Table 4. *Comparison of the dimension of the selenophthene and thiophthene molecules*

	X=Se	X=S
C(1) –C(2)	1·35 (3) Å	1·36 (2) Å
C(2) –C(3)	1·42 (2)	1·41 (2)
C(3) –C(3′)	1·36 (2)	1·36 (3)
C(1) –X	1·93 (2)	1·72 (1)
C(3′)–X	1·87 (1)	1·74 (1)
C(1)XC(3′)	86·1 (8)°	91·2°
XC(1)C(2)	111·4 (1·8)	116·5
C(1)C(2)C(3)	114·2 (1·4)	111·7
C(2)C(3)C(3′)	117·0 (1·1)	114·3
C(3)C(3′)X	111·1 (1·0)	110·2
C(2′)C(3′)X	131·8 (1·6)	135·5

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The Crystal Structure of Tetra(pyridine oxide)copper(II) Perchlorate

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The crystal structure of tetra(pyridine oxide)copper(II) perchlorate $Cu(PyO)_4(ClO_4)_2$ has been determined from three-dimensional X-ray diffraction data. Crystals are monoclinic with space group $P2_1/c$, and cell dimensions a=9·65, b=14·30, c=10·85 Å, β=122°0′. The structure was refined by Fourier and full-matrix least-squares methods on 1468 independent observed reflexions to R=10·4%. The structure is centrosymmetrical about the copper. Oxygen atoms from four pyridine oxide molecules form a square planar arrangement around the copper with Cu–O distances of 1·93 Å and 1·92 Å. The perchlorate ions occupy approximately octahedral positions, but are not involved in any coordination with the copper since the closest oxygen–metal approach is 3·38 Å.

Introduction

As part of a series of investigations into the role of polyanions in copper(II) complexes the structure of tetra(pyridine oxide)copper(II) perchlorate has been determined. Dark green coloured crystals in the form of hexagonal plates were prepared by Dr B. J. Hathaway (University of Essex).

A crystal of dimensions 0·4 mm × 0·3 mm × 0·2 mm was used for X-ray analysis. Three-dimensional Weissenberg data were collected for the crystal rotating about its b and c axes, allowing the observation of 1468 independent X-ray reflexions. Intensities were measured visually and converted to $|F|^2$ and $|F|$ by applying Lorentz and polarization corrections. No corrections were made for absorption or extinction.

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Crystal data

$Cu(C_5H_5NO)_4(ClO_4)_2$, M=642·9.

Monoclinic, a=9·65, b=14·30, c=10·85 all ±0·02 Å, β=122°0′±30′.

U=1269·7 Å^3, Z=2, D_m=1·66 g.cm^{-3}, D_c=1·681 g.cm^{-3}.

$F(000)$=654, Cu $K\alpha$, λ=1·542 Å, μ=38·4 cm^{-1}.

Absent reflexions, $h0l$ when $l=2n+1$, $0k0$ when $k=2n+1$; Space group $P2_1/c$.

Structure analysis

Since there are only two molecules of the complex in each unit cell, it was assumed that the molecules were centrosymmetric, with the copper atoms occupying special positions at 0,0,0 and $0,\frac{1}{2},\frac{1}{2}$. The positions of the light atoms were located from three-dimensional Patterson and Fourier sections. Several interpretations were possible, but all except one of the possibilities

were eliminated by structure-factor calculations in conjunction with bond-distance and angle calculations. The chosen trial structure gave a first residual $R=26{\cdot}3\%$.

Positional parameters and isotropic temperature factors were refined initially on an IBM 1620 computer, employing a least-squares program written by G.S.D. King (Union Carbide Ltd.), by use of a block-diagonal approximation. Reflexions too weak to be observed were omitted from the least-squares analysis, and initially all reflexions were given unit weights. The copper atom was included as Cu^+ with scattering factors from *International Tables for X-ray Crystallography* (1962), but the scattering factors used for the light atoms were those of Hanson, Herman, Lea & Skillman (1964).

Refinement was continued by use of a full-matrix least-squares method in the X-ray 63 system adapted for the SRC Chilton Atlas computer. The scattering factors for Cu^+ and Cl were modified for both the real and imaginary parts of anomalous dispersion. It was found that scattering factors for Cu^+ gave better agreement than those for Cu^{2+} or Cu, as would be expected from Pauling's electroneutrality principle. The final weighting scheme used was

$$w=\frac{1}{2(A)+|F_o|+(2/B)\,|F_o|^2}\,,$$

with values of $A=3{\cdot}6$ and $B=100$.

Anisotropic temperature factors were introduced first for copper atoms, and later for chlorine and oxygen atoms. In the final stages reflexions with $w\Delta F>5$ were assumed to be in error and were excluded from the least-squares matrix, though these reflexions are included in the residual, giving a final value of $R=10{\cdot}4\%$, based on the 1468 observed reflexions.

The final atomic parameters together with their standard deviations are set out in Tables 1 and 2. The

Table 1. *Final coordinates and standard deviations*

	x/a	*y/b*	*z/c*	$\sigma(x/a)$	$\sigma(y/b)$	$\sigma(z/c)$
Cu	0·0000	0·0000	0·0000	—	—	—
Cl	−0·2801	0·1101	−0·3828	0·0003	0·0002	0·0003
O(1)	0·0042	0·1164	0·0926	0·0008	0·0005	0·0007
O(11)	0·1761	0·0453	−0·0180	0·0008	0·0005	0·0008
O(21)	−0·2337	0·0176	−0·3868	0·0017	0·0008	0·0017
O(22)	−0·1536	0·1678	−0·3706	0·0011	0·0008	0·0012
O(23)	−0·3026	0·1291	−0·2700	0·0013	0·0009	0·0011
O(24)	−0·4278	0·1306	−0·5093	0·0012	0·0009	0·0011
N(1)	0·0947	0·1854	0·0877	0·0009	0·0006	0·0008
N(11)	0·2269	−0·0070	−0·0874	0·0009	0·0006	0·0009
C(1)	0·0284	0·2416	−0·0292	0·0011	0·0007	0·0010
C(2)	0·1220	0·3116	−0·0367	0·0013	0·0008	0·0012
C(3)	0·2808	0·3199	0·0726	0·0014	0·0009	0·0013
C(4)	0·3480	0·2629	0·1938	0·0013	0·0008	0·0012
C(5)	0·2511	0·1921	0·2001	0·0011	0·0007	0·0010
C(11)	0·1769	0·0176	−0·2249	0·0012	0·0007	0·0012
C(12)	0·2316	−0·0336	−0·2973	0·0015	0·0009	0·0014
C(13)	0·3447	−0·1026	−0·2272	0·0014	0·0009	0·0013
C(14)	0·3932	−0·1248	−0·0871	0·0015	0·0009	0·0014
C(15)	0·3332	−0·0744	−0·0150	0·0013	0·0008	0·0012

Table 2. *Final temperature factor parameters*

	B	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu		3·89	2·00	4·99	−0·51	3·38	−0·91
Cl		3·45	4·11	3·46	0·63	1·72	0·29
O(1)		4·06	3·13	4·69	−0·71	2·88	−1·05
O(11)		4·66	3·36	5·76	−0·40	3·90	−0·80
O(21)		10·36	6·38	14·33	1·92	7·16	1·31
O(22)		5·80	7·83	8·72	−0·50	4·44	0·31
O(23)		7·71	12·04	6·20	−2·45	4·62	−2·02
O(24)		5·73	10·69	5·52	1·25	1·68	0·78
N(1)	2·96 Å^2						
N(11)	3·14						
C(1)	3·36						
C(2)	4·41						
C(3)	4·89						
C(4)	4·23						
C(5)	3·35						
C(11)	3·67						
C(12)	5·00						
C(13)	4·56						
C(14)	5·03						
C(15)	4·03						

