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Ethyl 2-Cyano-5-phenyl-(2*E*,4*E*)-pentadienoate

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Abstract. $C_{14}H_{13}NO_2$, tetragonal, $I4_1/a$, a = 33·278 (4), c = 4·608 (1) Å, M_r = 227·27, Z = 16, D_m = 1·17 (flotation), D_x = 1·183 (1) Mg m^{-3}, μ(Cu $K\alpha$) = 0·65 mm^{-1}, m.p. 383–384 K. The structure was solved by direct methods and refined by full-matrix least-squares calculations. The final R was 0·042 for 1061 observed reflections. The molecule is planar, the carbonyl group being *s-trans* with respect to the cyano group.

Introduction. The title compound was synthesized by the Knoevenagel reaction (Cope, 1937) of ethyl cyanoacetate on cinnamaldehyde (Sá & Pitta, 1979).

Pale-yellow prismatic crystals were obtained by slowly evaporating a solution of the compound in ethanol. A crystal of approximate dimensions 0·1 × 0·2 × 0·2 mm was used for the data collection on a Nonius CAD-4 diffractometer. The cell parameters were refined from the setting angles of 25 reflections. The three-dimensional intensity data were collected using graphite-monochromated Cu $K\alpha$ radiation up to 2θ = 120°. The ω–2θ scanning mode with varying interval was used. Of the 1717 recorded independent reflections, 1061 were observed above background [$I > 3\sigma(I)$, where $\sigma(I)$ was based on counting statistics]. The data were reduced to structure factors without absorption correction. The structure was solved by application of *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978).

All the non-H atoms appeared clearly on the E map based on the set of phases giving the highest combined figure of merit. All the H atoms were placed at their calculated positions and were included in the refinement. Refinement was carried out by full-matrix least-squares calculations with anisotropic thermal parameters for the non-H atoms and for H atoms the thermal parameters of the atoms to which they are attached. The function minimized was $\sum w_i(k|F_o| - |F_c|)^2$ where $w_i = |\sigma(F_o)^2 + (0{\cdot}05F_o)^2|^{-1}$ for the observed and $w_i = 0$ for the unobserved reflections. The atomic scattering factors used were those of Cromer & Waber (1974). The final refinement cycle gave R = 0·058 for all reflections and R = 0·042 for the observed reflections only.

The final atomic parameters are given in Table 1.*

Discussion. Bond lengths and angles in ethyl 2-cyano-5-phenyl-(2*E*,4*E*)-pentadienoate are given in Fig. 1, with the atomic numbering.

Although the dihedral angle between the phenyl-ring and the pentadiene-chain planes is 6·4°, the molecule may be described by a mean plane through all non-H atoms whose equation referred to absolute coordinates is $-0{\cdot}1827\,X + 0{\cdot}6527\,Y - 0{\cdot}7352\,Z = 0{\cdot}0175$. The largest distances to this mean plane are observed for the C atoms of the phenyl ring in *ortho* and *meta* positions to the carbon C(6) bonded to the pentadiene chain, for the O atom of the carbonyl group and for the C atoms of the ethyl group and are respectively C(7) −0·124 (2), C(8) −0·127 (3), C(10) 0·101 (3), C(11) 0·087 (3), O(1) −0·120 (2), C(13) −0·113 (3) and C(14) −0·163 (3) Å.

The inclination of the phenyl and ethoxycarbonyl groups with respect to the mean plane of the penta-

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* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35919 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (*Hamilton,* 1959), *with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq} (Å^2)
O(1)	8111 (1)	2677 (1)	2912 (4)	8·1 (3)
O(2)	8716 (1)	2594 (1)	854 (3)	7·1 (3)
N	8718 (1)	1843 (1)	−4095 (5)	8·2 (3)
C(1)	8328 (1)	2514 (1)	1210 (5)	6·4 (3)
C(2)	8199 (1)	2188 (1)	−784 (5)	5·8 (3)
C(3)	7811 (1)	2067 (1)	−869 (5)	6·4 (3)
C(4)	7656 (1)	1754 (1)	−2643 (5)	6·3 (3)
C(5)	7271 (1)	1638 (1)	−2589 (5)	6·6 (3)
C(6)	7089 (1)	1321 (1)	−4285 (5)	6·0 (3)
C(7)	7308 (1)	1059 (1)	−6008 (5)	7·3 (3)
C(8)	7122 (1)	760 (1)	−7585 (6)	8·0 (3)
C(9)	6711 (1)	720 (1)	−7483 (6)	8·1 (3)
C(10)	6492 (1)	969 (1)	−5783 (6)	8·2 (4)
C(11)	6674 (1)	1265 (1)	−4173 (6)	7·0 (3)
C(12)	8493 (1)	2001 (1)	−2594 (5)	6·3 (3)
C(13)	8892 (1)	2888 (1)	2845 (7)	8·9 (4)
C(14)	9314 (1)	2874 (1)	2506 (11)	14·9 (4)

diene chain is defined by the torsion angles C(4)—C(5)—C(6)—C(7) 7·5, O(2)—C(1)—C(2)—C(3) −177·5, C(13)—O(2)—C(1)—C(2) −175·3 and C(1)—O(2)—C(13)—C(14) 168·6 °.

The cyano group is *s-trans* with respect to the carbonyl group. This conformation allows the ethoxy group to arrange itself in continuation of the zig-zag of the pentadiene chain as well as minimizing the molecular dipole moment.

All intermolecular contacts are longer than 3·3 Å.

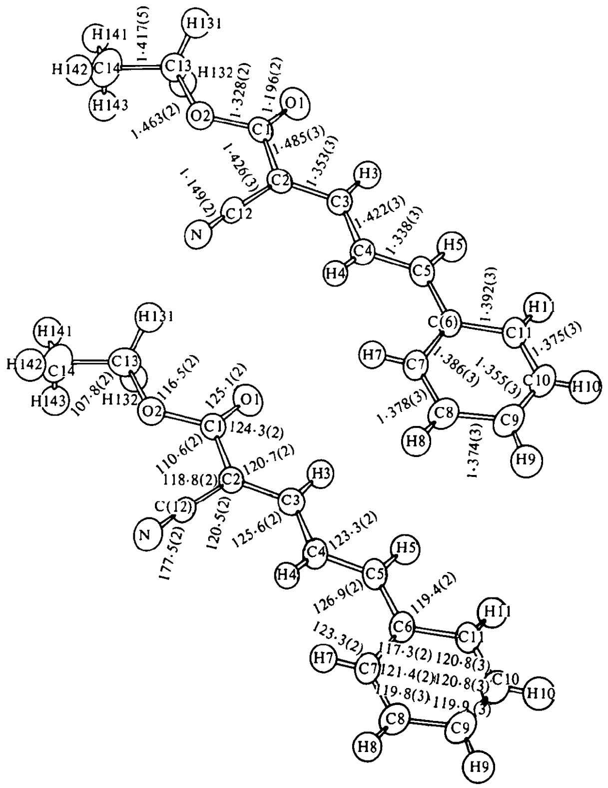

Fig. 1. Representation of the molecule giving bond lengths (Å), angles (°) and the atomic numbering.

All calculations were performed in this Institute on a PDP11/45 computer, using the Nonius SDP crystallographic programs (Frenz, 1978) unless otherwise mentioned in the text. This work has received support from CNPq, FAPESP and FINEP which is hereby gratefully acknowledged.

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