Table 4 (*cont.*)

Bond	Length
P ——O(3)	1·57 (2)
P ——C(6)	1·82 (2)
O(1)—C(1)	1·43 (2)
O(3)—C(3)	1·46 (2)
C(1)—C(2)	1·51 (3)
C(2)—C(3)	1·55 (3)
C(2)—C(4)	1·52 (4)
C(2)—C(5)	1·55 (3)
C(6)—C(7)	1·43 (3)
C(7)—C(8)	1·34 (3)
C(8)—C(9)	1·37 (4)
C(9)—C(10)	1·40 (3)
C(10)–C(11)	1·37 (3)
C(6)—C(11)	1·33 (3)

Table 5. *Intermolecular bond angles and standard deviations*

	Angle
O(1)—P ——O(2)	113·5 (0·9)°
O(1)—P ——O(3)	106·2 (0·8)
O(2)—P ——O(3)	111·4 (0·9)
O(1)—P ——C(6)	107·6 (0·9)
O(2)—P ——C(6)	111·7 (0·9)
O(3)—P ——C(6)	106·5 (0·9)
O(1)—C(1)—C(2)	113·8 (1·7)
O(3)—C(3)—C(2)	111·8 (1·5)
C(1)—C(2)—C(3)	105·8 (1·8)
C(1)—C(2)—C(4)	109·8 (2·0)
C(1)—C(2)—C(5)	113·2 (1·9)
C(3)—C(2)—C(4)	109·8 (2·0)
C(3)—C(2)—C(5)	104·8 (2·0)
P ——C(6)—C(7)	114·3 (1·7)
P ——C(6)—C(11)	121·0 (1·6)
C(6)—C(7)—C(8)	116·2 (2·0)
C(7)—C(8)—C(9)	120·1 (2·0)
C(8)—C(9)—C(10)	122·6 (2·0)
C(10)–C(11)–C(6)	118·2 (2·0)
C(11)–C(6)—C(7)	124·7 (2·0)
P–O(1)–C(1)	120·4 (1·5)
P–O(3)–C(3)	119·3 (1·5)
C(9)–C(10)–C(11)	118·0 (2·0)
C(4)– C(2)– C(5)	113·5 (2·0)

The single-bonded phosphorus–oxygen distance (mean 1·55 Å) and double-bonded phosphorus–oxygen distance (1·47 Å) agree with those found in several structures. Kraut & Jensen (1963) allot values of 1·56 and 1·49 Å respectively to these bonds. They have also observed that oxygen–phosphorus–oxygen angles increase with decreasing oxygen–phosphorus distances, and it can be seen that this confirmed by the present results. The P–C distance of $1{\cdot}82 \pm 0{\cdot}02$ Å compares satisfactorily with standard P–C distances ($1{\cdot}87 \pm 0{\cdot}02$ Å), while, in the phosphorinane ring, the C–C distances are the expected value (mean 1·53 Å) and the C–O distances (mean 1·45 Å) are not significantly different from the usually accepted value for this bond, 1·43 Å. Large valency angles for oxygen atoms have been found in organic phosphates (Svetich & Caughlan, 1965) and this is the case in the present structure (mean value 120°). The carbon valency angles are normal except for the C–C–C angle in the heterocyclic ring which is unusually small (105°).

Bond distances in the benzene ring give an average value of 1·37 Å and bond angles an average value of 120°.

References

ANDERSON, J. E. (1965). *Quart. Rev.* **19**, 426.

EDMUNDSON, R. S. (1964). *Tetrahedron*, **20**, 2781.

GEISE, H. J. (1967). *Rec. Trav. Chim. Pays-Bas*, **86**, 362.

HARGIS, J. H. & BENTRUDE, W. E. (1962). *Tetrahedron Letters*, **51**, 5365.

KILLEAN, R. C. G. (1967). *Acta Cryst.* **23**, 1109.

KILLEAN, R. C. G. & LAWRENCE, J. L. (1969). *Acta Cryst.* B**25**, 1750.

KITAIGORODSKI, A. I. (1957). *Theory of Crystal Structure Analysis*, Translation 1961, p. 249. New York: Heywood.

KRAUT, J. & JENSEN, L. H. (1963). *Acta Cryst.* **16**, 79.

SVETICH, G. W. & CAUGHLAN, C. N. (1965). *Acta Cryst.* **19**, 645.

Acta Cryst. (1971). B**27**, 192

The Crystal and Molecular Structure of Dichlorodiaquobis(dicyandiamide)copper(II)

BY A. CHIESI, L. COGHI, A. MANGIA, M. NARDELLI AND G. PELIZZI

Istituto di Chimica Generale, Università di Parma, Parma, Italy

(*Received* 9 *February* 1970)

Crystals of $Cu(OH_2)_2(C_2N_4H_4)_2Cl_2$ are triclinic ($P\bar{1}$): $a = 5{\cdot}42$ (1), $b = 6{\cdot}45$ (1), $c = 9{\cdot}31$ (1) Å, $\alpha = 74{\cdot}5$ (0·2), $\beta = 80{\cdot}4$ (0·3), $\gamma = 84{\cdot}7$ (0·3)°, $Z = 1$. The structure was solved and refined by means of three-dimensional Fourier methods (final $R = 8{\cdot}3$ %). The Cu^{II} atom lies on a centre of symmetry and is surrounded by a planar arrangement of two water molecules (Cu–O = 2·00 Å) and two nitrile nitrogen atoms (Cu–N = 1·92 Å) from two dicyandiamide molecules. Two chlorine atoms, in the *trans* position with respect to that plane (Cu–Cl = 2·87 Å), complete the coordination polyhedron to form an elongated octahedron.

The crystal structure of dichlorodiaquobis(dicyandiamide)copper(II) has been determined in order to study the behaviour of dicyandiamide in metal-coordination.

Dichlorodiaquobis(dicyandiamide)copper(II) occurs as blue-green triclinic platelets elongated along [100]. Cell constants, determined from Weissenberg and rota-

Table 1. *Final atomic fractional coordinates* ($\times 10^4$), *thermal parameters* ($\times 10^2$Å^2)* *with e.s.d.'s for non-hydrogen atoms*

	x/a (σ)	y/b (σ)	z/c (σ)	B_{11} (σ)	B_{22} (σ)	B_{33} (σ)	B_{12} (σ)	B_{13} (σ)	B_{23} (σ)
Cu	0 (–)	0 (–)	0 (–)	194 (8)	222 (2)	158 (0)	3 (4)	18 (3)	−12 (2)
Cl	2129 (4)	2697 (1)	1427 (1)	181 (7)	206 (1)	205 (1)	−19 (3)	0 (3)	−63 (1)
O	2634 (11)	−2299 (5)	625 (3)	207 (21)	142 (3)	216 (4)	−4 (9)	−10 (9)	−21 (5)
N(1)	2102 (14)	1235 (5)	−1855 (4)	156 (24)	215 (5)	148 (3)	−34 (10)	28 (10)	−28 (6)
N(2)	3720 (15)	2324 (6)	−4540 (4)	191 (25)	236 (6)	93 (2)	−31 (11)	17 (9)	−30 (6)
N(3)	8028 (17)	2178 (8)	−4398 (4)	216 (26)	342 (11)	188 (3)	−9 (15)	−49 (10)	14 (9)
N(4)	6559 (16)	3178 (4)	−6656 (4)	223 (27)	225 (5)	133 (3)	−57 (10)	42 (11)	−42 (6)
C(1)	3041 (15)	1735 (5)	−3099 (3)	123 (27)	135 (4)	128 (3)	−20 (10)	13 (10)	−25 (6)
C(2)	6105 (15)	2531 (5)	−5177 (4)	133 (28)	161 (4)	107 (3)	−18 (11)	25 (10)	−21 (6)

* The B_{ij} values refer to the formula: $\exp[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)]$ in which $b_{11}=\frac{1}{4}a^{*2}B_{11}$, $b_{12}=\frac{1}{2}a^*b^*B_{12}$, *etc.*

tion photographs (Cu $K\alpha$, $\bar{\lambda}=1{\cdot}5418$ Å) taken around the elongation axis, are as follows (standard deviations given in parentheses are in units of the last decimal figure):

$Cu(OH_2)_2(C_2N_4H_4)_2Cl_2$ $M=338{\cdot}7$
$a=5{\cdot}42(1)$, $b=6{\cdot}45(1)$, $c=9{\cdot}31(1)$ Å
$\alpha=74{\cdot}5(0{\cdot}2)$, $\beta=80{\cdot}4(0{\cdot}3)$, $\gamma=84{\cdot}7(0{\cdot}3)°$
$V=308{\cdot}8$ Å^3, $Z=1$, $D_m=1{\cdot}815$, $D_c=1{\cdot}821$ g.cm^{-3},
$F(000)=171$, $\mu=65{\cdot}96$ cm^{-1}
Space group: $P\bar{1}$(from structure analysis).

