Acta Cryst. (1968). B24, 1441

# Crystal and Molecular Structure of Cobalt(III) Tris(*N*,*N*-diethyldithiocarbamate)

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(*Received* 14 *November* 1967 *and in revised form* 28 *December* 1967)

Cobalt(III) tris(*N*,*N*-diethyldithiocarbamate) is monoclinic, space group $C2/c$ with four molecules in the unit cell, $a=14{\cdot}10$, $b=10{\cdot}26$, $c=17{\cdot}02$ Å, $\beta=110°8'$. The crystal structure has been determined by two-dimensional Patterson and Fourier syntheses and refined with three-dimensional data by anisotropic least-squares method, with a final agreement index for the observed reflexions $R_1=0{\cdot}062$. Each molecule is formed by three dithiocarbamic ligands coordinated to a cobalt atom through the sulphur atoms; except for the terminal methyl groups the molecule has nearly trigonal symmetry, with each $\ldots S_2CNC_2\ldots$ group approximately in a plane with the cobalt atom. Mean bond lengths: Co–S 2·258, S–C 1·704, C–N 1·319 Å. The $\ldots S_2C=NR_2$ form makes, as expected, an important contribution to the molecular structure. The intermolecular interactions are of the van der Waals type.

## Introduction

During recent years the amount of information available on the structures of dithiocarbamates has increased greatly; such information is important for the understanding of their biological activity and is also of theoretical interest in relation to the coordination geometry about the metal atom and the bond lengths and bond angles in dithiocarbamic ligands.

The crystal structure of cobalt(III) tris(*N*,*N*-diethyldithiocarbamate) was investigated as part of a research program on xanthates and dithiocarbamates developed in our laboratory with the object of gaining information on metal–sulphur bonds and on the structures of xanthic and dithiocarbamic ligands.

A preliminary study (optical and morphological data, X-ray crystallography and two-dimensional structure analysis) on this compound was published by Merlino & Troysi (1966). Accurate data on bond lengths and bond angles, however, are still lacking.

## Experimental

The crystals of $Co[S_2CN(C_2H_5)_2]_3$ are black and tabular (001). Optical and morphological data can be found in an earlier paper (Merlino & Troysi, 1966).

*Crystal data*

Cobalt(III) tris(*N*,*N*-diethyldithiocarbamate)
Co(III) $[S_2CN(C_2H_5)_2]_3$, M.W. 503·92
Crystal class: monoclinic prismatic
$a=14{\cdot}10\pm0{\cdot}02$, $b=10{\cdot}26\pm0{\cdot}02$, $c=17{\cdot}02\pm0{\cdot}03$ Å, $\beta=110°8'\pm15'$.
Unit-cell volume $U=2311{\cdot}8$ Å$^3$, $Z=4$, molecular symmetry $C_2$–2, $D_m=1{\cdot}44$ (determined with a Westphal type balance), $D_c=1{\cdot}448$ g.cm$^{-3}$, $F(000)=1056$.
$\mu=12{\cdot}9$ cm$^{-1}$ (Mo $K\alpha$).

The systematic absences ($hkl$ with $h+k=2n+1$, $h0l$ with $h=2n+1$ and $l=2n+1$, $0k0$ with $k=2n+1$) correspond to space groups $Cc$ and $C2/c$. The use of intensity statistics (Howells, Phillips & Rogers, 1950) and the absence of a detectable piezo-electric effect led to the conclusion that the space group is probably $C2/c$; this choice was subsequently confirmed by structure analysis.

The three-dimensional intensity data were recorded with zirconium-filtered Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å), by means of Buerger precession photographs, with the multiple-exposure technique and integration process. A crystal of the compound was reduced to nearly spherical shape with a mean diameter of 0·86 mm. ($\mu R=0{\cdot}55$ for Mo $K\alpha$ radiation). Six layers with [010] as precessing axis ($k=0$ through 5) and four layers with [100] as precessing axis ($h=0$ through 3) were taken.

A total of 1089 independent reflexions were observed. The intensities, measured with a Nonius microdensitometer, ranged from 1 to 2400 and were processed by a program written by Catani & Zanazzi (1965).

Corrections were made for Lorentz and polarization factors, and for the absorption factor by the three-constants formula proposed by Palm (1964) for spherical crystals.

## Determination of the structure

The structure (Merlino & Troysi, 1966) was determined with two-dimensional data (reflexions $h0l$ and $0kl$) by Patterson and Fourier methods and refined by two-dimensional difference syntheses to a reliability index $R_1=\Sigma\,||F_o|-|F_c||/\Sigma\,|F_o|=0{\cdot}17$. Among the atoms of the asymmetric unit, the cobalt atom, one nitrogen atom and one carbon atom are in special positions 4($e$) along the twofold axis; all the other atoms are in general positions 8($f$).

## Refinement

The atomic coordinates obtained in the preceding phase have been refined by a full-matrix least-squares program (Busing, Martin & Levy, 1962) with individual temperature factors and constant weights, including also among the refined parameters the ten scale factors. After the fourth cycle ($R_1=0{\cdot}16$) new positions for two carbon atoms [C(6) and C(7)], incorrectly located in the preceding phase, were determined by a three-dimensional Fourier difference synthesis, calculated with a program written by Domenicano & Giucci. Two further least-squares refinement cycles with individual isotropic temperature factors reduced the reliability index from $R_1=0{\cdot}16$ to $R_1=0{\cdot}096$.

At this point, before anisotropic refinement, the 002 and 112 reflexions were rejected because they had anomalously large discrepancies (the first is partially trapped by the beam stop and the second is on the limit of the blind region). Anisotropic temperature factors were then introduced in the form

$$\exp\{-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})\}.$$

Two further least-squares cycles reduced the reliability index to $R_1=0{\cdot}062$, whereas $R_2=[\sum w||F_o|-|F_c||^2/\sum w|F_o|^2]^{1/2}$ was 0·074. In the last four refinement

Table 1. *Observed and calculated structure factors*

Reflexions for which *FO* is listed as 0 were not recorded. Those marked with an asterisk were given zero weight.

