Two cations show N—H···Cl contacts of about 3·20 Å corresponding to hydrogen bonds of intermediate strength. These cations point with their methyl groups towards the next $Sb_2Cl_9^{3-}$ layers and are expected to constitute the bonding in the **a** direction by electrostatic interactions. The third cation which occupies the free space in a twelve-membered $(\text{—Sb—Cl—})_6$ ring shows a very weak bifurcated hydrogen bond. The large thermal parameters show this cation to be disordered.

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Structure of (6,13-Diacetyl-5,12-dimethyl-1,4,8,11-tetraazacyclotetradeca-4,6,11,13-tetraenato)nickel(II) 7,7,8,8-Tetracyano-*p*-quinodimethane (1/1), $C_{16}H_{22}N_4NiO_2.C_{12}H_4N_4$*

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Abstract. $M_r = 565·3$, monoclinic, $P2_1/n$, $a = 7·111\,(1)$, $b = 14·191\,(3)$, $c = 13·062\,(4)$ Å, $\beta = 93·21\,(2)°$, $V = 1316·0\,(5)$ Å^3, $Z = 2$, $D_x = 1·43$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 0·776$ mm^{-1}, $F(000) = 588$, $T = 293$ K, final $R = 0·045$ for 1405 observed reflections. The nickel(II) ion lies in a square-planar environment surrounded by the four N atoms of the tetradentate macrocyclic ring [Ni—N distances are 1·828 (3) and 1·887 (3) Å]. The structure consists of mixed stacks of neutral DADMTANi and TCNQ molecules. The molecules stack alternately in infinite columns, parallel to the *a* axis, the spacing and dihedral angle between the mean planes being 3·45 (5) Å and 6·1 (5)° respectively. The electrical conductivity of the title complex measured along the shortest axis of the crystal is 0·01 Ω^{-1} m^{-1}, which is larger than the value obtained for iodine-doped DADMTANi.

Introduction. Much attention has been focused on the study of materials that, by virtue of their crystalline packing arrangements, exhibit anisotropy in certain intensive variables including electrical, optical and magnetic behaviour (Ferraro, 1982). Among these systems are macrocyclic complexes that exhibit upon doping an increase in their electrical conductivity (Petersen, Schramm, Stojakovic, Hoffman & Marks, 1977; Phillips & Hoffman, 1977) and a great variety of complexes formed with TCNQ whose properties vary from insulators to conductors. Most of these complexes are purely organic; few examples are reported that contain an inorganic complex (Inoue & Inoue, 1983).

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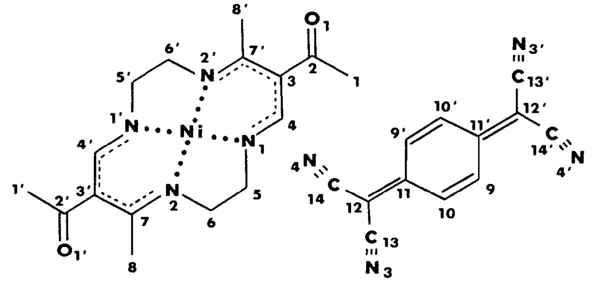
We have now prepared the title compound, DADMTANi–TCNQ, measured its room-temperature electrical conductivity and determined its crystal structure. The electrical conductivity of DADMTANi increases with iodine doping (López-Morales & Bukowski, 1981).



Experimental. Preparation by mixing hot solutions of nickel complex and of TCNQ in chloroform and slowly cooling to room temperature. DADMTANi prepared as described by Jäger (1968). Black prismatic crystal $0\cdot15 \times 0\cdot23 \times 0\cdot44$ mm. Nicolet *R*3 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from 25 machine-centred reflections with $6\cdot4 < 2\theta < 23\cdot9°$, 1886 reflections with $3 < 2\theta < 45°$ for two octants, 1405 independent with $I > 2\cdot5\sigma(I)$, $R_{\text{int}} = 0\cdot020$, index range h 0–8, k 0–16, $l \pm 15$, ω-scan mode, variable scan speed, scan width $1\cdot0°$ (θ), two standard reflections monitored every 50 measurements, Lp correction, empirical absorption correction based on reflection intensity measurements at different azimuthal angles, transmission range 0·836–1·0. Structure solved by heavy-atom method. Least-squares refinement of all non-H atoms treated anisotropically; H atoms riding on bonded C with fixed isotropic temperature factor, $U = 0\cdot06$ Å^2. $\sum w|\Delta F|^2$ minimized with $w = 1/[\sigma^2(F_o) + 0\cdot001(F_o)^2]$; in final cycle $\Delta/\sigma \leq 0\cdot4$, residual electron density within $\pm 0\cdot4$ e Å^{-3}. Final $R = 0\cdot045$, $wR = 0\cdot055$. Scattering factors from *International Tables for X-ray Crystallography* (1974). Isotropic extinction parameter $X = 0\cdot00187$. All computations performed on a Nova 4 computer and plots drawn on a Tektronix plotter with *SHELXTL* (Sheldrick, 1981).

