Related literature. Methyl 3-(2-ethoxyphenyl)-2-cyanopropenoate (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-cyanocinnamic acid esters of which the title compound is one. It crystallizes 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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Structure of *o,o'*-Bis(1-oxo-2,3-dihydro-1*H*-2-benzopyrrol-2-yl)diphenyl Disulfide

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Abstract. *N,N'*-(2,2'-Dithiodiphenylene)bis(2,3-dihydro-1*H*-2-benzopyrrol-1-one), $C_{28}H_{20}N_2O_2S_2$, M_r = 480.60, monoclinic, $P2_1/a$, $a = 25.05$ (3), $b = 6.203$ (2), $c = 15.89$ (2) Å, $\beta = 109.6$ (1)°, $V = 2326$ Å^3, $Z = 4$, $D_x = 1.372$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.12$ cm^{-1}, $F(000) = 1000$, $T = 293$ K, $R = 0.068$ for 2084 reflections with $I > 2\sigma(I)$. The investigation establishes the structure of the title compound with an S—S bond length of 2.053 (2) Å and S—C distances of 1.781 (6) and 1.787 (6) Å.

Experimental. During an investigation of the synthesis of the ligand (3), formed *via* the reaction of the potassium salt of 2-amino(thiophenol) (1) with phthalaldehyde, we observed that the product was contaminated with a small amount of another compound which, on the basis of microanalytical and spectroscopic data, was determined to be the disulfide derivative (5). In order to confirm the identity of the compound, a single-crystal X-ray structure determination of (5) was carried out.

2-Amino(thiophenol) (5.01 g, 40 mmol) was added to potassium ethoxide formed by the addition of potassium (1.56 g, 40 mmol) to absolute ethanol (20 cm^3). Phthalaldehyde (2.683 g, 20 mmol) was added in small portions to this solution with stirring, and the solution stirred for 2 h at room temperature and then refluxed and stirred for a further 30 min. After cooling to 313 K, a bright-yellow solid suspended in a yellow solution was formed. To this suspension, $NaBH_4$ (0.74 g) was added portionwise. The reaction mixture was stirred at room temperature for 2 h to give a colourless solution and the solvent then removed by distillation under reduced pressure. Water (100 cm^3) was added to the yellow product and the solution acidified (pH 4) with 1*M* HCl to give a yellow oil. The oil was extracted with trichloromethane (3 × 50 cm^3) and the extracts combined and dried with anhydrous $MgSO_4$ for 24 h. Removal of both $MgSO_4.xH_2O$ and solvent gave a pale-yellow oily product, a mixture of (3) and (5) (6.15 g, 87.2%). The mixture of (3) and (5) (1.26 g, 5 mmol) was dissolved in dichloromethane (25 cm^3), cooled to 263 K and treated with a solution of ethanedinitrile

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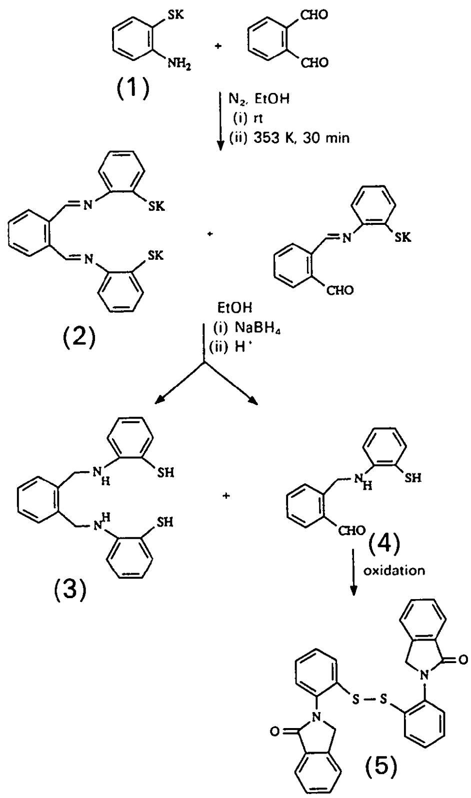


dioxide [prepared from 5,5-dichloroglyoxime (0.785 g, 5 mmol) and 0.5*M* aqueous Na_2CO_3 (25 cm^3)] in dichloromethane (30 cm^3). The mixture was kept at 263 K for a further 10 h and then warmed to room temperature. The pale-yellow solid was filtered off, washed with cold dichloromethane and the dichloromethane solution cooled to 263 K to afford pale-yellow crystals of the title compound (5).

