*Acta Cryst.* (1969). **B25**, 2175

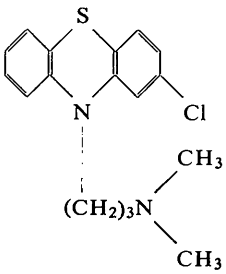
# The Crystal and Molecular Structure of Chlorpromazine

BY J. J. H. McDOWELL

*Department of Physics, University of Cape Town, Republic of South Africa*

(*Received* 11 *December* 1968)

The crystal structure of chlorpromazine,



has been determined from three-dimensional X-ray diffraction data and refined by least-squares methods, with individual anisotropic temperature factors. The space group is *Pbca* with $a = 23{\cdot}50 \pm 0{\cdot}04$, $b = 15{\cdot}20 \pm 0{\cdot}02$ and $c = 9{\cdot}23 \pm 0{\cdot}01$ Å. The molecule is folded about the S–N axis and the angle between the best planes for the two benzene rings was found to be 139·4°. The C–S–C angle is 97·3 ± 0·3 °, the C–S bonds are 1·75 ± 0·01 Å and C–Cl is 1·74 ± 0·01 Å. The contraction of the C–S bond, the size of the C–S–C angle and the folding of the molecule are characteristic of a number of related compounds, and can be explained by assuming the participation of *d* orbitals in the bonding of S.

## Introduction

Chlorpromazine is 3-chloro-10-(3′-dimethylamino-n-propyl)-phenothiazine which, since its first introduction into clinical psychiatry by Delay & Deniker (1952) has been one of the best known derivatives of phenothiazine (Colloque International Chlorpromazine, Paris, 1955). It is the valuable drug largactil used in general medicine (for relief of nausea and vomiting, and radiation sickness), psychiatry (to control schizophrenic or manic states) and in surgery and anaesthesia (to modify or prevent traumatic and haemorrhagic shock) (Buxton Hopkin, 1955; Courvoisier, Fournel, Ducrot, Kolsky & Koetschet, 1953; Takayanagi, 1964).

The determination of its crystal structure was undertaken as part of a general study of the group of phenothiazine and some of its important derivatives. It is hoped that knowledge of the molecular structures will help in the understanding of the biochemical action of these compounds in man.

## Solution and refinement of the structure

Needle-shaped crystals were formed from a solution of powdered chlorpromazine dissolved in low boiling point petroleum spirit. Because of the deleterious effects of light, air and X-rays and the consequent difficulties, the following procedure was finally adopted, with successful results: (1) the crystals were grown in darkness under an atmosphere of nitrogen; (2) single crystals were sealed in nitrogen-filled Lindemann-glass capillaries; and (3) exposure to light was avoided as far as possible throughout the photographic process. X-ray rotation and equi-inclination Weissenberg photographs, with the use of Ni-filtered Cu $K\alpha$ radiation, taken about the *y* and *z* axes, gave an orthorhombic system with $a = 23{\cdot}50 \pm 0{\cdot}04$, $b = 15{\cdot}20 \pm 0{\cdot}02$, $c = 9{\cdot}23 \pm 0{\cdot}01$ Å. The conditions for non-extinction are $hk0$, $h = 2n$; $0kl$, $k = 2n$; $h0l$, $l = 2n$; $h00$, $h = 2n$; $0k0$, $k = 2n$; $00l$, $l = 2n$; which lead uniquely to the space group *Pbca*. $D_m = 1{\cdot}289$ g.cm$^{-3}$, $D_c = 1{\cdot}285$ g.cm$^{-3}$ for 8 molecules per unit cell (Feil, Linck & McDowell, 1965; Falkenberg & Ringertz, 1967).

Eight layer-lines ($hk0$ to $hk7$) were photographed by use of the standard multiple-film technique. On each film $I_{(hkl)}$, $I_{(h\bar{k}l)}$, plus either $I_{(hkl)}$ or $I_{(hkl)}$ were measured; for *Pbca* these are all equal. Thus by collating their symmetry relationships, about 6000 measured intensities were reduced, by averaging, to 1895 independent reflexions. Scale factors for the individual layer-lines were obtained by the following two methods: (*a*) the crystal was rotated about the *y* axis and a five-film Weissenberg gave 120 independent $h1l$ intensities with which to set the *z* axis photographs on to the same scale, (*b*) Wilson's (1942) method of obtaining absolute *K*'s for each layer-line was used. The second method was found to give results more closely consistent with the refined scale factors obtained by the least-squares. All the usual correction factors were applied, but absorption was neglected since the value of $\mu R < 0{\cdot}5$.

A three-dimensional Patterson function, sharpened in accordance with the formula

$$|F|^2=\frac{(\sin^2\theta/\lambda^2+0\cdot16)F_o^2\exp(B\sin^2\theta/\lambda^2)}{(\Sigma f_i)^2},$$

was calculated. The S–S and the Cl–Cl inversion peaks and the eight corresponding S–Cl peaks were correctly located and a three-dimensional Fourier synthesis phased on S and Cl gave initial values of the coordinates of the C and N atoms. Structure factors were

Table 1. *Observed and calculated structure factors*

Within each group the columns, reading from left to right, contain the values of *h*, $K|F_o|$ and $F_c$.