| H | K | L | FO | FC | H | K | L | FO | FC | H | K | L | FO | FC | H | K | L | FO | FC | H | K | L | FO | FC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 137.5 | 167.9 | 4 | 0 | 18 | 14.1 | -20.9 | -11 | 1 | 7 | 22.1 | -21.3 | -5 | 1 | 22 | 20.3 | -21.4 | -2 | 2 | 9 | 81.1 | 82.0 |
| 4 | 0 | 0 | 86.1 | -78.5 | -4 | 0 | 18 | 29.0 | -28.7 | -17 | 1 | 7 | 23.0 | -24.1 | -9 | 1 | 22 | 18.9 | 20.7 | -4 | 2 | 9 | 42.8 | 49.5 |
| 8 | 0 | 0 | 82.7 | 87.8 | -8 | 0 | 18 | 28.4 | -24.9 | -19 | 1 | 7 | 17.1 | -17.9 | -3 | 1 | 23 | 15.4 | -16.4 | 6 | 2 | 9 | 51.7 | 54.2 |
| 10 | 0 | 0 | 53.0 | 48.9 | -10 | 0 | 18 | 56.9 | -60.6 | 1 | 1 | 8 | 115.4 | -108.7 | -5 | 1 | 23 | 19.2 | -22.0 | -6 | 2 | 9 | 21.6 | -22.9 |
| 14 | 0 | 0 | 18.6 | 18.1 | -12 | 0 | 18 | 39.6 | -45.9 | -3 | 1 | 8 | 34.6 | 33.9 | -7 | 1 | 23 | 16.2 | -16.3 | -8 | 2 | 9 | 41.8 | 41.5 |
| 16 | 0 | 0 | 19.6 | 19.5 | 2 | 0 | 20 | 30.7 | 32.6 | 5 | 1 | 8 | 28.0 | 28.0 | 2 | 2 | 0 | 0. | -250.2 | 10 | 2 | 9 | 29.3 | -29.1 |
| 0 | 0 | 2* | 137.5 | -241.1 | -2 | 0 | 20 | 13.7 | 15.5 | 9 | 1 | 8 | 29.3 | -31.0 | 4 | 2 | 0 | 122.6 | -123.1 | -12 | 2 | 9 | 16.1 | -15.1 |
| 2 | 0 | 2 | 175.0 | -174.8 | -4 | 0 | 20 | 14.6 | 15.7 | 11 | 1 | 8 | 24.5 | -26.5 | 6 | 2 | 0 | 34.0 | -35.0 | 0 | 2 | 10 | 17.5 | -14.2 |
| -2 | 0 | 2 | 310.7 | -375.7 | -10 | 0 | 20 | 17.3 | 18.9 | -11 | 1 | 8 | 27.6 | -27.5 | 8 | 2 | 0 | 17.0 | 15.2 | 2 | 2 | 10 | 23.1 | 10.6 |
| 4 | 0 | 2 | 109.7 | 113.3 | -12 | 0 | 20 | 34.5 | 38.9 | -1 | 1 | 9 | 100.8 | 102.3 | 10 | 2 | 0 | 23.7 | 22.8 | -2 | 2 | 10 | 44.6 | 42.3 |
| -6 | 0 | 2 | 44.1 | -45.7 | -14 | 0 | 20 | 17.1 | 15.8 | 3 | 1 | 9 | 24.3 | -21.2 | 12 | 2 | 0 | 23.3 | -21.7 | 4 | 2 | 10 | 18.0 | 17.6 |
| 8 | 0 | 2 | 85.2 | -86.7 | 0 | 0 | 22 | 20.9 | -21.5 | -3 | 1 | 9 | 100.0 | 109.7 | 14 | 2 | 0 | 17.3 | -17.1 | -4 | 2 | 10 | 68.3 | 72.1 |
| -8 | 0 | 2 | 158.8 | -169.5 | -4 | 0 | 22 | 26.3 | -23.4 | -5 | 1 | 9 | 37.7 | 39.3 | 16 | 2 | 0 | 16.1 | -19.0 | -6 | 2 | 10 | 102.7 | 105.3 |
| 10 | 0 | 2 | 62.1 | -62.1 | -6 | 0 | 22 | 20.3 | -20.5 | 7 | 1 | 9 | 25.7 | 23.4 | 0 | 2 | 1 | 89.2 | 101.0 | 8 | 2 | 10 | 30.2 | 30.9 |
| -10 | 0 | 2 | 93.2 | -91.2 | 1 | 1 | 0 | 0. | -3.0 | -7 | 1 | 9 | 84.2 | 81.3 | 2 | 2 | 1 | 75.5 | -77.1 | -8 | 2 | 10 | 70.7 | 73.0 |
| 12 | 0 | 2 | 51.6 | -50.9 | 3 | 1 | 0 | 118.0 | -120.2 | -9 | 1 | 9 | 87.7 | 86.9 | -2 | 2 | 1 | 0. | 109.5 | 10 | 2 | 10 | 20.2 | 20.8 |
| 14 | 0 | 2 | 28.9 | -26.1 | 5 | 1 | 0 | 61.1 | 61.2 | 11 | 1 | 9 | 22.5 | 20.6 | 4 | 2 | 1 | 18.8 | -16.5 | -10 | 2 | 10 | 28.2 | 28.7 |
| -14 | 0 | 2 | 25.6 | 22.5 | 7 | 1 | 0 | 24.3 | 21.6 | -11 | 1 | 9 | 27.8 | 26.6 | -4 | 2 | 1 | 48.0 | -42.0 | -12 | 2 | 10 | 27.4 | 27.7 |
| 0 | 0 | 4 | 228.6 | 252.7 | 9 | 1 | 0 | 29.3 | -30.2 | 13 | 1 | 9 | 21.8 | 19.8 | 6 | 2 | 1 | 46.7 | 44.7 | 0 | 2 | 11 | 44.1 | 43.7 |
| 2 | 0 | 4 | 18.8 | 22.1 | 11 | 1 | 0 | 46.4 | -47.3 | 15 | 1 | 9 | 10.4 | 10.4 | -6 | 2 | 1 | 88.4 | 83.2 | 2 | 2 | 11 | 52.4 | 52.6 |
| -2 | 0 | 4 | 135.7 | 138.6 | 15 | 1 | 0 | 26.7 | 22.9 | 1 | 1 | 10 | 55.4 | 52.3 | 8 | 2 | 1 | 32.7 | 37.0 | -2 | 2 | 11 | 20.0 | -21.7 |
| -4 | 0 | 4 | 139.2 | -122.4 | 1 | 1 | 1 | 0. | 132.7 | -1 | 1 | 10 | 36.5 | 34.8 | -8 | 2 | 1 | 29.1 | 28.8 | 4 | 2 | 11 | 42.0 | -40.4 |
| 6 | 0 | 4 | 97.4 | 100.1 | -1 | 1 | 1 | 0. | 257.2 | 3 | 1 | 10 | 58.1 | 62.9 | 10 | 2 | 1 | 50.0 | -51.4 | -4 | 2 | 11 | 34.2 | -34.6 |
| -6 | 0 | 4 | 27.1 | -26.8 | 3 | 1 | 1 | 45.8 | -43.7 | -3 | 1 | 10 | 50.2 | -48.6 | -10 | 2 | 1 | 16.3 | -15.7 | 6 | 2 | 11 | 42.5 | -43.5 |
| 8 | 0 | 4 | 105.5 | 115.0 | -3 | 1 | 1 | 112.9 | 111.6 | 5 | 1 | 10 | 57.6 | -61.7 | 12 | 2 | 1 | 18.3 | -18.0 | -10 | 2 | 11 | 35.9 | -37.8 |
| -8 | 0 | 4 | 156.6 | 164.6 | 5 | 1 | 1 | 27.4 | -33.1 | -7 | 1 | 10 | 26.2 | 24.3 | -12 | 2 | 1 | 42.4 | -40.5 | 0 | 2 | 12 | 20.6 | -19.5 |
| 10 | 0 | 4 | 81.4 | 77.2 | -5 | 1 | 1 | 86.3 | 79.8 | 9 | 1 | 10 | 25.9 | 29.3 | 14 | 2 | 1 | 27.3 | 26.3 | 2 | 2 | 12 | 52.8 | -52.5 |
| -10 | 0 | 4 | 154.3 | 168.4 | 7 | 1 | 1 | 26.0 | 26.2 | -9 | 1 | 10 | 24.5 | -20.7 | 16 | 2 | 1 | 16.2 | 13.1 | -2 | 2 | 12 | 19.9 | 18.0 |
| 12 | 0 | 4 | 26.0 | 23.5 | -7 | 1 | 1 | 96.4 | 98.3 | 1 | 1 | 11 | 28.3 | 24.5 | -16 | 2 | 1 | 19.9 | 18.5 | 4 | 2 | 12 | 46.2 | -40.7 |
| -12 | 0 | 4 | 39.5 | 40.2 | 9 | 1 | 1 | 36.1 | 39.3 | -1 | 1 | 11 | 15.5 | -17.4 | 0 | 2 | 2 | 154.1 | 136.4 | 6 | 2 | 12 | 45.9 | -47.5 |
| 0 | 0 | 6 | 260.4 | -307.9 | -9 | 1 | 1 | 43.2 | 44.3 | -3 | 1 | 11 | 22.4 | -21.3 | 2 | 2 | 2 | 34.4 | -28.3 | -6 | 2 | 12 | 28.1 | -27.2 |
| 2 | 0 | 6 | 128.4 | -133.5 | 13 | 1 | 1 | 23.7 | 25.8 | 5 | 1 | 11 | 51.8 | -50.8 | -2 | 2 | 2 | 51.1 | 44.1 | 8 | 2 | 12 | 18.3 | -18.6 |
| -2 | 0 | 6 | 93.1 | -104.2 | 15 | 1 | 1 | 22.3 | 23.7 | -5 | 1 | 11 | 24.3 | -22.3 | 4 | 2 | 2 | 41.9 | 44.6 | -8 | 2 | 12 | 40.9 | -41.4 |
| 4 | 0 | 6 | 62.1 | -55.2 | 17 | 1 | 1 | 16.0 | 15.7 | 7 | 1 | 11 | 35.4 | -35.9 | -4 | 2 | 2 | 144.6 | 159.1 | -10 | 2 | 12 | 21.6 | -22.2 |
| 6 | 0 | 6 | 62.1 | -59.9 | 1 | 1 | 2* | 75.8 | 153.7 | -7 | 1 | 11 | 22.8 | -24.1 | 6 | 2 | 2 | 21.8 | 24.5 | -12 | 2 | 12 | 32.6 | -32.0 |
| -6 | 0 | 6 | 61.6 | 58.2 | -1 | 1 | 2 | 19.8 | -13.7 | 9 | 1 | 11 | 27.5 | -27.3 | -6 | 2 | 2 | 133.2 | 140.2 | -14 | 2 | 12 | 46.1 | -49.0 |
| 8 | 0 | 6 | 72.5 | -69.7 | 3 | 1 | 2 | 23.8 | 21.5 | -9 | 1 | 11 | 97.2 | -96.8 | -8 | 2 | 2 | 59.0 | 60.3 | -16 | 2 | 12 | 23.4 | -22.3 |
| -8 | 0 | 6 | 40.4 | -39.8 | -3 | 1 | 2 | 16.9 | 6.1 | 11 | 1 | 11 | 19.9 | -19.0 | 10 | 2 | 2 | 32.1 | 33.4 | 0 | 2 | 13 | 47.3 | -49.4 |
| 10 | 0 | 6 | 22.5 | -22.6 | 5 | 1 | 2 | 43.9 | 35.8 | -11 | 1 | 11 | 82.0 | -83.6 | -10 | 2 | 2 | 24.6 | 27.8 | -2 | 2 | 13 | 29.4 | 29.8 |
| -10 | 0 | 6 | 88.4 | -89.8 | -5 | 1 | 2 | 36.8 | 37.3 | 13 | 1 | 11 | 15.8 | -10.4 | 12 | 2 | 2 | 36.3 | 33.7 | 4 | 2 | 13 | 41.2 | 37.7 |
| -12 | 0 | 6 | 49.0 | -48.2 | 7 | 1 | 2 | 60.3 | -61.0 | -13 | 1 | 11 | 21.6 | -19.8 | 14 | 2 | 2 | 31.9 | 32.5 | -4 | 2 | 13 | 41.8 | 42.3 |
| -14 | 0 | 6 | 17.8 | -17.6 | -7 | 1 | 2 | 45.9 | -44.9 | 1 | 1 | 12 | 83.4 | -83.6 | 16 | 2 | 2 | 19.1 | 18.4 | 6 | 2 | 13 | 25.6 | 27.6 |
| -16 | 0 | 6 | 24.7 | -22.1 | 9 | 1 | 2 | 22.3 | 24.5 | -1 | 1 | 12 | 23.8 | -25.8 | 2 | 2 | 3 | 74.3 | 65.1 | -6 | 2 | 13 | 24.2 | -18.8 |
| -18 | 0 | 6 | 27.3 | -23.5 | 11 | 1 | 2 | 42.7 | 45.4 | 3 | 1 | 12 | 27.9 | 28.3 | -2 | 2 | 3 | 31.0 | -31.8 | -8 | 2 | 13 | 28.3 | -29.5 |
| 0 | 0 | 8 | 184.1 | 201.0 | -11 | 1 | 2 | 39.5 | 39.3 | -3 | 1 | 12 | 24.6 | 26.7 | -4 | 2 | 3 | 59.8 | -52.7 | 10 | 2 | 13 | 15.7 | -14.0 |
| 2 | 0 | 8 | 42.7 | 42.9 | -13 | 1 | 2 | 20.7 | 21.4 | 5 | 1 | 12 | 26.0 | 26.7 | 6 | 2 | 3 | 63.0 | -62.0 | 0 | 2 | 14 | 54.2 | 54.5 |
| -2 | 0 | 8 | 314.5 | 330.8 | 15 | 1 | 2 | 15.8 | -13.7 | -7 | 1 | 12 | 22.0 | -21.8 | 8 | 2 | 3 | 20.6 | -21.2 | 2 | 2 | 14 | 43.3 | 43.5 |
| 4 | 0 | 8 | 37.8 | -34.7 | 1 | 1 | 3 | 55.4 | 43.3 | 9 | 1 | 12 | 16.8 | -16.9 | -8 | 2 | 3 | 74.7 | -76.3 | -2 | 2 | 14 | 37.3 | 37.2 |
| -4 | 0 | 8 | 27.6 | 26.5 | -1 | 1 | 3 | 81.4 | -84.5 | -9 | 1 | 12 | 32.5 | -33.1 | 10 | 2 | 3 | 17.6 | 16.8 | 4 | 2 | 14 | 43.6 | 38.8 |
| 6 | 0 | 8 | 21.9 | 21.6 | 3 | 1 | 3 | 74.2 | 81.4 | -11 | 1 | 12 | 24.6 | 22.3 | -12 | 2 | 3 | 19.7 | 23.1 | -4 | 2 | 14 | 29.0 | 25.1 |