Three-dimensional intensity data were determined photometrically on integrated and non-integrated Weissenberg photographs taken around [100], up to the fourth layer (multiple-film technique, Cu $K\alpha$). After correction for Lorentz and polarization factors, the intensities were placed on the same relative scale using a set of short oscillation ($\Delta\omega=20°$) Weissenberg photographs, all registered with the same exposure on the same film. The absolute scale was then established by Wilson's method.

The structure was solved by the heavy-atom method and refined by means of Booth's differential synthesis with anisotropic thermal parameters down to a final $R=8{\cdot}3\%$. All the hydrogen atoms except those of water molecules were located from the $\varrho_o-\varrho_c$ final map. Final coordinates with thermal parameters are listed in Tables 1 and 2. The comparison between observed and calculated peak shapes is shown in Table 3, while the observed and calculated structure factors are compared in Table 4.

Table 2. *Coordinates* ($\times 10^4$) *for the hydrogen atoms of dicyandiamide with their isotropic B values*

	x/a	y/b	z/c	B
H(1)	0	2167	−4833	2·48
H(2)	8000	2000	−3333	2·48
H(3)	8333	3000	−7167	1·93
H(4)	5000	3333	−7167	1·93

Fig. 1 shows a clinographic projection of the structure. The copper(II) atom lies on a centre of symmetry and is surrounded by a planar arrangement of two oxygen atoms from two water molecules and two nitrogen atoms from two dicyandiamide molecules at the corners of a distorted square. Dicyandiamide behaves as a monodentate ligand through nitrile nitrogen atoms. The coordination is completed by two chlorine atoms, which are on opposite sides with respect to the square, so that the whole polyhedron appears as an elongated octahedron.

Table 3. *Atomic peak heights* (e.Å^{-3}) *and curvatures* (e.Å^{-5}) *with their e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Cu	obs.	71·9	504	750	789	−155	−40	−23
	calc.	72·7	503	757	788	−162	−46	−27
Cl	obs.	38·6	273	432	422	−115	−36	−30
	calc.	38·9	270	430	423	−113	−37	−28
O	obs.	14·5	99	151	135	−30	−13	−3
	calc.	14·6	99	150	137	−31	−13	−4
N(1)	obs.	13·2	89	125	166	−25	6	−14
	calc.	13·4	89	126	166	−26	6	−14
N(2)	obs.	12·5	79	123	130	−25	0	−6
	calc.	12·6	78	125	128	−26	−1	−6
N(3)	obs.	10·3	62	92	102	−15	−20	−6
	calc.	10·4	60	95	102	−17	−19	−6
N(4)	obs.	11·8	73	122	115	−37	−5	−12
	calc.	11·9	72	125	113	−37	−5	−12
C(1)	obs.	11·3	74	130	129	−41	−16	−8
	calc.	11·5	72	130	128	−41	−15	−8
C(2)	obs.	11·3	77	124	132	−31	−7	−8
	calc.	11·6	76	124	133	−31	−8	−8
	e.s.d.	0·2	2	3	3	2	1	1

Distances in the coordination polyhedron agree well with those found in other octahedral copper(II) complexes, *e.g.*: Cu–O = 1·97, Cu–N = 1·99, Cu–Cl = 2·84 Å in bis(semicarbazide)copper(II) (Nardelli, Fava Gasparri, Boldrini & Giraldi Battistini, 1965); Cu–O = 1·935, Cu–Cl = 2·960 Å in bis(biuret)copper(II) dichloride (Freeman & Smith, 1966); Cu–O = 1·986, 1·931, Cu–N = 1·979, 1·996 Å in bis(L-histidine)copper(II) dinitrate dihydrate (Evertsson, 1969).

The dicyandiamide molecule is practically planar; the maximum displacement from the least-squares mean plane being 0·011 Å for C(2). Coordination appears not to affect the structural parameters of that ligand as can be seen from the data quoted in Table 5,

Table 4. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$10F_o$	$10F_c$
2	0	0	225	-330
3	0	0	113	126
4	0	0	173	194
0	1	0	340	376
1	1	0	150	203
1	$\bar{1}$	0	360	358
2	1	0	11-	45
2	$\bar{1}$	0	226	207
3	1	0	191	231
3	$\bar{1}$	0	62	50
4	1	0	183	225
4	$\bar{1}$	0	72	67
0	2	0	506	-551
1	2	0	209	208
1	$\bar{2}$	0	331	361
2	2	0	513	622
2	$\bar{2}$	0	389	399
3	2	0	143	145
3	$\bar{2}$	0	179	178
4	2	0	24	23
4	$\bar{2}$	0	121	114
0	3	0	138	115
1	3	0	174	141
1	$\bar{3}$	0	181	154
2	3	0	257	240
2	$\bar{3}$	0	175	164
3	3	0	103	107
3	$\bar{3}$	0	157	139
4	3	0	29	35
4	$\bar{3}$	0	132	120
0	4	0	374	369
1	4	0	82	75
1	$\bar{4}$	0	174	157
2	4	0	126	-108
2	$\bar{4}$	0	142	121
3	4	0	183	159
3	$\bar{4}$	0	19-	12
4	4	0	210	195
4	$\bar{4}$	0	29-	27
0	5	0	71	75
1	5	0	69	75
1	$\bar{5}$	0	113	107
2	5	0	96	98
2	$\bar{5}$	0	166	153
3	5	0	124	138
3	$\bar{5}$	0	119	112
4	5	0	105	96
4	$\bar{5}$	0	26-	20
0	6	0	38-	-11
1	6	0	170	167
1	$\bar{6}$	0	29-	-13
2	6	0	196	204
2	$\bar{6}$	0	64	61
3	6	0	17-	25
3	$\bar{6}$	0	174	181
4	6	0	42	-37
4	$\bar{6}$	0	81	89
0	7	0	80	94
1	7	0	107	111
1	$\bar{7}$	0	54	47
2	7	0	47	53
2	$\bar{7}$	0	20-	-6
3	7	0	12-	-5
3	$\bar{7}$	0	7-	36
1	8	0	7-	-23
0	0	1	181	185
1	0	1	380	441
1	0	$\bar{1}$	360	461
2	0	1	153	-173
2	0	$\bar{1}$	220	224
3	0	1	109	128
3	0	$\bar{1}$	82	-71
4	0	1	300	400
4	0	$\bar{1}$	164	178
0	1	1	192	218
0	1	$\bar{1}$	278	315
1	1	1	139	158
1	$\bar{1}$	1	420	454
1	1	$\bar{1}$	9-	3
1	$\bar{1}$	$\bar{1}$	240	313
2	1	1	118	118
2	$\bar{1}$	1	333	325
2	1	$\bar{1}$	14-	29
2	$\bar{1}$	$\bar{1}$	290	291
3	1	1	149	209
3	$\bar{1}$	1	27	23
3	1	$\bar{1}$	175	195
3	$\bar{1}$	$\bar{1}$	223	218
4	1	1	171	226
4	$\bar{1}$	1	37	-21
4	1	$\bar{1}$	226	278
4	$\bar{1}$	$\bar{1}$	21-	7
0	2	1	492	615
0	2	$\bar{1}$	60	-36
1	2	1	262	285
1	$\bar{2}$	1	192	174
1	2	$\bar{1}$	17-	41
1	$\bar{2}$	$\bar{1}$	150	-143
2	2	1	242	258
2	$\bar{2}$	1	574	632

