C11—C2—O11	108.6 (4)	107.2 (2)
C4—C5—O11	105.6 (4)	106.2 (2)
C3—C2—O11	103.1 (3)	104.2 (1)
C4—C5—C6	114.4 (4)	112.2 (2)
C2—O11—C5	110.2 (3)	109.8 (2)
C6—C5—O11	107.9 (4)	109.6 (2)
C5—C6—I	109.3 (3)	
C5—C6—N7		116.5 (2)
C6—N7—C8		110.0 (2)
C6—N7—C9		110.7 (2)
C6—N7—C10		106.4 (2)
C8—N7—C9		110.8 (2)
C8—N7—C10		109.3 (2)
C9—N7—C10		109.6 (2)
C5—O11—C2—C3	25.2 (4)	15.5 (2)
O11—C2—C3—C4	−38.9 (4)	−35.1 (2)
C2—C3—C4—C5	38.0 (4)	40.6 (2)
C3—C4—C5—O11	−23.8 (4)	−32.7 (2)
C4—C5—O11—C2	−1.2 (4)	10.7 (2)
C2—O11—C5—C6	121.6 (4)	132.1 (2)
O11—C5—C6—X	175.3 (3) (X = I)	87.8 (2) (X = N7)

Hydrogen bond for (2)

$A—B\cdots C$	$A\cdots C$	$B\cdots C$	$A—B\cdots C$
O12—H012$\cdots$I^{i}	3.416 (2)	2.50 (4)	153 (3)

Symmetry code: (i) $x, \frac{3}{2} - y, \frac{1}{2} + z$.

Table 3. *Pseudorotational parameters* (°) *for all published muscarine ions and compounds* (1) *and* (2)

The pseudorotational parameters were calculated by *PLATON* (Spek, 1991) according to the method described by Rao, Westhof & Sundaralingam (1981).

Compound	Phase	Amplitude	$T-E$
(1)	340.5 (4)	40.8 (3)	E_3
(2)	356.8 (2)	41.5 (1)	$^4T_3$
MUSCAI^a	31.6	26.6	$^4T_5$
MUSCHL^b	169.6 (1)	39.2 (1)	^3E
MUSPIC^c	−5.7 (1)	41.5 (1)	$^4T_3$
MUSTPB^d	0 (2)	36.3 (9)	$^4T_3$

Notes: (*a*) muscarine iodide (Jellinek, 1957); (*b*) muscarine chloride (Frydenvang & Jensen, 1990); (*c*) muscarine picrate (Frydenvang & Jensen, 1993); (*d*) muscarine tetraphenylborate (Frydenvang & Jensen, 1993).

Single crystals of (1) were obtained from a solution of the compound in 2-propanol. Single crystals of compound (2) were obtained by vapour diffusion of dimethoxymethane into a solution of the compound in methanol. The parameters of the benzene-ring H atoms in (1) and the H atoms of the aromatic rings and the quaternary ammonium group in (2) were not refined. Only the coordinates of the remaining H atoms were refined. In (2), high difference electron density is observed close to the iodide ion. Data reduction: *SDP BEGIN* (B. A. Frenz & Associates, Inc., 1982). Program(s) used to solve structure: *SDP PATTERSON, DIRDIF.* Program(s) used to refine structure: *SDP LSFM.* Molecular graphics: *ORTEP* (Johnson, 1976).

The two compounds were kindly donated by Dr M. De Amici, University of Milano, Italy.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71300 (61 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1063]

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Structure of the Charge-Transfer Complexes 2,4,5,7-Tetranitro-9-fluorenone–1-Ethylnaphthalene (1/1) (I) and 2,4,5,7-Tetranitro-9-fluorenone–3,6-Dimethylphenanthrene (1/1) (II)

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Abstract

In each structure the asymmetric unit consists of two molecules whose parallel rings are at van der Waals distances and stack, alternately, along the *a* axis for (I) and along the *n*-glide direction for (II). The carbon-skeleton geometry of the fluorenone within both charge-transfer complexes is not significantly altered from that of the parent 2,4,5,7-tetranitro-9-fluorenone.

