and Witte & Wölfel (1958), are given in Table 2. Here $F'_o$ and $F_o$ are respectively the observed structure factors before and after correcting for extinction. The values have been scaled so that $F_o(400)$, which has been found to have negligible extinction, is equal to the absolute value given by Renninger.

## Concluding remarks

The results indicate that this method of correcting for extinction will work in practice. The main advantage of the method is that it accounts for both primary and secondary extinction in a crystal of any shape. It is applicable to a good accuracy when the extinction is not greater than 25%, but is unsatisfactory for low Bragg angles, say for $2\theta<20°$, depending upon the accuracy of the intensity measurements.

The present experimental arrangement is defective in one respect. The axis of oscillation remaining vertical for all angles of $\varphi$ prevents $\varrho_\varphi$ from being measured over the whole range from $\varphi=90°$ to 0°. (The Cox–Shaw factor becomes zero for $\varphi=0°$). The most sensitive portion of the curve connecting $\varrho'_\varphi/\varrho'_\perp$ versus $\varphi$ is between $\varphi=20°$ and 0°, and hence the inability to make measurements in this range is a disadvantage. This can be overcome either by rotating the entire diffractometer about the polarized beam or by rotating the plane of polarization. An apparatus for accomplishing the latter, designed with a view to making the experimental procedure as efficient as possible, is now under construction at the Royal Institution.

I am indebted to Prof. Dame Kathleen Lonsdale for provision of facilities and for many helpful suggestions. I am also grateful to Dr H. J. Milledge for the loan of the diamonds and for discussions. Thanks are due to Mr E. Nave for his valuable help in setting up the apparatus. The work was carried out during the tenure of a D.S.I.R. Special Research Grant.

## References

Batterman, B. W. (1959). *Phys. Rev.* Lett. **2**, 47.
Bones, R. (1956). *J. Sci. Instrum.* **33**, 241.
Chandrasekhar, S. (1956). *Acta Cryst.* **9**, 954.
Chandrasekhar, S. & Weiss, R. J. (1957). *Acta Cryst.* **10**, 598.
Chandrasekaran, K. S. (1955). *Acta Cryst.* **8**, 361.
Chandrasekaran, K. S. (1959). *Acta Cryst.* **12**, 916.
Cochran, W. (1953). *Acta Cryst.* **6**, 260.
Compton, A. H. & Allison, S. K. (1935). *X-rays in Theory and Experiment.* New York: Van Nostrand.
Hume-Rothery, W., Brown, P. J., Forsyth, J. B. & Taylor, W. H. (1958). *Phil. Mag.* **3**, 1466.
James, R. W. (1954). *The Optical Principles of Diffraction of X-rays.* London: Bell.
Lonsdale, K. (1948). *Acta Cryst.* **1**, 12.
Ramaseshan, S. & Ramachandran, G. N. (1953). *Acta Cryst.* **6**, 364.
Ramaseshan, S. & Ramachandran, G. N. (1954). *Proc. Indian Acad. Sc.* A, **39**, 20.
Renninger, M. (1952). *Acta Cryst.* **5**, 711.
Vand, V. (1955). *J. Appl. Phys.* **26**, 1191.
Weiss, R. J. & De Marco, J. J. (1958). *Rev. Mod. Phys.* **30**, 59.
Weiss, R. J. & De Marco, J. J. (1959). *Phys. Rev.* Lett. **2**, 148.
Witte, H. & Wölfel, E. (1958). *Rev. Mod. Phys.* **30**, 51.

---

*Acta Cryst.* (1960). **13**, 594

# The Crystal Structure of *bis*-Biuret-Cadmium Chloride

By Luigi Cavalca, Mario Nardelli and Giovanna Fava
*Structural Chemistry Laboratory, Institute of Chemistry, University of Parma, Italy*

(*Received* 2 *December* 1959)

*Bis*-biuret-cadmium chloride, $CdCl_2 . 2\, C_2H_5N_3O_2$, is monoclinic ($P2_1/c$) with

$$a=3\cdot704,\ b=19\cdot96,\ c=8\cdot20\ \text{Å};\ \beta=111\cdot1°;\ Z=2\ .$$

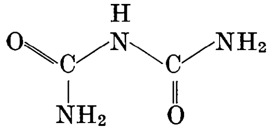
The crystal structure has been determined by Fourier methods using $0kl$, $1kl$ and $2kl$ reflections. The cadmium atom coordinates octahedrally with four Cl and two O atoms of two different biuret molecules (Cd–Cl = 2·55 and 2·62 Å, Cd–O = 2·34 Å). The biuret molecule is planar and has a 'trans' configuration.

## 1. Introduction

Biuret, $NH(CONH_2)_2=Bu$, forms two kinds of coordination compounds with divalent metals. Among those which can be obtained in an alkaline medium, the potassium *bis*-biuret-cuprate tetrahydrate has recently been studied by Freeman, Smith & Taylor (1959), who found that two biuret molecules are coordinated as bidentate ligands, through the nitrogen atoms of the amino-groups, by a copper atom.

In a neutral medium, coordination compounds of the kind $M^{II}X_2.2Bu$, in which $M^{II}X_2 = CuCl_2$, $CuSO_4$, $Cu(NO_3)_2$, $NiCl_2$, $NiSO_4$ (Schiff, 1898), $CdCl_2$ (Schenck, 1904), are formed. The crystal structure of *bis*-biuret-cupric chloride has also been determined by Freeman, Smith & Taylor (1959); in this compound the biuret is already a bidentate ligand but the coordination probably occurs through the two oxygen atoms.

In this paper the results of the study of the crystal structure of $CdCl_2.2Bu$ (isostructural with the corresponding Hg-compound (Nardelli & Chierici, 1960)) are reported. The complete structural study has showed that in this case the biuret is a mono-dentate ligand and its configuration is *trans*:



## 2. Experimental

*Bis*-biuret-cadmium chloride forms very slender and flattened colourless needles, which are optically biaxial and usually twinned on (001). The unit cell constants are:

$$a = 3{\cdot}704 \pm 0{\cdot}005,\ b = 19{\cdot}96 \pm 0{\cdot}03,\ c = 8{\cdot}20 \pm 0{\cdot}01\ \text{Å},$$
$$\beta = 111{\cdot}1° \pm 0{\cdot}2°,\ Z = 2,\ \text{space group } P2_1/c\,.$$

The intensity data were obtained photometrically from 0*kl*, 1*kl* and 2*kl* integrated and non-integrated Weissenberg photographs (Cu $K\alpha$ radiation) using the multiple-film technique. An untwinned sample, cut from a twinned crystal was used. The cross-section was rectangular (0·12 mm. along $z^*$, 0·008 mm. along $y^*$) and the relative intensities were corrected for absorption by Albrecht's (1939) method. The variation in size of spots on non-equatorial Weissenberg photographs was taken into account following Scouloudi (1953).

After correction for Lorentz, polarization and velocity factors, the $F_o^2(0kl)$ values were put on an absolute scale by Wilson's (1942) method. This gave a scale factor and an isotropic temperature factor which were in quite good agreement with the final ones (scale factor for $F_o$'s, from Wilson 2·571, final value 2·502; $B$ from Wilson 2·1 Å$^2$, final value 1·8 Å$^2$). The 1*kl* and 2*kl* structure factors were successively put on an absolute scale by comparison with the calculated values.

123 0*kl* (possible 196), 237 1*kl* (possible 372), and 213 2*kl* (possible 325) reflections were observed. The majority of the unobserved reflections had $k+l$ odd; this is because the Cd atom lies on a symmetry centre.

## 3. The structure determination

Locating the Cd atom at the origin of the unit cell allowed us to solve the 'phase problem' in a straightforward way. A first $\varrho_0(Y, Z)$ was calculated using the $F_o(0kl)$'s with $k+l$ even, which are influenced strongly by the Cd atom, taken with positive signs. This projection was sufficient to obtain the $y$ and $z$ coordinates of the Cl atom. A succeeding projection, calculated with all the $F_o(0kl)$'s and the signs determined by the contributions of the heavy atoms alone, showed well-resolved peaks corresponding to the light atoms. On examining the resultant projection it was evident that the coordination around the Cd was of the same kind as that previously found in *bis*-acetamide-cadmium chloride, $CdCl_2.2\,OCNH_2CH_3$, (Cavalca, Nardelli & Coghi, 1957) and in *bis*-urea-cadmium chloride, $CdCl_2.2\,OC(NH_2)_2$, (Nardelli, Cavalca & Fava, 1957). It was therefore easy to calculate the $x$ coordinate of the Cl atom.

