Acta Cryst. (1964). **17**, 633

The Crystal Structure of Monopyridinecopper(II) Acetate

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(*Received* 16 *January* 1963)

The structure of the orthorhombic monopyridinecopper(II) acetate has been determined by three dimensional Patterson, Fourier and (F_o-F_c)-syntheses and by least-squares refinement. The copper atoms are bridged in pairs by four acetate groups to form of a binuclear molecule, $Cu_2(CH_3.CO_2)_4.2C_5H_5N$, similar to those found in the monoclinic form and in copper acetate monohydrate. In all three structures, the copper atom is 0·22 Å out of the plane of the four oxygen atoms. The distorted octahedral coordination around Cu is completed by the nitrogen atom of the pyridine molecule (Cu–N: 2·19 Å) and by the second copper atom (Cu–Cu: 2·645 Å). The binuclear molecule as a whole possesses a centre of symmetry.

Introduction

Barclay & Kennard (1961) have reported the structure of the monoclinic form of monopyridinecopper(II) acetate, $Cu_2(CH_3.CO_2)_4.2C_5H_5N$. Their paper is interesting in connection with the earlier published crystal structure of a compound of the same composition but differing from the monoclinic form by symmetry both of the structure and of the molecules (Hanic, Štempelová & Hanicová, 1961). This structure was described as an orthorhombic form. The model of the structure was built up from the two-dimensional data, but the accuracy of the determination of the atomic positions was limited owing to the overlap of atoms. Therefore after collecting the three-dimensional data, we carried out the three-dimensional refinement.

Crystal data

Monopyridinecopper(II) acetate, in the form of dark-green well developed crystals, was supplied by Dr J. Krätsmar-Šmogrovič. Cell dimensions, symmetry and space group were determined from oscillation, precession and Weissenberg photographs with Cu *K* radiation:

$$a=13·08\pm0·02,\quad b=8·59\pm0·01,\quad c=19·57\pm0·04\ \text{Å}$$

Space group *Pbca*, $Z=4$

The integrated intensities of 686 independent reflexions were determined by photometric measurements of the diffraction spots on Weissenberg photographs. In order to establish the characteristics of the film a selected interval of the reciprocal lattice plane including some intensive spots was simultaneously exposed on three films, the exposure time being increased regularly. The integral intensity of the selected spot was plotted against the time and this intensity-time dependence was used for correction of all intensities on the Weissenberg photographs. The absorption was negligible. The correction of the intensities for the Lorenz and polarization factors was carried out in the usual way.

Determination of the structure

The approximate model of the structure was built up from two-dimensional Patterson and electron-density projections (Hanic, Štempelová & Hanicová, 1961). First, the refinement of the atomic coordinates was carried out by means of the three-dimensional Fourier- and (F_o-F_c)-syntheses (Hanic, Štempelová & Hanicová, 1961) with the phases of the structure factors calculated from the two-dimensional data. The structure factors and the three-dimensional electron density syntheses were calculated on the electronic computer BESK using the programs SNUSKMUS and SUPERMUS III (Åsbrink, Blomqvist & Westman, 1961). The parameters obtained for the atoms are given in Table 1.

Further refinement of the parameters of the structure has been carried out by a least-squares program on the computer FACIT EDB (Åsbrink & Brändén,

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Table 1. *Preliminary atomic parameters*

Atom	*x/a*	*y/b*	*z/c*
Cu	0·033	0·075	0·055
O(1)	0·392	0·072	0·433
O(2)	0·166	0·032	0·009
O(3)	0·449	0·363	0·093
O(4)	0·491	0·257	0·499
N	0·090	0·197	0·141
C(1)	0·027	0·232	0·196
C(2)	0·072	0·311	0·250
C(3)	0·165	0·372	0·249
C(4)	0·226	0·341	0·195
C(5)	0·179	0·250	0·145
C(6)	0·180	0·481	0·453
C(7)	0·206	0·038	0·437
C(8)	0·467	0·249	0·057
C(9)	0·436	0·104	0·092

1963). We started with the set of atomic positions as given above. The scattering factors for the atoms were taken from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955). The coordinates and the individual isotropic temperature factor of each atom were refined together with scale factors for the different equi-inclination Weissenberg photographs. The equation proposed by Hughes (1941) was applied to express the weight function. For the computing of shift u_K of the parameters during the Kth cycle of refinements the Curtis schema was taken:

$$u_K = \alpha v_K + \beta u_{K-1}$$

where v_K is the full shift computed in the Kth cycle, u_{K-1} is the shift of the parameter in the $(K-1)$th cycle. The constants α and β are equal to 0·8 and 0·2 respectively. During seven cycles of refinement the reliability index $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$ decreased from 0·182 to 0·098. The last two cycles were calculated without the contribution of the copper. Including all 276 zero reflexions the final R index after a further five cycles was found to be 0·129.

The final coordinates of the atoms and their individual isotropic temperature factors together with their standard diviations are listed in Table 2.

In Table 3 the final set of observed and calculated structure factors for orthorhombic $Cu_2(CH_3.CO_2)_4.2C_5H_5N$ is shown.

Description of the structure

The structure of the orthorhombic form of $Cu_2(CH_3.CO_2)_4.2C_5H_5N$ (1) is similar to that of the monoclinic form (2) (Barclay & Kennard, 1961) and to the structure of $Cu_2(CH_3.CO_2)_4.2H_2O$ (3) (van Niekerk & Schoening, 1953). All three complexes are built up of binuclear molecules, but their symmetry is different. The binuclear molecules (1) and (3) possess a centre of symmetry. The symmetry of binuclear units of (2) is related to a twofold axis of rotation. Consequently, the two pyridine molecules of the binuclear unit (1) lie in the same plane, in contradistinction to the monoclinic form (2), where the planes of the two pyridine rings are inclined to each other at an angle of 60°.

The similarity of the three binuclear complexes studied follows from tha data in Table 4. The intermolecular distances indicate that both structures (2) and (1) are built up of molecules. Only van der Waals forces act between the pyridine rings, oxygen atoms and methyl groups of neighbouring molecules. The nearest contacts between atoms of neighbouring pyridine rings in (1) are 3·70–4·14Å. Carbon atoms of methyl groups show the nearest distances to atoms of another molecule, 3·44–3·59 Å. The corresponding distances in the structure (2) are 4·48–4·72 Å (Barclay & Kennard, 1961). In the hydrate complex each 'molecule' is linked, apart from van der Waals forces, by eight hydrogen bonds (2·82–2·89 Å) to four neighbouring molecules at different levels (van Niekerk & Schoening, 1953).