Table 3(*a*). *Observed and calculated structure factors*

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
0	0	4	33.6	32.9	3	1	-8	24.4	-26.9	3	2	8	6.2	7.8	3	3	-9	10.0	9.9	8	4	-9	7.6	-6.0	5	6	-9	6.7	4.2
0	0	6	6.2	-5.8	3	1	-9	3.4	-2.9	3	2	9	5.7	6.8	3	3	-10	11.9	10.7	8	4	-10	12.0	13.1	5	6	-10	23.3	20.1
0	0	8	33.2	26.8	3	1	-10	4.8	3.0	3	2	-2	94.7	91.4	3	3	-11	17.3	16.2	8	4	-12	10.7	9.4	5	6	-11	12.8	-11.7
0	0	10	25.0	24.0	3	1	-11	7.1	4.3	3	2	-4	40.1	38.8	3	3	-12	10.9	9.3	9	4	-3	7.6	-5.6	6	6	3	6.8	7.4
1	0	4	40.3	42.7	3	1	-12	5.1	4.9	3	2	-5	8.7	-6.7	4	3	5	10.1	-11.6	9	4	-8	18.8	18.9	6	6	-8	38.8	42.5
1	0	6	16.1	-15.5	4	1	5	8.7	9.5	3	2	-6	28.6	26.1	4	3	6	8.7	8.0	9	4	-9	8.7	6.8	6	6	-9	10.2	-9.4
1	0	10	18.4	17.7	4	1	7	10.0	9.7	3	2	-8	27.4	28.4	4	3	8	5.7	6.5	9	4	-10	17.8	16.5	6	6	-10	8.2	8.7
1	0	-4	29.5	-28.3	4	1	8	5.5	6.2	3	2	-9	10.7	11.2	4	3	-7	28.0	29.7	9	4	-11	5.9	5.2	7	6	-7	20.7	-21.7
1	0	-6	42.8	41.7	4	1	-5	47.6	49.4	3	2	-10	11.9	11.3	4	3	-8	9.6	9.8	10	4	-7	8.4	7.5	7	6	-8	23.3	24.2
1	0	-8	17.5	14.9	4	1	-6	10.1	-9.9	3	2	-11	9.2	-9.2	4	3	-9	16.4	18.0	10	4	-8	20.1	17.9	7	6	-9	7.0	6.7
1	0	-10	14.2	-15.3	4	1	-7	10.7	9.1	3	2	-12	8.3	7.6	4	3	-10	12.3	9.2	11	4	-6	12.7	11.2	7	6	-10	8.8	10.7
2	0	2	88.1	83.1	4	1	-8	11.8	10.9	3	2	-13	4.4	-5.1	4	3	-11	10.1	11.1	0	5	7	18.1	17.8	7	6	-11	11.2	10.6
2	0	4	38.0	38.1	4	1	-11	24.0	22.2	4	2	5	21.4	20.9	5	3	4	7.3	-9.1	0	5	8	7.6	-6.6	8	6	-7	8.8	8.6
2	0	6	36.4	36.3	4	1	-13	7.6	7.9	4	2	7	5.2	-5.2	5	3	5	10.3	7.2	0	5	9	21.5	20.2	8	6	-8	14.3	15.4
2	0	8	10.5	10.4	5	1	0	6.9	-3.4	4	2	-6	29.1	27.2	5	3	6	5.6	-4.0	1	5	7	11.4	-13.1	8	6	-9	16.0	13.5
2	0	-4	63.5	69.5	5	1	3	7.0	7.1	4	2	-7	24.7	25.7	5	3	-7	37.8	37.6	1	5	-7	30.4	31.0	8	6	-10	13.1	14.0
2	0	-6	30.4	25.8	5	1	7	6.1	5.0	4	2	-8	24.0	25.2	5	3	-9	28.2	32.7	1	5	-8	13.4	-16.2	9	6	-7	13.9	14.1
2	0	-8	18.0	-16.8	5	1	-6	3.7	5.0	4	2	-9	17.9	-17.6	5	3	-10	18.6	-16.0	2	5	7	14.5	12.9	9	6	-8	18.3	18.7
2	0	-12	11.3	11.9	5	1	-7	10.2	9.6	4	2	-10	23.3	24.6	5	3	-11	13.4	12.2	2	5	-7	18.9	20.8	0	7	5	5.3	4.4
3	0	2	12.0	-11.8	5	1	-8	8.8	9.3	4	2	-11	4.6	-3.2	5	3	-12	8.5	-6.9	2	5	-8	10.5	-12.8	0	7	6	1.4	-2.9
3	0	4	3.5	-2.1	5	1	-9	37.5	44.9	5	2	7	7.9	6.3	5	3	-13	10.0	10.9	2	5	-9	4.4	-4.2	1	7	-7	18.2	19.7
3	0	6	29.7	26.0	5	1	-10	6.9	-7.8	5	2	-7	29.3	-31.7	6	3	4	8.2	7.9	3	5	7	23.3	26.7	1	7	-8	16.9	-17.9
3	0	8	17.0	15.0	5	1	-11	17.2	18.1	5	2	-8	24.6	25.0	6	3	5	12.9	12.4	3	5	-5	32.1	35.9	2	7	5	20.4	20.5
3	0	-2	78.5	80.7	5	1	-12	5.7	-4.3	5	2	-9	17.8	-20.5	6	3	-7	13.3	11.2	3	5	-6	5.9	-2.3	2	7	-7	18.3	20.0
3	0	-6	37.0	-40.6	5	1	-13	6.3	4.4	5	2	-10	31.1	27.8	6	3	-8	23.9	-24.7	3	5	-7	17.8	17.6	3	7	-7	25.8	24.2
3	0	-8	7.3	7.4	6	1	2	4.8	7.1	5	2	-11	5.6	4.6	6	3	-9	28.5	33.1	3	5	-8	14.5	12.2	3	7	-8	27.9	26.9
3	0	-10	24.7	25.6	6	1	4	7.2	7.4	5	2	-12	14.3	13.6	6	3	-11	18.5	15.9	3	5	-9	11.8	10.4	4	7	-7	18.8	19.4
3	0	-12	8.4	7.0	6	1	5	7.5	9.1	5	2	-13	4.1	3.3	6	3	-13	8.3	6.4	3	5	-11	6.8	5.2	5	7	0	2.1	-2.2
4	0	2	17.3	16.6	6	1	6	3.1	-3.3	6	2	4	9.9	10.9	7	3	0	7.2	-6.0	4	5	-7	38.4	38.8	6	7	-7	26.0	24.0
4	0	4	10.7	-9.6	6	1	-2	4.5	2.3	6	2	-7	12.3	-10.5	7	3	2	13.6	-12.3	4	5	-8	5.9	3.5	6	7	-8	15.9	13.9
4	0	8	10.6	9.7	6	1	-7	48.5	52.2	6	2	-8	28.0	28.2	7	3	-7	29.5	32.5	4	5	-9	15.0	18.2	7	7	-7	24.6	24.3
4	0	-6	10.7	8.2	6	1	-8	17.4	-16.9	6	2	-9	21.5	22.0	7	3	-8	11.8	-12.0	4	5	-10	6.4	-3.6	0	4	0	4.8	-3.2
4	0	-8	17.6	19.6	6	1	-9	31.8	33.8	6	2	-10	19.2	17.4	7	3	-9	17.0	16.1	5	5	5	20.5	20.5	0	6	0	62.2	69.6
5	0	2	28.1	27.1	7	1	2	11.3	-11.1	6	2	-12	14.7	12.1	7	3	-11	9.6	7.0	5	5	-7	40.1	37.7	0	8	0	69.6	75.4
5	0	4	29.0	27.7	7	1	3	9.5	7.5	7	2	-8	16.1	15.1	7	3	-12	7.1	6.4	5	5	-8	16.6	-17.3	0	10	0	48.4	58.9
5	0	-6	70.5	80.9	7	1	5	6.9	9.7	7	2	-9	6.7	-6.2	8	3	0	6.7	4.8	5	5	-9	4.7	-3.8	0	12	0	13.8	14.3
5	0	-8	15.0	15.8	7	1	-7	12.0	11.3	7	2	-10	8.8	8.9	8	3	1	10.0	7.4	5	5	-13	6.6	7.6	0	14	0	13.2	-10.0
5	0	-10	26.5	24.3	7	1	-8	15.0	-14.7	7	2	-11	5.8	-5.9	8	3	-7	26.7	31.8	6	5	2	6.9	-6.7	0	16	0	28.3	26.1
5	0	-12	13.3	10.6	7	1	-9	8.1	-6.7	7	2	-12	9.6	9.3	8	3	-8	11.0	10.7	6	5	3	9.4	9.1	0	18	0	10.1	13.1
6	0	4	24.2	24.4	7	1	-11	11.2	7.4	8	2	-1	5.9	-4.4	8	3	-9	9.2	-8.7	6	5	5	12.2	13.6	1	3	0	121.5	143.4
6	0	6	12.8	13.6	7	1	-13	6.7	7.5	8	2	-7	19.5	-18.6	8	3	-13	7.5	8.5	6	5	-6	5.8	-5.5	1	4	0	85.9	96.6
6	0	-8	29.0	32.6	8	1	2	4.2	-4.6	8	2	-8	11.4	15.2	9	3	-7	11.7	12.0	6	5	-7	7.5	7.0	1	5	0	30.8	-36.4
6	0	-10	22.4	24.6	8	1	4	3.7	-5.8	8	2	-9	27.1	-25.3	9	3	-11	12.3	12.0	6	5	-9	13.1	11.3	1	6	0	59.7	68.7
6	0	-12	13.4	12.5	8	1	-5	2.1	0.7	8	2	-10	11.9	10.1	9	3	-12	6.4	-5.7	6	5	-10	6.6	-6.8	1	7	0	45.1	-61.0
7	0	2	22.0	19.2	8	1	-7	1.8	-1.8	8	2	-11	10.3	9.4	9	3	-13	9.6	10.1	6	5	-11	9.9	7.6	1	8	0	19.9	18.1
7	0	4	7.3	-5.8	8	1	-9	16.7	17.0	8	2	-12	16.7	15.9	10	3	-1	6.4	6.1	6	5	-13	5.4	5.4	1	9	0	20.7	20.0
7	0	-8	34.8	43.0	8	1	-11	25.4	25.1	8	2	-13	7.8	7.9	10	3	-3	8.0	8.2	7	5	-7	29.5	30.7	1	10	0	26.8	27.7
