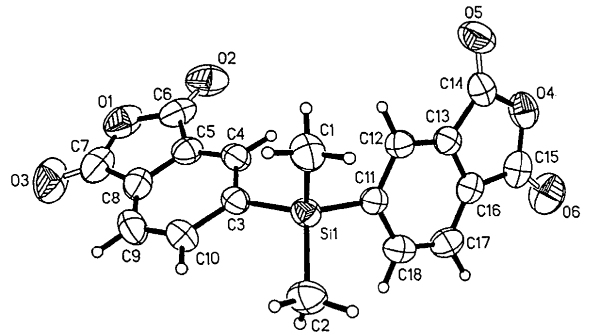

Fig. 1. *ORTEP* plot of the title compound showing the atom-labeling scheme and 50% probability thermal ellipsoids.

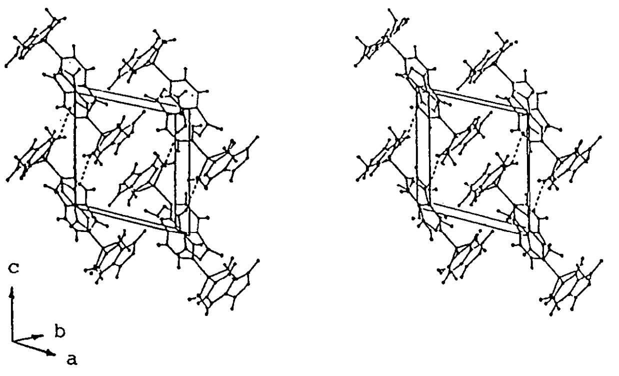

Fig. 2. Stereoview of the unit-cell packing of the title compound.

Related literature. The demand for materials which possess high thermal stability coupled with exceptional physical properties and, especially, outstanding electrical resistance has greatly increased in recent times. Aromatic polyimides are a class of compounds which possess these characteristics and have been extensively studied by Mittal (1984) and Bessonov, Kolton, Kudryavtsev & Louis (1987). Spectroscopic investigation of polyimides is, at best, difficult. Thus, the study of monomeric molecules, which are structurally similar to the repeat unit present in polyimides, has been undertaken. Solid-state ^{13}C NMR data, analyzed by Dinan, Schwartz, Wolfe, Pratt & St Clair (1989) and Dinan, Wolfe, Schwartz, Pratt & St Clair (1990), indicate that alignment of monomeric species occurs in a manner which is consistent with strong electrical interactions between electron-rich and electron-deficient portions of these molecules.

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Structure of Ethyl 2-Cyano-3-(3-hydroxy-4-methoxyphenyl)propenoate

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Abstract. $C_{13}H_{13}NO_4$, $M_r = 247.25$, monoclinic, $P2_1/n$, $a = 9.755$ (5), $b = 12.849$ (9), $c = 10.420$ (8) Å, $\beta = 104.16$ (4)°, $V = 1266$ (2) Å^3, $Z = 4$, $D_x = 1.30$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.91$ cm^{-1}, $F(000) = 520$, $T = 296$ K, $R = 0.052$ for 1690 observed unique reflections with $I > 3\sigma(I)$. The molecules are linked by O—H···N hydrogen bonds [O···N($\frac{1}{2} - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) 2.88 Å, O—H···N 159°].

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	B_{eq}
O(1)	0.2674 (2)	0.1722 (1)	0.7380 (2)	5.05 (8)
O(2)	0.3841 (2)	0.3604 (1)	0.7623 (2)	4.39 (8)
O(3)	0.9434 (2)	−0.1380 (1)	1.0011 (2)	4.45 (7)
O(4)	0.7783 (2)	−0.2627 (1)	0.9564 (2)	3.61 (7)
N(1)	0.4568 (2)	−0.1769 (2)	0.8360 (2)	4.3 (1)
C(1)	0.4416 (3)	0.4629 (2)	0.7706 (3)	4.8 (1)
C(2)	0.4088 (2)	0.1818 (2)	0.7871 (2)	3.21 (9)
C(3)	0.4883 (2)	0.0936 (2)	0.8229 (2)	3.19 (9)
C(4)	0.6354 (2)	0.0999 (2)	0.8753 (2)	3.06 (9)
C(5)	0.6981 (2)	0.1981 (2)	0.8889 (3)	3.8 (1)
C(6)	0.6188 (2)	0.2876 (2)	0.8534 (3)	3.9 (1)
C(7)	0.4740 (2)	0.2802 (2)	0.8016 (2)	3.31 (9)
C(8)	0.7282 (2)	0.0117 (2)	0.9163 (2)	3.12 (8)
C(9)	0.7017 (2)	−0.0916 (2)	0.9167 (2)	3.00 (8)
C(10)	0.5650 (2)	−0.1385 (2)	0.8723 (2)	3.18 (9)
C(11)	0.8228 (2)	−0.1643 (2)	0.9631 (2)	3.10 (9)
C(12)	0.8867 (3)	−0.3419 (2)	0.9935 (3)	4.0 (1)
C(13)	0.8108 (3)	−0.4440 (2)	0.9806 (4)	6.0 (1)

