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Crystallography of the Even *n*-Alkanes: Structure of $C_{20}H_{42}$

BY S. C. NYBURG AND A. R. GERSON

Department of Chemistry, King's College, University of London, Strand, London WC2R 2LS, England

(*Received* 26 *June* 1991; *accepted* 18 *September* 1991)

Abstract

n-Eicosane, $M_r = 282.55$, triclinic, $P\bar{1}$, $a = 4.293\ (5)$, $b = 4.84\ (1)$, $c = 27.35\ (9)$ Å, $\alpha = 85.3\ (3)$, $\beta = 68.2\ (1)$, $\gamma = 72.6\ (1)^\circ$, $V = 503.2$ Å^3, $Z = 1$, $D_x = 0.932$ Mg m^{-3}, $\lambda(\text{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 0.34$ mm^{-1}, $F(000) = 162$, $T = 300$ K, $wR = 0.074$, $R = 0.090$ for 649 significant reflections out of a total of 1251 [$I > 2.5\sigma(I)$]. The triclinic even *n*-alkanes up to $n = 22$ form an isostructural series.

Introduction

It is a matter of some difficulty to obtain accurate X-ray crystal structure analyses of long-chain *n*-alkanes. By virtually whatever method they are crystallized, they invariably adopt a thin platy habit, a consequence of their underlying layer structure. Such thin plates are often slightly warped and this prevents their precise alignment on a diffractometer. The best orientation matrix obtainable usually yields cell parameters with errors substantially larger than those normally acceptable. Nevertheless, accepting these limitations, it is possible to obtain X-ray structure analyses which, although not of the highest accuracy, can yield useful structural information. We report the X-ray crystal structure analysis of *n*-eicosane, $C_{20}H_{42}$. Its relation to the crystal structures of other even *n*-alkanes is given in the *Discussion*.

Experimental

A large crystalline agglomeration (25 × 10 × 4 mm) of *n*-$C_{20}H_{42}$ (Aldrich Chemical Company Ltd) was grown from *n*-dodecane, $C_{12}H_{20}$, by slow cooling (< 0.01 K per day) over a period of two weeks. From this, a flat plate was cut (dimensions 1.5 × 0.5 × 0.1 mm) and mounted on a Picker four-circle diffractometer. Using Ni-filtered Cu $K\alpha$ radiation, unit-cell dimensions were derived from 26 reflections, $34.0 < 2\theta < 80.0^\circ$. Data were collected in the range $3.0 < 2\theta$

0108-7681/92/010103-04$03.00

Table 1. *Atomic fractional coordinates and equivalent isotropic temperture factors* (Å²) *for* $C_{20}H_{42}$

	x	y	z	B_{iso}
C(1)	0.095 (3)	0.280 (2)	−0.4469 (3)	9.0 (7)
C(2)	0.136 (2)	0.081 (2)	−0.4032 (3)	8.0 (6)
C(3)	0.071 (2)	0.240 (2)	−0.3528 (3)	7.2 (6)
C(4)	0.112 (2)	0.039 (2)	−0.3086 (3)	6.9 (6)
C(5)	0.047 (2)	0.200 (2)	−0.2582 (3)	6.9 (6)
C(6)	0.085 (2)	0.000 (2)	−0.2141 (3)	6.6 (5)
C(7)	0.019 (2)	0.159 (2)	−0.1636 (3)	6.8 (6)
C(8)	0.057 (2)	−0.039 (2)	−0.1197 (3)	6.4 (5)
C(9)	−0.008 (2)	0.120 (2)	−0.0693 (3)	6.5 (6)
C(10)	0.032 (2)	−0.079 (2)	−0.0252 (2)	6.7 (6)
H(1*A*)	−0.164	0.416	−0.4343	11 (3)
H(1*B*)	0.272	0.404	−0.4572	11 (3)
H(1*C*)	0.132	0.171	−0.4812	12 (3)
H(2*A*)	−0.043	−0.041	−0.3945	11 (3)
H(2*B*)	0.392	−0.060	−0.4161	10 (2)
H(3*A*)	−0.186	0.381	−0.3398	8 (2)
H(3*B*)	0.250	0.362	−0.3614	10 (2)
H(4*A*)	−0.067	−0.083	−0.2999	9 (2)
H(4*B*)	0.369	−0.102	−0.3215	10 (3)
H(5*A*)	−0.209	0.342	−0.2455	9 (2)
H(5*B*)	0.227	0.320	−0.2668	10 (2)
H(6*A*)	−0.096	−0.120	−0.2056	9 (2)
H(6*B*)	0.340	−0.142	−0.2269	7 (2)
H(7*A*)	−0.237	0.301	−0.1509	8 (2)
H(7*B*)	0.200	0.279	−0.1722	8 (2)
H(8*A*)	−0.124	−0.158	−0.1110	8 (2)
H(8*B*)	0.312	−0.181	0.1325	8 (2)
H(9*A*)	−0.263	0.262	−0.0565	8 (2)
H(9*B*)	0.173	0.240	−0.0781	9 (2)
H(10*A*)	−0.144	−0.204	−0.0170	9 (2)
H(10*B*)	0.289	−0.219	−0.0380	8 (2)

