along the *a* axis. The intramolecular O···O and intermolecular O···O distances in the hydrogen bonds are 2·605 (8) and 2·755 (11) Å, respectively.

Related literature. The isolation and identification of the title compound have been reported (Inoue, Inoue & Chen, 1981; Rao & Kingston, 1982). However, the structure proposed was erroneous, since the hydroxyl group in the naphthoquinone ring was bonded to C(5) instead of C(8).

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Acta Cryst. (1991). C**47**, 1129–1131

Structure of a New Electrically Conducting Charge-Transfer Complex: 1,4-Phenylenebis(diazenecarbonitrile)–3,3′4,4′-Tetramethyl-2,2′,5,5′-tetrathiafulvalene (1/1)

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Abstract. 1,4-Phenylenebis(diazenecarbonitrile) (1) [Kachkurova, Ashkinadze, Shamsutdinova & Kazitsyna (1987). *Zh. Org. Khim.* **23**, 1629–1634] and tetramethyltetrathiafulvalene (2) [Ferraris, Poehler, Bloch & Cowan (1973). *Tetrahedron Lett.* **27**, 2553–2556] form a charge-transfer complex, $C_8H_4N_6.C_{10}H_{12}S_4$, $M_r = 444{\cdot}62$, triclinic, $P\bar{1}$, $a = 7{\cdot}656$ (2), $b = 8{\cdot}074$ (2), $c = 9{\cdot}236$ (2) Å, $\alpha = 89{\cdot}67$ (2), $\beta = 88{\cdot}16$ (2), $\gamma = 62{\cdot}58$ (2)°, $V = 506{\cdot}5$ Å^3, $Z = 1$, $D_x = 1{\cdot}46$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70930 Å, $\mu = 4{\cdot}7$ cm^{-1}, $F(000) = 230$, $T = 296$ K, $R = 0{\cdot}036$ for 1919 reflections with $F_o^2 > 3{\cdot}0\sigma(F_o^2)$. The planar π-electron systems of both organic molecules form a mixed stack with the double bond of (2) lying above the benzene ring of (1) at an intermolecular distance of 3·45 Å. The two N=N—CN groups of (1) are in an *anti* configuration; the stereochemistry of the N=N—CN group is *trans* with an N—N—C angle of 113·0 (1)° and an N—C=N angle of 170·7 (3)°. The thermally activated single-crystal conductivity is 10^{-3}–10^{-5} S cm^{-1} between 300 and 180 K.

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Experimental. Crystals of the title compound (1–2) were obtained by slowly cooling a solution of (1) and (2) in dichloromethane. The crystal used for data collection was a black needle with dimensions 0·02 × 0·05 × 0·40 mm. Preliminary examination and data collection were performed on an Enraf–Nonius CAD-4 computer-controlled diffractometer. Cell constants and the orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $15 < \theta < 17°$, measured by a computer-controlled diagonal slit method of centering. There were no systematic absences. Intensities were measured using ω–2θ scans of 1–4° min^{-1} in ω and a scan width of $(0{\cdot}9 + 0{\cdot}4\tan\theta)°$. Data were collected to a maximum of 56·0° in 2θ. No absorption correction was applied. The range of reflections was $-10 < h < 10$, $-10 < k < 10$, $0 < l < 12$. Three representative reflections were measured every 120 min and remained constant within experimental error. A total of 2582 reflections were collected, of which 2434 were unique. $R_{int} = 0{\cdot}02$. 1919 reflections having intensities greater than three times their standard deviation were used in the

Table 1. *Positional parameters and equivalent isotropic displacement factors*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
S(1)	0·78200 (6)	−0·01500 (6)	0·43120 (5)	3·12 (1)
S(2)	0·40618 (7)	0·12851 (7)	0·28877 (6)	3·38 (1)
N(1)	0·8689 (2)	0·5610 (2)	0·3037 (2)	3·93 (4)
N(2)	0·6910 (2)	0·6023 (2)	0·2832 (2)	3·66 (4)
N(3)	1·0476 (3)	0·6445 (3)	0·1091 (3)	5·41 (5)
C(1)	0·4079 (3)	0·5859 (3)	0·3744 (2)	3·54 (5)
C(2)	0·6021 (3)	0·5477 (2)	0·3968 (2)	3·17 (4)
C(3)	0·9541 (3)	0·6127 (3)	0·1918 (3)	4·04 (5)
C(4)	0·6953 (3)	0·4620 (3)	0·5239 (2)	3·47 (4)
C(5)	0·5971 (3)	0·1495 (2)	0·1952 (2)	3·10 (4)
C(6)	0·7682 (3)	0·0863 (2)	0·2609 (2)	2·96 (4)
C(7)	0·5396 (2)	0·0231 (2)	0·4414 (2)	2·83 (4)
C(8)	0·5485 (3)	0·2405 (3)	0·0501 (3)	4·58 (6)
C(9)	0·9513 (3)	0·0932 (3)	0·2080 (3)	3·95 (5)
H(1)	0·824 (3)	0·439 (3)	0·535 (2)	1·9 (5)*
H(2)	0·347 (3)	0·646 (3)	0.288 (2)	2·05 (2)*
H(3)	0·420 (4)	0·337 (4)	0·052 (3)	5·1 (8)*
H(4)	1·059 (3)	−0·026 (3)	0·195 (3)	3·9 (7)*
H(5)	0·939 (3)	0·144 (3)	0·126 (3)	3·6 (6)*
H(6)	0·993 (3)	0·166 (3)	0·272 (3)	3·6 (6)*
H(7)	0·647 (3)	0·255 (3)	0·013 (3)	3·6 (6)*
H(8)	0·528 (4)	0·171 (4)	−0·014 (3)	6·0 (8)*