h	k	l	$10F_o$	$10F_c$
2	2	$\bar{1}$	232	241
2	$\bar{2}$	$\bar{1}$	83	73
3	2	1	126	103
3	$\bar{2}$	1	112	114
3	2	$\bar{1}$	300	356
3	$\bar{2}$	$\bar{1}$	354	349
4	2	1	101	-84
4	$\bar{2}$	1	199	-165
4	2	$\bar{1}$	95	87
4	$\bar{2}$	$\bar{1}$	93	83
0	3	1	360	346
0	3	$\bar{1}$	83	78
1	3	1	235	239
1	$\bar{3}$	1	44	35
1	3	$\bar{1}$	263	259
1	$\bar{3}$	$\bar{1}$	246	229
2	3	1	56	77
2	$\bar{3}$	1	132	126
2	3	$\bar{1}$	329	324
2	$\bar{3}$	$\bar{1}$	22-	4
3	3	1	64	64
3	$\bar{3}$	1	245	233
3	3	$\bar{1}$	107	98
3	$\bar{3}$	$\bar{1}$	17-	-10
4	3	1	111	93
4	$\bar{3}$	1	180	163
4	3	$\bar{1}$	58	-46
4	$\bar{3}$	$\bar{1}$	220	202
0	4	1	31-	-26
0	4	$\bar{1}$	307	305
1	4	1	82	-64
1	$\bar{4}$	1	95	85
1	4	$\bar{1}$	250	232
1	$\bar{4}$	$\bar{1}$	416	468
2	4	1	147	121
2	$\bar{4}$	1	166	-136
2	4	$\bar{1}$	68	50
2	$\bar{4}$	$\bar{1}$	171	151
3	4	1	209	196
3	$\bar{4}$	1	176	156
3	4	$\bar{1}$	19-	-2
3	$\bar{4}$	$\bar{1}$	97	-70
4	4	1	231	217
4	$\bar{4}$	1	256	240
4	4	$\bar{1}$	113	105
4	$\bar{4}$	$\bar{1}$	111	103
0	5	1	37-	-25
0	5	$\bar{1}$	139	124
1	5	1	43	35
1	$\bar{5}$	1	171	148
1	5	$\bar{1}$	82	75
1	$\bar{5}$	$\bar{1}$	93	82
2	5	1	209	188
2	$\bar{5}$	1	107	100
2	5	$\bar{1}$	29-	20
2	$\bar{5}$	$\bar{1}$	235	222
3	5	1	184	172
3	$\bar{5}$	1	51	51
3	5	$\bar{1}$	112	104
3	$\bar{5}$	$\bar{1}$	183	171
4	5	1	55	54
4	$\bar{5}$	1	42	41
4	5	$\bar{1}$	148	152
4	$\bar{5}$	$\bar{1}$	26-	-11
0	6	1	119	114
0	6	$\bar{1}$	37-	-16
1	6	1	225	219
1	$\bar{6}$	1	79	74
1	6	$\bar{1}$	29-	39
1	$\bar{6}$	$\bar{1}$	126	-104
2	6	1	71	81
2	$\bar{6}$	1	179	179
2	6	$\bar{1}$	154	161
2	$\bar{6}$	$\bar{1}$	115	106
3	6	1	18-	1
3	$\bar{6}$	1	60	61
3	6	$\bar{1}$	108	104
3	$\bar{6}$	$\bar{1}$	155	159
4	6	1	24-	-5
4	$\bar{6}$	1	15-	-4
4	6	$\bar{1}$	19-	-18
4	$\bar{6}$	$\bar{1}$	17-	28
0	7	1	111	121
0	7	$\bar{1}$	71	79
1	7	1	61	90
1	$\bar{7}$	1	22-	-25
1	7	$\bar{1}$	102	95
1	$\bar{7}$	$\bar{1}$	73	73
2	7	1	23-	11
2	$\bar{7}$	1	31	31
2	7	$\bar{1}$	75	79
2	$\bar{7}$	$\bar{1}$	21-	13
3	7	1	13-	5
3	7	$\bar{1}$	8-	16
3	$\bar{7}$	$\bar{1}$	9-	9
1	8	1	14-	-24
0	0	2	283	346
1	0	2	227	-225
1	0	$\bar{2}$	440	535
2	0	1	311	364

h	k	l	$10F_o$	$10F_c$
2	0	$\bar{2}$	103	118
3	0	2	222	294
3	0	$\bar{2}$	109	110
4	0	2	62	71
4	0	$\bar{2}$	69	74
0	1	2	17	47
0	1	$\bar{2}$	341	411
1	1	2	393	487
1	$\bar{1}$	2	128	123
1	1	$\bar{2}$	339	419
1	$\bar{1}$	$\bar{2}$	221	-186
2	1	2	401	465
2	$\bar{1}$	2	57	45
2	1	$\bar{2}$	117	118
2	$\bar{1}$	$\bar{2}$	239	249
3	1	2	124	126
3	$\bar{1}$	2	145	137
3	1	$\bar{2}$	55	-43
3	$\bar{1}$	$\bar{2}$	370	408
4	1	2	45	-39
4	$\bar{1}$	2	182	168
4	1	$\bar{2}$	137	138
4	$\bar{1}$	$\bar{2}$	147	141
0	2	2	26	48
0	2	$\bar{2}$	45	54
1	2	2	800	866
1	$\bar{2}$	2	320	344
1	2	$\bar{2}$	19-	13
1	$\bar{2}$	$\bar{2}$	157	-132
2	2	2	111	105
2	$\bar{2}$	2	174	175
2	2	$\bar{2}$	253	258
2	$\bar{2}$	$\bar{2}$	221	180
3	2	2	174	-153
3	$\bar{2}$	2	140	130
3	2	$\bar{2}$	230	233
3	$\bar{2}$	$\bar{2}$	199	179
4	2	2	183	160
4	$\bar{2}$	2	60	56
4	2	$\bar{2}$	137	131
4	$\bar{2}$	$\bar{2}$	243	220
0	3	2	334	322
0	3	$\bar{2}$	55	-55
1	3	2	51	63
1	$\bar{3}$	2	131	103
1	3	$\bar{2}$	151	136
1	$\bar{3}$	$\bar{2}$	377	405
2	3	2	75	-33
2	$\bar{3}$	2	241	232
2	3	$\bar{2}$	297	281
2	$\bar{3}$	$\bar{2}$	88	73
3	3	2	118	95
3	$\bar{3}$	2	232	209
3	3	$\bar{2}$	236	226
3	$\bar{3}$	$\bar{2}$	65	-49
4	3	2	268	244
4	$\bar{3}$	2	106	93
4	3	$\bar{2}$	29-	26
4	$\bar{3}$	$\bar{2}$	78	72
0	4	2	316	312
0	4	$\bar{2}$	247	246
1	4	2	247	-215
1	$\bar{4}$	2	28-	13
1	4	$\bar{2}$	230	224
1	$\bar{4}$	$\bar{2}$	277	271
2	4	2	95	89
2	$\bar{4}$	2	28-	17
2	4	$\bar{2}$	99	93
2	$\bar{4}$	$\bar{2}$	139	128
3	4	2	361	341
3	$\bar{4}$	2	136	124
3	4	$\bar{2}$	20-	14
3	$\bar{4}$	$\bar{2}$	96	85
4	4	2	67	64
4	$\bar{4}$	2	190	179
4	4	$\bar{2}$	29-	8
4	$\bar{4}$	$\bar{2}$	29-	-25
0	5	2	84	88
0	5	$\bar{2}$	241	258
1	5	2	184	168
1	$\bar{5}$	2	133	126
1	5	$\bar{2}$	113	108
1	$\bar{5}$	$\bar{2}$	29-	11
2	5	2	167	152
2	$\bar{5}$	2	29-	20
2	5	$\bar{2}$	38	-35
2	$\bar{5}$	$\bar{2}$	101	93
3	5	2	118	103
3	$\bar{5}$	2	18-	-2
3	5	$\bar{2}$	18-	18
3	$\bar{5}$	$\bar{2}$	159	153
4	5	2	29-	-4
4	$\bar{5}$	2	71	68
4	5	$\bar{2}$	113	103
4	$\bar{5}$	$\bar{2}$	123	116
0	6	2	38-	35
0	6	$\bar{2}$	36-	-8
1	6	2	221	204
1	$\bar{6}$	2	144	142
1	6	$\bar{2}$	52	46