| -6 | 0 | 8 | 21.1 | -18.7 | -3 | 1 | 3 | 140.2 | -121.9 | 1 | 1 | 13 | 48.8 | 41.4 | -16 | 2 | 3 | 16.7 | -14.7 | -6 | 2 | 14 | 33.8 | 35.5 |
| 8 | 0 | 8 | 32.7 | 29.9 | -5 | 1 | 3 | 34.1 | -28.9 | -1 | 1 | 13 | 36.6 | 37.6 | 0 | 2 | 4 | 91.0 | 94.4 | -14 | 2 | 14 | 18.0 | 22.2 |
| -8 | 0 | 8 | 90.0 | 91.1 | 7 | 1 | 3 | 78.9 | -81.3 | 3 | 1 | 13 | 49.1 | 48.3 | 2 | 2 | 4 | 75.6 | -73.0 | -16 | 2 | 14 | 26.8 | 28.3 |
| -10 | 0 | 8 | 69.9 | 73.2 | -7 | 1 | 3 | 107.0 | -105.9 | -3 | 1 | 13 | 47.2 | 45.8 | -2 | 2 | 4 | 86.1 | 76.3 | 2 | 2 | 15 | 21.4 | 18.0 |
| 12 | 0 | 8 | 18.8 | 17.3 | 9 | 1 | 3 | 64.3 | -65.2 | 5 | 1 | 13 | 37.9 | 39.5 | 4 | 2 | 4 | 117.5 | -115.0 | -2 | 2 | 15 | 17.6 | -15.9 |
| 14 | 0 | 8 | 23.6 | 18.6 | -9 | 1 | 3 | 124.8 | -131.4 | -5 | 1 | 13 | 16.7 | -20.3 | -4 | 2 | 4 | 80.1 | -76.8 | 4 | 2 | 15 | 26.3 | -23.7 |
| -14 | 0 | 8 | 25.8 | -26.6 | 11 | 1 | 3 | 48.9 | -50.7 | 7 | 1 | 13 | 23.7 | 21.3 | 6 | 2 | 4 | 76.0 | -79.1 | -4 | 2 | 15 | 47.2 | -49.5 |
| -18 | 0 | 8 | 24.2 | 20.7 | -11 | 1 | 3 | 54.1 | -54.1 | -7 | 1 | 13 | 18.0 | -19.7 | -6 | 2 | 4 | 107.4 | -108.2 | -8 | 2 | 15 | 22.0 | 21.6 |
| 0 | 0 | 10 | 106.5 | -102.5 | 13 | 1 | 3 | 21.3 | -25.1 | -9 | 1 | 13 | 21.2 | 20.1 | 8 | 2 | 4 | 55.5 | -56.7 | -2 | 2 | 16 | 30.0 | -29.6 |
| 2 | 0 | 10 | 30.7 | 29.2 | 15 | 1 | 3 | 11.1 | -12.7 | -11 | 1 | 13 | 45.7 | 48.0 | -8 | 2 | 4 | 56.5 | -60.7 | -4 | 2 | 16 | 53.1 | -54.5 |
| -2 | 0 | 10 | 112.6 | -124.9 | 1 | 1 | 4 | 93.2 | -91.6 | -13 | 1 | 13 | 29.3 | 29.4 | 10 | 2 | 4 | 32.3 | -35.4 | -6 | 2 | 16 | 50.3 | -52.0 |
| -4 | 0 | 10 | 73.0 | -64.7 | -1 | 1 | 4 | 96.5 | -103.3 | -17 | 1 | 13 | 23.2 | 21.5 | -10 | 2 | 4 | 37.6 | -39.9 | -8 | 2 | 16 | 44.7 | -45.2 |
| 6 | 0 | 10 | 45.8 | -44.3 | 3 | 1 | 4 | 77.8 | -80.9 | -19 | 1 | 13 | 15.7 | 15.7 | 12 | 2 | 4 | 28.4 | -24.5 | 0 | 2 | 17 | 37.2 | -37.0 |
| 8 | 0 | 10 | 27.9 | -26.3 | -3 | 1 | 4 | 14.9 | 13.4 | 1 | 1 | 14 | 34.4 | 32.8 | -12 | 2 | 4 | 58.8 | -59.5 | -4 | 2 | 17 | 39.4 | 38.8 |
| -8 | 0 | 10 | 100.7 | -103.1 | 5 | 1 | 4 | 86.4 | 91.5 | -1 | 1 | 14 | 27.9 | 26.1 | -14 | 2 | 4 | 32.7 | -33.3 | 4 | 2 | 18 | 16.4 | 7.3 |
| 10 | 0 | 10 | 41.7 | -36.5 | 9 | 1 | 4 | 22.4 | -22.9 | -3 | 1 | 14 | 33.6 | -32.2 | -16 | 2 | 4 | 16.7 | -13.8 | -6 | 2 | 18 | 33.9 | 35.1 |
| -10 | 0 | 10 | 126.2 | -138.7 | 11 | 1 | 4 | 22.6 | -23.1 | 5 | 1 | 14 | 33.1 | -30.5 | 2 | 2 | 5 | 37.6 | -31.4 | -8 | 2 | 18 | 34.9 | 36.6 |
| 12 | 0 | 10 | 34.4 | -30.1 | -11 | 1 | 4 | 34.0 | -29.5 | -5 | 1 | 14 | 19.2 | -20.9 | -2 | 2 | 5 | 41.8 | 38.8 | -10 | 2 | 18 | 27.8 | 25.9 |
| -12 | 0 | 10 | 32.1 | -29.9 | 1 | 1 | 5 | 99.6 | 96.9 | -7 | 1 | 14 | 31.8 | 32.9 | 4 | 2 | 5 | 34.4 | 32.7 | -12 | 2 | 18 | 18.2 | 17.4 |
| 0 | 0 | 12 | 54.2 | 53.5 | -1 | 1 | 5 | 102.9 | 100.6 | 9 | 1 | 14 | 15.9 | 18.3 | 6 | 2 | 5 | 77.9 | 81.2 | -14 | 2 | 18 | 15.6 | 17.1 |
| 2 | 0 | 12 | 29.3 | 29.1 | 3 | 1 | 5 | 67.2 | 62.3 | 1 | 1 | 15 | 30.6 | -28.7 | 8 | 2 | 5 | 16.2 | 18.5 | 0 | 2 | 19 | 31.5 | 29.6 |
| -2 | 0 | 12 | 81.0 | 85.3 | -3 | 1 | 5 | 51.4 | -43.7 | -1 | 1 | 15 | 63.3 | -63.7 | 10 | 2 | 5 | 25.2 | -25.5 | -4 | 2 | 19 | 21.5 | -22.1 |
| 4 | 0 | 12 | 65.6 | 63.1 | 5 | 1 | 5 | 70.6 | 70.8 | -3 | 1 | 15 | 65.6 | -66.3 | -16 | 2 | 5 | 16.7 | 16.7 | -8 | 2 | 19 | 19.0 | 19.7 |
| 6 | 0 | 12 | 33.7 | 33.4 | -5 | 1 | 5 | 50.9 | -50.7 | -5 | 1 | 15 | 40.8 | -40.5 | 0 | 2 | 6 | 31.9 | 31.8 | -12 | 2 | 19 | 18.0 | -16.8 |
| -6 | 0 | 12 | 54.7 | -54.2 | 7 | 1 | 5 | 81.5 | 86.0 | -19 | 1 | 15 | 16.6 | -17.4 | 2 | 2 | 6 | 120.9 | 119.8 | 0 | 2 | 20 | 18.2 | -17.6 |
| 8 | 0 | 12 | 40.4 | 37.3 | 9 | 1 | 5 | 54.1 | 54.7 | 1 | 1 | 16 | 32.8 | -31.5 | -2 | 2 | 6 | 79.9 | 78.0 | 2 | 2 | 20 | 15.4 | -12.2 |
| -8 | 0 | 12 | 22.1 | 19.8 | -9 | 1 | 5 | 86.3 | 82.1 | -1 | 1 | 16 | 27.3 | -24.7 | 4 | 2 | 6 | 107.5 | 111.2 | 4 | 2 | 20 | 23.9 | -17.9 |
| 10 | 0 | 12 | 15.9 | 17.5 | -11 | 1 | 5 | 68.2 | 67.5 | -3 | 1 | 16 | 24.0 | 22.8 | -4 | 2 | 6 | 38.2 | 29.7 | -14 | 2 | 20 | 20.2 | -18.9 |
| -10 | 0 | 12 | 111.0 | 119.3 | -13 | 1 | 5 | 34.5 | 37.2 | -5 | 1 | 16 | 25.4 | 26.5 | 6 | 2 | 6 | 73.1 | 73.6 | -16 | 2 | 20 | 22.5 | -20.0 |
| 12 | 0 | 12 | 15.2 | 12.4 | -15 | 1 | 5 | 21.8 | 22.6 | -9 | 1 | 16 | 24.7 | -22.9 | -6 | 2 | 6 | 25.4 | 25.5 | 4 | 2 | 21 | 19.6 | 11.7 |
| -12 | 0 | 12 | 78.4 | 78.7 | -17 | 1 | 5 | 17.3 | 17.8 | -1 | 1 | 17 | 17.2 | 11.2 | -12 | 2 | 6 | 23.4 | 28.8 | -4 | 2 | 21 | 26.9 | 26.6 |
| -18 | 0 | 12 | 26.2 | 18.0 | 1 | 1 | 6 | 56.2 | 51.3 | -3 | 1 | 17 | 37.3 | 36.4 | -14 | 2 | 6 | 43.4 | 43.9 | -8 | 2 | 21 | 25.2 | -21.7 |
| 0 | 0 | 14 | 78.1 | -78.3 | -1 | 1 | 6 | 22.5 | 16.4 | -5 | 1 | 17 | 32.0 | 35.1 | -16 | 2 | 6 | 31.8 | 32.3 | 0 | 2 | 22 | 9.2 | 12.7 |
| 2 | 0 | 14 | 61.8 | -61.4 | 3 | 1 | 6 | 40.5 | 38.5 | -7 | 1 | 17 | 27.1 | 28.0 | 0 | 2 | 7 | 40.6 | -38.4 | -2 | 2 | 22 | 23.1 | 20.5 |
| -2 | 0 | 14 | 89.2 | -90.5 | -3 | 1 | 6 | 26.9 | 28.1 | -9 | 1 | 17 | 44.7 | 44.7 | 2 | 2 | 7 | 20.8 | 18.4 | -4 | 2 | 22 | 20.4 | 19.0 |
| 4 | 0 | 14 | 22.1 | -22.4 | 5 | 1 | 6 | 23.6 | -24.9 | -11 | 1 | 17 | 38.1 | 36.2 | -2 | 2 | 7 | 60.4 | -60.6 | -6 | 2 | 23 | 19.9 | -17.8 |
| -4 | 0 | 14 | 39.9 | -43.6 | 7 | 1 | 6 | 44.9 | -46.4 | 1 | 1 | 18 | 33.5 | 33.4 | 6 | 2 | 7 | 47.8 | -48.1 | -6 | 2 | 24 | 17.7 | -18.9 |
| 6 | 0 | 14 | 19.4 | -21.5 | 9 | 1 | 6 | 23.0 | 27.1 | -1 | 1 | 18 | 22.1 | 23.3 | 10 | 2 | 7 | 34.2 | 37.0 | 1 | 3 | 0 | 67.4 | -69.6 |
| -6 | 0 | 14 | 35.6 | 39.5 | -9 | 1 | 6 | 22.1 | 21.8 | -5 | 1 | 18 | 15.3 | -19.4 | -12 | 2 | 7 | 24.2 | 26.6 | 3 | 3 | 0 | 32.1 | -23.3 |
| -10 | 0 | 14 | 35.0 | -36.1 | 11 | 1 | 6 | 33.2 | 35.9 | 3 | 1 | 19 | 22.5 | -21.2 | 14 | 2 | 7 | 16.5 | -13.3 | 5 | 3 | 0 | 31.5 | 30.2 |
| -12 | 0 | 14 | 35.8 | -34.0 | -11 | 1 | 6 | 21.4 | 16.5 | 5 | 1 | 19 | 15.9 | -16.9 | -16 | 2 | 7 | 17.6 | -17.1 | 1 | 3 | 1 | 60.1 | 48.6 |
| -18 | 0 | 14 | 28.7 | -24.9 | 15 | 1 | 6 | 13.2 | -12.3 | -9 | 1 | 19 | 19.3 | -19.7 | 0 | 2 | 8 | 95.8 | -97.2 | -1 | 3 | 1 | 49.4 | 45.7 |
| 0 | 0 | 16 | 36.3 | 34.6 | 1 | 1 | 7 | 145.3 | -142.1 | -11 | 1 | 19 | 41.5 | -41.3 | 2 | 2 | 8 | 48.4 | -48.0 | 3 | 3 | 1 | 149.3 | -143.3 |
| -2 | 0 | 16 | 71.1 | 73.8 | -1 | 1 | 7 | 170.8 | -179.7 | -13 | 1 | 19 | 27.4 | -26.5 | -2 | 2 | 8 | 57.8 | -62.2 | -3 | 3 | 1 | 287.0 | -295.6 |
| -4 | 0 | 16 | 56.9 | 55.4 | 3 | 1 | 7 | 46.2 | -49.1 | -1 | 1 | 20 | 26.3 | -25.1 | 4 | 2 | 8 | 42.1 | -38.2 | 5 | 3 | 1 | 118.8 | -137.8 |
| -6 | 0 | 16 | 31.4 | 31.6 | -3 | 1 | 7 | 76.4 | -82.2 | -5 | 1 | 20 | 17.3 | 19.1 | -4 | 2 | 8 | 155.5 | -155.5 | -5 | 3 | 1 | 281.8 | -244.3 |
| -8 | 0 | 16 | 24.7 | 19.1 | 5 | 1 | 7 | 35.0 | -37.7 | -9 | 1 | 20 | 15.8 | -17.6 | -10 | 2 | 8 | 19.5 | 19.3 | -7 | 3 | 1 | 40.7 | -40.8 |
| -10 | 0 | 16 | 51.9 | 51.3 | -5 | 1 | 7 | 20.7 | 19.2 | 1 | 1 | 21 | 21.2 | 20.2 | -14 | 2 | 8 | 24.0 | -22.5 | 9 | 3 | 1 | 39.6 | 41.9 |
| -12 | 0 | 16 | 17.8 | 20.5 | 7 | 1 | 7 | 25.1 | -24.5 | -1 | 1 | 21 | 14.7 | 13.2 | 0 | 2 | 9 | 21.1 | -24.5 | -9 | 3 | 1 | 18.5 | 17.8 |
| -2 | 0 | 18 | 18.5 | -17.2 | -9 | 1 | 7 | 30.3 | -31.2 | -3 | 1 | 21 | 16.1 | 14.8 | 2 | 2 | 9 | 64.8 | -66.4 | 11 | 3 | 1 | 31.1 | -35.6 |