The most characteristic interatomic distance for all three binuclear complexes is the Cu–Cu contact. This distance remains nearly constant in all the structures (1), (2) and (3) and its value varies from 2·630 to 2·645 Å.

The nearest neighbours of the copper atom are the four oxygen atoms from the four acetate groups. The mean Cu–O distance 1·96 Å in the structure (1) is a little shorter than those found in the structures (2) and (3): 1·98 and 1·97 Å respectively. The arrangement of the four oxygen atoms around the Cu atom is planar, but the Cu atom is displaced from the plane of these

Table 2. *Final atomic parameters, isotropic temperature factors, and standard deviations*

Atom	*x/a*	*y/b*	*z/c*	*B*
Cu	0·0331 ± 0·0002	0·0745 ± 0·0003	0·0549 ± 0·0001	+ 0·43 ± 0·03
O(1)	0·3890 ± 0·0009	0·0645 ± 0·0016	0·4146 ± 0·0006	+ 1·39 ± 0·23
O(2)	0·1660 ± 0·0008	0·0558 ± 0·0014	0·0088 ± 0·0006	+ 0·60 ± 0·20
O(3)	0·4437 ± 0·0009	0·3662 ± 0·0015	0·0911 ± 0·0006	+ 0·89 ± 0·23
O(4)	0·4986 ± 0·0008	0·2548 ± 0·0018	0·4991 ± 0·0007	+ 0·82 ± 0·21
N	0·0907 ± 0·0009	0·2021 ± 0·0016	0·1435 ± 0·0006	+ 0·06 ± 0·22
C(1)	0·0321 ± 0·0016	0·2304 ± 0·0028	0·1964 ± 0·0010	+ 2·21 ± 0·40
C(2)	0·0687 ± 0·0017	0·3115 ± 0·0027	0·2532 ± 0·0012	+ 2·61 ± 0·45
C(3)	0·1637 ± 0·0018	0·3697 ± 0·0028	0·2524 ± 0·0013	+ 2·70 ± 0·45
C(4)	0·2297 ± 0·0017	0·3416 ± 0·0027	0·1989 ± 0·0010	+ 2·26 ± 0·43
C(5)	0·1869 ± 0·0013	0·2587 ± 0·0026	0·1427 ± 0·0009	+ 1·21 ± 0·33
C(6)	0·1778 ± 0·0014	0·5063 ± 0·0024	0·4523 ± 0·0009	+ 1·04 ± 0·33
C(7)	0·2103 ± 0·0015	0·0187 ± 0·0025	0·4269 ± 0·0009	+ 1·44 ± 0·37
C(8)	0·4673 ± 0·0013	0·2451 ± 0·0022	0·0594 ± 0·0008	+ 0·63 ± 0·28
C(9)	0·4412 ± 0·0015	0·0901 ± 0·0026	0·0932 ± 0·0010	+ 1·99 ± 0·39