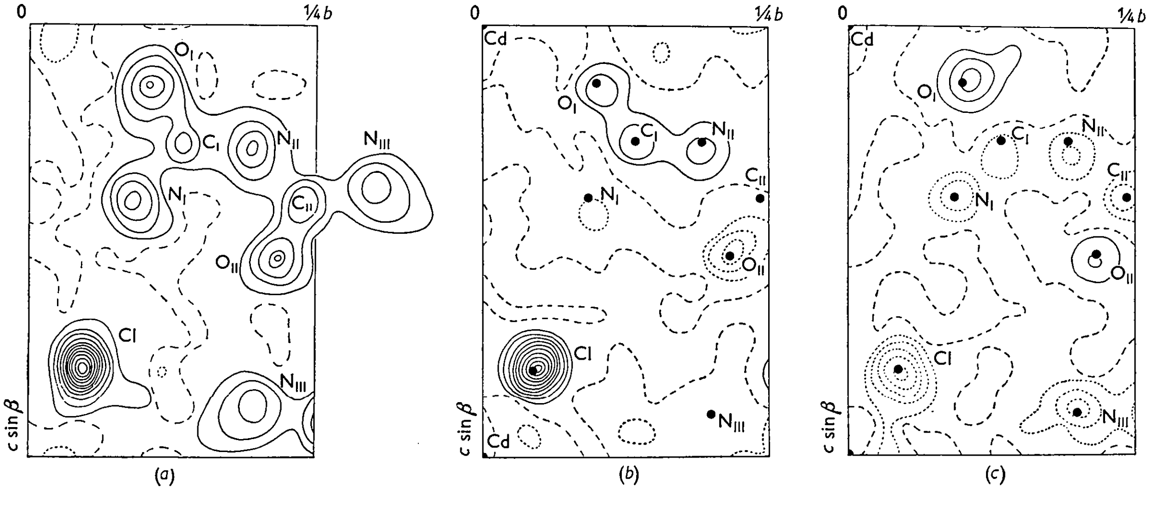


Fig. 1. Projection along [100] of: (*a*) $\varrho_0$, (*b*) $S_1$, (*c*) $C_1$, calculated without the Cd contributions. Contours at intervals of 2 e.Å$^{-2}$. The zero contour is broken, and the negative contours are dotted.

Table 1. *Atomic coordinates and their standard deviations*

| | $x/a$ | $y/b$ | $z/c$ | $x'$ | $y'$ | $z'$ | $\sigma(x)$ | $\sigma(y)$ | $\sigma(z)$ |
|---|---|---|---|---|---|---|---|---|---|
| Cd | 0 | 0 | 0 | 0 | 0 | 0 | — | — | — |
| Cl | 0·3234 | 0·0446 | −0·2053 | 1·803 | 0·890 | −1·570 | 0·005 | 0·005 | 0·011 |
| $O_I$ | 0·104 | 0·105 | 0·135 | −0·014 | 2·096 | 1·033 | 0·007 | 0·017 | 0·029 |
| $O_{II}$ | 0·842 | 0·214 | 0·543 | 1·511 | 4·271 | 4·154 | 0·020 | 0·025 | 0·031 |
| $N_I$ | 0·572 | 0·092 | 0·400 | 0·934 | 1·836 | 3·060 | 0·024 | 0·022 | 0·039 |
| $N_{II}$ | 0·354 | 0·197 | 0·284 | 0·472 | 3·932 | 2·173 | 0·022 | 0·023 | 0·029 |
| $N_{III}$ | 0·510 | 0·304 | 0·393 | 0·726 | 6·068 | 3·007 | 0·023 | 0·033 | 0·024 |
| $C_I$ | 0·337 | 0·130 | 0·268 | 0·456 | 2·595 | 2·050 | 0·031 | 0·038 | 0·048 |
| $C_{II}$ | 0·579 | 0·238 | 0·410 | 0·934 | 4·750 | 3·136 | 0·017 | 0·023 | 0·045 |

A series of projections of $\varrho_0$, $C_1$, $S_1$, $\varrho_1$, $C_2$, $S_2$, $\varrho_2$ (the suffixes 0, 1, 2 refer to the $0kl$, $1kl$ and $2kl$ reflections used for calculating the indicated functions respectively) along [100], calculated using the $F_o-F_{\text{Cd}}$ values and the signs determined by the heavy atoms, was useful in getting an improved distribution of the light atoms. The $\varrho_0$, $C_1$, and $S_1$ projections obtained at this stage are shown in Fig. 1.

The values of the $\varrho_0$, $C_1$, $S_1$, $C_2$, and $S_2$ functions at the $i$ points near the projections of the atomic centres were used to calculate the $\varrho(X, Y_i, Z_i)$ function. This function is specially influenced by series-termination errors because of the neglect of $3kl$ and $4kl$ reflections; nevertheless, it was valuable in giving the $x$ coordinates of the light atoms and in improving the other coordinates. To correct the series-termination errors, the $\{\varrho_o-\varrho_c\}(X, Y_i, Z_i)$ function was calculated several times until nearly stationary coordinates were obtained. At the end of these refinement operations the atomic coordinates were those reported in Table 1; in this table are also given the $x'$, $y'$, $z'$ (Å) coordinates referred to orthogonal axes $a, b, c'$, with $c'$ perpendicular to the $a$ and $b$ crystal axes.

The standard deviations $\sigma(y)$ and $\sigma(z)$ reported (Å) are the mean values of those estimated by Cruickshank's (1949) method from $\varrho_0(Y, Z)$, $\varrho_1(Y, Z)$, and $\varrho_2(Y, Z)$; $\sigma(x)$ was calculated by the same method from $\varrho(X, Y, Z)$. The standard deviations of the electron density in the three projections along [100] are: $\sigma(\varrho_0)=0{\cdot}51$, $\sigma(\varrho_1)=0{\cdot}69$, $\sigma(\varrho_2)=0{\cdot}68$ e.Å$^{-2}$ and for the three-dimensional data $\sigma(\varrho)=0{\cdot}30$ e.Å$^{-3}$.

In Table 2 are given the final values of $F_o$ and $F_c$.

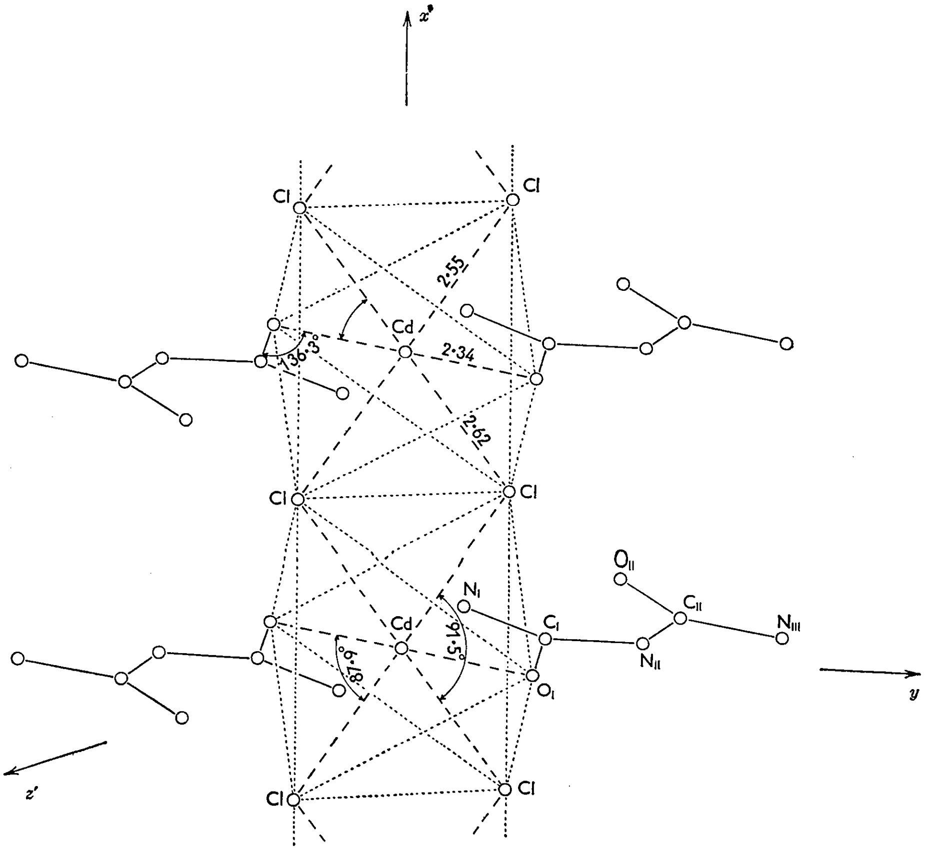


Fig. 2. A chain of coordination octahedra seen in clinographic projection.