Comment

As a continuation of a study in this laboratory of a series of $\pi-\pi^*$ charge-transfer complexes (Baughman, 1982, 1987; Shah & Baughman, 1993), the charge-transfer complexes of 2,4,5,7-tetranitro-9-fluorenone (TNF) with 1-ethylnaphthalene (1-ETNP) and 3,6-dimethylphenanthrene (3,6-DMP) were investigated. As a result of somewhat high B values for C24 and C25 in TNF:1-ETNP, a separate refinement assuming isotropic thermal parameters for C24 and C25 was performed. This produced no significant changes in bond distances and angles than noted when all non-H atoms were assumed to have anisotropic thermal parameters.

The average distances between the π systems of the nearly parallel molecules for TNF:1-ETNP and TNF:3,6-DMP are 3.45 (7) and 3.4 (2) Å, respectively, *i.e* the van der Waals distance (3.40 Å; Pauling, 1960). Rings in 1-ETNP, as well as the middle and end rings in the 3,6-DMP molecules, tend to be directly above the five- and six-membered rings in the TNF acceptor molecule. The asymmetric units stack as alternating acceptor/donor molecules generally along the *a* axis in TNF:1-ETNP and along the *n*-glide direction in TNF:3,6-DMP.

Distances and plane angles in the TNF portion of the title compounds are not significantly different from those in the parent TNF (Baughman, 1987). Since both of the complexes and the pure TNF crystals (Baughman, 1987) have centrosymmetric space groups, both + and − values of a torsional angle will be present in the unit cell. So, comparisons need to be made with either/both values. In addition, the asymmetric unit of TNF contains two conformers. The C5—C12—C11—C4 and C13—C12—C11—C10 torsional angles in the five-membered ring of TNF in TNF:1-ETNP have values of ±12 (1) and ±9.7 (7)°, respectively, and values of ±8 (1) and ±3.3 (7)° in the TNF portion of TNF:3,6-DMP. This set of torsional angles in the 1-ETNP complex compares more favorably with the corresponding angles in the *A* molecule of TNF [±17 (1) and ±8.5°, respectively]. However, the set of angles in the 3,6-DMP complex compares better with the TNF molecule *B* [±7.5 (9) and ±5.1°]. Future crystallographic and quantum-mechanical work in this series of compounds should help to demonstrate any patterns from which a better understanding of charge-transfer bonding may evolve.

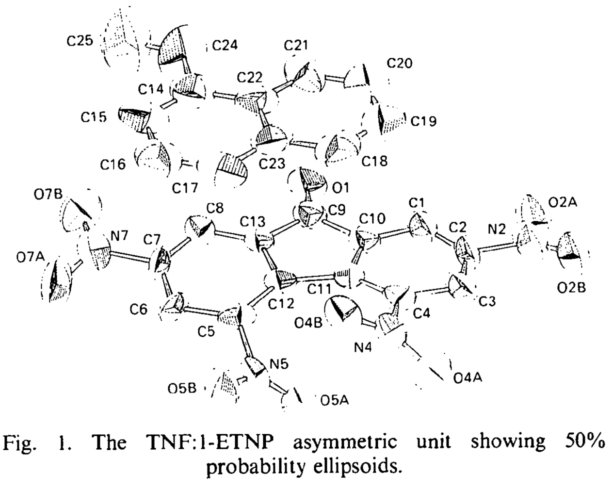

Fig. 1. The TNF:1-ETNP asymmetric unit showing 50% probability ellipsoids.

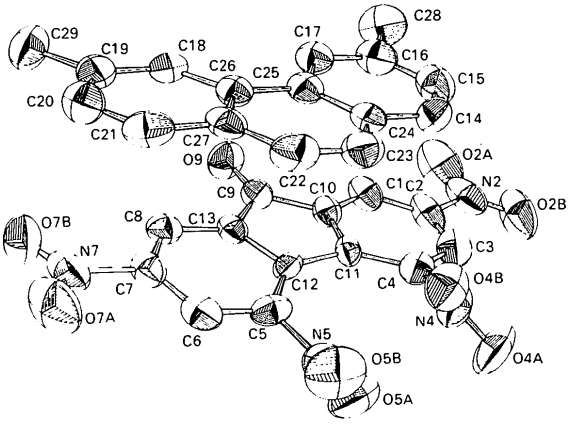

Fig. 2. The TNF:3,6-DMP asymmetric unit showing 50% probability ellipsoids.

