ORGANIC COMPOUNDS

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Bis[(*E*)-2-(2,4-dichloro-5-nitrostyryl)-1,3-benzothiazole] Hydrate

OSVALDO COX,* MARISOL CORDERO, SYLVIA PIÑEIRO AND SONGPING D. HUANG*

Department of Chemistry, University of Puerto Rico, PO Box 23346, San Juan, PR 00931, USA. E-mail: huang@zintl.chem.uprr.pr

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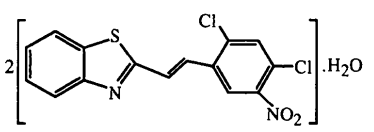
Abstract

The title compound, bis[(*E*)-2-(2,4-dichloro-5-nitrostyryl)-1,3-benzothiazole] hydrate, $2C_{15}H_8Cl_2N_2O_2S.H_2O$, was obtained from the condensation of 2-methylbenzothiazole with 2,4-dichloro-5-nitrobenzaldehyde. Single-crystal X-ray analysis showed that the asymmetric unit contains two crystallographically unique but structurally similar molecules. The dihedral angle between the benzothiazole fragment and the phenyl ring is 6.1 (4)° in molecule *A* and 19.5 (4)° in molecule *B*.

Comment

As part of our continued interest in the synthesis of benzothiazolo[3,2-*a*]quinolinium salts (Cox *et al.*, 1982; Alegría *et al.*, 1993) *via* the photochemically induced cyclization of 2-styrylbenzothiazoles, we carried out the reaction of 2-methylbenzothiazole with 2,4-dichloro-5-nitrobenzaldehyde in refluxing acetic anhydride. The ^1H and ^{13}C NMR, UV/VIS and elemental analysis data showed that the compound formed from the reaction is (*E*)-2-(2,4-dichloro-5-nitrostyryl)benzothiazole. In order to confirm the identity and to study the stereochemistry of this compound, a single-crystal X-ray structure analysis was carried out. It was found that the asymmetric unit contains two crystallographically unique but structurally similar molecules, along with one water molecule, (I).



(I)

The structures of the two molecules are very similar, but not superimposable (Fig. 1). For instance, the NO_2 group in molecule *B* is almost coplanar with the phenyl

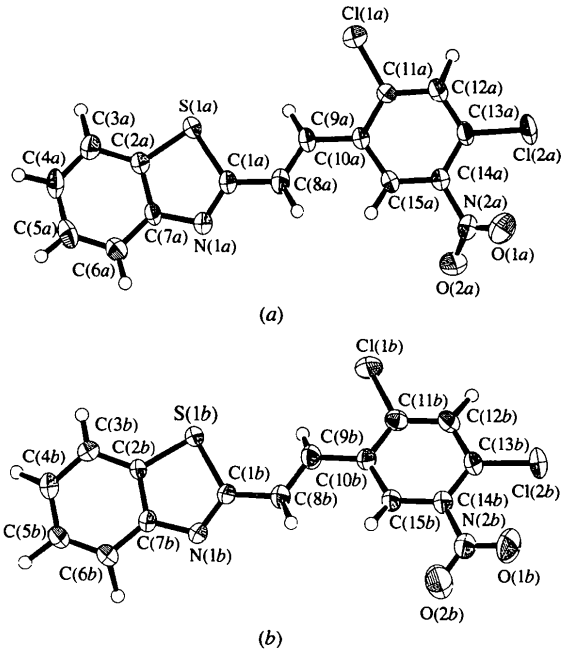


Fig. 1. An *ORTEP*II (Johnson, 1976) representation of molecules *A* and *B* in the unit cell, showing 50% probability displacement ellipsoids.

