

Fig. 2. Stereoscopic view of the unit cell with hydrogen bonding.

and N(6). There is no folding (the dihedral angle being 0.2°) about the C(4)–C(5) bond in the adenine ring system. The r.m.s. deviation of the nine atoms of the purine group from their least-squares plane is 0.003 \AA . The N(6) atom is displaced 0.04 \AA from the least-squares plane of the purine ring system. The seven atoms of the benzyl moieties (two rotamers) have an r.m.s. deviation of 0.011 \AA . The dihedral angle between the least-squares planes of the adenine and substituted benzyl moieties (two rotamers) has values of 86.2 and 87.8° .

Fig. 2 shows the packing of the molecules in the unit cell. The 2-chloro-6-fluorobenzyl rings stack over each other approximately perpendicular to the *b* direction of

the unit cell. There is no stacking observed between the adenine rings. The hydrogen-bonding interactions in the structure of arprinocid are also illustrated in Fig. 2. The adenine groups associate through N(6)–HN(6)A...N(7) and N(6)–HN(6)B...N(1) hydrogen bonds. These hydrogen-bond distances have values of 3.03 and 3.07 \AA , respectively, with the proton-to-acceptor distances being 2.15 and 2.15 \AA , respectively.

The authors would like to thank the National Institutes of Health (GM 32690) for financial support.

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Structure of [Bis(*p*-methoxyphenyl)](diethylaminocarbodithioato)iodine(III)

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(Received 8 July 1986; accepted 1 December 1986)

Abstract. $C_{19}H_{24}INO_2S_2$, $M_r=489.4$, monoclinic, $P2_1/c$, $a = 15.296 (1)$, $b = 14.085 (1)$, $c = 20.766 (1) \text{ \AA}$, $\beta = 106.813 (5)^\circ$, $V = 4282.67 \text{ \AA}^3$, $Z = 8$, $D_x = 1.518$, $D_m = 1.517 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 1.71 \text{ mm}^{-1}$, $F(000) = 1968$, $T = 298 \text{ K}$. Final $R = 0.067$ for 3317 independent non-zero reflections. Two discrete molecules in the asymmetric unit. Two covalent I–C_{ar} bonds [$2.09 (2)$ – $2.14 (2) \text{ \AA}$] and one secondary I...S bond [$2.927 (5)$, $2.902 (4) \text{ \AA}$] form T-shaped coordination geometry around each of the two I atoms. A very distorted tetragonal coordination around each I atom is complemented by a weak I...S interaction. The crystal cohesion may be attributed to van der Waals forces.

Introduction. The structure of the title compound (BPDI hereafter) has been determined within the framework of a systematic study of diaryliodanes, of which the structure of (2,2'-biphenylene)-(1-pyrrolidincarbodithioato)iodine(III) (BPPI) has already been reported (Bozopoulos & Rentzeperis, 1986).

Experimental. Yellow crystals, $0.30 \times 0.30 \times 0.40 \text{ mm}$, m.p. 381 – 383 K . D_m measured by flotation in carbon tetrachloride/chloroform. Computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. Cell parameters and standard deviations by least-squares analysis of measured θ angles of 100

strong reflections within the range 11–15°. Three-dimensional data, graphite-monochromated Mo $K\alpha$, θ - 2θ scan mode. Intensity statistics indicated $P2_1/c$. Three standard reflections exhibiting the same percentage linear decrease in intensity (30%). 7656 measured reflections, $\theta = 3$ –25°, index range $h = 0$ –16, $k = 0$ –15, $l = -21$ –21, 3317 with $I > 2\sigma(I)$, $R_{\text{int}} = 0.053$ from merging 484 symmetry-equivalent reflections. Correction for intensity drop, no absorption correction. I and S atoms located by direct methods with *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), remaining non-H atoms by Fourier synthesis. Full-matrix least-squares refinement using *F*, *XRAY72* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Four C atoms belonging to two ethyl groups could not be properly refined. The methylene atoms were found by difference Fourier synthesis at two different positions; the methyl atoms were placed at calculated positions. All these atoms were further included in the refinement with constant positions, population parameter 0.5 and isotropic temperature factor $B = 10.0 \text{ \AA}^2$. H atoms at calculated positions (no refinement) with isotropic temperature factors equal to those of bonded C atoms. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), $w = (F_o/80)^2$ for $F_o \leq 80$, $w = (80/F_o)^2$ for $F_o > 80$. $R = 0.067$, $wR = 0.068$, $S = 3.60$, $(\Delta/\sigma)_{\text{max}} = 0.29$, $(\Delta/\sigma)_{\text{mean}} = 0.04$, $\Delta\rho = -1.37$ – 1.69 e \AA^{-3} in the region of the two disordered ethyl groups.