| $h$ | $K\|F_o\|$ | $F_c$ |
|---|---|---|
| **h00** | | |
| 4 | 70 | -55 |
| 6 | 47 | -44 |
| 8 | 102 | -106 |
| 10 | 45 | 31 |
| 12 | 174 | -177 |
| 14 | 18 | 16 |
| 16 | 11 | -13 |
| 18* | 37 | 35 |
| 20 | 4 | -5 |
| 22 | 25 | 22 |
| 24* | 23 | 20 |
| 26* | 3 | 4 |
| 28* | 3 | -3 |
| 30 | 12 | -14 |
| **h10** | | |
| 4 | 81 | 82 |
| 6 | 85 | 78 |
| 8 | 67 | 46 |
| 10 | 24 | 14 |
| 12 | 65 | -55 |
| 14* | 30 | 27 |
| 16* | 3 | 5 |
| 18 | 15 | -17 |
| 20 | 14 | -13 |
| 22 | 13 | 13 |
| 24 | 22 | -25 |
| **h20** | | |
| 0† | 67 | 108 |
| 2 | 29 | 27 |
| 4 | 37 | 41 |
| 6 | 54 | -44 |
| 8 | 28 | -24 |
| 10 | 56 | -53 |
| 12 | 102 | -103 |
| 14* | 25 | 22 |
| 16* | 3 | 4 |
| 18 | 46 | 46 |
| 20 | 21 | 18 |
| 22 | 18 | 22 |
| **h30** | | |
| 2 | 7 | 10 |
| 4 | 110 | 116 |
| 6 | 65 | -58 |
| 8 | 59 | 58 |
| 10* | 28 | -26 |
| 12 | 3 | 2 |
| 14 | 35 | -36 |
| 16* | 21 | -20 |
| 18* | 4 | 1 |
| 20 | 9 | -8 |
| 22 | 15 | 15 |
| **h40** | | |
| 0* | 68 | 70 |
| 2* | 2 | 7 |
| 4 | 100 | 103 |
| 6 | 11 | 9 |
| 8 | 47 | -42 |
| 10 | 41 | -45 |
| 12 | 67 | -65 |
| 14 | 18 | -17 |
| 16 | 7 | -7 |
| 18 | 31 | 33 |
| 20 | 10 | 10 |
| 22 | 13 | 11 |
| **h50** | | |
| 2 | 61 | -55 |
| 4 | 110 | 114 |
| 6 | 33 | 28 |
| 8 | 59 | 50 |
| 10 | 11 | -11 |
| 12 | 27 | 27 |
| 14 | 11 | -9 |
| 16 | 35 | -27 |
| 18 | 38 | -38 |
| 20 | 7 | -5 |
| 22* | 11 | 11 |
| 24* | 3 | -5 |
| 26 | 7 | 9 |
| **h60** | | |
| 2 | 67 | 67 |
| 4 | 26 | 12 |
| 6 | 95 | -93 |
| 8 | 11 | -12 |
| 10 | 28 | -22 |
| 12 | 31 | 30 |
| 14 | 10 | -9 |
| 16 | 13 | -16 |
| 18* | 20 | 19 |
| 20* | 4 | 7 |
| 22* | 11 | 12 |
| 24* | 3 | 6 |
| 26* | 3 | -1 |
| 28 | 8 | -10 |
| **h70** | | |
| 4 | 82 | 86 |
| 6 | 28 | -23 |
| 8 | 41 | 45 |
| 10 | 19 | -19 |
| 12 | 8 | 7 |
| 14 | 23 | 21 |
| 16 | 68 | -66 |
| 18 | 26 | 27 |
| 20 | 16 | -16 |
| 22* | 11 | -8 |
| 24* | 3 | -3 |
| 26 | 5 | 6 |
| 28 | 13 | 15 |
| **h80** | | |
| 0 | 35 | -35 |
| 2 | 85 | 62 |
| 4 | 77 | -71 |
| 6 | 40 | 35 |
| 8 | 25 | -20 |
| 10 | 16 | -17 |
| 12 | 7 | -7 |
| 14 | 49 | -48 |
| 16 | 61 | 62 |
| 18 | 8 | -8 |
| 20 | 15 | 19 |
| **h90** | | |
| 4* | 48 | 48 |
| 6* | 3 | -3 |
| 8* | 3 | -3 |
| 10 | 35 | 33 |
| 12 | 20 | 19 |
| 14 | 45 | -45 |
| 16 | 11 | -10 |
| 18 | 21 | -22 |
| 20 | 11 | 10 |
| 22* | 6 | -6 |
| 24* | 3 | 1 |
| 26 | 10 | 10 |
| **h,10,0** | | |
| 0 | 19 | -14 |
| 2 | 36 | 39 |
| 4 | 29 | -29 |
| 6* | 29 | 24 |
| 8* | 4 | 2 |
| 10 | 51 | -51 |
| 12 | 18 | 15 |
| 14 | 21 | -20 |
| 16* | 31 | 31 |
| 18* | 4 | 5 |
| 20 | 9 | 12 |
| 22 | 8 | -9 |
| 24 | 5 | -7 |
| **h,11,0** | | |
| 2* | 34 | 38 |
| 4* | 4 | -4 |
| 6* | 44 | 38 |
| 8* | 4 | 1 |
| 10 | 32 | 33 |
| 12* | 22 | -22 |
| 14* | 4 | -7 |
| 16* | 4 | -4 |
| 18* | 32 | -30 |
| 20* | 3 | 3 |
| 22 | 7 | 5 |
| **h,12,0** | | |
| 0 | 67 | -67 |
| 2 | 49 | 47 |
| 4 | 9 | -7 |
| 6 | 24 | -23 |
| 8* | 15 | -16 |
| 10* | 4 | -7 |
| 12 | 35 | 33 |
| 14* | 4 | 5 |
| 16 | 8 | 8 |
| 18 | 11 | -10 |
| 20 | 13 | 13 |
| **h,13,0** | | |
| 2 | 26 | 25 |
| 4 | 7 | -4 |
| 6 | 27 | 28 |
| 8 | 11 | -13 |
| 10 | 7 | 8 |
| 12 | 20 | -21 |
| **h,14,0** | | |
| 0 | 60 | -51 |
| 2 | 37 | 33 |
| 4* | 11 | -11 |
| 6* | 4 | 1 |
| 8* | 4 | 4 |
| 10 | 8 | -7 |
| 12 | 13 | 14 |
| 14 | 8 | -6 |
| **h,15,0** | | |
| 2 | 13 | 11 |
| 4 | 12 | -11 |
| 6 | 24 | 25 |
| 8 | 7 | -8 |
| 10* | 11 | 10 |
| 12* | 3 | -4 |
| 14* | 9 | -11 |
| 16* | 3 | 4 |
| 18 | 10 | -12 |
| **h,16,0** | | |
| 2 | 18 | -17 |
| 4* | 13 | 13 |
| 6* | 3 | -1 |
| 8* | 3 | 1 |
| 10* | 3 | -1 |
| 12* | 3 | 1 |
| 14 | 10 | 8 |
| **h,17,0** | | |
| 2 | 20 | 19 |
| 4 | 10 | -8 |
| 6 | 5 | -3 |
| 8 | 5 | -6 |
| 10 | 12 | -10 |
| **h,18,0** | | |
| 0 | 22 | 22 |
| 2* | 15 | -13 |
| 4* | 3 | 1 |
| 6* | 3 | 2 |
| 8 | 11 | -8 |
| 10 | 13 | 13 |
| **h,19,0** | | |
| 4 | 5 | 6 |
| **h11** | | |
| 5 | 63 | 64 |
| 6 | 48 | 48 |
| 7 | 45 | -43 |
| 8 | 63 | 68 |
| 9 | 19 | 14 |
| 10 | 34 | 33 |
| 11 | 79 | -81 |
| 12 | 40 | -41 |
| 13 | 13 | -15 |
| 14 | 43 | -39 |
| 15 | 14 | -14 |
| 16* | 18 | -22 |
| 17* | 3 | 1 |
| 18 | 13 | -13 |
| 19 | 27 | 32 |
| 20* | 16 | -17 |
| 21* | 3 | 1 |
| 22 | 20 | 26 |
| 23 | 15 | 17 |
| **h21** | | |
| 4* | 52 | -44 |
| 5† | 103 | -80 |
| 6 | 9 | 7 |
| 7 | 9 | 7 |
| 8 | 10 | 8 |
| 9 | 45 | 43 |
| 10 | 47 | -43 |
| 11 | 8 | -9 |
| 12 | 50 | 41 |
| 13* | 29 | 31 |
| 14* | 2 | -3 |
| 15 | 27 | -25 |
| 16* | 14 | 13 |
| 17* | 3 | 3 |
| 18 | 21 | -10 |
| 19 | 16 | -18 |
| 20* | 8 | -8 |
| 21* | 3 | 3 |
| 22 | 10 | -10 |
| **h31** | | |
| 1† | 159 | 124 |
| 2† | 216 | 264 |
| 3 | 49 | 37 |
| 4 | 81 | 73 |
| 5 | 23 | -13 |
| 6 | 21 | 14 |
| 7 | 100 | |
| 8 | 15 | 14 |
| 9 | 4 | 3 |
| 10 | 12 | -9 |
| 11 | 43 | -44 |
| 12 | 36 | 37 |
| 13 | 11 | -13 |
| 14 | 48 | -46 |
| 15 | 4 | -3 |
| 16* | 20 | -20 |
| 17* | 3 | 1 |
| 18 | 14 | -15 |
| 19* | 18 | 19 |
| 20* | 3 | 6 |
| 21 | 3 | 3 |
| 22 | 14 | -17 |
| 23 | 14 | 16 |
| 24* | 7 | 9 |
| 25* | 3 | 3 |
| 26 | 9 | 12 |
| **h41** | | |
| 0† | 99 | -81 |
| 1† | 160 | -106 |
| 2 | 48 | -47 |
| 3 | 87 | 88 |
| 4 | 12 | -3 |
| 5 | 17 | -7 |
| 6 | 27 | -18 |
| 7 | 28 | 30 |
| 8 | 35 | 35 |
| 9 | 21 | 25 |
| 10 | 10 | 11 |
| 11 | 32 | -31 |
| 12 | 25 | 29 |
| 13* | 34 | -34 |
| 14* | 2 | -1 |
| 15 | 30 | -32 |
| 16 | 26 | 28 |
| 17 | 20 | 23 |
| 18 | 9 | -7 |
| 19 | 28 | -32 |
| 20* | 5 | -7 |
| 21* | 3 | -2 |
| 22* | 6 | 10 |
| 23* | 3 | 1 |
| 24* | 11 | -14 |
| 25* | 2 | -4 |
| 26 | 5 | -8 |
| 27 | 7 | 9 |
| **h51** | | |
| 1 | 27 | 18 |
| 2 | 3 | -1 |
| 3 | 89 | 78 |
| 4 | 85 | 73 |
| 5 | 12 | -12 |
| 6 | 19 | -11 |
| 7 | 22 | -22 |
| 8 | 15 | -13 |
| 9 | 56 | -54 |
| 10 | 35 | 34 |
| 11 | 38 | -37 |
| 12* | 21 | -20 |
| 13* | 3 | |
| 14 | 27 | -28 |
| 15 | 7 | -6 |
| 16 | 55 | -64 |
| 17 | 12 | 14 |
| 18 | 30 | 34 |
| 19* | 20 | 24 |
| 20* | 3 | -1 |
| 21* | 9 | 13 |
| 22* | 3 | 5 |
| 23* | 3 | 1 |
| 24* | 10 | 13 |
| 25* | 2 | 1 |
| 26* | 2 | 4 |
| 27 | 4 | -5 |
| 28 | 6 | 8 |
| **h61** | | |
| 0 | 7 | -4 |
| 1* | 29 | -13 |
| 2* | 2 | -1 |
| 3 | 99 | 98 |
| 4 | 64 | -63 |
| 5 | 52 | -38 |
| 6 | 109 | 97 |
| 7 | 58 | 49 |
| 8 | 47 | -39 |
| 9* | 26 | 21 |
| 10* | 2 | 2 |
| 11 | 22 | -19 |
| 12 | 39 | 40 |
| 13* | 26 | -27 |
| 14* | 3 | 1 |
| 15 | 58 | -66 |
| 16 | 16 | 16 |
| 17 | 11 | 13 |
| 18 | 40 | -46 |
| 19 | 14 | -15 |
| 20* | 10 | 10 |
| 21* | 3 | -1 |
| 22* | 3 | -8 |
| 23* | 3 | 2 |
| 24* | 2 | -7 |
| 25* | 2 | 2 |
| 26* | 2 | -3 |
| 27 | 6 | 10 |
| **h71** | | |
| 1 | 31 | 29 |
| 2 | 31 | 27 |
| 3 | 16 | 17 |
| 4 | 17 | -11 |
| 5 | 21 | -21 |
| 6 | 56 | 59 |
| 7 | 26 | 19 |
| 8 | 32 | 31 |
| 9 | 42 | -40 |
| 10* | 12 | 8 |
| 11* | 2 | -3 |
| 12* | 3 | -3 |
| 13 | 27 | -28 |
| 14 | 33 | -33 |
| 15 | 7 | 7 |
| 16 | 5 | -7 |
| 17 | 25 | 28 |
| 18* | 11 | -13 |
| 19* | 3 | -4 |
| 20 | 14 | 15 |
| 21 | 7 | 11 |
| **h81** | | |
| 0 | 32 | -23 |
| 1 | 31 | -26 |
| 2 | 24 | -22 |
| 3 | 48 | 45 |
| 4 | 76 | -70 |
| 5 | 19 | 22 |
| 6 | 45 | 38 |
| 7 | 40 | 37 |
| 8 | 15 | 17 |
| 9* | 6 | 9 |
| 10* | 2 | 7 |
| 11* | 3 | -2 |
| 12* | 34 | 34 |
| 13* | 3 | 3 |
| 14* | 3 | -2 |
| 15 | 30 | -33 |
| 16* | 39 | 42 |
| 17* | 3 | -1 |
| 18 | 20 | -21 |
| 19 | 19 | -20 |
| 20 | 14 | -16 |
| 21* | 4 | 8 |
| 22* | 3 | -1 |
| 23* | 2 | 2 |
| 24 | 8 | -9 |
| **h91** | | |
| 2 | 19 | -17 |
| 3 | 27 | 23 |
| 4 | 29 | 27 |
| 5 | 54 | -48 |
| 6 | 31 | 25 |
| 7 | 24 | 24 |
| 8 | 39 | -33 |
| 9 | 37 | -37 |
| 10 | 19 | -22 |
| 11 | 24 | 21 |
| 12 | 21 | -23 |
| 13 | 6 | 7 |
| 14* | 11 | 10 |
| 15* | 3 | 4 |
| 16* | 22 | 24 |
| 17* | 3 | 4 |
| 18 | 22 | -27 |
| 19 | 4 | -7 |
| 20 | 9 | 9 |
| 21 | 11 | 15 |
| 22 | 6 | 9 |
| **h,10,1** | | |
| 0 | 15 | -15 |
| 1 | 15 | 18 |
| 2 | 27 | -27 |
| 3 | 29 | 28 |
| 4* | 23 | 23 |
| 5* | 3 | 5 |
| 6 | 11 | -7 |
| 7 | 12 | 12 |
| 8* | 17 | 17 |
| 9* | 3 | 0 |
| 10* | 18 | 16 |
| 11* | 3 | -1 |
| 12 | 11 | -9 |
| 13 | 7 | -8 |
| 14 | 18 | 18 |
| 15 | 13 | -15 |
| 16* | 10 | -12 |
| 17* | 3 | 1 |
| 18* | 8 | -7 |
| 19* | 3 | -2 |
| 20* | 3 | 3 |
| 21* | 2 | -3 |
| 22 | 5 | -10 |
| **h,11,1** | | |
| 2 | 9 | 9 |
| 3 | 6 | -9 |
| 4 | 10 | -9 |
| 5 | 12 | -12 |
| 6 | 14 | 16 |
| 7 | 33 | 31 |
| 8 | 11 | -10 |
| 9 | 26 | -23 |
| 10 | 20 | 11 |
| 11* | 16 | 16 |
| 12* | 3 | -7 |
| 13* | 14 | -16 |
| 14* | 3 | -1 |
| 15* | 3 | 6 |
| 16 | 6 | -8 |
| 17* | 9 | 12 |
| 18* | 3 | 4 |
| 19 | 10 | -8 |
| **h,12,1** | | |
| 0 | 43 | -51 |
| 1 | 18 | 19 |
| 2 | 15 | 14 |
| 3 | 14 | -15 |
| 4 | 6 | -7 |
| 5 | 18 | 18 |
| 6 | 5 | 8 |
| 7 | 6 | 8 |
| 8* | 6 | -8 |
| 9* | 3 | -1 |
| 10* | 3 | 1 |
| 11 | 13 | 11 |
| 12 | 29 | 32 |
| 13* | 16 | -18 |
| 14* | 3 | -1 |
| 15* | 11 | 12 |
| 16* | 3 | -4 |
| 17 | 14 | -17 |
| **h,13,1** | | |
| 1 | 12 | -13 |
| 2 | 19 | -20 |
| 3 | 10 | 13 |
| 4 | 10 | 11 |
| 5 | 5 | -6 |
| 6* | 16 | -17 |
| 7* | 3 | 3 |
| 8* | 15 | 18 |
| 9* | 3 | -6 |
| 10* | 9 | -9 |
| 11* | 3 | 2 |
| 12* | 3 | -5 |
| 13* | 3 | -1 |
| 14* | 3 | 1 |
| 15* | 3 | 7 |
| 16* | 2 | -6 |
| 17* | 2 | 2 |
| 18* | 2 | 2 |
| 19 | 2 | 3 |
| 20 | 12 | 15 |
| **h,14,1** | | |
| 1 | 22 | 26 |
| 2 | 20 | -24 |
| 3 | 15 | -17 |
| 4 | 27 | 34 |
| 5 | 11 | 13 |
| 6* | 11 | -11 |
| 7* | 3 | 2 |
| 8* | 11 | -11 |
| 9* | 3 | -5 |
| 10 | 13 | 15 |
| 11* | 10 | 13 |
| 12* | 3 | -2 |
| 13 | 11 | -13 |
| 14* | 10 | 12 |
| 15* | 2 | 2 |
| 16 | 5 | -7 |
| 17 | 8 | -12 |
| **h,15,1** | | |
| 1 | 4 | 8 |
| 2 | 6 | -6 |
| 3* | 11 | -13 |
| 4* | 3 | 6 |
| 5 | 9 | 10 |
| 6 | 7 | 7 |
| 7 | 10 | -12 |
| 8* | 10 | -12 |
| 9* | 3 | -1 |
| 10 | 6 | -6 |
| 11* | 7 | 6 |
| 12* | 2 | 1 |
| 13* | 2 | 1 |
| 14* | 2 | 1 |
| 15 | 6 | 6 |
| **h,16,1** | | |
| 0 | 16 | 20 |
| 1 | 12 | 17 |
| 2 | 15 | -19 |
| 3* | 8 | -10 |
| 4* | 3 | 3 |
| 5* | 10 | 15 |
| 6* | 3 | 6 |
| 7* | 2 | -2 |
| 8* | 2 | 1 |
| 9* | 2 | -3 |
| 10* | 7 | 7 |
| 11* | 2 | 5 |
| 12* | 2 | -5 |
| 13* | 6 | -12 |
| 14* | 2 | 4 |
| 15 | 4 | 8 |
| **h,17,1** | | |
| 2* | 8 | -11 |
| 3* | 2 | -6 |
| 4 | 8 | 12 |
| 5 | 5 | 8 |
| 6 | 8 | -11 |
| 7 | 6 | -10 |
| 8 | 6 | 8 |
| 9* | 6 | 9 |
| 10* | 2 | -3 |
| 11* | 2 | -4 |
| 12 | 4 | 7 |
| **h,19,1** | | |
| 6 | 5 | -1 |
| **h02** | | |
| 2* | 76 | 90 |
| 3† | 216 | 273 |
| 4 | 120 | 121 |
| 5 | 102 | 110 |
| 6 | 43 | -40 |
| 7 | 39 | 35 |
| 8 | 48 | -39 |
| 9 | 29 | 24 |
| 10 | 29 | -34 |
| 11 | 33 | -32 |
| 12* | 42 | 35 |
| 13* | 3 | -1 |
| 14 | 20 | 27 |
| 15 | 58 | -59 |
| 16* | 16 | -13 |
| 17* | 3 | -2 |
| 18 | 16 | 20 |
| 19* | 33 | -33 |
| 20* | 4 | -3 |
| 21* | 4 | 3 |
| 22* | 4 | 5 |
| 23* | 4 | 5 |
| 24* | 3 | -4 |
| 25 | 8 | 9 |
| 26 | 9 | -10 |
| 27 | 12 | 14 |
| **h12** | | |
| 1 | 37 | -40 |
| 2 | 60 | -61 |
| 3 | 38 | -47 |
| 4 | 64 | 61 |
| 5 | 18 | -11 |
| 6 | 45 | -45 |
| 7 | 41 | 49 |
| 8* | 19 | 7 |
| 9* | 2 | 7 |
| 10 | 55 | -54 |
| 11 | 24 | 22 |
| 12* | 27 | 20 |
| 13* | 3 | -3 |
| 14 | 15 | -13 |
| 15* | 16 | -17 |
| 16* | 3 | 2 |
| 17 | 19 | 21 |
| 18 | 7 | 3 |
| 19* | 6 | 4 |
| 20* | 4 | 5 |
| 21* | 4 | -3 |
| 22* | 4 | -3 |
| 23* | 4 | -2 |
| 24* | 13 | 14 |
| 25* | 3 | -3 |
| 26* | 3 | -2 |
| 27 | 6 | -4 |
| **h22** | | |
| 0 | 85 | 89 |
| 1 | 168 | -159 |
| 2 | 70 | -70 |
| 3 | 126 | 135 |
| 4 | 129 | 131 |
| 5 | 52 | 60 |
| 6 | 38 | -33 |
| 7 | 46 | 46 |
| 8 | 47 | -49 |
| 9 | 92 | 87 |
| 10 | 10 | 14 |
| 11 | 83 | -75 |
| 12 | 20 | -6 |
| 13 | 8 | -5 |
| 14 | 28 | 26 |
| 15 | 53 | -54 |
| 16 | 46 | -47 |
| 17 | 30 | -32 |
| 18 | 12 | 8 |
| 19* | 18 | -19 |
| 20* | 4 | -1 |
| 21* | 18 | 20 |
| 22* | 4 | -2 |
| 23* | 12 | 12 |
| 24* | 4 | 4 |
| 25* | 3 | 6 |
| 26* | 3 | -1 |
| 27 | 9 | 13 |
