Crystal Structure of β-Copper Phthalocyanine

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The crystal structure of the β-polymorph of copper phthalocyanine has been determined from three-dimensional *X*-ray diffraction data. A total of 1691 structure amplitudes was used in the refinement which resulted in $R = 7{\cdot}3\%$ and standard deviation of bond lengths of 0·005 Å. The structure is isomorphous with those of the metal-free [1] and nickel [2] phthalocyanines.

THE crystal structure of the phthalocyanines has been known since the determination by Robertson and Woodward of the metal-free [1] and nickel [2] compounds, and the unit-cell dimensions [3] of a number of others, but no detailed work appears to have been done on copper phthalocyanine which is the derivative of greatest commercial importance. As it was desired to investigate more fully the relationship between the various polymorphs, a more accurate study of the structure of the

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[1] J. M. Robertson, *J. Chem. Soc.*, 1936, 1195.
[2] J. M. Robertson and I. Woodward, *J. Chem. Soc.*, 1937, 219.
[3] J. M. Robertson, *J. Chem. Soc.*, 1935, 615.

β-polymorph, which is the normal stable form at room temperature and pressure, was necessary. A summary of the results of this work, together with a preliminary description of the structures of some of the other polymorphs, was presented at the Seventh Congress of the International Union of Crystallography in Moscow.[4]

EXPERIMENTAL

Sublimation of a freshly prepared batch of copper phthalocyanine at ordinary pressure resulted in an abundant crop of acicular crystals elongated along *b*. A crystal of cross-section approximately 0·01 × 0·05 mm.² was used for obtaining Weissenberg photographs about *b* only (zero and four layers). The fragile acicular crystal shape prevented reasonable photographs being obtained about other axes. Nine films were exposed at each setting, in batches of three, and the intensities of the spots were estimated by comparison with a time-exposure calibrated film strip. Geometrical and polarisation corrections were applied and the relative scaling of the layers was carried out on the computer. The thin section of the crystal traversed by the *X*-ray beam rendered correction for absorption unnecessary, and no anomalies which could be attributed to extinction arose in the course of the work.

Crystal Data.—$CuC_{32}N_8H_{16}$, $M = 575{\cdot}67$, Monoclinic, $a = 19{\cdot}407$, $b = 4{\cdot}790$, $c = 14{\cdot}628$ Å, $\beta = 120°\ 56'$, $U = 1166$ Å³, $D_m = 1{\cdot}63$, $Z = 2$, $D_c = 1{\cdot}639$, $F(000) = 586$. Space group $P2_1/a$. Cu K_α-radiation ($\lambda = 1{\cdot}5418$ Å at 20°).

Refinement of the Structure.—The crystal structure was assumed to be similar to those found by Robertson and Woodward,[1,2] so commencing with the atomic co-ordinates of the metal-free phthalocyanine, successive cycles of structure-factors and least-squares calculations were carried out on our Pegasus computer, using programs written by Cruickshank and Pilling.[5] After five cycles using $F(h0l)$ terms only, the R factor over the 272 observed structure amplitudes was 9·5%. Six further cycles using all the 1691 observed $F(hkl)$ terms reduced R to 7·3%. During this process hydrogen atoms were included at calculated positions assuming C–H 1·08 Å, but these were not refined Anisotropic temperature factors were also calculated. A further 708 planes which gave either zero or unobserved intensities on the *X*-ray photographs all gave very small calculated structure amplitudes.

After the final refinement cycle, the shifts indicated for the positional and thermal parameters were all less than ¼ of the standard deviations which were 0·006 Å for x, y, and z, and 0·25 Å² for the diagonal B_{ij} values. The final atomic parameters are listed in Table 1, and the agreement between the observed and calculated structure amplitudes is shown in Table 2.

TABLE 1

Atomic parameters

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{13}
Cu	0	0	0	2·61	2·54	2·20	−0·11	0·06	1·19
C(1)	−0·0095	−0·4009	0·1451	2·49	2·61	1·94	−0·07	0·06	1·34
C(2)	0·0324	−0·4937	0·2563	2·77	2·63	2·25	0·34	0·43	1·49
C(3)	0·0135	−0·6928	0·3090	3·13	2·54	2·87	−0·06	0·23	1·67
C(4)	0·0668	−0·7296	0·4166	3·27	3·31	2·84	0·13	0·89	1·63
C(5)	0·1378	−0·5687	0·4706	3·20	2·85	2·12	0·45	0·66	1·03
C(6)	0·1557	−0·3747	0·4179	2·83	2·76	2·36	0·32	0·64	1·07
C(7)	0·1020	−0·3342	0·3084	2·33	2·09	2·11	0·18	0·21	1·20
C(8)	0·1021	−0·1547	0·2292	2·46	2·31	2·08	0·09	0·12	1·15
C(9)	0·1582	0·1968	0·1788	2·46	1·96	2·31	0·21	−0·07	1·31
C(10)	0·2216	0·3910	0·2023	2·41	2·16	2·25	−0·10	−0·20	1·36
C(11)	0·2954	0·4490	0·2919	2·95	2·96	2·12	−0·44	−0·34	1·11
C(12)	0·3424	0·6523	0·2852	2·64	3·85	2·35	−0·79	−0·82	1·10
C(13)	0·3187	0·7928	0·1884	3·01	3·06	2·65	−0·91	−0·77	1·71
C(14)	0·2445	0·7358	0·0971	2·95	3·42	2·86	−0·52	−0·25	1·78
C(15)	0·1982	0·5336	0·1064	2·26	2·63	2·08	−0·19	−0·05	1·14
C(16)	0·1196	0·4164	0·0282	2·08	2·10	2·16	−0·34	−0·12	1·01
N(1)	−0·0798	−0·5072	0·0724	2·32	2·77	1·99	−0·40	0·05	1·09
N(2)	0·0326	−0·1945	0·1316	2·24	1·99	1·89	−0·07	0·04	1·14
N(3)	0·1598	0·0294	0·2521	2·48	2·44	1·92	−0·34	−0·12	1·20
N(4)	0·0987	0·2144	0·0738	1·88	2·31	1·69	−0·14	0·02	0·66
H(3)	−0·0409	−0·8146	0·2673						
H(4)	0·0542	−0·8832	0·4600						
H(5)	0·1784	−0·6002	0·5549						
H(6)	0·2103	−0·2538	0·4597	Mean isotropic B 7·94					
H(11)	0·3156	0·3364	0·3654						
H(12)	0·3988	0·7064	0·3555						
H(13)	0·3582	0·9456	0·1850						
H(14)	0·2247	0·8450	0·2306						

The values of B in this Table are defined in the expression

$$\exp -\tfrac{1}{4}(h^2a^{*2}B_{11} + 2hka^*b^*B_{12} + \cdots)$$

used in the structure amplitude calculations.

Mean estimated standard deviations for C and N atoms are $\sigma(x)$, $\sigma(y)$, $\sigma(z) = 0{\cdot}006$ A; $\sigma(B_{ij}) = 0{\cdot}25$ Å².

DISCUSSION

Description of the Structure.—The numbering of the atoms used in this paper, and the bond lengths and inter bond angles found are given in Figure 1. The equation

[4] C. J. Brown, *Acta Cryst.*, 1966, **21**, *A*137.
[5] D. W. J. Cruickshank and D. E. Pilling in Computing Methods and the Phase Problem in *X*-Ray Crystal Analysis, Pergamon Press, Oxford, 1961, p. 32.

