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The Crystal Structure of Lower Paraffins

IV. Refinement of the Crystal Structures of Pentane and Octane

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The crystal structures of pentane and octane have been refined from partial three-dimensional X-ray diffraction data. The refinement consisted of difference syntheses and full matrix least squares refinement of positional and thermal parameters. The cell constants have also been redetermined. The unit cell dimensions are:

pentane: $a = 4.10 \pm 0.02$ Å, $b = 9.076 \pm 0.018$ Å, $c = 14.859 \pm 0.012$ Å

octane: $a = 4.22 \pm 0.02$ Å, $b = 4.79 \pm 0.02$ Å, $c = 11.02 \pm 0.02$ Å, $\alpha = 94.7 \pm 0.2°$, $\beta = 84.3 \pm 0.2°$, $\gamma = 105.8 \pm 0.2°$

In pentane the average C–C bond length is 1.533 ± 0.006 Å, the C–C repeat distance is 2.539 ± 0.006 Å, and the average C–C–C angle is $112.1 \pm 0.3°$. The shortest intermolecular C–C distances are 3.90 ± 0.01 Å, 3.95 ± 0.01 Å, and 3.96 ± 0.01 Å. In octane the average C–C bond length is 1.525 ± 0.007, the C–C repeat distance is 2.545 ± 0.007, and the average C–C–C angle is $113.3 \pm 0.6°$. The shortest intermolecular C–C distance is 3.65 ± 0.01 Å.

The crystal structures of pentane and octane have been determined previously from two-dimensional X-ray data in this laboratory.[1,2] Pentane crystallizes in the orthorhombic system, space group *Pbcn*. Octane crystallizes in the triclinic system, space group $P\bar{1}$.

In connection with the two-dimensional work, equi-inclination Weissenberg photographs were taken of zero, first, second, and third layer lines about the *a*-axes for both pentane and octane. The intensities were estimated visually, but the partial three-dimensional data were not fully utilized in the refinements owing to scarce computing facilities. Since then the situation has changed, and it was decided to carry through a three-dimensional refinement including all available data totalling about 300 independant reflections for pentane and 500 independant reflections for octane.

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REFINEMENT

1. Cell dimensions. To secure maximum accuracy in bond lengths we found it necessary to redetermine the unit cell dimensions. For pentane the *b*- and *c*-axes were found from a Weissenberg diagram using KCl powder lines on a separate film for calibration of diffraction angles. The *a*-axis could only be determined from oscillation diagrams, and hence the accuracy of this axis is not as high as of the other ones.

The cell constants for octane have been redetermined partly from a Weissenberg diagram with KCl as an external standard, and partly by a least squares adjustment to the observed line positions in a Debye-Scherrer powder diagram taken at approximately $-80°C$, and using α-SiO_2 as an internal standard. Due to serious overlap, only 14 of the strongest powder lines of octane up to a diffraction angle of $2\theta = 41°$ could be utilized in the refinement.

The unit cell dimensions with estimated standard deviations are:

Pentane: $a = 4.10 \pm 0.02$ Å, $b = 9.076 \pm 0.018$ Å, $c = 14.859 \pm 0.012$ Å
Octane: $a = 4.22 \pm 0.02$ Å, $b = 4.79 \pm 0.02$ Å, $c = 11.02 \pm 0.02$ Å,
$\alpha = 94.7 \pm 0.2°$, $\beta = 84.3 \pm 0.2°$, $\gamma = 105.8 \pm 0.2°$.

2. Coordinates and temperature factors. Three-dimensional $(F_o - F_c)$ syntheses were calculated to locate the hydrogen atoms. The hydrogens all appeared as sharp peaks, and no false peaks of nearly the same electron density were observed. The reliability factor based on these coordinates and the fractional coordinates and temperature factors from the two-dimensional work [1,2] for carbon was 0.161 for pentane and 0.132 for octane.

The positional parameters for all atoms were then refined by the full matrix least squares method.[3] Anisotropic temperature factors were used for the carbon atoms, whereas the individual thermal parameters for the hydrogen atoms were kept isotropic throughout the process. The first 4 cycles reduced the *R*-value to 0.158 and 0.125 for pentane and octane respectively. A recheck of the intensity data at this stage revealed a few false β-reflections. When these were eliminated and a few very strong reflections obviously suffering from extinction were excluded from the calculations, the reliability factor after 4 cycles of refinement dropped to the value 0.084 for pentane and 0.092 for octane. The final positional parameters and their standard deviations are given in Table 1 for pentane and Table 2 for octane.

The anisotropic thermal parameters were analysed. The root mean square amplitude of the motion along the principle axes of the thermal ellipsoid and the components of this motion along the crystallographic axes are given in Tables 3 and 4. In these tables are also included the isotropic temperature factors for the hydrogen atoms.

Interatomic distances and angles calculated from the parameter values in Table 1 and 2 are given in Table 5 for pentane and in Table 6 for octane. In Table 7 and 8 are listed observed and calculated structure factors for the two compounds.

Table 1. Pentane. Positional parameters and their standard deviations in fractions of corresponding cell edges.

	x	y	z
C_1	−0.1959	0.1325	0.0878
	(0.0015)	(0.0006)	(0.0003)
C_2	−0.0923	0.0374	0.1685
	(0.0016)	(0.0005)	(0.0003)
C_3	0.0000	0.1313	0.2500
	(0.0000)	(0.0007)	(0.0000)
H_1	−0.255	0.0739	0.0384
	(0.015)	(0.0054)	(0.0035)
H_2	0.015	0.1986	0.0616
	(0.018)	(0.0074)	(0.0042)
H_3	−0.443	0.2029	0.1015
	(0.017)	(0.0075)	(0.0038)
H_4	0.090	−0.0290	0.1521
	(0.014)	(0.0060)	(0.0033)
H_5	−0.297	−0.0294	0.1897
	(0.023)	(0.0082)	(0.0044)
H_6	0.227	0.1983	0.2283
	(0.015)	(0.0061)	(0.0036)