| **h32** | | |
| 1 | 128 | -126 |
| 2 | 43 | 27 |
| 3 | 127 | 113 |
| 4 | 64 | 53 |
| 5 | 25 | -28 |
| 6 | 18 | -13 |
| 7 | 10 | -3 |
| 8 | 61 | 61 |
| 9 | 19 | 20 |
| 10 | 30 | -32 |
| 11 | 37 | 31 |
| 12 | 16 | 13 |
| 13 | 30 | 29 |
| 14* | 8 | -7 |
| 15* | 3 | -2 |
| 16 | 21 | -17 |
| 17 | 12 | -16 |
| 18* | 20 | 21 |
| 19* | 4 | -3 |
| 20* | 15 | -16 |
| 21* | 4 | -3 |
| 22* | 4 | 2 |
| 23 | 14 | -15 |
| **h42** | | |
| 0 | 71 | 62 |
| 1 | 28 | 20 |
| 2 | 59 | -51 |
| 3 | 53 | 52 |
| 4 | 60 | -50 |
| 5* | 22 | 22 |
| 6* | 2 | 9 |
| 7 | 74 | 69 |
| 8 | 22 | 18 |
| 9 | 6 | 17 |
| 10 | 38 | 32 |
| 11 | 25 | 21 |
| 12 | 11 | 1 |
| 13* | 5 | -10 |
| 14* | 3 | 2 |
| 15* | 72 | -74 |
| 16* | 3 | 5 |
| 17 | 7 | -7 |
| 18* | 8 | -6 |
| 19* | 4 | 1 |
| 20* | 4 | -7 |
| 21* | 4 | 4 |
| 22* | 4 | 4 |
| 23* | 3 | 3 |
| 24* | 3 | -1 |
| 25 | 8 | 8 |
| **h52** | | |
| 1 | 55 | -52 |
| 2* | 36 | -28 |
| 3* | 2 | 4 |
| 4 | 43 | 39 |
| 5 | 70 | -57 |
| 6 | 84 | -83 |
| 7 | 47 | 45 |
| 8 | 66 | 64 |
| 9 | 14 | -13 |
| 10 | 36 | -29 |
| 11* | 41 | 45 |
| 12* | 3 | -2 |
| 13 | 37 | 33 |
| 14 | 6 | -6 |
| 15* | 10 | 5 |
| 16* | 3 | -2 |
| 17 | 23 | 20 |
| 18 | 26 | 27 |
| 19 | 27 | -29 |
| 20 | 17 | -18 |
| 21 | 6 | -7 |
| 22 | 11 | 12 |
| 23 | 6 | -8 |
| 24 | 6 | -8 |
| 25 | 6 | -3 |
| **h62** | | |
| 0 | 66 | -61 |
| 1 | 27 | 22 |
| 2 | 20 | 16 |
| 3 | 62 | 55 |
| 4 | 37 | -26 |
| 5 | 67 | 65 |
| 6 | 67 | 56 |
| 7* | 27 | -15 |
| 8* | 3 | 1 |
| 9 | 24 | -22 |
| 10* | 17 | -10 |
| 11* | 3 | -2 |
| 12 | 3 | -1 |
| 13 | 23 | -21 |
| 14* | 10 | -10 |
| 15* | 3 | 1 |
| 16 | 18 | 17 |
| 17 | 16 | -14 |
| 18* | 20 | -20 |
| 19* | 4 | -4 |
| 20* | 4 | 9 |
| 21* | 4 | 1 |
| 22* | 3 | -3 |
| 23* | 3 | 3 |
| 24* | 3 | -2 |
| 25 | 10 | 13 |
| **h72** | | |
| 1* | 42 | -39 |
| 2* | 2 | 2 |
| 3 | 19 | -15 |
| 4 | 13 | -13 |
| 5* | 3 | -12 |
| 6 | 23 | -25 |
| 7 | 42 | 41 |
| 8* | 24 | 28 |
| 9* | 3 | 7 |
| 10 | 23 | -20 |
| 11 | 18 | 14 |
| 12 | 25 | 24 |
| 13 | 20 | 20 |
| 14* | 30 | -32 |
| 15* | 4 | 2 |
| 16* | 41 | 43 |
| 17* | 4 | -1 |
| 18* | 19 | -19 |
| 19* | 4 | 1 |
| 20* | 10 | -10 |
| 21 | 3 | -5 |
| 22 | 7 | 9 |
| 23 | 11 | -14 |
| **h82** | | |
| 0 | 30 | -30 |
| 1 | 56 | 61 |
| 2 | 11 | 11 |
| 3 | 25 | -19 |
| 4 | 24 | 20 |
| 5 | 12 | 14 |
| 6 | 11 | -8 |
| 7 | 26 | 26 |
| 8 | 12 | 9 |
| 9 | 4 | 6 |
| 10 | 32 | -32 |
| 11 | 4 | 5 |
| 12 | 29 | 28 |
| 13* | 34 | -38 |
| 14* | 4 | -1 |
| 15* | 4 | 2 |
| 16 | 14 | -11 |
| 17* | 16 | -19 |
| 18* | 4 | -2 |
| 19* | 4 | 4 |
| 20 | 5 | 1 |
| 21 | 8 | 7 |
| **h92** | | |
| 1* | 31 | -34 |
| 2* | 3 | -8 |
| 3 | 10 | 6 |
| 4 | 15 | -9 |
| 5* | 17 | -17 |
| 6* | 3 | 4 |
| 7* | 3 | 7 |
| 8 | 26 | 23 |
| 9 | 14 | 16 |
| 10 | 18 | -15 |
| 11 | 15 | 17 |
| 12 | 13 | -11 |
| 13 | 22 | 22 |
| 14 | 23 | 26 |
| 15* | 11 | 11 |
| 16* | 3 | 4 |
| 17* | 3 | 3 |
| 18* | 4 | 2 |
| 19 | 10 | -13 |
| 20 | 12 | -14 |
| **h,10,2** | | |
| 0 | 55 | -53 |
| 1 | 34 | 37 |
| 2 | 50 | 51 |
| 3 | 32 | -28 |
| 4 | 27 | -27 |
| 4 | 23 | 25 |
| 6* | 6 | -8 |
| 7* | 3 | 3 |
| 11 | 11 | 12 |
| 12 | 13 | 16 |
| 13 | 37 | -42 |
| 14 | 11 | -13 |
| 15 | 11 | 10 |
| 16 | 12 | 14 |
| 17 | 8 | -12 |
| 18 | 7 | -9 |
| 19 | 11 | 12 |
| **h,11,2** | | |
| 1 | 17 | -18 |
| 2 | 15 | -17 |
| 3* | 15 | -13 |
| 4* | 3 | -1 |
| 5 | 4 | 5 |
| 6 | 4 | 10 |
| 7* | 9 | -8 |
| 8* | 4 | -3 |
| 9 | 22 | 21 |
| 10 | 6 | -9 |
| 11* | 6 | 7 |
| 12* | 4 | 3 |
| 13 | 19 | 23 |
| 14 | 5 | -4 |
| 15* | 5 | 6 |
| 16* | 4 | -3 |
| 17* | 10 | -11 |
| 18* | 3 | 5 |
| 19* | 3 | 1 |
| 20* | 3 | 3 |
| 21 | 14 | -17 |
| **h,12,2** | | |
| 0 | 12 | 13 |
| 1* | 44 | 49 |
| 2* | 4 | 7 |
| 3 | 18 | -17 |
| 4 | 23 | -24 |
| 5 | 12 | 14 |
| 6* | 19 | 19 |
| 7* | 4 | 6 |
| 8* | 4 | 5 |
| 9* | 23 | -27 |
| 10* | 4 | -7 |
| 11* | 4 | -3 |
| 12* | 4 | -1 |
| 13 | 11 | -14 |
| 14 | 12 | -14 |
| 15 | 17 | 17 |
| 16 | 13 | 16 |
| **h,13,2** | | |
| 1 | 15 | -19 |
| 2 | 13 | 12 |
| 3 | 9 | -9 |
| 4 | 10 | -9 |
| 5 | 20 | 25 |
| 6 | 17 | 19 |
| 7 | 13 | -13 |
| 8 | 18 | -18 |
| 9 | 17 | 22 |
| 10* | 16 | 17 |
| 11* | 4 | 3 |
| 12* | 4 | -2 |
| 13* | 3 | 5 |
| 14* | 3 | -4 |
| 15* | 3 | 2 |
| 16* | 3 | 2 |
| 17* | 10 | -12 |
| 18* | 3 | -7 |
| 19* | 3 | 2 |
| 20 | 7 | 12 |
| **h,14,2** | | |
| 0 | 27 | 29 |
| 1 | 17 | 20 |
| 2 | 22 | -23 |
| 3* | 11 | -12 |
| 4* | 4 | -3 |
| 5* | 4 | -2 |
| 6* | 4 | 4 |
| 7* | 4 | 1 |
| 8* | 4 | 2 |
| 9* | 4 | -7 |
| 10* | 10 | 9 |
| 11* | 3 | 6 |
| 12* | 10 | -9 |
| 13* | 3 | -2 |
| 14* | 3 | 5 |
| 15* | 3 | -5 |
| 16* | 4 | 4 |
| 17* | 3 | 1 |
| 18* | 2 | -1 |
| 19 | 7 | 12 |
| **h,15,2** | | |
| 1* | 4 | 4 |
| 2* | 4 | 1 |
| 3* | 18 | -19 |
| 4* | 3 | -10 |
| 5* | 3 | 7 |
| 6* | 14 | 15 |
| 7* | 3 | -2 |
| 8 | 14 | -18 |
| 9 | 8 | 11 |
| 10 | 5 | 9 |
| 11 | 5 | 2 |
| 12* | 7 | -12 |
| 13* | 3 | 2 |
| 14 | 8 | 12 |
| **h,17,2** | | |
| 1* | 3 | 3 |
| 2* | 3 | -5 |
| 3* | 11 | -9 |
| 4* | 3 | -7 |
| 5* | 3 | 9 |
| 6 | 9 | 14 |
| **h,18,2** | | |
| 5* | 6 | -6 |
| 6* | 2 | -7 |
| 7* | 2 | 1 |
| 8 | 5 | 6 |
| **h13** | | |
| 3 | 55 | -61 |
| 4 | 75 | 92 |
| 5 | 62 | 55 |
| 6 | 14 | -14 |
| 7 | 55 | -55 |
| 8 | 76 | 80 |
| 9 | 86 | 87 |
| 10 | 41 | -39 |
| 11 | 34 | 32 |
| 12 | 30 | 32 |
| 13 | 44 | 43 |
| 14 | 22 | -21 |
| 15 | 7 | 3 |
| 16 | 38 | -35 |
| 17* | 32 | -28 |
| 18* | 3 | -3 |
| 19 | 18 | -18 |
| 20 | 16 | -13 |
| 21* | 20 | -17 |
| 22* | 3 | 6 |
| 23* | 3 | 4 |
| 24* | 3 | 2 |
| 25 | 9 | -6 |
| 26* | 10 | 10 |
| 27* | 2 | 2 |
| 28 | 6 | 3 |
| **h23** | | |
| 0 | 33 | 37 |
| 1 | 40 | -39 |
| 2 | 22 | -17 |
| 3* | 33 | -25 |
| 4* | 1 | -1 |
| 5 | 16 | 16 |
| 6 | 13 | -15 |
| 7 | 13 | -11 |
| 8 | 43 | -35 |
| 9 | 36 | -34 |
| 10* | 11 | 9 |
| 11* | 2 | 11 |
| 12 | 32 | 33 |
| 13* | 18 | 17 |
| 14* | 3 | -1 |
| 15* | 3 | -7 |
| 16 | 16 | 14 |
| 17 | 24 | 23 |
| 18 | 13 | -12 |
| 19 | 13 | 10 |
| 20 | 9 | 8 |
| 21* | 9 | -8 |
| 22* | 3 | -2 |
| 23 | 10 | -8 |
| 24* | 9 | -9 |
| 25* | 3 | -2 |
| 26* | 2 | 4 |
| 27 | 2 | 3 |
| 28 | 6 | -5 |
| **h33** | | |
| 1 | 67 | -76 |
| 2 | 32 | -32 |
| 3 | 76 | -83 |
| 4 | 56 | 63 |
| 5 | 72 | 70 |
| 6 | 49 | 47 |
| 7 | 67 | 59 |
| 8 | 46 | 45 |
| 9 | 14 | 12 |
| 10* | 50 | -43 |
| 11* | 2 | -5 |
| 12 | 27 | -24 |
| 13* | 31 | 29 |
| 14* | 3 | -3 |
| 15* | 3 | -1 |
| 16* | 27 | -24 |
| 17* | 3 | 6 |
| 18 | 9 | 9 |
| 19 | 13 | -12 |
| 20 | 26 | -26 |
| 21 | 14 | -11 |
| 22* | 24 | 23 |
| 23* | 3 | -5 |
| 24* | 3 | -1 |
| 25 | 11 | -11 |
| 26* | 6 | 7 |
| 27* | 2 | 7 |
| 28 | 4 | 6 |
| **h43** | | |
| 0 | 71 | -76 |
| 1 | 17 | -18 |
| 2 | 77 | 77 |
| 3 | 34 | -36 |
| 4 | 86 | -93 |
| 5 | 48 | -45 |
| 6 | 66 | 61 |
| 7 | 10 | -14 |
| 8 | 5 | -2 |
| 9 | 9 | -10 |
| 10* | 10 | -5 |
| 11* | 2 | -4 |
| 12 | 33 | 31 |
| 13 | 23 | 21 |
| 14* | 49 | -49 |
| 15* | 3 | 1 |
| 16* | 48 | 42 |
| 17* | 3 | -5 |
| 18 | 16 | -14 |
| 19 | 15 | 12 |
| 20 | 5 | 8 |
| 21 | 8 | 9 |
| 22* | 16 | -16 |
| 23* | 3 | -3 |
| 24* | 6 | -7 |
| 25* | 2 | 3 |
| 26 | 5 | 6 |
| **h53** | | |
| 1* | 26 | 21 |
| 2* | 2 | -1 |
| 3 | 56 | -57 |
| 4 | 16 | 12 |
| 5 | 14 | 13 |
| 6 | 97 | 99 |
| 7 | 8 | -4 |
| 8 | 35 | 36 |
| 9 | 34 | 29 |
| 10 | 30 | -26 |
| 11 | 29 | 25 |
| 12* | 13 | -12 |
| 13* | 3 | 2 |
| 14 | 27 | -26 |
| 15 | 14 | 13 |
| 16 | 25 | 23 |
| 17 | 12 | -10 |
| 18 | 53 | -48 |
| 19* | 29 | -27 |
| 20* | 3 | 4 |
| 21 | 4 | -6 |
| 22 | 15 | 13 |
| **h63** | | |
| 0 | 123 | -132 |
| 1 | 61 | -67 |
| 2 | 33 | 28 |
| 3 | 15 | -18 |
| 4 | 21 | -21 |
| 5 | 39 | 31 |
| 6 | 31 | -26 |
| 7 | 37 | -33 |
| 8* | 44 | 43 |
| 9* | 2 | 1 |
| 10* | 2 | -1 |
| 11 | 8 | -9 |
| 12 | 52 | 51 |
| 13 | 4 | -7 |
| 14 | 15 | -13 |
| 15 | 37 | 35 |
| 16 | 21 | 19 |
| 17 | 9 | 9 |
| 18* | 4 | -3 |
| 19* | 3 | -6 |
| 20 | 8 | -4 |
| 21 | 6 | 9 |
| 22* | 10 | -9 |
| 23* | 2 | -1 |
| 24* | 13 | -12 |
| 25* | 2 | -3 |
| 26* | 2 | 4 |
| 27 | 6 | -6 |
| **h73** | | |
| 1 | 32 | -25 |
| 2 | 56 | 53 |
| 3 | 11 | 11 |
| 4 | 9 | -12 |
| 5 | 47 | -41 |
| 6 | 15 | 15 |
| 7 | 25 | 25 |
| 8* | 27 | -27 |
| 9* | 3 | -4 |
| 10 | 26 | 21 |
| 11 | 34 | 30 |
| 12 | 22 | -20 |
| 13 | 28 | 25 |
| 14 | 14 | -13 |
| 15 | 9 | -7 |
| 16 | 15 | -13 |
| 17 | 15 | -15 |
| 18 | 4 | 5 |
| 19 | 6 | -8 |
| 20* | 7 | 10 |
| 21* | 3 | -1 |
| 22* | 3 | -2 |
| 23* | 2 | -4 |
| 24 | 12 | 13 |
| **h83** | | |
| 0 | 34 | -22 |
| 1 | 24 | -20 |
| 2 | 10 | 2 |
| 3 | 11 | -10 |
| 4 | 27 | -25 |
| 5 | 20 | -17 |
| 6 | 4 | 8 |
| 7* | 23 | -16 |
| 8* | 3 | 3 |
| 9* | 3 | -1 |
| 10 | 27 | 26 |
| 11 | 37 | -34 |
| 12 | 29 | 24 |
| 13 | 4 | 2 |
| 14 | 16 | -12 |
| 15 | 28 | 27 |
| 16 | 4 | 8 |
| 17 | 4 | 6 |
| 18 | 15 | -13 |
| 19* | 14 | 11 |
| 20* | 3 | -6 |
| 21* | 3 | 2 |
| 22* | 10 | -9 |
| 23* | 2 | -1 |
| 24* | 2 | 1 |
| 25 | 5 | -5 |
| **h93** | | |
| 1 | 46 | -43 |
| 2 | 20 | 20 |
| 3* | 32 | 29 |
| 4* | 3 | -4 |
| 5* | 3 | 10 |
| 6 | 28 | 23 |
| 7 | 28 | 24 |
| 8* | 29 | -28 |
| 9* | 2 | -2 |
| 10 | 30 | 29 |
| 11 | 21 | 17 |
| 12 | 24 | -27 |
| 13 | 26 | -23 |
| 14 | 5 | 4 |
| 15* | 8 | -6 |
| 16* | 3 | -3 |
| 17* | 3 | -2 |
| 18* | 3 | -2 |
| 19* | 3 | -3 |
| 20* | 11 | 10 |
| 21* | 3 | -5 |
| 22 | 10 | -10 |
| 23 | 7 | -8 |
| 24 | 7 | 7 |
| 25 | 9 | 9 |
| **h,10,3** | | |
| 0* | 50 | -51 |
| 1* | 3 | 8 |
| 2* | 3 | 1 |
| 3 | 43 | -39 |
| 4* | 14 | -11 |
| 5* | 3 | -1 |
| 6* | 17 | 17 |
| 7* | 3 | -1 |
| 8* | 3 | -1 |
| 9 | 15 | -14 |
| 10 | 15 | 13 |
| 11 | 14 | 15 |
| 12* | 20 | 18 |
| 13* | 3 | -3 |
| 14* | 3 | -2 |
| 15* | 22 | 20 |
| 17* | 3 | -1 |
| 18 | 6 | -7 |
| 19* | 5 | 2 |
| 20* | 3 | 4 |
| 21 | 7 | 5 |
| 22 | 8 | -8 |
| 23 | 5 | -5 |
| 24 | 7 | -6 |
| **h,11,3** | | |
| 1 | 26 | -24 |
| 2 | 11 | 11 |
| 3 | 40 | 36 |
| 4 | 29 | -25 |
| 5 | 30 | -26 |
| 6 | 13 | 10 |
| 7 | 11 | 11 |
| 8* | 17 | -16 |
| 9* | 3 | -2 |
| 10 | 14 | 13 |
| 11 | 14 | 14 |
| 12* | 9 | -6 |
| 13* | 3 | -1 |
| 14* | 6 | -7 |
| 15* | 3 | -4 |
| 16 | 8 | 6 |
| 17* | 6 | -8 |
| 18* | 3 | -4 |
| 19* | 2 | -1 |
| 20* | 14 | 14 |
| 21* | 2 | 1 |
| 22 | 10 | -10 |
| **h,12,3** | | |
| 0* | 76 | 68 |
| 1* | 3 | -1 |
| 2 | 57 | -51 |
| 3* | 16 | -13 |
| 4* | 3 | 8 |
| 5* | 3 | -5 |
| 6* | 3 | -1 |
| 7* | 15 | -15 |
| 8* | 3 | 2 |
| 9 | 12 | -10 |
| 10* | 28 | 26 |
| 11* | 3 | 4 |
| 12* | 27 | -25 |
| 13* | 3 | 7 |
| 14* | 15 | 13 |
| 15* | 3 | 4 |
| 16* | 3 | 1 |
| 17* | 7 | 7 |
| 18 | 2 | 3 |
| 19 | 11 | 10 |
| 20 | 5 | -6 |
| 21 | 10 | -10 |
| 22 | 6 | -6 |
| **h,13,3** | | |
| 1* | 17 | 15 |
| 2* | 3 | 1 |
| 3 | 7 | -7 |
| 4 | 13 | -9 |
| 5 | 10 | -7 |
| 6 | 19 | 19 |
| 7 | 13 | 12 |
| 8 | 27 | -24 |
| 9 | 15 | -12 |
| 10 | 17 | 16 |
| 11 | 16 | 13 |
| 12 | 15 | -16 |
| 13 | 5 | -8 |
| 14 | 5 | 7 |
| 15* | 6 | -6 |
| 16* | 2 | 3 |
| 17* | 2 | -5 |
| 18 | 5 | -5 |
| **h,14,3** | | |
| 0 | 18 | 16 |
| 1 | 8 | 9 |
| 2 | 7 | -7 |
| 3 | 12 | -10 |
| 4* | 8 | -4 |
| 5* | 3 | -3 |
| 6 | 8 | -8 |
| 7 | 5 | -2 |
| 8* | 10 | 11 |
| 9* | 3 | 1 |
| 10* | 7 | 9 |
| 11* | 3 | 3 |
| 12 | 11 | -11 |
| 13 | 6 | -6 |
| 14 | 15 | 17 |
| 15 | 14 | 14 |
| 16 | 11 | -12 |
| **h,15,3** | | |
| 1* | 3 | -5 |
| 2 | 10 | 9 |
| 3* | 13 | 11 |
| 4* | 3 | 4 |
| 5 | 12 | -10 |
| 6 | 14 | -12 |
| 7 | 12 | 11 |
| 8* | 7 | 7 |
| 9* | 3 | 4 |
| 10* | 3 | -5 |
| 11 | 11 | -11 |
| **h,16,3** | | |
| 1* | 3 | -3 |
| 2* | 11 | -12 |
| 3* | 3 | -4 |
| 4* | 11 | 8 |
| 5* | 3 | -3 |
| 6* | 2 | -3 |
| 7* | 2 | -3 |
| 8* | 2 | 4 |
| 9* | 2 | 4 |
| 10* | 2 | 3 |
| 11* | 2 | -1 |
| 12* | 2 | -3 |
| 13* | 2 | 1 |
| 14 | 9 | 7 |
| **h,17,3** | | |
| 4* | 6 | 7 |
| 5* | 2 | 4 |
| 6 | 12 | -12 |

