C3*A*	0.0464 (6)	−0.0168 (5)	0.8582 (5)	0.068 (2)
C4*A*	0.1004 (6)	−0.0843 (5)	0.9446 (6)	0.059 (2)
C5*A*	0.2143 (6)	−0.0589 (5)	1.0225 (5)	0.062 (2)
C6*A*	0.2707 (5)	0.0410 (5)	1.0144 (4)	0.054 (2)
C1*B*	0.2462 (5)	0.3118 (4)	0.9931 (4)	0.043 (2)
C2*B*	0.1719 (5)	0.4015 (4)	0.9479 (4)	0.051 (2)
C3*B*	0.1475 (5)	0.4808 (5)	1.0118 (5)	0.059 (2)
C4*B*	0.1981 (5)	0.4680 (5)	1.1212 (4)	0.055 (2)
C5*B*	0.2714 (5)	0.3785 (5)	1.1665 (4)	0.058 (2)
C6*B*	0.2965 (5)	0.2996 (5)	1.1036 (4)	0.054 (2)
C7	0.2665 (5)	0.2257 (4)	0.9178 (4)	0.044 (2)
N1	0.4003 (4)	0.2221 (4)	0.9338 (4)	0.049 (2)
C8*A*	0.4376 (5)	0.3385 (5)	0.9126 (5)	0.058 (2)
C8*B*	0.4275 (5)	0.1323 (5)	0.8685 (4)	0.055 (2)
C9*A*	0.5740 (6)	0.3434 (6)	0.9306 (6)	0.092 (3)
C9*B*	0.3670 (7)	0.1499 (6)	0.7471 (5)	0.095 (3)

Table 2. *Geometric parameters* (Å, °)

C1*A*—C2*A*	1.381 (7)	C6*B*—C1*B*	1.387 (7)
C2*A*—C3*A*	1.390 (8)	Cl2—C4*A*	1.737 (7)
C3*A*—C4*A*	1.358 (8)	Cl3—C4*B*	1.733 (7)
C4*A*—C5*A*	1.385 (7)	C1*A*—C7	1.510 (8)
C5*A*—C6*A*	1.391 (8)	C1*B*—C7	1.531 (9)
C6*A*—C1*A*	1.387 (7)	N1—C7	1.517 (9)
C1*B*—C2*B*	1.369 (7)	N1—C8*A*	1.517 (9)
C2*B*—C3*B*	1.387 (9)	N1—C8*B*	1.502 (9)
C3*B*—C4*B*	1.374 (7)	C8*A*—C9*A*	1.541 (9)
C4*B*—C5*B*	1.362 (8)	C8*B*—C9*B*	1.531 (8)
C5*B*—C6*B*	1.377 (9)		
C1*A*—C2*A*—C3*A*	120.8 (4)	Cl2—C4*A*—C5*A*	118.9 (5)
C2*A*—C3*A*—C4*A*	119.5 (5)	Cl3—C4*B*—C5*B*	120.4 (5)
C3*A*—C4*A*—C5*A*	121.3 (6)	C2*A*—C1*A*—C7	117.4 (4)
C4*A*—C5*A*—C6*A*	118.9 (5)	C6*A*—C1*A*—C7	123.4 (4)
C5*A*—C6*A*—C1*A*	120.5 (4)	C2*B*—C1*B*—C7	117.8 (5)
C6*A*—C1*A*—C2*A*	119.0 (5)	C6*B*—C1*B*—C7	122.4 (5)
C1*B*—C2*B*—C3*B*	120.5 (5)	C1*A*—C7—N1	113.0 (5)
C2*B*—C3*B*—C4*B*	119.3 (5)	C1*B*—C7—N1	110.6 (5)
C3*B*—C4*B*—C5*B*	120.5 (6)	C1*A*—C7—C1*B*	111.9 (6)
C4*B*—C5*B*—C6*B*	120.6 (6)	C7—N1—C8*A*	108.3 (5)
C5*B*—C6*B*—C1*B*	119.4 (5)	C7—N1—C8*B*	112.9 (5)
C6*B*—C1*B*—C2*B*	119.7 (6)	C8*A*—N1—C8*B*	113.2 (6)
Cl2—C4*A*—C3*A*	119.8 (4)	N1—C8*A*—C9*A*	111.8 (5)
Cl3—C4*B*—C3*B*	119.1 (4)	N1—C8*B*—C9*B*	114.7 (5)

Crystal source: the compound was synthesized according to non-literature described techniques, by reaction of 4,4′-dichlorobenzhydryl bromide with diethylamine in nitromethane.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71164 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1018]

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Structure of Ethyl 2-Cyano-3-(4-methylphenyl)propenoate

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Abstract

The dihedral angle between the plane of the 4-methylbenzylidene group and the cyanopropenoic acid ethyl ester group is 3.33°. The molecule is nearly planar.

