similarity of the interionic relationships in the two $(MCl_4)^{2-}$ complexes suggests that there is little dependence of ground-state partial bonding on the metal d^n population. The shift of the optical charge-transfer transition to lower energy in the Fe compound parallels the greater ease of reduction of Fe^{II}.

We have also prepared the hitherto unknown $(CuCl_4)^{2-}$ analogue. Remarkably, it is not isostructural with the $(CoCl_4)^{2-}$ and $(FeCl_4)^{2-}$ complexes, although its triclinic cell parameters are very closely related to those of the monoclinic cells of the Co^{II} and Fe^{II} complexes. Preliminary X-ray and infrared data suggest two different environments for the dithiolium cations. It appears, therefore, that the known tendency of $(CuCl_4)^{2-}$ to occur as a considerably flattened tetrahedron is associated with further reorganization of the lattice. We hope to report the structure of the Cu^{II} complex in due course.

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Terephthalonitrile

BY COBY VAN RIJ AND DOYLE BRITTON

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Abstract. Eastman-Kodak $C_6H_4(CN)_2$ was recrystallized from glacial acetic acid; triclinic, $P\bar{1}$ (No. 2), $Z = 1$, $a = 7.318 (3)$, $b = 7.544 (3)$, $c = 3.841 (2)$ Å, $\alpha = 80.45 (3)$, $\beta = 93.57 (3)$, $\gamma = 127.44 (1)°$, $V = 165.8$ Å³, $D_c = 1.283$ g cm⁻³. (The standard setting has $a = 6.583$, $b = 7.318$, $c = 3.841$ Å, $\alpha = 93.57$, $\beta = 96.94$, $\gamma = 114.52°$. It can be obtained from the setting used in the main body of the paper by the transformation $\bar{1}10/100/001$.) The molecular dimensions are normal except that the nitrile groups are bent $0.5°$ out of the plane of the ring. The molecules are packed parallel to one another with antiparallel nitrile groups in contact.

Introduction. 526 independent reflections were collected for $0 < \theta \le 24°$ on a Hilger & Watts four-circle automated diffractometer with Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). The 11 most intense reflections appeared to suffer from extinction and were given zero weight in the least-squares calculations; for the 47 reflections for which I_o was < 0, $I = 0$ was used. The crystal used was $0.11 \times 0.32 \times 0.20$ mm; no absorption corrections were made ($\mu = 0.86$ cm⁻¹). The intensities of two check reflections decreased by 20% during the course of the data collection; corrections were made for this slow sublimation or decomposition. The space group was assumed to be $P\bar{1}$ since the molecule should have a center of symmetry. A trial structure was found from Patterson and Fourier maps and refined by full-matrix least squares, with anisotropic thermal parameters for the C and N atoms and isotropic thermal parameters fixed at 5.0 Å² for the H atoms. For details of the experimental arrangements and for the weights, calculations and programs used, see Chow & Britton (1974). The refinement converged with $R = 0.111$; if the 11 intense reflections are omitted, $R = 0.096$; if the 116 weakest reflections are omitted, which corresponds roughly to omitting those reflections with $I < 2\sigma(I)$, $R = 0.069$. The final positional parameters are given in Table 1.*

Table 1. *Positional parameters* ($\times 10^4$)

Estimated standard deviations for the final significant figures are given in parentheses.

	x	y	z
N	3032 (3)	−2799 (3)	3636 (5)
C(1)	1106 (4)	2275 (4)	76 (5)
C(2)	2179 (4)	1311 (4)	1330 (5)
C(3)	1070 (4)	−954 (4)	1244 (5)
C(4)	2171 (4)	−1973 (4)	2558 (6)
H(1)	1863 (47)	3902 (43)	20 (68)
H(2)	3782 (42)	2204 (41)	2259 (61)

Discussion. The bond distances and angles are shown in Fig. 1. A rigid-body analysis was made with the method of Schomaker & Trueblood (1968); the r.m.s. difference between the observed B_{ij} values and those calculated from the rigid-body model is $0 \cdot 15$ Å^2. The corrected and uncorrected bond distances are compared in Table 2. The bond distances are all normal within experimental error. The internal angle opposite the nitrile group in the benzene ring is larger than those opposite the H atoms, in keeping with the greater electronegativity of the nitrile group.

Also shown in Fig. 1 are the deviations from the best plane through the heavy atoms weighted according to their atomic numbers. The molecule deviates slightly, but significantly, from planarity. If the best plane is drawn through the ring atoms only, the ring atoms all lie in the plane to within less than 0·001 Å, but the C and N atoms in the nitrile group are out of this plane by 0·010 and 0·026 Å respectively, which corresponds to a 0·5° bend of the nitrile group out of the plane of the ring. Although we believe that this bend is real, it should
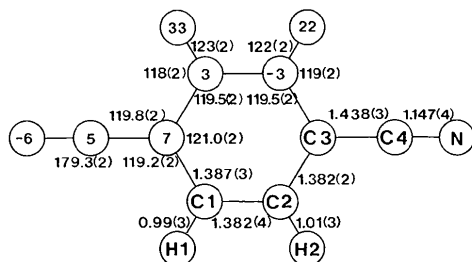
be noted that it might be a systematic error due to the approximate nature of the extinction corrections. To resolve this question convincingly would require data at higher angles, which in turn would require a low-temperature study.