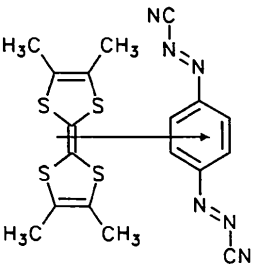
* Atoms were refined isotropically.

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

S(1)—C(6)	1·752 (1)	C(4)—H(1)	0·92 (2)
S(1)—C(7)	1·735 (1)	C(5)—C(6)	1·333 (2)
S(2)—C(5)	1·749 (1)	C(5)—C(8)	1·498 (2)
S(2)—C(7)	1·742 (1)	C(6)—C(9)	1·495 (2)
N(1)—N(2)	1·265 (2)	C(7)—C(7)	1·357 (3)
N(1)—C(3)	1·368 (2)	C(8)—H(3)	0·93 (3)
N(2)—C(2)	1·408 (2)	C(8)—H(7)	0·88 (2)
N(3)—C(3)	1·137 (2)	C(8)—H(8)	0·88 (3)
C(1)—C(2)	1·395 (2)	C(9)—H(4)	0·94 (2)
C(1)—C(4)	1·373 (2)	C(9)—H(5)	0·84 (2)
C(1)—H(2)	0·95 (2)	C(9)—H(6)	0·99 (2)
C(2)—C(4)	1·398 (2)		

C(6)—S(1)—C(7)	96·12 (7)	S(2)—C(5)—C(8)	116·0 (2)
C(5)—S(2)—C(7)	96·01 (7)	C(6)—C(5)—C(8)	127·0 (2)
N(2)—N(1)—C(3)	113·0 (1)	S(1)—C(6)—C(5)	116·8 (2)
N(1)—N(2)—C(2)	113·6 (1)	S(1)—C(6)—C(9)	115·3 (2)
C(2)—C(1)—C(4)	120·7 (1)	C(5)—C(6)—C(9)	127·9 (1)
C(2)—C(1)—H(2)	119· (1)	S(1)—C(7)—S(2)	114·05 (7)
C(4)—C(1)—H(2)	120· (1)	S(1)—C(7)—C(7)	123·0 (2)
N(2)—C(2)—C(1)	114·7 (2)	S(2)—C(7)—C(7)	123·0 (1)
N(2)—C(2)—C(4)	124·5 (1)	C(5)—C(8)—H(7)	111· (1)
C(1)—C(2)—C(4)	120·8 (2)	C(5)—C(8)—H(3)	111· (1)
N(1)—C(3)—N(3)	170·7 (3)	C(5)—C(8)—H(8)	112· (2)
C(1)—C(4)—C(2)	118·5 (2)	C(6)—C(9)—H(4)	113· (1)
C(1)—C(4)—H(1)	123· (1)	C(6)—C(9)—H(5)	113· (1)
S(2)—C(5)—C(6)	117·0 (1)	C(6)—C(9)—H(6)	114· (1)
C(2)—C(4)—H(1)	118· (1)		

refinements. The structure was solved with the Patterson method and subsequent difference Fourier syntheses. H atoms were located and their positions and isotropic thermal parameters were refined. Non-H atoms refined anisotropically. The structure was refined by full-matrix least squares where the function minimized is $\sum w(|F_o| - |F_c|)^2$ and the weight w is defined as $4F_o^2/\sigma^2(F_o^2)$. The standard deviation on intensities $\sigma(F_o^2)$ is defined as $\sigma^2(F_o^2) = [S^2(C + RB) + (pF_o^2)^2]/\mathrm{Lp}^2$ where S is the scan rate, C is the total integrated peak count, R is the ratio of scan time to background counting time, B is the total background count, Lp is the Lorentz–polarization factor and the parameter p is a factor introduced to downweight intense reflections. Here p was set to 0·020. The final cycle of refinement included 159 variable parameters and converged with



(2) (1)

Fig. 1. Charge-transfer complex between 1,4-phenylenebis-(diazenecarbonitrile) (1) and tetramethyltetrathiafulvalene (2).

