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Crystal structure of (*E*)-2,4-diiodo-6-(4-methyl-2-nitrostyryl)phenol, $C_{14}H_{10}I_2N_2O_3$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.32 \times 0.24 \times 0.17$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	4.22 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	27.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22474, 3432, 0.043
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2495
$N(\text{param})_{\text{refined}}$:	192
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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Abstract

$C_{14}H_{10}I_2N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 13.5219(8)$ Å, $b = 7.2772(4)$ Å, $c = 15.2709(8)$ Å, $\beta = 96.1761(18)^\circ$, $V = 1493.96(14)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0716$, $wR_{\text{ref}}(F^2) = 0.1888$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

4-methyl-2-nitroaniline (1 mmol, 0.152 g) and 3,5-diiodosalicylaldehyde (1 mmol, 0.373 g) were added to the solution of ethanol (10 mL), then the mixture was refluxed 4 hrs. After filtering the residue was dissolved in dichloromethane and the solution was evaporated slowly at room temperature to obtain colorless prismatic crystals suitable for X-ray structure determination.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.07267(4)	0.62072(7)	0.28165(3)	0.0449(2)
I2	0.16429(4)	0.36809(8)	−0.07512(3)	0.0503(2)
O3	0.3003(4)	0.5426(8)	0.3190(3)	0.0466(12)
H3	0.360888	0.534475	0.329995	0.070*
N2	0.4789(5)	0.4446(9)	0.2906(4)	0.0414(13)
C11	0.2122(6)	0.4164(10)	0.0580(5)	0.0419(16)
O1	0.5433(4)	0.3181(8)	0.5405(4)	0.0493(13)
O2	0.4665(4)	0.1969(9)	0.4230(4)	0.0513(13)
N1	0.5333(5)	0.2897(9)	0.4610(4)	0.0437(14)
C13	0.1737(6)	0.5160(10)	0.2001(5)	0.0387(15)
C14	0.2741(6)	0.4982(9)	0.2340(5)	0.0401(15)
C12	0.1421(6)	0.4741(10)	0.1132(5)	0.0428(16)
H12	0.075312	0.484080	0.091866	0.051*
C10	0.3117(6)	0.3965(10)	0.0883(5)	0.0421(16)
H10	0.357426	0.356486	0.051075	0.050*
C6	0.6070(6)	0.3670(10)	0.4093(5)	0.0398(15)
C5	0.5813(6)	0.4254(10)	0.3222(5)	0.0414(16)
C2	0.7810(6)	0.4291(10)	0.3985(5)	0.0421(16)
C8	0.4478(6)	0.4108(10)	0.2110(5)	0.0424(16)
H8	0.491813	0.367963	0.172845	0.051*
C9	0.3423(6)	0.4392(10)	0.1785(5)	0.0395(15)
C7	0.7063(6)	0.3712(10)	0.4469(5)	0.0426(16)
H7	0.721991	0.334531	0.505082	0.051*
C4	0.6571(6)	0.4867(10)	0.2748(5)	0.0422(16)
H4	0.641725	0.529894	0.217614	0.051*
C3	0.7549(6)	0.4842(11)	0.3118(5)	0.0469(17)
H3A	0.804680	0.520144	0.277819	0.056*
C1	0.8879(6)	0.4305(12)	0.4390(6)	0.0492(18)
H1A	0.908708	0.554868	0.451333	0.074*
H1B	0.929385	0.376407	0.398760	0.074*
H1C	0.893715	0.361150	0.492828	0.074*

Experimental details

The H atoms were positioned geometrically with $d(\text{C-H}) = 0.93\text{--}0.98\text{ \AA}$ and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier})$ or $1.5 U_{\text{eq}}(\text{methyl})$. Solving, refining and finishing of the crystal structure were finished by OLEX2 [2] and SHELX [3] program systems. The data completeness is 1.00 up to the diffraction angle of 55.01° .

Discussion

Schiff bases of salicylaldehyde with amines (anils) comprise a chemical system undergoing hydrogen-atom tautomerism between enol and keto forms and show the phenomena of solid state photochromism and thermochromism [4]. Similar to the crystal structure of the related Schiff base [5, 6], the titled compound with electron-withdrawing iodosubstituents (*cf.* the figure) clearly shows the enol form. The molecule displays an *E*-configuration at the central C7=N17 bond. The dihedral angle between the two substituted phenyl moieties (C1–C2–C3–C4–C5–C6 and C8–C9–C10–C11–C12–C13) is 39.09° . There is an intramolecular O–H $\cdots$ N hydrogen bond molecular structure of the title compound. All bond lengths and angles are in the expected ranges.

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