Fig. 3. Overlap diagram with pyrene molecule projected onto mean plane of NT.

Averaged bond lengths (Table 2) and angles (not listed) of naphthalenetetrone agree well with those reported earlier. There are small differences in the deviations from the mean planes. These differences are statistically significant and are ascribed to packing effects. Similar small differences have been found between the shapes of the non-planar benzo[c]phenanthrene molecule in its neat crystals and in the π-molecular compound benzo[c]phenanthrene–2,3-dichloro-5,6-dicyano-p-benzoquinone (Bernstein, Regev & Herbstein, 1977).

Table 2. *Comparisons of averaged bond lengths* (Å) *as defined in the molecular formulae*
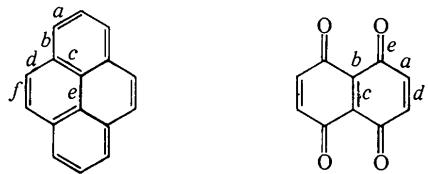
The results have not been corrected for thermal vibrations. Figures in parentheses are e.s.d.'s of average values.

| | Pyrene | | Naphthalenetetrone | |
Bond	Present results	Kai *et al.* (1978)	Present results	Herbstein & Kapon (1982)
a	1·383 (4)	1·385 (1)	1·468 (4)	1·474 (2)
b	1·400 (4)	1·402 (1)	1·505 (4)	1·502 (2)
c	1·419 (3)	1·422 (1)	1·350 (6)	1·349 (3)
d	1·434 (4)	1·436 (1)	1·318 (4)	1·325 (2)
e	1·419 (6)	1·427 (2)	1·216 (4)	1·215 (2)
f	1·315 (5)	1·352 (2)	—	—

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The Structure of p-Iodobenzoic Acid, C7H5IO2

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Abstract. $M_r = 237·0$, monoclinic, $P2_1/a$, $a = 30·357$ (2), $b = 6·0300$ (7), $c = 4·1561$ (4) Å, $\beta = 97·35$ (1)°, $V = 754·6$ (1) Å^3, $Z = 4$, $D_x = 2·148$ g cm^{-3}, monochromated Mo $K\alpha$, $\lambda = 0·71034$ Å, $\mu = 43·5$ cm^{-1}, $F(000) = 464$, $T = 300$ K. $R = 0·072$ for 1002 observed reflections. Two of the asymmetric units form a planar dimeric species. The compound is isostructural with the bromine analog.

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Introduction. As a prelude to the study in this laboratory of a series of compounds for which the *ortho-* and *para-*halogen-substituted benzoic acids are the precursors, it will be necessary to have reasonably accurate distance and angle information for each acid. The structures of the *ortho-*chloro, bromo and iodo substituted acids have been reported by Ferguson & Sim (1961, 1962) and Gougoutas (1977), respectively; *para-*chloro and bromo, by Miller, Paul & Curtin (1974) and Ohkura, Kashino & Haisa (1972). However, a complete single-crystal structure determination has not been reported to date for *p-*iodobenzoic acid (PIBA). Toussaint (1950) presented the lattice constants, but did not give the atomic coordinates. Takaki, Kurisu & Nakata (1982) analyzed the powder pattern, but presented no standard deviations and obtained an $R = 0.168$.

Table 1. *Final fractional unit-cell positional and thermal parameters*

Estimated standard deviations are given in parentheses for the least significant figures. E.s.d.'s elsewhere include the errors in the lattice constants. $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j$.

	x	y	z	$B_{eq}(\text{Å}^2)$
I	0·20932 (3)	0·2259 (2)	0·0791 (2)	4·81
O(1)	0·0097 (3)	0·760 (1)	0·287 (4)	6·41
O(2)	0·0560 (3)	0·993 (1)	0·576 (3)	5·97
C(1)	0·0860 (4)	0·681 (2)	0·333 (4)	4·40
C(2)	0·0785 (4)	0·489 (2)	0·159 (3)	4·26
C(3)	0·1138 (4)	0·358 (2)	0·092 (4)	5·05
C(4)	0·1562 (4)	0·424 (2)	0·183 (3)	4·15
C(5)	0·1649 (4)	0·620 (2)	0·351 (4)	4·24
C(6)	0·1302 (5)	0·755 (2)	0·420 (5)	5·01
C(7)	0·0484 (4)	0·825 (2)	0·409 (4)	4·61

Table 2. *Selected interatomic torsional angles* (°) (*signs as per Klyne & Prelog, 1960*)