TABLE 2

Observed and calculated structure amplitudes

h	k	l	$\|F_o\|$	F_c
0	0	1	129.5	122.8
0	0	2	29.2	−27.3
0	0	3	12.8	15.7
0	0	4	60.5	60.4
0	0	5	4.8	−1.9
0	0	6	5.7	−5.6
0	0	7	55.1	55.0
0	0	9	21.6	20.8
0	0	10	35.1	39.4
0	0	11	20.2	18.3
0	0	12	23.3	20.9
0	0	13	19.6	17.4
0	0	14	12.8	12.9
0	0	15	12.8	12.4
0	0	16	11.7	11.8
2	0	−16	12.8	13.0
2	0	−15	16.5	17.1
2	0	−14	24.0	24.5
2	0	−13	52.3	55.3
2	0	−12	20.2	19.9
2	0	−11	11.1	−11.2
2	0	−10	18.1	19.0
2	0	−9	34.2	36.5
2	0	−8	40.1	39.2
2	0	−7	51.0	52.1
2	0	−6	39.8	44.1
2	0	−5	73.4	76.3
2	0	−4	5.4	−4.8
2	0	−3	106.2	111.1
2	0	−2	67.9	70.2
2	0	−1	148.0	144.0
2	0	0	59.2	54.9
2	0	1	33.5	−32.5
2	0	2	56.6	53.6
2	0	3	33.5	36.6
2	0	4	48.5	51.6
2	0	5	12.2	−15.2
2	0	6	32.7	33.6
2	0	7	52.3	53.1
2	0	8	38.8	42.4
2	0	9	9.1	10.3
2	0	10	23.3	−21.9
2	0	12	25.2	25.6
2	0	13	10.5	8.6
2	0	14	19.6	17.8
2	0	15	18.1	19.1
4	0	−16	12.8	11.9
4	0	−15	20.2	20.5
4	0	−14	22.2	22.0
4	0	−11	29.6	30.1
4	0	−10	41.2	41.9
4	0	−9	38.1	39.5
4	0	−8	87.3	89.7
4	0	−7	79.0	75.1
4	0	−6	31.8	37.3
4	0	−5	9.1	11.3
4	0	−3	25.7	−20.8
4	0	−2	33.8	−31.3
4	0	−1	116.5	119.6
4	0	0	5.2	6.7
4	0	1	32.7	34.5
4	0	2	49.9	52.7
4	0	3	44.9	47.6
4	0	4	22.2	−17.1
4	0	5	17.4	19.2
4	0	6	66.0	58.6
4	0	7	38.5	34.6
4	0	8	25.2	21.5
4	0	9	33.1	33.8
4	0	10	18.1	16.6
4	0	11	7.0	6.4
4	0	12	10.5	10.5
4	0	13	14.8	15.3
4	0	14	10.5	10.0
6	0	−17	13.9	12.0
6	0	−16	20.9	21.5
6	0	−15	7.4	8.0
6	0	−14	11.7	−11.4
6	0	−12	28.7	28.7
6	0	−11	30.0	30.7
6	0	−10	29.2	30.5
6	0	−9	13.9	15.5
6	0	−8	52.3	49.2
6	0	−7	61.0	59.1
6	0	−6	64.9	66.0
6	0	−5	22.7	24.5
6	0	−4	44.0	46.1
6	0	−3	11.7	14.8
6	0	−1	8.5	9.0
6	0	0	70.6	75.3
6	0	1	10.5	12.2
6	0	2	23.9	−24.2
6	0	3	8.3	8.4
6	0	4	44.9	49.8
6	0	5	49.4	53.4
6	0	6	53.3	57.3
6	0	7	78.8	78.4
6	0	8	37.0	35.4
6	0	9	9.1	8.1
6	0	11	15.7	15.4
8	0	−18	7.4	7.7
8	0	−17	12.8	12.8
8	0	−13	46.8	50.9
8	0	−12	13.9	15.0
8	0	−11	10.5	−10.3
8	0	−10	10.5	10.9
8	0	−9	67.3	69.6
8	0	−8	63.6	67.4
8	0	−7	18.1	22.3
8	0	−6	38.8	41.2
8	0	−5	23.3	28.4
8	0	−4	35.5	39.0
8	0	−3	50.1	51.9
8	0	−2	35.5	35.8
8	0	−1	52.3	59.3
8	0	0	42.2	45.4
8	0	1	33.1	36.3
8	0	2	49.0	51.8
8	0	3	35.9	34.1
8	0	4	43.5	44.4
8	0	5	14.8	14.2
8	0	6	49.6	46.4
8	0	7	21.8	24.4
8	0	8	9.1	10.1
8	0	9	7.0	6.5
8	0	10	10.5	9.8
8	0	11	12.8	12.6
10	0	−18	11.7	13.5
10	0	−17	15.7	16.3
10	0	−16	27.2	27.9
10	0	−14	26.1	26.0
10	0	−13	40.5	39.4
10	0	−12	7.4	7.5
10	0	−11	31.4	29.8
10	0	−10	35.5	34.0
10	0	−9	38.5	38.1
10	0	−8	9.1	−9.5
10	0	−7	33.5	34.8
10	0	−6	53.6	56.4
10	0	−5	20.2	24.6
10	0	−4	45.3	49.0
10	0	−3	58.4	58.6
10	0	−2	53.6	54.9
10	0	−1	87.1	90.0
10	0	0	41.2	41.8
10	0	1	25.2	28.7
10	0	3	33.8	37.7
10	0	6	23.3	20.6
10	0	8	10.5	9.9
10	0	9	13.9	13.2
10	0	10	17.4	16.1
12	0	−18	10.5	11.6
12	0	−17	13.9	14.4
12	0	−16	18.1	18.8
12	0	−15	9.1	8.4
12	0	−14	40.5	42.3
12	0	−13	20.2	21.1
12	0	−12	28.7	31.5
12	0	−11	27.2	30.3
12	0	−10	31.8	34.9
12	0	−9	14.8	14.3
12	0	−8	6.1	−5.2
12	0	−6	5.9	−5.6
12	0	−5	34.8	36.3
12	0	−4	71.8	77.4
12	0	−3	16.5	20.8
12	0	−2	26.1	28.2
12	0	−1	37.0	37.0
12	0	0	41.2	42.1
12	0	1	21.6	20.2
12	0	2	9.1	16.6
12	0	3	23.3	21.4
12	0	4	20.2	−21.7
12	0	5	11.7	11.1
12	0	6	13.9	10.5
12	0	7	14.8	12.8
14	0	−18	10.5	11.7
14	0	−17	24.6	28.8
14	0	−14	26.1	27.9
14	0	−13	23.3	23.4
14	0	−12	11.7	13.0
14	0	−11	11.7	10.9
14	0	−10	25.2	24.5
14	0	−7	30.0	32.3
14	0	−6	33.8	33.8
14	0	−5	32.2	35.6
14	0	−4	14.8	16.2
14	0	−2	24.0	27.5
14	0	−1	25.2	24.4
14	0	0	15.7	14.7
14	0	1	16.5	−14.0
14	0	2	26.1	28.2
14	0	3	26.1	26.5
14	0	4	17.4	17.3
14	0	5	14.8	13.1
14	0	6	9.1	9.7
14	0	7	13.9	12.7
16	0	−18	9.1	8.6
16	0	−16	13.9	−12.7
16	0	−15	7.4	−8.0
16	0	−13	20.2	20.1
16	0	−12	20.2	23.2
16	0	−11	28.7	31.0
16	0	−10	28.7	28.3
16	0	−9	28.7	27.7
16	0	−8	14.8	14.9
16	0	−7	19.6	23.4
16	0	−6	27.2	27.7
16	0	−5	15.7	16.0
16	0	−4	9.1	−6.7
16	0	−3	23.3	22.7
16	0	−2	30.9	30.6
16	0	1	23.3	25.2
16	0	2	35.1	39.5
16	0	3	14.8	11.2
16	0	4	15.7	11.8
16	0	5	17.4	15.3