Table 2. Octane. Positional parameters and their standard deviations in fractions of corresponding cell edges.

	x	y	z
C_1	0.2998	0.2462	0.3804
	(0.0013)	(0.0009)	(0.0003)
C_2	0.1717	0.0183	0.2781
	(0.0011)	(0.0008)	(0.0003)
C_3	0.1378	0.1548	0.1618
	(0.0011)	(0.0007)	(0.0003)
C_4	0.0182	−0.0687	0.0576
	(0.0011)	(0.0007)	(0.0003)
H_1	0.336	0.1554	0.4466
	(0.011)	(0.0090)	(0.0047)
H_2	0.543	0.3680	0.3495
	(0.011)	(0.0090)	(0.0038)
H_3	0.106	0.3740	0.4098
	(0.014)	(0.0105)	(0.0047)
H_4	−0.053	−0.1217	0.3055
	(0.009)	(0.0076)	(0.0032)
H_5	0.339	−0.1169	0.2531
	(0.011)	(0.0090)	(0.0039)
H_6	0.364	0.2871	0.1356
	(0.009)	(0.0075)	(0.0031)
H_7	−0.014	0.2915	0.1841
	(0.011)	(0.0092)	(0.0038)
H_8	−0.197	−0.1891	0.0847
	(0.010)	(0.0079)	(0.0033)
H_9	0.197	−0.2019	0.0337
	(0.011)	(0.0089)	(0.0039)

Table 3. Pentane. Root mean square vibrational displacements in Å along the principal axes of the thermal ellipsoid, and the principal axes components in Å along the direct cell edges.

	Principal axis	Root mean square amplitude $\sqrt{\langle u^2 \rangle}$	Principal axis components in Å x	y	z		B
	1	0.225	0.225	0	0.009	H_1	2.28
C_1	2	0.175	0.007	0	−0.176	H_2	4.79
	3	0.182	0	0.182	0	H_3	3.89
						H_4	1.98
	1	0.169	0.019	0.023	0.166	H_5	6.13
C_2	2	0.256	0.253	−0.030	−0.025	H.	3.84
	3	0.226	0.023	0.223	−0.034		
	1	0.179	0.011	0.077	0.161		
C_3	2	0.228	0.223	0.035	−0.032		
	3	0.200	0.040	−0.178	0.083		

Table 4. Octane. Root mean square vibrational displacements in Å along the principal axes of the thermal ellipsoid, and the principal axes components in Å along the direct cell edges.

	Principal axis	Root mean square amplitude $\sqrt{\langle u^2 \rangle}$	Principal axis components in Å x	y	z		B
	1	0.168	0.036	0.030	0.162	H_1	2.38
C_1	2	0.289	0.272	−0.047	−0.071	H_2	2.24
	3	0.240	0.093	0.243	−0.035	H_3	4.27
						H_4	0.87
	1	0.160	0.029	0.004	0.155	H_5	2.45
C_2	2	0.272	0.249	−0.058	−0.073	H_6	0.62
	3	0.203	0.093	0.206	−0.004	H_7	2.29
						H_8	0.98
	1	0.171	0.027	0.022	0.167	H_9	2.07
C_3	2	0.260	0.247	−0.041	−0.054		
	3	0.193	0.076	0.197	−0.018		
	1	0.175	0.018	−0.009	0.172		
C_4	2	0.255	0.222	−0.079	−0.059		
	3	0.202	0.112	0.199	0.008		

DISCUSSION OF THE STRUCTURES

1. Pentane. From Table 5 it is seen that the C—C bonds in pentane have values close to the normal C—C sp^3-sp^3 single bond distance, 1.534 Å.[4] The C—C—C angles, however, are somewhat larger than the tetrahedral value: 112.1 ± 0.3°. The C—C repeat distance of 2.539 Å is also normal, and so are the shortest intermolecular distances of 3.90 Å, 3.95 Å, and 3.96 Å, which all

Table 5. Pentane. Interatomic distances and angles ± standard deviations.

$C_1-C_2 = 1.537 \pm 0.006$ Å	$C_1-H_1 = 0.94 \pm 0.07$ Å
$C_2-C_3 = 1.529 \pm 0.006$ Å	$C_1-H_2 = 1.12 \pm 0.07$ Å
$C_1-C_3 = 2.541 \pm 0.006$ Å	$C_1-H_3 = 1.21 \pm 0.07$ Å
$C_2-C_4 = 2.537 \pm 0.006$ Å	$C_2-H_4 = 0.99 \pm 0.07$ Å
	$C_2-H_5 = 1.08 \pm 0.07$ Å
$\angle C_1-C_2-C_3 = 112.0 \pm 0.3°$	$C_3-H_6 = 1.08 \pm 0.07$ Å
$\angle C_2-C_3-C_4 = 112.3 \pm 0.3°$	

are normal van der Waals' distances. The less accurate C—H bond length is found to be 1.07 ± 0.07 Å.

The particular mode of packing in the pentane structure as shown in Fig. 1 is different from any other paraffin structure hitherto published.[5] In

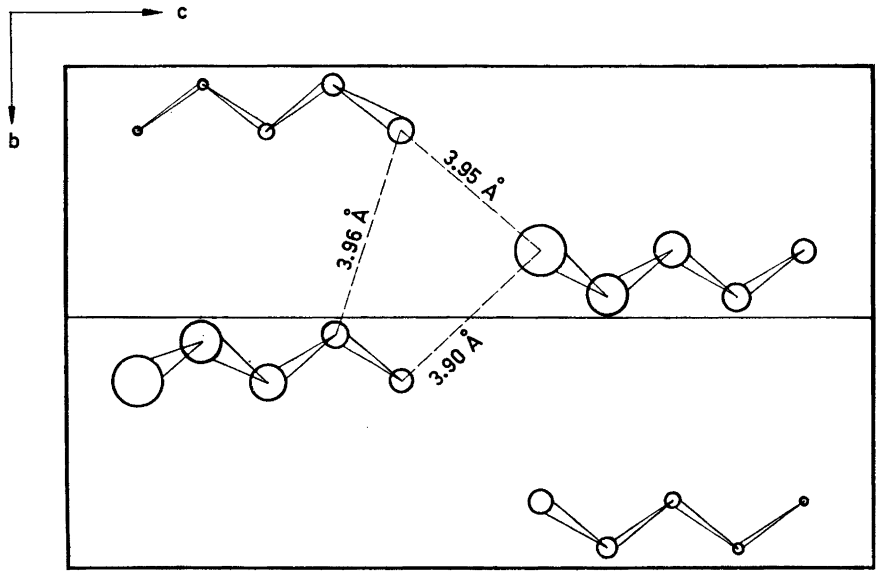

Fig. 1. Packing scheme for pentane. The shortest intermolecular distances are indicated.

