 | 149 | 128 | 180 |
| | | 8 | 86 | 82 | 0 |
| | | 9 | 129 | 106 | 270 |
| | | 10 | 47 | 68 | 180 |
| | | 11 | 41 | 33 | 270 |
| | | 13 | 47 | 57 | 270 |
| | | 15 | 149 | 125 | 90 |
| | | 16 | 144 | 139 | 180 |
| | | 17 | 94 | 74 | 90 |
| | | 18 | 38 | 40 | 180 |
| | | 19 | 13 | 11 | 90 |
| | | 20 | 92 | 90 | 180 |
| | | 21 | 74 | 64 | 90 |
| | | 22 | 22 | 17 | 0 |
| | | 23 | 38 | 12 | 270 |
| | | 25 | 63 | 81 | 90 |
| | | 26 | 62 | 67 | 0 |
| | | 29 | 46 | 44 | 90 |
| | | 30 | 84 | 87 | 0 |
| | | 32 | 21 | 15 | 0 |
| | | 33 | 26 | 24 | 90 |
| | | 34 | 21 | 26 | 0 |
| | | 35 | 37 | 35 | 90 |
| | | 37 | 17 | 17 | 90 |
| | | 38 | 40 | 36 | 0 |
| | | 39 | 37 | 26 | 90 |
| | | 40 | 19 | 21 | 0 |
| | | 41 | 19 | 35 | 270 |
| | | 48 | 32 | 34 | 180 |
| 3 | 4 | 0 | 51 | 50 | 180 |
| | | 1 | 31 | 34 | 99 |
| 3 | 4 | 2 | 150 | 138 | 171 |
| | | 3 | 43 | 48 | 8 |
| | | 5 | 68 | 63 | 82 |
| | | 6 | 68 | 70 | 139 |
| | | 7 | 46 | 46 | 125 |
| | | 8 | 47 | 59 | 236 |
| | | 9 | 19 | 26 | 75 |
| | | 10 | 32 | 20 | 128 |
| | | 11 | 60 | 5 | 234 |
| | | 12 | 60 | 67 | 254 |
| | | 13 | 57 | 59 | 210 |
| | | 14 | 112 | 102 | 44 |
| | | 15 | 32 | 45 | 248 |
| | | 16 | 43 | 60 | 238 |
| | | 18 | 93 | 94 | 38 |
| | | 19 | 55 | 52 | 182 |
| | | 20 | 28 | 27 | 315 |
| | | 21 | 65 | 53 | 124 |
| | | 22 | 59 | 59 | 39 |
| | | 23 | 90 | 107 | 181 |
| | | 24 | 23 | 20 | 302 |
| | | 25 | 44 | 53 | 161 |
| | | 26 | 16 | 18 | 89 |
| | | 27 | 35 | 41 | 187 |
| | | 28 | 37 | 36 | 1 |
| | | 29 | 35 | 36 | 108 |
| | | 30 | 37 | 43 | 179 |
| | | 31 | 18 | 13 | 160 |
| | | 32 | 19 | 18 | 20 |
| | | 34 | 17 | 2 | 185 |
| | | 35 | 25 | 27 | 82 |
| | | 36 | 23 | 28 | 17 |
| | | 37 | 48 | 51 | 344 |
| | | 38 | 29 | 35 | 270 |
| | | 39 | 22 | 14 | 7 |
| | | 40 | 25 | 25 | 50 |
| | | 41 | 32 | 29 | 326 |
| | | 42 | 16 | 25 | 298 |
| | | 43 | 16 | 30 | 37 |
| | | 44 | 24 | 15 | 150 |
| | | 45 | 32 | 31 | 329 |
| 3 | 5 | 0 | 159 | 161 | 0 |
| | | 1 | 65 | 63 | 261 |
| | | 2 | 101 | 98 | 182 |
| | | 3 | 60 | 53 | 239 |
| | | 4 | 49 | 48 | 12 |
| | | 5 | 54 | 58 | 154 |
| | | 6 | 21 | 36 | 137 |
| | | 7 | 31 | 34 | 208 |
| | | 8 | 34 | 51 | 50 |
| | | 9 | 60 | 62 | 138 |
| | | 10 | 56 | 63 | 107 |
| | | 11 | 67 | 80 | 234 |
| | | 12 | 55 | 59 | 113 |
| | | 13 | 30 | 33 | 155 |
| | | 14 | 37 | 51 | 72 |
| | | 15 | 31 | 38 | 271 |
| | | 16 | 68 | 75 | 113 |
| | | 18 | 55 | 52 | 358 |
| | | 20 | 56 | 56 | 109 |
| 3 | 5 | 21 | 51 | 50 | 45 |
| | | 22 | 29 | 32 | 39 |