Intensities were measured from a pale-yellow crystal with dimensions 0.58 × 0.25 × 0.08 mm on a Stoe Stadi-2 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Accurate cell parameters were obtained by a least-squares refinement of the optimized setting angles of 267 zero and upper-layer reflections in the angular range $7 \leq 2\theta \leq 30°$. Data were collected by the ω-scan technique. 4395 reflections were measured over the range $7 < 2\theta < 50°$ with $-29 \leq h \leq 29$, $0 \leq k \leq 7$, $0 \leq l \leq 18$. Monitored check reflections indicated no crystal movement or decay. The reflections merged to a unique set of 3840 reflections with 2084 having $I > 2\sigma(I)$ regarded as observed ($R_{\rm int} = 0.0314$).

Data were corrected for Lorentz and polarization effects. No absorption correction was applied to the data. All calculations were performed on a VAX 8650. Systematic absences were consistent with the space group $P2_1/a$. The structure was solved by direct methods using the *TREF* option of *SHELXS86* (Sheldrick, 1986); all subsequent calcula-

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

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
S(1)	0.29381 (6)	0.37066 (28)	0.25394 (11)	0.0563 (7)
S(2)	0.20722 (6)	0.33865 (27)	0.21062 (12)	0.0570 (7)
N(1)	0.38174 (17)	0.7189 (7)	0.28147 (28)	0.0378 (18)
N(2)	0.12834 (18)	0.7110 (8)	0.2147 (3)	0.0428 (20)
O(1)	0.42215 (15)	0.3842 (7)	0.32404 (24)	0.0494 (17)
O(2)	0.08298 (15)	0.3813 (7)	0.18400 (25)	0.0510 (17)
C(1)	0.38204 (29)	0.8915 (12)	0.2187 (5)	0.0478 (27)
C(2)	0.41991 (22)	0.7992 (9)	0.1721 (4)	0.0397 (23)
C(3)	0.43686 (28)	0.8853 (12)	0.1040 (4)	0.0517 (28)
C(4)	0.47272 (26)	0.7616 (12)	0.0737 (5)	0.054 (3)
C(5)	0.49126 (26)	0.5622 (12)	0.1074 (4)	0.0524 (29)
C(6)	0.47469 (24)	0.4771 (12)	0.1753 (4)	0.0438 (26)
C(7)	0.43880 (20)	0.5977 (10)	0.2069 (3)	0.0376 (22)
C(8)	0.41522 (21)	0.5438 (10)	0.2774 (4)	0.0377 (23)
C(9)	0.34902 (21)	0.7315 (10)	0.3400 (4)	0.0393 (23)
C(10)	0.35776 (27)	0.9046 (13)	0.3990 (4)	0.057 (3)
C(11)	0.3254 (3)	0.9236 (15)	0.4544 (5)	0.067 (3)
C(12)	0.2845 (3)	0.7699 (15)	0.4498 (5)	0.071 (4)
C(13)	0.27536 (29)	0.6028 (14)	0.3913 (5)	0.064 (3)
C(14)	0.30665 (21)	0.5816 (10)	0.3344 (3)	0.0406 (22)
C(15)	0.1337 (3)	0.8928 (11)	0.2771 (5)	0.0492 (28)
C(16)	0.09996 (23)	0.8171 (10)	0.3333 (4)	0.0458 (24)
C(17)	0.0888 (3)	0.9207 (15)	0.4029 (5)	0.068 (3)
C(18)	0.0527 (3)	0.8134 (19)	0.4404 (5)	0.076 (4)
C(19)	0.0286 (3)	0.6219 (19)	0.4085 (5)	0.073 (4)
C(20)	0.04127 (28)	0.5150 (15)	0.3415 (5)	0.058 (3)
C(21)	0.07734 (21)	0.6182 (11)	0.3037 (3)	0.0426 (23)
C(22)	0.09541 (22)	0.5473 (10)	0.2282 (4)	0.0386 (23)
C(23)	0.15061 (22)	0.7182 (11)	0.1440 (4)	0.0440 (25)
C(24)	0.13722 (25)	0.8881 (13)	0.0848 (4)	0.0526 (28)
C(25)	0.1565 (3)	0.8971 (18)	0.0136 (5)	0.074 (4)
C(26)	0.1883 (3)	0.7304 (18)	−0.0010 (5)	0.077 (4)
C(27)	0.2023 (3)	0.5588 (16)	0.0565 (5)	0.070 (4)
C(28)	0.18473 (23)	0.5532 (10)	0.1314 (4)	0.0477 (25)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