The packing of the molecules is shown in Fig. 2. The molecules are all parallel to each other and occur in layers that are parallel to (10$\bar{1}$); the layers are 3·10 Å apart. The individual molecules are tilted 14° out of the plane of the layers; the perpendicular distance between molecules is 3·49 Å. Within the layers the centers of the molecules approximate very closely a regular hexagonal closest-packed network.

The structure is similar to that of the β form of *p*-dichlorobenzene (Housty & Clastre, 1957), except that end-to-end Cl $\cdots$ Cl contacts within the layers are replaced by side-to-side antiparallel nitrile $\cdots$ nitrile contacts. It is apparently these antiparallel contacts that lead to the greater intermolecular attraction in the dinitrile (nitrile m.p. 222°C; chloride m.p. 53°C). However, a calculation of the Madelung energy to be expected from local charges of +0·15 and −0·15 e on the nitrile C and N atoms, respectively, gives only 400 cal mol^{-1} (Boeyens & Gafner, 1969). If we estimate the entropy of fusion to be 10 cal mol^{-1}(°C)$^{-1}$, this energy would only lead to a difference of 40°C in the melting points. The charges ($\pm 0 \cdot 15$ e) are based on a CNDO
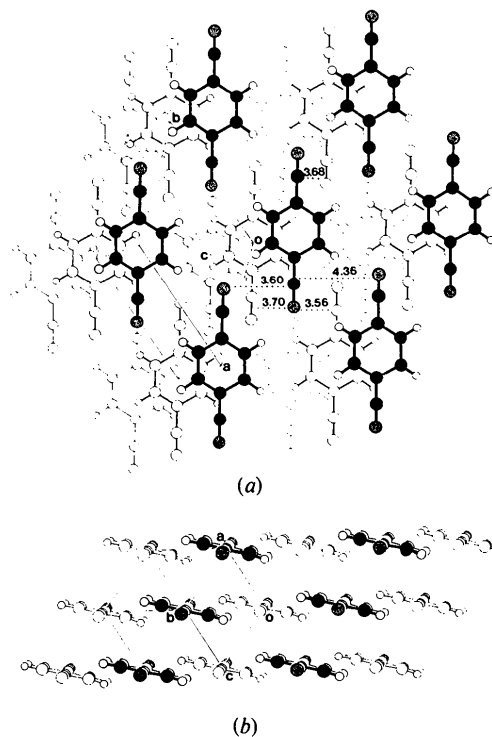

(a)

(b)

Fig. 2. The packing of terephthalonitrile. (a) A view perpendicular to (10$\bar{1}$). (b) A view along [1$\bar{1}$1]. The perpendicular distances (in Å) are shown between some of the parallel and antiparallel nitrile groups.

Fig. 1. Bond distances (Å) and angles (°) in terephthalonitrile. The deviations from the mean plane through the heavy atoms are given, in units of 0·001 Å, inside the circles.

Table 2. *Bond lengths* (Å) *in terephthalonitrile*

	Uncorrected	Corrected for rigid-body motion
C(1)—C(2)	1·382 (4)	1·385
C(2)—C(3)	1·382 (2)	1·391
C(1)—C(3′)	1·387 (3)	1·394
C(3)—C(4)	1·438 (3)	1·441
C(4)—N	1·147 (4)	1·148

molecular-orbital calculation (Segal, 1966; Pople & Beveridge, 1970), which gives +0·087 and −0·166 e for the charges on the C and N respectively. A more extensive experimental and theoretical comparison of the crystal energies of these two compounds would be a worthwhile undertaking.

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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.

The crystal structure of 6b,10b-dihydrobenzo[j]cyclobut[a]acenaphthylene, $C_{18}H_{12}$. Corrigenda. By A. C.

HAZELL and R. G. HAZELL, *Department of Inorganic Chemistry, Aarhus University, DK-8000 Aarhus C. Denmark*

A printer's error in the paper by Hazell & Hazell [*Acta Cryst.* (1977), B33, 360–365] is corrected: In Table 2 the fractional atomic coordinates (reported as multiplied by 10E) are multiplied by 10^4. Also, in Table 3 the units of T should be $Å^2 \times 10^{-4}$ and those of L, $(°)^2$.

All the relevant information is given in the abstract.

Bis(bis-η-cyclopentadienylmolybdenumdi-μ-oxo)phosphorus hexafluorophosphate: erratum. By KEITH

PROUT, M. CLAIRE COULDWELL and ROGER A. FORDER, *Chemical Crystallography Laboratory, Oxford University, 9 Parks Road, Oxford, OX1 3PD, England*

The abstract of Prout, Couldwell & Forder [*Acta Cryst.* (1977), B33, 218–221] contains a number of errors. The beginning of the abstract should read: **Abstract.** $[(C_5H_5)_2MoO_2PO_2Mo(C_5H_5)_2]PF_6$, $C_{20}H_{20}O_4F_6P_2Mo_2$, $M_r = 692·2$. Monoclinic, C2/c, $a = 17·079$ (9), $b = 23·619$ (13), $c = 12·399$ (7) Å, $\beta = 117·02$ (2)°, $U = 4455·7$ Å³, $D_c = 2·06$ g cm⁻³ for $Z = 8$. Mo Kα radiation, $\lambda = 0·71069$ Å, $\mu = 11$ cm⁻¹. The structure of the cation...

All information is given in the abstract.