Table 3. *Observed and calculated structure factors*

0	0	2	319.2	315.2
0	0	4	77.3	89,9
0	0	6	146.8	-173.4
0	0	8	121.5	-130.2
0	0	10	111.1	-106.8
0	0	12	42.5	31.3
0	0	14	91.0	92.0
0	0	16	133.7	130.6
0	0	18	83.2	69.2
0	0	20	168.7	185.2
0	0	22	74.2	75.9
1	0	2	86.8	-79.0
1	0	4	100.7	85.6
1	0	6	19.9	2.8
1	0	8	63.8	-52.4
1	0	10	51.1	-39.9
1	0	12	-	-0.4
1	0	14	-	25.7
1	0	16	-	-29.6
1	0	18	-	12.9
1	0	22	70,2	-65.6
2	0	0	225.4	300.4
2	0	2	168.8	201.9
2	0	4	45,4	24.3
2	0	6	118.1	-117.2
2	0	8	130.9	-124.0
2	0	10	95.2	-84.4
2	0	12	40.8	-22.0
2	0	14	-	17.8
2	0	16	88.0	82.5
2	0	18	105.3	117.7
2	0	20	118.0	114.3
2	0	22	46.1	53.5
3	0	2	149.5	-171.4
3	0	4	157.0	-178.6
3	0	6	75.0	-69.0
3	0	8	49.2	-38.7
3	0	10	60.9	45.8
3	0	12	104.4	90.0
3	0	14	88.4	72.2
3	0	16	48.0	42.6
3	0	18	-	21.1
3	0	20	-	-31.5
3	0	22	79.3	-80.6
4	0	0	149.7	149.8
4	0	2	115.2	116,6
4	0	4	90.9	75.7
4	0	6	-	-8.4
4	0	8	146.5	-147.9
4	0	10	45.8	-44.6
4	0	12	81.1	-71.9
4	0	14	48.8	27.6
4	0	16	103.5	99.8
4	0	18	118.0	110.9
4	0	20	87.7	73.4
5	0	2	188.8	-222.0
5	0	4	179.0	-193.0
5	0	6	159.9	-157.9
5	0	8	33.7	22.9
5	0	10	91.9	88.3
5	0	12	93.3	89.2
5	0	14	128.0	127.0
5	0	16	94.1	78.0
5	0	18	60.3	54.9
5	0	20	-	19.9
5	0	22	96.9	-96.9
6	0	0	111.0	91.1
6	0	2	134.7	130.8
6	0	4	151.6	140.1
6	0	6	83.5	68.2
6	0	8	-	5.2
6	0	10	43.7	-37.7
6	0	12	26.6	-15.0
6	0	14	20.7	24.6
6	0	16	96.3	89.0
6	0	18	98.2	97.7
6	0	20	39.0	49.2
7	0	2	156.6	-164.8
7	0	4	188.1	-201.9
7	0	6	128.1	-130.5
7	0	8	-	5.8
7	0	10	79.4	72.1
7	0	12	93.2	79.4
7	0	14	77.9	83.3
7	0	16	59.5	46.0
7	0	18	-	2 .5
7	0	20	77.2	-64.4
7	0	22	90.8	-108.5
8	0	0	101.8	-84.6
8	0	2	-	2.5
8	0	4	37.2	28.4
8	0	6	54.7	59.1
8	0	8	-	5.0
8	0	10	63.9	56.3
9	0	2	92.7	-84.6
9	0	4	156.3	-159.6
9	0	6	157.1	-184.6
9	0	8	-	19.6
9	0	10	74.8	64.0
9	0	12	80.4	82.7
9	0	14	141.1	140.7
9	0	16	102.3	95.8
9	0	18	-	9.7
9	0	20	73.4	-81.7
10	0	0	48.8	-58.0
10	0	2	41.6	-28.3
10	0	4	42.5	-31.0
10	0	6	54.5	72.8
10	0	8	75.7	72.3
10	0	10	-	33.2
10	0	12	-	30.4
10	0	14	54.4	46,3
11	0	2	-	30.3
11	0	4	52.5	-49.5
11	0	6	89.3	-96.9
11	0	8	63.6	-69.9
11	0	10	-	5.6
11	0	12	101.6	101.0
11	0	14	136.2	135.5
11	0	16	85.2	87.8
12	0	0	94.3	-69,0
12	0	2	66.7	-63.8
12	0	4	-	33.1
12	0	6	116.8	109.6
12	0	8	147.9	164.1
12	0	10	68.9	75.4
12	0	12	65.0	61.2
13	0	2	23.4	-33.7
13	0	4	-	20.2
13	0	6	32.7	-40.4
13	0	8	42.7	-55.7
13	0	10	-	1.6
13	0	12	63.8	68.0
14	0	0	148.3	-130.4
14	0	2	101.8	-89.0
14	0	4	-	-4.7
14	0	6	38.8	51.6
14	0	8	99.2	124.4
14	0	10	119.6	122.6
14	0	12	73.0	83.6
16	0	0	94.4	-102.7
16	0	2	70.5	-62.4
1	1	1	271.8	274.9
1	1	2	72.6	-68.5
1	1	3	134.9	113.5
1	1	4	38.4	36.9
1	1	5	22.4	-36.9
1	1	6	62.6	-59.4
1	1	7	135.2	-125.7
1	1	8	89.4	-85.7
1	1	9	133.2	-133.1
1	1	10	-	10.9
1	1	11	83.6	-87.3
11	1	12	54.0	56.8
1	1	13	-	-5.9
1	1	14	83.1	76.5
1	1	15	50.3	50.8
1	1	16	-	5.0
1	1	17	92.5	87.1
1	1	18	-	8.0
1	1	19	100.4	95.8
1	1	20	-	-16.5
1	1	21	91.9	76.6
1	1	22	74.4	-53.2