16	0	6	21.6	18.2
18	0	−17	7.4	−8.1
18	0	−15	15.7	15.7
18	0	−14	18.1	20.6
18	0	−13	9.1	8.8
18	0	−12	22.2	21.8
18	0	−11	26.1	25.7
18	0	−10	20.2	21.0
18	0	−9	11.7	10.2
18	0	−8	16.5	15.3
18	0	−7	26.1	25.2
18	0	−6	18.1	18.8
18	0	−5	40.5	39.4
18	0	−4	40.5	38.6
18	0	−3	18.1	17.0
18	0	−2	16.5	−14.2
18	0	0	13.9	11.9
18	0	1	17.4	19.0
18	0	2	14.8	13.6
18	0	3	13.9	13.1
18	0	4	7.4	7.9
20	0	−17	25.2	25.4
20	0	−15	13.9	−13.4
20	0	−14	11.7	10.8
20	0	−13	29.6	33.0
20	0	−12	21.6	20.3
20	0	−11	7.4	8.5
20	0	−10	22.2	24.4
20	0	−8	15.7	14.3
20	0	−7	25.2	25.5
20	0	−6	22.2	23.3
20	0	−3	10.5	10.8
20	0	−1	11.7	11.2
20	0	0	16.5	16.8
20	0	2	7.4	6.7
22	0	−15	20.2	19.5
22	0	−14	22.2	25.7
22	0	−13	19.6	22.4
22	0	−11	9.1	8.9
22	0	−9	10.5	−10.0
22	0	−8	7.4	−7.7
22	0	−6	18.9	18.5
22	0	−5	20.9	19.8
22	0	−4	17.4	19.2
22	0	−3	7.4	8.4
22	0	−2	10.5	9.5
22	0	−1	9.1	10.1
24	0	−14	10.5	11.4
24	0	−13	7.4	−8.8
24	0	−11	18.1	19.9
24	0	−9	7.4	−7.1
24	0	−8	7.4	6.2
24	0	−7	20.3	19.5
24	0	−6	9.4	7.7
24	0	−5	9.1	8.2
0	1	1	32.2	30.6
0	1	2	35.1	30.9
0	1	3	9.1	−7.3
0	1	4	4.8	−7.0
0	1	5	31.8	−27.7
0	1	6	35.5	−30.2
0	1	7	28.1	−24.1
0	1	9	8.0	10.8
0	1	10	11.3	14.9
0	1	11	7.2	6.2
0	1	13	6.7	−6.9
1	1	−16	17.4	15.2
1	1	−14	14.8	15.8
1	1	−13	15.7	15.6
1	1	−10	18.9	18.7
1	1	−9	39.2	40.6
1	1	−8	33.5	34.4
1	1	−7	17.4	19.6
1	1	−6	64.4	62.6
1	1	−5	52.2	50.2
1	1	−4	41.4	40.1
1	1	−3	30.9	30.4
1	1	−2	42.2	42.5
1	1	−1	11.1	9.3
1	1	0	14.8	−13.0
1	1	1	69.4	63.4
1	1	2	4.1	−5.3
1	1	3	61.6	59.6
1	1	4	59.4	56.9
1	1	5	47.3	47.7
1	1	6	8.7	12.2
1	1	7	34.8	39.9
1	1	8	40.1	41.2
1	1	9	16.5	17.7
1	1	10	25.7	28.1
1	1	11	39.8	44.0
1	1	12	17.4	17.9
1	1	13	8.0	8.7
1	1	14	9.8	11.6
1	1	15	13.9	12.3
2	1	−16	4.4	−4.4
2	1	−14	6.7	5.4
2	1	−13	20.2	17.7
2	1	−11	20.2	−20.9
2	1	−10	8.5	−7.5
2	1	−9	6.5	−5.8
2	1	−8	18.9	15.5
2	1	−7	9.2	7.6
2	1	−6	4.8	−5.1
2	1	−5	9.2	15.1
2	1	−4	25.2	19.7
2	1	−3	49.9	47.3
2	1	−2	49.6	−45.3
2	1	−1	22.7	−19.7
2	1	0	55.3	−49.0
2	1	1	24.0	−24.6
2	1	2	16.5	13.2
2	1	3	12.2	−10.1
2	1	4	29.6	−28.1
2	1	5	27.7	−33.3
2	1	7	13.7	10.7
2	1	8	20.9	20.3
2	1	9	34.8	38.4
2	1	11	18.1	−15.6
2	1	12	11.1	−9.5
2	1	15	8.0	−6.3
3	1	−17	10.7	10.4
3	1	−15	16.5	16.1
3	1	−14	10.5	9.5
3	1	−12	7.2	5.8
3	1	−11	17.4	16.4
3	1	−10	36.2	35.3
3	1	−9	27.2	30.7
3	1	−6	65.3	62.0
3	1	−5	64.9	62.5
3	1	−4	19.6	20.4
3	1	−3	34.8	34.1
3	1	−2	190.9	189.8
3	1	−1	71.8	76.9
3	1	0	31.4	32.3
3	1	1	13.7	17.2
3	1	2	72.5	72.8
3	1	3	8.9	9.9
3	1	5	45.9	48.5
3	1	6	35.9	35.9
3	1	7	28.7	28.1
3	1	8	30.9	29.1
3	1	9	48.8	47.9
3	1	10	36.2	35.2
3	1	11	18.1	16.5
3	1	12	10.5	10.8
3	1	13	15.7	13.3
3	1	14	5.2	5.2
4	1	−13	7.4	−9.3
4	1	−12	20.2	−19.7
4	1	−11	16.5	16.9
4	1	−10	14.8	13.4
4	1	−9	27.2	−22.9
4	1	−8	8.9	8.5
4	1	−7	6.1	7.4
4	1	−6	45.3	38.1
4	1	−5	15.9	14.3
4	1	−4	25.7	25.1
4	1	−3	113.9	−116.3
4	1	−2	152.6	−147.3
4	1	−1	33.7	39.9
4	1	0	20.2	19.6
4	1	1	12.8	11.8
4	1	2	12.8	11.6
4	1	3	23.3	25.5
4	1	4	13.9	12.7
4	1	6	19.6	21.0
4	1	7	10.5	−8.7
4	1	9	11.5	13.0
4	1	10	7.4	−8.5
4	1	14	9.6	−6.1
5	1	−17	9.8	10.5
5	1	−16	9.4	9.5
5	1	−15	25.2	26.1
5	1	−14	17.4	18.8
5	1	−13	11.5	14.4
5	1	−11	41.2	37.0
5	1	−10	29.6	28.4
5	1	−9	6.1	8.3
5	1	−8	19.6	21.3
5	1	−7	66.6	65.0
5	1	−6	32.7	30.1
5	1	−5	16.3	20.0
5	1	−4	74.0	80.2
5	1	−3	155.3	163.0
5	1	−2	58.6	67.1
5	1	−1	15.7	16.2
5	1	0	41.4	44.1
5	1	1	50.1	54.2
5	1	2	59.6	59.3
5	1	3	19.6	22.2
5	1	4	39.4	41.4
5	1	5	18.9	20.4
5	1	6	31.8	37.8
5	1	7	15.4	17.3
5	1	8	8.9	9.6
5	1	9	7.2	9.3
5	1	10	14.6	14.4
5	1	11	6.7	7.8
5	1	12	14.2	14.2
5	1	13	16.5	16.6
6	1	−17	4.4	−4.2
6	1	−16	10.0	−10.5
6	1	−15	8.1	−9.4
6	1	−14	16.5	17.6
6	1	−13	11.5	12.8
6	1	−12	7.2	−8.7
6	1	−11	22.2	19.2
6	1	−10	8.1	−9.4
6	1	−7	15.9	13.9
6	1	−6	7.4	−7.9
6	1	−5	47.9	−44.7
6	1	−4	18.1	15.6
6	1	−3	38.8	−37.5
6	1	−2	20.9	22.2
6	1	−1	30.9	31.7
6	1	0	6.7	10.2
6	1	1	27.7	28.2
6	1	2	29.6	27.7
6	1	3	26.6	22.3
6	1	4	20.9	−19.2
6	1	5	10.0	−12.1
6	1	8	20.2	20.9
6	1	10	7.0	−7.8
