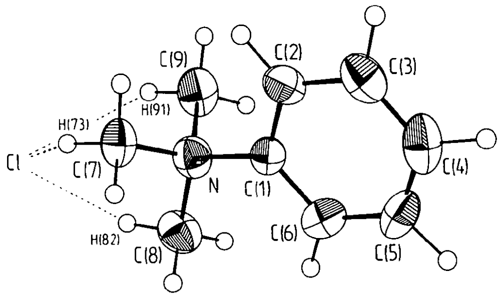

Fig. 1. Das $[N(CH_3)_3(C_6H_5)]^+$-Ion mit Ellipsoiden der thermischen Schwingung (50% Aufenthaltswahrscheinlichkeit, ausgenommen für die H-Atome).

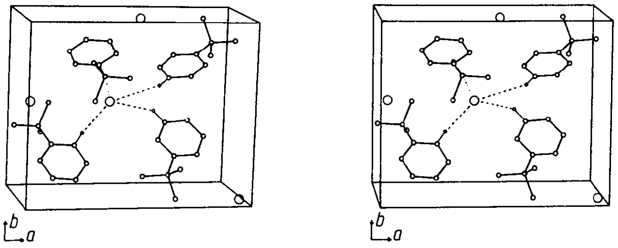

Fig. 2. Stereoskopische Ansicht der Elementarzelle. Punktiert: Verbindungslinie N···Cl um anzuzeigen, wie das Cl^--Ion in die drei Methylgruppen eingenistet ist. Gestrichelt: die drei kürzesten Cl···H-Kontakte mit Phenyl-H-Atomen.

$\leq h \leq 15$, $0 \leq k \leq 12$, $0 \leq l \leq 7$. Kontrollreflexe 401 und 130 zeigten Intensitätschwankungen < 3,3%. 896 unabhängige Reflexe gemessen, davon 161 mit $F < 5\sigma(F)$. Wegen nur geringer Absorptionseffekte keine Absorptionskorrektur. Strukturaufklärung durch 'direkte Methode', Verfeinerung durch Minimieren von $\sum(|F_o| - |F_c|)^2$, Einheitsgewichte, $(\Delta/\sigma)_{max} < 0{,}1$. Methylgruppen als starre Einheiten behandelt, alle C—H-Abstände auf 1,08 Å festgehalten. Restelektronendichte $|\Delta\rho| < 0{,}17$ e Å^{-3}. Keine Extinktionskorrektur. Atomformfaktoren: Cromer & Mann (1968). f', f'': Cromer & Liberman (1970). Rechenprogramme: Müller (1971), Sheldrick (1976), Johnson (1965). $R = 0{,}035$ für 735 Reflexe $F > 5\sigma(F)$, $R = 0{,}050$ für alle Reflexe. Die Atomparameter sind in Tabelle 1,* interatomare Abstände und Winkel in Tabelle 2 aufgeführt. Fig. 1 zeigt das $[N(CH_3)_3(C_6H_5)]^+$-Ion, Fig. 2 zeigt die Kristallpackung.

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* Die H-Atomkoordinaten, die Parameter für den anisotropen Temperaturfaktor und die Liste der beobachteten und berechneten Strukturfaktoren sind bei der British Library Document Supply Centre (Supplementary Publication No. SUP 52138: 7 pp.). hinterlegt. Kopien sind erhältlich durch: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *p*-Bis(phenylthio)benzene: a Redetermination

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Abstract. $C_{18}H_{14}S_2$, $M_r = 294{\cdot}4$, monoclinic, $P2_1/c$, $a = 5{\cdot}824\,(1)$, $b = 17{\cdot}771\,(3)$, $c = 7{\cdot}592\,(1)$ Å, $\beta = 110{\cdot}07\,(1)°$, $V = 738{\cdot}0\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}325$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 3{\cdot}39$ cm^{-1}, $F(000) = 308$, $T = 296$ K, final $R = 0{\cdot}049$ for 2257 unique reflections to $(\sin\theta)/\lambda = 0{\cdot}857$ Å^{-1} and with $F > 3\sigma(F)$. This is a new study of a known structure [Andreetti, Garbarczyk & Krolikowska

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S	−0·15839 (7)	0·10415 (3)	0·27664 (6)	59
C(1)	0·1136 (2)	0·12865 (8)	0·4604 (2)	37
C(2)	0·3365 (3)	0·09270 (8)	0·4948 (2)	41
C(3)	0·5399 (3)	0·11546 (10)	0·6428 (2)	50
C(4)	0·5236 (4)	0·17323 (11)	0·7584 (2)	59
C(5)	0·3031 (4)	0·20865 (10)	0·7263 (2)	59
C(6)	0·0978 (3)	0·18712 (9)	0·5783 (2)	48
C(7)	−0·0607 (2)	0·04596 (9)	0·1263 (2)	40
C(8)	−0·1638 (3)	−0·02498 (10)	0·0833 (2)	45
C(9)	−0·1041 (3)	−0·07093 (10)	−0·0422 (2)	46

Table 2. *Bond lengths* (Å) *and angles* (°)

Atoms related by inversion symmetry are indicated by primes.