Table 1 (*cont.*)

| H | K | L | FO | FC |
|---|---|---|---|---|
| 13 | 3 | 1 | 29.0 | -30.1 |
| -13 | 3 | 1 | 27.2 | -26.6 |
| 15 | 3 | 1 | 26.0 | -28.5 |
| -1 | 3 | 2 | 26.9 | 27.8 |
| 3 | 3 | 2 | 88.0 | -82.2 |
| -3 | 3 | 2 | 35.9 | 26.8 |
| -5 | 3 | 2 | 0. | 13.8 |
| -7 | 3 | 2 | 26.4 | 23.0 |
| -9 | 3 | 2 | 21.0 | 24.3 |
| 1 | 3 | 3 | 63.7 | -61.3 |
| -1 | 3 | 3 | 74.2 | -82.6 |
| 3 | 3 | 3 | 97.0 | 89.9 |
| -3 | 3 | 3 | 59.4 | 64.6 |
| 5 | 3 | 3 | 98.0 | 102.8 |
| -5 | 3 | 3 | 288.5 | 292.3 |
| 7 | 3 | 3 | 35.2 | 35.9 |
| -7 | 3 | 3 | 120.2 | 123.8 |
| 9 | 3 | 3 | 45.1 | 48.4 |
| 11 | 3 | 3 | 23.4 | 30.9 |
| -11 | 3 | 3 | 25.9 | 25.8 |
| 13 | 3 | 3 | 31.1 | 35.2 |
| -13 | 3 | 3 | 46.4 | 45.8 |
| 1 | 3 | 4 | 57.9 | 57.5 |
| -1 | 3 | 4 | 22.2 | 20.2 |
| -3 | 3 | 4 | 48.2 | 40.6 |
| -5 | 3 | 4 | 34.3 | 27.4 |
| -5 | 3 | 4 | 0. | 27.2 |
| 7 | 3 | 4 | 34.0 | -30.8 |
| -9 | 3 | 4 | 22.5 | -20.7 |
| -11 | 3 | 4 | 28.8 | -30.7 |
| 1 | 3 | 5 | 20.0 | -17.5 |
| -1 | 3 | 5 | 84.8 | 89.7 |
| 3 | 3 | 5 | 156.1 | -158.2 |
| -3 | 3 | 5 | 90.8 | -92.0 |
| 5 | 3 | 5 | 133.5 | -149.0 |
| -5 | 3 | 5 | 122.2 | -102.9 |
| 7 | 3 | 5 | 55.7 | -57.3 |
| -7 | 3 | 5 | 67.8 | -57.5 |
| -11 | 3 | 5 | 36.4 | -33.1 |
| -13 | 3 | 5 | 61.5 | -60.3 |
| -15 | 3 | 5 | 44.3 | -43.3 |
| 1 | 3 | 6 | 20.1 | 19.3 |
| 5 | 3 | 6 | 21.9 | 26.4 |
| -5 | 3 | 6 | 32.3 | -33.3 |
| -7 | 3 | 6 | 35.6 | -26.3 |
| 1 | 3 | 7 | 82.9 | 83.0 |
| -1 | 3 | 7 | 16.2 | 17.4 |
| 3 | 3 | 7 | 146.3 | 152.3 |
| -3 | 3 | 7 | 80.6 | 79.8 |
| 5 | 3 | 7 | 61.5 | 70.7 |
| -5 | 3 | 7 | 166.9 | 149.3 |
| -7 | 3 | 7 | 62.2 | 55.3 |
| -9 | 3 | 7 | 52.7 | -42.2 |
| -11 | 3 | 7 | 27.5 | -26.1 |
| -13 | 3 | 7 | 42.4 | 43.2 |
| -15 | 3 | 7 | 38.3 | 38.5 |
| -1 | 3 | 8 | 44.6 | -45.2 |
| 3 | 3 | 8 | 39.3 | 35.8 |
| -3 | 3 | 8 | 32.1 | -32.5 |
| 1 | 3 | 9 | 26.3 | -24.6 |
| -1 | 3 | 9 | 24.2 | 25.4 |
| 3 | 3 | 9 | 63.9 | -65.1 |
| -3 | 3 | 9 | 52.9 | -56.1 |
| 5 | 3 | 9 | 28.4 | -33.0 |
| -5 | 3 | 9 | 227.3 | -221.8 |
| -7 | 3 | 9 | 113.4 | -100.9 |
| -15 | 3 | 9 | 19.8 | -20.2 |
| 1 | 3 | 10 | 55.1 | -53.5 |
| -3 | 3 | 10 | 19.4 | 22.1 |
| -9 | 3 | 10 | 28.1 | 21.0 |
| -11 | 3 | 10 | 23.3 | 21.5 |
| 1 | 3 | 11 | 23.5 | 24.0 |
| -1 | 3 | 11 | 50.7 | -65.6 |
| 3 | 3 | 11 | 52.0 | 52.7 |
| 5 | 3 | 11 | 23.5 | 31.9 |
| -5 | 3 | 11 | 76.5 | 78.2 |
| 7 | 3 | 11 | 38.5 | 46.1 |
| -7 | 3 | 11 | 146.6 | 134.3 |
| -9 | 3 | 11 | 21.5 | 18.5 |
| -13 | 3 | 11 | 56.1 | 51.2 |
| -15 | 3 | 11 | 38.9 | 36.4 |
| 5 | 3 | 12 | 21.9 | -25.3 |
| -7 | 3 | 12 | 30.2 | 32.7 |
| -9 | 3 | 12 | 25.5 | -18.0 |
| 1 | 3 | 13 | 44.9 | -45.3 |
| -1 | 3 | 13 | 18.4 | -23.4 |
| 3 | 3 | 13 | 49.2 | -54.0 |
| -3 | 3 | 13 | 19.1 | 20.2 |
| 5 | 3 | 13 | 37.5 | -46.4 |
| -5 | 3 | 13 | 66.8 | -66.8 |
| -7 | 3 | 13 | 37.4 | -30.4 |
| -13 | 3 | 13 | 36.5 | -34.7 |
| -15 | 3 | 13 | 56.0 | -52.0 |
| -1 | 3 | 14 | 15.1 | 15.0 |
| -9 | 3 | 14 | 24.4 | -22.3 |
| 1 | 3 | 15 | 36.7 | 38.9 |
| -1 | 3 | 15 | 24.8 | 26.9 |
| 3 | 3 | 15 | 22.4 | 24.9 |
| -3 | 3 | 15 | 46.8 | 47.3 |
| -5 | 3 | 15 | 54.4 | 57.0 |
| -7 | 3 | 15 | 59.1 | 55.5 |
| -11 | 3 | 15 | 28.8 | -28.5 |
| -15 | 3 | 15 | 29.7 | 25.7 |
| -3 | 3 | 16 | 20.4 | -21.8 |
| -5 | 3 | 17 | 43.4 | -47.4 |
| -7 | 3 | 17 | 70.1 | -70.4 |
| -9 | 3 | 17 | 28.8 | -23.6 |
| 1 | 3 | 19 | 11.2 | 14.4 |
| -7 | 3 | 19 | 25.0 | 27.7 |
| -9 | 3 | 19 | 30.8 | 26.1 |
| -15 | 3 | 19 | 31.3 | 27.9 |
| 1 | 3 | 21 | 13.5 | -12.6 |
| -1 | 3 | 21 | 16.8 | -22.5 |
| -15 | 3 | 21 | 19.8 | -19.4 |
| 0 | 4 | 0 | 65.9 | 72.0 |
| 2 | 4 | 0 | 121.9 | 118.3 |
| 8 | 4 | 0 | 37.9 | -36.2 |
| -8 | 4 | 0 | 35.7 | -36.2 |
| 12 | 4 | 0 | 28.7 | 27.5 |
| -12 | 4 | 0 | 26.0 | 27.5 |
| 0 | 4 | 1 | 64.8 | -71.2 |
| 2 | 4 | 1 | 57.6 | -61.6 |
| -2 | 4 | 1 | 40.5 | -38.0 |
| 8 | 4 | 1 | 46.6 | 47.6 |
| -8 | 4 | 1 | 18.6 | -18.5 |
| 10 | 4 | 1 | 32.7 | -34.8 |
| 12 | 4 | 1 | 20.0 | -19.1 |
| 2 | 4 | 2 | 92.0 | -91.6 |
| -2 | 4 | 2 | 43.9 | -49.0 |
| -8 | 4 | 2 | 21.3 | -18.6 |
| 10 | 4 | 2 | 36.1 | -37.8 |
| 12 | 4 | 2 | 40.4 | -41.5 |
| -12 | 4 | 2 | 35.8 | -39.7 |
| 14 | 4 | 2 | 27.2 | -23.9 |
| 0 | 4 | 3 | 27.1 | 21.6 |
| 2 | 4 | 3 | 18.6 | -19.8 |
| -2 | 4 | 3 | 10.5 | 10.7 |
| 6 | 4 | 3 | 52.0 | -50.3 |
| 10 | 4 | 3 | 39.4 | 41.7 |
| -10 | 4 | 3 | 48.3 | 49.3 |
| 12 | 4 | 3 | 23.8 | 22.9 |
| -12 | 4 | 3 | 23.4 | 24.4 |
| 0 | 4 | 4 | 31.9 | -28.7 |
| 2 | 4 | 4 | 72.5 | 67.5 |
| -2 | 4 | 4 | 25.7 | -26.2 |
| 6 | 4 | 4 | 66.3 | 60.2 |
| 8 | 4 | 4 | 35.1 | 38.2 |
| -8 | 4 | 4 | 45.2 | 43.6 |
| 10 | 4 | 4 | 34.7 | 35.6 |
| -10 | 4 | 4 | 35.3 | 35.3 |
| 12 | 4 | 4 | 26.5 | 28.5 |
| -12 | 4 | 4 | 48.2 | 51.8 |
| -14 | 4 | 4 | 38.1 | 38.1 |
| 0 | 4 | 5 | 38.3 | -37.9 |
| 2 | 4 | 5 | 70.8 | -68.7 |
| -2 | 4 | 5 | 30.6 | 28.9 |
| 6 | 4 | 5 | 32.2 | 31.9 |
| 10 | 4 | 5 | 25.8 | -29.6 |
| -10 | 4 | 5 | 21.2 | -25.1 |
| -12 | 4 | 5 | 25.2 | -26.9 |
| 14 | 4 | 5 | 23.1 | 17.7 |
| 0 | 4 | 6 | 36.2 | -38.2 |
| 2 | 4 | 6 | 103.9 | -99.3 |
| -2 | 4 | 6 | 24.0 | -21.6 |
| 4 | 4 | 6 | 135.9 | -116.3 |
| 6 | 4 | 6 | 46.1 | -41.5 |
| -12 | 4 | 6 | 41.3 | -39.6 |
| -14 | 4 | 6 | 43.3 | -45.3 |
| -16 | 4 | 6 | 23.0 | -21.9 |
| 0 | 4 | 7 | 86.6 | 86.6 |
| 2 | 4 | 7 | 66.4 | 64.3 |
| -2 | 4 | 7 | 28.9 | 31.0 |
| 6 | 4 | 7 | 31.6 | -32.1 |
| -6 | 4 | 7 | 61.1 | -54.4 |
| 8 | 4 | 7 | 19.7 | -19.1 |
| 0 | 4 | 8 | 33.2 | 32.3 |
| 2 | 4 | 8 | 85.2 | 84.4 |
| -2 | 4 | 8 | 56.8 | 63.5 |
| 4 | 4 | 8 | 42.0 | 40.4 |
| -6 | 4 | 8 | 88.2 | 84.8 |
| -10 | 4 | 8 | 17.4 | -19.3 |
| -12 | 4 | 8 | 17.9 | 22.2 |
| 0 | 4 | 9 | 50.6 | -50.7 |
| 2 | 4 | 9 | 16.8 | -18.5 |
| 4 | 4 | 9 | 42.4 | 40.6 |
| 6 | 4 | 9 | 49.9 | 55.4 |
| -6 | 4 | 9 | 44.1 | -39.8 |
| 10 | 4 | 9 | 27.5 | -26.1 |
| -10 | 4 | 9 | 25.1 | -25.8 |
| 0 | 4 | 10 | 19.4 | -18.4 |
| 2 | 4 | 10 | 39.1 | -40.7 |
| -4 | 4 | 10 | 71.1 | -66.8 |
| -6 | 4 | 10 | 116.4 | -115.7 |
| 8 | 4 | 10 | 27.1 | -27.2 |
| -8 | 4 | 10 | 46.9 | -46.6 |
| 10 | 4 | 10 | 28.3 | -27.4 |
| -12 | 4 | 10 | 27.7 | -28.4 |
| -14 | 4 | 10 | 31.4 | -31.8 |
| 2 | 4 | 11 | 23.6 | 21.5 |
| -2 | 4 | 11 | 21.4 | -20.8 |
| 4 | 4 | 11 | 18.0 | -27.1 |
| -4 | 4 | 11 | 44.6 | -44.7 |
| -8 | 4 | 11 | 46.7 | 46.1 |
| 10 | 4 | 11 | 21.3 | 18.0 |
| -12 | 4 | 11 | 20.2 | 22.5 |
| 0 | 4 | 12 | 37.2 | 36.7 |
| 2 | 4 | 12 | 53.0 | 50.1 |
| -2 | 4 | 12 | 18.1 | -16.6 |
| 4 | 4 | 12 | 30.3 | 32.2 |
| 6 | 4 | 12 | 25.9 | 24.7 |
| -6 | 4 | 12 | 51.6 | 52.4 |
| 8 | 4 | 12 | 25.0 | 26.4 |
| -8 | 4 | 12 | 34.4 | 31.9 |
| -12 | 4 | 12 | 33.8 | 33.1 |
| -14 | 4 | 12 | 51.0 | 46.6 |
| -16 | 4 | 12 | 20.3 | 19.0 |
| 0 | 4 | 13 | 62.7 | -64.4 |
| 2 | 4 | 13 | 21.3 | -22.4 |
| -2 | 4 | 13 | 19.6 | 19.1 |
| -4 | 4 | 13 | 42.5 | 46.3 |
| 0 | 4 | 14 | 48.0 | -48.0 |
| 2 | 4 | 14 | 54.9 | -49.0 |
| -2 | 4 | 14 | 29.3 | -28.6 |
| -6 | 4 | 14 | 27.2 | -24.9 |
| -14 | 4 | 14 | 30.1 | -28.7 |
| -16 | 4 | 14 | 22.9 | -25.4 |
| 0 | 4 | 15 | 31.2 | 30.8 |
| 2 | 4 | 15 | 22.2 | 22.3 |
| -2 | 4 | 15 | 29.2 | 30.0 |
| 0 | 4 | 16 | 26.0 | 27.0 |
| 2 | 4 | 16 | 17.7 | 14.2 |
| -2 | 4 | 16 | 20.0 | 20.6 |
| -4 | 4 | 16 | 32.0 | 34.1 |
| -6 | 4 | 16 | 56.8 | 58.6 |
| -8 | 4 | 16 | 29.9 | 29.1 |
| 0 | 4 | 17 | 17.3 | -20.4 |
| 4 | 4 | 17 | 21.3 | 18.7 |
| -8 | 4 | 17 | 26.9 | -28.3 |
| -6 | 4 | 18 | 28.6 | -32.7 |
| -8 | 4 | 18 | 39.5 | -37.9 |
| 0 | 4 | 19 | 11.5 | 16.8 |
| -4 | 4 | 19 | 23.5 | -24.5 |
| 0 | 4 | 20 | 22.6 | 21.9 |
| 0 | 4 | 21 | 11.8 | -14.0 |
| -2 | 4 | 21 | 18.8 | -16.9 |
| -2 | 4 | 22 | 19.9 | -21.1 |
| 1 | 5 | 0 | 26.0 | 24.9 |
| 3 | 5 | 0 | 72.5 | 74.5 |
| 9 | 5 | 0 | 28.1 | -29.0 |
| 11 | 5 | 0 | 17.4 | 12.0 |
| 15 | 5 | 0 | 18.7 | -13.7 |
| 1 | 5 | 1 | 89.9 | 95.3 |
| -1 | 5 | 1 | 115.1 | 126.0 |
| 3 | 5 | 1 | 64.5 | 60.3 |
| -3 | 5 | 1 | 70.5 | 74.5 |
| 9 | 5 | 1 | 56.9 | 55.0 |
| -9 | 5 | 1 | 19.7 | 19.9 |
| 11 | 5 | 1 | 26.7 | 25.7 |
| -11 | 5 | 1 | 34.6 | 32.3 |
| -1 | 5 | 2 | 30.1 | 30.7 |
| -3 | 5 | 2 | 31.0 | -29.9 |
| 7 | 5 | 2 | 64.1 | 64.9 |
| 9 | 5 | 2 | 20.5 | -16.8 |
| 11 | 5 | 2 | 31.2 | -32.1 |
| -11 | 5 | 2 | 22.1 | -24.4 |
| 1 | 5 | 3 | 53.8 | -50.4 |
| -1 | 5 | 3 | 49.5 | -52.2 |
| 3 | 5 | 3 | 18.6 | -17.4 |
| -3 | 5 | 3 | 105.6 | -106.4 |
| 7 | 5 | 3 | 57.0 | -55.6 |
| 9 | 5 | 3 | 44.3 | -47.1 |
| -9 | 5 | 3 | 71.1 | -67.8 |
| 11 | 5 | 3 | 47.6 | -47.3 |
| -11 | 5 | 3 | 49.6 | -47.3 |
| -1 | 5 | 4 | 70.7 | -73.8 |
| 3 | 5 | 4 | 18.3 | 16.2 |
| -3 | 5 | 4 | 57.0 | -59.2 |
| 9 | 5 | 4 | 24.0 | 24.7 |
| 11 | 5 | 4 | 29.9 | 29.0 |
| -11 | 5 | 4 | 28.2 | 26.4 |
| -13 | 5 | 4 | 26.0 | 22.7 |
| 15 | 5 | 4 | 17.8 | -12.6 |
| 1 | 5 | 5 | 89.7 | 88.6 |
| -1 | 5 | 5 | 84.6 | 97.4 |
| 3 | 5 | 5 | 53.7 | 49.8 |
| 7 | 5 | 5 | 44.1 | 43.1 |
| 9 | 5 | 5 | 43.9 | 44.7 |
| -9 | 5 | 5 | 56.3 | 59.7 |
| 11 | 5 | 5 | 23.5 | 20.3 |
| -11 | 5 | 5 | 48.0 | 49.1 |
| -13 | 5 | 5 | 33.7 | 32.5 |
| 1 | 5 | 6 | 42.8 | -43.3 |
| 3 | 5 | 6 | 62.2 | -59.7 |
| -3 | 5 | 6 | 33.0 | 34.7 |
| 7 | 5 | 6 | 22.0 | 24.9 |
| -9 | 5 | 6 | 25.4 | 26.1 |
| 1 | 5 | 7 | 85.4 | -92.8 |