6	1	13	8.5	−7.2
7	1	−18	10.4	10.9
7	1	−17	12.8	12.7
7	1	−16	26.6	26.8
7	1	−15	13.3	13.2
7	1	−14	16.5	13.1
7	1	−13	16.3	13.7
7	1	−12	32.7	29.7
7	1	−11	43.6	43.3
7	1	−10	41.8	39.4
7	1	−9	48.3	47.5
7	1	−8	40.1	37.5
7	1	−7	32.5	32.0
7	1	−6	15.0	15.0
7	1	−5	15.7	19.4
7	1	−4	19.6	18.8
7	1	−3	36.2	38.5
7	1	−2	29.2	30.7
7	1	−1	62.1	63.2
7	1	0	40.3	42.8
7	1	1	64.4	66.4
7	1	2	12.9	14.9
7	1	3	27.6	27.8
7	1	4	33.5	34.5
7	1	5	33.1	33.7
7	1	7	7.2	−7.5
7	1	8	11.5	10.1
7	1	9	8.5	9.0
7	1	10	11.1	11.8
7	1	11	13.7	13.6
7	1	12	13.9	11.3
8	1	−16	6.7	5.9
8	1	−15	8.3	8.2
8	1	−14	12.8	10.9
8	1	−13	12.6	−7.9
8	1	−12	7.2	7.7
8	1	−10	16.3	−17.2
8	1	−9	6.1	8.1
8	1	−8	8.9	7.8
8	1	−7	18.9	19.0
8	1	−6	15.9	−13.7
8	1	−5	15.0	14.4
8	1	−4	22.9	−27.8
8	1	−3	24.6	−23.8
8	1	−2	46.4	46.4
8	1	−1	10.5	9.4
8	1	0	13.7	−13.2
8	1	1	16.5	15.2
8	1	2	25.2	28.2
8	1	4	18.1	−19.0
8	1	6	14.6	−17.2
8	1	7	14.6	−16.2
8	1	11	8.5	−7.4
9	1	−18	12.4	13.1
9	1	−17	14.1	13.4
9	1	−15	8.1	8.7
9	1	−14	28.1	27.1
9	1	−13	16.3	13.9
9	1	−12	36.2	34.2
9	1	−11	34.8	35.4
9	1	−10	41.8	43.7
9	1	−9	27.2	28.4
9	1	−8	32.2	31.4
9	1	−7	12.2	13.8
9	1	−4	24.4	24.1
9	1	−3	29.6	31.9
9	1	−2	55.9	59.4
9	1	−1	16.5	20.1
9	1	0	8.5	9.5
9	1	1	18.1	19.5
9	1	2	33.5	34.3
9	1	3	33.1	31.9
9	1	4	18.9	18.5
9	1	5	39.8	40.4
9	1	6	16.3	18.3
9	1	7	16.5	16.4
9	1	8	8.5	7.1
9	1	9	15.2	13.0
9	1	10	5.5	5.1
10	1	−17	8.3	8.0
10	1	−16	9.8	10.9
10	1	−15	11.7	12.2
10	1	−13	18.1	−16.5
10	1	−12	8.9	9.9
10	1	−11	7.0	7.5
10	1	−9	20.2	−16.9
10	1	−7	5.7	−4.8
10	1	−6	20.2	−20.0
10	1	−5	12.9	13.0
10	1	−4	20.9	21.7
10	1	−3	8.5	7.9
10	1	−0	14.2	13.7
10	1	1	5.9	5.8
10	1	2	8.1	−8.9
10	1	3	24.6	−24.6
10	1	4	25.7	−27.1
10	1	7	8.5	9.9
11	1	−18	8.9	7.8
11	1	−17	5.2	4.6
11	1	−16	7.6	6.0
11	1	−15	30.0	29.5
11	1	−12	7.2	7.1
11	1	−11	22.2	25.1
11	1	−10	29.6	30.9
11	1	−9	28.7	28.9
11	1	−8	37.4	36.9
11	1	−7	18.9	20.7
11	1	−6	24.6	24.6
11	1	−5	25.2	27.4
11	1	−4	28.1	30.0
11	1	−3	35.9	40.4
11	1	−2	18.9	20.1
11	1	−1	6.1	−5.7
11	1	0	24.6	27.6
11	1	1	29.6	32.3
11	1	2	24.6	28.3
11	1	3	8.9	9.7
11	1	4	44.8	48.4
11	1	5	32.7	35.3
11	1	6	15.5	13.1
11	1	7	8.5	7.8
11	1	8	14.6	12.9
11	1	9	13.5	12.0
12	1	−18	9.8	8.2
12	1	−17	14.1	14.0
12	1	−15	8.1	−11.4
12	1	−12	17.8	−16.7
12	1	−11	22.7	−20.5
12	1	−9	11.5	11.1
12	1	−8	14.2	10.9
12	1	−7	13.7	10.8
12	1	−4	7.4	7.0
12	1	−2	10.5	−11.4
12	1	−1	15.5	−14.7
12	1	0	8.1	8.2
12	1	2	16.3	−15.2
12	1	3	7.2	−5.7
12	1	5	7.4	7.9
13	1	−18	12.6	13.8
13	1	−17	10.4	8.0
13	1	−16	15.2	15.5
13	1	−15	10.5	9.8
13	1	−14	11.7	−11.9
13	1	−12	20.2	20.7
13	1	−11	39.8	37.4
13	1	−10	15.5	14.1
13	1	−9	33.1	30.9
13	1	−8	40.5	38.5
13	1	−7	34.8	33.8
13	1	−6	20.2	22.6
13	1	−5	31.8	31.9
13	1	−4	34.8	36.3
13	1	−3	38.8	38.7
13	1	−2	25.7	27.0
13	1	−1	37.7	40.3
13	1	0	22.2	22.7
13	1	1	11.5	10.7
13	1	3	20.2	19.2
13	1	4	28.1	28.3
13	1	5	8.3	7.3
13	1	6	9.8	11.5
13	1	7	12.2	12.0
13	1	8	17.4	18.0
14	1	−17	5.2	4.9
14	1	−16	10.7	−10.2
14	1	−15	8.3	−9.1
14	1	−14	7.0	−5.8
14	1	−10	11.5	9.5
14	1	−6	13.3	−13.3
14	1	−4	13.3	−12.1
14	1	−2	18.9	18.0
14	1	−1	15.5	15.6
14	1	0	7.2	−7.1
14	1	1	22.7	−23.0
14	1	2	7.2	8.5
14	1	4	6.7	5.7
14	1	5	8.3	8.3
15	1	−18	8.9	8.5
15	1	−17	12.2	10.9
15	1	−16	22.2	20.2
15	1	−15	10.5	10.8
15	1	−14	22.2	22.8
15	1	−13	18.1	17.0
15	1	−12	14.6	13.2
15	1	−10	25.7	25.7
15	1	−9	28.1	27.3
15	1	−8	14.1	13.4
15	1	−7	22.2	22.9
15	1	−6	30.9	30.5
15	1	−5	26.6	29.4
15	1	−4	34.8	37.7
15	1	−3	41.2	42.6
15	1	−2	25.7	25.5
15	1	−1	7.4	7.8
15	1	1	8.9	7.9
15	1	3	15.0	14.1
15	1	4	7.6	7.8
15	1	5	11.3	11.4
15	1	6	9.2	7.9
16	1	−18	5.5	5.2
16	1	−15	11.1	12.5
16	1	−13	17.4	−18.2
16	1	−12	11.7	−10.9
16	1	−11	20.2	18.7
16	1	−10	25.7	23.3
16	1	−8	16.3	14.1
16	1	−7	16.3	−12.8
16	1	−6	18.1	−15.3
16	1	−5	14.6	12.4
16	1	−2	16.3	−14.4
16	1	−1	7.2	6.7
16	1	0	7.4	−5.1
16	1	2	16.5	16.5
17	1	−18	9.2	8.6
17	1	−17	13.9	14.4
17	1	−16	21.6	22.4
17	1	−15	24.0	24.8
17	1	−12	20.2	21.7
17	1	−11	18.1	18.8