S—C(1)	1·769 (2)	C(5)—C(6)	1·384 (3)
S—C(7)	1·773 (2)	C(1)—C(6)	1·395 (2)
C(1)—C(2)	1·389 (2)	C(7)—C(8)	1·386 (2)
C(2)—C(3)	1·384 (2)	C(8)—C(9)	1·387 (3)
C(3)—C(4)	1·375 (3)	C(7)—C(9′)	1·395 (2)
C(4)—C(5)	1·375 (3)		
C(1)—S—C(7)	104·7 (1)	C(2)—C(3)—C(4)	120·6 (2)
S—C(1)—C(2)	124·2 (1)	C(3)—C(4)—C(5)	119·6 (2)
S—C(1)—C(6)	116·8 (1)	C(4)—C(5)—C(6)	120·8 (2)
S—C(7)—C(8)	118·2 (1)	C(1)—C(6)—C(5)	119·9 (2)
S—C(7)—C(9′)	122·2 (1)	C(8)—C(7)—C(9′)	119·5 (2)
C(2)—C(1)—C(6)	119·0 (1)	C(7)—C(8)—C(9)	120·4 (2)
C(1)—C(2)—C(3)	120·2 (1)	C(7)—C(9′)—C(8′)	120·1 (2)

(1981). *Cryst. Struct. Commun.* **10**, 789–792; $R = 0{\cdot}054$, 1387 reflections to $(\sin\theta)/\lambda = 0{\cdot}609\ \text{Å}^{-1}$, $I > 2\sigma(I)$]. The cell parameter b is smaller than that previously reported by 0·41 Å; bond-length e.s.d.'s for the present analysis are 0·002–0·003 Å compared with 0·004–0·007 Å for the previous study. An atomic group of C(7), S, C(1) and C(4) forms a plane through a center of inversion. This plane makes dihedral angles of 14·0 (4) and 56·6 (1)° with the terminal phenyl and central benzene rings, respectively. The deviations from the best least-squares planes for this atomic group, the phenyl ring and the benzene ring are in each case less than 0·004 (1) Å.

Experimental. The title compound was prepared by the reaction of *p*-diiodobenzene with sodium benzenethiolate (Migita, Shimizu, Asami, Shiobara, Kato & Kosugi, 1980); its purity was ≥ 99% from liquid chromatographic analysis. Single crystals were grown by evaporation from *n*-hexane solution and are rhombohedral plates parallel to the (010) plane. Crystal with dimensions 0·38 × 0·45 × 0·48 mm, data collection on a Rigaku AFC-5 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; scan mode θ–2θ, scan speed 10° min^{-1} in θ, scan width $1{\cdot}6° + 0{\cdot}5° \tan\theta$; cell dimensions from 24 reflections, $9{\cdot}8 < \theta < 16{\cdot}5°$; Lorentz and polarization corrections, no absorption correction; $(\sin\theta)/\lambda < 0{\cdot}857\ \text{Å}^{-1}$ ($0 \le h \le 9$, $0 \le k \le 30$, $-12 \le l \le 12$); three standard reflections (441, 162 and 124) monitored every 150 reflections, variation of intensity within 2%; 4266 reflections measured, 3983 unique reflections ($R_{\rm int} = 0{\cdot}036$), of which 2257 with $F > 3\sigma(F)$ were used in refinement. Structure solved by direct methods (Main, Woolfson & Germain, 1972). Full-matrix least-squares refinement on F with weights $w = 1/\sigma^2(F)$, non-H atoms refined anisotropically; all H atoms located in difference Fourier maps and refined isotropically. 120 parameters. $R = 0{\cdot}049$, $wR = 0{\cdot}047$, $S = 1{\cdot}41$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}28$; $(\Delta\rho)_{\rm max} = 0{\cdot}3$, $(\Delta\rho)_{\rm min} = -0{\cdot}3\ \text{e Å}^{-3}$. Atomic scattering factors, f' and f'' from *International Tables for X-ray Crystallography* (1974). Computer programs from *UNICS*3 (Sakurai & Kobayashi, 1979). Final atomic parameters are given in Table 1,* bond lengths and angles in Table 2, according to the numbering scheme given in Fig. 1. The crystal packing is shown in Fig. 2.