* Indicates a reflexion with $I_o=0$, $|F_o|=\frac{1}{3}|F_{\min}|$ of the appropriate layer line for these reflexions.

† Indicates $|F_o|$ omitted for these reflexions in the last three refinement cycles because the intensities were too high to measure accurately.

## Table 1 (*cont.*)

**h,18,3**

| | | |
|---|---|---|
| 0 | 6 | 6 |
| 1* | 2 | 2 |
| 2 | 10 | -10 |
| 3* | 2 | 3 |
| 4 | 10 | 10 |
| 5 | 5 | -5 |

**h04**

| | | |
|---|---|---|
| 1 | 24 | -41 |
| 2* | 50 | -75 |
| 3 | 34 | 29 |
| 4 | 9 | -16 |
| 5 | 64 | -70 |
| 6 | 8 | -3 |
| 7 | 41 | 42 |
| 8 | 93 | 93 |
| 9 | 26 | 31 |
| 10 | 41 | 39 |
| 11 | 50 | -52 |
| 12 | 14 | -12 |
| 13 | 34 | 31 |
| 14 | 18 | 17 |
| 15 | 54 | -57 |
| 16* | 3 | -3 |
| 17 | 32 | 27 |
| 18 | 7 | -4 |
| 19 | 13 | 13 |
| 20 | 16 | -12 |
| 21 | 9 | -8 |
| 22 | 25 | -21 |
| 23 | 7 | 7 |
| 24 | 8 | -7 |
| 25 | 6 | -4 |

**h14**

| | | |
|---|---|---|
| 1 | 14 | 18 |
| 2 | 25 | 35 |
| 3 | 15 | -11 |
| 4 | 42 | -51 |
| 5 | 17 | -17 |
| 6 | 17 | 8 |
| 7 | 37 | 38 |
| 8* | 2 | -1 |
| 9* | 2 | -4 |
| 10 | 23 | 21 |
| 11 | 8 | 7 |
| 12 | 15 | 16 |
| 13 | 27 | -27 |
| 14 | 16 | -15 |
| 15 | 14 | 15 |
| 16 | 21 | -18 |
| 17* | 3 | 3 |
| 18 | 17 | 17 |
| 19 | 28 | -24 |
| 20* | 3 | -6 |
| 21 | 8 | 7 |
| 22 | 11 | 10 |
| 23* | 3 | -3 |
| 24 | 6 | -7 |

**h24**

| | | |
|---|---|---|
| 0 | 51 | -63 |
| 1 | 19 | -16 |
| 2 | 8 | -7 |
| 3 | 12 | 16 |
| 4 | 93 | -103 |
| 5 | 12 | 8 |
| 6 | 29 | 22 |
| 7 | 23 | 22 |
| 8 | 60 | 55 |
| 9 | 22 | -18 |
| 10 | 39 | 38 |
| 11* | 3 | 3 |
| 12 | 50 | 47 |
| 13 | 15 | 12 |
| 14 | 6 | 1 |
| 15 | 10 | -7 |
| 16 | 25 | 19 |
| 17 | 27 | 27 |
| 18 | 20 | -17 |
| 19 | 21 | -20 |
| 20 | 31 | -26 |
| 21* | 3 | -3 |
| 22 | 15 | -13 |

**h34**

| | | |
|---|---|---|
| 1 | 4 | 2 |
| 2 | 16 | -22 |
| 3 | 31 | -29 |
| 4 | 22 | -20 |
| 5 | 71 | -69 |
| 6 | 13 | -13 |
| 7 | 121 | 112 |
| 8 | 21 | -17 |
| 9 | 57 | -49 |
| 10 | 29 | -26 |
| 11 | 29 | 27 |
| 12 | 17 | 13 |
| 13* | 3 | -2 |
| 14 | 9 | -7 |
| 15 | 10 | -7 |
| 16 | 36 | 30 |
| 17 | 22 | 17 |
| 18* | 3 | -4 |
| 19 | 25 | -19 |
| 20 | 14 | 11 |
| 21 | 18 | 15 |
| 22 | 12 | 10 |
| 23 | 10 | -7 |
| 24 | 10 | -9 |

**h44**

| | | |
|---|---|---|
| 0 | 112 | -120 |
| 1 | 21 | -26 |
| 2 | 9 | 6 |
| 3 | 15 | 15 |
| 4 | 5 | -2 |
| 5 | 33 | -32 |
| 6 | 18 | -11 |
| 7 | 25 | -26 |
| 8 | 48 | 41 |
| 9 | 18 | 19 |
| 10 | 12 | 9 |
| 11 | 31 | -31 |
| 12 | 49 | 44 |
| 13 | 11 | 9 |
| 14 | 21 | 18 |
| 15 | 29 | 26 |
| 16 | 23 | -20 |
| 17 | 7 | 5 |
| 18 | 21 | -19 |
| 19 | 18 | -12 |
| 20 | 14 | -11 |
| 21* | 3 | -4 |
| 22 | 8 | -7 |
| 23 | 9 | 5 |

**h54**

| | | |
|---|---|---|
| 1 | 19 | 18 |
| 2 | 15 | 12 |
| 3 | 12 | 8 |
| 4 | 34 | -33 |
| 5 | 51 | -49 |
| 6 | 8 | -6 |
| 7 | 58 | 55 |
| 8 | 27 | -22 |
| 9 | 34 | -30 |
| 10 | 49 | -45 |
| 11 | 34 | 29 |
| 12 | 32 | 28 |
| 13 | 38 | -36 |
| 14 | 5 | 2 |
| 15 | 5 | 6 |
| 16 | 29 | 25 |
| 17* | 3 | 2 |
| 18 | 10 | 7 |
| 19 | 9 | -9 |
| 20 | 6 | 7 |
| 21 | 16 | 16 |
| 22 | 4 | -4 |
| 23 | 11 | -12 |
| 24 | 4 | -4 |
| 25 | 9 | 10 |
| 26* | 2 | -3 |
| 27 | 5 | -7 |

**h64**

| | | |
|---|---|---|
| 0 | 22 | -22 |
| 1 | 5 | 2 |
| 2 | 50 | -50 |
| 3 | 55 | -52 |
| 4 | 12 | 8 |
| 5 | 17 | -10 |
| 6 | 20 | 17 |
| 7 | 26 | 22 |
| 8 | 42 | 37 |
| 9 | 12 | 10 |
| 10 | 22 | 22 |
| 11* | 3 | 4 |
| 12* | 3 | 3 |
| 13 | 13 | -9 |
| 14 | 5 | -6 |
| 15 | 22 | 19 |
| 16* | 3 | -5 |
| 17 | 10 | -9 |
| 18* | 3 | -1 |
| 19* | 3 | -3 |
| 20 | 13 | -14 |

**h74**

| | | |
|---|---|---|
| 1 | 19 | -17 |
| 2 | 38 | -41 |
| 3 | 63 | 64 |
| 4 | 15 | -16 |
| 5 | 61 | -61 |
| 6 | 40 | -35 |
| 7 | 29 | 30 |
| 8 | 12 | -10 |
| 9 | 37 | -34 |
| 10* | 3 | 3 |
| 11 | 26 | 22 |
| 12 | 10 | 7 |
| 13 | 19 | -16 |
| 14 | 24 | 23 |
| 15* | 3 | 4 |
| 16 | 10 | -9 |
| 17 | 17 | 14 |
| 18 | 18 | 17 |
| 19 | 12 | -10 |
| 20 | 7 | 5 |
| 21 | 12 | 11 |
| 22 | 8 | 6 |
| 23 | 10 | -10 |
| 24* | 2 | -5 |
| 25 | 6 | 6 |
| 26 | 6 | -7 |

**h84**

| | | |
|---|---|---|
| 1 | 36 | 28 |
| 2 | 8 | 6 |
| 3 | 38 | -36 |
| 4 | 38 | -36 |
| 5 | 31 | 28 |
| 6 | 12 | 12 |
| 7 | 5 | -2 |
| 9 | 31 | -30 |
| 10 | 32 | 29 |
| 11 | 31 | 30 |
| 12 | 5 | 1 |
| 13 | 15 | -12 |
| 14 | 5 | -5 |
| 15 | 11 | 10 |
| 16 | 5 | 5 |
| 17 | 10 | -8 |
| 18 | 12 | -12 |
| 19 | 9 | 8 |
| 20* | 3 | -2 |
| 21* | 3 | -5 |
| 22 | 11 | -12 |
| 23* | 2 | -2 |
| 24 | 7 | 7 |

**h94**

| | | |
|---|---|---|
| 1 | 16 | 16 |
| 2* | 2 | 1 |
| 3 | 18 | -17 |
| 4 | 24 | -25 |
| 5* | 3 | 2 |
| 6 | 25 | -25 |
| 7 | 5 | 8 |
| 8 | 15 | -12 |
| 9 | 16 | -18 |
| 10 | 15 | -14 |
| 11 | 15 | 11 |
| 12 | 23 | 23 |
| 13 | 18 | -14 |
| 14 | 11 | -9 |
| 15* | 3 | 5 |
| 16 | 20 | 17 |
| 17* | 3 | 5 |
| 18 | 9 | 7 |
| 19 | 5 | 4 |
| 20 | 12 | 12 |

**h,10,4**

| | | |
|---|---|---|
| 0 | 34 | 29 |
| 1 | 41 | 38 |
| 2 | 26 | -26 |
| 3 | 20 | -16 |
| 4 | 21 | 19 |
| 5 | 19 | 19 |
| 6* | 3 | 3 |
| 7* | 3 | -3 |
| 8 | 11 | -9 |
| 9 | 25 | -22 |
| 10 | 12 | 15 |
| 11 | 18 | 14 |
| 12* | 3 | -4 |
| 13 | 5 | -5 |
| 14* | 3 | 1 |
| 15 | 8 | 9 |
| 16 | 5 | -8 |
| 17 | 14 | -16 |
| 18 | 9 | -10 |
| 19 | 11 | 10 |
| 20* | 3 | -3 |
| 21* | 2 | -1 |
| 22* | 2 | -1 |
| 23* | 2 | -5 |
| 24 | 3 | 2 |