h	k	l	$10F_o$	$10F_c$
1	$\bar{6}$	$\bar{2}$	98	88
2	6	2	77	86
2	$\bar{6}$	2	145	139
2	6	$\bar{2}$	81	82
2	$\bar{6}$	$\bar{2}$	29	28
3	6	2	83	-65
3	$\bar{6}$	2	14-	21
3	6	$\bar{2}$	69	68
3	$\bar{6}$	$\bar{2}$	34	40
4	6	2	90	84
4	$\bar{6}$	2	5-	-16
4	$\bar{6}$	$\bar{2}$	122	136
0	7	2	34-	40
0	7	$\bar{2}$	25-	-14
1	7	2	26-	-6
1	$\bar{7}$	2	50	45
1	7	$\bar{2}$	51	58
1	$\bar{7}$	$\bar{2}$	131	134
2	7	2	49	48
2	$\bar{7}$	2	77	75
2	7	$\bar{2}$	113	122
2	$\bar{7}$	$\bar{2}$	103	103
3	7	2	65	71
3	$\bar{7}$	$\bar{2}$	9-	5
4	7	2	70	82
0	8	2	21-	16
1	8	2	16-	3
0	0	3	63	-33
1	0	3	135	-143
1	0	$\bar{3}$	319	396
2	0	3	160	153
2	0	$\bar{3}$	379	417
3	0	3	500	509
3	0	$\bar{3}$	124	129
4	0	3	125	137
4	0	$\bar{3}$	27-	25
0	1	3	268	271
0	1	$\bar{3}$	75	69
1	1	3	372	444
1	$\bar{1}$	3	92	-73
1	1	$\bar{3}$	403	512
1	$\bar{1}$	$\bar{3}$	106	-55
2	1	3	207	223
2	$\bar{1}$	3	162	136
2	1	$\bar{3}$	238	245
2	$\bar{1}$	$\bar{3}$	133	122
3	1	3	81	72
3	$\bar{1}$	3	262	264
3	1	$\bar{3}$	32	-22
3	$\bar{1}$	$\bar{3}$	272	285
4	1	3	27	28
4	$\bar{1}$	3	213	207
4	1	$\bar{3}$	46	57
4	$\bar{1}$	$\bar{3}$	204	187
0	2	3	405	437
0	2	$\bar{3}$	299	318
1	2	3	349	373
1	$\bar{2}$	3	341	384
1	2	$\bar{3}$	22-	-15
1	$\bar{2}$	$\bar{3}$	101	86
2	2	3	254	257
2	$\bar{2}$	3	162	152
2	2	$\bar{3}$	54	56
2	$\bar{2}$	$\bar{3}$	97	-81
3	2	3	167	-135
3	$\bar{2}$	3	111	-82
3	2	$\bar{3}$	188	176
3	$\bar{2}$	$\bar{3}$	181	170
4	2	3	36	33
4	$\bar{2}$	3	112	100
4	2	$\bar{3}$	128	111
4	$\bar{2}$	$\bar{3}$	209	194
0	3	3	105	98
0	3	$\bar{3}$	221	221
1	3	3	107	87
1	$\bar{3}$	3	251	245
1	3	$\bar{3}$	26-	-22
1	$\bar{3}$	$\bar{3}$	249	231
2	3	3	222	207
2	$\bar{3}$	3	168	156
2	3	$\bar{3}$	109	101
2	$\bar{3}$	$\bar{3}$	255	232
3	3	3	180	165
3	$\bar{3}$	3	52	46
3	3	$\bar{3}$	249	238
3	$\bar{3}$	$\bar{3}$	60	52
4	3	3	117	94
4	$\bar{3}$	3	35	38
4	3	$\bar{3}$	66	61
4	$\bar{3}$	$\bar{3}$	29-	18
0	4	3	109	-96
0	4	$\bar{3}$	60	66
1	4	3	233	202
1	$\bar{4}$	3	29-	-4
1	4	$\bar{3}$	273	296
1	$\bar{4}$	$\bar{3}$	119	109
2	4	3	181	162
2	$\bar{4}$	3	65	58
2	4	$\bar{3}$	160	152
2	$\bar{4}$	$\bar{3}$	290	280

h	k	l	$10F_o$	$10F_c$
3	4	3	200	177
3	$\bar{4}$	3	195	182
3	4	$\bar{3}$	49	-37
3	$\bar{4}$	$\bar{3}$	111	97
4	4	3	123	104
4	$\bar{4}$	3	124	123
4	4	$\bar{3}$	75	78
4	$\bar{4}$	$\bar{3}$	52	52
0	5	3	210	204
0	5	$\bar{3}$	116	119
1	5	3	201	189
1	$\bar{5}$	3	65	62
1	5	$\bar{3}$	184	186
1	$\bar{5}$	$\bar{3}$	52	50
2	5	3	28-	36
2	$\bar{5}$	3	28-	20
2	5	$\bar{3}$	65	63
2	$\bar{5}$	$\bar{3}$	31	-31
3	5	3	20-	4
3	$\bar{5}$	3	85	81
3	5	$\bar{3}$	70	-61
3	$\bar{5}$	$\bar{3}$	131	124
4	5	3	99	88
4	$\bar{5}$	3	94	93
4	5	$\bar{3}$	121	117
4	$\bar{5}$	$\bar{3}$	148	146
0	6	3	277	302
0	6	$\bar{3}$	106	112
1	6	3	30-	3
1	$\bar{6}$	3	128	130
1	6	$\bar{3}$	56	-46
1	$\bar{6}$	$\bar{3}$	113	106
2	6	3	72	-53
2	$\bar{6}$	3	122	116
2	6	$\bar{3}$	22-	-2
2	$\bar{6}$	$\bar{3}$	48	-41
3	6	3	72	63
3	$\bar{6}$	3	12-	5
3	6	$\bar{3}$	150	170
3	$\bar{6}$	$\bar{3}$	52	46
4	6	$\bar{3}$	87	90
4	$\bar{6}$	$\bar{3}$	39	49
0	7	3	34-	9
0	7	$\bar{3}$	20-	18
1	7	3	27-	-12
1	$\bar{7}$	3	60	68
1	7	$\bar{3}$	14-	-9
1	$\bar{7}$	$\bar{3}$	114	118
2	7	3	53	71
2	$\bar{7}$	$\bar{3}$	130	137
3	7	3	128	122
3	$\bar{7}$	$\bar{3}$	8-	6
4	7	3	36	43
0	8	3	84	-92
1	8	3	68	66
2	8	3	119	145
0	0	4	84	80
1	0	4	138	142
1	0	$\bar{4}$	89	79
2	0	4	342	386
2	0	$\bar{4}$	255	260
3	0	4	177	190
3	0	$\bar{4}$	255	252
4	0	4	23-	15
4	0	$\bar{4}$	29-	26
0	1	4	217	224
0	1	$\bar{4}$	233	224
1	1	4	299	305
1	$\bar{1}$	4	60	-38
1	1	$\bar{4}$	308	337
1	$\bar{1}$	$\bar{4}$	127	107
2	1	4	161	146
2	$\bar{1}$	4	133	121
2	1	$\bar{4}$	265	263
2	$\bar{1}$	$\bar{4}$	22-	2
3	1	4	87	78
3	$\bar{1}$	4	213	216
3	1	$\bar{4}$	55	40
3	$\bar{1}$	$\bar{4}$	194	198
4	1	4	112	111
4	$\bar{1}$	4	150	146
4	1	$\bar{4}$	113	-90
4	$\bar{1}$	$\bar{4}$	261	259
0	2	4	254	275
0	2	$\bar{4}$	244	253
1	2	4	141	112
1	$\bar{2}$	4	236	240
1	2	$\bar{4}$	289	283
1	$\bar{2}$	$\bar{4}$	266	258
2	2	4	123	-102
2	$\bar{2}$	4	67	-54
2	2	$\bar{4}$	55	53
2	$\bar{2}$	$\bar{4}$	163	149
3	2	4	230	212
3	$\bar{2}$	4	18-	-3
3	2	$\bar{4}$	84	-76
3	$\bar{2}$	$\bar{4}$	52	52
4	2	4	259	238
4	$\bar{2}$	4	193	199
4	2	$\bar{4}$	188	172

