

## Monoclinic

 $P2_1/c$  $a = 6.527 (1) \text{ \AA}$  $b = 17.257 (3) \text{ \AA}$  $c = 13.990 (3) \text{ \AA}$  $\beta = 93.58 (3)^\circ$  $V = 1572.7 (5) \text{ \AA}^3$  $Z = 4$  $D_x = 1.298 \text{ Mg m}^{-3}$  $D_m = 1.30 (1) \text{ Mg m}^{-3}$  $D_m$  measured by flotation in bromoform/cyclohexane

## Data collection

Syntex  $P2_1$  diffractometer $\theta/2\theta$  scans

Absorption correction: none

3757 measured reflections

3457 independent reflections

1155 reflections with

 $I > 2\sigma(I)$  $R_{\text{int}} = 0.033$ 

Cell parameters from 15 reflections

 $\theta = 13\text{--}22^\circ$  $\mu = 0.087 \text{ mm}^{-1}$  $T = 293 (2) \text{ K}$ 

Prism

 $0.40 \times 0.25 \times 0.20 \text{ mm}$ 

Dark green

 $\theta_{\text{max}} = 27.59^\circ$  $h = 0 \rightarrow 7$  $k = 0 \rightarrow 22$  $l = -18 \rightarrow 18$ 

2 standard reflections

frequency: 100 min

intensity decay: none

## Refinement

Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.063$  $wR(F^2) = 0.140$  $S = 1.309$ 

3457 reflections

209 parameters

H atoms not refined

 $w = 1/[\sigma^2(F_o^2) + (0.1145P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.002$  $\Delta\rho_{\text{max}} = 0.388 \text{ e \AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.193 \text{ e \AA}^{-3}$ 

Extinction correction:

*SHELXL93* (Sheldrick, 1993)

Extinction coefficient:

0.004 (3)

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1099). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1997). **C53**, 1359–1362*p*-Iodobenzaldehyde

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Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C1	1.339 (4)	C8—C10	1.370 (5)
O1—C7	1.390 (4)	C8—C9	1.407 (5)
O2—C14	1.340 (4)	C10—C11	1.371 (5)
O2—C11	1.374 (4)	C11—C12	1.377 (5)
N1—C1	1.301 (4)	C12—C13	1.369 (5)
N3—C14	1.324 (4)	C13—C14	1.372 (5)
C1—C8	1.411 (5)		
C1—O1—C7	104.6 (3)	O1—C1—C8	120.9 (3)
C14—O2—C11	107.3 (3)	C10—C8—C9	123.3 (3)
C1—N1—C6	104.7 (3)	C10—C8—C1	121.9 (3)
C14—N3—C17	121.0 (3)	C9—C8—C1	114.7 (3)
C14—N3—C15	120.1 (4)	C8—C10—C11	130.1 (3)
C17—N3—C15	118.9 (4)	C10—C11—O2	119.7 (3)
N1—C1—O1	114.5 (3)	C10—C11—C12	132.4 (4)
N1—C1—C8	124.6 (4)	O2—C11—C12	107.9 (3)
N1—C1—C8—C10	175.7 (3)	C8—C10—C11—O2	−3.9 (6)
N1—C1—C8—C9	−3.1 (5)	C15—N3—C14—O2	−176.6 (3)
C1—C8—C10—C11	−179.2 (4)		

H atoms were located from a difference Fourier map but were not refined.  $U_{\text{iso}}$  values were set to  $1.2U_{\text{eq}}$  of the parent atom.

Data collection: Syntex  $P2_1$  software. Cell refinement: Syntex  $P2_1$  software. Data reduction: *XP21* (Pavelčík, 1987). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

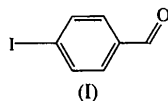
## Abstract

The molecular structure of the title compound,  $\text{C}_7\text{H}_5\text{IO}$ , is normal. There are two independent molecules in the asymmetric unit. Both kinds of molecules pack in similar two-dimensional sheets, in which the most striking feature is short distances [3.068 (4) and 3.074 (4)  $\text{\AA}$ ] between the O and I atoms in adjacent molecules, both of which can be thought of as an interaction between the Lewis base,  $-\text{CHO}$ , and the Lewis acid, I.

## Comment

Aromatic nitroso compounds usually crystallize as dimers with N—N bonds although they are monomeric in solution. *p*-Iodonitrosobenzene is unusual in that it is monomeric in the solid (Webster, 1956). This appears to be the result of an intermolecular  $\text{NO} \cdots \text{I}$  interaction that is comparable in energy to the  $\text{NO}—\text{NO}$  dimerization. We have determined the structure of the title compound, (I), to examine whether a similar  $\text{CO} \cdots \text{I}$  inter-

action occurs. The structures of *p*-bromo- and *p*-chlorobenzaldehyde have been determined previously (Britton, 1994) and found not to contain CO···X interactions.



