Experimental

Chasmanthinine was isolated from the roots of *A. chasmanthum*, collected from Rescuta Top in Azad Jammu Kashmir, using a method described previously (Parvez *et al.*, 1998). The pure compound was recrystallized from ethanol at room temperature by slow evaporation.

Crystal data

$C_{36}H_{49}NO_9$
$M_r = 639.78$
Orthorhombic
$P2_12_12_1$
$a = 8.300\,(2)$ Å
$b = 14.813\,(5)$ Å
$c = 27.654\,(6)$ Å
$V = 3400.0\,(16)$ Å^3
$Z = 4$
$D_x = 1.250$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 20–30°$
$\mu = 0.727$ mm^{-1}
$T = 293\,(1)$ K
Prismatic
0.50 × 0.43 × 0.40 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5751 measured reflections
5037 independent reflections
4727 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$

$\theta_{\rm max} = 67.6°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = -30 \rightarrow 33$
3 standard reflections every 200 reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.051$
$wR(F^2) = 0.126$
$S = 1.045$
5002 reflections
459 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 2.05P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.010$
$\Delta\rho_{\rm max} = 0.237$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.201$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.1 (3)

Based on the systematic absences of: $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$, and $00l$, $l = 2n + 1$, the space group was uniquely determined to be $P2_12_12_1$ (No. 19). Friedel pairs (2383) were collected to $\theta_{\rm max} = 60°$ and were not merged. Two methoxy groups attached to C4 and C16 were disordered and their atoms were allowed to refine at two locations with site-occupancy factors of 0.50. The phenyl ring was refined as a regular hexagon. The H atoms were included at geometrically idealized positions with C—H and O—H distances of 0.95 Å. Non-H atoms were given anisotropic displacement parameters.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1328). Services for accessing these data are described at the back of the journal. A unit-cell packing diagram has also been deposited.

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$\alpha\alpha'$-Diselenocyanato-*ortho*-xylene[†]

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Abstract

Molecules of the title compound, $C_{10}H_8N_2Se_2$, stack in a one-dimensional ladder configuration, with relatively short Se···N interactions between neighbours.

Comment

The title compound, (I), is commonly used in the synthesis of cyclophanes incorporating selenium (Akabori

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[†] Alternative name: 1,2-phenylenebis(methylselenocyanate).

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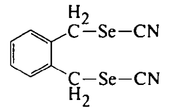
& Kumagai, 1989; Higughi *et al.*, 1987). Selenocyanates in general are used as intermediates in the synthesis of diorganoselenates (Toshimitsu & Uemura, 1987).



(I)

The structure of $\alpha\alpha'$-diselenocyanato-*ortho*-xylene (Fig. 1) shows the expected geometry around selenium confirming the stereochemical activity of the non-bonding valence-electron pairs, with C—Se—C bond angles of 95.9 (3) and 98.7 (3)° around Se1 and Se2, respectively. These compare favourably with other known selenocyanates, for example, 96.0° in benzyl selenocyanate and 94.0° in 4-nitrobenzyl selenocyanate (Maartmann-Moe, Sanderud & Songstad, 1984), 95.1° in phenacyl selenocyanate (Maartmann-Moe, Nevstad & Songstad, 1986) and 94.4° in diselenocyanato-*para*-benzene (McDonald & Pettit, 1970). The Se—CN bond lengths of 1.794 (9) and 1.775 (9) Å observed in the present structure are shorter than those observed previously, which range from 1.829 (Maartmann-Moe *et al.*, 1984) to 1.845 Å (Maartmann-Moe *et al.*, 1986), while the C—N bonds of 1.168 (14) and 1.172 (14) Å are longer than those observed previously, which range from 1.125 (Maartmann-Moe *et al.*, 1986) to 1.149 Å (McDonald & Pettit, 1970).

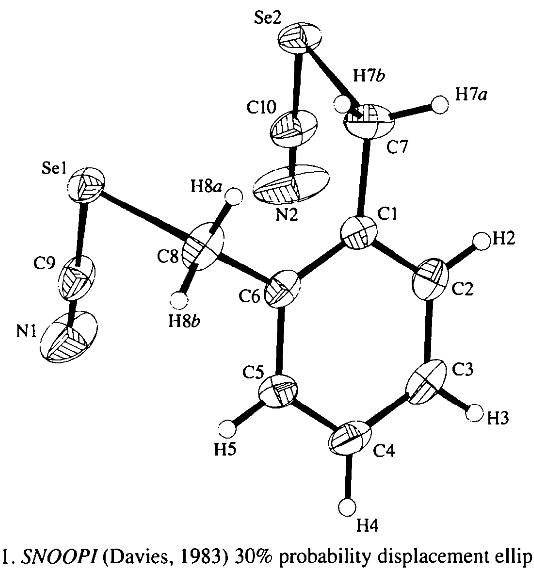


Fig. 1. *SNOOPI* (Davies, 1983) 30% probability displacement ellipsoid plot of $C_6H_4(CH_2SeCN)_2$.

$C_6H_4(CH_2SeCN)_2$ packs in a stacked arrangement stabilized by interactions between Se atoms on one molecule and the nitrile group on its neighbour; Se1···N1^i 2.969 (9) and Se2···N2^i 2.985 (8) Å [symmetry code: (i) x, $y-1$, z]. Both Se atoms on a single molecule interact with the same neighbouring molecule resulting in the formation of a ladder-like one-dimensional chain (Fig. 2). Neighbouring chains have their Se—CN groups pointing in opposite directions. Longer interactions between Se and N atoms on neighbouring chains at around 3.68–3.88 Å stabilize the three-dimensional structure. Similar weak interactions have been noted previously (Maartmann-Moe *et al.*, 1984).