TABLE 2 (*Continued*)

h	k	l	$\|F_o\|$	F_c
17	1	-10	11.5	12.3
17	1	-9	7.2	7.8
17	1	-8	28.1	27.7
17	1	-7	16.3	14.6
17	1	-6	8.9	8.3
17	1	-5	8.9	7.5
17	1	-4	16.3	14.5
17	1	-3	16.3	14.5
17	1	-2	18.1	18.5
17	1	-1	11.7	11.0
17	1	0	11.1	11.1
17	1	1	10.5	8.3
17	1	2	13.9	13.9
17	1	3	5.5	5.7
17	1	4	5.7	6.9
17	1	5	11.3	8.7
18	1	-12	15.5	15.0
18	1	-11	23.3	23.4
18	1	-9	11.7	-10.0
18	1	-7	16.3	-17.2
18	1	-5	11.7	-10.7
18	1	-3	8.9	6.6
18	1	-2	15.5	16.1
18	1	-1	10.5	10.1
18	1	0	8.0	-8.9
19	1	-17	9.6	9.3
19	1	-16	7.4	7.8
19	1	-15	9.4	10.1
19	1	-14	7.6	7.3
19	1	-13	17.4	19.0
19	1	-12	16.5	17.4
19	1	-11	18.9	20.2
19	1	-9	11.7	10.1
19	1	-8	16.5	14.9
19	1	-6	12.8	-11.2
19	1	-4	24.0	26.9
19	1	-3	15.5	13.9
19	1	-1	17.4	17.4
19	1	0	20.2	20.8
19	1	1	9.2	7.9
20	1	-17	8.0	7.7
20	1	-16	5.2	6.3
20	1	-14	5.7	-6.6
20	1	-10	13.3	-13.8
20	1	-2	9.4	9.5
20	1	-1	8.9	-8.0
20	1	0	8.5	-8.7
21	1	-16	12.6	14.0
21	1	-15	5.7	5.5
21	1	-14	10.4	9.8
21	1	-13	12.6	13.9
21	1	-12	9.4	10.2
21	1	-10	8.0	9.4
21	1	-9	25.2	26.6
21	1	-8	8.0	5.9
21	1	-6	17.4	15.6
21	1	-5	19.6	19.2
21	1	-2	14.1	13.6
21	1	-1	12.8	12.1
21	1	0	8.5	7.3
22	1	-14	5.2	-5.7
22	1	-13	5.9	-7.6
22	1	-11	5.5	4.2
22	1	-10	7.0	6.6
22	1	-9	7.0	6.9
22	1	-8	7.4	-7.3
22	1	-7	10.0	-11.0
22	1	-6	5.7	-6.2
22	1	-5	6.7	8.4
22	1	-4	10.4	11.5
22	1	-3	5.7	5.4
23	1	-14	8.5	6.7
23	1	-13	5.2	4.6
23	1	-12	14.6	14.8
23	1	-11	8.5	8.5
23	1	-10	16.5	15.8
23	1	-9	18.1	16.7
23	1	-8	18.1	17.8
23	1	-7	8.5	10.2
23	1	-6	5.9	7.1
23	1	-5	5.2	6.7
23	1	-4	4.1	3.3
23	1	-3	8.0	8.1
24	1	-11	5.0	4.9
24	1	-7	6.1	6.1
0	2	0	29.6	28.7
0	2	1	33.5	41.0
0	2	2	45.3	42.0
0	2	3	54.9	50.1
0	2	4	80.3	75.5
0	2	5	23.3	21.9
0	2	6	14.4	14.7
0	2	7	26.1	24.9
0	2	8	51.2	49.4
0	2	9	18.9	17.4
0	2	11	24.6	22.0
0	2	12	16.5	14.1
0	2	13	13.5	11.4
0	2	15	10.9	10.8
1	2	-12	13.5	-11.4
1	2	-11	15.7	-12.8
1	2	-10	10.9	-9.8
1	2	-8	7.4	8.8
1	2	-7	17.4	-14.1
1	2	-6	18.1	14.4
1	2	-5	7.0	7.1
1	2	-4	17.4	14.0
1	2	-3	12.8	-7.5
1	2	-2	4.1	4.2
1	2	-1	8.9	8.8
1	2	1	6.7	5.3
1	2	2	3.3	-3.3
1	2	3	24.0	-20.3
1	2	4	26.1	21.9
1	2	5	31.4	29.1
1	2	6	21.6	17.1
1	2	7	13.7	-9.8
1	2	8	6.5	-5.4
1	2	9	17.4	15.3
1	2	11	7.0	7.9
1	2	12	10.0	-10.5
2	2	-16	9.1	7.6
2	2	-15	5.9	5.4
2	2	-13	22.7	22.5
2	2	-12	38.5	39.0
2	2	-11	42.7	42.7
2	2	-10	18.9	16.0
2	2	-9	45.9	45.1
2	2	-8	27.2	23.9
2	2	-6	30.5	26.8
2	2	-5	54.0	54.3
2	2	-4	30.0	27.8
2	2	-3	6.5	-7.0
2	2	-2	37.0	34.8
2	2	-1	48.3	49.7
2	2	0	24.0	26.7
2	2	1	65.8	63.3
2	2	2	55.9	54.4
2	2	3	57.3	56.2
2	2	4	52.3	53.7
2	2	5	37.4	35.5
2	2	6	28.7	28.7
2	2	7	16.5	16.4
2	2	8	23.3	21.8
2	2	9	8.5	-10.1
2	2	10	7.0	8.3
2	2	11	18.1	16.9
2	2	12	7.6	8.4
2	2	14	12.2	11.5
3	2	-15	8.0	5.5
3	2	-13	6.5	-7.0
3	2	-10	18.9	-16.5
3	2	-9	32.2	32.8
3	2	-8	18.5	19.6
3	2	-7	13.5	10.7
3	2	-5	16.6	15.7
3	2	-3	17.9	-21.6
3	2	-2	29.6	26.1
3	2	-1	10.7	-9.7
3	2	1	24.6	22.7
3	2	2	9.8	10.0
3	2	6	6.1	-5.5
3	2	8	22.2	19.5
3	2	9	8.5	-7.9
3	2	10	8.5	-6.3
3	2	11	10.2	-10.3
4	2	-16	9.4	8.3
4	2	-15	10.9	10.2
4	2	-14	22.2	24.3
4	2	-13	15.0	13.8
4	2	-12	22.4	26.6
4	2	-11	22.4	26.8
4	2	-10	21.6	19.3
4	2	-9	27.7	27.3
4	2	-8	29.2	27.8
4	2	-7	45.7	41.5
4	2	-6	37.4	32.2
4	2	-5	42.9	37.3
4	2	-4	33.1	32.3
4	2	-2	10.4	-10.2
4	2	0	35.1	32.9
4	2	1	66.2	66.0
4	2	2	37.0	37.3
4	2	3	60.8	59.0
4	2	4	33.1	36.1
4	2	5	44.9	44.3
4	2	6	24.0	23.3
4	2	7	14.8	12.4
4	2	10	16.5	16.6
4	2	11	13.9	12.5
4	2	12	9.2	8.3
4	2	13	10.7	9.4
5	2	-15	5.5	6.8
5	2	-14	15.9	12.8
5	2	-12	8.5	6.4
5	2	-11	12.0	-9.4
5	2	-10	11.3	10.1
5	2	-9	19.6	16.7
5	2	-7	18.9	15.0
5	2	-5	13.9	13.7
5	2	-4	34.6	-31.6
5	2	-2	26.1	-28.5
5	2	-1	4.6	5.6
5	2	0	39.8	31.9
5	2	1	11.7	-8.5
5	2	2	37.4	-34.6
5	2	3	11.7	-8.8
5	2	4	5.5	6.5
5	2	6	8.0	-8.3
5	2	7	17.4	16.8
5	2	8	8.5	-8.4
5	2	9	17.4	-15.4
5	2	13	7.8	-7.3
6	2	-17	6.5	5.9
6	2	-16	5.9	5.7
6	2	-14	8.0	10.2
6	2	-13	27.2	24.5
6	2	-12	41.4	36.7
6	2	-11	7.0	6.7
6	2	-10	8.0	8.9
6	2	-9	31.4	27.3
6	2	-8	41.2	35.9
6	2	-7	24.0	23.5
6	2	-6	15.7	14.9
6	2	-5	22.7	23.0
6	2	-4	42.2	38.4
6	2	-3	24.6	25.4
6	2	-2	56.8	54.9
6	2	-1	31.4	29.8
6	2	0	53.8	49.6
6	2	1	25.7	23.7
6	2	2	20.2	21.9
6	2	3	39.2	39.1
6	2	4	27.2	29.1
6	2	5	11.3	13.6
6	2	6	8.5	-9.0
6	2	7	24.6	25.0
6	2	8	35.1	38.1
6	2	9	26.1	25.6
6	2	10	13.5	13.2
6	2	11	14.6	12.9
6	2	12	12.4	10.4
7	2	-16	13.3	14.3
7	2	-15	9.6	8.1
7	2	-13	8.5	8.3
7	2	-11	22.2	-20.2
7	2	-10	11.3	11.9
7	2	-9	7.6	-6.5
7	2	-8	5.7	-6.3
7	2	-6	43.5	38.6
7	2	-5	41.2	-35.3
7	2	-4	70.8	-66.5
7	2	-2	10.5	12.1
7	2	-1	11.1	8.8
7	2	2	15.2	-12.8
7	2	4	8.0	8.4
7	2	5	10.4	-11.0
7	2	7	8.5	10.5
7	2	8	6.7	-5.8
7	2	11	7.2	7.5
8	2	-17	17.4	15.7
8	2	-16	13.9	13.1
8	2	-15	23.3	22.8
8	2	-14	15.9	15.2
8	2	-13	7.0	11.2
8	2	-12	12.0	9.3
8	2	-10	10.4	-6.6
8	2	-9	16.5	14.3
8	2	-8	32.7	20.6
8	2	-7	21.6	22.6
8	2	-6	52.3	52.4
8	2	-5	124.8	122.0
8	2	-4	67.9	66.3
8	2	-2	29.2	29.8
8	2	-1	35.5	35.8
8	2	0	20.3	20.6
8	2	1	8.7	-6.3
8	2	2	37.0	37.6
8	2	3	21.6	22.3
8	2	4	8.0	10.2
8	2	5	15.5	14.0
8	2	6	24.6	26.7
8	2	7	27.2	28.5
8	2	8	17.4	17.1
8	2	9	13.2	10.7
8	2	10	16.1	16.3
8	2	11	15.4	13.7
9	2	-17	6.1	-5.5
9	2	-15	12.0	11.0
9	2	-12	17.4	-17.3
9	2	-11	10.9	-11.1
9	2	-10	18.1	-15.5
9	2	-9	6.5	7.5
9	2	-8	19.6	16.4
9	2	-6	21.6	-19.1
9	2	-5	37.4	-34.7
9	2	-4	18.1	16.0
9	2	-3	6.5	6.3
9	2	-2	14.2	14.9
9	2	0	6.8	6.2
9	2	1	18.1	16.3
9	2	2	15.0	14.4
9	2	3	6.7	-6.8
9	2	4	22.2	-23.0
9	2	6	24.6	24.8
10	2	-17	15.2	13.4
10	2	-15	12.0	10.7
10	2	-14	14.4	14.7
10	2	-13	12.2	10.1
10	2	-12	11.1	9.7
10	2	-11	15.5	14.7
10	2	-10	29.0	27.9
10	2	-9	25.2	23.7
10	2	-8	27.7	26.7
10	2	-7	46.2	42.4
10	2	-6	50.1	45.8
10	2	-5	48.3	46.2
10	2	-3	30.5	32.2
10	2	-2	30.5	31.4
10	2	-1	35.5	33.0
10	2	0	27.2	28.1
10	2	1	39.4	39.0
10	2	2	29.0	30.4
10	2	6	8.3	8.0
10	2	7	18.1	18.6