Discussion. Final positional parameters and equivalent isotropic temperature coefficients for the non-H atoms are given in Table 1.* Selected interatomic distances and angles are in Table 2. A clinographic projection of the BPD1 asymmetric unit, consisting of two molecules, is shown in Fig. 1. Two covalent I–C_{ar} bonds and one secondary I...S bond form a T-shaped coordination around each of the two I atoms [angles in Table 2; max. distances from mean planes 0.171 (3) and 0.058 (8) Å respectively for the two molecules]. The I–C_{ar} covalent bonds 2.14 (1), 2.09 (2), 2.14 (2) and 2.12 (1) Å are comparable with the expected value of 2.05 Å (Pauling, 1960). The four interactions I(1)...S(1) = 2.927 (5), I(1)...S(2) = 3.285 (4), I(2)...S(3) = 2.902 (4) and I(2)...S(4) = 3.268 (4) Å are longer than the sum, 2.37 Å, of the covalent radii, but significantly shorter than the sum, 3.78 Å, of the van der Waals radii (Bondi, 1964). They are comparable with the lengths

* Lists of structure amplitudes, anisotropic thermal parameters, coordinates and isotropic temperature factors for H atoms, interatomic distances and angles not listed in Table 2, selected short intermolecular distances, C–H bond distances and least-squares-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43595 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (\AA^2)