2	1	0	148.7	-119.9
2	1	1	33.0	31.0
2	1	2	91.2	82.4
2	1	3	56.2	-53.8
2	1	4	60.2	-54.9
2	1	5	-	-8.2
2	1	6	58.5	-60.1
2	1	7	85.1	-80.2
2	1	8	59.2	-49.1
2	1	9	-	20.3
2	1	10	94.0	91.3
2	1	11	-	5.2
2	1	12	20.9	24.9
2	1	13	50.1	56.1
3	1	1	55.3	47.6
3	1	2	40.4	38.6
3	1	3	-	-6.9
3	1	4	-	-9.9
3	1	5	66.9	- 60.1
3	1	6	94.7	-80.9
3	1	7	150.8	-129.8
3	1	8	-	-
3	1	9	148.4	-138.1
3	1	10	-	11.9
3	1	11	95.4	-94.4
3	1	12	20.9	19.0
3	1	13	43.0	-33.1
3	1	14	65.1	62.0
3	1	15	41.9	42.5
3	1	16	46.6	47.5
3	1	17	66.0	65.9
4	1	0	36.9	27.1
4	1	1	46.2	-58.2
4	1	2	25.8	29.4
4	1	3	63.4	-82.0
4	1	4	49.2	-43.7
4	1	5	105.2	-107.3
4	1	6	15.7	-17.9
4	1	7	88.4	-87.1
4	1	8	38.8	38.4
4	1	9	15.7	-22.0
4	1	10	77.2	77.4
4	1	11	53.7	60.4
4	1	12	55.0	54.3
4	1	13	112.9	113.4
4	1	14	-	7.8
4	1	15	108.4	109.0
4	1	16	41.9	-40.8
4	1	17	65.9	62.3
5	1	1	44.1	49.6
5	1	2	18.3	-19.1
5	1	3	84.2	84.2
5	1	4	-	0.3
5	1	5	58.1	47.9
5	1	6	52.1	-51.9
5	1	7	-	-9.4
5	1	8	-	-3.6
5	1	9	65.1	-61.8
5	1	10	-	-2.8
5	1	11	73.2	-74.3
5	1	12	-	-5.6
5	1	13	50.3	-44,4
5	1	14	20.9	33.2
5	1	15	-	14.6
5	1	16	-	15.6
5	1	17	65.9	77.2
5	1	18	-	2.5
5	1	19	83.7	77.0
6	1	0	89.3	-78.6
6	1	1	66.3	-64.0
6	1	2	-	7.9
6	1	3	231.7	-175.9
6	1	4	37.0	-33.2
6	1	5	167.2	-156.2
6	1	6	-	-1.4
6	1	7	50.0	-59.5
6	1	8	53.5	57.7
6	1	9	45.1	39.3
6	1	10	46.4	48.7
6	1	11	83.7	92.5
6	1	12	66.5	46.5
6	1	13	85.3	91.6
6	1	14	-	-6.8
6	1	15	62.8	64.0
6	1	16	41.9	-46.8
7	1	1	51.8	50.7
7	1	2	-	-4.1
7	1	3	20.9	25.8
7	1	4	49.4	-50.1
7	1	5	-	7.7
7	1	6	-	-11.7
7	1	7	31.4	-25.9
7	1	8	31.4	33.5
8	1	0	38.9	-38.1
8	1	1	38.9	-41.8
8	1	2	35.7	-39.2
8	1	3	113.5	-113.5
8	1	4	49.2	-46.9
8	1	5	119.6	-120.3
8	1	6	-	28.0
8	1	7	82.7	-86.5
8	1	8	52.1	46.7
8	1	9	-	12.8
8	1	10	-	23.7
8	1	11	31.4	35.5
8	1	12	-	18.0
8	1	13	83.7	81.1
8	1	14	-	19.0
8	1	15	65.9	71.7
9	1	1	66.7	-70.8
9	1	2	25.8	-29.7
9	1	3	50.8	-51.6
9	1	4	-	-1.2
9	1	5	40.2	-34.5
9	1	6	-	16.9
9	1	7	-	18.7
9	1	8	38.1	42.1
10	1	1	30.4	-38.9
10	1	2	30.7	-28.5
10	1	3	84.9	-84.1
10	1	4	-	-4.9
10	1	5	115.7	-121.9
10	1	6	-	19.1
10	1	7	64.2	-64.7
10	1	8	80.6	83.0
10	1	9	-	-16.1
10	1	10	-	-10.2
10	1	11	83.7	78.5
10	1	12	31.4	44.0
10	1	13	88.9	100,6
10	1	14	-	14,0
10	1	15	100.4	110.2
11	1	1	29.5	-39.3
11	1	2	-	-20.4
11	1	3	-	-28.5
11	1	4	-	11.0
11	1	5	-	22.0
11	1	6	65.5	64.6
11	1	7	41.9	50.9
11	1	8	-	-6.7
11	1	9	62.8	61.8
12	1	0	41.9	-44.3
13	1	1	95.9	-94.7
13	1	2	-	28.8
13	1	3	31.4	-38.6
13	1	4	31.4	34.3
13	1	5	-	28.3
13	1	6	-	28.0
13	1	7	94.2	93.0
13	1	8	-	10.6
13	1	9	88.9	94.9
15	1	1	130.3	-119.8
15	1	2	52.3	48.4
15	1	3	73.2	-66.2
0	2	0	44.9	34.9
0	2	1	-	-0.9
0	2	2	206.6	211. 1
0	2	3	264.9	-252.2
0	2	4	-	8.5
0	2	5	98.5	-106.1
0	2	6	136.2	-149.9
0	2	7	118.4	-136.7
0	2	8	132.9	-151.4
0	2	9	-	2.0
0	2	10	73.8	-59.1
0	2	11	72.7	63.4
0	2	12	30.3	-26.5
0	2	13	140.8	127.4
0	2	14	26.5	-24.7
0	2	15	56.8	51.3
0	2	16	-	15.9
0	2	17	45.0	40.0
0	2	18	99.3	83.6
0	2	19	47.0	-46.8
0	2	20	-	6.3
0	2	21	62.8	-63.1