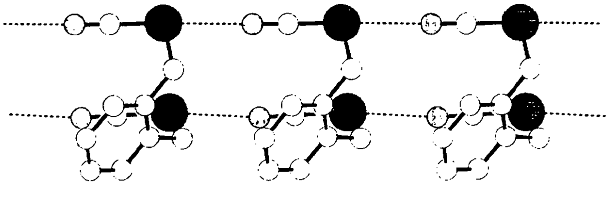


Fig. 2. Packing in $C_6H_4(CH_2SeCN)_2$ showing ladder configuration of an individual chain. C, Se and N atoms are represented by open, large shaded and small shaded circles, respectively.

Experimental

$\alpha\alpha'$-Diselenocyanato-*ortho*-xylene was prepared by dissolving the appropriate molar quantities of potassium selenocyante and $\alpha\alpha'$-dichloro-*ortho*-xylene in dry ethanol and stirring the solution under nitrogen overnight at room temperature. Water was then added to the solution and the ethanol was removed by evaporation *in vacuo*. Extraction with three portions of dichloromethane yielded the title compound as a white solid. Single crystals were formed by slow evaporation of a dichloromethane solution. ^{1}H NMR (recorded on a Jeol FX200 spectrometer in $CDCl_3$): δ 7.26 (4H, *m*), 4.06 (4H, *s*); m.p. 392 K

Crystal data

$C_{10}H_8N_2Se_2$
$M_r = 314.10$
Monoclinic
$P2_1/c$
$a = 8.060(2)$ Å
$b = 5.9206(9)$ Å
$c = 22.638(6)$ Å
$\beta = 91.99(3)°$
$V = 1079.6(4)$ Å^3
$Z = 4$
$D_x = 1.932$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 11.634–15.891°$
$\mu = 6.809$ mm^{-1}
$T = 293(2)$ K
Needle
0.60 × 0.50 × 0.38 mm
Colourless

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.075$, $T_{max} = 0.025$
1881 measured reflections
1711 independent reflections

1381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.105$
$\theta_{max} = 25°$
$h = -3 \rightarrow 9$
$k = 0 \rightarrow 7$
$l = -26 \rightarrow 26$
2 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 0.986$
1711 reflections
119 parameters
H atoms fixed
$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 4.2404P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.809$ e Å^{-3}
$\Delta\rho_{min} = -0.622$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0065 (11)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Se1—C9	1.794 (9)	C1—C7	1.498 (8)
Se1—C8	1.978 (7)	C6—C8	1.501 (8)
Se2—C10	1.775 (9)	C9—N1	1.168 (14)
Se2—C7	1.987 (6)	C10—N2	1.172 (12)
C9—Se1—C8	95.9 (3)	C1—C6—C8	124.4 (4)
C10—Se2—C7	98.7 (3)	C1—C7—Se2	114.6 (5)
C2—C1—C7	116.7 (4)	C6—C8—Se1	115.3 (4)
C6—C1—C7	123.3 (4)	N1—C9—Se1	178.4 (7)
C5—C6—C8	115.6 (4)	N2—C10—Se2	176.5 (8)

The relatively high R_{int} value suggests that the ψ-scan absorption correction used in this study may not be an entirely adequate description of the absorption in this system.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993). Cell refinement: *CAD*-4-*PC*. Data reduction: *CAD*-4 *Software* (Enraf–Nonius, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SNOOPI* (Davies, 1983). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1107). Services for accessing these data are described at the back of the journal.

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1-[*N*-(4-Methyl-2-pyridyl)aminomethylidene]-2(1*H*)-naphthalenone

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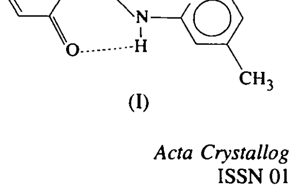
Abstract

The crystal structure of the title compound, $C_{17}H_{14}N_2O$, has been determined and reveals an almost planar structure. Each molecule contains a strong intramolecular O···N hydrogen bond [2.532 (3) Å].

Comment

Schiff bases have been extensively used as ligands in the field of coordination chemistry (Calligaris, Nardin & Randaccio, 1972; Garnovski, Nivorozhkin & Minkin, 1993; Maggio, Pizzino & Romano, 1974; Morassi, Bertini & Sacconi, 1973). The Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen, Schmidt & Flavian, 1964; Moustakali, Mavridis & Hadjoudis, 1978; Hadjoudis *et al.*, 1987). Photochromism is produced by intramolecular proton transfer associated with a change in the π-electron configuration (Barbara, Rentzepis & Brus, 1980; Hadjoudis, 1981; Higelin & Sixl, 1983). Thermochromism is also due to a change in the π-electron configuration induced by a proton transfer and the proton transfer can occur in the ground state (Cohen *et al.*, 1964). Interest in studies on photochromic compounds has been increasing ever since the potential applications of photochromic materials were realised in various areas such as the control and measurement of radiation intensity, optical computers and display systems (Dürr, 1989; Dürr & Bouas-Laurent, 1990).

Our structural investigations of Schiff bases (Elerman, Svoboda & Fuess, 1991; Elerman *et al.*, 1992, 1994, 1995, 1997; Elmali *et al.*, 1995; Kevran, Elmali & Elerman, 1996; Elmali & Elerman, 1997) have led us to examine the title compound, (I).



(I)