

between the planes are given in Table 4(b). These dihedral angles are naturally in close agreement with those in Gly-L-Trp (Pasternak, 1956) and Ac-L-Trp-OMe (Cotrait & Barrans, 1974). On the other hand, the dihedral angles between the carboxyl plane and the indole ring of the tryptophans which have no peptide linkage at the amino N atom are usually much larger than in these three peptides: 70.6° in L-tryptophan hydrochloride (Takigawa, Ashida, Sasada & Kakudo, 1966), 53.2° in DL-tryptophan formate (Bye, Mostad & Rømming, 1973), 40.1° in 5-hydroxy-DL-tryptophan (Wakahara, Kido, Fujiwara & Tomita, 1973), and 61.5° in DL-tryptophan ethyl ester hydrochloride (Vijayalakshmi & Srinivasan, 1975).

The prominent feature of the crystal structure is the alternate stacking of the polar layer composed of the carboxyl and peptide groups and the non-polar layer of the indole ring along [010]. The N atom of the indole ring is not involved with the hydrogen bond. In the polar layer, two hydrogen bonds are observed: between O(1) and O(3) [$\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$], and between N(2) and O(2) [$x, y, -1 + z$], the distances being 2.580 and 3.110 Å respectively.

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SHORT COMMUNICATION

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Single-crystal X-ray structure determination of ethylene at 85 K. By GERARD J. H. VAN NES and AAFJE VOS, *Laboratorium voor Structuurchemie, Rijksuniversiteit Groningen, Zernikelaan, Paddepoel, Groningen, The Netherlands*

(Received 21 January 1977; accepted 28 January 1977)

Crystalline ethylene at 85 K is monoclinic, $a = 4.626$ (1), $b = 6.620$ (2), $c = 4.067$ (2) Å, $\beta = 94.39$ (2)°, space group $P2_1/n$, $Z = 2$. Refinement of C and H parameters results in an $R = 0.059$ for reflexions up to $\sin \theta/\lambda = 1.07$ Å⁻¹.

Ethylene and other small hydrocarbons attract considerable attention because sophisticated theoretical calculations can relatively easily be done on these molecules in both the free and solid states. Up to now theoretical calculations on, for instance, solid ethylene are hampered by the lack of sufficiently reliable parameters (Elliott & Leroi, 1973). In the present paper we publish a single crystal X ray study of ethylene as part of an accurate study of the electron density distribution in single crystals of C₂H₂, C₂H₄ and C₂H₆.

Spherical ($\phi = 0.58 \pm 0.02$ mm) single crystals (mosaic spread $< 0.4^\circ$) of C₂H₄ were grown *in situ* at 85 K on an

Enraf-Nonius CAD-4 diffractometer. The reflexion symmetry $I(hkl) = I(h\bar{k}l)$ and the systematic extinctions $h0l$ ($h + l = 2n + 1$) and $0k0$ ($k = 2n + 1$) indicated the space group $P2_1/n$, in agreement with Elliott & Leroi (1973), Ligthart (1975) and Brecher & Halford (1961). Cell dimensions were determined from θ , ϕ , ω and κ reflexion angles, measured on the diffractometer.

The observed values of $a = 4.626$ (1), $b = 6.620$ (2), $c = 4.067$ (2) Å and $\beta = 94.39$ (2)° are consistent with those given by Ligthart (1975). Intensities were collected by the θ - 2θ scan technique with graphite-monochromatized Mo $K\alpha$

radiation. All reflexions up to $\sin \theta/\lambda = 1.07 \text{ \AA}^{-1}$ were measured at two different ψ values. From these data the average intensities of 1295 independent reflexions were calculated. This set includes 801 reflexions with $I > 3\sigma(I)$, $\sigma(I)$ being obtained from the variations of the intensities of equivalent reflexions. A small correction ($\pm 1\%$) deduced from the variation of the reference intensities (measured every 30 min) was made, in addition to the usual corrections for Lorentz and polarization effects.