| -1 | 5 | 7 | 87.7 | -93.6 |
| 3 | 5 | 7 | 61.9 | -59.7 |
| -3 | 5 | 7 | 71.5 | -70.3 |
| 5 | 5 | 7 | 28.5 | -23.9 |
| 9 | 5 | 7 | 18.9 | -20.8 |
| -9 | 5 | 7 | 24.8 | -25.1 |
| -11 | 5 | 7 | 41.3 | -42.0 |
| 1 | 5 | 8 | 49.6 | 50.8 |
| 3 | 5 | 8 | 21.3 | 17.8 |
| 5 | 5 | 8 | 49.6 | -49.1 |
| 7 | 5 | 8 | 41.9 | -44.9 |
| -9 | 5 | 8 | 22.9 | -21.9 |
| 1 | 5 | 9 | 59.1 | 54.0 |
| -1 | 5 | 9 | 50.4 | 52.0 |
| 3 | 5 | 9 | 20.4 | 20.7 |
| -3 | 5 | 9 | 69.2 | 72.4 |
| -7 | 5 | 9 | 59.2 | 62.2 |
| -9 | 5 | 9 | 41.1 | 43.1 |
| 11 | 5 | 9 | 23.1 | 24.2 |
| -11 | 5 | 9 | 26.9 | 25.6 |
| 1 | 5 | 10 | 17.1 | -19.4 |
| -1 | 5 | 10 | 25.6 | 25.5 |
| -3 | 5 | 10 | 37.7 | 40.0 |
| 5 | 5 | 10 | 49.0 | 51.6 |
| -5 | 5 | 10 | 38.1 | -30.2 |
| -7 | 5 | 10 | 44.8 | -45.9 |
| 9 | 5 | 10 | 24.8 | -26.1 |
| 11 | 5 | 10 | 14.4 | -15.4 |
| 1 | 5 | 11 | 26.5 | -26.8 |
| -1 | 5 | 11 | 36.6 | -37.5 |
| 5 | 5 | 11 | 26.9 | -23.4 |
| -5 | 5 | 11 | 51.1 | -51.4 |
| -7 | 5 | 11 | 26.9 | -28.2 |
| 9 | 5 | 11 | 25.5 | -31.2 |
| -9 | 5 | 11 | 51.8 | -50.8 |
| -11 | 5 | 11 | 51.8 | -48.6 |
| -13 | 5 | 11 | 26.3 | -24.4 |
| 1 | 5 | 12 | 42.8 | 42.3 |
| -3 | 5 | 12 | 62.1 | -65.8 |
| -5 | 5 | 12 | 35.1 | -35.1 |
| -7 | 5 | 12 | 19.6 | 15.8 |
| -13 | 5 | 12 | 22.2 | 16.8 |
| 1 | 5 | 13 | 46.7 | 45.8 |
| -1 | 5 | 13 | 34.6 | 34.9 |
| 3 | 5 | 13 | 30.2 | 31.0 |
| -3 | 5 | 13 | 32.7 | 36.7 |
| -5 | 5 | 13 | 20.6 | -17.4 |
| 7 | 5 | 13 | 18.6 | 20.4 |
| -7 | 5 | 13 | 20.7 | 15.5 |
| -9 | 5 | 13 | 16.1 | 21.1 |
| -11 | 5 | 13 | 31.7 | 28.3 |
| -13 | 5 | 13 | 27.2 | 26.3 |
| 1 | 5 | 14 | 44.3 | -43.9 |
| -1 | 5 | 14 | 28.2 | -29.5 |
| -3 | 5 | 14 | 18.7 | 19.6 |
| 5 | 5 | 14 | 20.9 | 19.1 |
| -5 | 5 | 14 | 28.6 | 27.6 |
| -11 | 5 | 14 | 21.4 | 18.1 |
| 1 | 5 | 15 | 32.3 | -32.4 |
| -1 | 5 | 15 | 46.5 | -48.8 |
| -3 | 5 | 15 | 31.3 | -32.9 |
| -5 | 5 | 15 | 30.0 | -26.7 |
| 1 | 5 | 16 | 15.8 | 19.0 |
| 5 | 5 | 16 | 21.9 | -24.0 |
| -7 | 5 | 16 | 23.1 | 24.4 |
| -9 | 5 | 16 | 16.6 | 17.4 |
| -13 | 5 | 16 | 16.8 | -14.2 |
| -1 | 5 | 17 | 18.3 | 18.4 |
| -3 | 5 | 17 | 20.5 | 16.5 |
| -5 | 5 | 17 | 23.2 | 24.8 |
| -7 | 5 | 17 | 23.5 | 22.4 |
| -9 | 5 | 17 | 23.4 | 27.7 |
| -11 | 5 | 17 | 23.9 | 22.5 |
| -3 | 5 | 18 | 28.9 | 26.5 |
| -7 | 5 | 18 | 22.9 | -22.8 |
| -9 | 5 | 18 | 19.9 | -22.6 |
| 1 | 5 | 19 | 12.7 | -13.8 |
| -1 | 5 | 19 | 11.5 | -10.4 |
| -7 | 5 | 19 | 19.6 | -16.6 |
| 1 | 5 | 20 | 12.6 | 13.2 |
| -1 | 5 | 20 | 16.7 | 17.0 |
| -5 | 5 | 20 | 26.4 | -27.4 |
| 1 | 5 | 21 | 17.4 | 16.5 |
| -1 | 5 | 21 | 19.6 | 17.5 |
| 0 | 6 | 0 | 167.2 | -199.4 |
| 2 | 6 | 0 | 63.0 | -64.5 |
| -2 | 6 | 1 | 41.1 | 39.4 |
| 0 | 6 | 2 | 92.7 | 110.1 |
| 2 | 6 | 2 | 44.2 | 46.3 |
| -2 | 6 | 2 | 98.0 | 96.6 |
| 0 | 6 | 3 | 61.9 | 58.0 |
| -2 | 6 | 3 | 41.1 | 38.5 |
| 0 | 6 | 4 | 92.8 | -94.1 |
| 2 | 6 | 4 | 21.4 | -20.0 |
| -2 | 6 | 4 | 45.7 | -49.3 |
| 2 | 6 | 5 | 18.3 | 18.5 |
| -2 | 6 | 5 | 40.7 | -42.3 |
| 0 | 6 | 6 | 96.6 | 101.6 |
| 2 | 6 | 6 | 44.6 | 43.8 |
| -2 | 6 | 6 | 62.1 | 65.0 |
| 0 | 6 | 7 | 22.7 | -20.1 |
| 2 | 6 | 7 | 54.2 | -50.5 |
| -2 | 6 | 7 | 19.0 | -18.2 |
| 0 | 6 | 8 | 86.8 | -86.2 |
| -2 | 6 | 8 | 101.5 | -109.2 |
| 0 | 6 | 10 | 49.5 | 48.9 |
| -2 | 6 | 10 | 57.8 | 58.4 |
| -2 | 6 | 11 | 56.8 | 59.4 |
| 0 | 6 | 12 | 31.4 | -28.5 |
| -2 | 6 | 12 | 42.4 | -44.0 |
| 2 | 6 | 13 | 33.3 | 27.9 |
| 0 | 6 | 14 | 38.0 | 36.5 |
| 2 | 6 | 14 | 20.1 | 20.5 |
| -2 | 6 | 14 | 36.5 | 38.6 |
| 0 | 6 | 15 | 27.8 | -26.6 |
| 0 | 6 | 16 | 14.8 | -16.1 |
| -2 | 6 | 16 | 34.7 | -33.0 |
| 2 | 6 | 20 | 11.8 | -13.6 |
| 1 | 7 | 0 | 26.3 | -27.7 |
| 3 | 7 | 0 | 37.5 | -37.5 |
| 1 | 7 | 1 | 75.5 | -79.9 |
| -1 | 7 | 1 | 104.6 | -105.5 |
| -3 | 7 | 1 | 44.1 | -45.3 |
| 1 | 7 | 2 | 40.4 | -38.9 |
| -3 | 7 | 2 | 52.6 | 52.1 |
| 1 | 7 | 3 | 44.2 | 44.4 |
| -1 | 7 | 3 | 91.6 | 98.1 |
| -3 | 7 | 3 | 25.1 | 23.5 |
| 1 | 7 | 4 | 44.9 | 43.6 |
| -1 | 7 | 4 | 53.9 | 55.1 |
| 1 | 7 | 5 | 52.1 | -51.4 |
| -1 | 7 | 5 | 62.5 | -66.7 |
| 3 | 7 | 5 | 22.0 | -23.0 |
| -3 | 7 | 5 | 42.6 | -38.5 |
| 3 | 7 | 6 | 40.2 | 39.1 |
| 1 | 7 | 7 | 59.2 | 59.2 |
| -1 | 7 | 7 | 87.3 | 89.3 |
| -3 | 7 | 7 | 31.4 | 29.5 |
| 1 | 7 | 8 | 14.7 | 16.4 |
| -1 | 7 | 8 | 15.3 | -16.4 |
| -3 | 7 | 8 | 60.6 | -59.4 |
| 1 | 7 | 9 | 14.0 | -16.2 |
| -1 | 7 | 9 | 70.3 | -72.8 |
| -3 | 7 | 9 | 47.0 | -42.1 |
| 1 | 7 | 10 | 33.4 | -29.3 |
| -1 | 7 | 10 | 29.7 | -33.5 |
| 3 | 7 | 10 | 18.7 | -18.4 |
| -3 | 7 | 10 | 22.3 | 23.9 |
| -1 | 7 | 11 | 27.6 | 29.6 |
| -3 | 7 | 11 | 52.6 | 55.3 |
| 1 | 7 | 12 | 23.8 | 21.3 |
| -1 | 7 | 12 | 18.6 | 19.8 |
| 3 | 7 | 12 | 27.9 | -25.0 |
| 1 | 7 | 13 | 16.1 | -14.6 |
| -1 | 7 | 13 | 27.2 | -29.7 |
| -3 | 7 | 13 | 23.7 | -28.3 |
| -3 | 7 | 14 | 17.4 | 21.1 |
| -1 | 7 | 15 | 22.5 | 26.5 |
| -3 | 7 | 15 | 33.5 | 34.6 |
| -3 | 7 | 16 | 23.5 | -23.1 |
| -1 | 7 | 17 | 11.9 | -11.2 |
| -3 | 7 | 17 | 27.9 | -26.9 |
| 1 | 7 | 18 | 13.8 | -14.0 |
| -1 | 7 | 18 | 16.5 | -17.2 |
| 0 | 8 | 0 | 33.8 | 28.9 |
| 2 | 8 | 0 | 57.2 | 54.0 |
| 0 | 8 | 1 | 23.9 | -25.7 |
| 2 | 8 | 1 | 13.8 | 16.4 |
| -2 | 8 | 1 | 31.7 | -30.5 |
| 0 | 8 | 2 | 52.2 | -49.3 |
| -2 | 8 | 2 | 34.1 | -35.9 |
| 0 | 8 | 3 | 27.9 | -25.7 |
| 2 | 8 | 3 | 59.2 | -60.0 |
| 2 | 8 | 4 | 33.5 | 34.6 |
| -2 | 8 | 4 | 33.0 | 34.7 |
| 0 | 8 | 6 | 23.3 | -21.0 |
| 2 | 8 | 6 | 34.0 | -32.6 |
| -2 | 8 | 6 | 34.2 | -37.3 |
| 0 | 8 | 7 | 28.4 | 27.8 |
| -2 | 8 | 7 | 31.1 | 31.7 |
| 0 | 8 | 8 | 36.4 | 34.3 |
| -2 | 8 | 8 | 31.2 | 32.1 |
| 0 | 8 | 9 | 11.0 | 14.5 |
| 2 | 8 | 9 | 41.5 | 41.5 |
| -2 | 8 | 9 | 30.5 | -31.9 |
| -2 | 8 | 10 | 41.3 | -45.9 |
| 0 | 8 | 11 | 44.1 | -43.9 |
| 2 | 8 | 11 | 31.7 | -31.0 |
| 2 | 8 | 12 | 18.1 | 17.2 |
| -2 | 8 | 12 | 15.8 | 16.4 |
| 2 | 8 | 14 | 16.1 | -14.6 |
| -2 | 8 | 14 | 16.0 | -16.5 |
| -2 | 8 | 15 | 21.4 | 22.6 |
| -2 | 8 | 16 | 15.6 | 16.2 |
| 0 | 8 | 17 | 19.7 | 20.7 |
| 2 | 8 | 17 | 15.9 | 14.1 |
| 1 | 9 | 0 | 49.4 | 47.7 |
| 3 | 9 | 1 | 31.1 | 32.7 |
| -3 | 9 | 1 | 34.9 | 32.2 |
| -1 | 9 | 2 | 30.6 | -29.9 |
| 3 | 9 | 2 | 51.9 | 49.9 |
| 3 | 9 | 3 | 39.2 | -38.9 |
| -3 | 9 | 3 | 38.4 | -39.5 |
| 1 | 9 | 4 | 24.4 | -25.5 |
| -3 | 9 | 4 | 22.2 | -22.5 |
| 3 | 9 | 5 | 29.4 | 28.6 |
| -3 | 9 | 5 | 19.4 | 21.5 |
| 1 | 9 | 6 | 17.6 | -20.2 |
| -1 | 9 | 6 | 19.5 | -18.3 |
| 3 | 9 | 7 | 30.8 | -29.7 |
| -3 | 9 | 7 | 16.1 | -13.7 |
| -1 | 9 | 8 | 39.0 | 40.7 |
| 3 | 9 | 8 | 21.9 | -24.6 |
| -3 | 9 | 8 | 27.4 | 27.7 |
| 3 | 9 | 9 | 21.8 | 23.3 |
| 1 | 9 | 10 | 39.4 | 36.6 |
| -1 | 9 | 14 | 15.7 | -14.9 |
| -3 | 9 | 16 | 17.0 | 18.7 |
| 0 | 10 | 1 | 48.9 | 50.5 |
| -2 | 10 | 1 | 35.8 | 38.4 |
| 2 | 10 | 2 | 45.1 | 43.9 |
| -2 | 10 | 2 | 20.6 | 19.5 |
| 2 | 10 | 4 | 33.2 | -34.5 |
| 0 | 10 | 5 | 20.5 | 22.7 |
| 2 | 10 | 5 | 23.4 | 22.6 |
| 2 | 10 | 6 | 22.6 | 23.5 |
| 0 | 10 | 7 | 45.9 | -46.9 |
| 2 | 10 | 7 | 27.5 | -27.1 |
| -2 | 10 | 7 | 25.4 | -27.2 |
| 2 | 10 | 8 | 33.5 | -32.0 |
| 0 | 10 | 9 | 22.7 | 23.1 |
| -2 | 10 | 9 | 24.5 | 23.8 |
| 0 | 10 | 10 | 20.0 | 20.9 |
| 2 | 10 | 10 | 30.3 | 29.0 |
| 0 | 10 | 12 | 16.9 | -16.6 |
| 2 | 10 | 12 | 15.4 | -17.6 |
| 0 | 10 | 13 | 15.3 | 17.4 |
| 2 | 10 | 13 | 15.0 | 14.5 |
| 0 | 10 | 14 | 13.6 | 10.4 |
| 2 | 10 | 14 | 16.2 | 13.2 |
| 0 | 10 | 15 | 19.7 | -18.6 |
| -2 | 10 | 15 | 18.0 | -16.7 |
| 0 | 10 | 16 | 12.5 | -12.7 |
| 1 | 11 | 0 | 17.0 | -19.6 |
| 1 | 11 | 1 | 22.6 | -22.5 |
| -1 | 11 | 1 | 14.6 | -16.6 |
| 3 | 11 | 1 | 27.9 | -30.5 |
| 1 | 11 | 3 | 41.5 | 41.2 |
| -1 | 11 | 3 | 28.3 | 28.2 |
| 3 | 11 | 3 | 23.5 | 24.2 |
| -3 | 11 | 3 | 20.8 | 18.7 |
| -1 | 11 | 4 | 25.9 | 22.6 |
| 1 | 11 | 5 | 28.8 | -26.9 |
| -1 | 11 | 5 | 32.1 | -31.3 |
| -3 | 11 | 5 | 19.5 | -20.8 |
| 1 | 11 | 6 | 16.7 | 18.7 |
| -3 | 11 | 6 | 15.5 | -17.2 |
| 1 | 11 | 7 | 21.1 | 18.6 |
| 1 | 11 | 8 | 15.7 | -14.1 |
| -1 | 11 | 8 | 12.6 | -11.2 |
| 1 | 11 | 9 | 30.3 | -29.0 |
| 1 | 11 | 11 | 28.8 | 27.2 |
| -1 | 11 | 11 | 25.1 | 23.3 |
| -3 | 11 | 12 | 20.1 | 20.1 |
| 1 | 11 | 13 | 18.3 | -15.2 |
| -1 | 11 | 13 | 16.2 | -16.1 |
| 1 | 11 | 14 | 19.9 | 16.5 |
| 1 | 11 | 15 | 13.9 | 11.1 |
| 1 | 11 | 16 | 11.1 | -4.5 |
| -1 | 11 | 17 | 11.3 | -11.1 |
| 0 | 12 | 0 | 15.9 | 14.5 |
| 2 | 12 | 0 | 20.1 | 15.4 |
| 0 | 12 | 2 | 34.4 | -31.5 |
| 2 | 12 | 2 | 22.9 | -25.8 |
| 0 | 12 | 3 | 16.4 | -17.2 |
| 0 | 12 | 4 | 35.4 | 35.1 |
| 2 | 12 | 4 | 21.0 | 19.1 |
| -2 | 12 | 4 | 20.1 | 27.3 |
| 0 | 12 | 6 | 15.9 | -17.0 |
| -2 | 12 | 6 | 22.3 | -22.6 |
| 2 | 12 | 7 | 17.3 | 15.9 |
| 0 | 12 | 8 | 17.3 | 15.7 |
| 0 | 12 | 10 | 22.1 | -20.8 |
| 2 | 12 | 10 | 17.0 | -17.1 |
| -2 | 12 | 10 | 17.3 | -16.0 |
| -2 | 12 | 11 | 15.7 | -16.1 |
| 0 | 12 | 12 | 15.4 | 16.7 |
| -2 | 12 | 12 | 18.6 | 15.7 |
| 1 | 13 | 1 | 14.3 | 17.1 |
| -1 | 13 | 1 | 14.7 | 12.0 |
| -1 | 13 | 2 | 14.8 | 12.1 |
| 1 | 13 | 3 | 21.7 | -22.1 |
| -1 | 13 | 3 | 26.5 | -27.2 |
| -1 | 13 | 4 | 14.1 | -14.8 |
| -1 | 13 | 5 | 21.7 | 22.7 |
| -1 | 13 | 7 | 13.7 | -10.5 |
| -1 | 13 | 9 | 14.4 | 13.8 |
| -1 | 13 | 10 | 11.5 | 10.6 |
| -1 | 13 | 11 | 15.7 | -14.8 |