10	2	8	13.1	12.4
10	2	9	12.4	11.5
10	2	10	15.7	17.2
11	2	-17	8.5	7.8
11	2	-12	7.0	-4.7
11	2	-7	33.5	30.2
11	2	-1	19.6	19.4
11	2	0	35.5	32.6
11	2	1	6.7	-6.8
11	2	2	22.2	-20.6
11	2	3	8.5	-8.3
11	2	4	8.5	6.6
11	2	5	8.3	7.6
12	2	-16	13.1	12.7
12	2	-15	21.6	20.5
12	2	-14	20.2	17.8
12	2	-13	38.8	34.2
12	2	-12	42.7	40.2
12	2	-9	14.6	13.8
12	2	-8	35.5	36.4
12	2	-7	14.4	13.8
12	2	-6	33.8	32.3
12	2	-5	27.7	25.8
12	2	-4	7.6	7.1
12	2	-3	27.7	28.5
12	2	-2	32.2	30.9
12	2	-1	20.2	18.7
12	2	1	22.2	22.3
12	2	2	18.9	18.3
12	2	3	11.1	10.7
12	2	4	19.6	19.4
12	2	5	15.9	15.9
12	2	7	13.1	10.4
12	2	8	12.2	10.6
13	2	-13	10.5	10.0
13	2	-11	22.2	-20.5
13	2	-9	26.6	25.3
13	2	-8	25.7	23.6
13	2	-6	8.0	8.1
13	2	-4	22.7	-19.8
13	2	0	7.0	-6.8
13	2	2	8.5	-8.9
13	2	3	10.7	-12.4
14	2	-17	9.4	9.2
14	2	-16	14.6	12.4
14	2	-15	16.1	16.0
14	2	-14	13.9	12.5
14	2	-13	21.6	21.4
14	2	-12	11.1	10.5
14	2	-11	17.4	15.6
14	2	-10	31.4	27.8
14	2	-9	34.8	33.1
14	2	-8	24.6	22.1
14	2	-6	18.9	20.0
14	2	-5	10.9	10.7
14	2	-3	11.8	-11.1
14	2	-1	27.2	24.9
14	2	0	35.1	34.2
14	2	1	31.4	31.3
14	2	2	26.1	29.0
14	2	3	22.7	20.8
14	2	4	11.7	10.5
14	2	6	11.1	9.2
15	2	-17	12.4	-13.8
15	2	-15	12.8	11.4
15	2	-12	6.8	-6.9
15	2	-10	8.5	9.6
15	2	-9	17.4	15.7
15	2	-7	15.5	-13.5
15	2	-5	17.4	-16.3
15	2	-4	8.5	-9.2
15	2	-3	11.1	-11.9
15	2	-2	7.0	6.4
15	2	2	10.2	-10.2
15	2	6	5.7	4.1
16	2	-15	6.7	7.6
16	2	-14	21.6	20.3
16	2	-13	25.2	25.3
16	2	-11	17.4	14.4
16	2	-10	19.6	17.9
16	2	-9	22.2	20.3
16	2	-7	12.0	11.7
16	2	-6	27.2	25.9
16	2	-5	7.0	7.4
16	2	-3	18.9	19.4
16	2	-2	27.2	25.8
16	2	-1	17.4	17.0
16	2	0	15.0	13.6
16	2	1	22.7	22.9
16	2	2	14.8	13.1
16	2	4	5.5	5.6
16	2	5	9.4	6.6
17	2	-16	7.4	7.5
17	2	-8	14.1	-13.2
17	2	-7	15.7	15.2
17	2	-5	15.7	-16.2
17	2	0	12.6	12.2
17	2	3	5.5	5.3
18	2	-16	8.7	8.3
18	2	-15	10.9	8.7
18	2	-14	13.5	11.6
18	2	-12	9.8	7.7
18	2	-11	14.4	12.5
18	2	-10	6.8	5.1
18	2	-9	15.0	14.7
18	2	-8	23.3	22.0
18	2	-7	30.0	30.8
18	2	-6	26.1	24.1
18	2	-5	15.0	13.7
18	2	-4	10.5	8.8
18	2	-3	10.2	10.0
18	2	-2	17.4	16.8
18	2	-1	14.8	13.5
18	2	1	10.0	9.7
18	2	2	5.2	4.0
18	2	3	7.0	7.0
19	2	-7	6.5	5.4
19	2	-3	12.0	10.1
19	2	-1	9.4	8.5
19	2	0	9.6	9.2
20	2	-16	14.2	12.8
20	2	-15	10.5	9.6
20	2	-14	7.8	7.5
20	2	-10	16.5	14.9
20	2	-9	21.6	20.3
20	2	-8	14.6	13.6
20	2	-7	14.1	12.8
20	2	-6	18.9	17.7
20	2	-5	16.5	15.6
20	2	-4	13.1	13.2
20	2	-3	10.9	8.9
20	2	-2	12.6	11.8
21	2	-14	4.6	4.3
21	2	-13	7.8	7.0
21	2	-8	9.8	9.4
21	2	-4	5.0	5.8
21	2	-3	5.5	6.1
22	2	-14	4.4	4.8
22	2	-12	9.6	8.9
22	2	-11	9.1	8.3
22	2	-10	11.8	11.2
22	2	-9	12.4	11.3
22	2	-7	8.5	8.5
22	2	-6	9.1	9.8
22	2	-5	5.2	5.4
22	2	-3	12.2	9.6
0	3	2	8.1	-8.3
0	3	3	16.5	15.0
0	3	5	10.9	-10.4
0	3	7	12.0	-10.9
0	3	8	10.9	10.7
0	3	9	14.2	-12.2
0	3	11	16.5	-16.4
0	3	12	9.4	-8.3
1	3	-13	6.1	6.6
1	3	-12	7.0	8.4
1	3	-10	29.0	28.0
1	3	-9	17.4	18.2
1	3	-8	13.9	13.2
1	3	-7	30.9	31.5
1	3	-6	37.7	35.5
1	3	-5	47.7	41.3
1	3	-4	41.8	37.4
1	3	-3	43.3	40.8
1	3	-1	4.1	4.8
1	3	0	12.0	12.2
1	3	2	41.8	44.2
1	3	3	49.7	45.2
1	3	4	18.1	17.4
1	3	5	18.9	19.1
1	3	6	39.4	39.6
1	3	7	37.4	36.3
1	3	9	22.7	21.9
1	3	10	20.2	17.8
1	3	11	11.7	10.8
1	3	12	11.3	11.7
1	3	13	11.3	10.7
2	3	-14	5.0	5.7
2	3	-12	13.3	12.0
2	3	-10	14.4	13.8
2	3	-8	16.5	-14.5
2	3	-6	11.3	-12.1
2	3	-4	20.2	-17.6
2	3	-2	9.4	-9.4
2	3	-1	21.6	-23.6
2	3	0	7.2	6.6
2	3	1	22.2	23.8
2	3	3	6.8	-6.0
2	3	4	18.9	-17.9
2	3	5	5.5	6.5
2	3	6	16.5	-13.6
2	3	8	14.2	-12.3
2	3	9	14.6	-11.8
2	3	12	5.9	6.4
2	3	13	3.1	-2.6
3	3	-14	14.4	13.4
3	3	-13	16.5	17.4
3	3	-12	9.6	10.0
3	3	-11	22.2	19.9
3	3	-10	24.6	23.1
3	3	-8	21.6	18.3
3	3	-7	27.7	27.3
3	3	-6	30.5	27.5
3	3	-5	17.4	16.0
3	3	-4	39.4	38.2
3	3	-3	42.0	40.0
3	3	-2	22.2	22.7
3	3	-1	32.7	31.8
3	3	0	22.2	23.4
3	3	1	35.9	30.9
3	3	2	42.4	46.7
3	3	3	21.6	20.6
3	3	5	17.4	16.0
3	3	6	37.4	38.2
3	3	9	24.6	25.6
3	3	10	22.7	21.9
3	3	11	14.2	11.9
4	3	-12	9.6	9.9
4	3	-10	7.8	-6.9
4	3	-9	24.0	-23.8
4	3	-8	7.4	-10.0
4	3	-7	24.6	-22.6
4	3	-6	11.3	-8.0
4	3	-4	11.3	9.7
4	3	-3	13.7	11.1
4	3	-2	15.4	12.6
4	3	1	16.5	-12.9
4	3	4	8.7	7.9
4	3	6	15.2	-13.8
4	3	9	16.5	18.1
5	3	-15	10.2	11.9
5	3	-14	22.7	23.5
5	3	-13	24.0	24.7
5	3	-12	16.5	16.9
5	3	-11	22.7	20.0
5	3	-10	7.8	8.6
5	3	-8	5.9	-5.4
5	3	-7	27.2	25.6
5	3	-6	33.5	33.3
5	3	-5	26.1	23.6
5	3	-4	30.5	31.6
5	3	-3	40.1	38.3
5	3	-2	37.4	33.3
5	3	-1	23.3	24.4
5	3	0	48.3	42.0
5	3	1	44.9	42.2
5	3	2	12.2	13.1
5	3	3	12.2	11.8
5	3	4	28.1	34.0
5	3	5	29.2	32.2
5	3	7	14.4	-11.6
5	3	8	15.4	14.9
5	3	9	21.6	19.4
5	3	10	14.2	14.3
6	3	-15	8.3	-8.5
6	3	-14	12.8	-12.9
6	3	-12	6.5	-5.9
6	3	-7	6.8	6.5
6	3	-6	8.0	8.6
6	3	-5	7.4	8.1
6	3	-4	14.4	14.2
6	3	-3	9.6	9.5
6	3	-1	13.9	-12.3
6	3	0	10.5	9.8
6	3	2	14.6	13.6
6	3	3	17.4	16.3
6	3	4	13.7	12.0
6	3	5	15.4	-15.7
6	3	7	8.5	8.0
6	3	10	5.7	5.4
7	3	-16	8.5	9.5
7	3	-15	19.6	19.2
7	3	-14	14.6	15.1
7	3	-13	18.9	17.8
7	3	-12	20.3	17.4
7	3	-10	14.2	13.0
7	3	-9	27.7	26.6
7	3	-8	24.6	20.8
7	3	-7	8.7	8.1
7	3	-6	18.9	17.4
7	3	-5	34.8	34.3
7	3	-4	21.6	19.8
7	3	-3	26.1	26.0
7	3	-2	20.9	19.9
7	3	-1	24.0	23.6
7	3	0	30.5	29.7
7	3	1	38.1	40.5
7	3	2	39.4	43.3
7	3	3	30.0	29.1
7	3	4	13.9	11.7
7	3	7	13.5	12.0
7	3	8	15.5	15.7
7	3	9	13.9	13.6
7	3	10	5.0	4.1
8	3	-16	3.3	-4.1
8	3	-14	6.7	-5.3
8	3	-12	7.8	11.1
8	3	-11	8.0	10.9
8	3	-10	17.4	16.9
8	3	-9	6.1	5.9
8	3	-8	16.5	-17.1
8	3	-7	8.9	-8.1
8	3	-6	12.6	12.1
8	3	-5	37.7	36.4
8	3	-3	18.9	16.7
8	3	-1	5.9	-7.3
8	3	0	6.1	-7.6
8	3	3	15.0	-14.7
8	3	5	8.0	7.5
9	3	-15	12.6	11.3
9	3	-14	16.5	17.3
9	3	-13	16.1	17.8
9	3	-12	10.9	9.0
9	3	-11	29.2	26.4
9	3	-10	28.7	30.7
9	3	-7	29.6	31.0
9	3	-6	48.5	44.6
9	3	-5	12.2	10.6
9	3	-4	19.6	21.5
9	3	-3	20.9	23.3
9	3	-2	6.7	6.5
9	3	-1	23.3	22.9
9	3	0	25.2	26.2
9	3	1	19.6	17.3
9	3	2	15.2	13.1