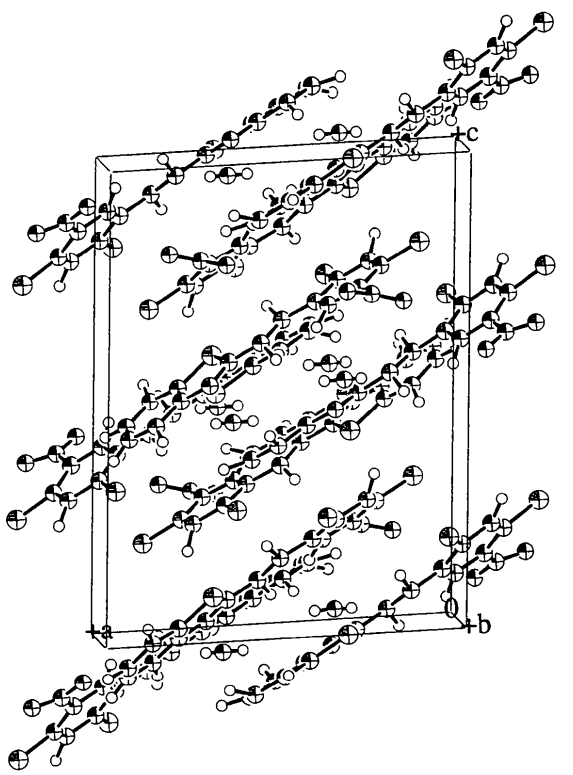


Fig. 2. Packing diagram of the title compound.

ring, while the NO_2 group in molecule *A* is more out of the phenyl plane. The dihedral angles are 15.1 (4) and 44.9 (3)° in molecules *B* and *A*, respectively. In addition, the conformations of the two molecules are slightly different; the dihedral angle between the benzothiazole fragment and the phenyl ring is 6.1 (4)° in *A* and 19.5 (4)° in *B*. The molecules are packed in the unit cell in a pseudo-layered fashion, with the water molecules situated in the layer gap (Fig. 2). No hydrogen bonding is found between the organic molecules and the solvent. The structure is consistent with the spectroscopic data. All bond distances and angles are in the normal ranges.

Experimental

When 2,4-dichloro-5-nitrobenzaldehyde (1.50 g, 6.82 mmol) and 2-methylbenzothiazole (0.909 g, 6.02 mmol) were refluxed in acetic anhydride (35 ml), a yellow solid was obtained (Cox *et al.*, 1982; Muir, Cox, Rivera, Cadiz & Medina, 1992). The crude product (1.59 g, 77% yield) was recrystallized from acetone to give yellow single crystals (m.p. 441–443 K).

Crystal data

$2C_{15}H_8Cl_2N_2O_2S.H_2O$
M_r = 720.43
Monoclinic
$P2_1/c$
a = 13.081 (2) Å
b = 13.473 (2) Å
c = 17.148 (3) Å
β = 93.73 (1)°
V = 3015.8 (7) Å^3
Z = 4
D_x = 1.587 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 15 reflections
θ = 10–11°
μ = 0.579 mm^{-1}
T = 295.2 K
Parallelepiped
0.31 × 0.27 × 0.26 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6665 measured reflections
6494 independent reflections
3476 reflections with $I > 3\sigma(I)$

R_{int} = 0.0454
θ_{max} = 26.29°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 16$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity decay: 3.11%

Refinement

Refinement on F
R = 0.0706
wR = 0.0936
S = 2.161
3476 reflections
407 parameters
H atoms not refined
$w = 1/\sigma^2(F_o) = 1/[\sigma^2(F_o) + 0.0009/4F_o^2]$
$(\Delta/\sigma)_{max}$ = 0.0004

$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}
Extinction correction: Zachariasen type 2, Gaussian isotropic
Extinction coefficient: 2.676 (2) × 10^{-7}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

The crystal deteriorated during data collection, possibly due to loss of water. H atoms were placed in calculated positions.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1995). Cell refinement: *CAD-4-PC*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: TA1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Euniciniatin

ABIMAEL D. RODRÍGUEZ,* EDUVIGIS GONZÁLEZ AND SONGPING D. HUANG*

Department of Chemistry, University of Puerto Rico, PO Box 23346, San Juan, PR 00931, USA. E-mail: huang@zintl.chem.uprr.pr

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Abstract

Euniciniatin [4(*S*),7(*R*)-epoxy-3(*R*)-hydroxy-1(*R*),11(*S*)-dolabella-8(*Z*),12(18)-dien-13-one; (1*R*,3*R*,4*S*,7*R*,11*S*)-3-hydroxy-12-isopropylidene-15-oxa-1,4,8-trimethyltri-