C(6)–C(1)–C(7)–O(1)	172 (2)	C(1)–C(7)–O(1)···O(2^i)	179 (1)
C(6)–C(1)–C(7)–O(2)	−8 (2)	C(1)–C(7)–O(2)···O(1^i)	179 (1)
C(2)–C(1)–C(7)–O(1)	−3 (2)	C(7)–O(1)···O(2^i)–C(7^i)	1 (2)
		C(2)–C(1)–C(7)–O(2)	177 (1)

Symmetry code: (i) $-x$, $2·0-y$, $1·0-z$.
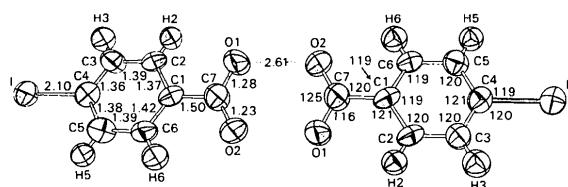


Fig. 1. Inversion-related pair of PIBA molecules. Distances given on the left molecule have a deviation of 0·02 Å; angles on the right, 1°.

Experimental. Colorless crystal 0·075 × 0·35 × 0·40mm selected from those precipitated from an acetone solution, as ethanolic solutions typically yielded much thinner crystals which produced ambiguous single-crystal information. *hkl* and $\overline{h}kl$ octants ($2\theta \le 45°$, $\overline{32} \le h \le 32, 0 \le k \le 6, 0 \le l \le 4$) collected with a diffractometer designed and built in the Ames Laboratory and described elsewhere (Rohrbaugh & Jacobson, 1974; Baughman & Jacobson, 1975). 1345 reflections yielded 1002 averaged, Lp-corrected, independent, reflections with $F_o > 3\sigma(F_o)$ (Lawton & Jacobson, 1968). Absorption correction applied ($T_{max} = 0·72; T_{min} = 0·18$) using a φ-scan technique (Karcher & Jacobson, 1980). Standard Patterson and Fourier analyses (Powell & Jacobson, 1980). Ring hydrogens placed at C—H bond length of 0·95 Å while bisecting the C—C—C angle and assigned an isotropic temperature factor of 4·50 Å^2. As the acid H was not seen in a difference map, nor could its position be accurately inferred, this H atom was not considered in the investigation. Refinement by full-matrix least-squares procedure (Lapp & Jacobson, 1979) minimizing $\sum\omega(|F_o| - |F_c|)$ where $\omega = 1/\sigma_F^2$, converged at $R = 0·072$, $R_w = 0·096$, $S = 3·54$, once a final set of H positions were assigned. Anistropic thermal parameters used for all non-hydrogen atoms. Scattering factors from Hanson, Herman, Lea & Skillman (1964) modified for the real and imaginary parts of anomalous dispersion (Templeton, 1962), for H from Stewart, Davidson & Simpson (1965). Final difference map (Powell & Jacobson, 1980) showed no unaccountable residual electron density. $(\Delta/\sigma)_{max} = 0·02$.

Discussion. Atomic positions, distances and plane angles are presented in Table 1 and Fig. 1, respectively; torsional angles, in Table 2.* These data show that PIBA is isostructural with the bromine analog. It is frequently the case with crystalline carboxylic acids that a pair of molecules is connected through the carboxyl group by hydrogen bonds. The O(1)···O(2) intermolecular distance of 2·61 (1) Å [twice the oxygen van der Waals radius is 2·80 Å (Pauling, 1960)] and the values of the C(7)—O(1)···O(2) and C(7)—O(2)···O(1) angles of 115·4 (9) and 119·8 (8)°, respectively, show that this is also the case with PIBA. While most of the corresponding interatomic distances and angles in the six *ortho-* and *para-*substituted benzoic acids are within 3σ of each other, the C(2)—C(1)—C(7)—O(1) torsional angles are not. The values in *o*-Cl, *o*-Br, *o*-I, *p*-Cl, *p*-Br and *p*-I benzoic acids are 13·7, 18·3, 17, 5·7, 5·8 and 3°,

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38833 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

respectively. When a center of inversion exists in a crystal, this implies that each molecule with a (+) value for a torsional angle has a (−)-valued counterpart. As this is the case for all six of the acids the (+) values have been cited.

A drawing of a pair of hydrogen-bonded molecules related by a center of inversion is given in Fig. 1 (Johnson, 1971). These two molecules are essentially planar as the absolute values of the maximum, minimum and average of the magnitudes of the deviations from a least-squares plane, defined by all 20 non-hydrogen atoms, are 0·077, 0·007 and 0·035Å, respectively. This, along with the values of the C(2)—C(1)—C(7)—O(1) angles, indicates that PIBA with its inversion-related partner is the most planar of the six *para* or *ortho* halogen-substituted benzoic acids.

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