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3,5-Diiodo-4-oxo-1,4-dihydropyridine-1-acetic acid

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.136
Data-to-parameter ratio = 19.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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In the title compound, $C_7H_5I_2NO_3$, catemeric hydrogen bonding is present involving the carboxyl group of one molecule and the ketone group of a translationally related neighbour.

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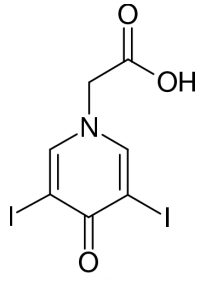
Comment

The title compound, (I) is used, in the form of its diethanolamine salt, as a radiopaque medium where it has the proprietary name Diodone. It is used for a variety of diagnostic procedures including urography (Reynolds, 1996). The main structural interests in this simple molecule are whether or not the carboxylate group is involved in dimer or catemer formation (Beyer & Price, 2000) and if the carboxylate group is coplanar or somewhat perpendicular to the ring. Dimer formation is common for monocarboxylic acids (Haas & Brenner, 1966) although acetic acid is known to form catemers (Jönsson, 1971). When a second H-atom acceptor group is present in a monocarboxylic acid, the tendency is for catemer formation (Thompson *et al.*, 1998) and this can also be detected by a combination of solid-state IR and Raman spectroscopy (Vanderhoff *et al.*, 1990).



(I)

As shown in Fig. 1, the 4-pyridone ring adopts a planar conformation with endocyclic torsion angles varying from −4.3 (6) to 4.9 (6)°. The angle between the mean plane of the six ring atoms and a plane through the carboxylate group (C1, C7, O2 and O3) is 70.6 (1)°.

In (I), intermolecular hydrogen bonding results in continous chains of molecules along the *a* axis (Fig. 2). Steric and electronic effects due to the presence of two I atoms attached to positions 3 and 5 on the ring does not prevent O1 attached to position 4 from acting as the hydrogen-bond acceptor. The lengths of the two C—I bonds are not significantly different but due to hydrogen bonding the length of the O1═C4 double bond is significantly greater than the O3═C7 double bond (Tables 1 and 2).

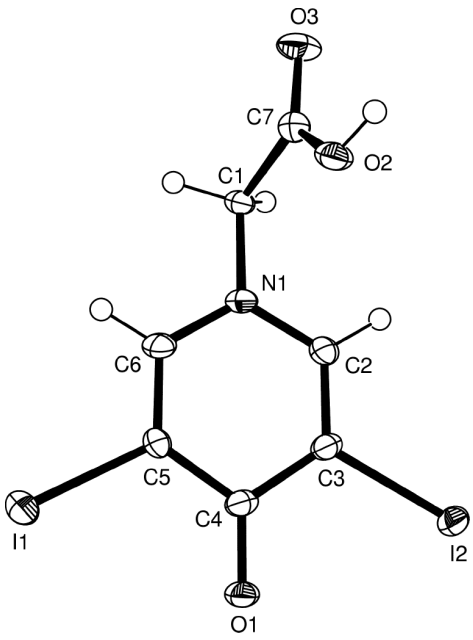


Figure 1
The atomic arrangement in the title molecule (50% probability displacement ellipsoids).

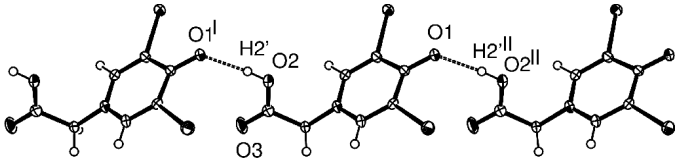


Figure 2
The catemers viewed normal to the (010) plane. [Symmetry codes: (I) $-1+x, y, z$; (II) $1+x, y, z$.]

Experimental

The title compound was obtained from Aldrich and was recrystallized from aqueous ethanol and chloroform.

Crystal data

$C_7H_5I_2NO_3$
M_r = 404.92
Monoclinic, $P2_1/n$
a = 8.5577 (2) Å
b = 10.7752 (3) Å
c = 11.0632 (3) Å
β = 97.3860 (9)°
V = 1011.68 (5) Å^3
Z = 4
D_x = 2.658 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2295 reflections
θ = 2.7–27.5°
μ = 6.19 mm^{-1}
T = 150 (2) K
Lozenge, colourless
0.40 × 0.10 × 0.05 mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
φ and ω scans to fill Ewald sphere
Absorption correction: multi-scan using multiple and symmetry-related data measurements *via* the program *SORTAV* (Blessing, 1995)
T_{min} = 0.191, T_{max} = 0.747
8844 measured reflections
2295 independent reflections
2145 reflections with $I > 2\sigma(I)$
R_{int} = 0.106
θ_{max} = 27.5°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.136
S = 1.05
2295 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0986P)^2 + 0.3356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.29 e Å^{-3}
$\Delta\rho_{min}$ = −4.11 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

I1—C5	2.085 (4)	O2—C7	1.305 (6)
I2—C3	2.094 (4)	O3—C7	1.206 (6)
O1—C4	1.253 (5)		
O3—C7—O2	125.7 (5)	O2—C7—C1	115.9 (4)
O3—C7—C1	118.4 (4)		
C6—N1—C1—C7	115.3 (5)	N1—C1—C7—O3	174.8 (4)
C2—N1—C1—C7	−69.5 (6)	N1—C1—C7—O2	−5.7 (6)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2′···O1^{i}	0.84	1.83	2.665 (5)	174
C1—H1A···O1^{ii}	0.99	2.58	3.503 (6)	156
C2—H2···O3^{iii}	0.95	2.22	3.095 (6)	152
C6—H6···O1^{ii}	0.95	2.35	3.217 (6)	151

Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $1-x, 1-y, -z$.

The H atoms were initially placed in calculated positions and thereafter allowed to ride on their attached atoms with common isotropic displacement parameters which converged to 0.026 (2) Å^2. The position of the hydroxy H atom was determined by rotation about the C—O bond to maximize electron density. Both the maximum and minimum residual electron densities on a final difference fourier map were less than 1 Å from the I2 atom.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997).

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References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Beyer, T. & Price, S. L. (2000). *J. Phys. Chem. B*, **104**, 2647–2655.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Haas, D. & Brenner, S. A. (1966). *Acta Cryst.* **20**, 709–713.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Jönsson, P. G. (1971). *Acta Cryst.* B**27**, 893–898.

Reynolds, J. E. F. (1996). Editor. *Martindale: The Extra Pharmacopoeia*, 31st ed., p. 1009. London: The Pharmaceutical Press.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Thompson, H. W., Brunskill, A. P. J. & Lalancette, R. A. (1998). *Acta Cryst.* C**54**, 829–831.
Vanderhoff, P. A., Lalancette, R. A. & Thompson, H. W. (1990). *J. Org. Chem.* **55**, 1696–1698.