all other orthorhombic crystals of paraffins, the molecules are mutually parallel with their chain axes normal to the basal plane. The chain axes of the pentane molecules are neither parallel to the *c*-axis, nor to each other. Hence, the pentane molecules do not show the characteristics of typical long chain molecules.

The anisotropic thermal vibrations of the carbon atoms very well fit into the packing scheme. All the longest axes of the vibration ellipsoides lie approximately in the *ac*-plane normal to the plane of the molecule, and the shortest axes are closely parallel to the direction of the molecular chain.

Table 6. Octane. Interatomic distances and angles ± standard deviations.

$C_1-C_2 = 1.534 \pm 0.007$ Å	$C_1-H_1 = 0.93 \pm 0.04$ Å
$C_2-C_3 = 1.522 \pm 0.007$ Å	$C_1-H_2 = 1.07 \pm 0.04$ Å
$C_3-C_4 = 1.529 \pm 0.007$ Å	$C_1-H_3 = 1.15 \pm 0.04$ Å
$C_4-C_5 = 1.516 \pm 0.007$ Å	$C_2-H_4 = 1.03 \pm 0.04$ Å
$C_1-C_3 = 2.569 \pm 0.007$ Å	$C_2-H_5 = 1.08 \pm 0.04$ Å
$C_2-C_4 = 2.549 \pm 0.007$ Å	$C_3-H_6 = 1.02 \pm 0.04$ Å
$C_3-C_5 = 2.528 \pm 0.007$ Å	$C_3-H_7 = 1.03 \pm 0.04$ Å
	$C_4-H_8 = 0.97 \pm 0.04$ Å
$\angle C_1-C_2-C_3 = 114.4 \pm 0.6°$	$C_4-H_9 = 1.12 \pm 0.04$ Å
$\angle C_2-C_3-C_4 = 113.3 \pm 0.6°$	
$\angle C_3-C_4-C_5 = 112.2 \pm 0.6°$	

2. *Octane.* Within the limits of accuracy, the average C—C bond length of 1.525 ± 0.007 Å in octane is the same as in pentane. Table 6 seems to indicate a trend towards alternating long and short C—C bonds. The difference, however, is only 0.012 Å, and according to the standard deviations given, this difference is probably not significant. It should also be mentioned that the accuracy in cell dimensions is somewhat lower in octane than in pentane, and that the bond lengths are more sensitive to errors in cell dimensions in a triclinic than in an orthorhombic structure. For pentane the standard deviation in the C—C bond length due to the estimated standard deviation in cell dimensions was found to be 0.001 Å, and thus negligible. For octane, however, the standard deviation due to uncertainty in fractional coordinates and estimated errors in cell axes and angles are of the same magnitude, both 0.005 Å for the C—C bond.

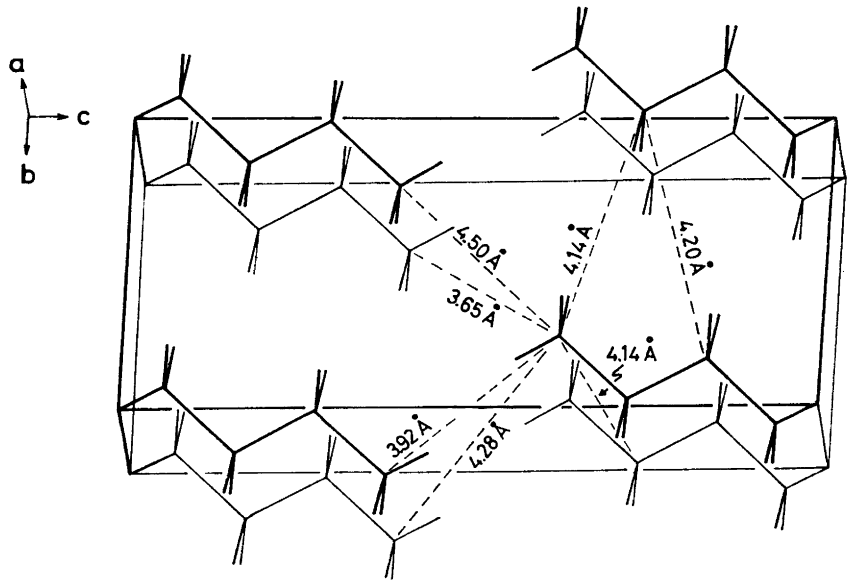

Fig. 2. Packing scheme for octane. The shortest intermolecular distances are indicated.

Table 7. Pentane. Observed and calculated structure factors.