Table 2. *Bond lengths* (Å) *and angles* (°) *for n*-$C_{20}H_{42}$

C(1)—C(2)	1.50 (1)	C(1)—C(2)—C(3)	113.4 (7)
C(2)—C(3)	1.53 (1)	C(2)—C(3)—C(4)	113.3 (7)
C(3)—C(4)	1.52 (1)	C(3)—C(4)—C(5)	113.0 (7)
C(4)—C(5)	1.53 (1)	C(4)—C(5)—C(6)	113.1 (7)
C(5)—C(6)	1.51 (1)	C(5)—C(6)—C(7)	113.4 (7)
C(6)—C(7)	1.53 (1)	C(6)—C(7)—C(8)	113.5 (7)
C(7)—C(8)	1.50 (1)	C(7)—C(8)—C(9)	113.5 (7)
C(8)—C(9)	1.53 (1)	C(8)—C(9)—C(10)	113.4 (6)
C(9)—C(10)	1.51 (1)	C(9)—C(10)—C(10)′	113.6 (6)
C(10)—C(10)′	153 (1)		

Table 3. *Anisotropic temperature factors for n*-$C_{20}H_{42}$ ($Å^2 \times 100$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	13.2 (9)	12.8 (7)	7.4 (6)	−4.0 (8)	−2.9 (5)	1.0 (5)
C(2)	11.5 (8)	10.2 (6)	8.1 (5)	−2.8 (7)	−2.9 (5)	0.2 (5)
C(3)	10.1 (7)	9.0 (6)	7.7 (5)	−3.1 (6)	−2.4 (5)	1.0 (5)
C(4)	10.5 (8)	8.1 (5)	7.5 (5)	−3.4 (7)	−2.7 (5)	0.7 (5)
C(5)	10.1 (7)	8.4 (5)	7.3 (5)	−3.3 (6)	−2.6 (5)	0.6 (4)
C(6)	8.9 (7)	7.8 (5)	7.8 (6)	−2.5 (6)	−2.4 (5)	0.9 (5)
C(7)	9.4 (7)	8.2 (5)	8.0 (6)	−3.2 (6)	−2.7 (5)	1.1 (5)
C(8)	9.2 (7)	7.7 (5)	7.1 (5)	−2.9 (6)	−2.4 (5)	0.6 (4)
C(9)	9.5 (8)	8.0 (5)	7.2 (5)	−3.2 (6)	−2.7 (5)	0.8 (5)
C(10)	9.3 (7)	8.1 (5)	7.6 (5)	−2.8 (6)	−2.3 (5)	0.9 (4)

< 110.0°, each reflection being scanned over 2° in θ–2θ-scan mode. The intensity of a standard reflection was measured every 25 reflections and showed no significant decay. The index ranges were h −4 to 4, k 0 to 3 and l −25 to 24. Friedel-related reflections were not measured. No absorption corrections were made. Phase sets in $P\bar{1}$ were generated by *SOLVER* (Gabe, Le Page, Charland, Lee & White, 1989). The second of these in order of merit gave the carbon backbone. Hydrogen positions were located assuming a C—H bond length of 1.0 Å. The fractional coordinates and anisotropic temperature factors of the carbon atoms and the isotropic temperature factors of the hydrogen atoms were refined using the *NRCVAX* package (Gabe, Le Page, Charland, Lee & White, 1989). Anomalous dispersion was taken into account for the carbon atoms but not the hydrogen atoms, with scattering factors taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 99).