Table 2. *Calculated and observed structure factors*

A negative sign after an $|F_o|$ must be interpretated as ‘less than’

| l | $10\lvert F_o\rvert$ | $10\,F_c$ |
|---|---|---|
| | 0 0 l | |
| 2 | 397 | 354 |
| 4 | 583 | 590 |
| 6 | 277 | 266 |
| 8 | 187 | 263 |
| | 0 1 l | |
| 1 | 992 | 1005 |
| 2 | 401 | 390 |
| 3 | 307 | 314 |
| 4 | 94- | -157 |
| 5 | 735 | 759 |
| 6 | 125- | 33 |
| 7 | 280 | 299 |
| 8 | 122- | -262 |
| 9 | 97- | 293 |
| | 0 2 l | |
| 0 | 563 | 735 |
| 1 | 269 | -266 |
| 2 | 323 | 296 |
| 3 | 79- | -68 |
| 4 | 654 | 679 |
| 5 | 111- | 71 |
| 6 | 571 | 564 |
| 7 | 132- | 151 |
| 8 | 235 | 239 |
| 9 | 95- | -79 |
| | 0 3 l | |
| 1 | 972 | 1042 |
| 2 | 66- | 0 |
| 3 | 591 | 578 |
| 4 | 97- | -98 |
| 5 | 867 | 894 |
| 6 | 317 | 321 |
| 7 | 132- | 109 |
| 8 | 121- | -70 |
| 9 | 262 | 298 |
| | 0 4 l | |
| 0 | 825 | 1000 |
| 1 | 346 | 384 |
| 2 | 848 | 885 |
| 3 | 749 | -766 |
| 4 | 741 | 753 |
| 5 | 113- | 83 |
| 6 | 394 | 426 |
| 7 | 131- | 106 |
| 8 | 120- | 153 |
| 9 | 92- | -139 |
| | 0 5 l | |
| 1 | 726 | 695 |
| 2 | 365 | 342 |
| 3 | 623 | 619 |
| 4 | 215 | -244 |
| 5 | 438 | 370 |
| 6 | 128- | 133 |
| 7 | 287 | 304 |
| 8 | 118- | -156 |
| 9 | 271 | 303 |
| | 0 6 l | |
| 0 | 378 | 372 |
| 1 | 754 | 764 |
| 2 | 576 | 568 |
| 3 | 91- | 51 |
| 4 | 772 | 756 |
| 5 | 117- | 71 |
| 6 | 441 | 452 |
| 7 | 131- | 42 |
| 8 | 243 | 277 |
| 9 | 85- | -30 |
| | 0 7 l | |
| 1 | 983 | 958 |
| 2 | 374 | 327 |
| 3 | 854 | 832 |
| 4 | 426 | -394 |
| 5 | 394 | 394 |
| 6 | 169 | 162 |
| 7 | 310 | 338 |
| 8 | 113- | -66 |
| 9 | 80- | 165 |
| | 0 8 l | |
| 0 | 389 | 427 |
| 1 | 637 | 540 |
| 2 | 866 | 836 |
| 3 | 98- | -113 |
| 4 | 439 | 426 |
| 5 | 122- | -110 |
| 6 | 349 | 359 |
| 7 | 128- | -57 |
| 8 | 307 | 352 |
| 9 | 74- | -118 |
| | 0 9 l | |
| 1 | 453 | 409 |
| 2 | 94- | 8 |
| 3 | 586 | 614 |
| 4 | 114- | -120 |
| 5 | 207 | 221 |
| 6 | 181 | 168 |
| 7 | 542 | 564 |
| 8 | 106- | 6 |
| 9 | 67- | 151 |
| | 0 10 l | |
| 0 | 916 | 834 |
| 1 | 93- | 17 |
| 2 | 883 | 876 |
| 3 | 108- | 9 |
| 4 | 221 | 236 |
| 5 | 128- | 54 |
| 6 | 326 | 313 |
| 7 | 123- | 107 |
| 8 | 314 | 383 |
| 9 | 56- | -29 |
| | 0 11 l | |
| 1 | 706 | 659 |
| 2 | 105- | -61 |
| 3 | 728 | 725 |
| 4 | 122- | -126 |
| 5 | 219 | 156 |
| 6 | 131- | 39 |
| 7 | 460 | 454 |
| 8 | 95- | -98 |
| | 0 12 l | |
| 0 | 103- | 41 |
| 1 | 232 | -179 |
| 2 | 910 | 840 |
| 3 | 117- | 35 |
| 4 | 224 | 183 |
| 5 | 131- | 22 |
| 6 | 331 | 311 |
| 7 | 115- | -56 |
| 8 | 270 | 310 |
| | 0 13 l | |
| 1 | 305 | 242 |
| 2 | 116- | -42 |
| 3 | 649 | 660 |
| 4 | 322 | 287 |
| 5 | 487 | 367 |
| 6 | 126- | -54 |
| 7 | 320 | 346 |
| 8 | 79- | 63 |
| | 0 14 l | |
| 0 | 301 | 277 |
| 1 | 180 | -194 |
| 2 | 747 | 668 |
| 3 | 126- | -187 |
| 4 | 475 | 486 |
| 5 | 131- | 50 |
| 6 | 282 | 277 |
| 7 | 103- | 34 |
| 8 | 194 | 222 |
| | 0 15 l | |
| 1 | 510 | 470 |
| 2 | 126- | -136 |
| 3 | 587 | 546 |
| 4 | 132- | 113 |
| 5 | 129- | 71 |
| 6 | 224 | -228 |
| 7 | 288 | 300 |
| | 0 16 l | |
| 0 | 330 | 344 |
| 1 | 189 | -187 |
| 2 | 354 | 309 |
| 3 | 386 | 345 |
| 4 | 411 | 383 |
| 5 | 125- | -82 |
| 6 | 236 | 230 |
| 7 | 84- | -76 |
| | 0 17 l | |
| 1 | 459 | 443 |
| 2 | 131- | 61 |
| 3 | 381 | 352 |
| 4 | 154 | 163 |
| 5 | 327 | 333 |
| 6 | 102- | -122 |
| 7 | 175 | 202 |
| | 0 18 l | |
| 0 | 391 | 361 |
| 1 | 212 | -197 |
| 2 | 427 | 377 |
| 3 | 129- | 121 |
| 4 | 317 | 321 |
| 5 | 111- | -117 |
| 6 | 311 | 318 |
| | 0 19 l | |
| 1 | 438 | 397 |
| 2 | 216 | -214 |
| 3 | 169 | 135 |
| 4 | 119 | 83 |
| 5 | 286 | 285 |
| 6 | 78- | -109 |
| | 0 20 l | |
| 0 | 569 | 588 |
| 1 | 127- | -47 |
| 2 | 220 | 181 |
| 3 | 149 | 110 |
| 4 | 199 | 221 |
| 5 | 91- | 80 |
| 6 | 175 | 206 |
| | 0 21 l | |
| 1 | 348 | 354 |
| 2 | 117- | -65 |
| 3 | 196 | 194 |
| 4 | 97- | 54 |
| 5 | 327 | 358 |
| | 0 22 l | |
| 0 | 413 | 441 |
| 1 | 112- | -70 |
| 2 | 156 | 134 |
| 3 | 98- | -49 |
| 4 | 284 | 278 |
| | 0 23 l | |
| 1 | 237 | 209 |
| 2 | 95- | 22 |
| 3 | 117 | 141 |
| 4 | 64- | 24 |
| | 0 24 l | |
| 0 | 356 | 351 |
| 1 | 87- | 55 |
| 2 | 123 | 124 |
| 3 | 65- | -48 |
| | 0 25 l | |
| 1 | 223 | 241 |
| 2 | 54- | 53 |
| | 1 0 l | |
| 0 | 161 | 132 |
| 2 | 904 | 1036 |
| -2 | 1216 | 974 |
| 4 | 196 | 249 |
| -4 | 1103 | 1018 |
| 6 | 778 | 889 |
| -6 | 165 | 182 |
| 8 | 148 | 173 |
| -8 | 489 | 497 |
| -10 | 71- | -44 |
| | 1 1 l | |
| 0 | 105 | -142 |
| 1 | 980 | 1059 |
| -1 | 674 | 688 |
| 2 | 70- | -81 |
| -2 | 1527 | 1270 |
| 3 | 393 | 436 |
| -3 | 986 | 860 |
| 4 | 100- | -144 |
| -4 | 76- | 3 |
| 5 | 314 | 309 |
| -5 | 214 | 337 |
| 6 | 119- | -22 |
| -6 | 105- | -21 |
| 7 | 338 | 423 |
| -7 | 387 | 420 |
| 8 | 86- | 70 |
| -8 | 117- | -149 |
| -9 | 361 | 434 |
| -10 | 71- | 20 |
| | 1 2 l | |
| 0 | 738 | 857 |
| 1 | 306 | -308 |
| -1 | 229 | -238 |
| 2 | 737 | 749 |
| -2 | 846 | 744 |
| 3 | 87- | 81 |
| -3 | 235 | 182 |
| 4 | 189 | 158 |
| -4 | 753 | 700 |
| 5 | 117 | 115 |
| -5 | 200 | -179 |
| 6 | 344 | 368 |
| -6 | 291 | 320 |
| 7 | 109- | -2 |
| -7 | 117- | 29 |
| 8 | 86- | 200 |
| -8 | 468 | 550 |
| -9 | 101- | 9 |
| -10 | 148 | 238 |
| | 1 3 l | |
| 0 | 535 | -626 |
| 1 | 1198 | 1293 |
| -1 | 240 | 225 |