TABLE 2 (*Continued*)

h	k	l	$\lvert F_o \rvert$	F_c
9	3	3	24·6	25·3
9	3	4	14·4	13·1
9	3	6	23·3	24·5
9	3	7	19·6	19·0
9	3	8	5·9	5·3
9	3	9	10·2	9·3
10	3	−14	5·2	−4·0
10	3	−13	5·9	5·2
10	3	−12	10·9	11·7
10	3	−11	6·5	6·9
10	3	−9	10·0	−11·5
10	3	−8	24·0	23·6
10	3	−7	13·5	11·1
10	3	−6	5·9	6·3
10	3	−5	12·6	−12·5
10	3	−3	25·2	−25·0
10	3	−1	13·5	−13·2
10	3	0	24·0	−24·1
10	3	2	10·0	8·8
10	3	3	17·4	18·2
10	3	4	7·6	8·6
10	3	5	6·2	−6·8
10	3	6	5·7	−7·8
11	3	−16	12·8	13·9
11	3	−14	6·7	7·7
11	3	−13	10·2	12·1
11	3	−12	7·8	7·6
11	3	−11	23·3	21·9
11	3	−10	15·5	14·5
11	3	−8	19·6	19·5
11	3	−7	68·6	64·7
11	3	−6	48·3	43·8
11	3	−5	5·9	−5·2
11	3	−4	16·5	15·3
11	3	−3	14·8	14·0
11	3	−1	6·2	−6·7
11	3	0	19·6	20·2
11	3	1	24·6	25·9
11	3	2	15·7	14·6
11	3	3	17·4	17·4
11	3	4	14·8	16·2
11	3	5	20·2	20·6
11	3	6	13·9	13·9
11	3	8	10·5	10·6
12	3	−15	5·7	−6·2
12	3	−13	12·4	11·5
12	3	−11	6·7	7·2
12	3	−8	22·2	−19·6
12	3	−7	31·4	−30·9
12	3	−5	10·0	−10·3
12	3	−4	13·9	14·3
12	3	−3	6·2	−5·3
12	3	−2	6·2	−5·8
12	3	0	7·8	8·6
12	3	1	10·2	10·2
12	3	4	5·7	4·7
12	3	5	6·5	−6·8
13	3	−16	6·7	5·8
13	3	−15	6·8	7·8
13	3	−14	11·7	12·4
13	3	−13	9·2	8·8
13	3	−12	14·8	14·0
13	3	−11	10·0	10·5
13	3	−10	6·7	5·6
13	3	−9	27·2	23·3
13	3	−8	39·2	39·5
13	3	−7	28·7	27·0
13	3	−6	10·0	−11·2
13	3	−5	17·4	17·4
13	3	−4	31·4	33·0
13	3	−3	12·8	10·3
13	3	−1	15·5	14·6
13	3	0	20·2	20·7
13	3	1	10·0	11·9
13	3	2	16·5	19·3
13	3	3	7·0	9·2
13	3	5	11·3	11·2
13	3	6	8·2	9·2
14	3	−10	14·2	−12·5
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18	4	−4	4·8	3·8
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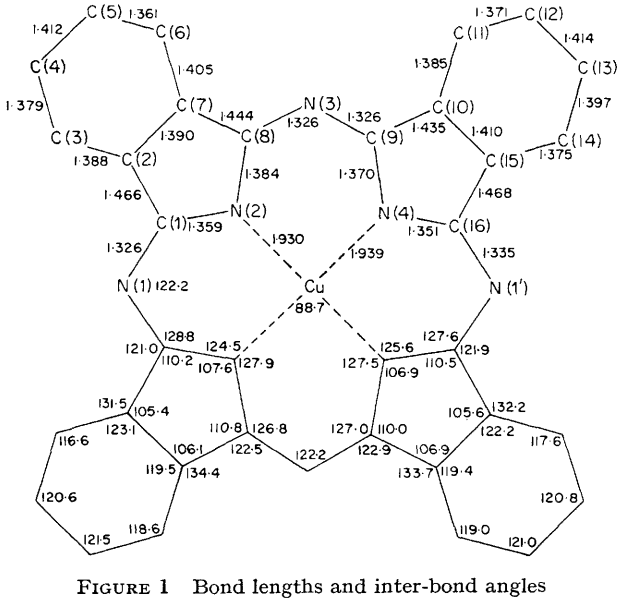