The final R was 0.090 with wR of 0.074, $w = 1/\sigma^2(F_o)$. The goodness of fit was 10.2. Refinement of the 112 parameters was based on F values. The maximum Δ/σ for the final refinement cycle was less than 0.0005. The highest density on the ΔF map was 0.13 and the largest hole −0.15 e Å^{-3}. Three strong reflections were excluded owing to their extremely asymmetric background, $0,\bar{1},\bar{1}\bar{1}$ [abs($F_o - F_c$) = 37], $1\bar{1}0$ [abs($F_o - F_c$) = 35] and 111 [abs($F_o - F_c$) = 96].

Table 1 gives final fractional coordinates and equivalent isotropic thermal factors.* Table 2 gives C—C bond distances and C—C—C angles. The structure is illustrated in Fig. 2(*b*). The bond lengths alternate slightly. The standard deviations indicate this may not be significant although a similar observation was made in the structure of C_8H_{18} (Mathisen, Norman & Pedersen, 1967). Table 3 shows the anisotropic thermal parameters of $C_{20}H_{42}$

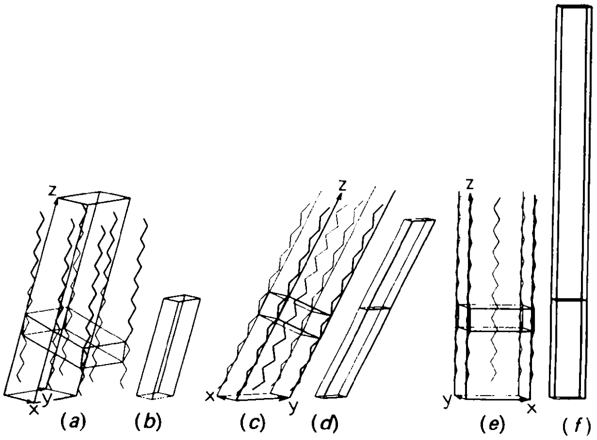

Fig. 1. A comparison of the three subcells in even *n*-alkanes up to $C_{36}H_{74}$. (*a*) The packing in triclinic, $P\bar{1}$, $Z = 1$ unit cells. The subcell is nearly monoclinic with space group *B*2/*m*. (*b*) The triclinic unit cell for $C_{18}H_{38}$. (*c*) The packing in $C_{36}H_{74}$, monoclinic $P2_1/a$, $Z = 2$ which has an orthorhombic subcell of space group *Pbcn*. (*d*) The unit cell for monoclinic $C_{36}H_{74}$. (*e*) The packing in orthorhombic $C_{36}H_{74}$, $Z = 4$, space group $Pca2_1$. The subcell is identical to that of the monoclinic form of $C_{36}H_{74}$. The difference between the two structures is in the end group packing. (*f*) The bilayer unit cell of orthorhombic $C_{36}H_{74}$.

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* A iist of structure factors has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54589 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Anisotropic temperature factors for n*-$C_{18}H_{38}$ ($Å^2 \times 100$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	11.48	9.75	8.33	−3.19	−4.57	−0.05
C(2)	12.58	8.25	6.18	−2.57	−5.14	0.00
C(3)	9.58	5.90	7.26	−2.39	−4.21	0.05
C(4)	9.07	6.43	6.18	−2.30	−3.37	−0.05
C(5)	8.34	5.79	6.18	−2.21	−3.02	0.05
C(6)	8.34	6.11	6.45	−2.21	−3.81	0.05
C(7)	7.83	5.57	6.18	−2.12	−3.28	0.00
C(8)	7.53	4.93	6.18	−2.04	−3.28	0.05
C(9)	7.02	5.68	6.72	−2.12	−3.37	0.05

and Table 4 those of $C_{18}H_{38}$ (Nyburg & Lüth, 1972). They are comparable in magnitude and sign. In both cases the main vibration is normal to the plane of the chain backbone with the magnitude of vibration increasing towards the ends of the carbon chain (Fig. 2). The latter trend is also shown by the isotropic thermal parameters of the hydrogen atoms.

The closest H···H distances for $C_{20}H_{42}$ are [2.7 (1) Å] between adjacent alkane chains and are of similar magnitude to the distances given for $C_{18}H_{38}$ (Nyburg & Lüth, 1972).