Comment

2-Cyano-3-(2-methoxyphenyl)propenoic acid methyl ester (Nakatani, Hayashi & Hidaka, 1992) crystallizes in a non-centrosymmetric space group and has a large second-harmonic generation (SHG) efficiency. We have synthesized a series of substituted α-cyano cinnamic acid esters. The title compound is one of them, which happens to crystallize in a centrosymmetric space group and therefore has no non-linear optical properties. This has been confirmed by SHG efficiency measurements on a powder sample using the method of Kurtz & Perry (1968).

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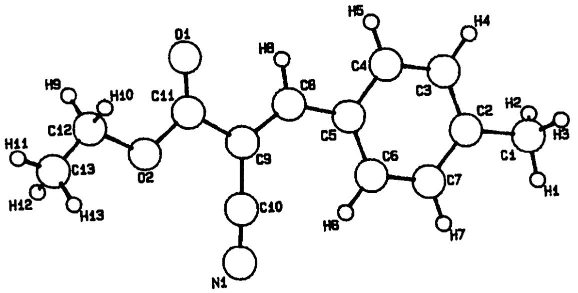

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) diagram of the title compound.

Experimental

Crystal data

$C_{13}H_{13}NO_2$
$M_r = 215.25$
Monoclinic
$P2_1/c$
$a = 6.882\ (5)$ Å
$b = 17.956\ (3)$ Å
$c = 9.584\ (7)$ Å
$\beta = 96.06^\circ$
$V = 1178\ (1)$ Å^3
$Z = 4$
$D_x = 1.21$ Mg m^{-3}
$D_m = 1.214$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14\text{-}15^\circ$
$\mu = 0.77$ mm^{-1}
$T = 296$ K
Prismatic
0.6 × 0.5 × 0.5 mm
Colorless
Crystal source: grown from alcohol solution

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
$T_{\min} = 0.69$, $T_{\max} = 1.00$
2879 measured reflections
2803 independent reflections
1211 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.016$
$\theta_{\max} = 27^\circ$
$h = -8 \rightarrow 8$
$k = -22 \rightarrow 0$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 66.67 min
intensity variation: 20% decrease (correction applied)

Refinement

Refinement on F
Final $R = 0.057$
$wR = 0.067$
$S = 0.823$
1211 reflections
145 parameters
H-atom parameters not refined

Unit weights applied
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\text{eq}} = 8\pi^2/3(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}
O(1)	1.0824 (5)	−0.0994 (2)	0.5071 (4)	8.18 (9)
O(2)	1.1179 (4)	−0.2035 (2)	0.6313 (4)	7.59 (9)
N(1)	0.7791 (6)	−0.2264 (2)	0.8507 (5)	7.5 (1)
C(1)	0.1000 (7)	0.0793 (3)	0.8581 (6)	7.5 (1)
C(2)	0.2736 (6)	0.0407 (3)	0.8083 (5)	5.4 (1)
C(3)	0.3803 (6)	0.0757 (3)	0.7137 (5)	6.1 (1)
C(4)	0.5423 (6)	0.0412 (3)	0.6677 (5)	5.8 (1)
C(5)	0.6006 (6)	−0.0287 (2)	0.7148 (4)	4.73 (9)
C(6)	0.4921 (7)	−0.0640 (3)	0.8087 (5)	6.1 (1)
C(7)	0.3320 (7)	−0.0289 (3)	0.8548 (3)	6.8 (1)
C(8)	0.7705 (6)	−0.0606 (2)	0.6562 (4)	5.2 (1)
C(9)	0.8651 (6)	−0.1254 (2)	0.6786 (4)	5.0 (1)
C(10)	0.8158 (6)	−0.1814 (2)	0.7738 (5)	5.4 (1)
C(11)	1.0326 (6)	−0.1400 (3)	0.5946 (5)	6.1 (1)
C(12)	1.2830 (8)	−0.2232 (3)	0.5503 (8)	12.1 (2)
C(13)	1.344 (1)	−0.2871 (4)	0.578 (1)	15.3 (3)

Table 2. *Bond distances* (Å) *and angles* (°)