8	0	2	18.5	18.3	8	1	-13	8.5	7.9	9	2	-2	15.6	15.5	10	3	-6	7.0	6.9	7	5	-9	38.5	35.8	1	11	0	21.9	21.1
8	0	-8	9.0	9.0	9	1	0	5.2	-4.9	9	2	-7	15.0	-13.7	10	3	-7	11.5	9.2	7	5	-10	7.7	-6.0	1	12	0	21.7	21.7
8	0	-10	9.6	5.9	9	1	1	12.7	13.5	9	2	-8	17.6	18.1	10	3	-8	8.0	-9.2	8	5	1	14.6	12.9	1	13	0	8.4	5.9
9	0	2	5.8	5.9	9	1	2	2.8	3.5	9	2	-9	11.0	9.2	10	3	-9	23.3	21.3	8	5	3	8.8	8.1	1	15	0	3.1	4.5
9	0	-4	12.2	14.0	9	1	-2	5.6	-6.7	9	2	-10	22.0	20.2	10	3	-11	11.8	11.3	8	5	-1	4.1	-3.7	1	16	0	21.2	17.9
9	0	-8	5.2	-5.9	9	1	-3	7.1	7.5	9	2	-11	9.2	9.0	10	3	-12	5.6	4.8	8	5	-5	18.7	19.7	1	18	0	7.6	8.7
9	0	-10	20.6	19.1	9	1	-6	4.5	4.2	9	2	-12	12.6	13.6	11	3	-5	13.9	13.2	8	5	-7	31.8	34.8	2	0	0	85.0	-104.3
9	0	-12	16.7	18.9	9	1	-7	21.7	21.8	9	2	-13	7.0	4.3	11	3	-6	11.4	-11.2	8	5	-8	9.1	-8.2	2	1	0	60.7	-71.2
10	0	0	5.8	6.5	9	1	-9	26.9	26.2	10	2	0	5.8	6.4	11	3	-7	15.9	16.8	9	5	-1	7.7	6.3	2	2	0	35.6	-32.9
10	0	-4	4.0	-3.1	9	1	-10	6.2	-7.8	10	2	-5	5.9	-5.4	11	3	-9	11.8	12.8	9	5	-5	10.4	13.9	2	3	0	71.3	-81.0
10	0	-8	12.7	12.3	9	1	-11	16.7	16.9	10	2	-7	5.7	4.4	12	3	-5	6.5	10.9	9	5	-7	8.2	11.1	2	4	0	28.4	24.9
10	0	-10	27.3	24.7	9	1	-13	6.9	6.1	10	2	-8	17.2	17.1	12	3	-7	11.1	14.7	9	5	-8	6.1	4.9	2	5	0	11.4	10.2
10	0	-12	19.3	18.2	10	1	-6	5.7	-4.7	10	2	-10	19.9	21.9	0	4	8	9.7	-9.5	9	5	-10	7.6	7.8	2	7	0	12.6	12.9
11	0	-8	18.0	18.6	10	1	-7	20.6	21.3	10	2	-11	6.8	-6.6	1	4	6	27.3	32.5	9	5	-11	17.8	19.3	2	8	0	28.5	30.5
11	0	-10	16.4	14.7	10	1	-8	12.3	-10.9	10	2	-12	6.4	7.1	1	4	8	18.5	20.3	10	5	-1	9.1	11.8	2	9	0	5.1	8.1
12	0	-6	8.8	8.9	10	1	-9	14.6	14.5	11	2	-2	5.4	6.3	1	4	-7	10.7	-8.9	10	5	-3	10.4	9.4	2	11	0	18.9	-15.6
0	1	8	23.4	29.3	10	1	-10	4.0	-2.5	11	2	-5	4.8	5.3	1	4	-8	13.2	15.2	10	4	-4	5.1	-2.9	2	12	0	23.2	21.3
1	1	1	80.0	75.9	10	1	-11	11.2	11.6	11	2	-6	22.3	23.4	1	4	-10	16.7	16.4	10	5	-5	12.4	9.0	2	14	0	25.0	22.7
1	1	6	7.1	11.4	10	1	-12	6.5	6.2	11	2	-8	21.2	20.4	2	4	5	16.5	-16.9	10	5	-7	14.0	11.7	2	15	0	11.0	8.9
1	1	7	26.6	26.3	10	1	-13	8.0	9.2	11	2	-9	11.2	-11.4	2	4	8	12.2	10.2	10	5	-8	11.0	9.6	2	16	0	16.0	13.2
1	1	8	25.7	25.5	11	1	-5	20.2	20.4	11	2	-10	9.2	10.7	2	4	-7	9.7	-10.6	10	5	-9	23.6	24.9	3	0	0	71.3	81.0
1	1	9	7.9	5.6	11	1	-7	5.9	6.5	11	2	-11	5.1	-5.7	2	4	-8	42.1	49.1	10	5	-10	7.9	6.1	3	1	0	21.7	-20.5
1	1	-3	88.4	89.0	11	1	-8	7.8	-7.2	12	2	-6	5.1	8.2	3	4	5	23.6	-24.9	10	5	-11	9.7	12.9	3	2	0	5.4	3.6
1	1	-4	20.1	-17.3	11	1	-9	15.9	15.4	12	2	-7	7.0	-7.7	3	4	6	17.0	17.8	11	5	-4	4.9	-5.1	3	3	0	18.3	-16.4
1	1	-5	12.5	-12.8	11	1	-11	12.7	13.5	12	2	-8	9.5	12.9	3	4	-7	12.4	12.3	11	5	-5	8.8	9.1	3	4	0	23.5	-23.8
1	1	-7	3.6	-2.1	12	1	-6	4.9	5.2	12	2	-9	7.3	-7.9	3	4	-9	11.2	8.5	11	5	-7	17.3	17.8	3	5	0	21.8	-20.8
1	1	-8	17.0	17.4	12	1	-8	3.7	3.7	0	3	5	37.5	36.3	3	4	-10	12.8	-11.4	11	5	-9	12.3	15.0	3	6	0	31.8	-28.9
1	1	-9	22.9	23.2	0	2	2	46.0	43.2	0	3	7	28.1	29.7	4	4	6	12.9	11.0	0	6	5	16.9	17.6	3	7	0	14.5	12.6
1	1	-10	5.9	-7.0	0	2	7	15.2	-16.3	0	3	8	7.4	6.5	4	4	-8	2.1	-3.1	0	6	6	4.6	3.9	3	8	0	54.3	63.3
2	1	6	6.1	8.9	0	2	8	21.6	24.1	0	3	9	16.8	15.4	4	4	-10	22.4	21.4	1	6	-7	13.6	13.6	3	10	0	30.9	34.5
2	1	8	4.5	-5.0	0	2	9	12.7	12.1	0	3	10	10.5	-10.7	4	4	-12	12.3	11.6	1	6	-8	14.1	12.8	3	11	0	18.0	-14.2
2	1	9	11.5	9.5	0	2	10	10.1	8.1	1	3	8	6.9	4.1	5	4	3	7.3	-5.3	2	6	0	7.1	8.0	3	12	0	20.2	19.3
2	1	-1	83.1	79.7	1	2	9	9.2	-7.9	1	3	-7	33.0	31.5	5	4	4	9.7	9.0	2	6	6	12.8	13.1	3	13	0	14.2	-10.5
2	1	-4	25.1	-23.3	1	2	-7	24.7	-23.9	1	3	-8	13.1	15.0	5	4	6	9.6	9.1	0	6	7	11.6	11.0	3	16	0	15.5	14.0
2	1	-5	15.5	-14.6	1	2	-8	23.1	26.6	1	3	-10	6.8	7.2	5	4	-7	17.5	-19.4	1	6	8	9.9	8.5	4	0	0	58.7	65.6
2	1	-6	8.0	5.6	1	2	-9	28.3	-25.6	2	3	7	14.2	13.4	5	4	-8	33.6	36.9	2	6	5	9.9	-8.2	4	1	0	13.8	-14.3
2	1	-7	27.9	25.7	1	2	-10	11.2	9.7	2	3	8	8.3	-8.2	5	4	-9	12.3	-11.5	2	6	-7	7.6	6.3	4	2	0	57.0	64.1
2	1	-8	10.0	-11.4	2	2	5	6.5	8.0	2	3	-7	26.7	29.8	5	4	-10	22.3	20.3	2	6	-8	22.6	25.7	4	3	0	49.4	52.4
2	1	-9	21.2	18.6	2	2	7	22.7	27.1	2	3	-8	11.2	-7.9	6	4	4	11.1	12.8	2	6	-9	8.9	-7.8	4	4	0	34.1	32.4
2	1	-10	15.2	-15.2	2	2	-4	23.2	24.4	2	3	-9	10.0	8.0	6	4	-7	11.6	-10.7	3	6	5	30.4	30.1	4	5	0	16.3	13.9
3	1	6	5.6	3.8	2	2	-5	8.2	10.4	2	3	-10	17.6	-16.0	6	4	-8	33.7	32.8	4	6	4	9.0	8.9	4	6	0	29.9	24.4
3	1	9	5.3	4.9	2	2	-6	42.2	41.4	3	3	5	14.1	14.4	7	4	4	11.8	14.9	4	6	-8	2.5	-2.3	4	7	0	18.6	-15.6
3	1	-3	39.9	33.5	2	2	-7	24.2	-25.2	3	3	6	6.6	6.8	7	4	-1	7.2	-5.1	4	6	-9	11.8	9.8	4	8	0	50.7	53.5
3	1	-4	25.1	23.6	2	2	-8	19.0	17.2	3	3	7	11.9	11.8	7	4	-9	9.6	-9.1	4	6	-10	20.8	20.4	4	10	0	39.3	35.4
3	1	-5	45.0	49.4	2	2	-9	1.9	3.4	3	3	-2	68.5	80.6	7	4	-10	5.1	-5.2	5	6	4	8.8	9.3	4	11	0	16.7	11.4
3	1	-6	32.6	31.0	3	2	5	10.3	-11.4	3	3	-7	19.8	17.9	7	4	-11	14.0	-13.8	5	6	-7	23.7	25.2	4	12	0	12.1	9.5
3	1	-7	38.6	42.7	3	2	6	8.0	8.5	3	3	-8	17.6	-17.5	7	4	-12	8.9	8.2	5	6	-8	27.7	31.1	4	16	0	11.7	12.3