cycles Cruickshank's weighting system was used, modified by downweighting the weaker reflexions:

$$w=\frac{1}{(3/|F_{\max}|)\ |F_o|^2+|F_o|+2|F_{\min}|}\quad \text{if } |F_o|\geq 2|F_{\min}|$$

$$w=a|F_o|^2\qquad\qquad \text{if } |F_o|<2|F_{\min}|\,,$$

where $|F_{\min}|=14{\cdot}0$, $|F_{\max}|=320{\cdot}0$ and the constant $a$ was such as to ensure that the two functions assumed the same value for $|F_o|=2|F_{\min}|$. With such a weighting system the average $w(F_o-F_c)^2$ was approximately constant over the whole range of $F_o$ values. In the last refinement cycle all the parameter shifts were well within the corresponding standard deviations.

Table 2. *Positional parameters in fractional coordinates and, in parentheses, the significant figures of their standard deviations*

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| Co | 0·5000 | 0·2517 (2) | 0·2500 |
| S(1) | 0·3735 (2) | 0·3987 (2) | 0·2235 (1) |
| S(2) | 0·4005 (2) | 0·0794 (3) | 0·2495 (1) |
| S(3) | 0·4470 (2) | 0·2785 (2) | 0·1098 (1) |
| N(1) | 0·5000 | −0·1439 (10) | 0·2500 |
| N(2) | 0·2936 (6) | 0·4502 (8) | 0·0593 (4) |
| C(1) | 0·5000 | −0·0169 (12) | 0·2500 |
| C(2) | 0·4072 (12) | −0·2158 (12) | 0·2479 (7) |
| C(3) | 0·4186 (15) | −0·2661 (13) | 0·3373 (8) |
| C(4) | 0·3605 (6) | 0·3848 (8) | 0·1210 (4) |
| C(5) | 0·2953 (8) | 0·4366 (11) | −0·0281 (4) |
| C(6) | 0·2290 (9) | 0·5517 (14) | 0·0775 (5) |
| C(7) | 0·3444 (11) | 0·5503 (13) | −0·0529 (7) |
| C(8) | 0·1237 (9) | 0·5587 (19) | 0·0161 (8) |

The following atomic scattering curves were used: cobalt from Freeman & Watson (1961); sulphur from Dawson (1960); carbon and nitrogen from Freeman (1959).