**h,11,4**

| | | |
|---|---|---|
| 1 | 17 | 14 |
| 2 | 16 | 14 |
| 3 | 17 | -15 |
| 4 | 21 | -17 |
| 5* | 3 | -1 |
| 6* | 3 | -1 |
| 7* | 3 | -2 |
| 8 | 21 | -22 |
| 9 | 14 | 14 |
| 10* | 3 | 3 |
| 11* | 3 | -1 |
| 12* | 3 | 5 |
| 13 | 13 | -11 |
| 14* | 3 | 4 |
| 15* | 3 | 3 |
| 16 | 12 | 12 |
| 17* | 3 | 1 |
| 18 | 8 | 7 |
| 19* | 3 | -1 |
| 20* | 2 | 1 |
| 21* | 2 | -3 |
| 22* | 2 | -2 |

**h,12,4**

| | | |
|---|---|---|
| 1 | 10 | -6 |
| 2 | 16 | -16 |
| 3 | 14 | -13 |
| 4 | 18 | 17 |
| 5 | 20 | 17 |
| 6 | 8 | 8 |
| 7 | 11 | -9 |
| 8 | 5 | -4 |
| 9* | 3 | 4 |
| 10 | 5 | 6 |
| 11 | 11 | 9 |
| 12 | 8 | -6 |
| 13 | 9 | -10 |
| 14* | 3 | 2 |
| 15* | 3 | 6 |
| 16 | 11 | -11 |
| 17 | 9 | -7 |
| 18* | 2 | 2 |
| 19 | 6 | 8 |

**h,13,4**

| | | |
|---|---|---|
| 1 | 10 | 8 |
| 2 | 15 | -14 |
| 3 | 18 | -20 |
| 4 | 12 | -11 |
| 5 | 11 | 11 |
| 6* | 3 | -6 |
| 7 | 11 | -10 |
| 8* | 3 | -2 |
| 9 | 13 | 14 |
| 10* | 3 | -1 |
| 11 | 10 | -10 |
| 12* | 3 | -4 |
| 13* | 3 | 6 |
| 14 | 9 | 12 |

**h,14,4**

| | | |
|---|---|---|
| 1* | 3 | -4 |
| 2 | 9 | 7 |
| 3* | 3 | 1 |
| 4* | 3 | 5 |
| 5* | 3 | -1 |
| 6* | 3 | 1 |
| 7 | 12 | -11 |
| 8* | 3 | -7 |
| 9 | 8 | 7 |
| 10* | 3 | 1 |
| 11* | 3 | -4 |
| 12* | 3 | 2 |
| 13* | 3 | -3 |
| 14* | 2 | -2 |
| 15 | 7 | 8 |
| 16 | 7 | -7 |

**h,15,4**

| | | |
|---|---|---|
| 5 | 18 | 21 |
| 6 | 10 | -11 |
| 7 | 18 | -23 |
| 8* | 3 | 1 |
| 9 | 18 | 21 |
| 10* | 3 | 3 |
| 11 | 14 | -14 |
| 12* | 2 | 3 |
| 13 | 6 | 2 |
| 14* | 2 | 2 |
| 15* | 2 | -1 |
| 16 | 4 | 2 |

**h,16,4**

| | | |
|---|---|---|
| 0 | 19 | 20 |
| 1* | 3 | -5 |
| 2 | 12 | -12 |
| 3 | 8 | 9 |
| 4* | 3 | 5 |
| 5* | 3 | -6 |
| 6* | 3 | -3 |
| 7* | 2 | 1 |
| 8* | 2 | 2 |
| 9* | 2 | 2 |
| 10* | 2 | 1 |
| 11 | 7 | -7 |
| 12* | 2 | -4 |
| 13 | 9 | 13 |

**h,17,4**

| | | |
|---|---|---|
| 6 | 8 | -9 |
| 7 | 11 | -14 |
| 8 | 4 | -6 |
| 9 | 7 | 10 |
| 10 | 6 | 11 |

**h,18,4**

| | | |
|---|---|---|
| 1 | 7 | -13 |
| 2* | 2 | -2 |
| 3 | 5 | 9 |

**h15**

| | | |
|---|---|---|
| 1 | 15 | -16 |
| 2 | 37 | -49 |
| 3 | 22 | -25 |
| 4 | 4 | 1 |
| 5 | 34 | -39 |
| 6 | 79 | -89 |
| 7 | 53 | 57 |
| 8 | 17 | 16 |
| 9* | 2 | -3 |
| 10 | 43 | 45 |
| 11 | 33 | 34 |
| 12 | 38 | 38 |
| 13 | 7 | 5 |
| 14 | 25 | 20 |
| 15 | 8 | -9 |
| 16 | 24 | 22 |
| 17 | 21 | 20 |
| 18 | 25 | 21 |
| 19 | 20 | -17 |
| 20 | 19 | -17 |
| 21 | 8 | -5 |
| 22 | 8 | 8 |
| 23 | 16 | -14 |
| 24 | 19 | -19 |

**h25**

| | | |
|---|---|---|
| 0 | 38 | -41 |
| 1 | 13 | 15 |
| 2 | 59 | 72 |
| 3 | 9 | 8 |
| 4 | 36 | -39 |
| 5 | 13 | -14 |
| 6 | 15 | 14 |
| 7 | 19 | -18 |
| 8 | 19 | 19 |
| 9 | 8 | 7 |
| 10 | 37 | -37 |
| 11 | 14 | -14 |
| 12 | 7 | -7 |
| 13 | 16 | -14 |
| 14 | 28 | -27 |
| 15 | 35 | 35 |
| 16 | 22 | 20 |
| 17 | 8 | -8 |
| 18* | 3 | 1 |
| 19 | 9 | -8 |
| 20 | 7 | 5 |
| 21 | 10 | 9 |
| 22 | 8 | 6 |

**h35**

| | | |
|---|---|---|
| 1 | 32 | -34 |
| 2 | 46 | -48 |
| 3 | 18 | 17 |
| 4 | 9 | 10 |
| 5 | 13 | -14 |
| 6 | 82 | -90 |
| 7 | 15 | -11 |
| 8 | 29 | 29 |
| 9 | 38 | 39 |
| 10 | 9 | 8 |
| 11 | 30 | 29 |
| 12 | 31 | 29 |
| 13 | 5 | -5 |
| 14 | 18 | -17 |
| 15* | 3 | 1 |
| 16 | 15 | 15 |
| 17 | 12 | -11 |
| 18 | 11 | 5 |
| 19* | 3 | -2 |
| 20 | 11 | 9 |
| 21* | 3 | -3 |
| 22* | 2 | -3 |
| 23 | 11 | -11 |
| 24 | 9 | -9 |

**h45**

| | | |
|---|---|---|
| 0 | 12 | 14 |
| 1 | 21 | 19 |
| 2 | 33 | 34 |
| 3 | 9 | -8 |
| 4* | 2 | -3 |
| 5 | 18 | -20 |
| 6 | 26 | -23 |
| 7 | 18 | -17 |
| 8* | 2 | 4 |
| 9 | 9 | 8 |
| 10 | 41 | -40 |
| 11 | 22 | 21 |
| 12 | 17 | 15 |
| 13 | 17 | -16 |
| 14* | 3 | 1 |
| 15* | 3 | 4 |
| 16* | 3 | 2 |
| 17 | 10 | 9 |
| 18* | 3 | -5 |
| 19* | 3 | 4 |
| 20* | 3 | 1 |
| 21* | 3 | 2 |
| 22 | 8 | 7 |

**h55**

| | | |
|---|---|---|
| 1 | 26 | -25 |
| 2 | 17 | 17 |
| 3 | 3 | -1 |
| 4* | 2 | 3 |
| 5 | 24 | -21 |
| 6 | 45 | -47 |
| 7 | 22 | 16 |
| 8 | 16 | -13 |
| 9 | 10 | 7 |
| 10 | 11 | 8 |
| 11 | 29 | 24 |
| 12 | 11 | -10 |
| 13 | 7 | 7 |
| 14 | 28 | 26 |
| 15* | 3 | -5 |
| 16 | 8 | -4 |
| 17 | 8 | 7 |
| 18 | 23 | 19 |
| 19* | 3 | -4 |
| 20* | 2 | 2 |
| 21 | 9 | -10 |
| 22 | 7 | -7 |
| 23 | 7 | -7 |
| 24 | 8 | -6 |
| 25 | 7 | 6 |

**h65**

| | | |
|---|---|---|
| 0 | 34 | -34 |
| 1 | 32 | 28 |
| 2 | 48 | 48 |
| 3 | 18 | -14 |
| 4 | 15 | -8 |
| 5 | 30 | -29 |
| 6 | 16 | 12 |
| 7 | 12 | -11 |
| 8 | 18 | -16 |
| 9 | 17 | -15 |
| 10 | 12 | -11 |
| 11 | 8 | 6 |
| 12* | 3 | -1 |
| 13* | 3 | -4 |
| 14 | 26 | -22 |
| 15 | 8 | 6 |
| 16 | 8 | 8 |
| 17* | 3 | 2 |
| 18 | 13 | -12 |
| 19 | 17 | 15 |
| 20 | 16 | 14 |
| 21* | 2 | 4 |
| 22* | 2 | 2 |
| 23* | 2 | -5 |
| 24 | 6 | 6 |

**h75**

| | | |
|---|---|---|
| 1 | 14 | -11 |
| 2 | 13 | -7 |
| 3 | 7 | -5 |
| 4 | 21 | -17 |
| 5 | 24 | 24 |
| 6 | 24 | 22 |
| 7 | 14 | -11 |
| 8 | 30 | -32 |
| 9 | 21 | 21 |
| 10 | 8 | 8 |
| 11* | 3 | -1 |
| 12* | 3 | -1 |
| 13* | 3 | -5 |
| 14 | 12 | 11 |
| 15 | 9 | 8 |
| 16 | 24 | 20 |
| 17 | 15 | -14 |
| 18 | 12 | -9 |
| 19* | 3 | -3 |
| 20* | 2 | 2 |
| 21 | 7 | -7 |
| 22 | 10 | -10 |

**h85**

| | | |
|---|---|---|
| 0 | 19 | -20 |
| 1 | 42 | 40 |
| 2 | 25 | 19 |
| 3 | 4 | -3 |
| 4* | 2 | 1 |
| 5 | 12 | -11 |
| 6* | 2 | 3 |
| 7 | 19 | -19 |
| 8 | 15 | 11 |
| 9 | 24 | -24 |
| 10 | 18 | -13 |
| 11 | 36 | 34 |
| 12 | 15 | -12 |
| 13* | 3 | -1 |
| 14 | 12 | -9 |
| 15* | 3 | -3 |
| 16* | 3 | -2 |
| 17 | 12 | 11 |
| 18* | 3 | 1 |
| 19 | 12 | 12 |
| 20 | 12 | 13 |
| 21* | 2 | -3 |
| 22* | 2 | -1 |
| 23 | 10 | -8 |

**h95**

| | | |
|---|---|---|
| 1 | 24 | 20 |
| 2 | 8 | 3 |
| 3 | 45 | -43 |
| 4 | 67 | -68 |
| 5* | 3 | 2 |
| 6 | 48 | 45 |
| 7 | 16 | 16 |
| 8 | 32 | -30 |
| 9 | 12 | 12 |
| 10 | 23 | 19 |
| 11 | 7 | -6 |
| 12 | 7 | 7 |
| 13* | 3 | -1 |
| 14* | 3 | 3 |
| 15* | 3 | 1 |
| 16 | 10 | 9 |
| 17* | 2 | -2 |
| 18 | 13 | -10 |
| 19 | 4 | -4 |
| 20 | 9 | 9 |
| 21* | 2 | -3 |
| 22 | 8 | -7 |
| 23 | 10 | 9 |

**h,10,5**

| | | |
|---|---|---|
| 0 | 42 | 42 |
| 1 | 31 | -31 |
| 2* | 3 | -6 |
| 3* | 3 | 4 |
| 4 | 13 | 11 |
| 5 | 13 | -9 |
| 6 | 15 | -12 |
| 7 | 32 | -29 |
| 8* | 3 | -4 |
| 9 | 16 | 16 |
| 10 | 7 | -7 |
| 11 | 11 | -10 |
| 12 | 16 | -15 |
| 13* | 3 | 6 |
| 14* | 3 | 5 |
| 15 | 10 | 9 |
| 16 | 8 | -8 |
| 17* | 2 | -1 |
| 18 | 11 | 8 |
| 19 | 6 | 6 |
| 20* | 2 | -2 |
| 21* | 2 | 1 |
| 22 | 6 | 6 |

**h,11,5**

| | | |
|---|---|---|
| 1 | 14 | 12 |
| 2 | 22 | 18 |
| 3 | 13 | -12 |
| 4 | 29 | -26 |
| 5 | 13 | 12 |
| 6 | 24 | 22 |
| 7* | 3 | -4 |
| 8 | 34 | -31 |
| 9 | 8 | 7 |
| 10 | 27 | 26 |
| 11 | 15 | -14 |
| 12 | 10 | -10 |
| 13* | 3 | 4 |
| 14 | 14 | 16 |
| 15* | 2 | 3 |
| 16* | 2 | -5 |
| 17 | 15 | -14 |
| 18 | 4 | -1 |

**h,12,5**

| | | |
|---|---|---|
| 1 | 12 | 10 |
| 2 | 15 | 10 |
| 3* | 3 | -4 |
| 4 | 12 | 9 |
| 5 | 15 | -13 |
| 6* | 3 | -4 |
| 7* | 3 | 2 |
| 8 | 8 | -7 |
| 9* | 3 | 2 |
| 10 | 8 | -6 |
| 11* | 2 | 1 |
| 12 | 13 | -13 |
| 13* | 2 | 2 |
| 14 | 9 | 9 |
| 15 | 4 | 4 |
| 16 | 8 | -8 |
| 17* | 2 | 1 |
| 18 | 3 | 3 |

**h,13,5**

| | | |
|---|---|---|
| 1* | 3 | -4 |
| 2 | 9 | 8 |
| 3* | 3 | 3 |
| 4 | 5 | -4 |
| 5 | 8 | 8 |
| 6 | 8 | 6 |
| 7 | 9 | -8 |
| 8 | 8 | -6 |
| 9 | 9 | 9 |
| 10 | 8 | 8 |
| 11 | 12 | -11 |

**h,14,5**

| | | |
|---|---|---|
| 0 | 26 | 22 |
| 1 | 15 | -16 |
| 2 | 20 | -19 |
| 3 | 8 | 7 |
| 4 | 20 | 18 |
| 5* | 2 | 1 |
| 6* | 2 | -2 |
| 7 | 11 | -11 |
| 8 | 13 | -10 |
| 9* | 2 | 2 |
| 10* | 2 | -1 |
| 11 | 6 | -6 |
| 12 | 6 | -7 |
| 13 | 13 | 12 |
| 14* | 2 | 5 |
| 15* | 2 | -3 |
| 16* | 1 | -7 |
| 17 | 5 | 2 |