| 2 | 73- | 21 |
| -2 | 359 | 297 |
| 3 | 564 | 545 |
| -3 | 1234 | 1171 |
| 4 | 102- | 82 |
| -4 | 312 | -302 |
| 5 | 360 | 393 |
| -5 | 236 | 224 |
| 6 | 118- | 0 |
| -6 | 106- | -50 |
| 7 | 340 | 404 |
| -7 | 380 | 420 |
| 8 | 84- | 69 |
| -8 | 115- | 32 |
| -9 | 215 | 262 |
| -10 | 68- | 6 |
| | 1 4 l | |
| 0 | 531 | 585 |
| 1 | 297 | -285 |
| -1 | 51- | 153 |
| 2 | 873 | 918 |
| -2 | 623 | 581 |
| 3 | 569 | 571 |
| -3 | 207 | 185 |
| 4 | 520 | 536 |
| -4 | 865 | 830 |
| 5 | 245 | -257 |
| -5 | 184 | -160 |
| 6 | 343 | 388 |
| -6 | 357 | 350 |
| 7 | 107- | 39 |
| -7 | 263 | 320 |
| 8 | 193 | 241 |
| -8 | 289 | 311 |
| -9 | 163 | -154 |
| -10 | 113 | 149 |
| | 1 5 l | |
| 0 | 157 | -120 |
| 1 | 300 | 318 |
| -1 | 335 | 284 |
| 2 | 78- | 109 |
| -2 | 547 | 532 |
| 3 | 572 | 586 |
| -3 | 960 | 890 |
| 4 | 105- | -57 |
| -4 | 84- | -100 |
| 5 | 425 | 444 |
| -5 | 802 | 872 |
| 6 | 117- | -152 |
| -6 | 110- | 17 |
| 7 | 188 | 218 |
| -7 | 341 | 381 |
| 8 | 78- | 139 |
| -8 | 114- | 32 |
| -9 | 161 | 204 |
| -10 | 62- | -122 |
| | 1 6 l | |
| 0 | 661 | 707 |
| 1 | 283 | -302 |
| -1 | 62- | -56 |
| 2 | 850 | 812 |
| -2 | 655 | 612 |
| 3 | 208 | 152 |
| -3 | 75- | -61 |
| 4 | 451 | 460 |
| -4 | 548 | 540 |
| 5 | 188 | -217 |
| -5 | 306 | -278 |
| 6 | 223 | 222 |
| -6 | 373 | 428 |
| 7 | 201 | 198 |
| -7 | 173 | 159 |
| 8 | 75- | 171 |
| -8 | 256 | 313 |
| -9 | 94- | -120 |
| -10 | 141 | 248 |
| | 1 7 l | |
| 0 | 434 | -346 |
| 1 | 630 | 596 |
| -1 | 932 | 874 |
| 2 | 333 | 299 |
| -2 | 154 | 136 |
| 3 | 653 | 656 |
| -3 | 523 | 512 |
| 4 | 109- | 22 |
| -4 | 359 | -334 |
| 5 | 448 | 467 |
| -5 | 506 | 516 |
| 6 | 115- | -115 |
| -6 | 174 | 208 |
| 7 | 248 | 308 |
| -7 | 259 | 278 |
| 8 | 71- | 135 |
| -8 | 111- | 85 |
| -9 | 164 | 184 |
| | 1 8 l | |
| 0 | 913 | 882 |
| 1 | 81- | 22 |
| -1 | 355 | 352 |
| 2 | 278 | 264 |
| -2 | 730 | 730 |
| 3 | 220 | 164 |
| -3 | 208 | 213 |
| 4 | 517 | 507 |
| -4 | 318 | 315 |
| 5 | 317 | -317 |
| -5 | 105- | -105 |
| 6 | 232 | 236 |
| -6 | 568 | 577 |
| 7 | 98- | 81 |
| -7 | 146 | 182 |
| 8 | 188 | 277 |
| -8 | 201 | 229 |
| -9 | 198 | -163 |
| | 1 9 l | |
| 0 | 255 | -203 |
| 1 | 370 | 349 |
| -1 | 1141 | 1133 |
| 2 | 265 | 230 |
| -2 | 160 | -104 |
| 3 | 430 | 394 |
| -3 | 181 | 187 |
| 4 | 113- | 2 |
| -4 | 179 | -199 |
| 5 | 373 | 313 |
| -5 | 526 | 549 |
| 6 | 112- | -74 |
| -6 | 117- | 125 |
| 7 | 94- | 143 |
| -7 | 311 | 385 |
| 8 | 59- | 30 |
| -8 | 106- | 45 |
| -9 | 223 | 293 |
| | 1 10 l | |
| 0 | 700 | 691 |
| 1 | 92- | -132 |
| -1 | 86- | -138 |
| 2 | 332 | 290 |
| -2 | 745 | 685 |
| 3 | 108- | 60 |
| -3 | 95- | -44 |
| 4 | 700 | 648 |
| -4 | 325 | 329 |
| 5 | 118- | 14 |
| -5 | 111- | -135 |
| 6 | 259 | 300 |
| -6 | 441 | 515 |
| 7 | 90- | 23 |
| -7 | 115- | 51 |
| -8 | 103- | 124 |
| -9 | 77- | -3 |
| | 1 11 l | |
| 0 | 190 | -116 |
| 1 | 412 | 387 |
| -1 | 892 | 920 |
| 2 | 103- | -7 |
| -2 | 447 | 368 |
| 3 | 494 | 494 |
| -3 | 253 | 220 |
| 4 | 117- | 34 |
| -4 | 106- | 4 |
| 5 | 415 | 461 |
| -5 | 231 | 470 |
| 6 | 106- | 19 |
| -6 | 118- | 19 |
| 7 | 84- | 154 |
| -7 | 305 | 319 |
| -8 | 98- | -40 |
| -9 | 114 | 130 |
| | 1 12 l | |
| 0 | 660 | 608 |
| 1 | 102- | -40 |
| -1 | 97- | 97 |
| 2 | 108- | 16 |
| -2 | 536 | 520 |
| 3 | 114- | -122 |
| -3 | 175 | 187 |
| 4 | 561 | 590 |
| -4 | 385 | 384 |
| 5 | 114- | 106 |
| -5 | 117- | 44 |
| 6 | 102- | 85 |
| -6 | 567 | 638 |
| 7 | 77- | -33 |
| -7 | 111- | -140 |
| -8 | 94- | 105 |
| -9 | 61- | 45 |
| | 1 13 l | |
| 0 | 103- | 112 |
| 1 | 525 | 485 |
| -1 | 665 | 666 |
| 2 | 210 | -252 |
| -2 | 177 | -164 |
| 3 | 526 | 449 |
| -3 | 281 | 280 |
| 4 | 118- | 6 |
| -4 | 113- | 140 |
| 5 | 303 | 251 |
| -5 | 348 | 360 |
| 6 | 97- | 72 |
| -6 | 117- | -215 |
| 7 | 69- | 85 |
| -7 | 253 | 308 |
| -8 | 88- | -19 |
| | 1 14 l | |
| 0 | 570 | 548 |
| 1 | 111- | 66 |
| -1 | 107- | -141 |
| 2 | 489 | 461 |
| -2 | 459 | 434 |
| 3 | 118- | 102 |
| -3 | 188 | -212 |
| 4 | 577 | 535 |
| -4 | 344 | 288 |
| 5 | 108- | 87 |
| -5 | 118- | 134 |
| 6 | 168 | 150 |
| -6 | 381 | 376 |
| 7 | 57- | -69 |
| -7 | 102- | 30 |
| -8 | 80- | 78 |
| | 1 15 l | |
| 0 | 335 | 323 |
| 1 | 114- | 75 |
| -1 | 510 | 479 |
| 2 | 117- | -57 |
| -2 | 210 | -192 |
| 3 | 492 | 467 |
| -3 | 323 | 304 |
| 4 | 113- | -22 |
| -4 | 244 | 219 |
| 5 | 361 | 360 |
| -5 | 660 | 670 |
| 6 | 82- | 30 |
| -6 | 110- | -24 |
| -7 | 185 | 185 |
| -8 | 70- | -4 |
| | 1 16 l | |
| 0 | 362 | 307 |
| 1 | 254 | 219 |
| -1 | 115- | -53 |
| 2 | 421 | 363 |
| -2 | 421 | 389 |
| 3 | 329 | -314 |
| -3 | 118- | -94 |
| 4 | 275 | 258 |
| -4 | 386 | 379 |
| 5 | 162 | 141 |
| -5 | 114- | 151 |
| 6 | 161 | 189 |
| -6 | 305 | 350 |
| -7 | 181 | -193 |
| -8 | 152 | 225 |
| | 1 17 l | |
| 0 | 118- | 106 |
| 1 | 478 | 435 |
| -1 | 431 | 392 |
| 2 | 117- | -64 |
| -2 | 231 | -197 |
| 3 | 384 | 332 |
| -3 | 333 | 370 |
| 4 | 104- | -71 |
| -4 | 117- | 17 |
| 5 | 253 | 253 |
| -5 | 235 | 225 |
| 6 | 61- | 78 |
| -6 | 99- | 12 |
| -7 | 168 | 208 |
| | 1 18 l | |
| 0 | 505 | 516 |
| 1 | 256 | 209 |
| -1 | 118- | -8 |
| 2 | 348 | 353 |
| -2 | 381 | 350 |
| 3 | 182 | -156 |
| -3 | 117- | -63 |
| 4 | 244 | 229 |
| -4 | 240 | 206 |