H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}
0	0	2	24.19	-25.47	1	1	14	5.73	-5.59	1	10	1	3.46	3.37	2	8	10	3.46	-3.64
0	0	6	15.13	-14.50	1	2	1	24.11	29.36	1	10	2	2.78	-2.90	2	9	1	3.46	3.84
0	0	4	18.97	-19.85	1	2	2	10.41	10.97	1	10	3	2.40	-2.35	2	9	3	3.67	-4.09
0	0	8	8.81	-8.46	1	2	3	5.56	-5.85	1	11	0	5.56	-5.67	2	9	4	2.61	2.28
0	0	10	4.21	-3.77	1	2	4	14.37	-13.27	1	11	1	2.49	-2.20	2	9	8	1.98	-2.18
0	0	12	25.58	25.21	1	2	5	16.52	-16.81	1	11	2	3.25	3.09	2	9	9	1.90	-2.72
0	0	16	4.55	-4.89	1	2	6	7.00	5.36	2	0	2	14.96	-16.23	2	10	1	1.52	-1.97
0	0	14	9.02	-8.43	1	2	7	8.64	-8.36	2	0	4	10.16	8.41	2	10	2	1.52	2.11
0	0	18	2.53	-2.73	1	2	8	7.59	6.52	2	0	6	7.25	7.73	2	10	3	3.96	3.77
0	2	0	29.83	28.74	1	2	10	3.96	-3.71	2	0	8	5.02	5.82	3	0	2	3.12	3.40
0	2	2	18.46	-19.02	1	2	11	10.58	10.55	2	0	10	11.21	-11.32	3	0	4	11.63	-10.96
0	2	3	20.31	-20.45	1	2	13	5.61	5.15	2	0	14	3.75	-4.69	3	0	8	7.46	7.78
0	2	4	13.61	-13.12	1	2	15	4.21	-4.23	2	1	2	17.87	16.75	3	0	10	2.99	-3.72
0	2	5	21.37	-20.17	1	2	16	3.16	-3.14	2	1	3	3.75	-4.54	3	1	0	3.33	-2.37
0	2	6	30.77	30.15	1	2	17	4.30	-4.50	2	1	4	6.62	-6.17	3	1	2	4.72	-4.66
0	2	7	16.73	15.10	1	3	1	20.15	-20.93	2	1	5	4.09	-2.65	3	1	3	7.97	8.37
0	2	8	11.55	-9.83	1	3	2	2.78	-3.05	2	1	6	1.18	.38	3	1	4	7.29	6.38
0	2	9	6.74	6.46	1	3	3	3.79	2.80	2	1	7	2.32	-2.37	3	1	8	4.00	4.07
0	2	10	6.49	-5.71	1	3	4	14.50	-13.56	2	1	8	2.99	3.35	3	1	9	5.56	-6.02
0	2	11	11.72	11.70	1	3	5	2.28	.98	2	1	9	2.74	-1.58	3	1	10	3.84	-3.86
0	2	12	6.83	6.92	1	3	6	21.03	21.36	2	1	10	9.82	-9.75	3	1	12	1.69	-1.42
0	2	13	14.88	-14.97	1	3	8	7.29	-7.54	2	1	11	4.64	4.16	3	1	15	2.07	3.40
0	2	14	4.51	-3.99	1	3	11	6.74	6.84	2	1	12	2.23	2.07	3	2	1	2.02	-1.57
0	2	15	2.74	-2.40	1	3	13	7.29	-7.44	2	1	13	2.91	2.66	3	2	2	6.03	5.63
0	2	16	4.85	4.51	1	3	16	2.32	-2.44	2	1	14	5.56	6.11	3	2	3	5.48	-4.51
0	4	0	13.82	-13.66	1	3	18	3.96	5.33	2	1	16	2.23	-2.58	3	2	4	6.53	-6.06
0	4	1	4.51	-3.99	1	4	1	3.54	3.46	2	1	17	1.35	-1.57	3	2	5	6.03	5.07
0	4	2	7.92	-7.36	1	4	2	9.78	-8.94	2	2	0	4.55	3.58	3	2	7	4.26	4.22
0	4	3	2.66	2.55	1	4	3	6.15	-6.59	2	2	1	5.48	-4.27	3	2	8	3.88	4.35
0	4	4	5.57	-5.47	1	4	4	16.31	-16.86	2	2	2	4.51	-4.55	3	2	9	3.25	-3.64
0	4	5	15.17	13.93	1	4	5	7.80	6.78	2	2	3	19.89	17.93	3	2	10	4.05	-5.01
0	4	6	32.50	32.16	1	4	6	3.46	2.91	2	2	4	3.58	-3.20	3	2	14	1.48	2.78
0	4	7	15.17	-15.88	1	4	7	4.30	4.08	2	2	6	10.12	9.56	3	3	2	5.73	5.96
0	4	8	8.68	-7.69	1	4	8	12.26	11.97	2	2	8	4.47	-1.70	3	3	3	7.38	5.63
0	4	9	2.23	-1.19	1	4	9	4.39	-5.06	2	2	9	9.90	-10.73	3	3	4	5.23	-5.58
0	4	10	4.55	-3.83	1	4	14	4.13	-4.29	2	2	11	3.54	4.26	3	3	5	2.07	-1.65
0	4	11	3.54	4.01	1	4	15	2.87	-2.36	2	2	12	2.23	2.99	3	3	6	2.61	-1.77
0	4	12	6.53	-6.98	1	4	16	4.30	-4.07	2	2	15	5.82	6.28	3	3	8	2.87	-3.09
0	4	13	3.20	-3.54	1	4	17	1.77	1.92	2	3	1	10.83	9.92	3	3	9	4.43	-4.31