The observed and calculated factors are compared in Table 1. Tables 2 and 3 respectively give the final positional and thermal parameters with their standard deviations.

## Description and discussion of the structure

The structure consists of enantiomorphous molecules $Co[S_2CN(C_2H_5)_2]_3$ (Fig. 1). The interatomic distances and bond angles were calculated by the *ORFFE* program of Busing, Martin & Levy (1964). Intramolecular distances and bond angles are reported in Tables 4 and 5 respectively and in Fig. 1. The closest intermolecular distances are reported in Table 8 and in Fig. 2. In the Tables and Figures the atoms of the different asymmetric units are related to the symmetry equivalent atoms of the fundamental unit as follows:

Table 3. *Thermal parameters and, in parentheses, the significant figures of their standard deviations*

| | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|
| Co | 0·0049 (1) | 0·0067 (2) | 0·0029 (1) | 0·0 | 0·0013 (1) | 0·0 |
| S(1) | 0·0069 (2) | 0·0114 (3) | 0·0031 (1) | 0·0014 (2) | 0·0019 (1) | −0·0001 (1) |
| S(2) | 0·0062 (1) | 0·0098 (3) | 0·0039 (1) | −0·0015 (2) | 0·0015 (1) | 0·0005 (1) |
| S(3) | 0·0058 (1) | 0·0088 (2) | 0·0032 (1) | 0·0008 (2) | 0·0018 (1) | −0·0002 (1) |
| N(1) | 0·0107 (10) | 0·0073 (11) | 0·0044 (4) | 0·0 | 0·0007 (5) | 0·0 |
| N(2) | 0·0069 (5) | 0·0111 (9) | 0·0037 (3) | 0·0018 (7) | 0·0016 (3) | 0·0004 (4) |
| C(1) | 0·0073 (10) | 0·0071 (10) | 0·0039 (5) | 0·0 | 0·0008 (6) | 0·0 |
| C(2) | 0·0156 (12) | 0·0106 (11) | 0·0059 (5) | −0·0086 (13) | 0·0012 (7) | −0·0001 (6) |
| C(3) | 0·0246 (21) | 0·0187 (18) | 0·0075 (6) | −0·0156 (20) | 0·0034 (9) | 0·0018 (8) |
| C(4) | 0·0056 (5) | 0·0077 (8) | 0·0036 (3) | −0·0001 (8) | 0·0017 (3) | 0·0001 (4) |
| C(5) | 0·0079 (6) | 0·0160 (13) | 0·0030 (3) | −0·0010 (10) | 0·0011 (3) | 0·0011 (5) |
| C(6) | 0·0108 (9) | 0·0254 (22) | 0·0039 (4) | 0·0093 (14) | 0·0017 (5) | −0·0007 (7) |
| C(7) | 0·0128 (11) | 0·0199 (18) | 0·0065 (6) | −0·0050 (15) | 0·0042 (7) | 0·0012 (9) |
| C(8) | 0·0075 (19) | 0·0346 (34) | 0·0090 (7) | 0·0061 (16) | 0·0015 (7) | −0·0005 (13) |

Table 4. *Bond distances, with their standard deviations*

| Bond | Distance | e.s.d. | Mean | e.s.d. | Calculated |
|---|---|---|---|---|---|
| Co—S(2) | 2·255 Å | 0·003 Å | 2·258 Å | 0·002 Å | 2·26 Å |
| Co—S(1) | 2·260 | 0·003 | | | |
| Co—S(3) | 2·258 | 0·002 | | | |
| S(2)-C(1) | 1·714 | 0·008 | 1·704 | 0·005 | 1·698 |
| S(1)-C(4) | 1·703 | 0·007 | | | |
| S(3)-C(4) | 1·695 | 0·009 | | | |
| C(1)-N(1) | 1·304 | 0·015 | 1·319 | 0·009 | 1·314 |
| C(4)-N(2) | 1·327 | 0·010 | | | |
| N(1)-C(2) | 1·492 | 0·016 | 1·496 | 0·008 | |
| N(2)-C(5) | 1·502 | 0·010 | | | |
| N(2)-C(6) | 1·486 | 0·014 | | | |
| C(2)-C(3) | 1·562 | 0·019 | | | |
| C(5)-C(7) | 1·490 | 0·016 | | | |
| C(6)-C(8) | 1·495 | 0·015 | | | |

| | | | | |
|---|---|---|---|---|
| i | atom at | $1-x$ | $y$ | $\frac{1}{2}-z$ |
| ii | atom at | $x$ | $-y$ | $\frac{1}{2}+z$ |
| iii | atom at | $x$ | $1+y$ | $z$ |
| iv | atom at | $1-x$ | $1-y$ | $-z$ |
| v | atom at | $x$ | $1-y$ | $\frac{1}{2}+z$ |
| vi | atom at | $\frac{1}{2}-x$ | $-\frac{1}{2}+y$ | $\frac{1}{2}-z$ |
| vii | atom at | $-x$ | $1-y$ | $-z$ |
| viii | atom at | $\frac{1}{2}-x$ | $\frac{1}{2}-y$ | $-z$ |

Fig. 2 illustrates the arrangement of the molecules in the unit cell, as seen along [100]. The packing of the molecules does not present outstanding peculiarities; the weak intermolecular interactions, which are of the van der Waals type, explain very well the softness of the crystals.

Each complex molecule is formed by three dithiocarbamic ligands octahedrally coordinated to a cobalt

Table 5. *Bond angles, with standard deviations*

| | Bond angle | e.s.d. |
|---|---|---|
| S(2)-Co—S(2$^{\text{i}}$) | 76°46′ | 9′ |
| S(1)-Co—S(3) | 75 55 | 5 |
| Co—S(2)-C(1) | 86 50 | 22 |
| Co—S(1)-C(4) | 86 59 | 17 |
| Co—S(3)-C(4) | 87 5 | 14 |
| S(2)-C(1)-S(2$^{\text{i}}$) | 109 34 | 42 |
| S(2)-C(1)-N(1) | 125 13 | 21 |
| S(1)-C(4)-S(3) | 110 2 | 26 |
| S(1)-C(4)-N(2) | 124 35 | 38 |
| S(3)-C(4)-N(2) | 125 24 | 35 |
| C(1)-N(1)-C(2) | 119 37 | 35 |
| C(2)-N(1)-C(2$^{\text{i}}$) | 120 46 | 1°11′ |
| C(4)-N(2)-C(5) | 119 13 | 43 |
| C(4)-N(2)-C(6) | 120 33 | 38 |
| C(5)-N(2)-C(6) | 119 30 | 41 |
| N(1)-C(2)-C(3) | 109 32 | 1°1 |
| N(2)-C(5)-C(7) | 112 0 | 49 |
| N(2)-C(6)-C(8) | 114 50 | 55 |

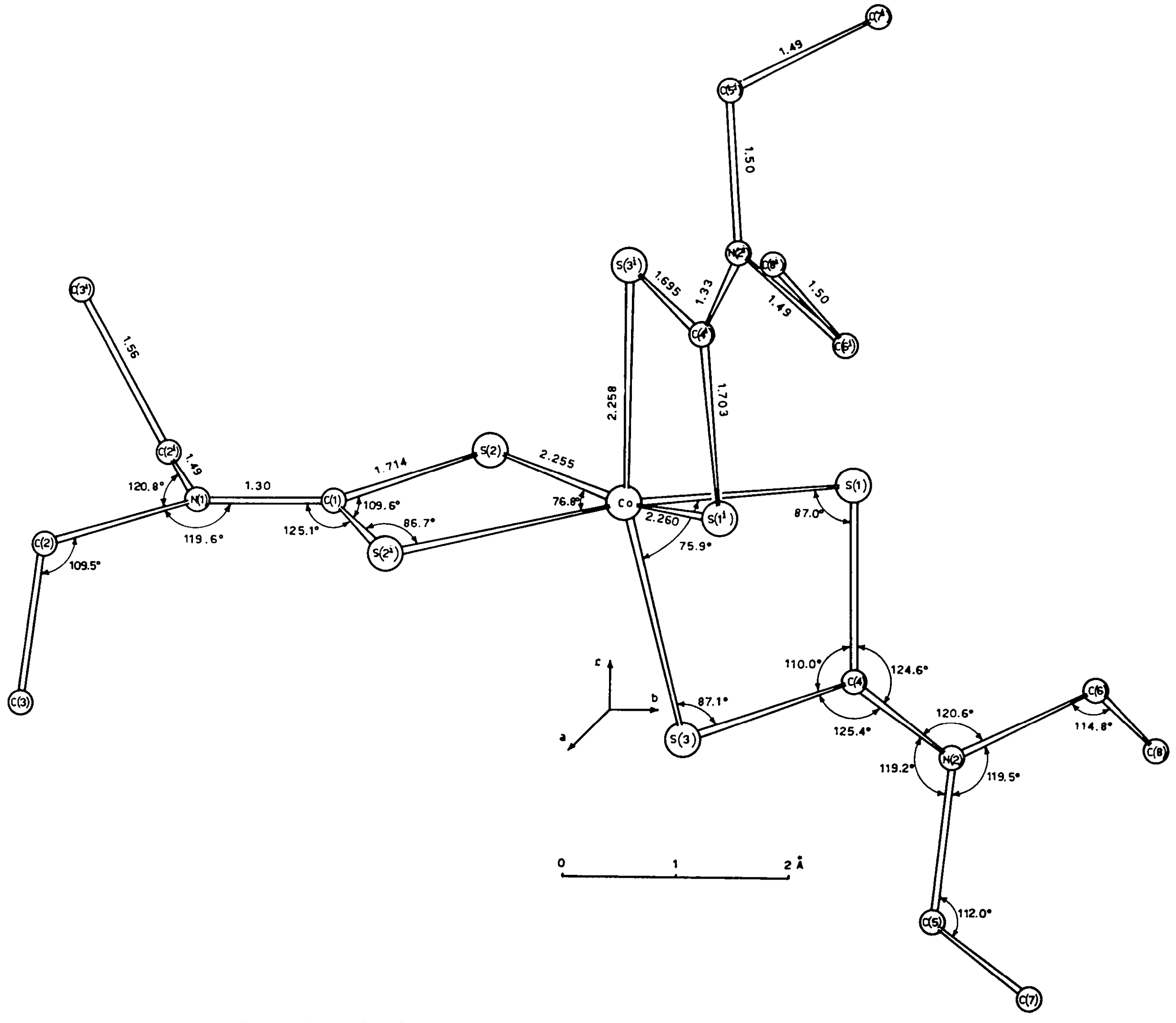


Fig. 1. The molecular structure of cobalt(III) tris(*N*,*N*-diethyldithiocarbamate).

atom through the sulphur atoms. Distances and angles in the cobalt coordination sphere are reported in Table 6: the octahedron formed by the sulphur atoms is distorted with trigonal symmetry. Except for the terminal methyl groups, the whole molecule has nearly trigonal symmetry and the deviations are probably due to the intermolecular interactions which assure the best packing in the crystal.

Table 6. *Distances and angles in the cobalt coordination sphere*

Values of interatomic distances and bond angles not crystallographically independent are given in parentheses: they are reported to emphasize the trigonal symmetry.

| | | | |
|---|---|---|---|
| Co —S(1) | 2·260 Å | S(1) –Co–S(3) | 75° 54′ |
| Co —S(2) | 2·255 | S(2) –Co–S(2$^{i}$) | 76 48 |
| Co —S(3) | 2·258 | S(1$^{i}$)–Co–S(3$^{i}$) | (75 54) |
| S(1) –S(3) | 2·779 | S(1) –Co–S(2) | 94 17 |
| S(2) –S(2$^{i}$) | 2·801 | S(2) –Co–S(3$^{i}$) | 94 31 |
| S(3$^{i}$)–S(1$^{i}$) | (2·779) | S(3$^{i}$)–Co–S(1) | 94 38 |
| S(1$^{i}$)–S(1) | 3·366 | S(1) –Co–S(1$^{i}$) | 96 17 |
| S(3) –S(2) | 3·366 | S(2) –Co–S(3) | 96 27 |
| S(2$^{i}$)–S(3$^{i}$) | (3·366) | S(2$^{i}$)–Co–S(3$^{i}$) | (96 27) |
| S(1) –S(2) | 3·310 | | |
| S(2) –S(3$^{i}$) | 3·314 | | |
| S(3$^{i}$)–S(1) | 3·321 | | |

The distances between sulphur atoms belonging to different dithiocarbamic ligands, 3·31 and 3·37 Å, correspond to the shortest values observed for S···S contacts and are comparable to those observed in zinc ethylxanthate, 3·38 Å (Ikeda & Hagihara, 1966), in nickel bis(dithiocarbamate), 3·41 and 3·44 Å (Fava Gasparri, Nardelli & Villa, 1967), in bisthioureanickel thiocyanate, 3·40 and 3·45 Å (Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966), in $K_2Co(NCS)_4.4H_2O$, 3·43 Å (Zhdanov & Zvonkova, 1950), in rhodanine, 3·47 Å (van der Helm, Lessor & Merritt, 1960), and are consistent with the value 1·72–1·73 Å proposed by van der Helm, Lessor & Merritt (1960) for the van der Waals radius of the sulphur atom.