The structure of (I) contains two molecules in the asymmetric unit in  $P2_1$ . There are no systematic extinctions in the  $h0l$  data, so one can be certain about the space group. However, the two independent molecules are related by pseudosymmetry, which can be described in various ways. For example, molecule *A* at  $y \sim \frac{1}{8}$  is approximately converted to molecule *B* at  $y \sim \frac{3}{8}$  by a pseudo-glide operation of  $0.44 + x, \frac{1}{2} - y, z - 0.02$ . Conversely, molecule *B* at  $y \sim \frac{3}{8}$  is converted to molecule *A* at  $y \sim \frac{1}{8}$  by a pseudo-glide operation of  $0.56 + x, \frac{1}{2} - y, z + 0.02$ . Alternatively, molecule *B* at  $y \sim \frac{3}{8}$  is converted to molecule *A* at  $y \sim \frac{5}{8}$  by a pseudo-center at  $0.72, \frac{1}{2}, 0.49$ , while molecule *A* at  $y \sim \frac{1}{8}$  is converted to molecule *B* at  $y \sim \frac{7}{8}$  by a pseudo-center at  $0.78, \frac{1}{2}, 0.51$ . The overall arrangement might be described as 'pseudo- $P2_1/a$ '.

The atom labeling and anisotropic displacement ellipsoids are shown in Fig. 1. The bond lengths and angles (constrained to be the same in both independent molecules) are similar to those in the analogous chloride and bromide compounds.

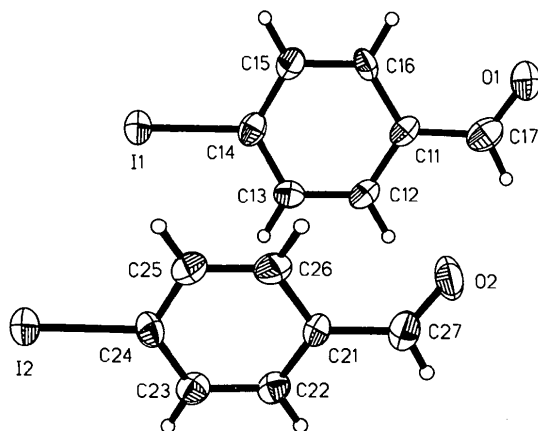


Fig. 1. The two independent molecules of the title compound. Displacement ellipsoids are shown at the 50% probability level. H atoms are shown as spheres of arbitrary size.

The CO···I interaction was found; the molecules form chains linked through these interactions. The parameters of interest in the CO···I contact are the CO···I angle, the O···I distance and the O···IC angle.

If this interaction is thought of as the Lewis base O approaching the Lewis acid I, the values of these parameters are expected to be about 120°, significantly less than the sum of the van der Waals radii (equal to 3.55 Å), and about 180°, respectively. In the title compound, the values are 119.2(3)°, 3.068(4) Å and 174.5(4)° at C11—O1···I1—C14, and 118.5(3)°, 3.074(4) Å and 174.0(3)° at C21—O2···I2—C24. The comparable values in the analogous nitroso compound are 125°, 3.18 Å and 175° (Webster, 1956). A similar interaction was found in 1-aldehyde-4'-methoxy-3,5-diiodophenylamine (Cody & Hazel, 1976) where the values are 139°, 3.26 Å and 174°. The structure of 4-iodo-2-nitrobenzaldehyde was only determined in projection (Coppens & Schmidt, 1964), but it appears from the projection that a similar interaction also occurs in this compound.

The chains (see above) are arranged in two-dimensional sheets perpendicular to the *b* axis, as shown in Fig. 2. Each independent molecule forms a sheet only with its own kind so there are two independent, but very similar sheets. Sheets of molecule *A* occur at  $y \sim \frac{1}{8}$  and  $\frac{5}{8}$ , related by the  $2_1$  axis; sheets of molecule *B* occur at  $y \sim \frac{3}{8}$  and  $\frac{7}{8}$ , also related by the  $2_1$  axis. The individual molecules *A* and *B* are tilted 8.4(3)° and 10.3(3)°, respectively, from the (010) plane. The sheets at  $\frac{1}{8}$  and  $\frac{3}{8}$  are related by a pseudo-glide plane; the benzene rings in the molecules in these two sheets are 18.7(2)° away from being parallel to each other. The sheets at  $\frac{3}{8}$  and