Table 3(*a*) (*cont.*)

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
5	0	0	28.6	26.9	6	3	-1	36.7	37.2	6	1	1	45.8	52.7	10	3	-2	11.1	9.1	3	12	-3	21.6	-21.7	2	15	-3	27.5	23.5
5	2	0	56.2	53.0	6	4	-1	22.9	21.6	6	2	1	13.7	13.8	10	8	-2	8.6	10.4	3	13	-3	29.4	28.5	2	16	-3	12.7	-9.6
5	3	0	23.1	19.2	6	5	-1	8.7	8.7	6	3	1	10.9	11.0	11	3	-2	6.0	7.1	3	15	-3	18.4	20.7	2	1	3	25.0	-24.1
5	4	0	52.4	52.7	6	6	-1	38.3	-41.2	6	5	1	8.1	-8.7	0	3	2	59.6	-61.0	4	1	-3	67.2	69.5	2	2	3	41.3	-44.1
5	5	0	18.5	14.0	6	7	-1	10.9	12.5	6	7	1	16.2	16.1	0	4	2	30.7	32.0	4	2	-3	14.1	13.7	2	3	3	52.5	56.4
5	6	0	30.9	28.6	6	8	-1	20.9	-17.5	6	9	1	23.0	25.3	0	5	2	57.8	53.9	4	3	-3	41.0	38.5	2	4	3	21.8	-23.4
5	8	0	42.4	35.5	6	9	-1	9.8	8.5	7	2	1	15.3	14.8	0	6	2	39.3	37.1	4	4	-3	42.9	41.4	2	5	3	27.8	29.2
5	9	0	21.7	20.5	6	10	-1	12.2	10.0	7	3	1	19.1	18.1	0	7	2	24.9	25.2	4	6	-3	32.0	-31.6	2	6	3	16.5	15.2
5	11	0	16.5	15.1	6	13	-1	10.9	10.5	7	5	1	18.6	17.1	0	8	2	20.3	21.7	4	7	-3	31.7	35.0	2	7	3	11.6	9.3
5	14	0	16.7	16.0	7	1	-1	3.7	-4.8	7	6	1	6.8	-6.5	0	9	2	8.5	5.3	4	8	-3	0.8	-2.8	2	8	3	15.8	16.0
6	0	0	25.6	21.0	7	2	-1	16.6	-19.2	2	3	-2	8.0	-10.3	0	10	2	21.9	19.6	4	9	-3	49.4	54.9	2	9	3	20.0	25.2
6	1	0	16.3	-12.7	7	3	-1	33.9	33.6	2	4	-2	9.1	-15.4	0	11	2	26.6	-26.8	4	11	-3	27.5	25.5	2	11	3	15.2	16.6
6	2	0	28.7	24.8	7	5	-1	11.9	12.0	2	5	-2	14.5	-14.5	0	12	2	21.2	20.4	4	12	-3	8.6	-8.1	2	12	3	12.1	-10.5
6	3	0	17.3	-13.7	7	7	-1	15.8	14.7	2	6	-2	18.7	15.9	0	13	2	12.6	-11.2	4	15	-3	8.8	8.1	2	13	3	20.0	22.9
6	4	0	27.1	23.2	7	13	-1	11.4	14.1	2	7	-2	2.0	-3.3	0	14	2	16.1	14.6	5	1	-3	26.3	27.6	2	14	3	14.5	14.0
6	7	0	33.3	28.8	8	1	-1	26.2	25.0	2	10	-2	31.6	40.9	0	15	2	15.6	13.8	5	2	-3	28.1	26.9	2	15	3	10.8	9.6
6	8	0	23.6	16.0	8	4	-1	12.1	-10.1	2	13	-2	7.9	-7.4	0	16	2	23.0	19.7	5	3	-3	51.6	60.3	2	16	3	10.2	11.5
6	12	0	15.5	13.5	8	6	-1	13.1	10.0	2	15	-2	9.8	8.7	1	1	2	16.8	-15.8	5	4	-3	30.5	30.3	3	1	3	32.9	33.8
6	14	0	10.0	9.3	8	7	-1	16.7	15.7	2	16	-2	11.4	15.7	1	2	2	64.9	73.7	5	5	-3	35.9	34.5	3	2	3	27.4	-29.8
7	0	0	41.4	36.0	8	8	-1	10.5	8.9	3	1	-2	38.1	34.5	1	3	2	24.7	-20.3	5	6	-3	24.6	-25.6	3	3	3	21.5	25.0
7	2	0	10.9	8.6	8	9	-1	23.4	24.5	3	4	-2	52.5	50.6	1	4	2	30.9	26.8	5	7	-3	19.7	18.2	3	5	3	15.6	-17.3
7	4	0	18.3	-15.6	8	12	-1	5.9	5.0	3	5	-2	8.8	-10.0	1	5	2	26.8	24.5	5	8	-3	15.0	-15.0	3	6	3	13.7	16.5
7	5	0	14.9	-11.2	9	1	-1	19.3	18.0	3	6	-2	30.0	33.4	1	6	2	26.2	26.2	5	9	-3	10.5	9.2	3	7	3	18.4	20.9
7	6	0	11.7	7.9	9	2	-1	16.3	16.4	3	7	-2	40.9	-45.4	1	7	2	26.6	-24.3	5	10	-3	27.6	26.6	3	9	3	31.9	40.7
7	7	0	12.0	9.4	9	7	-1	11.4	11.0	3	8	-2	58.0	64.2	1	8	2	70.5	68.3	5	12	-3	17.8	16.1	3	10	3	10.9	-9.3
7	8	0	22.9	18.8	9	9	-1	10.6	12.0	3	9	-2	19.0	17.9	1	10	2	36.8	40.0	6	1	-3	29.2	28.3	4	1	3	19.8	17.2
7	9	0	21.7	-17.7	10	6	-1	0.9	0.2	3	10	-2	41.9	49.2	1	12	2	20.1	17.0	6	2	-3	15.3	-14.2	4	2	3	30.2	36.1
7	10	0	21.3	15.3	0	4	1	4.9	4.0	3	11	-2	13.0	14.3	1	15	2	11.1	10.7	6	3	-3	21.9	23.1	4	3	3	21.3	23.9
8	0	0	21.4	17.1	0	5	1	48.2	48.3	3	12	-2	28.0	28.2	1	16	2	16.5	14.9	6	4	-3	13.8	14.9	4	5	3	21.2	21.8
8	1	0	10.1	10.2	0	6	1	14.6	-14.6	3	14	-2	16.8	14.0	1	3	-2	17.4	-15.9	6	5	-3	37.4	41.4	4	6	3	0.2	-0.4
8	4	0	8.7	8.3	0	7	1	19.4	17.6	3	16	-2	12.0	15.5	1	5	-2	21.8	-19.9	6	6	-3	2.8	-2.9	4	7	3	20.3	22.7
8	5	0	15.1	-13.0	0	8	1	7.1	6.7	4	0	-2	51.8	51.6	1	6	-2	27.0	24.5	6	9	-3	4.1	-4.0	5	2	3	10.9	-9.8
8	6	0	12.7	12.7	0	9	1	27.2	24.3	4	1	-2	18.6	-17.4	1	7	-2	4.1	-5.2	6	10	-3	8.5	10.2	5	3	3	22.8	18.8
8	7	0	10.6	-8.1	0	10	1	13.2	13.7	4	2	-2	30.8	31.6	1	8	-2	11.5	-11.5	6	11	-3	7.5	8.4	5	5	3	31.4	39.9
8	8	0	12.1	10.2	0	11	1	17.1	15.6	4	3	-2	39.3	37.7	1	9	-2	13.8	-8.7	6	13	-3	17.7	19.2	5	6	3	16.5	18.8
8	10	0	13.2	11.5	0	13	1	29.8	24.3	4	4	-2	76.4	77.7	1	12	-2	21.6	23.1	7	1	-3	24.7	27.6	6	1	3	24.2	21.2
8	11	0	8.3	6.4	0	14	1	11.8	9.8	4	5	-2	32.3	27.0	1	16	-2	16.3	15.6	7	2	-3	16.7	-15.7	6	2	3	15.4	-13.4
9	4	0	17.3	15.2	0	15	1	24.2	22.0	4	6	-2	51.2	52.1	2	1	2	0.7	1.2	7	3	-3	24.1	22.0	4	0	-4	11.2	12.2
9	6	0	12.2	9.1	0	17	1	14.2	13.5	4	7	-2	5.3	-4.2	2	2	2	7.0	5.8	7	4	-3	12.7	8.8	4	1	-4	17.4	20.4
9	7	0	10.2	7.4	1	2	1	63.1	-59.5	4	8	-2	40.6	40.8	2	3	2	12.6	13.5	7	5	-3	11.4	10.6	4	3	-4	32.2	30.2
1	3	-1	43.9	-47.7	1	4	1	12.4	-10.1	4	10	-2	17.9	17.1	2	4	2	49.6	55.3	7	6	-3	10.1	-10.9	4	4	-4	37.8	44.7
1	4	-1	39.9	-36.2	1	5	1	48.0	52.7	4	11	-2	15.2	15.6	2	5	2	15.2	-16.0	7	7	-3	29.1	26.1	4	5	-4	17.2	17.0
1	5	-1	15.3	13.6	1	6	1	8.1	12.1	4	12	-2	30.9	30.9	2	6	2	37.7	40.8	7	9	-3	28.3	27.0	4	6	-4	52.7	61.3
1	6	-1	4.6	2.1	1	7	1	44.2	38.4	4	14	-2	14.2	17.0	2	7	2	15.6	-14.1	7	11	-3	20.3	19.0	4	7	-4	29.9	31.7
1	7	-1	50.1	48.6	1	9	1	43.1	40.0	4	16	-2	11.2	13.0	2	8	2	33.7	36.2	7	13	-3	9.3	10.2	4	8	-4	3.1	-2.7
1	8	-1	20.1	-18.6	1	10	1	20.6	-17.4	5	0	-2	51.6	51.6	2	10	2	13.7	9.7	8	1	-3	21.4	21.7	4	9	-4	7.6	8.3
1	9	-1	50.2	48.9	1	11	1	11.8	10.3	5	1	-2	12.3	-13.7	2	11	2	17.8	18.1	8	2	-3	10.7	11.1	4	12	-4	19.4	19.4
1	10	-1	15.8	-16.3	1	12	1	12.6	-11.8	5	2	-2	22.7	21.1	2	12	2	17.5	19.3	8	3	-3	26.4	26.9	4	14	-4	14.3	17.2
1	11	-1	20.4	26.6	1	13	1	27.8	24.2	5	3	-2	12.3	10.5	2	13	2	14.2	15.2	8	6	-3	15.8	-14.4	5	0	-4	38.0	41.3