0	4	17	3.25	3.66	1	5	4	4.55	-3.61	2	3	3	10.96	-9.60	3	3	10	4.09	4.83
0	6	0	7.76	7.96	1	5	5	14.58	13.96	2	3	4	12.43	-11.30	3	3	14	1.73	2.44
0	6	2	3.84	-2.59	1	5	6	9.15	9.34	2	3	7	2.99	.70	3	3	15	1.35	2.40
0	6	3	4.17	3.59	1	5	7	15.88	-16.80	2	3	8	6.01	8.68	3	4	1	3.16	3.31
0	6	5	15.97	16.06	1	5	8	6.09	-5.81	2	3	9	3.58	-4.72	3	4	2	6.11	6.59
0	6	6	2.99	-2.52	1	5	9	3.69	1.86	2	3	10	2.23	-2.37	3	4	3	7.00	-6.60
0	6	7	17.95	-18.42	1	6	1	5.49	-5.57	2	3	11	4.59	5.01	3	4	5	3.29	2.46
0	6	9	2.28	-1.98	1	6	2	2.70	2.20	2	3	13	2.99	2.80	3	4	7	2.32	2.31
0	6	12	4.17	3.88	1	6	3	5.84	-6.22	2	3	15	3.25	-3.44	3	4	9	3.75	-4.50
0	6	15	1.69	2.19	1	6	5	11.73	12.71	2	3	16	3.84	-4.36	3	4	10	3.33	-3.83
0	8	0	7.67	6.78	1	6	6	9.90	9.91	2	4	0	4.26	5.64	3	4	11	2.07	2.47
0	8	1	8.26	-8.57	1	6	7	8.85	8.05	2	4	1	3.75	-3.03	3	4	13	1.01	1.86
0	8	5	4.05	3.15	1	6	9	6.20	-6.19	2	4	2	9.95	9.24	3	4	14	1.64	3.19
0	8	6	11.72	-10.75	1	6	10	3.16	-2.56	2	4	4	11.59	-12.15	3	5	1	1.77	2.18
0	8	7	3.20	-3.20	1	6	11	2.91	-3.42	2	4	5	3.84	3.87	3	5	2	4.26	3.13
0	8	8	2.53	2.61	1	6	13	1.90	-1.53	2	4	8	6.74	-7.01	3	5	3	6.57	-6.46
0	8	11	5.18	5.29	1	7	0	5.35	6.57	2	4	9	2.23	2.27	3	5	4	4.34	-2.57
0	8	12	4.17	4.59	1	7	1	3.25	-3.43	2	4	10	6.57	6.91	3	5	8	1.64	-1.76
0	8	13	4.47	-5.40	1	7	2	2.19	-3.24	2	4	12	2.19	2.15	3	5	9	4.13	4.70
0	10	0	6.15	-6.58	1	7	4	3.25	3.02	2	4	14	2.91	2.08	3	5	10	2.44	2.44
0	10	1	7.38	-7.57	1	7	5	8.09	7.50	2	5	3	9.36	-9.08	3	5	11	1.35	-1.71
0	10	2	2.32	2.13	1	7	6	6.07	-6.52	2	5	4	4.26	-5.61	3	6	1	5.06	5.09
0	10	4	1.94	1.62	1	7	7	8.77	-8.24	2	5	5	8.30	8.30	3	6	3	5.65	-6.56
1	0	4	10.87	10.62	1	7	8	3.54	2.79	2	5	7	6.41	6.34	3	6	9	3.08	-3.32
1	0	8	6.36	-6.25	1	7	11	1.94	2.06	2	5	8	4.72	4.01	3	6	11	2.70	3.93
1	0	10	14.37	-14.49	1	7	12	3.46	3.49	2	5	9	6.36	-6.74	3	7	2	3.41	-3.49
1	0	12	5.90	5.23	1	7	13	2.19	-1.73	2	5	15	3.03	-2.83	3	7	3	2.49	-2.59
1	0	14	9.86	9.77	1	7	14	2.07	-2.16	2	6	3	10.24	-10.17	3	7	4	3.29	3.58
1	0	18	1.73	-2.11	1	8	1	3.29	3.74	2	6	5	4.43	4.83	3	7	8	2.40	2.75
1	1	1	15.09	-19.28	1	8	2	3.54	3.67	2	6	7	2.78	-2.76	3	7	9	1.64	1.73
1	1	2	17.95	-20.24	1	8	3	3.84	-4.14	2	6	9	7.25	7.47	3	7	10	2.19	-2.95
1	1	3	5.10	3.96	1	8	4	4.34	4.49	2	7	3	6.36	-6.89	3	8	1	1.52	1.45
1	1	4	6.20	-6.02	1	8	8	5.56	-5.14	2	7	4	3.25	2.93	3	8	2	2.28	-2.34
1	1	5	11.76	-9.87	1	8	9	2.61	-2.73	2	7	5	4.93	4.94	3	8	3	3.25	-3.91
1	1	6	6.66	6.77	1	8	10	1.77	-1.94	2	7	7	3.41	3.49	3	8	5	1.98	2.70
1	1	7	5.44	3.50	1	8	11	2.95	2.88	2	7	8	3.67	-2.84	3	8	7	1.35	2.01
1	1	8	2.53	-2.87	1	8	13	1.26	1.81	2	7	9	5.31	-5.27	3	9	3	2.11	3.36
1	1	10	7.92	-7.65	1	9	1	7.33	-7.52	2	8	1	2.74	-2.22	3	9	4	1.10	1.85
1	1	11	7.54	7.02	1	9	4	2.53	2.42	2	8	2	4.55	-4.15	3	9	5	.84	-1.35
1	1	12	12.98	12.90	1	9	6	4.34	-3.87	2	8	4	5.27	5.57					
1	1	13	5.56	-5.56	1	9	11	4.00	4.24	2	8	8	3.54	3.26					