**h,15,5**

| | | |
|---|---|---|
| 1* | 2 | -1 |
| 2* | 2 | -4 |
| 3* | 2 | 1 |
| 4 | 4 | -5 |
| 5 | 12 | 9 |
| 6 | 9 | 5 |
| 7 | 7 | -8 |
| 8* | 2 | 3 |
| 9* | 2 | 1 |
| 10* | 2 | 4 |
| 11* | 2 | -2 |
| 12* | 2 | -7 |
| 13 | 7 | 2 |

**h,16,5**

| | | |
|---|---|---|
| 0 | 16 | 17 |
| 1* | 2 | 2 |
| 2 | 10 | -9 |
| 3* | 2 | 1 |
| 4 | 9 | 9 |
| 5 | 5 | -6 |
| 6 | 5 | -6 |
| 7 | 5 | 5 |
| 8 | 5 | -6 |
| 9* | 2 | -5 |
| 10 | 4 | 8 |
| 11* | 1 | 1 |
| 12 | 6 | -9 |

**h,17,5**

| | | |
|---|---|---|
| 1* | 1 | -1 |
| 2* | 2 | -2 |
| 3 | 5 | 5 |

**h06**

| | | |
|---|---|---|
| 1 | 30 | -43 |
| 2 | 36 | 40 |
| 3 | 16 | -14 |
| 4 | 32 | -34 |
| 5 | 21 | -23 |
| 6 | 19 | 24 |
| 7 | 28 | -25 |
| 8 | 17 | -22 |
| 9 | 14 | -11 |
| 10 | 26 | -25 |
| 11 | 16 | 15 |
| 12 | 51 | 52 |
| 13 | 13 | 11 |
| 14 | 16 | -12 |
| 15 | 18 | 16 |
| 16* | 2 | -1 |
| 17 | 17 | 15 |
| 18 | 15 | -13 |
| 19 | 15 | -14 |
| 20 | 6 | 5 |
| 21 | 22 | 22 |
| 22 | 10 | 10 |
| 23 | 7 | -7 |
| 24 | 7 | -7 |
| 25 | 9 | -9 |

**h16**

| | | |
|---|---|---|
| 1 | 13 | -15 |
| 2 | 11 | 12 |
| 3 | 25 | 29 |
| 4 | 6 | -8 |
| 5 | 15 | -15 |
| 6 | 6 | -3 |
| 7 | 11 | -11 |
| 8* | 1 | 1 |
| 9 | 7 | -7 |
| 10* | 2 | -2 |
| 11 | 13 | 11 |
| 12 | 9 | -9 |
| 13 | 15 | 16 |
| 14 | 4 | 4 |
| 15 | 4 | -4 |
| 16 | 19 | 17 |
| 17 | 14 | -13 |
| 18 | 13 | -13 |
| 19 | 4 | 5 |
| 20 | 12 | 12 |
| 21* | 2 | 1 |
| 22 | 10 | -10 |
| 23* | 2 | -1 |
| 24* | 2 | 3 |
| 25 | 5 | 7 |

**h26**

| | | |
|---|---|---|
| 1 | 21 | -21 |
| 2* | 1 | 1 |
| 3 | 11 | 10 |
| 4 | 8 | 7 |
| 5 | 53 | -57 |
| 6* | 2 | -4 |
| 7 | 24 | -25 |
| 8 | 5 | 8 |
| 9 | 12 | -14 |
| 10 | 13 | -15 |
| 11 | 17 | -17 |
| 12* | 2 | 3 |
| 13 | 44 | 43 |
| 14 | 16 | -14 |
| 15 | 7 | -6 |
| 16 | 11 | 12 |
| 17 | 23 | 21 |
| 18* | 2 | 2 |
| 19 | 17 | 12 |
| 20* | 2 | -3 |
| 21* | 2 | 1 |
| 22 | 9 | 10 |
| 23* | 2 | -4 |
| 24 | 7 | -8 |
| 25 | 11 | 13 |

**h36**

| | | |
|---|---|---|
| 1 | 13 | -12 |
| 2 | 9 | -12 |
| 3 | 34 | 34 |
| 4 | 8 | -3 |
| 5 | 14 | 14 |
| 6 | 20 | 16 |
| 7 | 10 | -10 |
| 8 | 29 | -27 |
| 9 | 22 | -22 |
| 10 | 29 | 30 |
| 11 | 26 | 28 |
| 12 | 26 | -29 |
| 13 | 34 | -34 |
| 14 | 19 | 18 |
| 15* | 2 | -1 |
| 16* | 2 | 2 |
| 17 | 7 | -6 |
| 18 | 13 | -12 |
| 19* | 2 | -3 |
| 20 | 7 | 8 |
| 21 | 10 | 10 |
| 22 | 7 | -7 |
| 23* | 2 | -3 |
| 24 | 7 | 8 |
| 25 | 9 | 11 |

**h46**

| | | |
|---|---|---|
| 0 | 17 | 13 |
| 1* | 1 | -1 |
| 2 | 6 | -4 |
| 3 | 28 | -27 |
| 4 | 7 | 8 |
| 5 | 25 | -23 |
| 6* | 2 | 3 |
| 7 | 22 | -17 |
| 8 | 11 | -10 |
| 9 | 29 | -29 |
| 10 | 9 | 7 |
| 11 | 33 | 29 |
| 12 | 15 | -13 |
| 13 | 18 | 18 |
| 14 | 8 | -6 |
| 15 | 8 | 4 |
| 16 | 8 | 5 |
| 17 | 18 | 16 |
| 18 | 6 | 5 |
| 19 | 6 | 4 |
| 20 | 7 | 7 |
| 21* | 2 | 1 |
| 22 | 8 | -9 |
| 23 | 16 | -15 |

**h56**

| | | |
|---|---|---|
| 1 | 17 | -18 |
| 2 | 25 | 24 |
| 3 | 46 | 47 |
| 4 | 32 | -32 |
| 5 | 12 | -9 |
| 6 | 27 | 29 |
| 7 | 26 | 29 |
| 8 | 36 | -37 |
| 9 | 44 | -47 |
| 10 | 39 | 40 |
| 11 | 8 | 5 |
| 12 | 10 | -8 |
| 13 | 21 | -17 |
| 14 | 5 | -5 |
| 15 | 11 | -9 |
| 16* | 2 | 1 |
| 17 | 9 | 8 |
| 18 | 8 | -7 |
| 19* | 2 | 1 |
| 20 | 9 | 9 |
| 21 | 15 | 15 |
| 22 | 7 | -6 |
| 23* | 2 | -2 |
| 24 | 4 | 3 |

**h66**

| | | |
|---|---|---|
| 0 | 11 | 11 |
| 1 | 13 | 13 |
| 2 | 12 | 13 |
| 3 | 34 | -31 |
| 4* | 2 | -8 |
| 5 | 20 | -23 |
| 6* | 2 | -4 |
| 7 | 20 | -20 |
| 8 | 23 | -20 |
| 9 | 12 | -8 |
| 10 | 9 | 7 |
| 11 | 29 | 31 |
| 12* | 2 | -2 |
| 13 | 11 | 8 |
| 14* | 2 | 1 |
| 15* | 2 | 4 |
| 16 | 4 | 1 |
| 17* | 2 | 1 |
| 18* | 2 | -1 |
| 19 | 10 | 9 |
| 20* | 2 | 5 |
| 21 | 6 | -6 |

**h76**

| | | |
|---|---|---|
| 1 | 36 | 35 |
| 2 | 23 | 22 |
| 3* | 2 | -3 |
| 4 | 24 | -24 |
| 5 | 10 | 9 |
| 6 | 32 | 32 |
| 7* | 2 | -6 |
| 8 | 26 | -26 |
| 9 | 42 | -42 |
| 10 | 13 | 10 |
| 11* | 2 | 8 |
| 12* | 2 | -2 |
| 13 | 17 | -15 |
| 14 | 9 | -8 |
| 15* | 2 | 3 |
| 16 | 9 | 10 |
| 17* | 2 | -3 |
| 18 | 13 | -12 |
| 19* | 2 | 1 |
| 20 | 15 | 14 |
| 21 | 9 | 9 |
| 22 | 13 | -12 |

**h86**

| | | |
|---|---|---|
| 0 | 15 | 15 |
| 1 | 16 | 13 |
| 2 | 22 | -21 |
| 3 | 8 | -6 |
| 4 | 34 | 33 |
| 5* | 2 | -2 |
| 6 | 12 | -12 |
| 7 | 17 | -18 |
| 8* | 2 | -5 |
| 9 | 10 | 13 |
| 10* | 2 | 1 |
| 11* | 2 | 1 |
| 12 | 13 | -14 |
| 13 | 9 | -7 |
| 14 | 15 | 16 |
| 15 | 22 | 22 |
| 16 | 9 | -9 |
| 17 | 6 | -6 |
| 18 | 6 | 4 |
| 19* | 2 | 2 |
| 20* | 2 | -1 |
| 21* | 1 | -5 |
| 22 | 4 | 4 |

**h96**

| | | |
|---|---|---|
| 1 | 9 | 7 |
| 2 | 8 | -3 |
| 3 | 24 | 23 |
| 4* | 2 | -3 |
| 5 | 4 | -4 |
| 6 | 24 | 25 |
| 7* | 2 | -3 |
| 8 | 19 | -20 |
| 9 | 19 | -17 |
| 10 | 18 | 18 |
| 11 | 8 | -6 |
| 12 | 15 | -15 |
| 13 | 8 | -7 |
| 14 | 8 | 5 |
| 15* | 2 | 1 |
| 16* | 2 | -3 |
| 17 | 3 | -3 |
| 18* | 2 | -1 |
| 19* | 2 | 3 |
| 20* | 1 | -2 |
| 21 | 10 | 9 |

**h,10,6**

| | | |
|---|---|---|
| 0 | 9 | 8 |
| 1 | 9 | 9 |
| 2 | 6 | -7 |
| 3 | 11 | -10 |
| 4* | 2 | -5 |
| 5 | 9 | 7 |
| 6 | 10 | 10 |
| 7* | 2 | -4 |
| 8* | 2 | -1 |
| 9 | 12 | 10 |
| 10* | 2 | -2 |
| 11* | 2 | 2 |
| 12 | 7 | -7 |
| 13 | 13 | -13 |
| 14 | 9 | 11 |
| 15 | 17 | 18 |
| 16 | 4 | -5 |
| 17* | 2 | -3 |
| 18 | 5 | 6 |

**h,11,6**

| | | |
|---|---|---|
| 1* | 2 | -3 |
| 2 | 7 | -7 |
| 3* | 2 | 3 |
| 4 | 15 | 15 |
| 5 | 24 | 24 |
| 6 | 17 | -19 |
| 7 | 15 | -16 |
| 8 | 14 | 13 |
| 9* | 2 | -4 |
| 10* | 2 | 1 |
| 11 | 10 | -11 |
| 12 | 9 | -11 |
| 13* | 2 | 0 |
| 14 | 8 | 10 |
| 15* | 2 | 1 |
| 16 | 6 | -6 |
| 17 | 5 | -5 |
| 18* | 1 | -1 |
| 19 | 9 | 9 |

**h,12,6**

| | | |
|---|---|---|
| 0 | 16 | 11 |
| 1 | 17 | 16 |
| 2* | 2 | 1 |
| 3 | 9 | -9 |
| 4* | 2 | -2 |
| 5 | 6 | -5 |
| 6 | 11 | -11 |
| 7* | 2 | 1 |
| 8 | 6 | 5 |
| 9 | 7 | 6 |
| 10 | 7 | 8 |
| 11* | 2 | 2 |
| 12 | 9 | -9 |
| 13 | 3 | -2 |
| 14* | 2 | 3 |
| 15* | 2 | 4 |
| 16* | 1 | -1 |
| 17 | 5 | -5 |

**h,13,6**

| | | |
|---|---|---|
| 1 | 8 | 8 |
| 2* | 2 | -1 |
| 3* | 2 | -3 |
| 4 | 11 | 10 |
| 5 | 17 | 16 |
| 6 | 11 | -10 |
| 7 | 19 | -21 |
| 8 | 7 | 8 |
| 9 | 7 | 7 |
| 10* | 2 | 3 |
| 11 | 7 | -9 |

**h,14,6**

| | | |
|---|---|---|
| 1 | 5 | -5 |
| 2 | 6 | -6 |
| 3* | 2 | 3 |
| 4 | 10 | 10 |
| 5* | 2 | 3 |
| 6* | 2 | -5 |
| 7* | 2 | -1 |
| 8* | 2 | 2 |
| 9* | 2 | 2 |
| 10* | 2 | 1 |
| 11* | 2 | -1 |
| 12* | 1 | -4 |
| 13 | 5 | 5 |

**h,15,6**

| | | |
|---|---|---|
| 6 | 5 | -7 |
| 7* | 2 | -2 |
| 8 | 7 | 7 |
| 9* | 1 | 1 |
| 10 | 5 | -6 |
| 11 | 8 | -10 |

**h,16,6**

| | | |
|---|---|---|
| 0 | 14 | -13 |
| 1 | 8 | -9 |
| 2 | 8 | 9 |
| 3* | 1 | 4 |
| 4 | 5 | -4 |
| 5* | 1 | -1 |

**h17**

| | | |
|---|---|---|
| 1 | 5 | 4 |
| 2 | 10 | -9 |
| 3 | 45 | 51 |
| 4 | 9 | -6 |
| 5 | 25 | -24 |
| 6 | 37 | -41 |
| 7 | 17 | 19 |
| 8* | 2 | 2 |
| 9 | 44 | -45 |
| 10* | 2 | 3 |
| 11 | 10 | -10 |
| 12 | 7 | 4 |
| 13 | 16 | -17 |
| 14* | 3 | 3 |
| 15* | 3 | 8 |
| 16 | 15 | 13 |
| 17* | 3 | 3 |
| 18* | 3 | -1 |
| 19* | 2 | -1 |
| 20 | 7 | 6 |
| 21 | 18 | 20 |

**h27**

| | | |
|---|---|---|
| 0 | 8 | 3 |
| 1 | 19 | 18 |
| 2 | 5 | -2 |
| 3 | 21 | -24 |
| 4 | 19 | 20 |
| 5 | 15 | 14 |
| 6* | 2 | 1 |
| 7 | 20 | 22 |
| 8 | 20 | -21 |
| 9 | 13 | -10 |
| 10 | 4 | -1 |
| 11 | 21 | 20 |
| 12* | 3 | 2 |
| 13 | 8 | -8 |
| 14* | 3 | -4 |
| 15 | 16 | -14 |
| 16* | 3 | -1 |
| 17 | 7 | -7 |
| 18* | 3 | -1 |
| 19* | 2 | 5 |
| 20 | 9 | 8 |
| 21* | 2 | -1 |
| 22 | 5 | -6 |