Experimental

Compound (I)

Crystal data

$C_{13}H_4N_4O_9.C_{12}H_{12}$
M_r = 516.42
Monoclinic
$P2_1/c$
a = 7.131 (1) Å
b = 10.852 (4) Å
c = 29.553 (8) Å
β = 91.85 (2)°
V = 2286 (1) Å^3
Z = 4
D_x = 1.499 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 25 reflections
θ = 10.1-26.6°
μ = 1.005 mm^{-1}
T = 290 K
Prism
0.21 × 0.15 × 0.14 mm
Deep red
Crystal source: 1:1 mol mixture of parent compounds in 1:1 toluene-acetone

Data collection

Siemens $P3$ diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6348 measured reflections
3174 independent reflections
1745 observed reflections [$F \geq 3\sigma(F)$ and $I \geq 3\sigma(I)$]
R_{int} = 0.079
$\theta_{\max}$ = 55°
$h = 0 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -32 \rightarrow 32$
3 standard reflections monitored every 50 reflections intensity variation: ±7%

Refinement

Refinement on F
Final R = 0.063
wR = 0.046
S = 3.77
1745 reflections
343 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max}$ = 0.01
$\Delta\rho_{\max}$ = 0.3 e Å^{-3}
$\Delta\rho_{\min}$ = −0.1 e Å^{-3}
Atomic scattering factors from Hanson, Herman, Lea & Skillman (1964) for non-H atoms and from Stewart, Davidson & Simpson (1965) for H atoms

Compound (II)

Crystal data

$C_{13}H_4N_4O_9.C_{16}H_{14}$
$M_r = 566.48$
Monoclinic
$P2_1/n$
$a = 12.840\ (4)$ Å
$b = 15.072\ (4)$ Å
$c = 14.439\ (3)$ Å
$\beta = 114.54\ (2)°$
$V = 2542\ (1)$ Å^3
$Z = 4$
$D_x = 1.480$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å

Cell parameters from 25 reflections
$\theta = 12.0–44.7°$
$\mu = 0.963$ mm^{-1}
$T = 290$ K
Prism
0.15 × 0.13 × 0.10 mm
Deep red
Crystal source: 1:1 mol mixture of parent compounds in 1:1 toluene–acetone

Data collection

Siemens *P*3 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6752 measured reflections
3376 independent reflections
1441 observed reflections [$F \geq 3\sigma(F)$ and $I \geq 3\sigma(I)$]
$R_{\rm int} = 0.054$

$\theta_{\rm max} = 55°$
$h = 0 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 15$
3 standard reflections monitored every 50 reflections
intensity variation: ±4%

Refinement

Refinement on F
Final $R = 0.050$
$wR = 0.031$
$S = 1.90$
1441 reflections
379 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max} = 0.01$