| | | 23 | 17 | 19 | 83 |
| | | 24 | 14 | 27 | 200 |
| | | 25 | 70 | 61 | 26 |
| | | 27 | 23 | 25 | 41 |
| | | 28 | 35 | 17 | 57 |
| | | 29 | 36 | 38 | 353 |
| | | 35 | 25 | 24 | 137 |
| | | 36 | 39 | 31 | 255 |
| | | 38 | 23 | 6 | 125 |
| 3 | 6 | 0 | 16 | 1 | 0 |
| | | 1 | 22 | 21 | 246 |
| | | 2 | 33 | 36 | 27 |
| | | 3 | 64 | 61 | 298 |
| | | 4 | 64 | 55 | 220 |
| | | 5 | 15 | 10 | 34 |
| | | 6 | 19 | 20 | 182 |
| | | 7 | 53 | 60 | 306 |
| | | 8 | 57 | 61 | 201 |
| | | 9 | 30 | 27 | 87 |
| | | 10 | 38 | 25 | 149 |
| | | 12 | 22 | 34 | 208 |
| | | 13 | 71 | 66 | 119 |
| | | 17 | 41 | 31 | 305 |
| | | 19 | 25 | 30 | 120 |
| | | 20 | 27 | 18 | 145 |
| | | 23 | 40 | 43 | 163 |
| | | 24 | 24 | 18 | 4 |
| | | 25 | 26 | 26 | 261 |
| | | 27 | 38 | 47 | 159 |
| | | 28 | 44 | 53 | 21 |
| | | 29 | 40 | 30 | 306 |
| | | 30 | 25 | 28 | 28 |
| | | 31 | 24 | 25 | 153 |
| | | 32 | 42 | 45 | 354 |
| | | 34 | 23 | 17 | 7 |
| | | 35 | 20 | 28 | 238 |
| | | 36 | 23 | 22 | 338 |
| | | 37 | 25 | 21 | 64 |
| | | 39 | 23 | 15 | 353 |
| | | 40 | 21 | 13 | 54 |
| | | 41 | 36 | 26 | 68 |
| 3 | 7 | 4 | 46 | 46 | 155 |
| | | 5 | 38 | 4 | 106 |
| | | 6 | 18 | 15 | 244 |
| | | 7 | 32 | 33 | 238 |
| | | 8 | 28 | 29 | 122 |
| | | 9 | 28 | 29 | 82 |
| | | 11 | 40 | 42 | 242 |
| | | 12 | 37 | 42 | 43 |
| | | 13 | 24 | 30 | 99 |
| | | 14 | 39 | 46 | 29 |
| | | 15 | 31 | 32 | 269 |
| | | 16 | 54 | 59 | 38 |
| | | 20 | 55 | 56 | 40 |
| | | 21 | 19 | 29 | 228 |
| | | 22 | 19 | 21 | 337 |
| 3 | 8 | 0 | 32 | 43 | 180 |
| | | 2 | 22 | 25 | 9 |
| 3 | 8 | 4 | 35 | 35 | 187 |
| | | 5 | 25 | 32 | 209 |
| | | 6 | 22 | 29 | 43 |
| | | 8 | 21 | 24 | 128 |
| | | 9 | 36 | 36 | 249 |
| | | 11 | 16 | 19 | 86 |
| | | 12 | 44 | 51 | 77 |
| | | 13 | 32 | 29 | 10 |
| | | 15 | 25 | 25 | 79 |
| | | 16 | 23 | 17 | 312 |
| | | 18 | 25 | 31 | 171 |
| | | 19 | 23 | 19 | 60 |
| | | 20 | 14 | 10 | 39 |
| | | 21 | 21 | 28 | 354 |
| | | 23 | 16 | 19 | 25 |
| | | 25 | 20 | 23 | 295 |
| | | 27 | 20 | 20 | 19 |
| | | 28 | 23 | 21 | 83 |
| 3 | 9 | 0 | 45 | 47 | 180 |
| 4 | 4 | 0 | 97 | 130 | 0 |
| | | 2 | 37 | 31 | 180 |
| | | 3 | 30 | 31 | 270 |
| | | 4 | 54 | 53 | 0 |
| | | 5 | 90 | 66 | 90 |
| | | 6 | 13 | 18 | 0 |
| | | 7 | 45 | 41 | 90 |
| | | 9 | 78 | 54 | 90 |
| | | 10 | 39 | 54 | 180 |
| | | 11 | 69 | 55 | 90 |
| | | 12 | 98 | 101 | 180 |
| | | 13 | 18 | 17 | 270 |
| | | 14 | 30 | 28 | 180 |
| | | 15 | 27 | 32 | 90 |
| | | 16 | 94 | 95 | 180 |
| | | 17 | 34 | 32 | 270 |
| | | 20 | 18 | 5 | 180 |