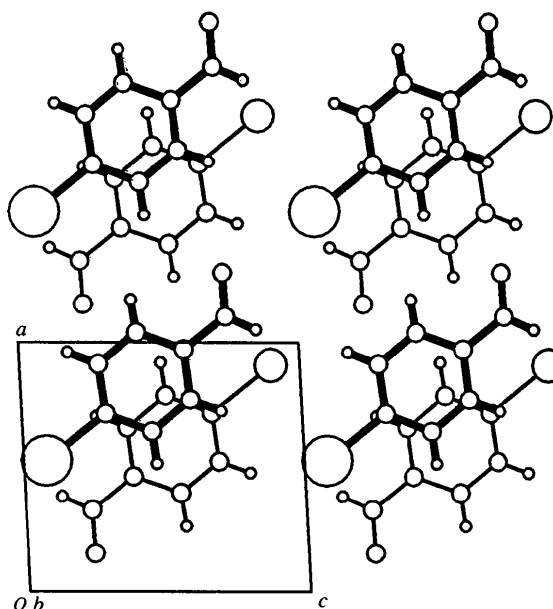


Fig. 2. A view of the packing of the title compound down the *b* axis, showing half the contents of the unit cell. The upper layer is all molecule *A*; the lower layer, which is crystallographically independent, is all molecule *B*. The two layers are related by a pseudo inversion center at 0.72, 0.50, 0.49. Two other layers, related by real  $2_1$  axes, are not shown.

$\frac{5}{8}$  are related by a pseudo-center; the rings in the molecules in these two sheets are  $2.0(3)^\circ$  away from being parallel to each other.

Using the van der Waals radii suggested by Pauling (1960), there are only eight intermolecular distances shorter than normal. Each I atom is involved in four of these short distances. There are the very short I...O distances already mentioned. There are short I...C distances [3.814(4) and 3.809(4) Å] to the C atoms attached to the O atom; these are incidental to the I...O distances. There are I1...H12 (3.274 Å) and I2...H22 (3.306 Å) distances between molecules in adjacent chains. There are I1...C27 [3.801(9) Å] and I2...C17 [3.777(9) Å] distances between pairs of molecules related by the pseudo-centers. The interactions leading to the latter pair of distances appear to be strong enough to pull the I and CHO groups out of the plane of the ring towards each other. The I atoms are 0.039(4) and 0.071(4) Å, the aldehyde C atoms 0.068(9) and 0.035(9) Å, and the aldehyde O atoms 0.134(10) and 0.111(10) Å out of the planes of the rings, all in the direction of the pseudo-centrally related molecule. The aldehyde distances arise from a combination of bending out of the ring plane and twisting with respect to the ring plane. The shortest C...C distance between atoms in the two rings is 3.422(9) Å.

## Experimental

Crystals of the title compound were obtained from Pfaltz & Bauer, Inc.

### Crystal data

C<sub>7</sub>H<sub>5</sub>IO  
 $M_r = 232.01$   
 Monoclinic  
 $P2_1$   
 $a = 6.9122(6)$  Å  
 $b = 13.1517(11)$  Å  
 $c = 7.8098(7)$  Å  
 $\beta = 93.324(1)^\circ$   
 $V = 708.77(11)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 2.174(1)$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 2948 reflections  
 $\theta = 2.6\text{--}24.1^\circ$   
 $\mu = 4.43$  mm<sup>-1</sup>  
 $T = 293(2)$  K  
 Plate  
 $0.40 \times 0.35 \times 0.20$  mm  
 Colorless

### Data collection

Siemens SMART area-detector diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scans (XPREP in SHELXTL; Sheldrick, 1994)  
 $T_{\min} = 0.25$ ,  $T_{\max} = 0.41$   
 3005 measured reflections

1927 independent reflections  
 1921 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$   
 $\theta_{\text{max}} = 24.0^\circ$   
 $h = -7 \rightarrow 7$   
 $k = -15 \rightarrow 11$   
 $l = -8 \rightarrow 6$   
 Intensity decay: <1%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.025$   
 $wR(F^2) = 0.0672$   
 $S = 1.09$   
 1927 reflections  
 175 parameters  
 H-atom positions not refined, isotropic  $U$ 's refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 1.103P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = -0.015$

$\Delta\rho_{\text{max}} = 0.46$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.62$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL93 (Sheldrick, 1993)  
 Extinction coefficient: 0.0051(6)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)  
 Absolute configuration: Flack (1983)  
 Flack parameter = 0.61(5)

Table 1. Selected geometric parameters (Å, °)

I1—C14	2.096(4)	C13—C14	1.397(6)
C11—C12	1.395(6)	C14—C15	1.395(6)
C11—C16	1.419(6)	C15—C16	1.364(7)
C11—C17	1.467(6)	C17—O1	1.220(6)
C12—C13	1.387(6)		
C12—C11—C16	117.8(4)	C13—C14—C15	120.6(4)
C12—C11—C17	119.7(4)	C16—C15—C14	119.9(4)
C16—C11—C17	122.5(4)	C15—C16—C11	121.2(4)
C13—C12—C11	121.7(4)	O1—C17—C11	123.7(4)
C12—C13—C14	118.8(4)		