The average C—C repeat distance of 2.545 ± 0.007 Å and the C—H bond length of 1.04 ± 0.04 Å are close to the corresponding values for pentane. The average C—C—C angle of $113.3 \pm 0.6°$ is somewhat larger than for pentane, the difference is probably again not significant.

The octane molecules pack in the extended trans configuration, *i.e.* the carbon skeletons are planar. They seem to form long chains in the [111]-direction, and in this direction the distance between two adjacent CH_3 groups

Table 8. Octane. Observed and calculated structure factors.

H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}
0	0	2	5.85	-6.29	1	-4	-8	1.04	1.35	1	2	-6	9.10	-9.45	2	-2	13	3.45	4.30
0	0	3	5.89	5.82	1	-4	-4	4.60	4.69	1	2	-5	1.70	-1.42	2	-1	-12	.67	.59
0	0	4	6.64	-5.90	1	-4	-3	3.17	3.50	1	2	-4	1.59	1.75	2	-1	-11	1.34	-1.11
0	0	6	1.75	-1.44	1	-4	-2	1.53	-1.59	1	2	-3	1.94	-2.16	2	-1	-10	4.48	-3.98
0	0	7	1.61	1.54	1	-4	-1	1.15	1.15	1	2	-2	4.10	4.17	2	-1	-6	1.69	1.33
0	0	9	9.23	-9.74	1	-4	0	1.67	-1.66	1	2	-1	2.50	1.10	2	-1	-5	.49	-.60
0	0	10	2.60	-2.49	1	-4	1	3.48	-3.76	1	2	0	.78	.96	2	-1	-4	1.11	1.14
0	0	11	1.59	1.67	1	-4	5	3.40	-3.28	1	2	1	1.11	-1.29	2	-1	-3	1.54	-1.23
0	0	12	1.30	-1.32	1	-4	6	3.45	-3.36	1	2	2	1.36	1.10	2	-1	-2	2.20	1.65
0	0	13	1.40	1.21	1	-4	7	.99	1.21	1	2	3	11.24	13.90	2	-1	1	1.28	-1.10
0	0	14	.66	.69	1	-4	8	.47	-.64	1	2	4	2.36	2.36	2	-1	2	2.15	1.77
0	1	-14	2.49	3.16	1	-4	10	2.53	2.86	1	2	5	1.77	-1.73	2	-1	4	2.41	-2.05
0	1	-12	.82	-.79	1	-3	-8	1.01	-1.53	1	2	6	1.80	1.90	2	-1	5	.87	1.06
0	1	-11	1.58	1.53	1	-3	-7	1.05	1.10	1	2	7	2.45	-2.66	2	-1	7	.74	-.69
0	1	-10	4.77	-4.94	1	-3	-6	1.08	-1.46	1	2	8	1.33	-1.27	2	-1	8	8.68	-8.66
0	1	-9	3.87	-4.19	1	-3	-5	2.12	2.12	1	2	12	3.60	-4.00	2	-1	9	2.27	-2.10
0	1	-7	.82	.79	1	-3	-4	8.14	9.78	1	3	-11	1.63	1.89	2	-1	10	1.98	1.88
0	1	-6	1.53	-1.29	1	-3	-3	.61	.63	1	3	-7	4.20	-4.74	2	-1	11	1.77	-1.81
0	1	-5	15.53	-15.32	1	-3	-2	.88	-.85	1	3	-6	4.86	-5.23	2	-1	12	2.62	2.32
0	1	-4	5.97	-5.71	1	-3	-1	.92	.91	1	3	-2	2.31	-2.46	2	-1	13	1.87	1.99
0	1	-3	7.15	7.25	1	-3	0	1.26	-1.68	1	3	-1	1.24	-1.23	2	0	-12	.63	.60
0	1	-2	9.85	-10.44	1	-3	1	.94	.38	1	3	0	1.25	1.27	2	0	-11	2.98	-2.79
0	1	1	4.15	-4.01	1	-3	2	1.04	-.73	1	3	1	2.17	-2.36	2	0	-10	2.77	-2.55
0	1	2	1.30	1.17	1	-3	3	1.24	1.15	1	3	2	4.76	4.98	2	0	-6	2.42	-1.96
0	1	3	1.21	-1.01	1	-3	4	1.45	-1.36	1	3	3	6.15	6.59	2	0	-5	.99	-.90
0	1	4	14.68	14.56	1	-3	5	8.40	-8.89	1	3	7	1.29	.89	2	0	-4	1.50	1.30
0	1	5	5.57	5.14	1	-3	6	1.98	-2.00	1	3	8	1.33	.86	2	0	-3	3.07	-2.68
0	1	6	3.66	-3.42	1	-3	7	1.79	1.69	1	3	10	1.61	1.19	2	0	-2	7.96	7.56
0	1	7	3.69	3.60	1	-3	8	1.49	-1.39	1	3	11	1.46	-1.72	2	0	0	1.95	-1.86
0	1	8	5.55	-5.44	1	-3	9	1.95	1.60	1	4	-7	2.41	-2.53	2	0	1	.95	.85
0	1	9	7.42	-7.61	1	-3	10	.84	.73	1	4	-3	2.36	-2.28	2	0	2	.67	-.53
0	1	10	.78	.83	1	-2	-9	4.72	-4.85	1	4	-2	4.12	-4.26	2	0	3	2.70	2.77
0	1	13	.95	-.76	1	-2	-8	1.63	-1.03	1	4	2	3.78	4.05	2	0	4	1.47	1.30
0	2	-13	.72	-.91	1	-2	-7	1.04	1.48	1	4	3	1.73	1.55	2	0	5	1.98	-1.84
0	2	-10	1.37	-1.73	1	-2	-6	2.06	-2.39	1	4	6	1.23	.80	2	0	6	2.69	2.87
0	2	-8	1.07	-.71	1	-2	-5	5.97	7.01	1	4	7	2.71	2.64	2	0	7	5.62	-5.62
0	2	-7	2.49	2.23	1	-2	-4	5.93	6.61	1	4	9	.49	.01	2	0	8	8.42	-8.39
0	2	-6	7.03	-6.90	1	-2	-3	1.32	-1.34	1	5	-3	2.98	-3.34	2	0	9	1.12	1.15
0	2	-5	12.04	-12.21	1	-2	1	2.74	2.82	1	5	-2	1.91	-2.10	2	0	13	.55	-.58
0	2	-4	1.98	1.88	1	-2	2	2.91	-2.90	1	5	5	.62	-.44	2	1	-11	1.98	-1.91
0	2	-2	1.36	-1.02	1	-2	3	4.31	4.60	2	-6	1	2.13	-2.27	2	1	-8	.83	.79
0	2	-1	5.15	4.85	1	-2	4	9.72	-10.11	2	-5	-4	2.13	1.83	2	1	-7	3.07	-2.62
