Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1976). B**32**, 2919

Strukturen von Kobalt(II)-Komplexen der Stöchiometrie $Co(CNR)_4J_2$ mit R=Phenyl bzw. 2,6-Diäthylphenyl. Corrigenda.

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(*Received* 14 *May* 1976; *accepted* 14 *May* 1976)

As pointed out by the Crystallographic Data Centre in Cambridge, a mistake has occurred in the lattice constants of $Co(CNC_6H_5)_4I_2$ [Baumann, Endres, Keller, Nuber & Weiss, *Acta Cryst.* (1975), B**31**, 40–44]. The correct values are: $a=12{\cdot}462$, $b=11{\cdot}058$, $c=11{\cdot}878$ Å, $\alpha=104{\cdot}64$, $\beta=93{\cdot}19$, $\gamma=107{\cdot}69°$.

All relevant details are given in the abstract.

Acta Cryst. (1976). B**32**, 2919

Ammonium perchlorate: reinvestigation of the crystal structure at 298 K.

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(*Received* 27 *April* 1976; *accepted* 27 *May* 1976)

The structure of ammonium perchlorate at 298K has been refined with the X-ray diffraction data of Peyronel & Pignedoli [*Acta Cryst.* (1975), B**31**, 2052–2056]. A satisfactory refinement was achieved in space group *Pnma*, in agreement with earlier observations at low temperatures.

A refinement of the structure of ammonium perchlorate, NH_4ClO_4, at room temperature has recently been reported by Peyronel & Pignedoli (1975) (hereafter referred to as PP), who conclude that the structure cannot be refined in the centrosymmetric space group *Pnma* by least-squares methods but can be refined in the space group $Pna2_1$. This conclusion is in disagreement with refinements at low temperatures (10 and 78K) of neutron diffraction data by Choi, Prask & Prince (1974) (hereafter referred to as CPP). CPP did not succeed in refining the structure at 298K because of the important contribution of H atoms to the neutron structure factors and the very large thermal amplitudes of the H atoms. The non-centrosymmetric structure reported by PP differs from a centrosymmetric one only in displacements of atomic positions from the mirror plane which are small compared with the amplitudes of thermal vibration. Furthermore, the absence of a center of symmetry at 298K and the presence of one at 78K imply a phase transition at some intermediate temperature for which there is no other definite physical evidence.

Because of the discrepancy between the structures reported by PP and CPP we have performed further least-squares refinement of the room-temperature structure in the centrosymmetric space group using the published data of PP. The unit-cell dimensions given by PP (based on $Pna2_1$) were converted to the equivalent values in the space group *Pnma*. The computer program *RFINE* 4 (Finger & Prince, 1975) was used throughout. Initially, unit weights were used for all reflections and H atoms were neglected. Starting parameters were the 10K parameters of CPP, with the thermal parameters converted to equivalent isotropic thermal parameters. Two cycles of refinement led to R indices of about 0·25. Comparison of observed and calculated structure factors showed a systematic difference which suggested a large amplitude of thermal motion in a direction parallel to the c axis (the b axis in space group $Pna2_1$, used by PP). It appeared that secondary extinction might also be a problem. Further refinement was carried out, therefore, including anisotropic temperature factors and an isotropic secondary extinction parameter (Zachariasen, 1968), with the path-length parameter, $\bar{T}$, treated as a constant. Three cycles of refinement reduced the R indices to $R_w=[\sum w(F_o-|F_c|)^2/\sum wF_o^2]^{1/2}=0{\cdot}047$ and $R=\sum|F_o-|F_c||/\sum F_o=0{\cdot}048$ for the 800 observed reflections. The effect of secondary extinction was very marked for certain strong reflections, with $F_{\rm corr}/F_{\rm calc}=0{\cdot}73$ for the 011 reflection. To give relatively less weight to strong reflections, two additional cycles were performed with weights given by $w=(\sigma^2+0{\cdot}0005I)^{-1}$, where I is the observed intensity. The final secondary extinction parameter $r^*{\rm d}A/\mu$ (Zachariasen, 1968) was $0{\cdot}1016\times10^{-6}$ and the scale factor was 1·1073; the other final parameters are given in Table 1.† A comparison of this centrosymmetric structure result ($R=0{\cdot}044$ for the 35 parameters) with the non-centrosymmetric structure refinement of PP ($R=0{\cdot}055$ for the 55 parameters) clearly shows that *Pnma* is the correct space group for room-temperature NH_4ClO_4, as it is at lower temperatures. The observed bond lengths, Cl–O(1) = 1·432 (3), Cl–O(2) = 1·424 (3), Cl–O(3) = 1·442 (2)