h	k	l	$10F_o$	$10F_c$
4	$\bar{2}$	$\bar{4}$	97	100
0	3	4	30	-22
0	3	$\bar{4}$	124	140
1	3	4	74	66
1	$\bar{3}$	4	288	302
1	2	$\bar{4}$	28-	1
1	$\bar{3}$	$\bar{4}$	213	187
2	3	4	244	242
2	$\bar{3}$	4	112	104
2	3	$\bar{4}$	60	55
2	$\bar{3}$	$\bar{4}$	323	329
3	3	4	232	210
3	$\bar{3}$	4	19-	18
3	3	$\bar{4}$	148	120
3	$\bar{3}$	$\bar{4}$	106	102
4	3	4	74	59
4	$\bar{3}$	4	58	51
4	3	$\bar{4}$	223	227
4	$\bar{3}$	$\bar{4}$	50	-39
0	4	4	38	38
0	4	$\bar{4}$	88	87
1	4	4	186	173
1	$\bar{4}$	4	30-	11
1	4	$\bar{4}$	87	59
1	$\bar{4}$	$\bar{4}$	41	40
2	4	4	388	388
2	$\bar{4}$	4	205	209
2	4	$\bar{4}$	99	90
2	$\bar{4}$	$\bar{4}$	86	80
3	4	4	18-	18
3	$\bar{4}$	4	175	176
3	4	$\bar{4}$	219	218
3	$\bar{4}$	$\bar{4}$	187	184
4	4	4	153	-120
4	$\bar{4}$	4	26-	-17
4	4	$\bar{4}$	48	40
4	$\bar{4}$	$\bar{4}$	114	111
0	5	4	249	248
0	5	$\bar{4}$	95	96
1	5	4	195	190
1	$\bar{5}$	4	29-	-3
1	5	$\bar{4}$	177	160
1	$\bar{5}$	$\bar{4}$	47	45
2	5	4	29-	23
2	$\bar{5}$	4	93	89
2	5	$\bar{4}$	49	56
2	$\bar{5}$	$\bar{4}$	67	-52
3	5	4	25	-15
3	$\bar{5}$	4	93	97
3	5	$\bar{4}$	60	64
3	$\bar{5}$	$\bar{4}$	68	67
4	5	4	60	62
4	$\bar{5}$	4	55	65
4	5	$\bar{4}$	13-	13
4	$\bar{5}$	$\bar{4}$	164	162
0	6	4	95	113
0	6	$\bar{4}$	64	74
1	6	4	112	98
1	$\bar{6}$	4	159	167
1	6	$\bar{4}$	22-	23
1	$\bar{6}$	$\bar{4}$	134	124
2	6	4	113	-97
2	$\bar{6}$	4	18-	-3
2	6	$\bar{4}$	70	76
2	$\bar{6}$	$\bar{4}$	97	93
3	6	4	83	75
3	$\bar{6}$	4	7-	-14
3	$\bar{6}$	$\bar{4}$	15-	-9
4	6	4	210	210
0	7	4	33-	-12
1	7	4	26-	26
1	$\bar{7}$	$\bar{4}$	70	70
2	7	4	83	89
2	$\bar{7}$	$\bar{4}$	118	115
3	7	4	84	83
4	7	4	41	51
0	8	4	64	75
1	8	4	16-	23
1	$\bar{8}$	$\bar{4}$	11-	10
2	8	4	67	92
0	0	5	47	38
1	0	5	260	270
1	0	$\bar{5}$	218	196
2	0	5	201	191
2	0	$\bar{5}$	159	147
3	0	5	78	67
3	0	$\bar{5}$	46	35
4	0	5	33	32
4	0	$\bar{5}$	210	199
0	1	5	463	545
0	1	$\bar{5}$	116	-98
1	1	5	130	117
1	$\bar{1}$	5	164	142
1	1	$\bar{5}$	177	174
1	$\bar{1}$	$\bar{5}$	229	220
2	1	5	127	-98
2	$\bar{1}$	5	271	248
2	1	$\bar{5}$	355	351
2	$\bar{1}$	$\bar{5}$	85	-68
3	1	5	58	57

h	k	l	$10F_o$	$10F_c$
3	$\bar{1}$	5	201	193
3	1	$\bar{5}$	77	57
3	$\bar{1}$	$\bar{5}$	55	57
4	1	5	193	196
4	$\bar{1}$	5	67	59
4	1	$\bar{5}$	34	39
4	$\bar{1}$	$\bar{5}$	132	117
0	2	5	239	246
0	2	$\bar{5}$	282	305
1	2	5	52	34
1	$\bar{2}$	5	55	43
1	2	$\bar{5}$	166	154
1	$\bar{2}$	$\bar{5}$	193	190
2	2	5	75	63
2	$\bar{2}$	5	91	79
2	2	$\bar{5}$	29-	7
2	$\bar{2}$	$\bar{5}$	200	195
3	2	5	179	172
3	$\bar{2}$	5	121	113
3	2	$\bar{5}$	175	151
3	$\bar{2}$	$\bar{5}$	120	120
4	2	5	219	221
4	$\bar{2}$	5	125	116
4	2	$\bar{5}$	62	56
4	$\bar{2}$	$\bar{5}$	36	-47
0	3	5	182	-160
0	3	$\bar{5}$	283	297
1	3	5	164	153
1	$\bar{3}$	5	202	196
1	3	$\bar{5}$	52	52
1	$\bar{3}$	$\bar{5}$	90	71
2	3	5	375	351
2	$\bar{3}$	5	68	65
2	3	$\bar{5}$	125	-125
2	$\bar{3}$	$\bar{5}$	293	288
3	3	5	193	176
3	$\bar{3}$	5	20-	-21
3	3	$\bar{5}$	189	171
3	$\bar{3}$	$\bar{5}$	215	212
4	3	5	28-	-6
4	$\bar{3}$	5	73	67
4	3	$\bar{5}$	191	191
4	$\bar{3}$	$\bar{5}$	28-	42
0	4	5	72	72
0	4	$\bar{5}$	38-	6
1	4	5	223	199
1	$\bar{4}$	5	202	194
1	4	$\bar{5}$	30-	1
1	$\bar{4}$	$\bar{5}$	48	45
2	4	5	159	148
2	$\bar{4}$	5	108	107
2	4	$\bar{5}$	181	177
2	$\bar{4}$	$\bar{5}$	84	78
3	4	5	93	91
3	$\bar{4}$	5	86	82
3	4	$\bar{5}$	102	94
3	$\bar{4}$	$\bar{5}$	123	121
4	4	5	30	-23
4	$\bar{4}$	5	22-	30
4	4	$\bar{5}$	80	75
4	$\bar{4}$	$\bar{5}$	146	158
0	5	5	212	223
0	5	$\bar{5}$	34-	-30
1	5	5	49	56
1	$\bar{5}$	5	26-	15
1	5	$\bar{5}$	119	101
1	$\bar{5}$	$\bar{5}$	191	170
2	5	5	29-	-29
2	$\bar{5}$	5	100	90
2	5	$\bar{5}$	247	236
2	$\bar{5}$	$\bar{5}$	29-	4
3	5	5	61	63
3	$\bar{5}$	5	134	147
3	5	$\bar{5}$	22	26
3	$\bar{5}$	$\bar{5}$	17-	-18
4	5	5	131	120
4	$\bar{5}$	$\bar{5}$	89	90
0	6	5	68	69
0	6	$\bar{5}$	63	69
1	6	5	30-	-4
1	$\bar{6}$	5	16-	2
1	6	$\bar{5}$	110	128
1	$\bar{6}$	$\bar{5}$	123	121
2	6	5	116	107
2	$\bar{6}$	$\bar{5}$	82	79
3	6	5	68	70
3	$\bar{6}$	$\bar{5}$	52	53
4	6	5	56	57
0	7	5	32-	5
1	7	5	132	131
1	$\bar{7}$	$\bar{5}$	47	-41
2	7	5	110	122
2	$\bar{7}$	$\bar{5}$	76	80
3	7	5	14-	12
4	7	5	33	-26
1	8	5	107	131
2	8	5	12-	-12
0	0	6	298	304
1	0	6	173	174
1	0	$\bar{6}$	72	62