Table 2 (*cont.*)

| l | $10\lvert F_o\rvert$ | $10\,F_c$ |
|---|---|---|
| 5 | 127 | 107 |
| -5 | 105- | 155 |
| -6 | 192 | 159 |
| -7 | 68- | -85 |
| | 1 19 1 | |
| 0 | 187 | 169 |
| 1 | 353 | 355 |
| -1 | 207 | 211 |
| 2 | 110- | 1 |
| -2 | 290 | -273 |
| 3 | 148 | 119 |
| -3 | 419 | 457 |
| 4 | 89- | 48 |
| -4 | 107- | 136 |
| 5 | 177 | 196 |
| -5 | 210 | 189 |
| -6 | 81- | 12 |
| -7 | 193 | 293 |
| | 1 20 1 | |
| 0 | 226 | 197 |
| 1 | 108- | -12 |
| -1 | 111- | 5 |
| 2 | 355 | 330 |
| -2 | 319 | 304 |
| 3 | 94- | -115 |
| -3 | 106- | -27 |
| 4 | 146 | 118 |
| -4 | 369 | 378 |
| -5 | 88- | 14 |
| -6 | 122 | 133 |
| | 1 21 1 | |
| 0 | 104- | 27 |
| 1 | 475 | 451 |
| -1 | 200 | 198 |
| 2 | 94- | -84 |
| -2 | 103- | -18 |
| 3 | 269 | 260 |
| -3 | 352 | 332 |
| 4 | 63- | 43 |
| -4 | 90- | 71 |
| -5 | 97 | 97 |
| | 1 22 1 | |
| 0 | 179 | 152 |
| 1 | 92- | -10 |
| -1 | 97- | 33 |
| 2 | 321 | 330 |
| -2 | 233 | 244 |
| 3 | 69- | -9 |
| -3 | 89- | 66 |
| -4 | 314 | 349 |
| -5 | 61- | 33 |
| | 1 23 1 | |
| 0 | 85- | -27 |
| 1 | 315 | 321 |
| -1 | 86- | 79 |
| 2 | 69- | -35 |
| -2 | 82- | 44 |
| -3 | 328 | 354 |
| -4 | 63- | -6 |
| | 1 24 1 | |
| 0 | 145 | 151 |
| 1 | 63- | -44 |
| -1 | 71- | -38 |
| -2 | 193 | 196 |
| -3 | 58- | -33 |
| | 2 0 1 | |
| 0 | 426 | 522 |
| 2 | 591 | 596 |
| -2 | 833 | 992 |
| 4 | 578 | 505 |
| -4 | 374 | -395 |
| 6 | 78- | 85 |
| -6 | 591 | 530 |
| -8 | 272 | 313 |
| -10 | 230 | 340 |
| | 2 1 1 | |
| 0 | 111 | 143 |
| 1 | 239 | 237 |
| -1 | 681 | 872 |
| 2 | 74- | -6 |
| -2 | 253 | -311 |
| 3 | 518 | 489 |
| -3 | 842 | 773 |
| 4 | 350 | 343 |
| -4 | 178 | 135 |
| 5 | 250 | 259 |
| -5 | 326 | 302 |
| 6 | 78- | -51 |
| -6 | 83- | 111 |
| 7 | 260 | 259 |
| -7 | 516 | 548 |
| -8 | 92- | -66 |
| -9 | 119 | 144 |
| -10 | 60- | -3 |
| | 2 2 1 | |
| 0 | 379 | 384 |
| 1 | 130 | -120 |
| -1 | 122 | 13 |
| 2 | 409 | 416 |
| -2 | 759 | 973 |
| 3 | 85- | -13 |
| -3 | 48- | 38 |
| 4 | 390 | 406 |
| -4 | 584 | 579 |
| 5 | 91- | 18 |
| -5 | 72- | 1 |
| 6 | 165 | 181 |
| -6 | 585 | 628 |
| 7 | 54- | -80 |
| -7 | 92- | -39 |
| -8 | 297 | 323 |
| -9 | 81- | -29 |
| -10 | 118 | 134 |
| | 2 3 1 | |
| 0 | 86 | -50 |
| 1 | 202 | 179 |
| -1 | 594 | 694 |
| 2 | 300 | -313 |
| -2 | 41- | -60 |
| 3 | 764 | 756 |
| -3 | 462 | 365 |
| 4 | 170 | 159 |
| -4 | 62- | 89 |
| 5 | 193 | 207 |
| -5 | 452 | 468 |
| 6 | 77- | -83 |
| -6 | 84- | 77 |
| 7 | 253 | 270 |
| -7 | 441 | 465 |
| -8 | 92- | -148 |
| -9 | 100 | 99 |
| -10 | 58- | 132 |
| | 2 4 1 | |
| 0 | 470 | 461 |
| 1 | 77 | -98 |
| -1 | 387 | 490 |
| 2 | 625 | 572 |
| -2 | 401 | 486 |
| 3 | 87- | -67 |
| -3 | 248 | -172 |
| 4 | 455 | 440 |
| -4 | 412 | 385 |
| 5 | 162 | 177 |
| -5 | 247 | 234 |
| 6 | 181 | 180 |
| -6 | 450 | 455 |
| 7 | 49- | -110 |
| -7 | 246 | -307 |
| -8 | 266 | 278 |
| -9 | 79- | 3 |
| -10 | 131 | 173 |
| | 2 5 1 | |
| 0 | 158 | 167 |
| 1 | 699 | 730 |
| -1 | 423 | 577 |
| 2 | 136 | -147 |
| -2 | 53- | -100 |
| 3 | 486 | 448 |
| -3 | 480 | 519 |
| 4 | 214 | 200 |
| -4 | 67- | -90 |
| 5 | 129 | 139 |
| -5 | 525 | 532 |
| 6 | 74- | -10 |
| -6 | 242 | 228 |
| 7 | 45- | 143 |
| -7 | 394 | 461 |
| -8 | 89- | -164 |
| -9 | 191 | 287 |
| -10 | 53- | 82 |
| | 2 6 1 | |
| 0 | 478 | 469 |
| 1 | 141 | 114 |
| -1 | 142 | 127 |
| 2 | 433 | 396 |
| -2 | 627 | 593 |
| 3 | 89- | -68 |
| -3 | 409 | -485 |
| 4 | 315 | 305 |
| -4 | 599 | 660 |
| 5 | 87- | 25 |
| -5 | 255 | 242 |
| 6 | 220 | 252 |
| -6 | 458 | 492 |
| -7 | 93- | 38 |
| -8 | 200 | 212 |
| -9 | 75- | 37 |
| -10 | 104 | 182 |
| | 2 7 1 | |
| 0 | 452 | 457 |
| 1 | 692 | 590 |
| -1 | 368 | 336 |
| 2 | 235 | -209 |
| -2 | 63- | -115 |
| 3 | 454 | 393 |
| -3 | 383 | 356 |
| 4 | 93- | 43 |
| -4 | 74- | -186 |
| 5 | 406 | 388 |
| -5 | 380 | 389 |
| 6 | 69- | -104 |
| -6 | 89- | 146 |
| -7 | 252 | 269 |
| -8 | 87- | -146 |