Discussion. Final positional parameters and equivalent isotropic temperature factors are given in Table 1.* The molecular structure with numbering is illustrated in Fig. 1. Bond lengths and angles for non-H atoms are listed in Table 2.

The asymmetric unit comprises half of a DADMTANi molecule and half of a TCNQ molecule with the other halves related by the centres of symmetry coinciding with Ni for DADMTANi and the centre of the ring for TCNQ.

Fig. 2 shows a general view of the structure along **a**.* The structure consists of stacks in which DADMTANi and TCNQ molecules alternate in infinite columns parallel to the *a* axis.

The NiN_4 coordination is required to be planar by the crystallographic inversion centre. However, the two independent Ni—N bond lengths are significantly different. This difference may arise because of interactions between neighbouring molecules in the stacks.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$) *for non-hydrogen atoms*

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
Ni	5000	10000	0	32 (1)
N(1)	4958 (4)	8803 (2)	516 (2)	36 (1)
N(2)	5445 (4)	10420 (2)	1363 (2)	34 (1)
N(3)	1522 (6)	1342 (3)	3178 (3)	69 (2)
N(4)	246 (7)	3270 (3)	608 (4)	78 (2)
O(1)	5270 (5)	6794 (2)	−2377 (2)	70 (1)
C(1)	4896 (8)	6107 (3)	−778 (4)	65 (2)
C(2)	4972 (6)	6961 (3)	−1478 (3)	46 (2)
C(3)	4746 (5)	7903 (3)	−1073 (3)	38 (1)
C(4)	4895 (6)	8013 (3)	7 (3)	42 (1)
C(5)	4947 (6)	8752 (3)	1638 (3)	42 (1)
C(6)	5878 (6)	9623 (3)	2073 (3)	42 (1)
C(7)	5595 (5)	11286 (3)	1726 (3)	38 (1)
C(8)	6262 (6)	11430 (3)	2832 (3)	49 (2)
C(9)	141 (6)	−893 (3)	442 (3)	46 (2)
C(10)	473 (6)	−145 (3)	1040 (3)	44 (2)
C(11)	350 (6)	803 (3)	633 (3)	39 (1)
C(12)	647 (6)	1571 (3)	1260 (3)	45 (1)
C(13)	1143 (6)	1456 (3)	2328 (4)	50 (2)
C(14)	441 (7)	2513 (4)	895 (4)	53 (2)

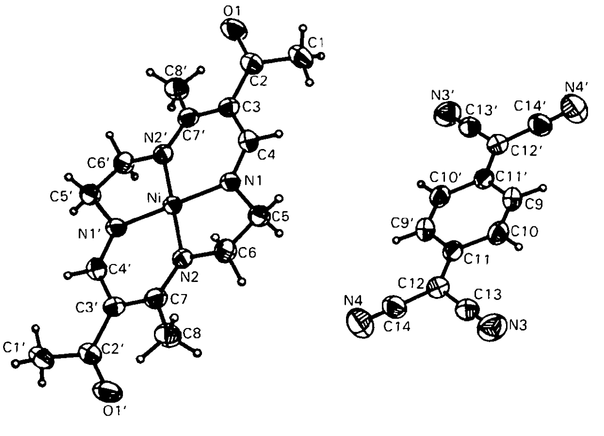


Fig. 1. The molecular conformation of DADMTANi–TCNQ, showing atom numbering. The thermal ellipsoids are drawn at 50% probability level.

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* Lists of structure amplitudes, anisotropic thermal parameters, calculated H-atom coordinates and least-squares planes calculations and a figure showing the structure projected along **b** have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42084 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* See deposition footnote.

The macrocyclic ring is not planar. It contains two five- and two six-membered rings. The five-membered ring [Ni—N(1)—C(5)—C(6)—N(2)] approximates more closely to a half-chair conformation than to an envelope. A pseudo-diad runs through Ni and the midpoint of the C(5)—C(6) bond. The mean of the five endocyclic torsion-angle moduli is 21·7°. The torsion angle around the C(5)—C(6) ethylene bridge is 33·8 (4)° and these two atoms are displaced by −0·178 (6) and 0·183 (6) Å from the plane of the five-membered ring. The pseudorotational angle (Altona, Geise & Romers, 1968) is $\Delta = 1{\cdot}7\ (4)°$ and $\varphi_m = 33{\cdot}8\ (4)°$ for this five-membered ring. The six-membered ring [Ni—N(2′)—C(7′)—C(3)—C(4)—N(1)] is essentially planar; maximum deviation is 0·077 (6) Å for C(3). The mean of the six endocyclic torsion-angle moduli is 6·8°. The distribution of bond lengths in this six-membered ring indicates extensive delocalization of the π electrons.