Discussion

Prior to the current analysis, the detailed crystal structures of three triclinic *n*-alkanes with an even number of carbon atoms were known. Those having fewer than 18 carbon atoms melt below 298 K and the structures of two such compounds are known: C_6H_{14} (177.7 K; Norman & Mathisen, 1961*b*) and C_8H_{18} (216.3 K; Norman & Mathisen, 1961*a*; Mathisen, Norman & Pedersen, 1967). The data were recorded photographically and not all reflections accessible to Cu *Kα* radiation were measured. The accuracy of these analyses was therefore limited. A more complete set of photographic data was collected from $C_{18}H_{38}$ (Hayashida, 1962). The structure was derived from these data by Nyburg & Lüth (1972).

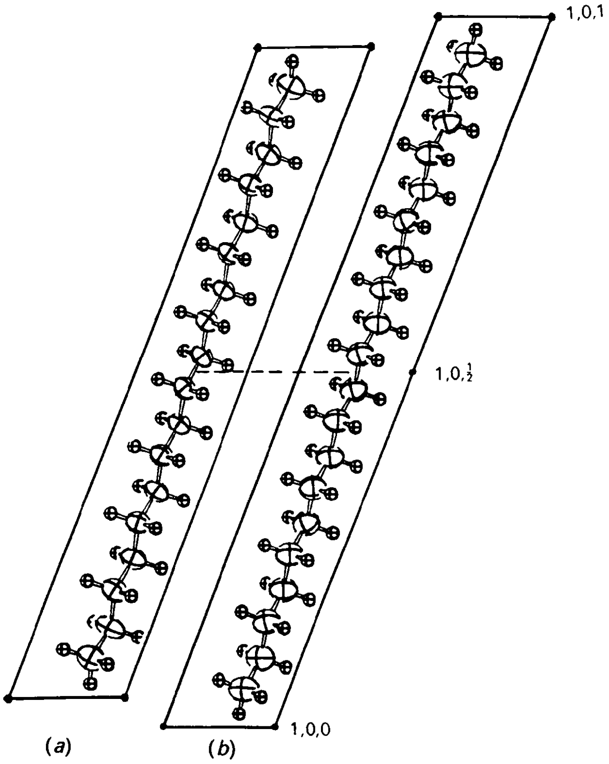

Fig. 2. *ORTEP* (Johnson, 1965) plots of the structures of (*a*) $C_{18}H_{38}$ and (*b*) $C_{20}H_{42}$ showing the differing inclinations of the central C—C bonds.

These three *n*-even crystal structures have space group $P\bar{1}$ with one centrosymmetric molecule per cell. They constitute a set from which, by interpolation and extrapolation, the unit cells of any other member can be predicted (Nyburg & Potworowski, 1973). For these predictions, it was assumed that the *a*, *b* and *γ* cell parameters of $C_{18}H_{38}$ would be the same throughout the set. This seems to be generally the case. The cell dimensions given for $C_{20}H_{42}$ fit well with those predicted: $a = 4.285$, $b = 4.82$, $c = 27.43$ Å, $\alpha = 85.66$, $\beta = 68.16$, $\gamma = 72.7°$. [The cell given in the *Abstract*, when reduced, is $a = 4.293$, $b = 4.84$, $c = 25.45$ Å, $\alpha = 90.74$, $\beta = 93.49$, $\gamma = 107.38°$ as compared to that previously determined from powder diffraction data (Crissman, Passaglia, Eby & Colson, 1970): $a = 4.281$, $b = 4.820$, $c = 25.52$ Å, $\alpha = 91.18$, $\beta = 93.52$, $\gamma = 107.35°$.] In these *n*-even alkanes the molecules are parallel and form layers in which they subtend 70° with the basal plane (Fig. 1*a*).

Powder diffraction has confirmed the cell-parameter prediction for $C_{22}H_{46}$ (Gerson, Roberts & Sherwood, 1991). Powder photographs of $C_{24}H_{50}$ and $C_{26}H_{54}$ (Hoffman & Decker, 1953) show long spacings *d*(001) in agreement with the predictions. Three forms have been reported for $C_{28}H_{58}$. A triclinic form grown from light petroleum ether at temperatures below 283 K has been reported (Boistelle, 1980) but no structural details published.