| -9 | 241 | 282 |
| -10 | 44- | 110 |
| | 2 8 1 | |
| 0 | 580 | 561 |
| 1 | 79- | 45 |
| -1 | 264 | 218 |
| 2 | 423 | 404 |
| -2 | 432 | 381 |
| 3 | 92- | 26 |
| -3 | 307 | -312 |
| 4 | 300 | 289 |
| -4 | 558 | 663 |
| 5 | 149 | 158 |
| -5 | 210 | 223 |
| 6 | 304 | 347 |
| -6 | 237 | 276 |
| -7 | 93- | -29 |
| -8 | 259 | 326 |
| -9 | 70- | -44 |
| | 2 9 1 | |
| 0 | 178 | 117 |
| 1 | 724 | 574 |
| -1 | 515 | 474 |
| 2 | 241 | -223 |
| -2 | 72- | 26 |
| 3 | 93- | 50 |
| -3 | 690 | 722 |
| 4 | 92- | 12 |
| -4 | 81- | -55 |
| 5 | 341 | 399 |
| -5 | 454 | 527 |
| 6 | 62- | -21 |
| -6 | 92- | 33 |
| -7 | 200 | 213 |
| -8 | 84- | -61 |
| -9 | 267 | 366 |
| | 2 10 1 | |
| 0 | 763 | 679 |
| 1 | 85- | -101 |
| -1 | 77- | -15 |
| 2 | 363 | 324 |
| -2 | 315 | 308 |
| 3 | 93- | -72 |
| -3 | 79- | -28 |
| 4 | 235 | 226 |
| -4 | 445 | 428 |
| 5 | 78- | 15 |
| -5 | 89- | 42 |
| 6 | 282 | 323 |
| -6 | 159 | 147 |
| -7 | 91- | -8 |
| -8 | 303 | 338 |
| -9 | 62- | -6 |
| | 2 11 1 | |
| 0 | 156 | 110 |
| 1 | 612 | 580 |
| -1 | 388 | 352 |
| 2 | 92- | 7 |
| -2 | 81- | -37 |
| 3 | 188 | 137 |
| -3 | 419 | 409 |
| 4 | 161 | 131 |
| -4 | 87- | 83 |
| 5 | 386 | 376 |
| -5 | 399 | 383 |
| 6 | 50- | -55 |
| -6 | 93- | -41 |
| -7 | 88- | 92 |
| -8 | 77- | -68 |
| -9 | 280 | 383 |
| | 2 12 1 | |
| 0 | 689 | 628 |
| 1 | 91- | 2 |
| -1 | 85- | -106 |
| 2 | 407 | 387 |
| -2 | 163 | 150 |
| 3 | 92- | 121 |
| -3 | 86- | 117 |
| 4 | 216 | 192 |
| -4 | 721 | 714 |
| 5 | 70- | 3 |
| -5 | 93- | -44 |
| -6 | 166 | 177 |
| -7 | 86- | 12 |
| -8 | 312 | 385 |
| -9 | 49- | -10 |
| | 2 13 1 | |
| 0 | 342 | -351 |
| 1 | 498 | 394 |
| -1 | 324 | 273 |
| 2 | 93- | 94 |
| -2 | 88- | 17 |
| 3 | 243 | 209 |
| -3 | 450 | 454 |
| 4 | 80- | -63 |
| -4 | 92- | 49 |
| 5 | 281 | 304 |
| -5 | 572 | 591 |
| -6 | 91- | 11 |
| -7 | 147 | 148 |
| -8 | 68- | 94 |
| | 2 14 1 | |
| 0 | 481 | 377 |
| 1 | 93- | 24 |
| -1 | 92- | 71 |
| 2 | 445 | 346 |
| -2 | 185 | 148 |
| 3 | 86- | -80 |
| -3 | 92- | 174 |
| 4 | 272 | 233 |
| -4 | 514 | 479 |
| 5 | 57- | -78 |
| -5 | 92- | -112 |
| -6 | 222 | 229 |
| -7 | 78- | -128 |
| -8 | 178 | 166 |
| | 2 15 1 | |
| 0 | 93- | -89 |
| 1 | 774 | 653 |
| -1 | 212 | 204 |
| 2 | 284 | 225 |
| -2 | 107 | 120 |
| 3 | 228 | 185 |
| -3 | 324 | 311 |
| 4 | 111 | -109 |
| -4 | 93- | 15 |
| 5 | 194 | 222 |
| -5 | 322 | 289 |
| -6 | 84- | -103 |
| -7 | 190 | 227 |
| -8 | 54- | 81 |
| | 2 16 1 | |
| 0 | 385 | 355 |
| 1 | 89- | 95 |
| -1 | 251 | -244 |
| 2 | 314 | 288 |
| -2 | 351 | 395 |
| 3 | 77- | 38 |
| -3 | 93- | 127 |
| 4 | 205 | 187 |
| -4 | 375 | 368 |
| -5 | 159 | -176 |
| -6 | 313 | 330 |
| -7 | 155 | 211 |
| -8 | 121 | 172 |
| | 2 17 1 | |
| 0 | 89- | -56 |
| 1 | 313 | 242 |
| -1 | 331 | 313 |
| 2 | 143 | 90 |
| -2 | 92- | -81 |
| 3 | 270 | 269 |
| -3 | 318 | 277 |
| 4 | 87 | -113 |
| -4 | 87- | 8 |
| -5 | 263 | 257 |
| -6 | 74- | -53 |
| -7 | 184 | 223 |
| | 2 18 1 | |
| 0 | 266 | 211 |
| 1 | 176 | 131 |
| -1 | 88- | -88 |
| 2 | 250 | 191 |
| -2 | 307 | 330 |
| 3 | 64- | 27 |
| -3 | 162 | 162 |
| -4 | 282 | 296 |
| -5 | 157 | -151 |
| -6 | 168 | 191 |
| -7 | 47- | 68 |
| | 2 19 1 | |
| 0 | 84 | -72 |
| 1 | 187 | 149 |
| -1 | 425 | 430 |
| 2 | 160 | 117 |
| -2 | 126 | 165 |
| 3 | 266 | 298 |
| -3 | 254 | 267 |
| -4 | 77- | 11 |
| -5 | 179 | 198 |
| -6 | 113 | -144 |
| | 2 20 1 | |
| 0 | 267 | 245 |
| 1 | 68- | -67 |
| -1 | 163 | -157 |
| 2 | 240 | 215 |
| -2 | 352 | 409 |
| -3 | 75- | 66 |
| -4 | 86 | 64 |
| -5 | 61- | 0 |
| -6 | 179 | 251 |
| | 2 21 1 | |
| 0 | 66- | -72 |
| 1 | 127 | 78 |
| -1 | 310 | 305 |
| 2 | 86 | 61 |
| -2 | 69- | 25 |
| -3 | 230 | 247 |
| -4 | 60- | 26 |
| -5 | 125 | 125 |
| | 2 22 1 | |
| 0 | 146 | 146 |
| 1 | 44- | 34 |
| -1 | 59- | 15 |
| -2 | 298 | 320 |
| -3 | 56- | 56 |
| -4 | 88 | 80 |
| | 2 23 1 | |
| -1 | 194 | 253 |
| -2 | 44- | -23 |