Table 4 (*cont.*)

h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$	h	k	l	10F$_o$	10F$_c$
2	0	6	237	220	1	4	6	67	67	3	1	7	157	153	1	5	7	171	168	1	3	8	47	-44	4	1	9	38	-27	2	1	10	11-	13
2	0	6	106	-97	1	4	6	110	96	3	1	7	55	46	2	5	7	78	83	1	3	8	171	166	4	1	9	122	118	2	1	10	95	92
3	0	6	18-	16	1	4	6	30	55	3	1	7	202	188	2	5	7	136	134	1	3	8	84	77	0	2	9	43	58	3	1	10	90	79
3	0	6	208	199	2	4	6	93	82	3	1	7	67	61	3	5	7	170	168	2	3	8	29-	26	0	2	9	93	84	3	1	10	39	39
4	0	6	39	-32	2	4	6	197	203	4	1	7	115	107	3	5	7	44	50	2	3	8	23-	9	1	2	9	158	147	4	1	10	20-	-6
4	0	6	189	183	2	4	6	24-	34	4	1	7	29-	9	4	5	7	66	73	2	3	8	121	113	1	2	9	137	131	0	2	10	193	196
0	1	6	233	233	2	4	6	29-	32	4	1	7	62	63	0	6	7	34-	47	2	3	8	117	-112	1	2	9	44	34	0	2	10	115	138
0	1	6	57	58	3	4	6	19-	-10	4	1	7	77	74	1	6	7	82	80	3	3	8	80	-70	1	2	9	75	62	1	2	10	69	58
1	1	6	71	57	3	4	6	15-	26	0	2	7	124	124	1	6	7	42	-28	3	3	8	158	168	2	2	9	183	187	1	2	10	122	130
1	1	6	235	225	3	4	6	114	108	0	2	7	56	-54	2	6	7	129	124	3	3	8	96	90	2	2	9	70	66	1	2	10	11-	25
1	1	6	28-	3	3	4	6	99	100	1	2	7	59	50	2	6	7	64	59	4	3	8	82	82	2	2	9	16-	28	1	2	10	88	81
1	1	6	316	313	4	4	6	73	70	1	2	7	30-	13	3	6	7	60	63	0	4	8	121	128	2	2	9	25-	-4	2	2	10	26-	-24
2	1	6	80	-58	4	4	6	17-	-9	1	2	7	139	141	4	6	7	9-	-5	0	4	8	87	92	3	2	9	56	-36	2	2	10	15-	27
2	1	6	318	315	4	4	6	60	56	1	2	7	64	59	0	7	7	76	84	1	4	8	77	-56	3	2	9	13-	9	2	2	10	19-	1
2	1	6	87	77	0	5	6	52	71	2	2	7	251	248	1	7	7	118	121	1	4	8	50	52	3	2	9	119	119	3	2	10	87	83
2	1	6	131	115	0	5	6	28-	-38	2	2	7	262	261	2	7	7	20-	27	1	4	8	109	119	4	2	9	26-	25	4	2	10	28	33
3	1	6	168	164	1	5	6	46	-36	2	2	7	165	160	3	7	7	15	-18	1	4	8	194	189	0	3	9	170	176	0	3	10	33-	43
3	1	6	23	33	1	5	6	36	39	2	2	7	45	35	0	0	8	38-	33	2	4	8	64	90	0	3	9	23-	10	1	3	10	27-	15
3	1	6	282	280	1	5	6	93	89	3	2	7	144	140	1	0	8	30-	9	2	4	8	15-	18	1	3	9	104	93	1	3	10	143	137
3	1	6	129	-109	1	5	6	219	214	3	2	7	120	116	1	0	8	252	239	2	4	8	26-	32	1	3	9	43	44	2	3	10	67	64
4	1	6	218	219	2	5	6	60	47	3	2	7	174	160	2	0	8	203	183	3	4	8	96	98	1	3	9	53	67	2	3	10	84	78
4	1	6	60	-47	2	5	6	173	168	3	2	7	254	244	2	0	8	116	103	3	4	8	48	47	1	3	9	64	62	3	3	10	60	74
4	1	6	104	99	2	5	6	105	112	4	2	7	32	-22	3	0	8	60	55	4	4	8	70	68	2	3	9	72	-57	4	3	10	87	92
4	1	6	129	125	2	5	6	92	98	4	2	7	37	41	3	0	8	41	32	0	5	8	41	-51	2	3	9	108	118	0	4	10	65	-77
0	2	6	33-	-3	3	5	6	170	168	4	2	7	23	20	4	0	8	100	98	1	5	8	29-	33	2	3	9	75	65	1	4	10	65	70
0	2	6	79	84	3	5	6	60	67	4	2	7	93	84	4	0	8	16-	35	1	5	8	85	87	3	3	9	68	73	1	4	10	43	44
1	2	6	83	62	3	5	6	16-	-13	0	3	7	106	114	0	1	8	38-	-4	2	5	8	121	123	3	3	9	35	34	2	4	10	194	203
1	2	6	177	158	4	5	6	116	116	0	3	7	95	102	0	1	8	118	111	2	5	8	186	187	4	3	9	132	128	2	4	10	96	111
1	2	6	111	95	4	5	6	47	48	1	3	7	273	278	1	1	8	30-	26	3	5	8	130	132	0	4	9	37	40	3	4	10	14-	20
1	2	6	259	260	0	6	6	37-	-9	1	3	7	29-	-16	1	1	8	202	198	3	5	8	9-	24	1	4	9	87	83	4	4	10	16-	4
2	2	6	75	67	1	6	6	29-	32	1	3	7	219	227	1	1	8	62	55	4	5	8	23-	15	1	4	9	47	40	0	5	10	50	63
2	2	6	29-	-11	1	6	6	70	65	1	3	7	30-	-2	1	1	8	121	98	0	6	8	31-	31	2	4	9	27-	0	1	5	10	146	135
2	2	6	255	252	2	6	6	100	99	2	3	7	70	68	2	1	8	132	127	1	6	8	134	131	2	4	9	173	171	1	5	10	53	-48
2	2	6	256	260	2	6	6	109	115	2	3	7	79	66	2	1	8	151	132	1	6	8	32	30	3	4	9	122	124	2	5	10	54	56
3	2	6	192	197	3	6	6	164	159	2	3	7	56	57	2	1	8	27-	17	2	6	8	35	42	4	4	9	90	79	3	5	10	11-	6
3	2	6	151	146	3	6	6	76	88	2	3	7	85	89	2	1	8	251	257	2	6	8	53	57	0	5	9	32-	25	1	6	10	64	65
3	2	6	47	46	4	6	6	23-	13	3	3	7	36	-23	3	1	8	220	231	3	6	8	14-	5	1	5	9	86	83	2	6	10	69	-83
3	2	6	20-	0	0	7	6	41	55	3	3	7	106	105	3	1	8	48	-47	4	6	8	23	32	1	5	9	38	38	0	0	11	22-	23
4	2	6	177	169	1	7	6	152	150	3	3	7	13-	-8	3	1	8	120	105	0	7	8	104	127	2	5	9	149	151	1	0	11	100	117
4	2	6	172	165	1	7	6	42	-38	3	3	7	77	73	3	1	8	91	91	1	7	8	43	38	2	5	9	16-	15	1	0	11	12-	-1
4	2	6	24-	2	2	7	6	76	79	4	3	7	64	58	4	1	8	55	53	2	7	8	16-	-29	3	5	9	15-	12	3	0	11	7-	22
4	2	6	39	38	2	7	6	14-	21	4	3	7	108	110	4	1	8	71	42	3	7	8	6-	6	4	5	9	39	-35	0	1	11	95	91
0	3	6	109	108	3	7	6	36	-31	4	3	7	147	141	4	1	8	18-	-25	0	0	9	114	105	0	6	9	118	138	1	1	11	118	118
0	3	6	167	187	4	7	6	10-	-13	0	4	7	81	97	0	2	8	120	122	1	0	9	29-	-13	1	6	9	32	29	1	1	11	45	45
1	3	6	219	208	0	0	7	218	219	0	4	7	182	177	0	2	8	108	116	1	0	9	157	147	1	6	9	68	73	1	1	11	43	32
1	3	6	101	88	1	0	7	269	267	1	4	7	93	86	1	2	8	281	278	2	0	9	28-	31	2	6	9	47	48	2	1	11	7-	4
1	3	6	128	119	1	0	7	86	69	1	4	7	86	90	1	2	8	111	100	2	0	9	158	155	3	6	9	11-	-7	3	1	11	11-	-19
1	3	6	28-	-26	2	0	7	70	-61	1	4	7	74	67	1	2	8	27-	14	3	0	9	120	185	1	7	9	8-	3	0	2	11	100	101
2	3	6	201	191	2	0	7	124	110	1	4	7	224	220	1	2	8	93	-79	3	0	9	12-	15	0	0	10	32-	-13	1	2	11	5-	3
2	3	6	48	-47	3	0	7	35	28	2	4	7	29-	-29	2	2	8	29-	31	0	1	9	47	-62	1	0	10	25-	34	1	2	11	142	150
2	3	6	112	102	3	0	7	19-	-9	2	4	7	22-	-13	2	2	8	70	61	0	1	9	199	207	1	0	10	108	104	2	2	11	20-	-18
2	3	6	126	123	4	0	7	136	135	2	4	7	18-	12	2	2	8	95	95	1	1	9	94	82	2	0	10	100	103	3	2	11	60	65
3	3	6	19	30	4	0	7	116	106	2	4	7	115	119	2	2	8	118	107	1	1	9	80	71	2	0	10	151	152	0	3	11	25-	39
3	3	6	115	107	0	1	7	188	193	3	4	7	37	41	3	2	8	55	60	1	1	9	141	118	3	0	10	95	94	1	3	11	50	-33
3	3	6	16-	10	0	1	7	44	51	3	4	7	67	63	3	2	8	137	129	1	1	9	95	85	0	1	10	53	61	1	3	11	106	108
3	3	6	234	233	1	1	7	49	-26	3	4	7	105	-95	3	2	8	126	120	2	1	9	249	253	0	1	10	125	125	2	3	11	20-	37
4	3	6	29-	0	1	1	7	273	269	4	4	7	163	156	3	2	8	133	137	2	1	9	59	-57	1	1	10	91	82	3	3	11	129	144
4	3	6	145	141	1	1	7	91	-78	4	4	7	58	58	4	2	8	31	39	2	1	9	22-	27	1	1	10	53	51	0	4	11	22-	37
4	3	6	53	57	1	1	7	293	268	0	5	7	39	50	4	2	8	21-	5	2	1	9	152	146	1	1	10	110	109	1	4	11	74	82
4	3	6	26-	29	2	1	7	122	114	0	5	7	56	55	4	2	8	115	117	3	1	9	89	86	1	1	10	58	47	1	4	11	10-	-24
0	4	6	202	207	2	1	7	171	139	1	5	7	84	-70	0	3	8	235	253	3	1	9	66	54	2	1	10	95	97	2	4	11	86	87
0	4	6	36-	29	2	1	7	196	187	1	5	7	76	88	0	3	8	90	96	3	1	9	9-	1-	2	1	10	21-	9	3	4	11	61	62
1	4	6	161	153	2	1	7	29-	18	1	5	7	10-	9	1	3	8	171	174	3	1	9	120	122										