Table 3 (*cont.*)

0	2	22	-	17.1	8	2	1	-	-16.8	4	3	6	25.1	21.4	9	3	4	-	21.7
0	2	23	126.5	-107.1	8	2	2	58.2	-68.3	4	3	7	35.8	-32.0	9	3	5	-	-9.4
1	2	1	-	-21.1	8	2	3	-	-2.2	4	3	8	122.5	125.7	9	3	6	58.6	52.0
1	2	2	124.6	116.7	8	2	4	64.4	-55.1	4	3	9	28.8	-33.4	9	3	7	-	-21.4
1	2	3	57.3	-60.8	9	2	1	88.4	-96.5	4	3	10	160.7	161.7	9	3	8	64.0	63.2
1	2	4	-	12.4	9	2	2	69.7	-71.6	4	3	11	14.2	12.4	9	3	9	26.6	27.2
1	2	5	31.5	-30.8	9	2	3	68.8	-67.1	4	3	12	99.1	108.6	9	3	10	21.3	17.1
1	2	6	-	-10.0	9	2	4	94.5	-77.7	4	3	13	34.9	27.4	9	3	11	29.4	31.9
1	2	7	44.0	-50.8	9	2	5	-	15.7	4	3	14	-	-17.1	9	3	12	37.5	-39.9
2	2	0	72.8	-70.7	9	2	6	-	-30.3	4	3	15	66.1	64.4	9	3	13	-	-4.6
2	2	1	94.7	-84.7	9	2	7	88.2	84.3	4	3	16	71.0	-74.0	9	3	14	51.5	-51.3
2	2	2	-	-25.3	9	2	8	41.8	-56.5	4	3	17	33.5	37.2	10	3	0	103.3	-120.8
2	2	3	86.3	-84.1	9	2	9	94.1	98.4	4	3	18	124.8	-111.6	10	3	1	-	-11.6
2	2	4	90.8	-84.5	9	2	10	-	6.3	4	3	19	-	-15.3	10	3	2	77.7	-85.4
2	2	5	106.6	-108.8	9	2	11	62.7	67.0	4	3	20	48.8	-48.5	10	3	3	65.2	-48.9
2	2	6	77.1	-68.1	9	2	12	73.2	83.9	5	3	1	41.3	38.7	10	3	4	-	11.0
2	2	7	115.0	-104.5	1	3	1	10.9	18.5	5	3	2	24.8	-31.0	10	3	5	-	-14.0
2	2	8	82.2	-83.2	1	3	2	43.0	-68.1	5	3	3	-	4.6	10	3	6	61.7	65.4
2	2	9	31.7	2.4	1	3	3	71.2	72.1	5	3	4	75.1	-77.4	10	3	7	35.1	-35.5
2	2	10	93.0	-93.0	1	3	4	231.2	-225.5	5	3	5	32.1	28.6	10	3	8	109.3	104.6
2	2	11	54.1	56.5	1	3	5	24.5	28.9	5	3	6	68.2	-71.0	10	3	9	-	16.1
2	2	12	62.6	-74.1	1	3	6	197.3	-183.1	5	3	7	26.9	29.6	10	3	10	143.6	134.2
2	2	13	112.2	123.2	1	3	7	-	-13.2	5	3	8	52.2	-53.8	10	3	11	-	8.4
2	2	14	-	-28.0	1	3	8	70.5	-71.8	5	3	9	-	-10.5	10	3	12	74.2	66.3
2	2	15	84.3	94.8	1	3	9	33.9	-29.8	5	3	10	-	3.4	10	3	13	62.9	47.0
3	2	1	87.1	-70.7	1	3	10	55.5	53.0	5	3	11	39.2	-37.6	10	3	14	-	-20.1
3	2	2	-	-5.6	1	3	11	17.2	-18.0	5	3	12	54.2	57.5	10	3	15	-	12.4
3	2	3	-	-3.3	1	3	12	147.4	130.6	5	3	13	-	-10.7	10	3	16	67.7	-60.5
3	2	4	73.3	-64.1	1	3	13	-	-18.4	5	3	14	69.9	71.2	11	3	1	-	8.3
3	2	5	52.4	-49.4	1	3	14	131.8	133.3	5	3	15	15.8	11.1	11	3	2	59.2	58.1
3	2	6	73.4	-65.7	1	3	15	-	14.6	5	3	16	49.5	54.6	11	3	3	-	24.5
3	2	7	31.5	32.1	1	3	16	96.8	99.9	5	3	17	-	2.4	11	3	4	61.9	62.1
3	2	8	-	14.0	1	3	17	44.5	42.3	5	3	18	-	3.5	11	3	5	-	-9.3
3	2	9	81.1	81.0	1	3	18	33.4	-22.0	5	3	19	-	27.6	11	3	6	57.6	53.9
3	2	10	-	10.6	1	3	19	43.0	32.8	5	3	20	40.0	-49.6	11	3	7	-	-8.3
3	2	11	86.5	91.5	1	3	20	104.7	-94.3	6	3	0	147.5	-138.8	11	3	8	67.0	61.1
4	2	0	39.5	-40.5	1	3	21	-	-3.0	6	3	1	31.6	-27.8	11	3	9	-	10.9
4	2	1	-	-6.0	2	3	0	12.0	1.2	6	3	2	152.5	-147.5	11	3	10	-	-9.9
4	2	2	-	-21.9	2	3	1	-	-5.8	6	3	3	-	-15.8	11	3	11	-	23.5
4	2	3	40.1	-36.1	2	3	2	68.6	-76.0	6	3	4	33.9	-34.3	11	3	12	83.6	-99.5
4	2	4	68.3	-59.8	2	3	3	70.9	74.7	6	3	5	26.9	-27.2	11	3	13	-	2.8
4	2	5	108.9	-105.4	2	3	4	136.0	-129.1	6	3	6	78.7	82.8	11	3	14	84.1	-86.1
4	2	6	62.3	-62.0	2	3	5	44.7	-47.4	6	3	7	-	-14.6	12	3	0	62.6	-60.0
4	2	7	47.4	-49.1	2	3	6	24.0	-20.4	6	3	8	136.5	145.7	12	3	1	-	19.0
4	2	8	57.9	-57.6	2	3	7	-	8.7	6	3	9	-	-0.3	12	3	2	71.3	-68.5
4	2	9	40.5	-31.1	2	3	8	33.8	38.3	6	3	10	134.0	140.6	12	3	3	-	-20.7
4	2	10	117.3	-124.3	2	3	9	27.7	-21.9	6	3	11	38.5	30.4	12	3	4	-	-24.8
4	2	11	51.2	62.3	2	3	10	56.0	60.9	6	3	12	52.8	60.0	12	3	5	-	-1.1
4	2	12	53.9	-57.6	2	3	11	-	11.4	6	3	13	26.2	31.7	12	3	6	60.8	55.4
4	2	13	72.7	70.5	2	3	12	40.2	38.1	6	3	14	29.6	-31.0	12	3	7	-	4.8
4	2	14	-	-14.7	2	3	13	-	-14.4	6	3	15	-	9.1	12	3	8	80.2	66.7
4	2	15	108.3	93.1	2	3	14	-	2.1	6	3	16	102.3	-93.8	12	3	9	-	10.4
5	2	1	91.3	-82.0	2	3	15	33.5	25.2	6	3	17	-	-17.7	12	3	10	67.4	51.2
5	2	2	43.5	-39.6	2	3	16	81.3	-82.4	6	3	18	117.2	-122.2	13	3	1	-	-18.0
5	2	3	52.0	-44.2	2	3	17	31.7	-26.8	6	3	19	-	-10.6	13	3	2	72.7	71.1
5	2	4	100.3	-98.7	2	3	18	109.2	-95.4	6	3	20	77.3	-63.1	13	3	3	-	-7.1
5	2	5	45.6	40.8	2	3	19	24.6	-28.8	7	3	1	43.0	47.7	13	3	4	132.9	127.1
5	2	6	45.8	-37.4	2	3	20	25.5	-26.2	7	3	2	57.4	-60.0	13	3	5	-	21.6
5	2	7	81.7	79.9	3	3	1	25.4	-29.6	7	3	3	-	11.7	13	3	6	86.7	89.2
5	2	8	24.9	-27.0	3	3	2	73.6	-77.9	7	3	4	41.9	-46.2	13	3	7	-	8.3
5	2	9	117.1	126.3	3	3	3	-	-1.3	7	3	5	34.1	-30.8	13	3	8	-	25.3
5	2	10	-	17.1	3	3	4	119.7	-123.9	8	3	0	186.3	-88.2	13	3	9	-	28.1
5	2	11	98.7	104.0	3	3	5	20.1	-25.4	8	3	1	-	-4.5	13	3	10	49.6	-45.9
5	2	12	62.2	66.0	3	3	6	89.7	-92.9	8	3	2	134.0	-140.3	13	3	11	-	31.6
6	2	0	50.0	37.1	3	3	7	-	-11.5	8	3	3	-	-15.0	13	3	12	79.5	-89.9
6	2	1	-	-5.4	3	3	8	85.3	-82.8	8	3	4	48.5	-39.9	14	3	0	67.0	-60.8
6	2	2	-	6.2	3	3	9	28.0	-28.8	8	3	5	-	0.1	15	3	1	35.0	-35.1
6	2	3	-	-26.8	3	3	10	29.1	33.1	8	3	6	61.9	62.1	15	3	2	91.1	83.3
6	2	4	-	15.7	3	3	11	26.5	-24.1	8	3	7	-	1.1	15	3	3	-	-23.8
6	2	5	53.8	-53.6	3	3	12	145.9	157.5	8	3	8	86.8	89.7	15	3	4	151.3	135.5
6	2	6	-	-12.1	3	3	13	-	10.9	8	3	9	62.5	-45.6	15	3	5	-	1.0
6	2	7	51.6	-49.6	3	3	14	123.1	135.5	8	3	10	134.0	140.6	15	3	6	91.2	96.9
6	2	8	-	-21.6	3	3	15	-	10.0	8	3	11	-	21.5	0	4	0	30.3	31.0
6	2	9	-	16.1	3	3	16	75.6	85.8	8	3	12	35.1	39.2	0	4	1	105.6	-108.3
6	2	10	66,3	-64.3	3	3	17	32.5	21.1	8	3	13	-	1.4	0	4	2	48.1	46.1
7	2	1	142.2	-139.7	3	3	18	26.5	7.0	8	3	14	42.1	-36.6	0	4	3	152.5	-178.8
7	2	2	50.0	-43.8	3	3	19	-	-15.3	8	3	15	-	1.4	0	4	4	34.5	24.0
7	2	3	80.6	-79.6	3	3	20	88.2	-50.5	8	3	16	99.5	-93.6	0	4	5	260.5	-238.0
7	2	4	73.9	-80.9	3	3	22	97.8	-80.0	8	3	17	-	2.1	0	4	6	112.1	112.2
7	2	5	-	-6.7	4	3	0	18.7	-19.4	8	3	18	109.9	-112.6	0	4	7	58.8	-58.9
7	2	6	49.9	-38.1	4	3	1	40.2	42.6	8	3	19	-	-2.7	0	4	8	30.8	31.7
7	2	7	89.2	101.5	4	3	2	81.4	-86.0	8	3	20	93.9	-94.6	0	4	9	39.1	34.5
7	2	8	-	-13.2	4	3	3	22.1	23.0	9	3	1	15.8	-17.3	0	4	10	-	-0,2
7	2	9	81.3	90.9	4	3	4	-	-6.4	9	3	2	21.3	-10.8	0	4	11	88.6	78.9
8	2	0	-	-29.4	4	3	5	41.3	-43.8	9	3	3	37.2	-29.1	0	4	12	-	-2.9