Related literature. The title compound was first synthesized by Migita *et al.* (1980). The crystal structure of poly-*p*-phenylene sulfide has been reported by Tabor, Magré & Boon (1971). An earlier determination of the present structure had $b = 18{\cdot}185\ (10)$ Å (Andreetti, Garbarczyk & Krolikowska, 1981) and we cannot explain the discrepancy. Molecular geom-

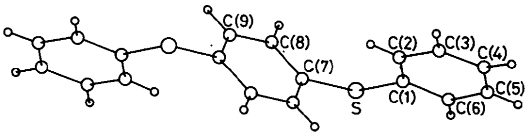

Fig. 1. Molecular geometry and atomic numbering.

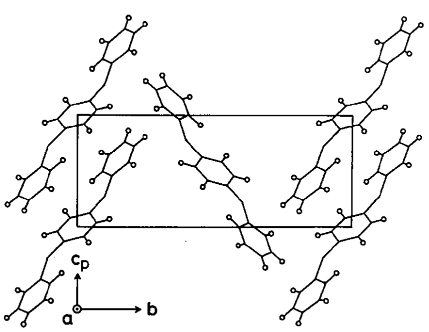

Fig. 2. The packing arrangement in the crystal viewed along **a**.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, non-essential bond lengths and angles, and least-squares-planes details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51951 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

etry in the present study is more precise, with bond-length e.s.d.'s 0·002–0·003 Å (*cf.* 0·004–0·007 Å) and valence-angle e.s.d.'s 0·1–0·2° (*cf.* 0·2 – 0·4°). Aromatic C—C distances here are all in the range 1·375 (2)–1·395 (2) Å compared with the slightly wider range of 1·365 (7)–1·402 (5) Å obtained in the earlier study.

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Structure of *N*-(*p*-Nitrophenyl)ethylenediamine

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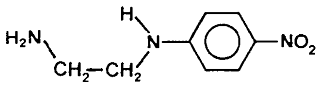
Abstract. $C_8H_{11}N_3O_2$, $M_r = 181{\cdot}19$, monoclinic, $P2_1/c$, $a = 10{\cdot}668\ (2)$, $b = 8{\cdot}958\ (2)$, $c = 10{\cdot}308\ (2)$ Å, $\beta = 115{\cdot}75\ (2)°$, $U = 887{\cdot}2\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}356$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}085$ cm^{-1}, $F(000) = 384$, $T = 295$ K, $R = 0{\cdot}054$ for 1136 observed reflections. All non-H atoms except C(8) and N(2) are coplanar, the maximum atomic deviation from the best plane being 0·08 Å. A comparison of C—C and N—C bond lengths in the aromatic ring indicates significant contributions from the quinonoid canonical forms owing to through-conjugation effects as also found in *p*-nitroaniline derivatives. An intermolecular hydrogen bond is formed between N(1)(x, y, z) and N(2)($-x$, $-\frac{1}{2}+y$, $\frac{3}{2}-z$) [N(1)···N(2) = 2·994 (4) Å and N(1)—H(9)··· N(2) = 170 (3)°].

Experimental. The title compound (1) was prepared by a previously reported method (Linsker & Evans, 1945). Crystals were grown as brown plates from boiling water. A crystal with approximate dimensions 0·15 × 0·50 × 0·55 mm was mounted on a Rigaku automated four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). Accurate unit-cell parameters were determined by a least-squares fit of 2θ values of 25 centered reflections in the 2θ range 20·1–28·6°. Intensities were measured by the θ–2θ scan technique with a scan rate of 4° min^{-1} in 2θ and a scan width of $\Delta(2\theta) = (2{\cdot}5 + 0{\cdot}70\tan\theta)°$. Background intensities were measured for 5 s at both ends of a scan. Four standard reflections (00$\bar{2}$, 04$\bar{4}$, 040, 500) were remeasured after every 60 reflections; no significant loss of intensity was observed throughout data collection. 1610 independent reflections ($R_{\rm int} = 0{\cdot}015$) were collected with 2θ up to 50·5° [$(\sin\theta)/\lambda = 0{\cdot}600$ Å^{-1}] and index range of $h = -11$ to 12, $k = 0$ to 10, $l = -12$ to 0. Corrections for Lorentz and polarization effects were applied to the intensity data; no absorption or extinction corrections were carried out.



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