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(±)-(3a*RS*,4*SR*)-9-Chloro-2-(2-chloro-4-nitrophenyl)-4-(4-chlorophenyl)-3a,4-diethoxy-6,8-dimethoxychromano[4,3-*d*]-$\Delta^{1,9b}$-1,2,3-thiadiazoline

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.115
Data-to-parameter ratio = 16.9

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

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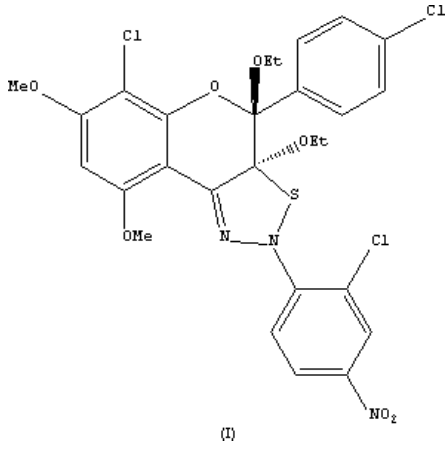
The title compound, $C_{27}H_{24}Cl_3N_3O_7S$, contains a thiadiazoline ring which is almost planar. The two ethoxy groups bonded at the 3a- and 4-positions are in a *trans* configuration.

Comment

Flavonoids continue to capture the interest of scientists from many different disciplines because these compounds possess a broad range of pharmacological properties, including anti-tumor, anti-inflammatory and antiviral activities. A variety of flavonoids have been shown to be antitumor agents in several animal models (Mattocks, 1986; Cassady *et al.*, 1990). We are interested in flavonoids as anticancer agents. The title compound, (I), a novel flavonoid containing nitrogen and sulfur, has been prepared in order to study its antitumor activity.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected geometric parameters are listed in Table 1. The two ethoxy groups bonded to atoms C2 and C3 are in a *trans* configuration. The C1═N2 bond distance suggests a partial double bond. The N1/S1/C2/C1/N2 ring is almost planar, presumably as a result of the conjugation of atoms S1 and N1 with the C1═N2 double bond. The lengths of the C1—C2, S1—C2, S1—N1 and N1—N2 bonds are similar to those found in other derivatives of thiadiazoline (Mohamed *et al.*, 2003; Glossman *et al.*, 2001).

The crystal structure is shown in Fig. 2. The layer sequence is *ABAB*, with layer *B* oriented antiparallel to layer *A*. This type of antiparallel alignment has been reported in the structural arrangement of diimidazolines (Brennan & McKee, 1999) and diones (Klein *et al.*, 1999). In the same layer, the molecules are linked through the C—H···O interactions (Table 2). Between the two layers there are short contacts, O7···Cl3(x, $-\frac{1}{2}-y$, $\frac{1}{2}+z$) of 3.154 (3) Å and O6···O6($1-x$, $-1-y$, $1-z$) of 2.807 (3) Å.

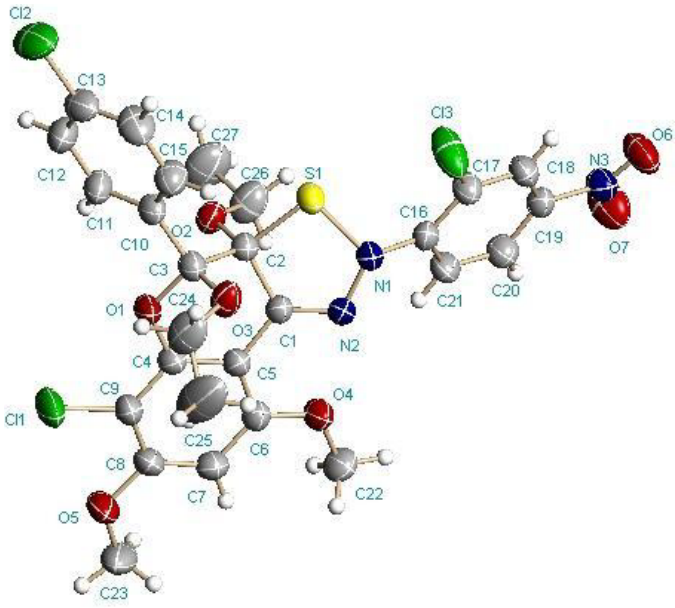


Figure 1
A view of the molecular structure of (I), showing 30% probability displacement ellipsoids.