Table 3 (*cont.*)

0	4	13	72.4	59.0
0	4	14	82.4	65.8
0	4	15	93.6	84.6
0	4	16	31.6	29.3
0	4	17	27.0	23.1
0	4	18	-	3.2
0	4	19	45.4	-27.7
0	4	20	-	-7.1
0	4	21	90.5	-86.3
0	4	22	-	3.0
0	4	23	95.4	-88.2
1	4	1	26.6	-36.2
1	4	2	20.6	25.4
1	4	3	42.0	-44.5
1	4	4	48.3	56.0
1	4	5	51.2	-56.5
1	4	6	48.0	49.4
1	4	7	-	-7.0
1	4	8	45.8	-29.0
1	4	9	-	-5.1
1	4	10	36.0	-30.1
2	4	0	25.5	-28.8
2	4	1	34.0	-32.2
2	4	2	45.7	-54.9
2	4	3	151.9	-126.8
2	4	4	84.3	76.2
2	4	5	179.7	-155.6
2	4	6	93.9	83.5
2	4	7	41.0	-42.3
2	4	8	43.4	51.5
2	4	9	-	-1.5
2	4	10	61.2	56.7
2	4	11	91.9	87.7
2	4	12	25.9	14.0
2	4	13	77.5	80.1
2	4	14	-	11.2
2	4	15	101.3	105.0
3	4	1	61.7	-57.7
3	4	2	22.8	16.1
3	4	3	86.1	-88.5
3	4	4	47.7	48.5
3	4	5	30.6	24.5
3	4	6	26.3	-21.8
3	4	7	67.0	68.9
3	4	8	18.9	-14.6
3	4	9	76.8	75.3
3	4	10	-	-4.7
3	4	11	43.0	45.4
3	4	12	-	5.4
3	4	13	35.9	28.0
3	4	14	-	21.1
3	4	15	-	-9.1
3	4	16	-	-22.9
3	4	17	95.9	-81.0
3	4	18	-	-19.4
3	4	19	82.8	-69.0
4	4	0	24.4	17.8
4	4	1	37.9	-35.9
4	4	2	-	8.3
4	4	3	61.9	-66.5
4	4	4	40.0	38.3
4	4	5	61.3	-61.9
4	4	6	-	18.3
4	4	7	70.4	-74.5
4	4	8	37.9	38.4
4	4	9	15.9	-21.0
4	4	10	42.0	36.7
4	4	11	52.8	52.2
4	4	12	49.1	44.5
4	4	13	100.6	108.5
4	4	14	-	17.6
4	4	15	74.7	75.5
5	4	1	108.0	-108.2
5	4	2	53.7	50.6
5	4	3	39.7	-39.0
5	4	4	43.8	45.6
5	4	5	61.8	62.6
5	4	6	-	-3.2
5	4	7	127.8	131.4
5	4	8	-	-27.4
5	4	9	117.1	119.1
5	4	10	-	-16.2
5	4	11	73.2	74.3
6	4	0	59.8	63.4
6	4	1	-	-2.6
6	4	2	49.5	52.6
6	4	3	44.9	-46.8
7	4	1	189.0	-177.9
7	4	2	-	25.1
7	4	3	75.0	-76.8
7	4	4	73.8	75.6
7	4	5	45.9	43.0
7	4	6	-	21.1
7	4	7	66.4	66.2
7	4	8	-	3.9
7	4	9	90.8	100.7
7	4	10	-	-15.2
7	4	11	65.5	70.9
9	4	1	107.9	-121.1
9	4	2	-	14.6
9	4	3	39.5	-48.9
9	4	4	-	3.0
9	4	5	-	20.8
9	4	6	-	-9.9
9	4	7	65.5	-70.5
9	4	8	-	27.2
9	4	9	115.7	112.5
9	4	10	-	12.1
9	4	11	78.7	98.0
1	5	1	85.7	-95.9
1	5	2	94.9	-110.0
1	5	3	80.9	-85.3
1	5	4	88.1	-104.2
1	5	5	12.3	24.4
1	5	6	68.0	-80.3
1	5	7	74.4	86.9
1	5	8	-	-11.8
1	5	9	78.3	83.5
2	5	0	52.8	-52.5
2	5	1	-	-7.1
2	5	3	-	18.2
2	5	4	-	-7.0
2	5	5	62.8	69.6
2	5	6	36.4	-37.6
2	5	7	51.7	42.7
3	5	1	98.2	-79.3
3	5	2	39.7	-37.4
3	5	3	69.8	-61.3
3	5	4	56.4	-55.3
3	5	5	-	10.4
3	5	6	74.8	-75.8
3	5	7	52.0	49.9
3	5	8	-	2.0
3	5	9	89.2	89.9
3	5	10	46.8	38.1
3	5	11	78.6	79.6
4	5	0	66.8	-50.9
4	5	1	-	17.2
4	5	2	-	12.5
4	5	3	-	32.0
4	5	4	-	-7.0
4	5	5	99.0	105.2
4	5	6	65.5	59.5
4	5	7	-	13.3
4	5	8	93.7	86.1
5	5	1	70.4	-62.3
6	5	0	141.6	-125.4
6	5	1	54.7	41.4
6	5	2	82.7	-75.0
6	5	3	91.4	79.3
6	5	4	-	16.4
6	5	5	101.9	88.8
6	5	6	68.8	62.9
8	5	0	113.2	-113.2
8	5	1	36.8	39.0
8	5	2	74.4	-71.2
8	5	3	115.2	120.7
8	5	4	-	23.9
0	6	0	178.2	-181.9
0	6	1	84.6	-74.2
0	6	2	167.8	-157.2
0	6	3	80.3	-91.6
0	6	4	20.5	-25.8
0	6	5	30.2	-61.4
0	6	6	29.5	27.2
0	6	7	26.4	-25.0
0	6	8	89.0	85.7
0	6	9	-	17.8
0	6	10	65.1	68.5
0	6	11	27.1	29.8
0	6	12	29.3	25.9
0	6	13	-	26.6
0	6	14	53.9	-53.6
0	6	15	-	-4.0
0	6	16	111.4	-87.0
0	6	17	-	-12.5
0	6	18	122.7	-117.2
1	6	1	16.4	-21.3
1	6	2	21.8	34.6
1	6	3	-	-8.2
1	6	4	36.8	53.5
1	6	5	21.3	-28.6
1	6	6	29.6	28.9
1	6	7	-	-26.1
1	6	8	4.7	37.0
2	6	0	282.0	-187.6
2	6	1	29.0	-21.9
2	6	2	164.3	-129.6
2	6	3	59.7	-59.2
2	6	4	51.2	-50.3
2	6	5	-	-12.2
2	6	6	49.9	59.2
2	6	7	-	-24.8
2	6	8	129.7	142.9
2	6	9	-	14.8
2	6	10	72.8	72.3
2	6	11	-	17.0
2	6	12	57.9	54.1
2	6	13	52.5	44.0
3	6	1	58.2	-52.8
3	6	2	46.5	38.2
3	6	3	-	8.5
3	6	4	56.6	50.4
3	6	5	-	15.6
3	6	6	82.5	8.4
3	6	7	-	7.0
3	6	8	-	20.4
3	6	9	-	29.0
3	6	10	-	-14.5
3	6	11	-	31.5
3	6	12	-	-39.5
3	6	13	-	-11.4
3	6	14	78.2	-83.7
4	6	0	110.7	-97.5
4	6	1	-	0.2
4	6	2	98.4	-82.8
4	6	3	-	-13.8
4	6	4	45.6	-42.5
4	6	5	28.4	-33.0
4	6	6	-	12.5
4	6	7	-	-29.6
4	6	8	54.5	54.5
4	6	9	-	23.3
4	6	10	73.0	84.9
4	6	11	-	6.8
4	6	12	-	41.9
4	6	13	-	33.8
4	6	14	-	-15.5
4	6	15	-	40.1
4	6	16	96.7	-74.8
5	6	1	61.0	-46.7
5	6	2	81.7	74.9
5	6	3	-	15.0
5	6	4	134.5	120.2
5	6	5	-	31.1
5	6	6	96.8	91.7
5	6	7	59.1	56.0
5	6	8	-	15.8
5	6	9	51.2	42.0
5	6	10	56.6	-51.3
5	6	11	-	29.4
5	6	12	101.3	-85.1
5	6	13	-	12.2
5	6	14	117.9	-90.2
6	6	0	84.0	-82.4
6	6	1	-	1.7
6	6	2	40.0	-34.7
6	6	3	-	24.1
6	6	4	34.6	-35.0
6	6	5	54.8	-53.2
6	5	6	37.5	--29.4
7	6	1	66.6	-61.1
7	6	2	135.8	130.9
7	6	3	38.1	-45.4
7	6	4	135.9	134.8
7	6	5	-	2.6
7	6	6	126.8	125.1
7	6	7	58.0	49.6
7	6	8	54.8	46.7
7	6	9	-	16.5
7	6	10	34.1	-39.9
7	6	11	-	-17.0
7	6	12	93.8	-102.9
7	6	13	-	2.4
7	6	14	118.2	-114.5
9	6	1	32.5	-37.4
9	6	2	61.9	61.8
9	6	3	-	-28.5
9	6	4	91.4	102.3
9	6	5	-	-1.0
9	6	6	86.1	98.9
9	6	7	43.3	49.2
9	6	8	-	34.1
9	6	9	-	36.5
9	66	10	-	-36.2
9	6	11	-	13.1
9	6	12	91.0	-78.2
9	6	13	-	18.7
9	6	14	100.3	-100.7