0	2	0	1.34	-1.13	1	-2	5	11.91	-12.28	2	-5	-3	.71	.52	2	1	-6	5.23	-4.78
0	2	1	2.45	2.36	1	-2	6	2.07	1.72	2	-5	-2	.67	-.37	2	1	-5	1.26	1.08
0	2	2	3.73	-3.42	1	-2	9	1.62	-1.65	2	-5	-1	.70	.61	2	1	-4	.88	-.56
0	2	3	7.25	6.50	1	-2	10	1.63	-1.43	2	-5	0	3.02	-2.61	2	1	-2	8.67	9.31
0	2	4	14.96	14.75	1	-2	11	1.40	1.25	2	-5	1	3.23	-2.85	2	1	-1	2.71	2.76
0	2	5	1.66	-1.41	1	-2	12	1.59	-1.51	2	-5	5	1.86	-1.47	2	1	0	.71	-.74
0	2	7	1.58	1.09	1	-2	13	2.36	2.18	2	-5	6	1.07	-.72	2	1	1	.62	-.68
0	2	8	4.60	-4.81	1	-1	-10	3.46	-3.56	2	-5	7	.49	.51	2	1	2	2.66	2.77
0	2	9	1.38	-1.45	1	-1	-9	5.32	-5.52	2	-5	8	.78	-.66	2	1	3	7.84	8.18
0	2	12	1.05	-.83	1	-1	-5	3.65	4.28	2	-5	9	1.01	1.55	2	1	4	1.58	-1.36
0	3	-10	2.28	1.95	1	-1	-3	1.62	1.78	2	-4	-9	.62	.61	2	1	7	6.34	-6.38
0	3	-9	.75	-.80	1	-1	1	4.99	-5.89	2	-4	-6	.76	-.72	2	1	8	2.36	-2.39
0	3	-7	.66	.51	1	-1	2	2.07	1.85	2	-4	-5	3.08	2.77	2	1	11	.90	-.81
0	3	-6	7.56	-7.55	1	-1	3	1.44	1.33	2	-4	-4	4.83	4.36	2	1	12	2.92	-3.22
0	3	-5	3.62	-3.65	1	-1	4	15.98	-15.69	2	-4	-3	.83	-.80	2	2	-11	.51	.31
0	3	-4	1.33	1.22	1	-1	5	5.76	-4.31	2	-4	-2	2.79	-2.88	2	2	-7	5.56	-5.55
0	3	-1	3.40	-3.37	1	-1	6	1.44	.81	2	-4	3	.75	.68	2	2	-6	3.16	-3.07
0	3	2	1.44	-1.73	1	-1	7	1.21	1.10	2	-4	4	3.07	-2.47	2	2	-5	1.55	1.42
0	3	3	8.62	8.37	1	-1	8	3.94	-3.18	2	-4	5	5.51	-4.93	2	2	-4	1.28	-.92
0	3	4	5.14	4.90	1	-1	9	9.66	-8.49	2	-4	9	2.79	2.53	2	2	-3	1.91	1.63
0	3	5	1.69	-1.75	1	-1	12	1.87	-1.25	2	-4	10	1.07	.93	2	2	-2	2.13	2.05
0	3	6	.94	.85	1	-1	13	4.02	3.61	2	-3	-9	1.37	-1.48	2	2	1	1.04	-1.21
0	3	8	.94	.33	1	-1	14	1.20	-1.37	2	-3	-5	6.61	6.04	2	2	2	8.17	8.28
0	3	11	.76	.84	1	0	-10	5.51	-5.64	2	-3	-4	2.91	2.62	2	2	3	5.34	5.07
0	4	-10	1.86	1.78	1	0	-9	2.87	-2.35	2	-3	-3	1.13	-1.07	2	2	4	2.17	-2.04
0	4	-9	.94	-.99	1	0	-5	3.46	-3.78	2	-3	0	3.99	3.34	2	2	5	1.37	1.38
0	4	-8	.97	1.05	1	0	-2	3.53	-3.99	2	-3	3	.71	.88	2	2	6	1.38	-1.15
0	4	-7	1.58	-1.59	1	0	2	3.60	4.16	2	-3	4	8.72	-8.15	2	2	7	1.33	-1.48
0	4	-6	3.62	-3.67	1	0	3	2.52	-2.35	2	-3	5	5.10	-4.78	2	2	10	1.13	.84
0	4	-2	3.27	-3.15	1	0	4	4.56	4.59	2	-3	6	2.19	1.89	2	2	11	2.63	-3.12
0	4	-1	3.40	-2.93	1	0	5	1.01	-.88	2	-3	7	.82	-.97	2	3	-9	.63	.63
0	4	0	1.40	1.08	1	0	7	2.77	2.64	2	-3	12	.43	-.71	2	3	-8	1.61	-1.60
0	4	1	1.12	-1.20	1	0	8	12.20	-11.05	2	-2	-10	2.90	-2.56	2	3	-7	3.57	-3.64
0	4	2	1.54	1.26	1	0	9	7.86	-7.09	2	-2	-9	2.19	-2.00	2	3	-6	.63	-.64
0	4	3	5.12	4.82	1	0	10	2.87	2.53	2	-2	-8	1.62	1.26	2	3	-5	2.06	-2.01
0	4	7	1.71	1.39	1	0	11	1.40	-1.08	2	-2	-7	1.36	-1.04	2	3	-2	2.12	-2.13
0	4	8	2.54	2.08	1	0	13	.86	.71	2	-2	-6	2.40	1.73	2	3	-1	1.32	1.31
0	4	9	.79	-.63	1	1	-11	1.98	-1.97	2	-2	-5	4.23	3.68	2	3	0	1.37	-1.35
0	5	-7	.69	-.75	1	1	-10	3.32	-3.68	2	-2	-2	1.33	-1.29	2	3	1	2.24	1.82
0	4	-4	.65	.57	1	1	-6	5.94	-5.93	2	-2	-1	8.18	8.37	2	3	2	6.71	6.67
0	5	-3	.99	-.84	1	1	-5	5.89	-6.09	2	-2	0	7.35	6.65	2	3	3	.76	.71
0	5	-2	4.37	-4.05	1	1	-4	3.39	3.82	2	-2	1	3.62	-3.10	2	3	6	.66	.66
0	5	-1	1.08	-.95	1	1	-3	3.77	-3.67	2	-2	2	2.91	2.77	2	3	7	1.88	1.38
0	5	0	.84	.59	1	1	-2	5.18	5.22	2	-2	3	3.11	-3.00	2	3	8	.71	-.70
0	5	1	.82	-.53	1	1	1	1.01	.66	2	-2	4	9.16	-9.01	2	3	9	1.58	.78
0	5	2	1.62	1.43	1	1	2	2.36	-2.36	2	-2	5	.97	-.82	2	4	-7	.67	-.77
0	5	3	.99	1.03	1	1	5	4.20	-4.67	2	-2	6	.78	.84	2	4	-5	.62	.59
1	-6	1	1.11	-1.50	1	1	6	2.99	2.91	2	-2	8	1.95	-2.26	2	4	-4	.97	-.91
1	-6	2	1.65	-2.43	1	1	8	8.56	-8.96	2	-2	9	3.36	-3.27	2	4	-2	.94	-.86
1	-5	-3	1.96	1.87	1	1	12	1.41	-1.27	2	-2	10	1.50	1.45	2	4	-1	.59	.62
1	-5	1	3.69	-3.65	1	1	13	2.32	-2.32	2	-2	11	1.09	-1.03	2	4	0	.67	-.64
1	-5	6	1.32	-1.49	1	2	-7	2.11	-2.13	2	-2	12	.91	.50	2	4	1	2.36	2.13