1	12	-1	8.1	-8.5	1	16	1	13.1	13.0	5	4	-2	11.3	8.4	2	14	2	12.4	12.6	8	7	-3	33.5	28.8	5	2	-4	17.7	19.6
2	2	-1	34.8	36.8	1	17	1	12.8	13.9	5	5	-2	17.2	-16.9	2	15	2	7.4	5.7	8	8	-3	10.3	-8.9	5	3	-4	6.3	4.8
2	3	-1	44.9	-52.5	2	1	1	79.5	70.4	5	6	-2	30.1	32.2	2	16	2	9.7	11.2	8	9	-3	28.7	27.2	5	4	-4	29.1	25.7
2	4	-1	5.6	-3.1	2	2	1	79.1	-75.3	5	7	-2	10.5	9.8	3	1	2	28.9	-24.9	8	11	-3	10.6	11.4	5	5	-4	11.4	-11.5
2	5	-1	33.5	29.2	2	3	1	36.1	32.3	5	8	-2	13.1	14.0	3	2	2	9.6	10.3	8	12	-3	6.9	6.7	5	6	-4	11.6	11.8
2	6	-1	65.1	-71.4	2	4	1	14.0	-13.7	5	11	-2	8.5	6.6	3	3	2	5.4	-7.9	8	13	-3	11.0	12.2	5	7	-4	17.6	18.9
2	7	-1	29.5	31.2	2	5	1	9.5	9.9	5	12	-2	9.9	10.4	3	4	2	39.5	41.4	9	2	-3	24.4	20.4	5	8	-4	13.7	15.9
2	8	-1	18.7	-19.0	2	6	1	9.4	9.0	5	13	-2	10.5	-9.2	3	6	2	24.3	25.9	9	3	-3	21.4	19.9	5	9	-4	15.2	15.0
2	9	-1	39.8	46.8	2	7	1	45.5	49.0	5	14	-2	11.6	12.5	3	7	2	22.0	21.2	9	5	-3	13.7	13.3	5	10	-4	10.2	13.3
2	10	-1	17.9	21.8	2	9	1	49.0	53.4	6	0	-2	34.0	34.9	3	9	2	13.4	14.7	9	7	-3	10.5	11.7	5	12	-4	11.9	13.4
2	11	-1	26.1	30.1	2	10	1	6.7	-5.7	6	2	-2	32.7	31.8	3	11	2	16.8	-12.5	9	12	-3	9.6	7.7	5	13	-4	6.5	-5.9
2	12	-1	13.7	13.9	2	11	1	19.4	20.9	6	3	-2	5.0	-4.5	3	12	2	17.9	18.9	11	1	-3	10.6	10.3	6	0	-4	39.6	41.0
3	1	-1	27.7	27.5	2	16	1	9.8	9.0	6	5	-2	12.7	-11.3	3	15	2	13.9	9.9	0	1	3	74.5	89.7	6	1	-4	5.6	-6.2
3	2	-1	19.2	17.9	2	17	1	11.4	14.1	6	6	-2	4.6	-4.5	3	16	2	8.7	8.3	0	2	3	3.8	2.6	6	2	-4	51.6	56.2
3	3	-1	45.3	37.4	3	1	1	57.4	51.3	6	8	-2	11.0	12.1	4	1	2	24.8	-24.4	0	3	3	63.9	67.2	6	3	-4	20.1	18.1
3	4	-1	44.2	41.2	3	2	1	9.2	7.7	6	10	-2	13.9	15.6	4	2	2	30.0	28.3	0	4	3	16.7	18.0	6	4	-4	29.8	29.1
3	5	-1	56.8	63.0	3	3	1	12.1	-13.4	7	0	-2	34.4	34.8	4	3	2	18.9	-20.7	0	5	3	26.8	21.6	6	5	-4	20.3	-21.1
3	6	-1	10.0	-8.0	3	4	1	16.8	16.8	7	1	-2	12.1	10.2	4	5	2	21.9	27.1	0	6	3	9.7	9.3	6	6	-4	13.9	13.4
3	7	-1	33.2	32.2	3	5	1	6.3	-7.1	7	2	-2	23.0	22.8	4	6	2	8.4	-9.9	0	7	3	26.5	22.0	6	7	-4	19.7	-18.4
3	9	-1	11.7	8.6	3	6	1	9.1	-9.9	7	3	-2	20.1	22.4	4	7	2	19.5	19.9	0	8	3	8.0	-4.9	6	8	-4	20.9	21.4
3	10	-1	10.6	14.0	3	7	1	26.1	27.4	7	4	-2	11.3	9.7	4	8	2	35.2	46.0	0	9	3	28.3	25.9	6	9	-4	17.2	-16.7
3	11	-1	22.4	25.1	3	8	1	15.7	-14.7	7	5	-2	14.5	-14.6	4	10	2	17.7	18.1	0	13	3	20.5	17.8	6	10	-4	28.6	26.7
3	12	-1	13.9	12.0	3	9	1	39.0	41.9	7	6	-2	9.2	9.8	4	11	2	20.2	-19.1	0	17	3	11.2	13.1	6	11	-4	9.3	11.0
3	13	-1	25.6	28.7	3	10	1	14.6	15.1	7	7	-2	11.5	-9.4	4	12	2	14.0	12.8	1	1	3	28.7	22.2	6	12	-4	13.1	12.3
3	15	-1	15.5	17.3	3	11	1	14.0	16.7	7	8	-2	36.6	33.7	5	1	2	17.4	16.4	1	2	3	23.8	24.5	7	0	-4	18.6	16.1
4	1	-1	64.3	55.6	3	13	1	10.6	12.4	7	9	-2	6.1	-5.0	5	2	2	23.1	25.9	1	3	3	36.3	33.4	7	1	-4	11.4	7.7
4	2	-1	2.6	-1.3	3	15	1	12.4	13.7	7	10	-2	16.8	15.1	5	3	2	14.9	14.9	1	5	3	40.6	45.0	7	2	-4	30.8	32.9
4	3	-1	33.2	33.5	4	1	1	8.4	-9.0	7	11	-2	12.0	10.7	5	4	2	11.7	12.3	1	7	3	35.6	37.1	7	3	-4	14.8	13.6
4	4	-1	4.8	1.8	4	2	1	21.1	19.0	7	13	-2	6.6	4.7	5	5	2	12.4	-11.6	1	13	3	27.3	25.1	7	4	-4	40.6	45.6
4	5	-1	44.0	42.8	4	3	1	24.1	22.8	8	0	-2	7.4	-7.0	5	8	2	40.8	42.4	1	15	3	19.7	19.9	7	5	-4	16.3	-16.6
4	6	-1	44.3	44.5	4	4	1	17.2	16.6	8	2	-2	24.3	21.6	5	9	2	9.4	-9.7	1	3	-3	47.4	39.7	7	6	-4	48.6	46.2
4	7	-1	44.9	45.5	4	5	1	42.0	47.6	8	3	-2	13.6	10.7	5	10	2	9.4	8.3	1	4	-3	23.2	-20.0	7	7	-4	27.7	-27.3
4	8	-1	30.0	31.7	4	7	1	15.8	15.3	8	4	-2	32.3	28.8	5	11	2	10.3	10.5	1	5	-3	31.0	25.3	7	8	-4	20.6	16.3
4	9	-1	37.6	36.8	4	8	1	9.6	7.2	8	5	-2	10.5	-12.1	6	2	2	15.9	14.8	1	6	-3	9.6	-7.8	7	10	-4	18.8	15.5
4	11	-1	21.5	21.0	4	9	1	11.1	9.6	8	6	-2	30.3	24.9	6	4	2	31.7	30.5	1	7	-3	43.6	40.7	7	11	-4	6.3	6.3
4	12	-1	15.5	-13.4	4	11	1	9.5	11.9	8	8	-2	13.1	13.4	6	6	2	17.7	15.7	1	8	-3	13.6	-12.8	7	12	-4	12.3	13.1
4	13	-1	19.8	15.9	4	13	1	20.5	24.4	8	10	-2	10.5	10.9	7	4	2	10.9	9.4	1	9	-3	38.7	39.3	7	14	-4	16.5	18.5
4	15	-1	13.7	14.3	4	15	1	8.2	11.1	8	11	-2	11.4	12.1	7	5	2	9.2	8.6	1	11	-3	20.4	19.2	8	1	-4	20.2	-22.8
5	1	-1	77.8	79.1	5	1	1	34.3	34.7	8	12	-2	12.4	14.0	7	7	2	12.9	14.8	1	12	-3	39.5	32.8	8	2	-4	13.5	13.7
5	2	-1	8.5	6.7	5	2	1	38.0	-40.1	9	0	-2	14.5	12.5	3	2	-3	38.0	-34.6	2	3	-3	0.2	0.1	8	3	-4	6.0	-5.1
5	3	-1	19.3	18.0	5	3	1	24.2	23.3	9	2	-2	15.9	15.5	3	3	-3	1.6	1.3	2	4	-3	53.1	-45.5	8	4	-4	36.5	34.4
5	4	-1	6.6	6.3	5	4	1	8.5	-8.8	9	3	-2	10.4	-8.4	3	4	-3	9.8	-7.0	2	5	-3	97.1	105.5	8	6	-4	22.7	21.1
5	6	-1	12.7	-12.6	5	5	1	17.3	17.7	9	4	-2	12.9	10.3	3	5	-3	71.2	77.8	2	6	-3	22.7	-21.8	8	9	-4	13.7	9.8
5	7	-1	25.2	24.6	5	6	1	25.2	32.2	9	5	-2	8.5	5.8	3	6	-3	3.2	1.9	2	7	-3	40.8	37.5	8	10	-4	15.3	14.0
5	8	-1	6.9	8.6	5	7	1	22.8	27.2	9	6	-2	7.3	8.3	3	7	-3	17.7	17.4	2	8	-3	8.8	-7.7	8	12	-4	17.4	17.0
5	9	-1	52.5	54.4	5	8	1	21.0	22.8	9	7	-2	12.5	11.7	3	8	-3	14.2	14.2	2	10	-3	5.5	6.5	8	14	-4	11.1	11.2
5	11	-1	18.0	17.3	5	9	1	16.3	17.3	9	8	-2	12.9	10.1	3	9	-3	23.7	26.7	2	11	-3	37.6	35.1	9	0	-4	15.9	14.0
6	1	-1	24.1	21.1	5	10	1	11.9	-11.1	9	10	-2	12.5	12.8	3	10	-3	12.5	-12.0	2	12	-3	27.7	23.3	9	1	-4	14.0	-11.0
6	2	-1	14.5	14.9	5	11	1	9.0	9.3	10	0	-2	20.3	20.1	3	11	-3	38.1	42.5	2	13	-3	27.2	22.3	9	2	-4	19.3	17.7