C(8)—O(1)	1.214 (6)	C(13)—C(12)	1.359 (10)
C(22)—O(2)	1.226 (6)	C(14)—C(13)	1.388 (7)
C(1)—N(1)	1.465 (7)	C(16)—C(15)	1.498 (8)
C(8)—N(1)	1.387 (7)	C(17)—C(16)	1.386 (9)
C(9)—N(1)	1.434 (6)	C(21)—C(16)	1.373 (8)
C(15)—N(2)	1.478 (7)	C(18)—C(17)	1.407 (12)
C(22)—N(2)	1.370 (7)	C(19)—C(18)	1.351 (11)
C(23)—N(2)	1.413 (6)	C(20)—C(19)	1.378 (10)
C(2)—C(1)	1.500 (7)	C(21)—C(20)	1.397 (8)
C(3)—C(2)	1.395 (8)	C(22)—C(21)	1.485 (7)
C(7)—C(2)	1.384 (8)	C(24)—C(23)	1.377 (8)
C(4)—C(3)	1.385 (9)	C(28)—C(23)	1.390 (8)
C(5)—C(4)	1.366 (9)	C(25)—C(24)	1.371 (9)
C(6)—C(5)	1.384 (8)	C(26)—C(25)	1.373 (11)
C(7)—C(6)	1.387 (8)	C(27)—C(26)	1.369 (11)
C(8)—C(7)	1.470 (7)	C(28)—C(27)	1.402 (8)
C(10)—C(9)	1.393 (9)	S(2)—S(1)	2.053 (2)
C(14)—C(9)	1.392 (7)	C(14)—S(1)	1.781 (6)
C(11)—C(10)	1.388 (8)	C(28)—S(2)	1.787 (6)
C(12)—C(11)	1.384 (10)		

C(8)—N(1)—C(1)	113.5 (4)	C(16)—C(15)—N(2)	102.6 (5)
C(9)—N(1)—C(1)	122.5 (4)	C(17)—C(16)—C(15)	129.5 (7)
C(9)—N(1)—C(8)	124.0 (4)	C(21)—C(16)—C(15)	109.2 (5)
C(22)—N(2)—C(15)	112.6 (5)	C(21)—C(16)—C(17)	121.3 (7)
C(23)—N(2)—C(15)	122.5 (5)	C(18)—C(17)—C(16)	116.7 (9)
C(23)—N(2)—C(22)	124.6 (5)	C(19)—C(18)—C(17)	121.7 (8)
C(2)—C(1)—N(1)	101.9 (5)	C(20)—C(19)—C(18)	121.6 (8)
C(3)—C(2)—C(1)	130.2 (6)	C(21)—C(20)—C(19)	117.5 (9)
C(7)—C(2)—C(1)	109.6 (5)	C(20)—C(21)—C(16)	121.1 (6)
C(7)—C(2)—C(3)	120.2 (6)	C(22)—C(21)—C(16)	109.9 (5)
C(4)—C(3)—C(2)	117.4 (7)	C(22)—C(21)—C(20)	129.0 (7)
C(5)—C(4)—C(3)	122.7 (7)	N(2)—C(22)—O(2)	125.6 (5)
C(6)—C(5)—C(4)	120.0 (7)	C(21)—C(22)—O(2)	128.6 (5)
C(7)—C(6)—C(5)	118.5 (7)	C(21)—C(22)—N(2)	105.7 (5)
C(6)—C(7)—C(2)	121.3 (6)	C(24)—C(23)—N(2)	119.8 (6)
C(8)—C(7)—C(2)	109.8 (5)	C(28)—C(23)—N(2)	121.1 (5)
C(8)—C(7)—C(6)	128.9 (6)	C(28)—C(23)—C(24)	119.1 (5)
N(1)—C(8)—O(1)	125.1 (5)	C(25)—C(24)—C(23)	121.5 (8)
C(7)—C(8)—O(1)	129.7 (5)	C(26)—C(25)—C(24)	119.6 (8)
C(7)—C(8)—N(1)	105.2 (5)	C(27)—C(26)—C(25)	120.4 (8)
C(10)—C(9)—N(1)	118.9 (5)	C(28)—C(27)—C(26)	120.2 (8)

