

## Short Communications

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*Acta Cryst.* (1976). B32, 2919

**Strukturen von Kobalt(II)-Komplexen der Stöchiometrie  $\text{Co}(\text{CNR})_4\text{J}_2$  mit R=Phenyl bzw. 2,6-Diäthylphenyl. Corrigenda.** By D. BAUMANN, H. ENDRES, H. J. KELLER, B. NUBER and J. WEISS, *Anorganisch-Chemisches Institut der Universität Heidelberg, Germany (BRD)*

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As pointed out by the Crystallographic Data Centre in Cambridge, a mistake has occurred in the lattice constants of  $\text{Co}(\text{CNC}_6\text{H}_5)_4\text{J}_2$  [Baumann, Endres, Keller, Nuber & Weiss, *Acta Cryst.* (1975), B31, 40–44]. The correct values are:  $a=12.462$ ,  $b=11.058$ ,  $c=11.878$  Å,  $\alpha=104.64$ ,  $\beta=93.19$ ,  $\gamma=107.69^\circ$ .

All relevant details are given in the abstract.

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**Ammonium perchlorate: reinvestigation of the crystal structure at 298 K.** By C. S. CHOI and H. J. PRASK, *Feltman Research Laboratory, Picatinny Arsenal, Dover, N.J. 07801, U.S.A. and Institute for Materials Research, National Bureau of Standards, Washington, D.C. 20234, U.S.A.* and E. PRINCE, *Institute for Materials Research, National Bureau of Standards, Washington, D.C. 20234, U.S.A.*

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The structure of ammonium perchlorate at 298 K has been refined with the X-ray diffraction data of Peyronel & Pignedoli [*Acta Cryst.* (1975), B31, 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,  $\text{NH}_4\text{ClO}_4$ , 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<sub>1</sub>*. This conclusion is in disagreement with refinements at low temperatures (10 and 78 K) of neutron diffraction data by Choi, Prask & Prince (1974) (hereafter referred to as CPP). CPP did not succeed in refining the structure at 298 K 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 298 K and the presence of one at 78 K 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<sub>1</sub>*) 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 10 K parameters of CPP, with the thermal parameters converted to equivalent isotropic thermal parameters. Two cycles of refinement led to *R* in-

stances 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<sub>1</sub>*, 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  $R_w = [\sum w(F_o - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.047$  and  $R = \sum |F_o - |F_c|| / \sum F_o = 0.048$  for the 800 observed reflections. The effect of secondary extinction was very marked for certain strong reflections, with  $F_{\text{corr}}/F_{\text{calc}} = 0.73$  for the 011 reflection. To give relatively less weight to strong reflections, two additional cycles were performed with weights given by  $w = (\sigma^2 + 0.0005I)^{-1}$ , where *I* is the observed intensity. The final secondary extinction parameter  $r^*dA/\mu$  (Zachariasen, 1968) was  $0.1016 \times 10^{-6}$  and the scale factor was 1.1073; the other final parameters are given in Table 1.† A comparison of this centrosymmetric structure result ( $R=0.044$  for the 35 parameters) with the non-centrosymmetric structure refinement of PP ( $R=0.055$  for the 55 parameters) clearly shows that *Pnma* is the correct space group for room-temperature  $\text{NH}_4\text{ClO}_4$ , 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)

† 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.