$\Delta\rho_{\rm max} = 0.2$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.1$ e Å^{-3}
Atomic scattering factors from Hanson, Herman, Lea & Skillman (1964) for non-H atoms and from Stewart, Davidson & Simpson (1965) for H atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$B_{\rm eq}$
Compound (I)				
O1	0.6954 (7)	1.4072 (3)	0.0452 (1)	5.7
O2*A*	0.4573 (8)	0.9390 (4)	0.0444 (2)	9.2
O2*B*	0.4926 (9)	0.8423 (5)	0.1072 (2)	9.0
O4*A*	0.6398 (7)	1.0670 (4)	0.2451 (1)	5.7
O4*B*	0.8692 (6)	1.1938 (4)	0.2339 (1)	4.9
O5*A*	0.5416 (6)	1.3284 (4)	0.2460 (1)	4.8
O5*B*	0.7488 (7)	1.4402 (4)	0.2830 (1)	6.0
O7*A*	0.8463 (7)	1.8240 (4)	0.2042 (2)	6.7
O7*B*	0.9238 (9)	1.8156 (4)	0.1340 (2)	8.8
N2	0.5007 (9)	0.9342 (6)	0.0841 (2)	7.3
N4	0.7316 (9)	1.1365 (5)	0.2213 (2)	4.5
N5	0.6725 (8)	1.4027 (5)	0.2478 (2)	4.8
N7	0.868 (1)	1.7711 (5)	0.1679 (3)	6.3
C1	0.5934 (8)	1.1527 (6)	0.0808 (2)	4.5
C2	0.5669 (8)	1.0482 (6)	0.1071 (2)	4.1
C3	0.6061 (8)	1.0439 (5)	0.1525 (2)	3.6
C4	0.6640 (8)	1.1498 (5)	0.1743 (2)	3.3
C5	0.7316 (8)	1.4546 (5)	0.2045 (2)	3.6
C6	0.7812 (9)	1.5784 (5)	0.2062 (2)	4.1
C7	0.8114 (9)	1.6372 (6)	0.1665 (2)	4.3
C8	0.7920 (9)	1.5826 (5)	0.1243 (2)	4.6
C9	0.6989 (9)	1.3798 (5)	0.0843 (2)	4.9
C10	0.6486 (8)	1.2572 (5)	0.1044 (2)	3.5
C11	0.6789 (8)	1.2605 (5)	0.1510 (2)	3.0
C12	0.7208 (8)	1.3912 (5)	0.1642 (2)	3.2
C13	0.7450 (8)	1.4580 (5)	0.1244 (2)	3.6
C14	0.870 (2)	0.7516 (8)	0.4433 (2)	6.6
C15	0.902 (1)	0.6433 (8)	0.4210 (3)	6.3
C16	0.879 (1)	0.6316 (8)	0.3738 (3)	7.2
C17	0.833 (1)	0.7312 (9)	0.3479 (2)	5.8
C18	0.757 (1)	0.9496 (9)	0.3437 (3)	7.1
C19	0.721 (1)	1.0583 (9)	0.3642 (4)	7.8
C20	0.738 (1)	1.0704 (8)	0.4111 (4)	7.4
C21	0.782 (1)	0.9703 (8)	0.4379 (2)	6.4
C22	0.8206 (9)	0.8555 (7)	0.4171 (2)	5.1
C23	0.8023 (9)	0.8455 (7)	0.3696 (2)	5.5
C24	0.884 (2)	0.7616 (8)	0.4938 (3)	10.3
C25	0.943 (2)	0.6690 (9)	0.5180 (3)	14.8
Compound (II)				
O2*A*	0.4719 (6)	0.9124 (4)	−0.0999 (5)	8.2
O2*B*	0.4866 (5)	0.7830 (4)	−0.1615 (4)	7.7
O4*A*	0.3823 (4)	0.5173 (3)	−0.0220 (4)	7.3
O4*B*	0.4745 (4)	0.5270 (3)	0.1433 (4)	5.6
O5*A*	0.2402 (4)	0.5207 (3)	0.0939 (4)	6.3
O5*B*	0.3358 (4)	0.4586 (3)	0.2417 (4)	6.4
O7*A*	0.2340 (4)	0.6237 (4)	0.4854 (4)	7.3
O7*B*	0.2278 (5)	0.7672 (4)	0.4795 (4)	6.2
O9	0.3623 (4)	0.9178 (3)	0.2139 (3)	4.4
N2	0.4687 (6)	0.8306 (5)	−0.1030 (5)	5.1
N4	0.4210 (6)	0.5598 (4)	0.0585 (5)	6.5
N5	0.2942 (5)	0.5226 (4)	0.1868 (5)	5.5
N7	0.2425 (5)	0.6953 (5)	0.4500 (5)	5.5
C1	0.4133 (6)	0.8413 (4)	0.0420 (5)	4.1
C2	0.4376 (6)	0.7868 (6)	−0.0241 (6)	3.2
C3	0.4367 (6)	0.6978 (6)	−0.0195 (5)	4.2
C4	0.4079 (6)	0.6566 (4)	0.0521 (5)	3.6
C5	0.3054 (5)	0.6098 (4)	0.2374 (5)	3.8
C6	0.2763 (5)	0.6121 (5)	0.3184 (5)	3.4
C7	0.2741 (5)	0.6923 (5)	0.3622 (5)	2.5
C8	0.2978 (5)	0.7713 (4)	0.3259 (5)	3.1
C9	0.3588 (6)	0.8402 (5)	0.1933 (5)	4.0
C10	0.3850 (5)	0.7987 (4)	0.1130 (5)	3.2
C11	0.3767 (5)	0.7063 (5)	0.1184 (5)	2.0
C12	0.3378 (5)	0.6850 (4)	0.1997 (4)	2.5
C13	0.3296 (5)	0.7650 (4)	0.2458 (5)	2.8
C14	0.6847 (6)	0.6741 (5)	0.2629 (6)	4.5
C15	0.7012 (6)	0.7533 (7)	0.2243 (5)	4.4
C16	0.6801 (6)	0.8340 (5)	0.2622 (6)	4.9
C17	0.6433 (6)	0.8323 (4)	0.3393 (5)	3.7
C18	0.5523 (5)	0.8267 (4)	0.4994 (5)	2.8
C19	0.5120 (5)	0.8231 (5)	0.5739 (5)	4.0
C20	0.5035 (5)	0.7396 (5)	0.6135 (4)	3.6
C21	0.5327 (6)	0.6647 (4)	0.5772 (6)	3.6
C22	0.6020 (6)	0.5881 (4)	0.4627 (6)	3.4
C23	0.6346 (6)	0.5902 (5)	0.3872 (6)	4.2
C24	0.6481 (5)	0.6722 (5)	0.3424 (5)	3.9
C25	0.6249 (5)	0.7527 (5)	0.3808 (5)	3.3
C26	0.5852 (5)	0.7506 (5)	0.4601 (5)	3.0
C27	0.5741 (5)	0.6676 (5)	0.5007 (5)	4.0
C28	0.7043 (6)	0.9203 (5)	0.2213 (5)	5.1
C29	0.4758 (6)	0.9063 (4)	0.6118 (5)	4.1