The data collection nominally covered more than a hemisphere of reciprocal space by a combination of three sets of exposures. Each set had a different  $\Phi$  angle for the crystal and each exposure covered  $0.3^\circ$  in  $\omega$ . The crystal-to-detector distance was 5.94 cm. Coverage of the unique set was approximately 98% complete to at least  $24^\circ$  in  $\theta$ . Crystal decay was monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections. In view of the pseudosymmetry and since the scattering is dominated by the I atoms, the two independent molecules were constrained in the least-squares refinement to have the same bond lengths and angles, and the C<sub>6</sub> ring was constrained to be planar. As the value of the Flack parameter was 0.61(5), the structure was refined as a racemic twin. The 0.61 is the fraction of the larger twin to which the reported parameters refer.

Data collection: ASTRO (Siemens, 1995). Cell refinement: SAINT (Siemens, 1995). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Sheldrick, 1994). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1329). Services for accessing these data are described at the back of the journal.

## References

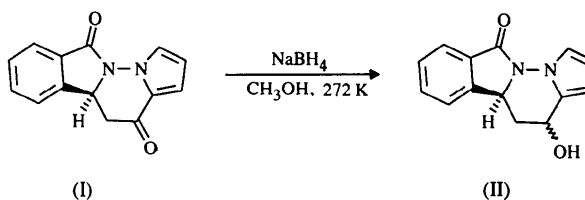
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Webster, M. S. (1956). *J. Chem. Soc.* pp. 2841–2845.

its structure was supported by <sup>1</sup>H and <sup>13</sup>C NMR spectral data. To confirm the *cis* configuration of the hydroxy group and the isoindole ring, the X-ray diffraction study of (II) was undertaken.



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### *cis*-6,10b,11,12-Tetrahydro-12-hydroxy-pyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindol-6-one

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#### Abstract

The title molecule, C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>, consists of a pyridazine ring fused to a five-membered pyrrole ring and to an isoindole moiety. The pyrrole ring and the isoindole moiety are planar, defining a dihedral angle of 35°, while the pyridazine ring adopts an envelope conformation. The isoindole ring and the hydroxy group are in a *cis* configuration.

#### Comment

It has been reported that isoindolo[2,1-*a*]quinoline derivatives similar to vinpocetine (Karpati & Szporny, 1976) have vasodilator activity (Ishihara, Kiyota & Goto, 1990). These results, in connection with our studies on polycyclic heterocycles containing a pyrrole ring, prompted us to search for pyrrolo analogues of this system. The stereoselective reduction of racemic 10b,11-dihydropyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindole-6(6*H*),12-dione, (I), with NaBH<sub>4</sub> in methanol at 272 K led to the title compound, (II) (Marchalín & Decroix, 1995). Only one diastereomer was isolated and

The bond lengths are consistent with average values in the usual sources [*International Tables for Crystallography* (1992, Vol. C, Table 9.5.1.1)]. Selected torsion angles are given in Table 1.

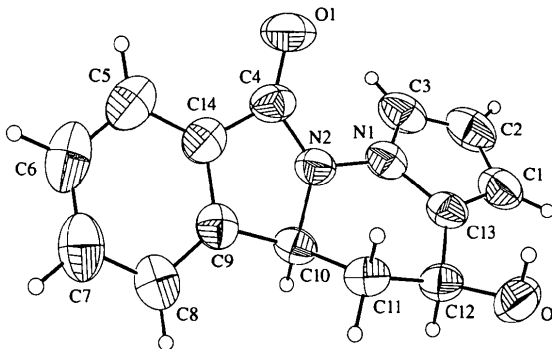


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

#### Experimental

The title compound was prepared by stereoselective reduction of racemic 10b,11-dihydropyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindole-6(6*H*),12-dione, (I), with NaBH<sub>4</sub> in methanol at 272 K.

#### Crystal data

C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>  
M<sub>r</sub> = 240.26  
Monoclinic  
P2<sub>1</sub>/n  
a = 8.386 (2) Å  
b = 8.149 (2) Å  
c = 17.267 (4) Å  
β = 99.04 (4)°  
V = 1165.3 (5) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.369 Mg m<sup>-3</sup>  
D<sub>m</sub> not measured

Mo Kα radiation  
λ = 0.71069 Å  
Cell parameters from 25 reflections  
θ = 17.3–21.6°  
μ = 0.094 mm<sup>-1</sup>  
T = 298 (2) K  
Block  
1.32 × 0.80 × 0.57 mm  
Colourless