The molecules are situated on centres of symmetry. Approximate coordinates for the C atom were deduced from

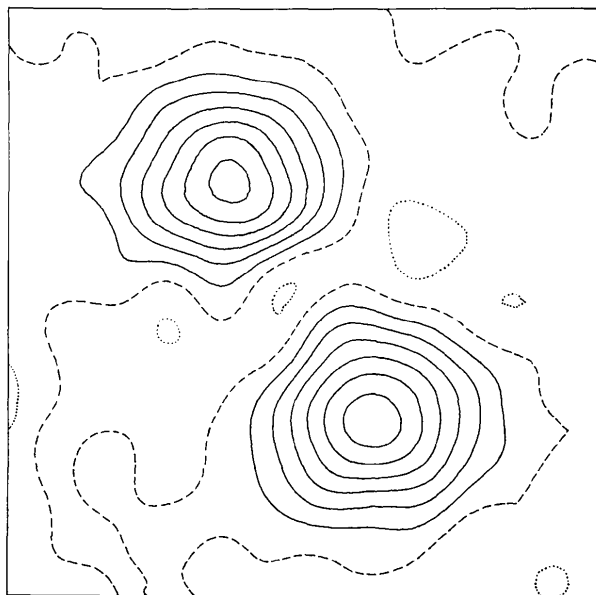


Fig. 1. X-ray difference density section perpendicular to the C=C bond (see text). Contours are at intervals of $0.1 e \text{ \AA}^{-3}$. Full lines are positive, dashed lines zero and dotted lines negative contours. The distance between the maxima is 1.15 \AA .

Table 1. Final fractional atomic coordinates and thermal (10^{-2} \AA^2) parameters

The temperature factor is of the form $\exp\{-2\pi^2[(h_i a^i)(h_j a^j)U^{ij}]\}$, where h_i and a^i are reflexion indices and reciprocal-unit-cell edges respectively. For hydrogen, $U^{ij} = U\delta_{ij}$.

C	x	-0.11633 (30)	U_{11}	3.993 (24)	U_{12}	0.130 (19)
	y	0.05406 (21)	U_{22}	3.372 (22)	U_{13}	0.641 (18)
	z	-0.04077 (35)	U_{33}	3.668 (24)	U_{23}	0.085 (19)
H(1)	x	-0.2317 (19)	$U = 6.48 (17)$			
	y	0.0326 (13)				
	z	-0.2537 (22)				
H(2)	x	-0.1854 (17)	$U = 6.48 (17)$			
	y	0.1651 (13)				
	z	0.1089 (19)				

the literature. After isotropic least-squares refinement, the H atoms were clearly seen in a section of the difference density perpendicular to C=C and at a distance of 0.55 \AA from the adjacent C atom (Fig. 1). For this and further refinements, all 1295 independent reflexions were used with unit weights. Scattering factors were taken from Cromer & Mann (1968) for C and from Stewart, Davidson & Simpson (1965) for H. The C positions (Table 1) were obtained from an anisotropic least-squares refinement in which only the C atoms were considered. In the further refinement the thermal parameters U_{ij} for C were varied plus the positions and the isotropic temperature factor for H, with the constraint $U_{ij}(\text{H1}) = U_{ij}(\text{H2})$. The residual $R = [\sum (F_o - F_c)^2 / \sum F_o^2]^{1/2}$ decreased from 0.104 to 0.059. Final parameters are listed in Table 1.* For the calculations the set of programs in the XRAY system (1975) was used.

The bond lengths and angles are C=C = $1.314 (6)$, C-H(1) = $0.992 (13)$, C-H(2) = $1.021 (9) \text{ \AA}$, H(1)-C-H(2) = $116.8 (7)$, H(1)-C-C' = $121.2 (5)$, H(2)-C-C' = $122.0 (5)^\circ$. Because of librational motion and bonding effects, the C=C and C-H bonds are shorter than the values of $1.336 (3)$ and $1.089 (3) \text{ \AA}$ obtained by electron diffraction (Kuchitsu, 1966, 1968). Further refinement, including multipoles (Stewart, 1976) and higher cumulants in the temperature factors (Johnson, 1968), to account for bonding effects and librational motion respectively, are in progress.

Part of the research has been supported by the Dutch Organization for the Advancement of Pure Research (ZWO). The computations were carried out on the CYBER 74-16 computer of the University of Groningen.

* A table of structure factors has been deposited with the editors. At the request of the authors, it will not be made available for general reference until the electron density calculations have been completed.

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