**h37**

| | | |
|---|---|---|
| 1 | 18 | 17 |
| 2* | 2 | 1 |
| 3 | 31 | 31 |
| 4 | 25 | -26 |
| 5 | 36 | -39 |
| 6 | 7 | 7 |
| 7 | 27 | 25 |
| 8 | 12 | -10 |
| 9 | 49 | -51 |
| 10 | 14 | -14 |
| 11 | 7 | 3 |
| 12* | 3 | 5 |
| 13* | 3 | 4 |
| 14 | 21 | 18 |
| 15* | 3 | -3 |
| 16* | 3 | 3 |
| 17 | 11 | 10 |
| 18* | 3 | 2 |
| 19 | 9 | -9 |
| 20* | 2 | -2 |
| 21 | 14 | 13 |

**h47**

| | | |
|---|---|---|
| 0 | 37 | -38 |
| 1 | 33 | 31 |
| 2 | 42 | 45 |
| 3 | 19 | -16 |
| 4 | 14 | -9 |
| 5 | 31 | 34 |
| 6 | 4 | 2 |
| 7 | 18 | 15 |
| 8 | 20 | -17 |
| 9 | 26 | -28 |
| 10* | 2 | -1 |
| 11 | 14 | 11 |
| 12* | 2 | -1 |
| 13 | 19 | -17 |
| 14 | 16 | -13 |
| 15 | 14 | 13 |
| 16 | 14 | 12 |
| 17 | 13 | -12 |
| 18 | 13 | -13 |
| 19* | 2 | 1 |
| 20 | 11 | 12 |

**h57**

| | | |
|---|---|---|
| 1 | 11 | 10 |
| 2 | 9 | 7 |
| 3* | 2 | -1 |
| 4 | 14 | -11 |
| 5 | 12 | 10 |
| 6 | 14 | 16 |
| 7* | 2 | -1 |
| 8 | 15 | -15 |
| 9 | 11 | -8 |
| 10 | 12 | -11 |
| 11 | 15 | -17 |
| 12 | 23 | 21 |
| 13* | 3 | -5 |
| 14 | 14 | -14 |
| 15 | 4 | 4 |
| 16 | 9 | 8 |
| 17 | 4 | -4 |
| 18* | 2 | 5 |
| 19 | 8 | 9 |
| 20* | 2 | 2 |
| 21 | 4 | 3 |

**h67**

| | | |
|---|---|---|
| 0 | 28 | 28 |
| 1 | 20 | 16 |
| 2* | 2 | 2 |
| 3 | 23 | -22 |
| 4 | 21 | -23 |
| 5 | 27 | 28 |
| 6 | 18 | 20 |
| 7 | 21 | 20 |
| 8* | 3 | 3 |
| 9 | 12 | -12 |
| 10 | 18 | -16 |
| 11 | 9 | 6 |
| 12* | 3 | 3 |
| 13 | 13 | -9 |
| 14 | 13 | -11 |
| 15 | 11 | 11 |
| 16* | 3 | 6 |
| 17 | 12 | -13 |
| 18* | 2 | -3 |
| 19 | 9 | -6 |

**h77**

| | | |
|---|---|---|
| 1 | 8 | 5 |
| 2* | 3 | 3 |
| 3 | 8 | -5 |
| 4 | 12 | -12 |
| 5 | 16 | 16 |
| 6* | 3 | 2 |
| 7 | 17 | -15 |
| 8 | 12 | -11 |
| 9* | 3 | -1 |
| 10 | 15 | 14 |
| 11 | 9 | -9 |
| 12 | 8 | -9 |
| 13* | 3 | 2 |
| 14 | 11 | 10 |
| 15* | 2 | -1 |
| 16* | 2 | 2 |
| 17* | 2 | 1 |
| 18* | 2 | -1 |
| 19 | 11 | 12 |

**h87**

| | | |
|---|---|---|
| 0 | 15 | -12 |
| 1 | 11 | -8 |
| 2 | 13 | 12 |
| 3 | 10 | -13 |
| 4* | 3 | 1 |
| 5 | 7 | 7 |
| 6 | 10 | 8 |
| 7 | 13 | 13 |
| 8* | 3 | 2 |
| 9 | 11 | 9 |
| 10 | 15 | -14 |
| 11 | 8 | -8 |
| 12 | 4 | -2 |
| 13* | 3 | -4 |
| 14* | 2 | -1 |
| 15* | 2 | 3 |
| 16* | 2 | -1 |
| 17 | 10 | -11 |
| 18 | 6 | -4 |
| 19* | 2 | -3 |
| 20 | 2 | 3 |

**h97**

| | | |
|---|---|---|
| 1 | 11 | 9 |
| 2* | 3 | 2 |
| 3* | 3 | -3 |
| 4* | 3 | 6 |
| 5 | 25 | 26 |
| 6 | 17 | -18 |
| 7 | 39 | -45 |
| 8* | 2 | -2 |
| 9 | 18 | 16 |
| 10 | 14 | 14 |
| 11 | 8 | -9 |
| 12 | 8 | -8 |
| 13* | 2 | 2 |
| 14 | 10 | 13 |
| 15 | 4 | 6 |
| 16 | 6 | -8 |
| 17 | 8 | -8 |
| 18* | 2 | 5 |
| 19 | 10 | 10 |

**h,10,7**

| | | |
|---|---|---|
| 1 | 18 | 18 |
| 2* | 3 | 7 |
| 3* | 3 | -4 |
| 4* | 3 | 3 |
| 5 | 9 | 9 |
| 6* | 3 | 4 |
| 7 | 19 | 20 |
| 8 | 14 | -14 |
| 9 | 12 | -12 |
| 10* | 2 | 2 |
| 11* | 2 | 2 |
| 12 | 6 | -6 |
| 13* | 2 | -1 |
| 14* | 2 | -2 |
| 15 | 6 | -3 |

**h,11,7**

| | | |
|---|---|---|
| 1* | 3 | 2 |
| 2 | 12 | -12 |
| 3 | 12 | -12 |
| 4* | 3 | 4 |
| 5 | 25 | 27 |
| 6* | 3 | 4 |
| 7 | 25 | -29 |
| 8* | 2 | 2 |
| 9 | 11 | 11 |
| 10* | 2 | 2 |
| 11 | 5 | -6 |
| 12* | 2 | -2 |
| 13 | 4 | 3 |
| 14* | 2 | 2 |
| 15* | 2 | -2 |
| 16 | 6 | 8 |

**h,12,7**

| | | |
|---|---|---|
| 0 | 13 | 11 |
| 1 | 17 | -16 |
| 2* | 3 | -6 |
| 3 | 14 | 15 |
| 4 | 14 | 14 |
| 5* | 2 | 2 |
| 6 | 12 | -12 |
| 7* | 2 | -1 |
| 8* | 2 | 1 |
| 9 | 9 | 10 |
| 10* | 2 | -2 |
| 11 | 12 | -11 |
| 12* | 2 | -2 |
| 13 | 8 | 8 |
| 14* | 2 | 2 |
| 15 | 10 | 11 |

**h,13,7**

| | | |
|---|---|---|
| 1* | 2 | 5 |
| 2* | 2 | 2 |
| 3 | 11 | -10 |
| 4 | 6 | -8 |
| 5 | 8 | 8 |
| 6 | 5 | 7 |
| 7 | 8 | -10 |
| 8* | 2 | 5 |
| 9 | 5 | 6 |

**h,14,7**

| | | |
|---|---|---|
| 1 | 8 | -10 |
| 2* | 2 | -3 |
| 3 | 7 | 7 |
| 4* | 2 | 4 |
| 5* | 2 | -2 |
| 6 | 6 | -2 |
| 7* | 2 | 6 |
| 8* | 2 | -3 |
| 9 | 5 | 5 |

**h,15,7**

| | | |
|---|---|---|
| 4 | 7 | 10 |

calculated with the analytical *f* values of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955).

The structure was refined by further Fourier and difference Fourier syntheses and five least-squares cycles with the use of the Busing, Martin & Levy (1962) program *ORFLS*. Initially an overall temperature factor of 3·8 obtained by Wilson's (1942) method was applied; subsequently individual isotropic temperature factors for each atom were used. The value of *R* for observed reflexions dropped to 18·9%. At this stage 675 unobserved reflexions, estimated as $\frac{1}{3}$ $|F_{\min}|$ of the appropriate layer line, were included and a further four cycles with anisotropic temperature factors for all atoms (excluding hydrogen atoms) were computed. In the last cycle the average parameter shifts expressed as fractions of the estimated standard deviations were about 0·3 for *x* coordinates, 0·5 for *y* and *z* coordinates. The refinement was terminated at this stage for financial reasons;* further, it was considered that the molecule was sufficiently stabilized for the purposes of this study. The final *R* index for 2560 reflexions is 13·5%. Observed and calculated structure factors are listed in Table 1. The *f* values used in the least-squares calculations are those given by Hanson, Herman, Lea & Skillman (1964). Final atomic coordinates and thermal-motion parameters and their standard deviations are given in Table 2.

## Discussion

Fig. 1 shows the structure of the molecule and bond lengths and angles. Table 3 gives interatomic distances and angles with associated standard deviations which were calculated from the results of the last refinement cycle by the Busing–Levy program *ORFFE* (Busing, Martin & Levy, 1964). The quoted errors include allowance for errors in cell dimensions. The C–C bond lengths within the benzene rings are all between 1·38 and 1·43 Å; the average value for each benzene ring is 1·40 Å which is in good agreement with the values reported for benzene (1·397 Å, Stoicheff, 1954; 1·392 Å, Cox, Cruickshank & Smith, 1958; 1·394 Å, Sutton, 1965).

Table 3. *Bond lengths and angles*

The estimated standard deviations in the bond lengths are all 0·01 Å. Deviations in the bond angles (×10) are given in parentheses.

| Bond | Length |
|---|---|
| C(1)—C(2) | 1·39 Å |
| C(2)—C(3) | 1·38 |
| C(3)—C(4) | 1·42 |
| C(4)—C(5) | 1·40 |
| C(5)—C(6) | 1·40 |
| C(6)—C(1) | 1·40 |
| C(7)—C(8) | 1·43 |
| C(8)—C(9) | 1·39 |
| C(9)—C(10) | 1·39 |
| C(10)–C(11) | 1·41 |
| C(11)–C(12) | 1·40 |
| C(12)–C(7) | 1·39 |
| C(7)—S | 1·75 |
| C(4)—S | 1·75 |
| C(12)–N(1) | 1·41 |

Table 2. *Final atomic fractional coordinates* (×10⁴) *and thermal parameters* (×10⁴) *with estimated standard deviations*

Temperature factor $=\exp\{-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})\}$ with $\beta_{11}=2\pi^2a^{*2}U_{11}$, $\beta_{12}=2\pi^2a^*b^*U_{12}$, *etc.* The least-squares standard errors are given in parentheses.

| | x | y | z | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Cℓ | 4520 (1) | $0199_5$(2) | 3710 (3) | 23 ($0_4$) | 102 (2) | 176 (4) | 20 (1) | 4 (1) | 11 (2) |
| S | 2164 (1) | 0515 (1) | 0643 (2) | 28 ($0_4$) | 41 (1) | 107 (3) | − 6 ($0_5$) | −16 (1) | −13 (1) |
| N(1) | 2687 (2) | 1874 (3) | 2481 (6) | 17 (1) | 29 (2) | 107 (9) | − 1 (1) | − 0 (2) | − 9 (3) |
| N(2) | 4192 (2) | 3325 (4) | 3565 (7) | 19 (1) | 58 (3) | 122 (10) | − 5 (1) | 10 (3) | − 5 (4) |
| C(1) | 3855 (3) | 0261 (5) | 2893 (8) | 23 (2) | 58 (4) | 88 (12) | 7 (2) | 10 (3) | 14 (5) |
| C(2) | 1344 (4) | 4537 (5) | 2131 (9) | 27 (2) | 55 (4) | 92 (13) | − 5 (2) | −17 (4) | 11 (5) |
| C(3) | 1870 (4) | 4617 (5) | 1478 (9) | 34 (2) | 36 (3) | 138 (14) | − 1 (2) | −21 (4) | 1 (5) |
| C(4) | 2802 (3) | 0398 (4) | 1597 (8) | 24 (2) | 32 (3) | 88 (11) | − 1 (2) | 5 (3) | − 5 (4) |
| C(5) | 3020 (3) | 1111 (4) | 2379 (7) | 20 (1) | 42 (3) | 50 (10) | 3 (2) | 6 (3) | − 5 (4) |
| C(6) | 3548 (3) | 1045 (5) | 3073 (7) | 21 (1) | 52 (4) | 58 (11) | 5 (2) | 11 (3) | −11 (4) |
| C(7) | 1807 (3) | 1156 (5) | 1926 (8) | 21 (2) | 45 (3) | 102 (12) | − 2 (2) | −15 (3) | 15 (5) |
| C(8) | 1206 (3) | 1052 (5) | 2119 (11) | 23 (2) | 48 (4) | 232 (17) | − 3 (2) | −19 (4) | 27 (6) |
| C(9) | 0921 (4) | 1579 (6) | 3109 (13) | 22 (2) | 53 (5) | 281 (20) | 5 (2) | 4 (5) | 27 (8) |
| C(10) | 1214 (4) | 2194 (6) | 3942 (11) | 24 (2) | 53 (4) | 233 (18) | 9 (2) | 15 (5) | 20 (7) |
| C(11) | 1807 (3) | 2288 (5) | 3780 (9) | 22 (2) | 39 (3) | 145 (13) | 6 (2) | 8 (3) | 17 (5) |
| C(12) | 2097 (3) | 1782 (4) | 2747 (8) | 18 (1) | 36 (3) | 104 (12) | − $0_4$(2) | − 7 (3) | 21 (4) |
| C(13) | 2973 (3) | 2709 (4) | 2992 (8) | 20 (1) | 34 (3) | 91 (11) | − 4 (2) | 2 (3) | −17 (4) |
| C(14) | 3394 (3) | 3028 (4) | 1840 (8) | 26 (2) | 42 (3) | 80 (11) | −10 (2) | 1 (3) | − 6 (4) |
| C(15) | 3802 (3) | 3713 (5) | 2522 (9) | 27 (2) | 43 (3) | 112 (13) | − 8 (2) | − 3 (4) | 2 (5) |
| C(16) | 4662 (4) | 2836 (7) | 2845 (15) | 24 (2) | 83 (6) | 395 (26) | 5 (3) | 28 (6) | −62 (11) |
| C(17) | 4426 (4) | 4024 (7) | 4476 (11) | 35 (2) | 89 (6) | 161 (16) | −17 (3) | −15 (5) | −28 (8) |

---

*At a later date further funds became available and the anisotropic least-squares refinement was continued using $F_{\text{obs}}$ data only until the final *R* obtained was 11.4%. Bond lengths and angles calculated at the end of the final cycle were not significantly different from the values given in Table 3.