Table 3(*a*) (*cont.*)

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
9	3	-4	14.0	11.9	2	4	4	10.5	13.6	5	1	-5	12.9	13.1	0	4	5	8.8	6.2	4	2	-5	32.4	31.0	1	2	6	17.2	22.6
9	4	-4	13.5	-10.8	2	5	4	9.0	8.8	5	2	-5	10.3	-13.4	0	5	5	16.4	13.0	4	3	-5	5.3	3.7	1	3	6	12.5	-12.9
9	5	-4	10.7	9.1	2	6	4	14.8	20.6	5	3	-5	45.7	51.3	0	8	5	15.3	12.4	4	4	-5	50.6	51.8	1	6	6	14.3	13.3
9	8	-4	17.7	16.1	2	7	4	8.5	-5.8	5	4	-5	12.7	11.4	0	9	5	18.4	17.0	4	5	-5	58.1	58.3	1	7	6	12.9	15.7
9	10	-4	13.6	14.0	2	8	4	22.9	31.9	5	5	-5	56.7	64.3	0	11	5	24.7	21.4	4	6	-5	16.0	-10.6	1	1	-6	27.2	-29.4
10	1	-4	19.2	17.0	2	10	4	17.3	21.4	5	6	-5	20.8	-23.7	0	12	5	10.8	-6.8	4	7	-5	33.9	29.5	1	2	-6	15.9	21.3
10	2	-4	13.6	11.8	2	12	4	13.5	13.3	5	7	-5	10.8	11.8	1	1	5	43.4	49.8	4	9	-5	25.8	20.3	1	3	-6	10.6	-8.3
10	3	-4	16.3	15.7	2	13	4	11.0	8.7	5	8	-5	5.9	7.0	1	2	5	13.9	12.7	4	10	-5	23.3	20.2	1	4	-6	17.7	18.9
10	6	-4	9.5	10.8	2	3	-4	26.1	-23.8	5	9	-5	12.5	-11.2	1	3	5	30.3	31.3	4	11	-5	19.6	19.2	1	5	-6	16.5	-16.0
10	7	-4	7.0	-6.5	2	4	-4	45.3	38.8	5	10	-5	8.7	7.9	1	4	5	17.6	20.5	4	12	-5	12.8	9.4	1	6	-6	15.5	14.6
10	8	-4	8.5	8.5	2	5	-4	27.7	-29.8	5	11	-5	18.9	14.9	1	5	5	14.6	19.1	4	15	-5	16.1	13.5	1	7	-6	7.2	8.0
11	2	-4	13.2	11.7	2	6	-4	31.7	29.4	5	13	-5	18.6	20.2	1	6	5	17.1	-20.6	6	0	-6	29.2	25.3	1	8	-6	24.2	29.5
11	4	-4	18.5	19.2	2	7	-4	35.7	-29.4	6	1	-5	42.7	44.8	1	7	5	19.7	28.0	6	1	-6	10.9	-7.6	1	9	-6	10.8	-13.2
11	6	-4	10.0	12.7	2	8	-4	56.0	58.7	6	2	-5	25.6	-23.4	1	8	5	17.8	-26.2	6	2	-6	26.3	29.4	1	10	-6	24.3	21.0
0	1	4	8.5	7.8	2	9	-4	18.2	-17.7	6	3	-5	36.6	38.8	1	9	5	15.0	20.1	6	3	-6	22.1	18.1	2	2	6	13.9	14.5
0	2	4	36.7	37.3	2	10	-4	35.2	35.5	6	4	-5	15.0	-14.1	1	2	-5	13.4	13.6	6	4	-6	52.2	60.8	2	3	6	20.8	-19.9
0	3	4	33.4	-34.1	2	11	-4	9.4	5.5	6	5	-5	27.2	27.7	1	3	-5	32.3	32.9	6	6	-6	13.3	40.0	2	4	6	18.4	19.2
0	4	4	53.0	61.8	2	12	-4	29.4	25.5	6	7	-5	20.1	22.2	1	4	-5	18.8	-18.0	6	7	-6	26.0	-25.6	2	8	6	19.9	21.8
0	5	4	18.8	15.0	2	13	-4	13.6	-11.3	6	8	-5	2.8	-3.6	1	5	-5	42.7	41.7	6	12	-6	22.2	18.7	2	3	-6	24.0	27.7
0	6	4	14.5	16.0	2	14	-4	8.7	10.4	6	10	-5	17.9	-15.2	1	6	-5	25.8	-24.9	7	0	-6	8.4	-9.8	2	4	-6	21.7	22.5
0	7	4	6.5	-5.7	2	15	-4	11.8	-10.5	6	11	-5	20.5	18.9	1	7	-5	28.6	26.4	7	1	-6	34.8	-29.6	2	5	-6	11.1	-10.4
0	10	4	6.0	-4.2	2	16	-4	14.7	13.8	6	12	-5	11.8	-11.5	1	8	-5	23.8	-25.7	7	2	-6	16.4	15.7	2	6	-6	35.6	38.4
0	11	4	12.7	-11.1	3	1	4	14.9	15.1	6	13	-5	14.1	15.4	1	9	-5	10.9	9.2	7	3	-6	7.7	-8.1	2	7	-6	13.9	-10.9
0	12	4	19.9	18.5	3	3	4	10.0	8.1	7	1	-5	30.6	29.8	1	10	-5	16.2	-13.0	7	4	-6	33.9	34.0	2	8	-6	24.1	29.9
0	14	4	17.6	20.1	3	4	4	18.9	20.4	7	2	-5	10.2	7.4	1	13	-5	28.1	24.6	7	6	-6	31.6	34.8	2	10	-6	29.3	21.8
1	1	4	22.9	-25.1	3	6	4	12.0	9.3	7	3	-5	21.9	22.4	1	15	-5	12.7	12.4	7	7	-6	14.4	13.0	2	11	-6	21.3	17.1
1	2	4	38.6	44.1	3	7	4	9.2	-8.3	7	5	-5	26.0	24.1	2	1	5	9.3	-10.9	7	10	-6	17.7	15.8	2	12	-6	30.6	25.5
1	3	4	20.2	-20.0	3	8	4	13.0	10.5	7	6	-5	15.9	-13.7	2	3	5	47.1	57.8	7	12	-6	22.7	20.0	2	14	-6	20.7	18.2
1	4	4	62.6	74.2	3	9	4	10.7	8.4	7	7	-5	29.8	28.2	2	5	5	34.6	43.2	8	0	-6	21.8	20.5	3	3	-6	15.5	13.4
1	5	4	7.6	4.6	3	12	4	17.0	19.2	7	8	-5	8.9	-9.1	2	3	-5	48.1	58.2	8	1	-6	12.0	-9.1	3	4	-6	56.0	69.5
1	6	4	12.2	13.7	3	14	4	12.5	14.9	7	9	-5	28.2	27.2	2	4	-5	47.4	-55.0	8	2	-6	13.6	12.2	3	6	-6	31.8	28.8
1	7	4	7.9	5.8	3	3	-4	57.5	62.9	7	11	-5	18.2	17.4	2	5	-5	44.2	47.8	8	3	-6	10.5	-8.6	3	7	-6	22.7	27.8
1	8	4	26.8	27.1	3	4	-4	60.4	59.6	7	12	-5	1.7	1.2	2	6	-5	9.2	-9.3	8	4	-6	3.9	-2.3	3	8	-6	17.5	15.2
1	9	4	10.9	-7.9	3	5	-4	28.5	-26.8	8	2	-5	10.9	10.7	2	7	-5	20.6	21.1	8	6	-6	17.9	19.0	3	12	-6	37.1	31.7
1	10	4	18.3	14.7	3	6	-4	51.8	52.1	8	3	-5	23.3	23.1	2	8	-5	21.5	18.8	8	8	-6	15.3	11.9	3	14	-6	34.5	29.6
1	11	4	19.8	-20.0	3	7	-4	9.0	-7.3	8	4	-5	7.1	6.4	2	9	-5	21.3	15.7	8	10	-6	29.4	25.5	4	3	-6	24.0	-26.0
1	14	4	9.7	10.0	3	8	-4	16.1	17.3	8	5	-5	20.5	19.7	2	10	-5	15.6	-16.3	8	12	-6	12.4	12.8	4	4	-6	35.1	34.4
1	15	4	9.3	8.2	3	9	-4	14.4	-13.5	8	6	-5	14.4	-12.4	2	11	-5	20.5	19.2	9	0	-6	25.3	22.2	4	5	-6	8.1	-8.4
1	16	4	9.8	10.5	3	10	-4	31.6	27.7	8	7	-5	17.5	16.9	2	13	-5	33.6	28.3	9	2	-6	15.5	14.8	4	6	-6	20.1	18.8
1	2	-4	28.7	24.2	3	11	-4	24.5	20.9	8	9	-5	12.5	12.3	2	14	-5	12.4	-11.8	9	3	-6	22.0	19.4	4	7	-6	55.7	53.0
1	3	-4	11.0	-10.2	3	12	-4	39.5	34.3	8	10	-5	10.4	9.2	2	15	-5	15.7	14.3	9	4	-6	15.7	14.0	4	9	-6	26.6	-22.7
1	4	-4	44.2	43.4	3	14	-4	26.6	22.0	8	11	-5	13.8	14.2	3	3	-5	32.0	32.7	9	6	-6	19.6	18.0	4	10	-6	25.2	20.3
1	5	-4	18.6	-18.8	3	15	-4	12.4	-10.8	8	12	-5	7.6	8.1	3	4	-5	8.5	7.8	9	7	-6	14.4	-12.5	4	11	-6	18.5	-12.4
1	6	-4	34.8	31.8	3	16	-4	12.6	12.8	8	13	-5	13.3	15.1	3	5	-5	43.4	35.9	10	0	-6	8.2	-9.0	5	2	-6	37.7	43.6
1	7	-4	8.6	-9.2	4	1	4	7.3	-4.9	9	1	-5	6.7	-4.8	3	6	-5	42.1	37.6	10	2	-6	21.2	26.3	5	3	-6	5.0	2.0
1	8	-4	21.3	20.3	4	3	4	7.3	-6.5	9	3	-5	27.6	25.7	3	7	-5	18.7	16.0	10	4	-6	25.2	30.2	5	4	-6	15.2	13.2
1	9	-4	17.2	-13.4	4	4	4	25.0	28.7	9	4	-5	7.6	5.6	3	8	-5	7.6	5.3	0	1	6	18.4	20.9	5	5	-6	21.7	-19.1
1	10	-4	15.3	14.5	4	7	4	20.5	21.3	9	5	-5	13.4	13.9	3	9	-5	33.4	30.9	0	2	6	11.4	14.0	5	6	-6	35.8	30.9
1	11	-4	13.8	-14.8	4	12	4	11.4	11.9	9	6	-5	9.7	4.8	3	10	-5	11.6	-9.7	0	3	6	23.7	26.4	5	7	-6	15.2	-13.6
1	12	-4	19.4	19.2	5	2	4	13.0	16.0	10	1	-5	12.7	14.2	3	11	-5	44.6	42.8	0	4	6	29.1	36.5	5	9	-6	17.0	-12.0
2	1	4	19.6	-20.1	5	7	4	11.3	14.2	10	3	-5	12.4	17.9	3	12	-5	6.9	4.0	0	9	6	13.5	12.4	5	10	-6	35.0	28.8
2	2	4	25.1	27.2	5	8	4	11.7	13.7	0	1	5	34.1	39.6	3	13	-5	9.8	10.8	0	10	6	8.3	-6.3					
2	3	4	6.7	-8.7	5	10	4	10.1	12.7	0	2	5	27.6	-29.6	3	15	-5	11.8	12.3	0	11	6	15.0	16.5					