O(1)—C(11)	1.189 (6)	C(4)—C(5)	1.379 (6)
O(2)—C(11)	1.315 (6)	C(5)—C(6)	1.383 (6)
O(2)—C(12)	1.485 (7)	C(5)—C(8)	1.467 (6)
N(1)—C(10)	1.140 (6)	C(6)—C(7)	1.382 (6)
C(1)—C(2)	1.502 (6)	C(8)—C(9)	1.340 (6)
C(2)—C(3)	1.376 (6)	C(9)—C(10)	1.422 (7)
C(2)—C(7)	1.373 (6)	C(9)—C(11)	1.497 (6)
C(3)—C(4)	1.387 (6)	C(12)—C(13)	1.242 (9)
C(11)—O(2)—C(12)	114.3 (5)	C(2)—C(7)—C(6)	121.8 (5)
C(1)—C(2)—C(3)	119.8 (5)	C(5)—C(8)—C(9)	132.2 (4)
C(1)—C(2)—C(7)	122.1 (5)	C(8)—C(9)—C(10)	124.9 (4)
C(3)—C(2)—C(7)	118.1 (5)	C(8)—C(9)—C(11)	117.0 (5)
C(2)—C(3)—C(4)	120.6 (5)	C(10)—C(9)—C(11)	118.1 (4)
C(3)—C(4)—C(5)	121.1 (5)	N(1)—C(10)—C(9)	179.0 (6)
C(4)—C(5)—C(6)	118.2 (4)	O(1)—C(11)—O(2)	124.5 (5)
C(4)—C(5)—C(8)	116.5 (4)	O(1)—C(11)—C(9)	124.7 (5)
C(6)—C(5)—C(8)	125.2 (4)	O(2)—C(11)—C(9)	110.8 (5)
C(5)—C(6)—C(7)	120.1 (5)	O(2)—C(12)—C(13)	111.6 (7)

The title compound was prepared by condensation of methylbenzaldehyde and ethyl cyanoacetate using piperidine as a catalyst. Data were collected using *CONTROL* (Molecular Structure Corporation, 1989). The structure was solved by direct methods using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H atoms were placed in geometrically calculated positions with C—H = 0.95 Å. All calculations were performed on a MicroVAX 3100 computer using the *MolEN* (Fair, 1990) program package. Fig. 1 was produced using *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71291 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1046]

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2,11-Dithia[3.3]metacyclophane-9-carboxylic Acid *tert*-Butyl Ester

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Abstract

X-ray structure determination of *tert*-butyl 3,11-dithiatricyclo[11.3.1.1^{5,9}]octadeca-1(17),5,7,9(18),13,15-hexaene-17-carboxylate reveals the molecules adopting a *syn* conformation with the sulfur-containing bridging chains in a boat–chair arrangement. The *syn*-orientated arene rings are tilted with respect to each other forming a dihedral angle of 22.9 (2)°.

Comment

The structure determination of the title compound was undertaken within the context of investigations on the synthesis, reactivity and conformation of intra-annular substituted cyclophanes (Vögtle, Grütze, Nätscher, Wieder, Weber & Grün, 1975).

In the solid state, there are four separate molecules per unit cell, each of them adopting a *syn* conformation, as seen in the parent compound 2,11-dithia[3.3]metacyclophane (Anker, Bushnell & Mitchell, 1979).

A dihedral angle of 22.9 (2)° between the arene rings indicates a slightly stronger strain compared with the unsubstituted compound (dihedral angle 20.6°). The methylene C atoms attached to the rings are displaced from the arene-ring planes. The intramolecular S···S distance of 6.37 Å is shorter than those of the unsubstituted compound (6.97 Å). The other bond lengths and angles are comparable to those of the unsubstituted compound.

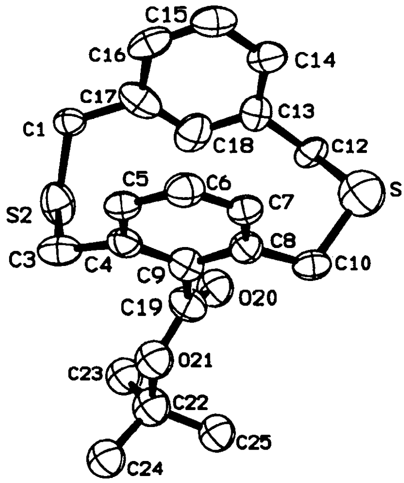

Fig. 1. *ORTEP* view (Johnson, 1965) of the title compound with thermal ellipsoids shown at the 50% probability level.

Experimental

Crystal data

$C_{21}H_{24}O_2S_2$
M_r = 372.54
Monoclinic
$P2_1/a$
a = 13.317 (5) Å
b = 7.624 (1) Å
c = 19.965 (8) Å
β = 104.15 (2)°
V = 1965.4 (8) Å^3
Z = 4
D_x = 1.26 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 15–18°
μ = 0.237 mm^{-1}
T = 293 K
Plate
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
5782 measured reflections
2322 independent reflections
2026 observed reflections $[F_\sigma > 3\sigma(F_o)]$

$R_{\rm int}$ = 0.0608
$\theta_{\rm max}$ = 22°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 8$
$l = -21 \rightarrow 21$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
Final R = 0.0558
wR = 0.0510
2026 reflections
229 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max}$ = 0.003

$\Delta\rho_{\rm max}$ = 0.266 e Å^{-3}
$\Delta\rho_{\rm min}$ = 0.259 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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