Table 3 (*cont.*)

| Bond | Length |
|---|---|
| C(5)—N(1) | 1·40 |
| N(1)—C(13) | 1·51 |
| C(13)-C(14) | 1·53 |
| C(14)-C(15) | 1·55 |
| C(15)-N(2) | 1·45 |
| N(2)—C(16) | 1·49 |
| N(2)—C(17) | 1·46 |
| Cl——C(1) | 1·74 |
| Cl——C(16) | 4·10 |
| Cl——Cl($\bar{1}$) | 3·34 |
| Cl——S | 6·24 |
| Cl——S(4) | 4·47 |
| Cl——S($\bar{4}$) | 6·26 |
| N(2)—S($\bar{3}$) | 5·37 |
| N(2)—S($\bar{2}$) | 5·45 |
| N(2)—Cl(3) | 4·71 |
| N(2)—Cl($\bar{3}$) | 5·28 |

| Angle | $\theta$ |
|---|---|
| C(6)—C(1)—C(2) | 124·1 (7) |
| C(1)—C(2)—C(3) | 117·0 (7) |
| C(2)—C(3)—C(4) | 121·7 (7) |
| C(3)—C(4)—C(5) | 119·3 (7) |
| C(4)—C(5)—C(6) | 120·3 (6) |
| C(5)—C(6)—C(1) | 117·5 (7) |
| C(12)-C(7)—C(8) | 119·4 (7) |
| C(7)—C(8)—C(9) | 119·5 (8) |
| C(8)—C(9)—C(10) | 120·9 (8) |
| C(9)—C(10)-C(11) | 119·9 (8) |
| C(10)-C(11)-C(12) | 119·8 (8) |
| C(11)-C(12)-C(7) | 120·4 (7) |
| C(4)—S——C(7) | 97·3 (3) |
| S——C(7)—C(12) | 120·9 (6) |
| C(7)—C(12)-N(1) | 116·9 (6) |
| C(12)-N(1)—C(5) | 118·4 (5) |
| N(1)—C(5)—C(4) | 118·1 (6) |
| C(5)—C(4)—S | 119·7 (5) |
| C(12)-N(1)—C(13) | 117·7 (5) |
| C(5)—N(1)—C(13) | 117·8 (5) |
| N(1)—C(13)-C(14) | 109·7 (5) |
| C(13)-C(14)-C(15) | 109·3 (6) |

Table 3 (*cont.*)

| Angle | $\theta$ |
|---|---|
| C(14)-C(15)-N(2) | 112·7 (6) |
| C(15)-N(2)—C(16) | 112·0 (8) |
| C(15)-N(2)—C(17) | 108·9 (7) |
| C(16)-N(2)—C(17) | 109·9 (7) |
| Cl——C(1)—C(2) | 118·7 (6) |
| Cl——C(1)—C(6) | 117·1 (6) |

The C–Cl bond length is 1·74 ± 0·01 Å; this is a typical C (aromatic)–Cl bond distance, although the value given by Sutton (1965) is 1·70 ± 0·01 Å. Palenik, Donohue & Trueblood (1968) tabulated twenty-six C–Cl bond distances reported between the years 1959 and 1968 in various aromatic molecules. Each distance involves a chlorine atom bonded to only one other atom. Of the bond lengths tabulated, four are equal to or greater than 1·76 Å, six have values between 1·71 and 1·72 Å, and sixteen have values between 1·73 and 1·75 Å. The average value of the twenty-six bonds reported is 1·737 ± 0·016 Å, which is in excellent agreement with the result reported above.

The S–C bonds are 1·75 ± 0·01 Å, which implies double-bond character of about 13%. This is close to other values in similar aromatic substances, *e.g.* thianthrene, 1·76 Å (Lynton & Cox, 1956); phenoxthionine, 1·75 Å (Hosoya, 1966); phenothiazine, 1·77 Å (Bell, Blount, Briscoe & Freeman, 1968). The C–S–C angle (97·3°) is much less than the C–N–C angle (118·4°). The implication of the difference in angles and the contraction of the S–C bond will be discussed in the next section.

The best planes for the two benzene rings were obtained by the method of Schomaker, Waser, Marsh & Bergman (1959), with the program *LSPLANE*, and are given by

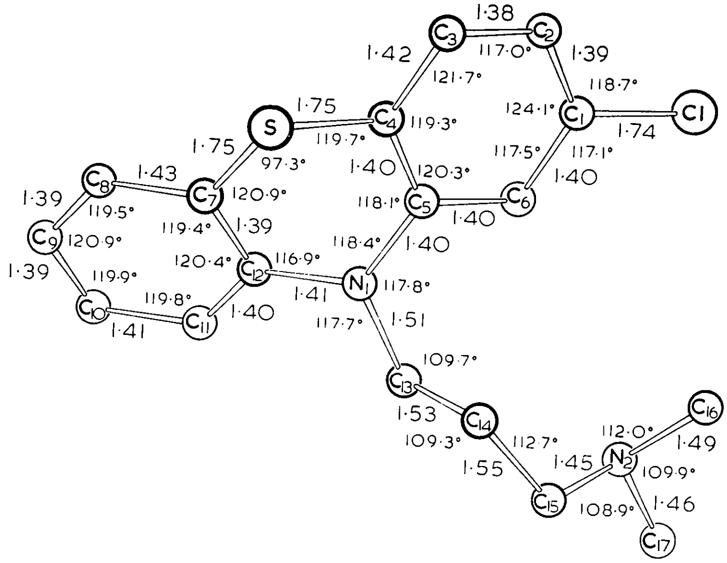


Fig. 1. Bond distances and angles.

$$-0{\cdot}4353x-0{\cdot}3576y+0{\cdot}8262z=-1{\cdot}8684 \text{ for } C(1)-C(6)\,,$$

$$0{\cdot}1603x-0{\cdot}6920y+0{\cdot}7039z=0{\cdot}7144 \text{ for } C(7)-C(12)\,.$$

The displacements of the atoms from these planes are given in Table 4. The deviations from planarity of the C atoms are not significant; the Cl atom is very close to the plane of the attached benzene ring. The fact that S is not in plane I, and N not in plane II may indicate that the steric effects of the 'tail' have slightly distorted the ring portion of the molecule so that it has no plane of symmetry through the S–N axis, as it could be expected to have in phenothiazine.*

Table 4. *Displacements of atoms from the best planes of the two benzene rings*

| Plane | Atom | Displacement |
|---|---|---|
| I C(1)–C(6) | C(1) | −0·011 Å |
| | C(2) | 0·004 |

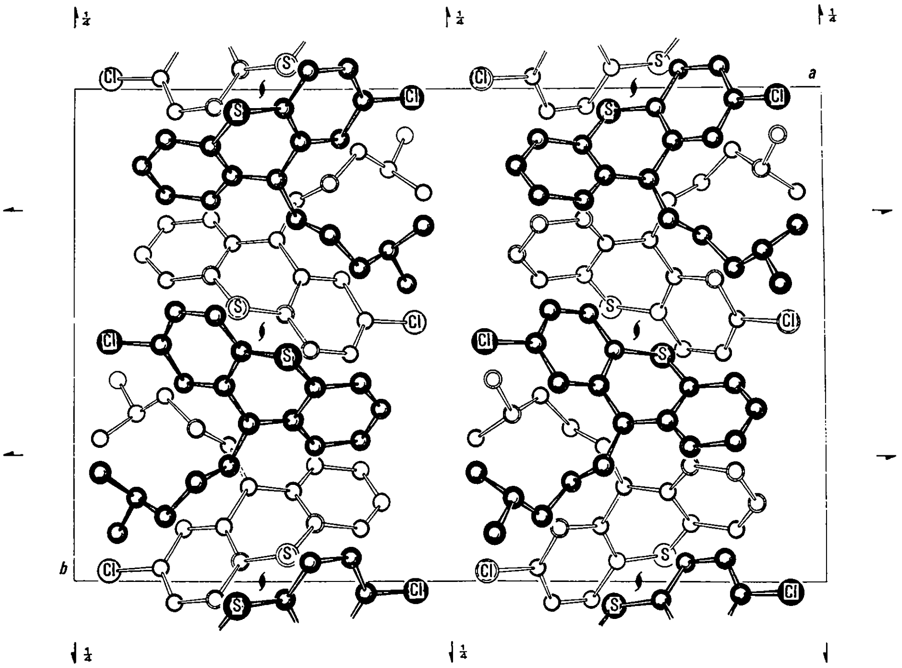


Fig. 2. The structure viewed down the *c* axis.

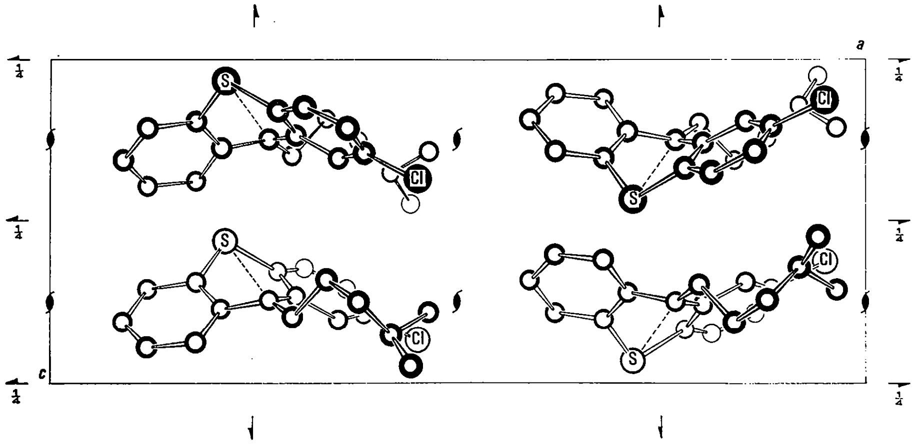


Fig. 3. Perspective drawing of half the contents of the unit cell (from 0 to *b*/2) viewed down the *b* axis. All molecules from 0 to *a*/2 are folded downwards about the S–N axis (dashed lines); all molecules from *a*/2 to *a* are folded upwards.

---

* This, however, is not the case. The author has just received a communication from Drs Bell, Blount, Briscoe & Freeman (1968) who have recently accurately determined the structure of phenothiazine. It is also not symmetrical.

Table 4 (*cont.*)

| Plane | Atom | Displacement |
|---|---|---|
| | C(3) | 0·001 |
| | C(4) | 0·003 |
| | C(5) | −0·011 |
| | C(6) | 0·014 |
| | S | −0·135 |
| | N(1) | −0·007 |
| | Cl | −0·035 |
| II C(7)–C(12) | C(7) | 0·001 |
| | C(8) | 0·010 |
| | C(9) | −0·009 |
| | C(10) | −0·004 |
| | C(11) | 0·015 |
| | C(12) | −0·014 |
| | S | −0·023 |
| | N(1) | −0·062 |

The dihedral angle between the two planes of the benzene rings is 139·4°, and is very close to that found by Hosoya (1966) for phenoxthionine. The folding of the molecule, the difference in the angles of the type C–N–C and C–S–C and the shortening of the C–S bond are characteristic of a number of heterocyclic compounds derived by replacing anthracene *meso*-CH groups by atoms *A* and *B*. It has been found that molecules are planar if both *A* and *B* are any of C, N or O, but folded if at least one of *A* and *B* is S, Se or Te. The X–S(Se,Te)–X angles which have been determined are in the range 93–100°. *e.g.* thianthrene (Lynton & Cox, 1956); phenothiazine (Cullinane & Rees, 1940; Wood, McCale & Williams, 1940; Bell, Blount, Briscoe & Freeman, 1968); phenoxthionine (Cullinane & Rees, 1940; Hosoya, 1966). According to Lynton & Cox (1956) and Hosoya (1963), this is explained by assuming the participation of *d* orbitals in the bonding of S and S–like atoms. The valence orbitals in atoms of the second period such as C, N and O are limited to 2*s* and 2*p* or hybrids of the two, but sulphur can be converted to the excited configuration $(3s)^2(3p)^3(3d)$. The folding of the molecule enables the sulphur atom to retain its 'natural' valency angle. The quantum mechanical treatment of Craig & Magnusson (1956) lends support to the above theory.

A few intermolecular distances are given at the end of Table 3. The only noteworthy distance is Cl–Cl($\bar{1}$), which is 3·34 Å implying that the effective van der Waals radius of chlorine is less than the ionic radius, 1·81 Å.

The molecular packing in the unit cell is given in Figs. 2 and 3. Four of the molecules are 'left-handed', while the other four are 'right-handed' enantiomorphs. Fig. 3, which represents half the contents of the unit cell from 0 to $b/2$, shows clearly that the molecules are arranged in layers of width $a/2$, with the axis of the fold alternately left and right in the $z$ direction.

Most of the calculations were done on an I.C.T. 1301 computer. The programs *ORFLS*, *ORFFE* and *LSPLANE* were carried out on an I.B.M. 360/40 computer.

Professor W. Schaffer of the University of Cape Town is thanked for his support and encouragement; appreciation is recorded to Mr M. H. Linck for his assistance, especially with the I.C.T. programs. The author is indebted to the Council of Scientific and Industrial Research for a computing grant, and gratefully acknowledges the invaluable assistance and skilled advice given by Dr G. Gafner, head of the crystallography division of the C.S.I.R.

## References

Bell, J. D., Blount, J. F., Briscoe, O. V. & Freeman, H. C. (1968). (Sydney). Private communication.

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). USAEC Report ORNL-TM-305, Oak Ridge National Laboratory, Tennessee.

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). USAEC Report ORNL-TM-306, Oak Ridge National Laboratory, Tennessee.

Buxton Hopkin, D. A. (1955). *Pharm. J.* **174**, 317.

*Colloque International Chlorpromazine* (1955). Paris: Doin et Cie.

Courvoisier, S., Fournel, J., Ducrot, R., Kolsky, M. & Koetschet, P. (1953). *Arch. Int. Pharmacodyn.* **92** (3–4), 305.

Cox, E. G., Cruickshank, D. W. J. & Smith, J. A. S. (1958) *Proc. Roy. Soc.* A**247**, 1.

Craig, D. P. & Magnusson, E. A. (1956). *J. Chem. Soc.* p. 4895.

Cullinane, N. M. & Rees, W. T. (1940). *Trans. Faraday Soc.* **36**, 507.

Delay, J. Deniker, P. & Harl, J. M. (1952). *Ann. Med. Psychol.* **2**, 111.

Falkenberg, G. & Ringertz, H. (1967). *Acta Cryst.* **23**, 1111.

Feil, D., Linck, M. H. & McDowell, J. J. H. (1965). *Nature, Lond.* **207**, 285.

Hanson, H. P., Herman, F., Lea, J. D. & Skillman, S. (1964). *Acta Cryst.* **17**, 1040.

Hosoya, S. (1963). *Acta Cryst.* **16**, 310.

Hosoya, S. (1966). *Acta Cryst.* **20**, 429.

Lynton, H. & Cox, E. G. (1956). *J. Chem. Soc.* p. 4886.

Palenik, G. H., Donohue, J. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 1139.

Schomaker, V., Waser, J., Marsh, R. E. & Bergman, G. (1959). *Acta Cryst.* **12**, 600.

Stoicheff, B. P. (1954). *Canad. J. Phys.* **32**, 339.

Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions*. Special Publication No. 18. London: The Chemical Society.

Takayanagi, I. (1964). *Drug. Res. (Arzneim-Forsch.)* **14**, 694.

Wilson, A. J. C. (1942). *Nature, Lond.* **150**, 151.

Wood, R. G., McCale, C. H. & Williams, G. (1941). *Phil. Mag.* **31**, 71.