Table 2. *Interatomic distances* (Å), *bond angles* (°) *and torsion angles* (°)

Signs for torsion angles as per Klyne & Prelog (1960).

Compound (I)			
O1—C9	1.192 (6)	C7—C8	1.382 (7)
O2*A*—N2	1.204 (6)	C8—C13	1.394 (6)
O2*B*—N2	1.211 (6)	C9—C10	1.505 (6)

O4*A*—N4	1.232 (5)	C9—C13	1.487 (7)
O4*B*—N4	1.210 (5)	C10—C11	1.387 (6)
O5*A*—N5	1.233 (5)	C11—C12	1.499 (6)
O5*B*—N5	1.226 (5)	C12—C13	1.396 (6)
O7*A*—N7	1.229 (6)	C14—C15	1.370 (8)
O7*B*—N7	1.192 (6)	C14—C22	1.405 (8)
N2—C2	1.481 (7)	C14—C24	1.496 (8)
N4—C4	1.463 (6)	C15—C16	1.404 (9)
N5—C5	1.473 (6)	C16—C17	1.359 (9)
N7—C7	1.509 (7)	C17—C23	1.416 (8)
C1—C2	1.391 (6)	C18—C19	1.354 (9)
C1—C10	1.381 (6)	C18—C23	1.396 (9)
C2—C3	1.363 (7)	C19—C20	1.40 (1)
C3—C4	1.376 (6)	C20—C21	1.375 (9)
C4—C11	1.391 (6)	C21—C22	1.419 (8)
C5—C6	1.390 (6)	C22—C23	1.410 (7)
C5—C12	1.376 (6)	C24—C25	1.30 (1)
C6—C7	1.360 (7)		