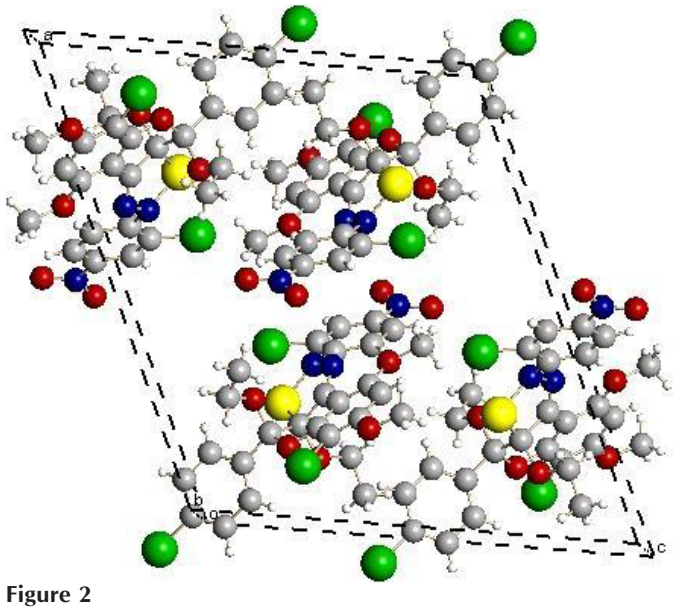


Figure 2
Packing diagram for (I), viewed down the *b* axis.

Experimental

N-(5,7-Dimethoxy-2-phenylchroman-4-ylidene)-*N*′-(4-nitrophenyl)-hydrazine (0.42 g, 1.0 mmol) was refluxed in $SOCl_2$ (8 ml) for 0.5 h, followed by evaporation to dryness *in vacuo*. The resulting slurry was treated with ethanol (8 ml) and refluxed for 0.5 h to yield the crude product of (I). The pure product was obtained through silica gel chromatography (0.34 g, yield 53.1%), and diffraction quality crytals were obtained by slow evaporation of an acetone/methanol solution at room temperature.

Crystal data

$C_{27}H_{24}Cl_3N_3O_7S$
M_r = 640.90
Monoclinic, $P2_1/c$
a = 18.285 (4) Å
b = 10.779 (3) Å
c = 16.152 (4) Å
β = 113.652 (4)°
V = 2916.0 (12) Å^3
Z = 4
D_x = 1.460 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3365 reflections
θ = 4.7–46.5°
μ = 0.44 mm^{-1}
T = 293 (2) K
Prism, yellow
0.51 × 0.34 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.525, T_{max} = 0.885
16 701 measured reflections
6336 independent reflections
3679 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 27.0°
$h = -12 \rightarrow 23$
$k = -13 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.115
S = 0.89
6336 reflections
374 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.016
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.771 (2)	N1—N2	1.380 (3)
S1—C2	1.809 (2)	N1—C16	1.401 (3)
Cl1—C9	1.717 (2)	N2—C1	1.281 (3)
Cl2—C13	1.739 (3)	C1—C2	1.516 (3)
Cl3—C17	1.723 (3)		
N1—S1—C2	91.62 (10)	N2—N1—S1	112.38 (14)
C2—S1—N1—N2	−6.43 (18)	N2—C1—C2—S1	−9.2 (3)
C2—S1—N1—C16	132.2 (2)	N1—S1—C2—C1	7.88 (16)
S1—N1—N2—C1	2.1 (3)	O2—C2—C3—O3	176.19 (17)
N1—N2—C1—C2	5.0 (3)	O2—C2—C3—C10	−58.7 (2)
N2—C1—C2—O2	114.8 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C25—H25*C*···O7^{i}	0.98	2.499	3.456 (5)	175

Symmetry code: (i) $-x+1, -y, -z+1$.

H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C_{Me}).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT-Plus-NT* (Bruker, 1997); data reduction: *SAINT-Plus-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

X-ray data were collected at the Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences.

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