Table 2. *Bond lengths* (Å) *and angles* (°) *for non-hydrogen atoms*

The e.s.d.'s are given in parentheses.

Ni—N(1)	1·828 (3)	Ni—N(2)	1·887 (3)
N(1)—C(4)	1·303 (5)	N(1)—C(5)	1·468 (5)
N(2)—C(6)	1·485 (5)	N(2)—C(7)	1·320 (5)
N(3)—C(13)	1·140 (6)	N(4)—C(14)	1·143 (7)
O(1)—C(2)	1·229 (6)	C(1)—C(2)	1·520 (6)
C(2)—C(3)	1·450 (6)	C(3)—C(4)	1·417 (6)
C(3)—C(7′)	1·445 (6)	C(5)—C(6)	1·499 (6)
C(7)—C(8)	1·509 (5)	C(11)—C(12)	1·372 (6)
C(9)—C(10)	1·331 (6)	C(12)—C(13)	1·428 (6)
C(10)—C(11)	1·447 (6)		
C(11)—C(9′)	1·434 (6)		
C(12)—C(14)	1·425 (7)		
N(1)—Ni—N(2)	87·2 (1)	N(1)—Ni—N(1′)	180·0
N(2)—Ni—N(2′)	180·0	N(1)—Ni—N(2′)	92·8 (1)
Ni—N(1)—C(4)	127·8 (3)	Ni—N(1)—C(5)	114·5 (2)
C(4)—N(1)—C(5)	117·7 (3)	Ni—N(2)—C(6)	111·5 (2)
Ni—N(2)—C(7)	129·7 (3)	C(6)—N(2)—C(7)	118·4 (3)
O(1)—C(2)—C(1)	115·8 (4)	O(1)—C(2)—C(3)	123·7 (4)
C(1)—C(2)—C(3)	120·4 (4)	C(2)—C(3)—C(4)	117·5 (4)
C(2)—C(3)—C(7′)	122·5 (3)	C(4)—C(3)—C(7′)	120·0 (4)
N(1)—C(4)—C(3)	127·0 (4)	N(1)—C(5)—C(6)	108·1 (3)
N(2)—C(6)—C(5)	108·6 (3)	N(2)—C(7)—C(8)	119·0 (3)
N(2)—C(7)—C(3′)	121·5 (3)	C(8)—C(7)—C(3′)	119·4 (4)
C(10)—C(9)—C(11′)	122·0 (4)	C(9)—C(10)—C(11)	121·4 (4)
C(10)—C(11)—C(12)	121·0 (4)	C(10)—C(11)—C(9′)	116·6 (4)
C(12)—C(11)—C(9′)	122·3 (4)	C(11)—C(12)—C(13)	120·9 (4)
C(11)—C(12)—C(14)	122·4 (4)	C(13)—C(12)—C(14)	116·7 (4)
N(3)—C(13)—C(12)	178·2 (5)	N(4)—C(14)—C(12)	178·8 (5)

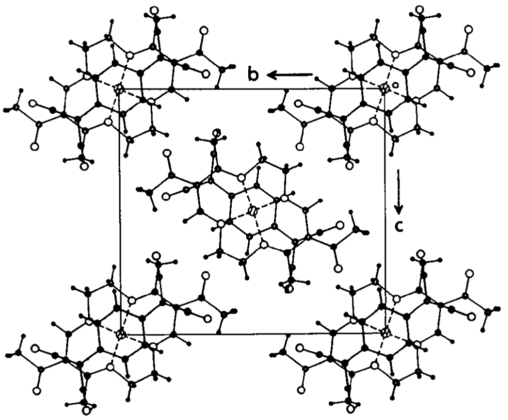

Fig. 2. Projection of the structure along **a**.

The molecular geometry of TCNQ, as shown in Table 2 and in Fig. 1, suggests, by comparison with TCNQ dimensions in other structures (Hoekstra, Spoelder & Vos, 1972; Goldberg & Shmueli, 1973; Ashwell, Eley, Wallwork & Willis, 1975), that TCNQ in the present structure is neutral. The quinonoid bond distance, C(11)—C(12) [1·372 (6) Å], agrees with that theoretically predicted for the neutral TCNQ molecule (Lowitz, 1967; Jonkman & Kommandeur, 1972). The TCNQ molecule is not quite planar, the $C-(CN)_2$ groups being bent slightly out of the plane of the ring, deviations from the mean plane being −0·187 (6) Å for N(4) and −0·033 (6) Å for N(3).

The arrangement of molecules in the crystal appears to be determined by van der Waals interactions.

The fact that DADMTANi and TCNQ molecules stack alternately is consistent with the relatively low electrical conductivity along **a** (0·01 Ω^{-1} m^{-1}), since it is well known that highly conducting compounds like 2,2′-bis(1,3-dithiole) (TTF)—TCNQ form homologous columnar stacks and that compounds with mixed stacks exhibit low conductivity (Hatfield, 1979).

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