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

O(1)—C(2)	1.354 (3)	C(3)—C(4)	1.407 (3)
O(2)—C(7)	1.350 (3)	C(4)—C(5)	1.395 (3)
O(2)—C(1)	1.425 (3)	C(4)—C(8)	1.449 (3)
O(3)—C(11)	1.195 (3)	C(5)—C(6)	1.385 (3)
O(4)—C(11)	1.332 (3)	C(6)—C(7)	1.387 (3)
O(4)—C(12)	1.450 (3)	C(8)—C(9)	1.353 (3)
N(1)—C(10)	1.142 (3)	C(9)—C(10)	1.433 (3)
C(2)—C(3)	1.372 (3)	C(9)—C(11)	1.491 (3)
C(2)—C(7)	1.407 (3)	C(12)—C(13)	1.497 (4)

C(7)—O(2)—C(1)	118.0 (2)	O(2)—C(7)—C(2)	114.3 (2)
C(11)—O(4)—C(12)	116.4 (2)	C(6)—C(7)—C(2)	119.6 (2)
O(1)—C(2)—C(3)	118.9 (2)	C(9)—C(8)—C(4)	131.5 (2)
O(1)—C(2)—C(7)	120.9 (2)	C(8)—C(9)—C(10)	125.1 (2)
C(3)—C(2)—C(7)	120.2 (2)	C(8)—C(9)—C(11)	118.6 (2)
C(2)—C(3)—C(4)	120.9 (2)	C(10)—C(9)—C(11)	116.3 (2)
C(5)—C(4)—C(3)	118.2 (2)	N(1)—C(10)—C(9)	179.0 (3)
C(5)—C(4)—C(8)	116.8 (2)	O(3)—C(11)—O(4)	124.6 (2)
C(3)—C(4)—C(8)	125.1 (2)	O(3)—C(11)—C(9)	124.6 (2)
C(6)—C(5)—C(4)	121.4 (2)	O(4)—C(11)—C(9)	110.9 (2)
C(5)—C(6)—C(7)	119.8 (2)	O(4)—C(12)—C(13)	106.2 (2)
O(2)—C(7)—C(6)	126.1 (2)		

Experimental. The title compound was prepared by condensation of 3-hydroxy-4-methoxybenzaldehyde and ethyl cyanoacetate using piperidine as a catalyst. A colorless transparent crystal, dimensions 1.5 × 0.5 × 0.3 mm, was grown from an alcohol solution and mounted in a random orientation on a glass fiber. Data were collected with a Rigaku AFC-5*R* diffractometer (*CONTROL* software; Molecular Structure Corporation, 1988). Cell constants were obtained by least-squares analysis of 20 reflection maxima ($3 \leq 2\theta \leq 50°$). $\omega/2\theta$ scans, scan width $(1.68 + 0.35\tan\theta)°$, $2\theta \leq 50°$ ($0 \leq h \leq 11$, $0 \leq k \leq 15$, $-12 \leq l \leq 11$). The intensities of 2348 unique reflections were measured. Three standard reflections were measured periodically and corrections were applied to the intensities to allow for the 1.0% drop in the mean standard intensities observed during data collection. An empirical absorption correction, based on azimuthal scans of three reflections, was applied (range of transmission factors 0.9015 to 1.0000 with an average value of 0.9650). The data were corrected for Lorentz and polarization factors. 1690 reflections with $I \geq 3\sigma(I)$ were obtained and used in the refinement.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and *DIRDIF* (Beurskens, 1984). H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined on F by the full-matrix least-squares technique with anisotropic thermal parameters for the C, N and O atoms. Final $R = 0.052$, $wR = 0.068$ and $S = 1.97$; $w = 1/\sigma^2(F_o)$; $(\Delta/\sigma)_{\max} = 0.006$; 163 parameters. In the final difference synthesis the largest height was 0.21 e Å^{-3}. Anomalous-dispersion corrections were not applied. All calculations were performed on a MicroVAX II computer using the *TEXSAN* (Molecular Structure Corporation, 1987) program package. Scattering factors were taken from Cromer & Waber (1974). The views of the molecule and unit cell were produced by the *PLUTO* program and are shown in Figs. 1 and 2, respectively. The atomic parameters are listed in Table 1,* with important bond lengths and bond angles in Table 2.

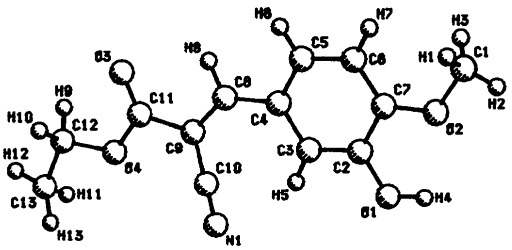

Fig. 1. Structure of the title compound.

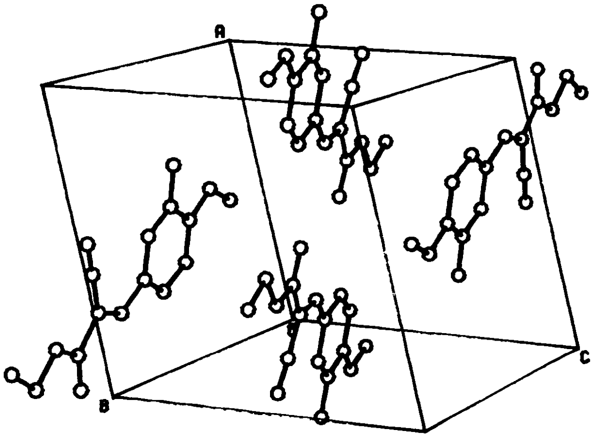

Fig. 2. Packing of the title molecules in the unit cell.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71080 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1043]

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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