four oxygen atoms toward the N atom of the pyridine ring (in the structure (3) towards the O atom of the water molecule). This displacement is 0·22 Å in all three structures. The corresponding Cu–N distances are: 2·19 Å in structure (1) and 2·12–2·13 Å in structure (2). The Cu–O distance in structure (3) is 2·20 Å. These five nearest atoms coordinate the Cu atom in the form of a tetragonal pyramid. The distorted octahedral coordination around the Cu atom is completed by Cu–Cu contact.

The acetate groups are planar. The mean bond lengths C–O are: 1·25 Å in structure (1), 1·24 Å in structure (2) and 1·33 Å in structure (3). The corresponding C–C distances are· 1·54, 1·51 and 1·52 Å respectively. The normals of the two planes of the acetate groups are perpendicular to each other. The

Table 4. *Bond distances and angles*

(1) Orthorhombic $Cu_2(CH_3CO_2)_4 . 2\ C_5H_5N$
(2) Monoclinic $Cu_2(CH_3 . CO_2)_4 . 2\ C_5H_5N$
(3) $Cu_2(CH_3 . CO_2)_4 . 2\ H_2O$

Bond	(1) (Å)	(2) (Å)	(3) (Å)
Cu–Cu	2·645 ± 0·003	2·630 ± 0·003	2·64
Cu–O(H_2O) or Cu–N	2·186 ± 0·008	2·122 ± 0·009	2·20
(pyridine ring)		2·129 ± 0·011	
Cu–O	1·979 ± 0·008	1·985 ± 0·010	1·96
(planar group)	1·965 ± 0·007	1·973 ± 0·010	1·99
	1·948 ± 0·008	1·985 ± 0·010	1·95
	1·928 ± 0·008	1·982 ± 0·010	1·98
C–O	1·248 ± 0·020	1·247 ± 0·016	1·33
(acetate group)	1·236 ± 0·019	1·247 ± 0·015	1·36
	1·250 ± 0·022	1·231 ± 0·021	1·29
	1·250 ± 0·023	1·231 ± 0·021	1·34
C–N	1·311 ± 0·023	1·377 ± 0·014	
(pyridine ring)	1·349 ± 0·020	1·379 ± 0·016	
C–C	1·396 ± 0·031	1·381 ± 0·018	
(pyridine ring)	1·339 ± 0·033	1·411 ± 0·019	
	1·379 ± 0·032	1·433 ± 0·021	
	1·426 ± 0·026	1·412 ± 0·018	
C–C	1·525 ± 0·025	1·467 ± 0·022	1·52
(acetate group)	1·549 ± 0·027	1·540 ± 0·026	1·52
Angle	(°)	(°)	(°)
O–Cu–O	88·7	89·0	
(planar group)	91·2	88·2	
	87·4		
	89·9		
O–C–O	127·8	127·3	116
(acetate group)	123·3	122·9	108
O–C–C	115·7	115·0	
(acetate group)	116·4	118·1	
	118·9		
	123·3		
C–N–C	119·2	117·9	
(pyridine ring)		116·4	
N–C–C	121·4	121·5	
(pyridine ring)	122·5	125·2	
C–C–C	119·7	113·7	
(pyridine ring)	121·6	120·5	
	115·3	124·2	
		116·5	

angles between the planes of the acetate groups and pyridine rings are 30°. There is no significant difference between the carbon–nitrogen and carbon–carbon bond lengths in the pyridine molecules. The bond angles vary from 114 to 125°, the bond lengths from 1·31 to 1·43 Å.