C2—N2—O2*A*	118.6 (6)	C1—C10—C9	126.4 (5)
C2—N2—O2*B*	116.7 (6)	C1—C10—C11	123.7 (5)
O2*A*—N2—O2*B*	124.7 (7)	C4—C11—C10	117.3 (5)
C4—N4—O4*A*	115.9 (5)	C4—C11—C12	134.9 (5)
C4—N4—O4*B*	118.6 (5)	C10—C11—C12	107.9 (5)
O4*A*—N4—O4*B*	125.5 (5)	C5—C12—C11	134.7 (5)
C5—N5—O5*A*	116.7 (5)	C11—C12—C13	107.5 (5)
C5—N5—O5*B*	118.6 (5)	C5—C12—C13	117.7 (5)
O5*A*—N5—O5*B*	124.6 (6)	C8—C13—C9	126.8 (5)
C7—N7—O7*A*	115.7 (7)	C8—C13—C12	122.8 (5)
C7—N7—O7*B*	117.5 (7)	C9—C13—C12	110.2 (5)
O7*A*—N7—O7*B*	126.7 (7)	C15—C14—C22	117.8 (7)
C10—C1—C2	115.4 (5)	C15—C14—C24	122.3 (8)
C1—C2—C3	123.4 (5)	C22—C14—C24	119.9 (7)
C1—C2—N2	118.2 (6)	C14—C15—C16	122.6 (8)
C3—C2—N2	118.4 (6)	C15—C16—C17	120.4 (8)
C2—C3—C4	118.8 (5)	C16—C17—C23	118.6 (7)
C3—C4—N4	116.6 (5)	C19—C18—C23	120.2 (8)
C3—C4—C11	121.0 (5)	C18—C19—C20	121.0 (9)
C11—C4—N4	122.0 (5)	C19—C20—C21	120.5 (8)
C12—C5—N5	123.5 (5)	C20—C21—C22	119.3 (7)
C6—C5—C12	121.5 (5)	C14—C22—C21	120.9 (7)
C6—C5—N5	114.6 (5)	C14—C22—C23	120.0 (7)
C5—C6—C7	117.9 (5)	C21—C22—C23	119.0 (7)
C6—C7—C8	124.3 (5)	C17—C23—C18	119.8 (8)
C6—C7—N7	118.5 (6)	C17—C23—C22	120.4 (7)
C8—C7—N7	117.2 (6)	O1—C9—C13	128.9 (5)
C7—C8—C13	115.5 (5)	O1—C9—C10	127.3 (5)
C10—C9—C13	103.8 (5)	C18—C23—C22	119.7 (8)
C9—C10—C11	109.7 (5)	C14—C24—C25	120 (1)

C1—C2—N2—O2*A*	−6 (1)	C6—C5—N5—O5*A*	141.7 (5)
C1—C2—N2—O2*B*	174.5 (7)	C6—C5—N5—O5*B*	−34.7 (7)
C3—C2—N2—O2*A*	176.5 (7)	C6—C7—N7—O7*A*	−14 (1)
C3—C2—N2—O2*B*	−3 (1)	C6—C7—N7—O7*B*	167.8 (7)
C3—C4—N4—O4*A*	−42.1 (7)	C8—C7—N7—O7*A*	165.4 (6)
C3—C4—N4—O4*B*	138.4 (5)	C8—C7—N7—O7*B*	−13 (1)
C11—C4—N4—O4*A*	145.6 (5)	C15—C14—C24—C25	6 (2)
C11—C4—N4—O4*B*	−33.8 (8)	C22—C14—C24—C25	−175 (1)
C12—C5—N5—O5*A*	−31.7 (8)	C13—C12—C11—C10	−9.7 (7)
C12—C5—N5—O5*B*	152.0 (6)	C5—C12—C11—C4	−12 (1)

Compound (II)

N2—O2*A*	1.233 (7)	C8—C13	1.379 (7)
N2—O2*B*	1.201 (7)	C13—C12	1.403 (8)
N2—C2	1.507 (9)	C13—C9	1.497 (8)
N4—O4*A*	1.235 (6)	C9—C10	1.474 (8)
N4—O4*B*	1.232 (6)	C9—O9	1.202 (7)
N4—C4	1.468 (7)	C14—C15	1.371 (9)
N5—O5*A*	1.228 (6)	C15—C16	1.405 (9)
N5—O5*B*	1.223 (6)	C16—C28	1.513 (8)
N5—C5	1.482 (7)	C16—C17	1.379 (8)
N7—O7*A*	1.219 (7)	C17—C25	1.403 (7)
N7—O7*B*	1.209 (7)	C25—C24	1.417 (8)
N7—C7	1.482 (8)	C24—C14	1.410 (8)
C1—C2	1.390 (8)	C24—C23	1.437 (8)
C2—C3	1.343 (8)	C23—C22	1.321 (8)
C3—C4	1.383 (8)	C22—C27	1.423 (8)
C4—C11	1.398 (7)	C27—C26	1.414 (7)
C11—C10	1.402 (7)	C26—C25	1.435 (7)