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† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31915 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final least-squares parameters obtained with weights* $w=(\sigma^2+0{\cdot}0005I)^{-1}$

Thermal parameters are the coefficients U_{ij} (in Å^2), multiplied by 10^4, in the form:

$$\exp\left[-(U_{11}h^2+U_{22}k^2+U_{33}l^2+2U_{12}hk+2U_{13}hl+2U_{23}kl)\right].$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	0·4318 (1)	0·2500	0·1916 (1)	289 (3)	286 (3)	989 (7)	0	13 (3)	0
O(1)	0·3142 (3)	0·2500	0·0664 (4)	540 (13)	522 (13)	1059 (0)	0	− 169 (14)	–
O(2)	0·5680 (3)	0·2500	0·1019 (5)	444 (12)	699 (17)	1351 (26)	0	248 (15)	0
O(3)	0·4203 (2)	0·0487 (2)	0·3036 (3)	485 (8)	324 (6)	1171 (15)	12 (6)	− 33 (9)	101 (8)
N	0·3191 (2)	0·2500	0·6645 (5)	323 (10)	366 (11)	1058 (23)	3	25 (13)	0

Å, are, within one standard deviation, the same as those of PP (obtained with the positions of the ammonium ions held fixed). The thermal ellipsoids of all atoms in Table 1 are highly anisotropic, with U_{33} two to three times larger than U_{11} or U_{22} throughout. The observed atomic thermal motions of the perchlorate group agree extremely well with values obtained by a rigid-body analysis with the method of Schomaker & Trueblood (1968), with an average difference in U_{ij} of 0·0002 Å^2. The librational motion of the perchlorate group, as obtained from the rigid-body refinement, is almost isotropic (7·1, 6·9, 6·5° for the amplitudes about principal axes), and causes the apparent Cl–O bond length to be shortened by about 0·02 Å. The translational motion, however, is extremely anisotropic and closely resembles the thermal ellipsoid of the N atom of the ammonium group (0·0291, 0·0281 and 0·0987 Å^2 for the three principal axes of **T**). It is possible that this relative large translational motion along **c** is correlated with the occurrence of highly temperature-dependent lattice modes observed *via* Raman scattering (Rosasco & Prask, 1975, 1976).

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Acta Cryst. (1976). B**32**, 2920

2-Aminopyridine and 3-aminopyridine. Errata.

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(*Received* 28 *May* 1976; *accepted* 28 *May* 1976)

The figures giving the bond distances and angles in 2-aminopyridine and 3-aminopyridine, by Chao, Schempp & Rosenstein [*Acta Cryst.* (1975), B**31**, 2922–2924 and 2924–2926], have been transposed. Fig. 1 of the paper on 3-aminopyridine shows the results for 2-aminopyridine and Fig. 2 of the paper on 2-aminopyridine shows those for 3-aminopyridine.

All the relevant information is given in the abstract.

We are grateful to Dr K. D. Holmes for bringing these errors to our attention.

Acta Cryst. (1976). B**32**, 2920

The crystal structure of [1,4-di-(*N*-pyridiniummethyl)benzene]$^{2+}$ (7,7,8,8-tetracyanoquinodimethane)$_4^{2-}$. Erratum.

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(*Received* 1 *June* 1976; *accepted* 4 *June* 1976)

In the previous paper on the title complex [*Acta Cryst.* (1975), B**31**, 1174–1178] the TCNQ stacking direction was erroneously described as parallel to **b**. It is, in fact, parallel to [110].

In our paper on the crystal structure of 1,4-di-(*N*-pyridiniummethyl)benzene(TCNQ)$_4$ (Ashwell, Wallwork, Baker & Berthier, 1975) we stated that the TCNQ molecules and anions were packed plane-to-plane in columns parallel to **b**. In fact the columns are parallel to [110], *i.e.* in the (001) plane and along the positive diagonal between **a** and **b**. The atomic coordinates, intra- and intermolecular distances and diagrams given in the paper are correct.

Reference

Ashwell, G. J., Wallwork, S. C., Baker, S. R. & Berthier, P. I. C. (1975). *Acta Cryst.* B**31**, 1174–1178.