Here and throughout this paper the e.s.d.'s are given in parentheses and refer to the last digit.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
I(1)	0.39797 (7)	0.11721 (8)	0.50725 (5)	4.8
S(1)	0.5711 (3)	0.2206 (4)	0.5458 (2)	5.7
S(2)	0.5500 (3)	0.0853 (3)	0.6495 (2)	5.4
C(1)	0.599 (1)	0.184 (1)	0.628 (1)	4.0
N(1)	0.657 (1)	0.234 (1)	0.673 (1)	5.8
C(2)	0.680 (2)	0.210 (2)	0.744 (1)	7.8
C(3)	0.756 (2)	0.142 (2)	0.770 (1)	10.6
C(4)	0.693 (2)	0.332 (2)	0.658 (1)	10.0
C(5)	0.778 (2)	0.319 (2)	0.647 (2)	13.8
C(6)	0.267 (1)	0.051 (1)	0.470 (1)	4.6
C(7)	0.251 (1)	−0.018 (1)	0.420 (1)	5.3
C(8)	0.168 (1)	−0.064 (1)	0.401 (1)	6.0
C(9)	0.099 (1)	−0.037 (1)	0.430 (1)	5.7
C(10)	0.113 (1)	0.035 (1)	0.477 (1)	6.1
C(11)	0.199 (1)	0.080 (1)	0.498 (1)	5.6
O(1)	−0.013 (1)	−0.077 (1)	0.411 (1)	8.2
C(12)	−0.004 (1)	−0.163 (2)	0.371 (1)	7.7
C(13)	0.368 (1)	0.213 (1)	0.423 (1)	3.9
C(14)	0.338 (1)	0.305 (2)	0.432 (1)	6.6
C(15)	0.316 (1)	0.371 (1)	0.379 (1)	5.4
C(16)	0.329 (1)	0.345 (2)	0.318 (1)	5.3
C(17)	0.357 (1)	0.255 (1)	0.307 (1)	6.3
C(18)	0.379 (1)	0.189 (1)	0.363 (1)	5.5
O(2)	0.3106 (9)	0.4148 (9)	0.2722 (7)	7.4
C(19)	0.327 (2)	0.396 (2)	0.209 (1)	9.9
I(2)	0.06093 (8)	0.34767 (9)	0.46740 (6)	5.2
S(3)	0.2135 (3)	0.4789 (3)	0.5051 (2)	4.9
S(4)	0.2329 (3)	0.3204 (3)	0.6026 (2)	5.6
C(20)	0.275 (1)	0.417 (1)	0.574 (1)	4.7
N(2)	0.355 (1)	0.445 (2)	0.604 (1)	12.0
C(21)	0.429	0.371	0.642	10.0*
C(21')	0.396	0.431	0.683	10.0*
C(22)	0.385	0.272	0.634	10.0*
C(22')	0.321	0.385	0.710	10.0*
C(23)	0.387	0.550	0.598	10.0*
C(23')	0.417	0.508	0.573	10.0*
C(24)	0.306	0.606	0.554	10.0*
C(24')	0.363	0.534	0.500	10.0*
C(25)	−0.060 (1)	0.263 (1)	0.437 (1)	4.7
C(26)	−0.066 (1)	0.188 (1)	0.394 (1)	5.7
C(27)	−0.144 (1)	0.133 (1)	0.374 (1)	6.5
C(28)	−0.216 (1)	0.156 (1)	0.401 (1)	4.9
C(29)	−0.210 (1)	0.229 (1)	0.447 (1)	4.9
C(30)	−0.131 (1)	0.282 (1)	0.464 (1)	4.9
O(3)	−0.2934 (9)	0.0982 (8)	0.3779 (6)	6.3
C(31)	−0.373 (1)	0.120 (1)	0.398 (1)	7.2
C(32)	0.017 (1)	0.434 (1)	0.380 (1)	4.3
C(33)	−0.055 (1)	0.497 (1)	0.372 (1)	6.0
C(34)	−0.082 (1)	0.557 (1)	0.317 (1)	6.2
C(35)	−0.032 (1)	0.556 (1)	0.274 (1)	5.4
C(36)	0.044 (1)	0.495 (1)	0.282 (1)	5.7
C(37)	0.069 (1)	0.437 (1)	0.336 (1)	5.1
O(4)	−0.051 (1)	0.613 (1)	0.218 (1)	7.2
C(38)	−0.135 (2)	0.667 (1)	0.201 (1)	8.1

$$B_{\text{eq}} = \frac{1}{3} \pi^2 \sum_i \sum_j a_i^* a_j^* a_i \cdot a_j$$

* Unrefined isotropic temperature factor.

Table 2. *Interatomic distances* (\AA) *and angles* ($^\circ$) *around the I atoms*

I(1)...S(1)	2.927 (5)	I(2)...S(3)	2.902 (4)
I(1)...S(2)	3.285 (4)	I(2)...S(4)	3.268 (4)
I(1)–C(6)	2.14 (1)	I(2)–C(25)	2.14 (2)
I(1)–C(13)	2.09 (2)	I(2)–C(32)	2.12 (1)
C(6)–I(1)–C(13)	93.2 (6)	C(25)–I(2)–C(32)	92.1 (6)
C(6)–I(1)...S(1)	176.0 (4)	C(25)–I(2)...S(3)	174.4 (4)
C(6)–I(1)...S(2)	125.8 (4)	C(25)–I(2)...S(4)	127.1 (4)
C(13)–I(1)...S(1)	83.8 (4)	C(32)–I(2)...S(3)	83.8 (4)
C(13)–I(1)...S(2)	139.5 (4)	C(32)–I(2)...S(4)	140.8 (4)
S(1)...I(1)...S(2)	56.6 (1)	S(3)...I(2)...S(4)	57.2 (1)