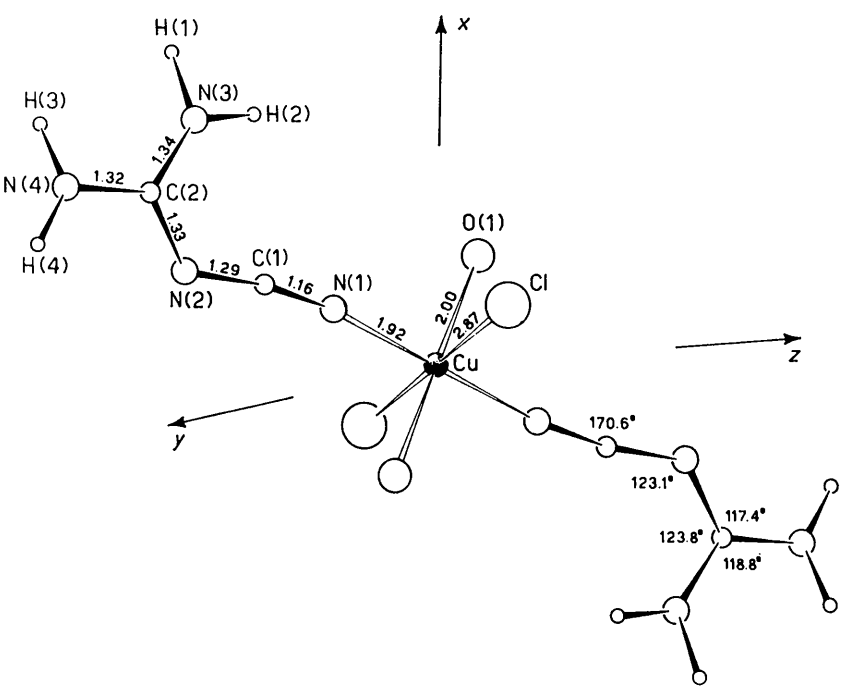

Fig. 1. Clinographic projection of the structure.

which concerns coordinated and uncoordinated dicyandiamide.

Fig. 2 shows the packing of the molecules, which is mainly determined by the following hydrogen-bonds:

O–H···Cl^{i}	3·18(1) Å	
O–H···Cl^{ii}	3·14(1) (not shown in figure)	
N(4)–H(4)···Cl^{iii}	3·30(1)	N(4)H(4)Cl^{iii} 160·7°
N(4)–H(3)···Cl^{iv}	3·28(1)	N(4)H(3)Cl^{iv} 166·4°

The other packing contacts less than 3·5 Å are as follows:

O···N(1^{i})	3·43(2) Å
O···N(3^{i})	3·49(1)
N(1)···N(3^{v})	3·40(1)
N(2)···N(3^{v})	3·08(2)
N(3)···C(1^{vi})	3·11(2)

i $= 1-x, \bar{y}, \bar{z}$
ii $= x, y-1, \bar{z}$
iii $= x, y, z-1$
iv $= x+1, y, z-1$
v $= x-1, y, z$
vi $= x+1, y, z$

The authors are indebted to the Consiglio Nazionale delle Ricerche (Rome) for financial support.