observed and calculated structure factors are given in Table 3, together with an agreement analysis.

Table 3(*b*). *Agreement analysis*

F_{obs}	R	Sin θ	R
0–10	25·8%	0·00–0·10	—
10–20	14·7	0·10–0·20	8·9%
20–30	10·6	0·20–0·30	12·0
30–40	10·7	0·30–0·40	11·5
40–50	11·4	0·40–0·50	11·9
50–60	12·7	0·50–0·60	14·7
60–70	12·4	0·60–0·70	15·8
70–80	7·6	0·70–0·80	12·8
80–90	10·0	0·80–0·90	12·9
90–100	6·3	0·90–1·00	20·7
100–110	17·3		

Discussion

A preliminary account of this work appeared in a recent communication (Brown, Lee & Melsom, 1968*b*).

The environment of each copper atom is shown in Fig. 1, bond lengths and angles are quoted in Tables 4 and 5, and the closest non-bonded distances are given in Tables 6 and 7. The equations of the planes of the molecule and distance of atoms from these planes are given in Table 8.

Table 4. *Bond lengths and their standard deviations*

	Bond length	σ
Cu —O(1)	1·93 Å	0·007 Å
Cu —O(11)	1·92	0·009
O(1)—N(1)	1·34	0·012
O(11)–N(11)	1·33	0·014
N(1)—C(1)	1·34	0·013
N(1)—C(5)	1·35	0·010
N(11)—C(11)	1·35	0·015
N(11)–C(15)	1·32	0·013
C(1)—C(2)	1·38	0·018
C(2)—C(3)	1·36	0·014
C(3)—C(4)	1·38	0·017
C(4)—C(5)	1·40	0·017
C(11)–C(12)	1·37	0·022
C(12)–C(13)	1·36	0·017
C(13)–C(14)	1·37	0·020
C(14)–C(15)	1·40	0·022
Cl——O(21)	1·40	0·013
Cl——O(22)	1·42	0·012
Cl——O(23)	1·38	0·015
Cl——O(24)	1·39	0·008

Table 5. *Bond angles and their standard deviations*

	Angle	σ
O(1)—Cu —O(11)	89·2°	0·4°
Cu —O(1)—N(1)	116·7	0·7
Cu —O(11)–N(11)	118·7	0·6
O(1)—N(1)—C(1)	119·0	0·7
O(1)—N(1)—C(5)	117·3	0·8
C(1)—N(1)—C(5)	123·7	0·9
N(1)—C(1)—C(2)	119·4	0·8
C(1)—C(2)—C(3)	118·9	1·1
C(2)—C(3)—C(4)	121·6	1·3
C(3)—C(4)—C(5)	118·8	0·9
C(4)—C(5)—N(1)	117·5	0·9
O(11)–N(11)–C(11)	117·2	0·8
O(11)–N(11)–C(15)	118·5	0·9
C(11)–N(11)–C(15)	124·0	1·1
N(11)–C(11)–C(12)	118·3	1·0
C(11)–C(12)–C(13)	120·3	1·3
C(12)–C(13)–C(14)	119·2	1·5
C(13)–C(14)–C(15)	120·2	1·1
C(14)–C(15)–N(11)	117·7	1·1
O(21)–Cl——O(22)	106·2	0·9
O(21)–Cl——O(23)	114·6	1·0
O(21)–Cl——O(24)	110·2	0·7
O(22)–Cl——O(23)	109·6	0·7
O(22)–Cl——O(24)	110·6	0·7
O(23)–Cl——O(24)	105·8	0·7

Table 6. *Intramolecular distances less then* 3 Å *which are not directly bonded*

Cu —N(1)	2·80 Å
Cu —N(11)	2·81
O(1)—O(11)	2·71
O(1)—C(1)	2·31
O(1)—C(5)	2·29
O(11)–N(1)	2·63
O(11)–C(5)	2·95
O(11)–C(11)	2·28
O(11)–C(15)	2·28
O(21)–O(22)	2·26
O(21)–O(23)	2·34
O(21)–O(24)	2·29
O(22)–O(23)	2·29
O(22)–O(24)	2·31

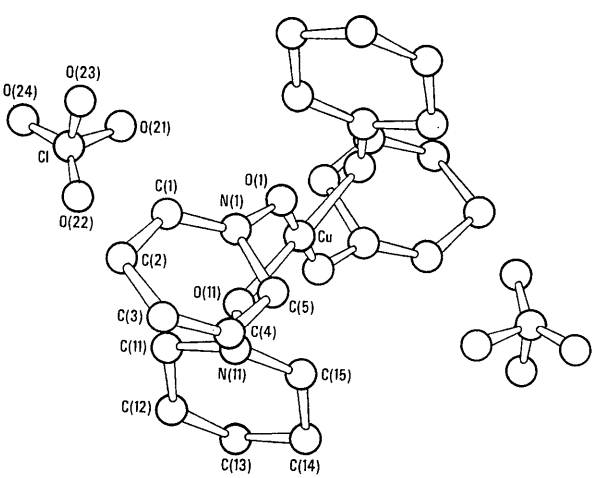

Fig. 1. Diagram of the structure showing the environment of each copper atom.