The atoms of each dithiocarbamic ligand, apart from the terminal methyl groups, lie approximately in a plane with the central cobalt atom (Table 7). The deviations from the plane are of the same order of those found in nickel, copper and zinc diethyldithiocarbamates by Bonamico, Dessy, Mariani, Vaciago & Zambonelli (1965), Bonamico, Dessy, Mugnoli, Vaciago &

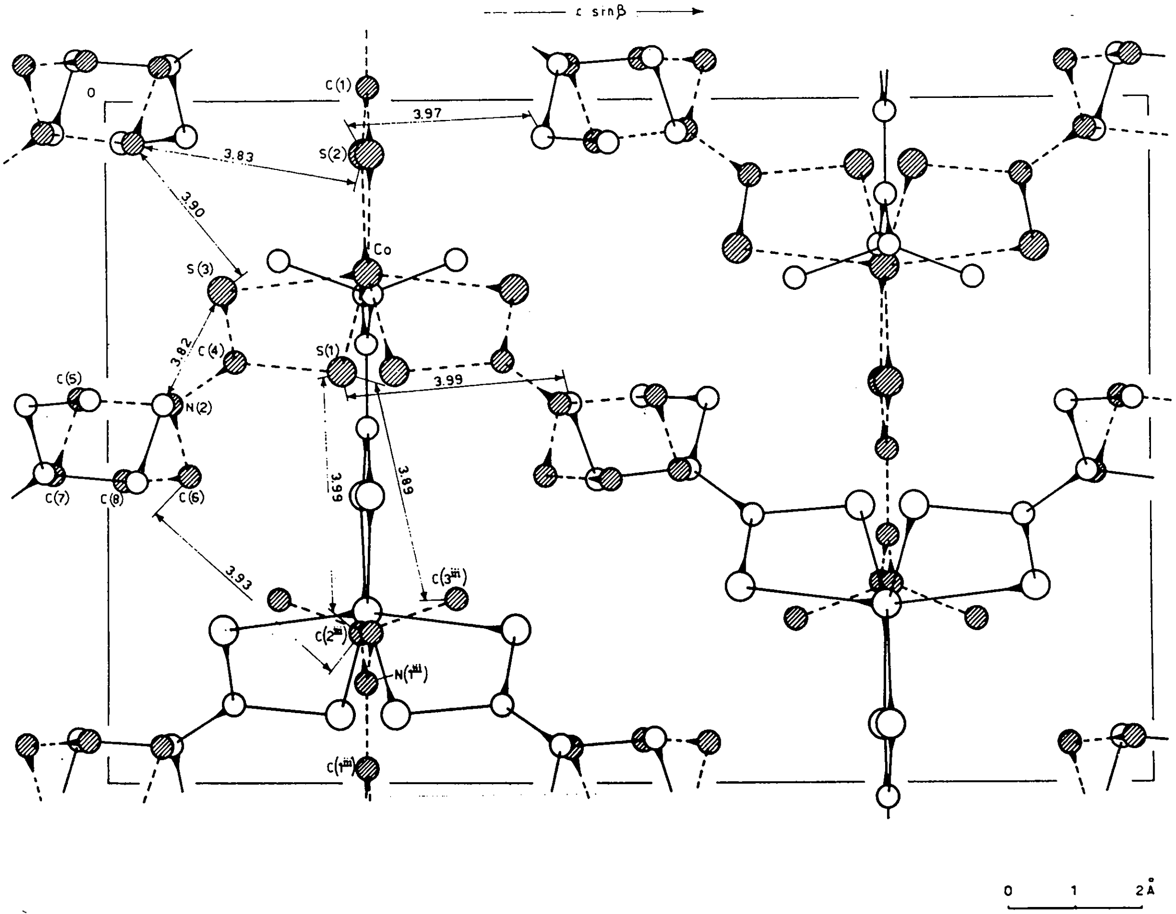
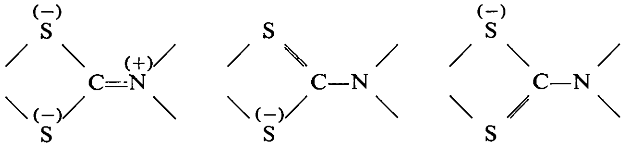


Fig. 2. [100] projection of the unit cell. Atoms and interatomic bonds in the two molecular units with central Co atoms at ($\frac{1}{2}$, 0·2517, $\frac{1}{4}$) and (0, 0·2483, $\frac{3}{4}$) are represented respectively as hatched circles and dashed lines.

Zambonelli (1965), and Bonamico, Mazzone, Vaciago & Zambonelli (1965).

The most interesting bonds are Co–S, S–C and C–N. The mean value of Co–S bond lengths is 2·258 Å, which is equal to the sum of the covalent radii of cobalt(III) and sulphur atoms, 1·22 and 1·04 Å respectively (Pauling, 1960). The mean values of S–C and C–N bond lengths are 1·704 and 1·319 Å respectively. Planarity, bond angle and bond length values indicate conjugation with $sp^2$ hybridization of the valence orbitals of C(1), N(1), C(4) and N(2) atoms. Bond distances in the diethyldithiocarbamate ligand, calculated in terms of the valence-bond theory, agree very well with those observed, assuming the following contributions of the resonance structures:



(I) 42% (II) 29% (III) 29%

Bond order-bond length curves were derived with the use of the Pauling relation:

$$r_x = r_1 - (r_1 - r_2)\frac{3x}{2x+1}\,.$$

It was assumed that for the S–C bond $r_1 = 1{\cdot}812$ Å and $r_2 = 1{\cdot}607$ Å (Pauling, 1960), for the C–N bond $r_1 = 1{\cdot}475$ Å (*International Tables for X-ray Crystallography*, 1962) and $r_2 = 1{\cdot}24$ Å, following the suggestion of Vaughan & Donohue (1952), Wheatley (1955), Hahn (1957), and Marsh, Bierstedt & Eichorn (1962). The calculated values are reported in Table 4 for comparison with the observed values.

In Table 9 the bond lengths within the dithiocarbamic group in various compounds are compared: it appears that the C–N bond always presents a high degree of double-bond character. The close similarity of the bond lengths (Table 9) in the M(ligand)$_3$ complex studied in this work and in the various other M(ligand)$_2$ complexes is noteworthy: it means that the contributions of the three resonance structures are nearly insensitive not only to the nature of the central metal atom but also to changes in stereochemistry.

Table 8. *Shortest intermolecular distances*

The shortest intermolecular S···C distances are given also in Fig. 2.

| | |
|---|---|
| S(1) ···C(2$^{iii}$) | 3·99 Å |
| S(1) ···C(3$^{iii}$) | 3·89 |
| S(1) ···C(7$^{v}$) | 3·99 |
| S(2) ···C(5$^{viii}$) | 3·83 |
| S(2) ···C(6$^{vi}$) | 3·97 |
| S(3) ···C(5$^{viii}$) | 3·90 |
| S(3) ···C(7$^{iv}$) | 3·82 |
| C(3) ···C(5$^{ii}$) | 3·75 |
| C(3) ···C(7$^{ii}$) | 3·80 |
| C(4) ···C(5$^{viii}$) | 3·98 |
| C(6) ···C(2$^{iii}$) | 3·93 |
| C(8) ···C(8$^{vii}$) | 3·55 |

As Alderman, Owston & Rowe (1962) first pointed out, the high contribution of the resonance structure (I) confirms the attribution, made by Chatt, Duncanson & Venanzi (1956), of the strong absorption band in the region 1542 – 1480 $\text{cm}^{-1}$ of the infrared spectra of dithiocarbamic acid derivatives, to a polar C–N partial double bond.

The two-dimensional Patterson and Fourier syntheses were calculated on the Calcolatrice Elettronica Pisana (C.E.P.). All the other calculations were performed on the IBM 7090 computer of the Centro Nazionale Universitario di Calcolo Elettronico (Pisa).

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Table 7. *Least-squares planes*

Equations in the form $Ax + By + Cz = D$, where $x$, $y$ and $z$ are fractional coordinates of the monoclinic cell, calculated after Schomaker, Waser, Marsh & Bergman (1959), with all weights equal to 1.

| Plane | $A$ | $B$ | $C$ | $D$ | Atoms defining the plane |
|---|---|---|---|---|---|
| I | 9·5194 | 7·4665 | −2·0240 | 6·0986 | Co, S(1), S(3), C(4), N(2), C(5), C(6) |
| II | 0·1710 | 0·0 | 16·4098 | 3·9270 | Co, S(2), S(2$^{i}$), C(1), N(1), C(2), C(2$^{i}$) |

Deviations from plane

| I | |
|---|---|
| Co | 0·033 Å |
| S(1) | −0·020 |
| S(3) | 0·014 |
| C(4) | −0·067 |
| N(2) | −0·043 |
| C(5) | 0·023 |
| C(6) | 0·060 |

| II | |
|---|---|
| Co | 0·0 Å |
| S(2) S(2$^{i}$) | ±0·012 |
| C(1) | 0·0 |
| N(1) | 0·0 |
| C(2) C(2$^{i}$) | ∓0·013 |

Table 9. *Comparison of the bond lengths within the dithiocarbamic group in various compounds*

| | S–C | | C–N | | N–C | |
|---|---|---|---|---|---|---|
| | Mean value | e.s.d. | Mean value | e.s.d. | Mean value | e.s.d. |
| $Ni[S_2CN(C_2H_5)_2]_2$. Bonamico, Dessy, Mariani *et al.* (1965) | 1·707 Å | 0·005 Å | 1·330 Å | 0·010 Å | 1·485 Å | 0·011 Å |
| $Cu[S_2CN(C_2H_5)_2]_2$. Bonamico, Dessy, Mugnoli *et al.* (1965) | 1·717 | 0·004 | 1·333 | 0·010 | 1·470 | 0·005 |
| $Cu[S_2CN(C_2H_5)_2]_2$. O'Connor & Maslen (1966) | 1·722 | 0·005 | 1·319 | 0·008 | 1·483 | 0·008 |
| $Zn[S_2CN(C_2H_5)_2]_2$. Bonamico, Mazzone *et al.* (1965) | 1·727 | 0·005 | 1·325 | 0·009 | 1·473 | 0·007 |
| $Ni[S_2CN(C_3H_7)_2]_2$. Peyronel & Pignedoli (1967) | 1·708 | 0·006 | 1·330 | 0·009 | 1·470 | 0·007 |
| $Co[S_2CN(C_2H_5)_2]_3$. Present work | 1·704 | 0·005 | 1·319 | 0·009 | 1·496 | 0·008 |
| $Zn[S_2CN(CH_3)_2]_2$. Klug (1966) | 1·722 | 0·010 | 1·347 | 0·018 | 1·472 | 0·015 |
| $CuS_2CN(C_2H_5)_2$. Hesse (1963) | 1·708 | 0·018 | 1·408 | 0·033 | 1·483 | 0·025 |
| $Ni(S_2CNH_2)_2$. Nardelli *et al.* (1967) | 1·686 | 0·017 | 1·375 | 0·021 | | |
| $C_6H_5N$ . $Zn[S_2CN(CH_3)_2]_2$. Fraser & Harding (1967) | 1·718 | 0·005 | 1·338 | 0·009 | 1·472 | 0·012 |
| $C_6H_5As[S_2CN(C_2H_5)_2]_2$. Bally (1967) | 1·670<br>1·769 | 0·014<br>0·014 | 1·345 | 0·021 | 1·483 | 0·015 |
| $Co(NO)[S_2CN(CH_3)_2]_2$. Alderman *et al.* (1962) | 1·75 | | 1·25 | | 1·5 | |
| $[(S_2CN(C_2H_5)_2]_2$. Karle *et al.* (1967) | 1·648<br>1·816 | 0·009<br>0·009 | 1·348 | 0·012 | 1·473 | 0·008 |

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