C10—C1	1.380 (7)	C27—C21	1.412 (8)
C11—C12	1.490 (7)	C21—C20	1.361 (7)
C12—C5	1.393 (7)	C20—C19	1.406 (8)
C5—C6	1.368 (7)	C19—C29	1.515 (7)
C6—C7	1.370 (7)	C19—C18	1.374 (7)
C7—C8	1.385 (8)	C18—C26	1.420 (7)

C2—N2—O2*A*	115.2 (8)	C8—C13—C9	126.6 (6)
C2—N2—O2*B*	117.2 (8)	C24—C14—C15	120.6 (7)
O2*A*—N2—O2*B*	127.6 (9)	C14—C15—C16	120.6 (7)
C4—N4—O4*A*	117.7 (7)	C15—C16—C28	119.3 (8)
C4—N4—O4*B*	117.6 (6)	C28—C16—C17	121.7 (8)
O4*B*—N4—O4*A*	124.6 (7)	C15—C16—C17	119.0 (7)
C5—N5—O5*A*	117.1 (6)	C16—C17—C25	122.4 (7)
C5—N5—O5*B*	117.0 (6)	C26—C18—C19	123.5 (6)
O5*B*—N5—O5*A*	125.9 (7)	C18—C19—C29	121.4 (7)
C7—N7—O7*A*	115.8 (8)	C29—C19—C20	120.4 (7)
C7—N7—O7*B*	117.9 (7)	C20—C19—C18	118.2 (7)
O7*B*—N7—O7*A*	126.2 (8)	C19—C20—C21	120.4 (6)
C10—C1—C2	116.0 (6)	C20—C21—C27	121.9 (6)
C1—C2—N2	117.7 (8)	C27—C22—C23	121.0 (7)
N2—C2—C3	119.0 (8)	C22—C23—C24	122.1 (7)
C1—C2—C3	123.2 (7)	C23—C24—C14	121.8 (8)
C2—C3—C4	119.7 (7)	C23—C24—C25	118.5 (7)
C3—C4—N4	115.7 (7)	C25—C24—C14	119.7 (7)
N4—C4—C11	123.2 (7)	C17—C25—C26	122.6 (8)
C3—C4—C11	120.9 (6)	C7—C8—C13	116.5 (6)
C12—C5—N5	120.8 (7)	C13—C9—O9	127.0 (7)
N5—C5—C6	116.0 (7)	O9—C9—C10	127.7 (7)
C12—C5—C6	123.0 (7)	C13—C9—C10	105.3 (6)
C5—C6—C7	118.9 (6)	C9—C10—C1	127.1 (6)
C6—C7—N7	119.2 (7)	C9—C10—C11	109.3 (6)
N7—C7—C8	118.6 (7)	C11—C10—C1	123.6 (6)
C6—C7—C8	122.2 (6)	C17—C25—C24	117.8 (6)
C10—C11—C12	108.4 (6)	C26—C25—C24	119.6 (7)
C10—C11—C4	116.4 (6)	C25—C26—C18	124.4 (8)
C12—C11—C4	135.2 (7)	C25—C26—C27	118.8 (7)
C11—C12—C5	137.3 (7)	C27—C26—C18	116.8 (6)
C5—C12—C13	114.9 (6)	C21—C27—C22	120.8 (7)
C13—C12—C11	107.8 (6)	C21—C27—C26	119.2 (7)
C8—C13—C12	124.3 (6)	C26—C27—C22	120.0 (7)
C12—C13—C9	109.1 (6)		

C1—C2—N2—O2*A*	1 (1)	C12—C5—N5—O5*B*	135.1 (7)
C1—C2—N2—O2*B*	−178.1(7)	C6—C5—N5—O5*A*	129.0 (7)
C3—C2—N2—O2*A*	−177.2 (8)	C6—C5—N5—O5*B*	−48.3 (8)
C3—C2—N2—O2*B*	3 (1)	C6—C7—N7—O7*A*	8.7 (9)
C3—C4—N4—O4*A*	−47 (1)	C6—C7—N7—O7*B*	−171.3 (7)
C3—C4—N4—O4*B*	129.8 (7)	C8—C7—N7—O7*A*	−172.8 (7)
C11—C4—N4—O4*A*	137.8 (7)	C8—C7—N7—O7*B*	7 (1)
C11—C4—N4—O4*B*	−46 (1)	C4—C11—C12—C5	−8 (1)
C12—C5—N5—O5*A*	−47.6 (9)	C10—C11—C12—C13	−3.3 (7)

