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2-[2-(5-Nitro-2-thienyl)ethenyl]-4*H*-3,1-benzoxazine-4-one, 2-[2-(4-nitrophenyl)-ethenyl]-4*H*-3,1-benzoxazine-4-one and 6,7-dimethoxy-2-[2-(4-nitrophenyl)ethenyl]-4*H*-3,1-benzoxazine-4-one

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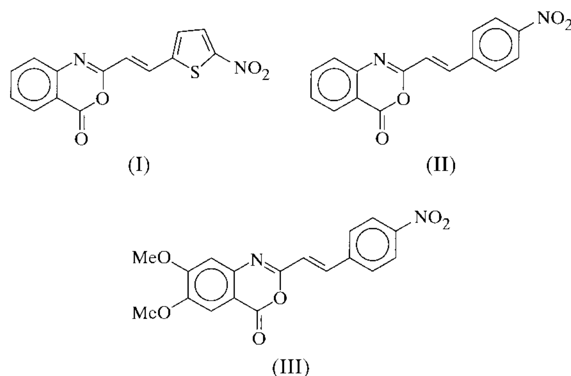
The three title compounds, *i.e.* $C_{14}H_8N_2O_4S$, (I), $C_{16}H_{10}N_2O_4$, (II), and $C_{18}H_{14}N_2O_6$, (III), respectively, are almost planar. The benzoxazinone and nitroaryl moieties are in a *trans* configuration with respect to the ethylene group. The nitro group in all three molecules is almost coplanar with the aryl substituent and shows quite strong conjugation with the rest of the π-system of the molecules. The molecules of (I) form layers along the crystallographic *b* axis, with a distance between adjacent layers of about 3.5 Å. However, the molecules of (II) and (III) form stacks, with distances between adjacent molecules of about 6.0 and 3.6 Å, respectively.



Experimental

The title compounds were synthesized according to the procedure of Misra *et al.* (1980) in good yields (<70%). Crystals appropriate for X-ray structure determination were grown by vacuum sublimation.

Compound (I)

Crystal data

$C_{14}H_8N_2O_4S$
M_r = 300.28
Orthorhombic, $Pca2_1$
a = 7.0127 (11) Å
b = 12.524 (2) Å
c = 14.477 (3) Å
V = 1271.5 (4) Å^3
Z = 4
D_x = 1.569 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
θ = 10–13°
μ = 0.273 mm^{-1}
T = 293 (2) K
Parallelepiped, yellow
0.40 × 0.20 × 0.20 mm

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
1938 measured reflections
1938 independent reflections
1073 reflections with $I > 2\sigma(I)$
θ_{max} = 30.06°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 20$
2 standard reflections every 98 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.100
S = 1.179
1903 reflections
190 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.25 (14)

Compound (II)

Crystal data

$C_{16}H_{10}N_2O_4$
M_r = 294.26
Monoclinic, *Cc*
a = 14.627 (3) Å
b = 7.491 (2) Å
c = 12.344 (5) Å
β = 98.71 (3)°
V = 1337.0 (7) Å^3
Z = 4
D_x = 1.462 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
θ = 9–10°
μ = 0.108 mm^{-1}
T = 213 (2) K
Parallelepiped, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Syntex $P2_1/PC$ diffractometer
$\theta/2\theta$ scans
2042 measured reflections
2042 independent reflections
1011 reflections with $I > 2\sigma(I)$
θ_{max} = 30.07°
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 10$
$l = -17 \rightarrow 17$
2 standard reflections every 98 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.178
S = 1.545
2004 reflections
199 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.06 (20)

Compound (III)

Crystal data

$C_{18}H_{14}N_2O_6$
$M_r = 354.31$
Triclinic, $P\overline{1}$
$a = 6.8484$ (10) Å
$b = 7.2348$ (14) Å
$c = 16.806$ (3) Å
$\alpha = 93.801$ (15)°
$\beta = 96.783$ (13)°
$\gamma = 105.321$ (13)°
$V = 793.3$ (2) Å^3
$Z = 2$
$D_x = 1.483$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 30 reflections
$\theta = 10$–13°
$\mu = 0.113$ mm^{-1}
$T = 208$ (2) K
Parallelepiped, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Syntex $P2_1/PC$ diffractometer
$\theta/2\theta$ scans
3051 measured reflections
2792 independent reflections
1723 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.00°$
$h = -6 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -19 \rightarrow 19$
2 standard reflections every 98 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.101$
$S = 1.108$
2727 reflections
237 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.0324P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Profile analysis of reflections using the method by Streltsov & Zavodnik (1989) was applied to the collected data sets for compounds (I) and (II). This procedure improved the data sets. All H atoms in the three compounds were found by difference Fourier syntheses and were then treated in a riding mode with $U_{iso} = nU_{eq}$ of the parent non-H atom ($n = 1.5$ for methyl groups and 1.2 for the other H atoms). Methyl H atoms in (III) were refined as connected to an idealizied CH_3 group with tetrahedral angles combined and with the possibility of rotation. The absolute structures could not be defined.

For all compounds, data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

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