H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}	H	K	L	F_{obs}	F_{calc}
2	4	2	1.80	1.84	3	-3	9	.51	-.55	3	-1	11	.82	.50	3	1	3	.99	.82
2	4	6	2.63	2.70	3	-3	11	.74	-.92	3	-1	12	1.61	1.42	3	1	4	.99	-.83
2	4	7	.75	.74	3	-2	-10	1.50	-1.58	3	0	-7	1.73	-1.68	3	1	5	1.37	1.41
3	-5	-5	.88	1.17	3	-2	-6	1.86	2.44	3	0	-6	1.65	-1.56	3	1	6	3.02	-3.25
3	-5	-4	.78	.89	3	-2	-3	.63	-.52	3	0	-5	1.07	1.11	3	1	7	2.42	-2.85
3	-5	-1	.84	-.94	3	-2	-2	2.31	2.45	3	0	-4	1.32	-1.32	3	1	11	3.10	-2.53
3	-5	0	1.78	-2.12	3	-2	-1	4.77	5.02	3	0	-3	2.27	2.06	3	2	-8	2.12	-2.06
3	-5	4	1.36	-1.22	3	-2	0	1.15	-.84	3	0	-2	5.73	5.87	3	2	-7	2.37	-2.32
3	-5	5	1.23	-1.27	3	-2	1	.43	.20	3	0	2	.87	.88	3	2	-6	.43	.14
3	-4	-6	.78	.70	3	-2	3	4.40	-3.66	3	0	3	1.74	1.62	3	2	-1	.53	.43
3	-4	-5	2.44	2.62	3	-2	4	1.79	-1.42	3	0	4	1.20	-1.11	3	2	0	1.24	-1.20
3	-4	-1	.99	-.89	3	-2	5	.40	.18	3	0	5	1.11	1.32	3	2	1	3.77	3.70
3	-4	2	.61	.61	3	-2	7	1.51	-1.62	3	0	6	1.19	-1.20	3	2	2	5.27	5.03
3	-4	3	1.01	-.98	3	-2	8	3.48	-4.07	3	0	7	4.80	-5.68	3	2	3	.94	-.75
3	-4	4	4.41	-4.58	3	-2	11	.54	-.76	3	0	8	1.28	-.67	3	2	4	.43	.17
3	-4	5	1.15	-.98	3	-2	12	3.31	3.01	3	0	9	.66	.61	3	2	6	1.04	-1.18
3	-4	6	.80	.81	3	-1	-10	.91	-1.03	3	0	10	.46	-.40	3	2	8	.40	-.27
3	-4	7	.80	-.75	3	-1	-7	.58	.58	3	0	11	.69	-.16	3	2	9	.87	.86
3	-4	8	1.33	1.81	3	-1	-3	.54	-.60	3	0	12	1.12	-.97	3	3	-5	.80	.25
3	-4	9	.82	1.04	3	-1	-2	3.64	5.81	3	1	-9	.40	.45	3	3	-4	1.08	-1.08
3	-3	-6	2.24	2.25	3	-1	-1	2.65	2.94	3	1	-8	.91	-.95	3	3	-3	2.31	-1.94
3	-3	-5	2.03	2.31	3	-1	0	.86	-1.02	3	1	-7	3.40	-3.45	3	3	-2	.40	.31
3	-3	-1	2.53	2.43	3	-1	2	.70	-.85	3	1	-6	.54	-.52	3	3	1	3.98	3.67
3	-3	0	.99	.72	3	-1	3	.69	-.33	3	1	-5	.59	.41	3	3	2	1.74	1.67
3	-3	1	.91	-.84	3	-1	6	.82	.75	3	1	-4	1.00	-.71	3	3	3	.43	-.41
3	-3	2	1.50	1.18	3	-1	7	4.20	-4.77	3	1	-3	3.50	3.17	3	3	5	.43	.49
3	-3	3	4.49	-3.83	3	-1	8	3.00	-3.45	3	1	-2	2.53	2.25	3	3	6	2.20	1.61
3	-3	4	5.16	-4.88	3	-1	9	1.38	1.49	3	1	1	.53	.40	3	4	-2	.69	.35
3	-3	5	.74	.62	3	-1	10	.91	-.92	3	1	2	4.82	5.00					

is only 3.65 Å. The other shortest intermolecular C—C distances are all normal, 3.92 Å, 4.14 Å, 4.20 Å, 4.28 Å, and 4.50 Å, as shown in Fig. 2.

The orientation of the vibration ellipsoides for the carbon atoms is as expected. The longest axis is approximately normal to the plane of the molecule, and the shortest axis in all the ellipsoides is very close to being parallel to the [111]-direction, the direction of the molecular chain.

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