Table 2 (*cont.*)

C(14)—C(9)—N(1)	120.8 (5)	C(27)—C(28)—C(23)	119.2 (6)
C(14)—C(9)—C(10)	120.2 (5)	C(14)—S(1)—S(2)	103.1 (2)
C(11)—C(10)—C(9)	119.9 (7)	C(28)—S(2)—S(1)	102.2 (2)
C(12)—C(11)—C(10)	119.2 (7)	C(9)—C(14)—S(1)	119.2 (4)
C(13)—C(12)—C(11)	120.8 (6)	C(13)—C(14)—S(1)	122.2 (5)
C(14)—C(13)—C(12)	121.1 (7)	C(23)—C(28)—S(2)	120.7 (4)
C(13)—C(14)—C(9)	118.7 (6)	C(27)—C(28)—S(2)	120.1 (6)

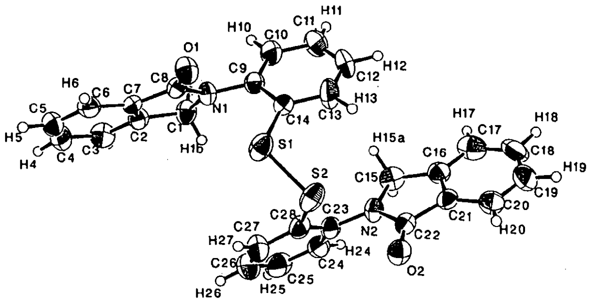
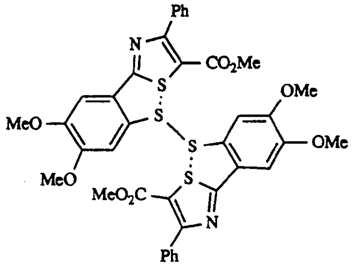


Fig. 1. A perspective view of the title compound with the atom-numbering scheme.

tions used *SHELX76* (Sheldrick, 1976) with full-matrix least-squares calculations on F magnitudes. There is an approximate noncrystallographic centre of symmetry relating the two halves of the molecule. All the non-H atoms were refined anisotropically. H atoms which appeared in Fourier maps were included and refined as isotropic atoms. Final $R = 0.068$ and $wR = 0.058$, $w = [\sigma^2(F) + 0.0038F^2]^{-1}$; ratio of maximum shift/σ = 0.035; 387 parameters refined. Maximum and minimum peak heights in the difference Fourier map were 0.30 and -0.27 e Å^{-3}, respectively. Atomic scattering factors as given in *SHELX*.

Final atomic coordinates are given in Table 1* with selected bond distances and angles in Table 2. A perspective view of the molecule is shown in Fig. 1.

Related literature. Hordvik (1966) gives a correlation between the S—S distance and the dihedral angle around it; the present values of 2.053 (2) Å and 81.6° agree with this. Ricci & Bernal (1970) have observed short intramolecular nearest-neighbour S···ONO contacts (2.636 and 2.588 Å) in the crystal structure of bis(*o*-nitrophenyl) disulfide. Non-bonding S···S interactions (3.117 and 3.110 Å) have also been observed in the thiazole disulfide (6) (Fodor, Szabo, Bernáth, Sohár, Argay, Kálmán & Tamas, 1988). In the title compound, the O(1)···S(1) and O(2)···S(2) contacts of 3.029 and 3.009 Å are also shorter than the sum of the van der Waals radii of S and O atoms (3.25 Å), but the interactions are much weaker.



(6)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71047 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1033]