Table 6 (*cont.*)

O(23)–O(24)	2·21
N(1)—C(2)	2·35
N(1)—C(3)	2·70
N(1)—C(4)	2·36
N(11)–C(14)	2·33
N(11)–C(12)	2·33
N(11)–C(13)	2·70
C(1)—C(3)	2·36
C(1)—C(4)	2·75
C(1)—C(5)	2·38
C(2)—C(4)	2·39
C(2)—C(5)	2·77
C(3)—C(5)	2·40
C(11)–C(15)	2·36
C(11)–C(14)	2·73
C(11)–C(13)	2·37
C(15)–C(12)	2·74
C(15)–C(13)	2·40
C(14)–C(12)	2·36

Table 7. *Intermolecular distances less than* 3·5 Å

	Distance	Symmetry operation applied to second atom
C(4)—O(24)	3·36 Å	(1)
C(5)—O(24)	3·16	(1)
C(3)—O(24)	3·44	(2)
N(1)—O(22)	3·39	(3)
C(1)—O(22)	3·31	(3)
O(22)–N(1)	3·39	(4)
O(22)–C(1)	3·31	(4)
O(24)–C(3)	3·44	(5)
C(2)—O(21)	3·39	(6)
C(3)—O(21)	3·36	(6)
O(24)–C(4)	3·36	(7)
O(24)–C(5)	3·16	(7)
C(12)–O(21)	3·45	(8)
C(13)–O(24)	3·38	(8)
O(21)–C(12)	3·45	(8)
O(24)–C(13)	3·38	(8)
O(21)–C(2)	3·39	(9)
O(21)–C(3)	3·36	(9)

Key to symmetry operations

(1) $1+x, \quad y, 1+z$
(2) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$
(3) $x, \frac{1}{2}-y, \frac{1}{2}+z$
(4) $x, \frac{1}{2}-y, z-\frac{1}{2}$
(5) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$
(6) $-x, \frac{1}{2}+y, -z-\frac{1}{2}$
(7) $x-1, \quad y, z-1$
(8) $-x, \quad -y, -z-1$
(9) $-x, y-\frac{1}{2}, -z-\frac{1}{2}$

The structure is centrosymmetric about the copper atoms. Oxygen atoms from four pyridine oxide molecules are in a square planar arrangement around the copper with Cu–O distances of 1·93 Å and 1·92 Å. These distances agree with the normal single bond values of 1·92–1·95 Å (Pauling, 1960) and compare with Cu–O distances of 1·89 and 1·90 Å in bis(*N*-t-butylsalicylaldiminato)copper(II) (Cheesman, Hall & Waters, 1966), 1·90 Å in bis(salicylaldiminato)copper-(II) (Baker, Hall & Waters, 1966) and 1·91 and 1·94 Å in copper ethyl acetoacetate (Barclay & Cooper, 1965).

The four pyridine oxide molecules form a swastika configuration around the copper, with the nitrogen atoms lying approximately in the copper-oxygen plane, but the benzenoid rings approximately perpendicular to this plane.

Bond lengths and angles in the pyridine rings appear to be normal when compared with the C–N distances of 1·34 Å and the C–C distances of 1·39–1·40 Å in pyridine itself (Bak, Hansen-Nygaard & Rastrup-Andersen, 1958), and C–N distances of 1·34 Å and C–C distances of 1·36–1·41 Å in di-μ-(pyridine oxide)-bis-(dichloro copper(II)) (Sager, Williams & Watson, 1967).

The N–O distances of 1·33 and 1·32 Å are slightly shorter than the value of 1·35 Å in di-μ-(pyridine oxide) bis(dichloro copper(II)) (Sager, Williams & Watson, 1967), 1·37 Å in pyridine *N*-oxide hydrochloride (Tsoucaris, 1961) and 1·39 Å in $(CH_3)_3NO$ (Caron, Palenik, Goldish & Donohue, 1964). Though this might indicate some double bond character in the present complex, the orbitals are not favourably orientated for π overlap.

The angles Cu–O(1)–N(1) and Cu–O(11)–N(11) (117° and 119° respectively) are greater than tetrahedral, presumably because repulsion between the bulky pyridine rings exceeds that between the lone pairs of electrons on the oxygen atoms.

Temperature factors of atoms in the pyridine rings increase from N(1) to O(3) and from N(11) to C(13), suggesting a certain amount of vibrational movement of the rings pivoted about the oxygen atoms.

The ClO_4^- ions occupy approximately octahedral positions relative to the square plane around the copper but the closest approaches between it and the copper are 3·38 Å from O(23) and 3·57 Å from O(21). This distance is too long to allow any bonding, and in any case, no oxygen atom is favourably situated geometrically for coordination, *i.e.* near the tetragonal axis. The perchlorate ion shows a fairly close approximation to tetrahedral symmetry with Cl–O distances in the range 1·38 Å to 1·42 Å (σ=0·015 Å) and bond angles in the range 106–115° (σ=1°). Three-dimensional difference Fourier sections have provided no alternative orientations of the ClO_4^- ion. The high temperature factors indicate considerable thermal motion. Analysis of the anisotopic temperature factors for the ClO_4^- group shows that O(23) and O(24) vibrate most in approximately the **b*** direction, whilst O(21) is vibrating most in the a^*c^* plane. The atoms O(22) and Cl show considerably less anisotropy. The results are interpreted in terms of distorting modes of vibration within the ion. Librational movement is thought to be confined to small angular oscillations of the ion.

It is clear from the above arguments that in this complex the perchlorate group is ionic, in contrast to the structures of Cu(ethylenediamine)$_2$(ClO$_4$)$_2$ (Pajunen, 1967) and Cu(ethylenediamine)$_2$(BF$_4$)$_2$ (Brown, Lee & Melsom, 1968(*a*)], where the polyanions are involved in weak coordination with the metal. This interaction has been termed 'semi-coordination', (Brown, Lee, Melsom, Hathaway, Procter & Tomlinson, 1967) and (Procter, Hathaway & Nichols, 1968), and causes considerable distortion of the tetrahedral symmetry of the polyanion. The slight distortions observed for Cu(PyO)$_4$(ClO$_4$)$_2$ could result from unsymmetrical interactions around the polyanion.

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Table 8. *The planes of the molecule*

Equations to planes

Atoms in plane	Equation of plane
N(1), C(1), C(2), C(3), C(4), C(5)	$-6{\cdot}00x+9{\cdot}08y+7{\cdot}79z=1{\cdot}80$
N(11), C(11), C(12), C(13), C(14), C(15)	$6{\cdot}62x+9{\cdot}30y-0{\cdot}95z=1{\cdot}53$
Cu, O(1), O(11)	$2{\cdot}15x-5{\cdot}59y+6{\cdot}94z=0{\cdot}00$

x, y and z refer to fractional coordinates of the unit cell axes a, b and c.

Distance of atoms from planes

	Distance from first plane		Distance from second plane
N(1)	0·00 Å	N(11)	0·01 Å
C(1)	0·00	C(11)	−0·20
C(2)	−0·11	C(12)	0·00
C(3)	0·14	C(13)	0·00
C(4)	−0·01	C(14)	0·02
C(5)	0·00	C(15)	0·02

Angles between planes

Ring N(1)···C(5) to ring N(11)···C(15)	=82·2°
Ring N(1)···C(5) to square plane Cu, O(1), O(11)	=88·7
Ring N(11)···C(15) to square plane Cu, O(1), O(11)	=79·1

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The Structure of an Organo-aluminum Ketimine Derivative $[Ph_2Al.N{=}CPh.C_6H_4Br]_2.2C_6H_6$

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Crystals of $[Ph_2Al.N{=}CPh.C_6H_4Br]_2.2C_6H_6$ are triclinic, space group $P\bar{1}$, with $Z=1$. The structure was solved by the heavy atom method and refined by least-squares to $R=10{\cdot}7\%$. The molecule contains a four-membered ring of alternating Al and N atoms, with Al–N = 1·92 Å, and N=C = 1·28 Å. The benzene molecules undergo large amplitude vibrations.

Introduction

Aluminum alkyls and aryls form adducts with nitriles, and on heating the adducts rearrange to give ketimine derivatives. Gibson & Hughes (1964) described the compound from triphenylaluminum and benzonitrile, and reported a degree of association in solution of 1·2–1·4. Wade and co-workers (Lloyd & Wade, 1965; Jennings, Lloyd & Wade, 1965; Wade & Wyatt, 1967) have obtained similar compounds by the same route, and also by elimination of hydrocarbon from ketimine adducts, reporting that the compounds are dimeric.

The present work was undertaken in order to establish the degree of association and molecular structure of these compounds. Shearer & Willis (reported in Coates & Wade, 1967) have determined the structure of a related compound, $[Bu^tMeC{=}N.AlMe_2]_2$.

Mr G. K. J. Gibson kindly supplied a sample of the rearrangement product from *p*-bromobenzonitrile and triphenylaluminum. The material was recrystallized from benzene, giving a solvate containing two molecules of benzene to each dimeric molecule of the aluminum compound. Crystals were sealed in capillaries for X-ray study.

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Crystal data

$Al_2N_2C_{50}H_{38}Br_2.2C_6H_6$, $M=1036{\cdot}9$, triclinic, $a=9{\cdot}88$, $b=14{\cdot}56$, $c=9{\cdot}85$ Å, $\alpha=107{\cdot}5°$, $\beta=95{\cdot}1°$, $\gamma=99{\cdot}0°$, $V=1321$ Å^3.
Space group $P\bar{1}$, $Z=1$, $D_x=1{\cdot}303$.

Structure determination

The cell dimensions were measured from zero layer precession photographs. Intensities were estimated visually from Cu $K\alpha$ Weissenberg photographs of reciprocal-lattice layers $hk0$ to $hk7$, giving 1854 independent structure amplitudes. The bromine and aluminum positions were obtained from a three-dimensional Patterson synthesis, and were consistent with the centrosymmetric space group. An electron density synthesis based on the Br and Al positions revealed the remaining non-hydrogen atoms of the molecule, but the benzene molecule appeared only as an annulus of electron density with no well-resolved peaks. After least-squares refinement of atomic positions and isotropic temperature factors a difference synthesis was