The conventional $R$ indices (observed reflections only) are:

$$R(0kl)=6\cdot60\%,\quad R(1kl)=9\cdot08\%,\quad R(2kl)=11\cdot14\%\,.$$

The scattering factors of Berghuis *et al.* (1955) were used for C, N, O, $Cl^-$, while for $Cd^{2+}$ the values given by Thomas & Umeda (1957) were employed.

An estimate of the accuracy of the coordinates is also given by the $R'$ indices calculated for the observed reflections with $k+l$ odd, in which the cadmium has no influence. These reflections are rather weak and their number is small (24 $0kl$, 59 $1kl$, 51 $2kl$); nevertheless, the $R'$ values are fairly small:

$$R'(0kl)=7\cdot76\%,\quad R'(1kl)=11\cdot99\%,\quad R'(2kl)=16\cdot19\%\,.$$

## 4. Discussion of the structure

The coordination polyhedron around the Cd atom is octahedral. Fig. 2 shows how these polyhedra are linked in chains parallel to [100]. The bond distances and angles concerning Cd are as follows (the coordinates are not given if the atom is at $x, y, z$):

$\text{Cd–Cl} = 2{\cdot}55 \pm 0{\cdot}01$ Å
$\text{Cd–Cl}(x-1, y, z) = 2{\cdot}62 \pm 0{\cdot}01$
$\text{Cd–O}_\text{I} = 2{\cdot}34 \pm 0{\cdot}02$
$\text{Cl–Cd–Cl}(x-1, y, z) = 91{\cdot}5 \pm 0{\cdot}3°$
$\text{Cd–Cl–Cd}(x+1, y, z) = 91{\cdot}5 \pm 0{\cdot}3$
$\text{O}_\text{I}\text{–Cd–Cl} = 87{\cdot}9 \pm 0{\cdot}5$
$\text{O}_\text{I}\text{–Cd–Cl}(x-1, y, z) = 88{\cdot}0 \pm 0{\cdot}5$

The standard deviations in distances and angles were calculated using the relations of Ahmed & Cruickshank (1953). The distances Cd–Cl are in agreement with the corresponding ones found in the coordination compounds formed by $CdCl_2$ and *bis*-acetamide (2·58, 2·66 Å; Cavalca, Nardelli & Coghi, 1957), *bis*-urea (2·64 Å; Nardelli, Cavalca & Fava, 1957), *mono*-methylurea, $CdCl_2.OC(NH_2)NHCH_3$, (2·58, 2·62 Å; Nardelli, Coghi & Azzoni, 1958). The distance Cd–O is a little larger than those found in the other compounds (2·23, 2·28, 2·18 Å respectively).

A diagrammatic projection of a part of a unit cell along [100] is given in Fig. 3.

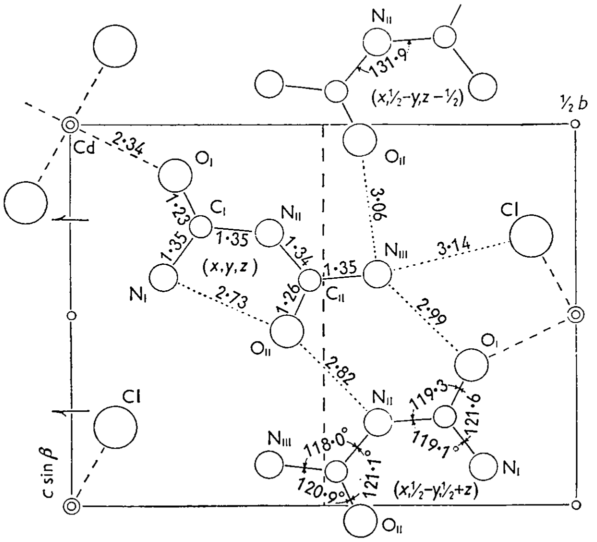


Fig. 3. Diagrammatic projection along [100] of a part of a unit cell.

The results confirm the *trans* configuration of the biuret molecule, and so an intramolecular hydrogen bond is possible, as suggested by Kogon (1957) from the study of infrared absorption spectra of some alkyl- and aryl-alkyl-biurets. The *trans* configuration with the indicated hydrogen bonding is present in dioxane solution of biuret too, as Kumler (1959) deduced from the dipole moment (3·27 Debyes) which is considerably less than that of urea (4·56 D.) and that of acetamide (3·90 D.). Because of this hydrogen bonding, the two oxygen atoms ($O_I$ and $O_{II}$) are not equivalent and only $O_I$ can be coordinated by the metal atom. The biuret molecule has the following bond lengths and angles:

$\text{O}_\text{I}\text{–C}_\text{I} = 1{\cdot}23 \pm 0{\cdot}05$ Å
$\text{C}_\text{I}\text{–N}_\text{I} = 1{\cdot}35 \pm 0{\cdot}05$
$\text{C}_\text{I}\text{–N}_\text{II} = 1{\cdot}35 \pm 0{\cdot}04$
$\text{N}_\text{II}\text{–C}_\text{II} = 1{\cdot}34 \pm 0{\cdot}04$
$\text{C}_\text{II}\text{–O}_\text{II} = 1{\cdot}26 \pm 0{\cdot}05$
$\text{C}_\text{II}\text{–N}_\text{III} = 1{\cdot}35 \pm 0{\cdot}04$
$\text{N}_\text{I} \cdots \text{O}_\text{II} = 2{\cdot}73 \pm 0{\cdot}04$
$\text{O}_\text{I}\text{–C}_\text{I}\text{–N}_\text{I} = 121{\cdot}6 \pm 2{\cdot}9°$
$\text{O}_\text{I}\text{–C}_\text{I}\text{–N}_\text{II} = 119{\cdot}3 \pm 2{\cdot}7$
$\text{N}_\text{I}\text{–C}_\text{I}\text{–N}_\text{II} = 119{\cdot}1 \pm 2{\cdot}8$
$\text{C}_\text{I}\text{–N}_\text{II}\text{–C}_\text{II} = 131{\cdot}9 \pm 3{\cdot}0$
$\text{N}_\text{II}\text{–C}_\text{II}\text{–O}_\text{II} = 121{\cdot}1 \pm 2{\cdot}3$
$\text{N}_\text{II}\text{–C}_\text{II}\text{–N}_\text{III} = 118{\cdot}0 \pm 2{\cdot}5$
$\text{O}_\text{II}\text{–C}_\text{II}\text{–N}_\text{III} = 120{\cdot}9 \pm 2{\cdot}5$
$\text{O}_\text{II} \cdots \text{N}_\text{I}\text{–C}_\text{I} = 82{\cdot}8 \pm 2{\cdot}1$
$\text{N}_\text{I} \cdots \text{O}_\text{II}\text{–C}_\text{II} = 85{\cdot}0 \pm 2{\cdot}0$