| | | 21 | 78 | 73 | 270 |
| | | 23 | 43 | 5 | 90 |
| | | 24 | 41 | 28 | 0 |
| | | 25 | 75 | 74 | 270 |
| | | 26 | 17 | 28 | 180 |
| | | 27 | 17 | 9 | 270 |
| | | 28 | 67 | 69 | 0 |
| | | 29 | 38 | 38 | 270 |
| | | 30 | 20 | 14 | 180 |
| | | 31 | 64 | 74 | 270 |
| | | 32 | 71 | 85 | 0 |
| | | 33 | 21 | 22 | 270 |
| | | 34 | 18 | 20 | 180 |
| | | 35 | 22 | 36 | 270 |
| | | 36 | 15 | 19 | 0 |
| | | 40 | 21 | 6 | 180 |
| | | 42 | 19 | 27 | 180 |
| | | 44 | 21 | 15 | 180 |
| | | 45 | 21 | 23 | 90 |
| | | 47 | 17 | 18 | 270 |
| 4 | 5 | 0 | 26 | 11 | 0 |
| | | 1 | 91 | 89 | 20 |
| | | 2 | 27 | 19 | 139 |
| | | 3 | 29 | 18 | 197 |
| 4 | 5 | 4 | 38 | 27 | 345 |
| | | 5 | 75 | 78 | 36 |
| | | 6 | 24 | 31 | 201 |
| | | 7 | 40 | 43 | 272 |
| | | 8 | 37 | 27 | 8 |
| | | 9 | 62 | 86 | 6 |
| | | 10 | 11 | 19 | 304 |
| | | 11 | 44 | 58 | 335 |
| | | 12 | 48 | 40 | 45 |
| | | 13 | 58 | 58 | 38 |
| | | 14 | 54 | 46 | 2 |
| | | 15 | 13 | 11 | 277 |
| | | 16 | 19 | 21 | 103 |
| | | 17 | 35 | 38 | 319 |
| | | 18 | 14 | 16 | 23 |
| | | 19 | 61 | 51 | 50 |
| | | 20 | 18 | 19 | 30 |
| | | 21 | 20 | 32 | 236 |
| | | 22 | 28 | 28 | 106 |
| | | 23 | 30 | 23 | 58 |
| | | 25 | 23 | 44 | 211 |
| | | 27 | 29 | 30 | 55 |
| | | 28 | 22 | 39 | 117 |
| | | 29 | 24 | 32 | 216 |
| | | 30 | 20 | 6 | 235 |
| | | 31 | 20 | 8 | 24 |
| | | 32 | 22 | 7 | 122 |
| | | 33 | 36 | 44 | 145 |
| | | 34 | 22 | 30 | 176 |
| | | 35 | 19 | 13 | 301 |
| | | 36 | 35 | 31 | 226 |
| | | 39 | 18 | 14 | 305 |
| | | 41 | 18 | 19 | 107 |
| | | 43 | 23 | 28 | 264 |
| 4 | 6 | 0 | 25 | 22 | 180 |
| | | 1 | 32 | 34 | 66 |
| | | 4 | 29 | 35 | 201 |
| | | 5 | 26 | 27 | 148 |
| | | 6 | 22 | 7 | 225 |
| | | 7 | 26 | 34 | 146 |
| | | 8 | 15 | 14 | 209 |
| | | 9 | 18 | 26 | 165 |
| | | 10 | 18 | 20 | 113 |
| | | 12 | 36 | 25 | 338 |
| | | 13 | 14 | 22 | 247 |
| | | 15 | 40 | 42 | 75 |
| | | 16 | 45 | 41 | 329 |
| | | 17 | 51 | 53 | 341 |
| | | 18 | 21 | 12 | 241 |
| | | 19 | 46 | 43 | 37 |
| | | 20 | 26 | 35 | 279 |
| | | 21 | 46 | 51 | 298 |
| | | 22 | 15 | 12 | 221 |
| | | 23 | 24 | 31 | 11 |
| | | 24 | 16 | 14 | 233 |
| | | 25 | 45 | 51 | 313 |
| | | 27 | 17 | 19 | 75 |
| | | 28 | 22 | 24 | 282 |
| | | 29 | 19 | 23 | 295 |
| | | 30 | 17 | 18 | 309 |
| 4 | 6 | 32 | 24 | 23 | 198 |
| | | 33 | 22 | 21 | 202 |
| | | 34 | 16 | 16 | 352 |
| | | 35 | 25 | 22 | 200 |
| | | 37 | 22 | 23 | 117 |
| | | 38 | 19 | 18 | 54 |
| | | 39 | 35 | 35 | 254 |
| 4 | 7 | 1 | 25 | 28 | 61 |