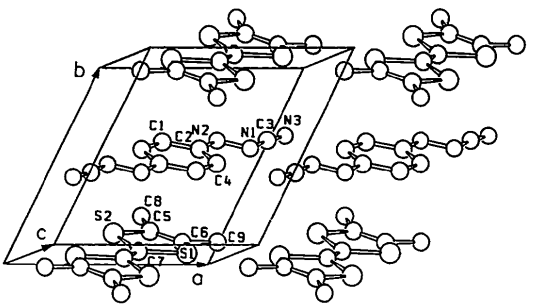


Fig. 2. Perspective view of the structure of (1–2).

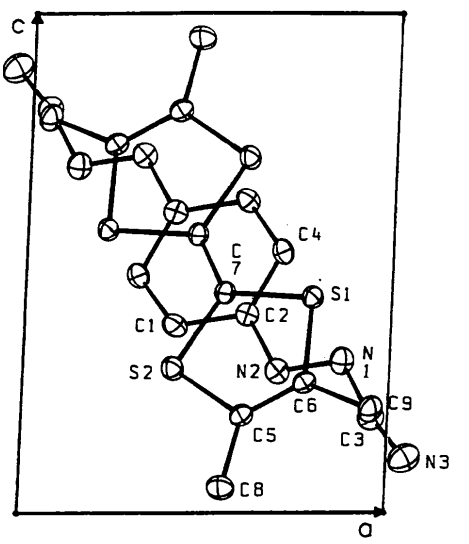


Fig. 3. [010] projection of the structure of (1–2).

unweighted and weighted residuals $R = 0{\cdot}036$ and $wR = 0{\cdot}042$. $S = 2{\cdot}03$. The conventional R for all reflections including unobserved ones was 0·052. Maximum and minimum peak heights in the final difference synthesis were 0·41 and $-0{\cdot}30$ e Å^{-3}. $(\Delta/\sigma)_{max} = 0{\cdot}004$. All calculations were performed on a VAX computer using *SDP/VAX* (Frenz, 1978). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1* gives the atomic coordinates and thermal parameters. Selected bond distances and bond angles are listed in Table 2. Fig. 1 shows the complex (1–2), Figs. 2 and 3 show projections of the crystal structure (Keller, 1988; Johnson, 1976).

* Tables of anisotropic displacement factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53747 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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SHORT COMMUNICATION

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. (1991). C**47**, 1131–1132

Structure of the complex of imidazole and picric acid. Corrigendum and addendum.

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Abstract

The crystallographic results of Soriano-García, Schatz-Levine, Toscano & Iribe [*Acta Cryst.* (1990), C**46**, 1556–1558] show unequivocally that this substance is the salt 'imidazolium picrate' and not a complex of some kind between the neutral moieties 'imidazole' and 'picric acid'.

This crystal structure was reported (Soriano-García, Schatz-Levine, Toscano & Iribe, 1990) under the title given above. However, the authors state "The imidazole ring is protonated and makes a dihedral angle of 112·6 (1)° with the six-membered ring of picric acid." This provides clear evidence for classifying the material as the salt, imidazolium picrate.

We have computed the *ORTEP* stereodiagram (Fig. 1; Johnson, 1965). The moieties are arranged in zigzag ribbons, with axes along [100]; each 'picrate' oxygen is hydrogen bonded to the two N—H groups of the imidazolium cation, the N(1)···O(1)···N(3) angle being 77·6 (4)°. Despite slight asymmetry in the hydrogen bonding {d[N(1)···O(1)] = 2·710 (4) and d[N(3)···O(1)] = 2·825 (5) Å}, the imidazolium cation does not deviate significantly from *mm*2 symmetry, in contrast to the situation in the neutral molecule (Craven, McMullan, Bell & Freeman, 1977). The mutual disposition of the ring planes of the components is such that π–π^* interaction is impossible; the moiety dimensions and the location of the H atoms show that this is not a hydrogen-bonded complex of neutral molecules, as in imidazole–5,5-diethylbarbituric acid (Hsu & Craven, 1974). Thus the material is not a 'complex' in any of the (here relevant) senses in which this term is abused (le Noble, 1974).

Somewhat similar confusions of nomenclature occur in the report of the crystal structure of pyridinium picrate (Talukdar & Chaudhuri, 1976).

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