FIGURE 1 Bond lengths and inter-bond angles

of the mean plane through the molecule, referred to standard orthogonal axes, is: $0{\cdot}6780X - 0{\cdot}6976Y - 0{\cdot}2317Z = 0$. The mean displacement of the carbon and nitrogen atoms out of this plane is 0·03 Å, randomly above and below. The normal to the mean plane of the ring makes an angle of 45·8° with *b*, hence adjacent phthalocyanine molecules along *a* are inclined to each other by 91·6°. The perpendicular distance between successive planes of molecules along *b* is 3·34 A. The packing of the rings and the closest inter-molecular contacts are shown in Figures 2 and 3.

The mean C–N bond length in the central sixteen-membered ring is 1·347 Å which is exactly that required for a bond order of 1·5. It is evident, though, that the C–N bonds attached to N(1) and N(3) of length 1·328 Å are shorter than those attached to N(2) and N(4), 1·366 Å. This could be due to the influence of the copper atom attracting N(2) and N(4), but is more likely to be due to the five-membered isoindole ring diverting some of the π electrons away from the N(2) and N(4) branches.

The dimensions of the benzene rings are normal with

mean C–C 1·391 Å, and the mean angle 120·0°, and as with the central sixteen-membered ring, all the bonds are equivalent.

The C–C bonds in the isoindole ring, with mean length 1·453 Å have a bond order of 1·25 and are therefore obviously linked with the π-electron resonating system. A full treatment of the phthalocyanine molecule by both VB and MO theories would be of value.

ordinates with four atoms in a square planar system, with two other atoms at right angles to the plane at a greater distance, thus forming a distorted octahedron,[6] *e.g.* in copper dimethyl glyoxime,[7] there are four Cu–N bonds in a plane at 1·94 Å, and one Cu–O at right angles at 2·43 Å. In bis(benzene azo-β-naphthol) copper,[8] however, there are two Cu–N at 2·01, and two Cu–O at 1·93 Å, forming an almost square planar system, with

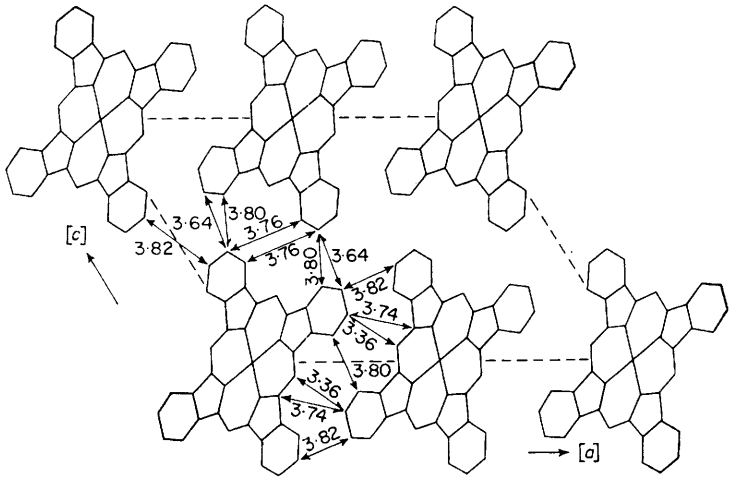

FIGURE 2 Diagrammatic projection on (001) showing intermolecular contacts

The Cu–N mean distance is 1·934 Å in a square-planar co-ordination system, with the next nearest copper contact to N(1) of the adjacent molecules along

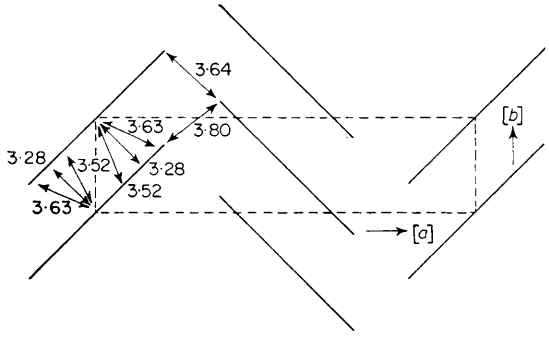

FIGURE 3 Diagrammatic projection on (001) showing the relative orientation of the phthalocyanine rings

b at 3·282 Å. These Cu–N(1) directions make angles of 89·6° with the Cu–N(2) bond and 86·2° with the Cu–N(4) bond. If these Cu–N(1) contacts are regarded as genuine interactions, then the copper co-ordination to nitrogen is distorted octahedral. Copper generally co-

two Cu–O interactions at 3·00 Å, at right angles to the plane. In tetramminecopper sulphate monohydrate,[9] there are four Cu–N bonds in a plane at 2·05 Å, and two Cu–O interactions at right angles at distances of 2·59 and 3·37 A. In copper phthalocyanine the co-ordinated N(1) atoms are each displaced out of the mean planes of their phthalocyanine rings in the direction of the copper atom by 0·066 Å, which is eleven times the standard deviation of the atomic co-ordinates.

It appears from preliminary work on the other polymorphs of copper phthalocyanine that the essential difference between the various polymorphic forms is that the copper co-ordinates to different nitrogen atoms of neighbouring rings, and this results in different packing arrangements between the rings and hence different crystal structures. In order to produce any given polymorphic form, it becomes necessary to block in some way the nitrogen atoms which are not required to co-ordinate. For example, the α-polymorph is readily formed by treating the β-polymorph with sulphuric acid and thus blocking the N(1) and N(3) atoms with sulphate residues; the copper then co-ordinates to N(2) or N(4).

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