The configuration assumed for the organic molecule is based on the following considerations:

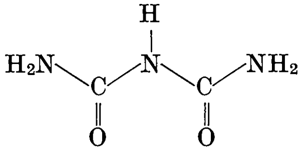
($a$) The values of the electron density at the maxima of the peaks attributed to the oxygen atoms are higher than those of the nitrogen atoms, e.g. the values for $\varrho_{\text{max.}}$ in $\varrho_0(Y, Z)$ projection are:

| | | $O_I$ | $O_{II}$ | $N_I$ | $N_{II}$ | $N_{III}$ | |
|---|---|---|---|---|---|---|---|
| $\varrho_{\text{max.}}$ | obs. | 10·1 | 9·2 | 8·4 | 8·1 | 7·6 | e.Å$^{-2}$ |
| | calc. | 9·9 | 9·8 | 8·6 | 7·9 | 7·1 | |

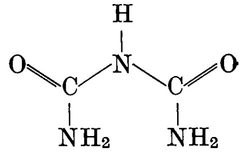
($b$) The interatomic distances O–C and N–C agree with those expected.

($c$) The *trans* configuration and the interatomic distances correspond to what has been found for the biuret hydrate by Hughes, Yakel & Freeman (1959).

($d$) The two *cis* configurations



(I)



(II)

can be ruled out because with (I) the biuret molecule should behave as a bidentate ligand, and with (II) the steric hindrance between the hydrogen atoms of the $NH_2$-groups should push these out of the plane of the molecule. The *trans* configuration is stabilized by a strong intramolecular hydrogen-bond.

The planarity of the molecule is indicated by the values of the angles at $C_I$ and $C_{II}$, which in both cases add up to 360°, and by the sum of the angles in the pentagon $N_IC_IN_{II}C_{II}O_{II}$, which is 539·9°. The equation of the least-squares plane of the molecule is:

$$1{\cdot}9288x' + 0{\cdot}0782y' - 1{\cdot}0000z' + 1 = 0$$

using the orthogonal coordinates of the atoms. The average deviation of all atoms from this plane is

0·04 Å, the greatest deviation being 0·06 Å for $N_{III}$. It is easy to calculate from this that the contribution to the structure factor from the organic molecule is a maximum for the $20\bar{2}$ reflection.

The distances and angles found in the organic molecule are in good agreement with those reported in the literature for the amide groups (Tomiie, Koo & Nitta, 1958). The angle $C_I$–$N_{II}$–$C_{II}$ is considerably larger than that required by trigonal $sp^2$ hybridization on nitrogen atom; the increased value can be justified by steric hindrance between $N_I$ and $O_{II}$.

The angle Cd–$O_I$–$C_I$ = 136·3° ± 2·8° is similar to the corresponding ones found in *bis*-urea- (134°), in *bis*-acetamide- (134°) and in *mono*-methylurea- (128°) cadmium chlorides.

Interaction of the different parallel chains occurs through the atoms of the organic molecules, which form hydrogen bonds of NH ··· O type. The most significant intermolecular distances are:

$N_I$–Cl $(1-x, \bar{y}, \bar{z})$ = 3·25 ± 0·03 Å
$N_I$–Cl $(x, y, 1+z)$ = 3·79 ± 0·04
$N_I$–Cl $(1+x, y, 1+z)$ = 3·55 ± 0·04
$N_{III}$—Cl $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·14 ± 0·03
$N_{III}$–$O_I$ $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·41 ± 0·03
$N_{III}$–$O_I$ $(1+x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 2·99 ± 0·03
$O_{II}$–$N_{II}$ $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·59 ± 0·03
$O_{II}$–$N_{II}$ $(1+x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 2·82 ± 0·04
$O_{II}$–$N_{III}$ $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·52 ± 0·04
$O_{II}$–$N_{III}$ $(1+x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·06 ± 0·04
$N_{III}$–$N_{II}$ $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·45 ± 0·04
$N_{III}$–$N_{II}$ $(1+x, \frac{1}{2}-y, \frac{1}{2}+z)$ = 3·58 ± 0·04

The distance between the planes of molecules separated by one unit cell translation along the $x$ axis is 3·29 Å.

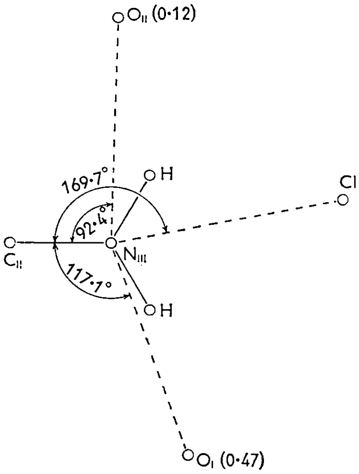


Fig. 4. Hydrogen-bonding around $N_{III}$.

An intermolecular hydrogen bond occurs between $N_{II}$ $(1+x, \frac{1}{2}-y, \frac{1}{2}+z)$ and $O_{II}$; this is indicated by the short value of the interatomic distance (2·82 Å), by the small distance (0·12 Å) of $O_{II}$ from the plane of the molecule containing the nitrogen donor-atom and by the angles

$$C_{II}\,(1+x, \tfrac{1}{2}-y, \tfrac{1}{2}+z)\text{–}N_{II}\,(1+x, \tfrac{1}{2}-y, \tfrac{1}{2}+z)\text{–}O_{II} = 103{\cdot}5^\circ$$

and

$$C_{I}\,(1+x, \tfrac{1}{2}-y, \tfrac{1}{2}+z)\text{–}N_{II}\,(1+x, \tfrac{1}{2}-y, \tfrac{1}{2}+z)\text{–}O_{II} = 123{\cdot}9^\circ\,.$$

The nearest neighbours of the $N_{III}$ atom are represented diagrammatically in Fig. 4, in which the values in brackets indicate the distances of these atoms from the plane containing $C_{II}N_{III}H_2$. The positions of the two H-atoms are deduced assuming that they lie in the plane of the molecule and form trigonal angles. From this diagram one can deduce that hydrogen bonding with $O_I$ is probable, because this atom is not very far from the plane of the molecule containing the H-donor. The angular values indicate that the other H-atom is more probably bound to $O_{II}$ than to Cl, but it may be bound to both of them.

The calculations in the final stages of this work were performed on IBM 650 using a financial support from *Consiglio Nazionale delle Ricerche*. The authors wish to thank Prof. L. H. Jensen who kindly provided the programs for the computer and the *Centro Calcoli e Servomeccanismi* (Università di Bologna) for its help and facilities.

## References

Ahmed, F. R. & Cruickshank, D. W. J. (1953). *Acta Cryst.* **6**, 385.

Albrecht, G. (1939). *Rev. Sci. Instrum.* **10**, 221.

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

Cavalca, L., Nardelli, M. & Coghi, L. (1957). *Nuovo Cimento*, *X*, **6**, 278.

Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.

Freeman, H. C., Smith, J. E. W. L. & Taylor, J. C. (1959). *Nature, Lond.* **184**, 707.

Hughes, E. W., Yakel, H. L. & Freeman, H. C. (1960). *Acta Cryst.* (in the press), private communication from Freeman.

Kogon, I. C. (1957). *J. Amer. Chem. Soc.* **79**, 2253.

Kumler, W. D. (1959). Private communication.

Nardelli, M., Cavalca, L. & Fava, G. (1957). *Gazz. Chim. Ital.* **87**, 1232.

Nardelli, M. & Chierici, I. (1960). *J. Chem. Soc.* p. 1952.

Nardelli, M., Coghi, L. & Azzoni, G. (1958). *Gazz. Chim. Ital.* **88**, 235.

Schenck, M. (1904). *Z. physiol. Chem.* **43**, 72.

Schiff, H. (1898). *Ann. Chem.* **299**, 243.

Scouloudi, H. (1953). *Acta Cryst.* **6**, 651.

Thomas, L. H. & Umeda, K. (1957). *J. Chem. Phys.* **26**, 293.

Tomiie, Y., Koo, C. H. & Nitta, I. (1958). *Acta Cryst.* **11**, 774.

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