Table 5. *Interatomic distances and bond angles in coordinated and uncoordinated dicyandiamide*

	I	II	III	IV
N(1)–C(1)	1·151 Å	1·192 Å	1·16 (1) Å	1·16 (1) Å
C(1)–N(2)	1·299	1·292	1·29 (1)	1·29 (1)
N(2)–C(2)	1·330	1·335	1·33 (1)	1·36 (1)
C(2)–N(3)	1·333	1·332	1·34 (1)	1·33 (1)
C(2)–N(4)	—	—	1·32 (1)	1·33 (1)
N(1)C(1)N(2)	175°	180°	170·6 (0·9)°	172·6 (0·6)°
C(1)N(2)C(2)	119	119	123·1 (0·7)	118·6 (0·6)
N(2)C(2)N(3)	123	124	123·8 (0·4)	124·4 (0·5)
N(2)C(2)N(4)	117	118	117·4 (0·7)	116·6(0·5)
N(3)C(2)N(4)	—	—	118·8 (0·7)	118·9 (0·6)

I Dicyandiamide (neutron diffraction) (Rannev, Ozerov, Datt & Kshnyakina, 1966).
II Dicyandiamide (X-ray diffraction) (Zvonkova, Krivnov & Khvatkina, 1964).
III Dichlorodiaquobis(dicyandiamide)copper(II) (present paper).
IV 1-(2-Aminoethyl)biguanidecyanoguanidinecopper(II) sulphate monohydrate (Coghi, Mangia, Nardelli, Pelizzi & Sozzi, 1968).

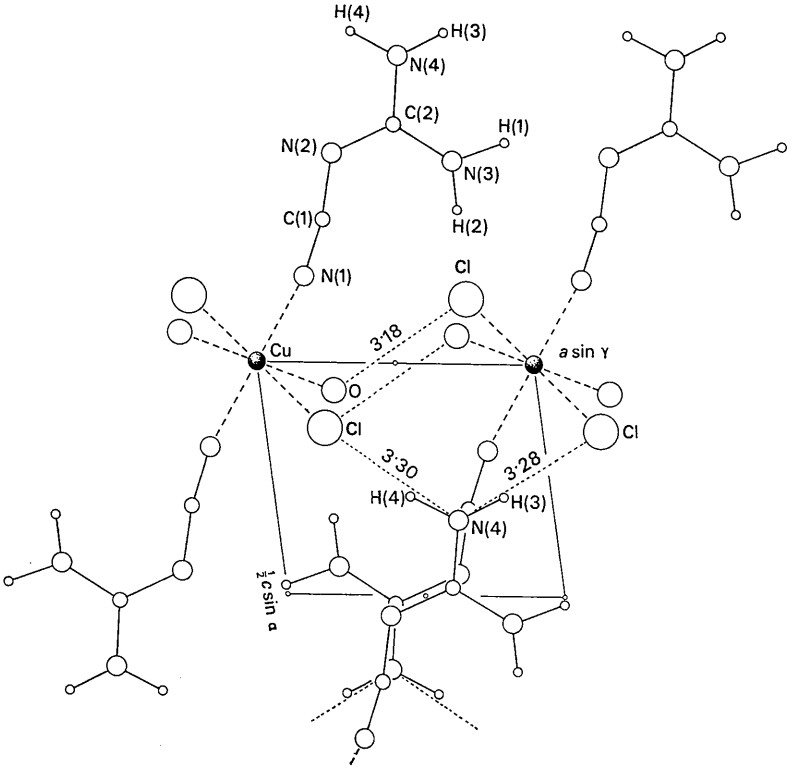

Fig. 2. Projection of the structure along [010].

References

COGHI, L., MANGIA, A., NARDELLI, M., PELIZZI, G. & SOZZI, L. (1968). *Chem. Comm.* p. 1475.

EVERTSSON, B. (1969). *Acta Cryst.* B**25**, 30.

FREEMAN, H. C. & SMITH, J. E. W. L. (1966). *Acta Cryst.* **20**, 153.

NARDELLI, M., FAVA GASPARRI, G., BOLDRINI, P. & GIRALDI BATTISTINI, G. (1965). *Acta Cryst.* **19**, 491.

RANNEV, N. V., OZEROV, R. P., DATT, I. D. & KSHNYAKINA, A. N. (1966). *Kristallografiya*, **11**, 175. *Soviet Physics-Crystallography*, **11**, 177.

ZVONKOVA, Z. V., KRIVNOV, V. YA. & KHVATKINA, A. N. (1964). *Doklady Akad. Nauk*, **155**, 2; see also *Kristallografiya*, **11**, 175.

Acta Cryst. (1971). B**27**, 197

The Crystal Structure of [2.2]Metaparacyclophane-1,9-diene

BY A. W. HANSON

Biochemistry Laboratory, National Research Council, Ottawa, Canada

(*Received* 26 *January* 1970)

[2.2]Metaparacyclophane-1,9-diene, $C_{16}H_{12}$, crystallizes in the orthorhombic system, space group *Pbca*, with $a=14{\cdot}91\pm1$, $b=18{\cdot}51\pm1$, $c=8{\cdot}133\pm5$ Å, $Z=8$. The intensity data were measured with a four-circle diffractometer and scintillation counter. The structure was determined by symbolic addition and refined by block-diagonal least-squares analysis of 1378 reflexions to a final R index of 0·046. The two aromatic rings are inclined to each other at 41°. Both show significant boat distortion, which is moderate for the *meta*-bridged ring, and severe for the *para*-bridged ring.

[2.2]Metaparacyclophane-1,9-diene (I; Hylton & Boekelheide, 1968) is one of a series of compounds prepared by Professor Boekelheide and his associates. Nuclear magnetic resonance studies of the material in solution fail to resolve the individual protons of the *para*-bridged ring, suggesting that the molecule has *mm*2 symmetry, with the two rings perpendicular to each other (Boekelheide, 1968). This conformation could, of course, be simulated by rapid 'flipping' of the molecule between two conformations related by the apparent symmetry. The X-ray analysis was undertaken in order to determine the conformation in the solid state. It is found that the rings are in fact by no means perpendicular, but are inclined to each other at 41°.

Experimental

Crystal data

$C_{16}H_{12}$ F.W. 204·3

Orthorhombic,
$a=14{\cdot}91\pm1$, $b=18{\cdot}51\pm1$, $c=8{\cdot}133\pm5$ Å

(Cu $K\alpha_1$, $\lambda=1{\cdot}5405$ Å), $V=2245$ Å^3, $D_m=1{\cdot}20$ g.cm^{-3}, $Z=8$, $D_x=1{\cdot}21$ g.cm^{-3}, $\mu=6{\cdot}3$ cm^{-1}.

Space group *Pbca* (D_{2h}^{15}) (from precession photographs. Systematic absences: $0kl$ for k odd, $h0l$ for l odd, $hk0$ for h odd).

The crystals supplied were colourless and translucent, displaying various faces of which [010] was the most prominent. The specimen used for data collection was a triangular plate, 0·3 mm thick, and 0·5 mm to the side. The material was found to decompose in conditions of moderate humidity, and the specimen was therefore enclosed in a thin-walled Lindemann glass capillary.

The intensities were measured with a Picker four-circle diffractometer and scintillation counter, using nickel-filtered Cu $K\alpha$ radiation with pulse-height discrimination. The θ–2θ scan method was used (2° for $2\theta<100°$, 3° otherwise), and background counts were measured at the beginning and end of each scan. Reflexions for which the net count was less than 10, or less than 20% of the gross count, were treated as unobserved. The yield of observed reflexions was rather low. In the range explored ($2\theta<130°$), 1380 of a possible 1904 reflexions were observed, and of these the net count exceeded four times the threshold value for only 956. Absorption corrections were not applied.

The structure was determined routinely by the symbolic addition procedure of Karle & Karle (1966). Refinement was by block-diagonal least-squares. The weighting scheme used was $w=w_1w_2$, where

$$w_1=F_o/10 \text{ for } F_o<10$$
$$=10/F_o \text{ for } F_o\geq10$$
$$w_2=2{\cdot}5\sin^2\theta \text{ for } \sin^2\theta<0{\cdot}4$$
$$=1 \text{ for } \sin^2\theta\geq0{\cdot}4$$

(The nominal minimum value of F_o is 2·5).

This weighting scheme was reasonably effective in removing trends in the magnitudes of the residuals.