The equation for the plane of the pyridine ring in structure (1) is:

$$0{\cdot}325x - 0{\cdot}845y + 0{\cdot}424z - 0{\cdot}115 = 0$$

The pyridine ring is nearly planar and there is a slight alternating displacement, ± 0·03 Å, of the atoms from the plane of the ring. The interatomic distances and bond angles of the pyridine ring are shown in Fig. 1.

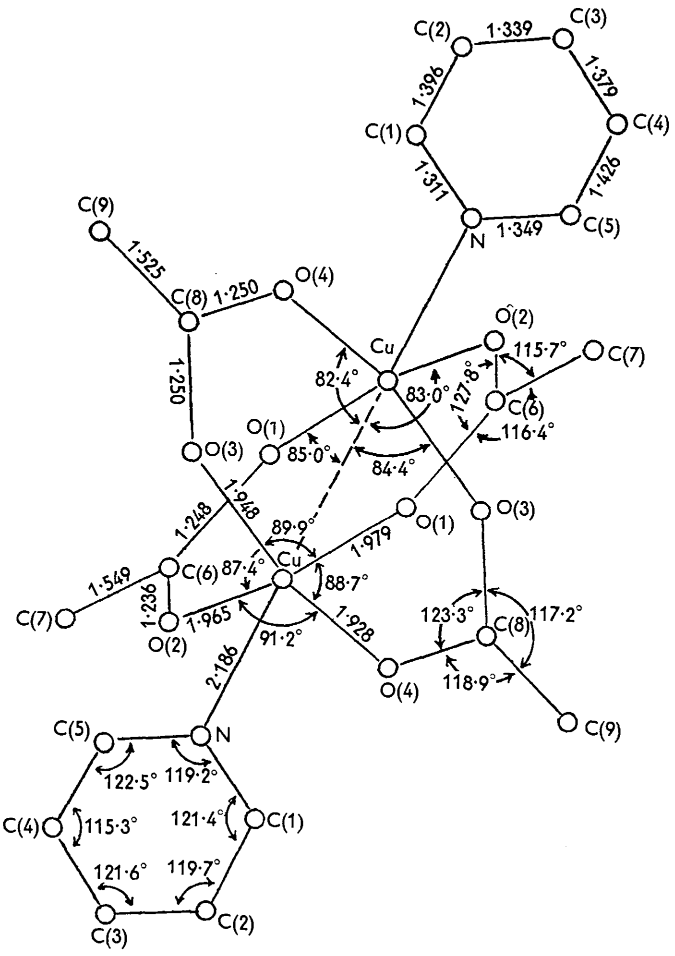
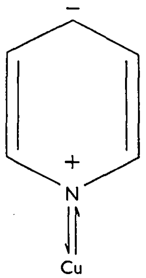


Fig. 1. Bond lengths (Å) and bond angles in the binuclear molecule.

The angles between the normals of the pyridine rings and the Cu–Cu bonds are 89°.

The assumption of the participation of the valence formula:



in the structure of the pyridinecopper(II) acetate, supported by Martin and Waterman (1959), does not seem to be confirmed by the established interatomic distances in the pyridine rings. No shortening of the bond lengths C(1)–C(2) and C(4)–C(5) in the pyridine rings was observed.

We are greatly indebted to Prof L. G. Sillén, Director of the Department of Inorganic Chemistry of the Royal Institute of Technology in Stockholm, for his help and interest in this work. We are most thankful to Dr G. Lundgren for his friendly help and also to Dr D. MacWhan. Dr J. Šmogrovič was kind enough to supply us with the crystals of monopyridine-copper(II) acetate.

Last but not least, we wish to thank the Swedish Board of Computing Machinery for providing us with free time on the computers BESK and FACIT EDB.

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Precise and Accurate Lattice Parameters by Film Powder Methods. III. An Exact Graphical Method for Axial (Vertical) Divergence Profiles for Cylindrical Diffraction Cameras*

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(*Received* 14 *February* 1962 *and in revised form* 1 *August* 1963)

An exact graphical method has been developed for calculating axial divergence profiles for cylindrical powder cameras. The method is exact in that no approximations are used in deriving the equations for calculating the profiles. The profiles can easily be plotted with an accuracy considerably greater than the best precision with which diffraction lines can be measured on film patterns (about 0·002° θ (Straumanis & Ievins, 1940)). This is important when these profiles are used to correct diffraction line measurements for axial divergence, so that the application of this systematic error correction will not limit the accuracy of the corrected data.

Various characteristics of axial divergence profiles for cases of practical interest are presented. In particular, it is shown that the use of extremely high angles (about 175° 2θ) as recommended by Straumanis & Weng (1955) causes negligible error (less than the 0·002° θ precision of measurement possible with sharp diffraction lines) due to axial divergence, provided the collimator dimensions are restricted as described, a spot focus source is used, and diffraction measurements are made in the equatorial plane of the diffraction pattern. Relaxation of these conditions may require correction for axial divergence; however, for cameras of the type described by Straumanis (not necessarily identical with Straumanis-type cameras), the correction for axial divergence will usually be less than the precision of measurement and may be neglected.

1. Introduction

The problem of axial (vertical) divergence has been treated in numerous ways with varying degrees of approximation and various quantitative conclusions. It is, however, generally agreed that: (1) the correction for axial divergence increases as 2θ approaches either 0° or 180°, and (2) the correction goes to zero in the range of about 90° to 120° 2θ (Eastabrook, 1952; Pike, 1957). Methods for calculating axial divergence profiles have been derived for two crystal X-ray

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* This work (other than the contribution by Dr E. R. Pike) was performed under Contract AT–(33–2)–1 with the U.S. Atomic Energy Commission.