3.053 and 3.162 Å found in BPPI (Bozopoulos & Rentzeperis, 1986). The I(1)⋯S(1) and I(2)⋯S(3) interactions are approximately aligned (see Table 2) with the main bonds I(1)–C(6) and I(2)–C(25) and may be considered as secondary bonds (Alcock, 1972). The other I⋯S interactions deviate significantly from linearity with the corresponding main bonds [angles C(13)–I(1)⋯S(2) = 139.5 (4) and C(32)–I(2)⋯S(4) = 140.8 (4)°], but still tend to complete the planar tetragonal coordination around I found in BPPI and other diaryliodonium compounds [Bozopoulos & Rentzeperis (1986) and literature cited therein].

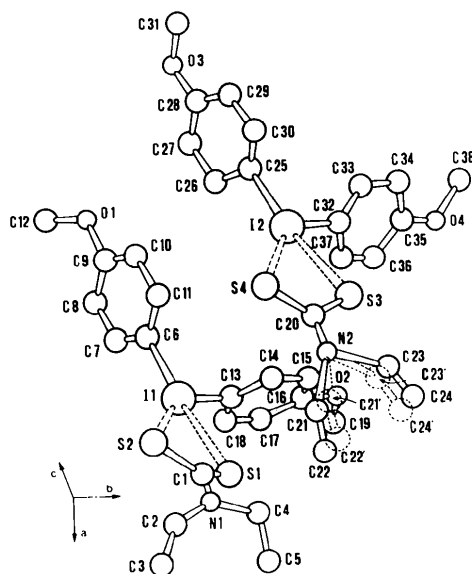


Fig. 1. Clinographic projection of the two independent molecules.

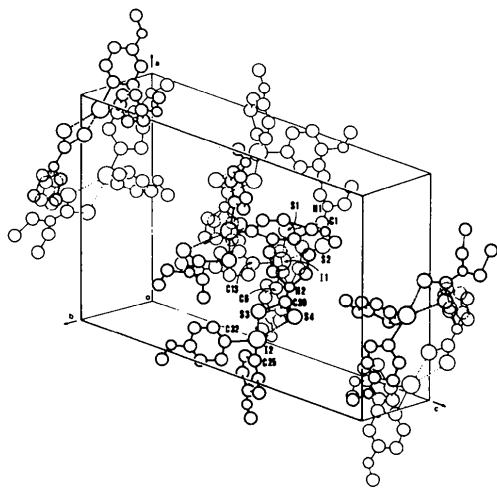


Fig. 2. Clinographic projection of the unit cell showing the molecular packing.

In the bidentate dithiocarbamate (DTC) ligand the mean S–C(N) bond length is 1.70 Å [range 1.68 (2)–1.71 (2) Å] indicating some double-bond character and is comparable with the values found in other DTC ligands (Coucovanis, 1970, 1979). The C–N bonds [1.30 (2) and 1.26 (2) Å] also have double-bond character.

Three of the four phenyl rings are almost perpendicular to the *T*-group plane [corresponding angles 82.5 (5), 82.3 (5) and 82.0 (5)°] while the deviation from near-perpendicularity of the fourth angle [67.8 (5)°] may be attributed to crystal packing forces (see also intermolecular distances in deposited material). The planes of the methoxy groups are inclined to the mean planes of the corresponding phenyl rings by 13.6 (17), 4.4 (10), 6.0 (12) and 6.2 (15)° respectively. The exocyclic angles around each of the C_{ar}–O bonds are unequal and enlarged on the side of the methyl groups owing to the steric hindrance between H atoms in the ring and those in the methyl groups (see deposited material). The largest tilt angle occurs at the C(9)–O(1)–C(12) group, where the difference of the exocyclic angles around C(9)–O(1) is the smallest (Bryan & White, 1982).

A clinographic projection of the unit cell showing the molecular packing is given in Fig. 2.

We thank Professor A. Varvoglis and Miss E. Kotali, Organic Chemistry Laboratory, University of Thessaloniki, for providing the crystals and for useful discussions, the Aristotle University of Thessaloniki for the use of its computer facilities.

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