There are no reports of any higher even *n*-alkanes having the single-layer triclinic structure. Nothing appears to be known of the structures of $C_{30}H_{62}$ through to $C_{34}H_{70}$ although, from powder photographs, they have been classified as orthorhombic [$C_{30}H_{62}$, Müller (1930); $C_{32}H_{66}$, Broadhurst (1962); $C_{34}H_{70}$, Piper & Malkin (1930)]. $C_{36}H_{74}$ has three known polymorphs. Two have had their structures determined from photographic data. First is a monoclinic form, space group $P2_1/a$, two molecules to the cell, crystallized from light petroleum solution of purity less than 99% (Shearer & Vand, 1956). This structure is in some ways analogous to the even triclinic set in that parallel molecules form single layers. However, the backbones of the chains lie in the *xz* plane and make 63° with the basal *xy* plane

Table 5. *Subcells of the known even n-alkanes* (Å, °)

	a	*b*	*c*	*α*	*β*	*γ*	Subcell space group
C_6H_{14}	7.88	4.70	2.56	90.8	88.6	73.9	*B2/m*
C_8H_{18}	8.01	4.79	2.54	91.2	89.2	72.9	*B2/m*
$C_{18}H_{38}$	8.14	4.82	2.55	90.8	89.1	71.6	*B2/m*
$C_{20}H_{42}$	8.15	4.84	2.54	90.3	89.2	71.5	*B2/m*
$C_{36}H_{74}$ monoclinic	4.95	7.42	2.55	90.0	90.0	90.00	*Pbcn*
$C_{36}H_{74}$ orthorhombic	4.96	7.42	2.54	90.0	90.0	90.00	*Pbcn*

(Fig. 1*c*). An analogous structure has been inferred from the powder data of a second form of $C_{28}H_{58}$ crystallized from dodecane (Gerson, Roberts & Sherwood, 1991). Second is an orthorhombic form of $C_{36}H_{74}$, space group $Pca2_1$, which has been grown from petroleum ether of high purity (Teare, 1959). This has a bilayer structure in which the molecules are in pairs of layers with their backbones normal to the basal plane and in which the unit cell spans two layers (Fig. 1*e*). A third polymorphic structure of $C_{36}H_{74}$ was found by Boistelle, Simon & Pepe (1976) by crysallization of $C_{36}H_{74}$ of purity greater than 95% from light petroleum. This latter structure is an alternating bilayer arrangement of the monoclinic structure. An analogous orthorhombic structure has been reported for a third form of $C_{28}H_{58}$ (Boistelle, Simon & Pepe, 1976).

The triclinic even *n*-alkanes, *n* less than or equal to 22, can be regarded as an isostructural series. However, in going from $C_{18}H_{38}$ to $C_{20}H_{42}$ it is of interest to note the differing inclinations of the central C—C bonds lying on the symmetry centres. The difference in inclination corresponds to a rotation of 180° of the molecule about its long axis (Fig. 2). This happens, of course, to preserve identical inclinations of the outermost C—C bond, thus ensuring the same, and evidently most favourable, end-group packing. Thus, as far as the inclination of the central C—C bond is concerned, the even *n*-alkanes C_6H_{14}, $C_{10}H_{22}$, $C_{14}H_{30}$, $C_{18}H_{38}$ and $C_{22}H_{46}$ form one series and C_8H_{18}, $C_{12}H_{26}$, $C_{16}H_{34}$ and $C_{20}H_{42}$, another.

We have examined the chain packing in the *n*-even alkanes. Alternate carbon atoms were replaced by the best-fitting straight line to give the direction of the edge of the subcell along the backbone. The spacing along the backbone is given by the separation of alternate carbon atoms along it. For all the *n*-even alkane $P\bar{1}$ triclinic structures the subcell is closely that, *B2/m*, proposed by Segerman (1965). The subcell parameters for the six known single molecular layer structures are given in Table 5. In the monoclinic, orthorhombic and bilayer structures of $C_{36}H_{74}$ the packing has an orthorhombic subcell of space group *Pbcn*, again one of those predicted by Segerman (1965). A comparison of the single-layer structures and their subcells is given in Fig. 1.

The authors gratefully acknowledge financial support provided by Exxon Chemicals Ltd.

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