For structure (I), 37 of the 38 non-H atoms were located by *MULTAN*76 (Main, Woolfson & Germain, 1976) using the coordinates for TNF, a two-dimensional estimate of the coordinates for 1-ETNP, and a *K* curve to compute the *E* values. For structure (II), all 42 non-H atoms were located by *MULTAN*76 using coordinates for TNF, a two-dimensional estimate of the coordinates for 3,6-DMP and a *K* curve to compute the *E* values. Aromatic H atoms were set at 1.00 Å from the respective C atoms while bisecting the C—C—C angle. The alkyl H atoms of (I) and the methyl H atoms of (II) were set in tetrahedral positions at 1.10 Å. For both structures, anisotropic thermal parameters were used for all but the H atoms, which were assigned the B_{eq} value of the C atom to which they are bonded. Programs used: *MULTAN*76; *ALLS* (Lapp & Jacobson, 1979); *FOUR* (Powell & Jacobson, 1980); *ORFFE* (Busing, Martin & Levy, 1964); *ORTEP* (Johnson, 1971).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes data, along with diagrams showing the molecular stacking for both compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71171 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1015]

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Structure of Hydralazine Hydrochloride

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Abstract

The hydrazine side chain of 1(2*H*)-phthalazinone hydrazone hydrochloride is nearly coplanar, with the phthalazine ring having a torsion angle N(2)—C(1)—N(4)—N(5) of −0.4 (5)°. The N(2) atom of the phthalazine ring is protonated and forms an intermolecular hydrogen bond with the amino N atom of the side chain. Another hydrogen bond is formed between the imino N atom of the side chain and the Cl ion.

Comment

1(2*H*)-Phthalazinone hydrazone, also known as hydralazine, is a potent therapeutic drug for hypertension (Perry, 1973). This compound causes site-specific DNA damage (Yamamoto & Kawanishi, 1991) and has mutagenic and/or carcinogenic potency (Toth, 1980; Parodi *et al.*, 1981). A complete structural analysis of this compound therefore seemed very important for understanding its biological function. In this study, the structure of the hydrochloride form of hydralazine has been determined.

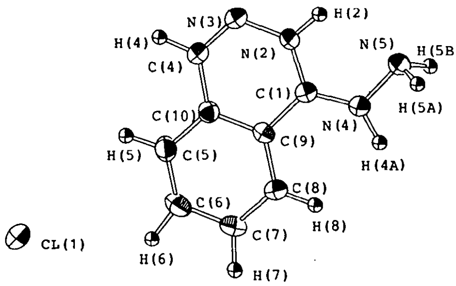

Fig. 1. Perspective view of 1(2*H*)-phthalazinone hydrazone hydrochloride with atomic numbering, viewed down the *x* axis.

Experimental

Crystal data

$C_8H_9N_4^+.Cl^-$
$M_r = 196.64$
Monoclinic
$P2_1/a$
$a = 6.644\ (2)$ Å
$b = 14.531\ (3)$ Å
$c = 9.413\ (1)$ Å
$\beta = 103.57\ (2)°$
$V = 883.4\ (3)$ Å^3
$Z = 4$
$D_x = 1.478$ Mg m^{-3}
$D_m = 1.478\ (1)$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 17 reflections
$\theta = 10.35\text{–}13.50°$
$\mu = 0.385$ mm^{-1}
$T = 296$ K
Needle
0.30 × 0.20 × 0.20 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω-2θ scans
Absorption correction: *DIFABS* (Walker & Stuart, 1983)
$T_{min} = 0.89$, $T_{max} = 1.03$
2299 measured reflections
2128 independent reflections
1184 observed reflections [$I > 3.00\sigma(I)$]

$R_{int} = 0.024$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 18$
$l = -12 \rightarrow 11$
3 standard reflections monitored every 150 reflections intensity variation: 5.08% decay