| | | 2 | 39 | 54 | 344 |
| | | 3 | 26 | 23 | 88 |
| | | 5 | 24 | 28 | 23 |
| | | 6 | 18 | 17 | 39 |
| | | 8 | 32 | 44 | 342 |
| | | 9 | 26 | 22 | 69 |
| | | 10 | 24 | 22 | 113 |
| | | 12 | 19 | 11 | 347 |
| | | 13 | 26 | 37 | 98 |
| | | 14 | 9 | 5 | 57 |
| | | 16 | 29 | 34 | 263 |
| | | 17 | 23 | 17 | 151 |
| | | 18 | 24 | 37 | 165 |
| | | 19 | 19 | 10 | 308 |
| | | 21 | 19 | 19 | 119 |
| | | 22 | 19 | 25 | 155 |
| | | 23 | 26 | 27 | 225 |
| | | 25 | 25 | 31 | 217 |
| | | 27 | 24 | 15 | 291 |
| | | 28 | 24 | 30 | 160 |
| | | 30 | 22 | 20 | 0 |
| | | 32 | 22 | 20 | 205 |
| 5 | 5 | 0 | 31 | 19 | 0 |
| | | 1 | 31 | 36 | 270 |
| | | 2 | 24 | 28 | 0 |
| | | 3 | 70 | 86 | 270 |
| | | 4 | 23 | 26 | 0 |
| | | 5 | 43 | 33 | 90 |
| | | 6 | 46 | 53 | 0 |
| | | 7 | 43 | 48 | 270 |
| | | 8 | 22 | 4 | 180 |
| | | 9 | 26 | 25 | 90 |
| | | 10 | 16 | 9 | 0 |
| | | 11 | 75 | 74 | 270 |
| | | 13 | 25 | 41 | 90 |
| | | 14 | 42 | 40 | 180 |
| | | 16 | 29 | 36 | 180 |
| | | 17 | 47 | 55 | 90 |
| | | 18 | 39 | 49 | 180 |
| | | 20 | 27 | 17 | 180 |
| | | 22 | 44 | 50 | 180 |
| | | 23 | 18 | 29 | 90 |
| | | 24 | 26 | 20 | 180 |
| | | 25 | 29 | 17 | 270 |
| | | 26 | 25 | 19 | 180 |
| | | 34 | 29 | 1 | 0 |
| | | 36 | 27 | 6 | 180 |
| | | 38 | 41 | 14 | 0 |
| 5 | 6 | 0 | 35 | 38 | 180 |
| | | 1 | 26 | 29 | 226 |
| | | 2 | 22 | 24 | 273 |
| | | 3 | 28 | 40 | 10 |
| 5 | 6 | 4 | 35 | 47 | 202 |
| | | 5 | 18 | 11 | 238 |
| | | 6 | 23 | 32 | 297 |
| | | 7 | 18 | 22 | 33 |
| | | 8 | 24 | 23 | 191 |
| | | 9 | 38 | 31 | 226 |
| | | 10 | 28 | 29 | 245 |
| | | 12 | 29 | 32 | 45 |
| | | 13 | 29 | 31 | 222 |
| | | 14 | 47 | 56 | 247 |
| | | 15 | 41 | 35 | 234 |
| | | 17 | 28 | 34 | 250 |
| | | 18 | 23 | 26 | 288 |
| | | 19 | 38 | 30 | 204 |
| | | 20 | 43 | 45 | 347 |
| | | 21 | 23 | 13 | 137 |
| | | 23 | 48 | 49 | 138 |
| | | 25 | 27 | 28 | 161 |
| | | 28 | 31 | 22 | 93 |
| 5 | 7 | 0 | 19 | 32 | 180 |
| | | 2 | 50 | 49 | 357 |
| | | 4 | 34 | 38 | 144 |
| | | 5 | 19 | 30 | 237 |
| | | 6 | 43 | 42 | 342 |
| | | 7 | 19 | 27 | 153 |
| | | 9 | 43 | 40 | 190 |
| | | 10 | 25 | 24 | 97 |
| | | 11 | 30 | 40 | 199 |
| 5 | 8 | 1 | 27 | 27 | 284 |

The standard deviations of the final atomic co-ordinates were derived from the least-squares residuals by application of the equation:

$$\sigma^2(x_i) = \sum_j w_j(\Delta F_j)^2/[(n - s) \sum_j w_j(\partial F_j/\partial x_i)^2].$$

The individual results have been listed by Paul.[25]

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