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# Crystal Structure of Octyl Benzoic Acid

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Octyl benzoic acid, $CH_3(CH_2)_7C_6H_4COOH$(OBA), Mr = 234.34, triclinic, $P\bar{1}$, $a = 13.6372$ Å, $b = 22.0910$ Å, $c = 7.6816$ Å, $V = 2156.69$ Å$^3$, $Z = 1$, $D_m = 1.079$ gm cm$^{-3}$, $D_c = 1.083$ gm cm$^{-3}$, $\mu = 0.07$ cm$^{-1}$, $F_{000} = 768$, (Mo$K\alpha$) = 0.71069 Å, final $R1$ and $wR2$ are 0.0456 and 0.1265 respectively.

*Keywords:* Crystal structure; octyl benzoic acid; nematic

## INTRODUCTION

The crystal structure studies of mesogens provide better insight into the understanding of physical properties and phase transitions. In view of this, the crystal structure study of octyl benzoic acid was undertaken. The compound melts into nematic state at 98.9°C and into isotropic phase at 111.6°C. The material exhibits very small positive dielectric anisotropy and it has very small dipole moment [1] as compared with other mesogens.

## EXPERIMENTAL

Clear needle shaped crystals of the title compound (procured from M/s Merck Ltd., England) were obtained from a solution in acetone. A crystal

*Corresponding author. The observed and calculated structure factor tables can be obtained from the authors.

with approximate size of $0.2 \times 0.1 \times 0.2$ mm was mounted on a Rigaku AFC7S diffractometer equipped with a graphite monochromated Mo$K\alpha$ X-ray source ($\lambda = 0.71069$ Å). The unit cell parameters were obtained by using the method of short vectors followed by least squares refinement of 15 reflections. All reflections could be indexed with respect to a triclinic cell. Lorentz and polarization corrections were applied. The structure was solved using SHELXS-86 [2]. The mean $|E^2 - 1|$ value for zonal and all data is as follows: 1.203 (0*kl*), 1.147 (*h*0*l*), 1.046 (*hk*0) and 1.050 for the rest of the data. These values indicate a centrosymmetric space group. Structure solution in $P\bar{1}$ did not give good combined figure of merit (0.087). This value was observed only twice among the 1024 phase sets determined by the program. The resulting *E*-map did not reveal the complete structure nor could be further expanded by difference fourier map after a few cycles of refinement. As the *R* indices also did not show any improvement, the solution was tried in the non-centric space group *P*1. The combined figure of merit (0.0151), which is very much better than the earlier one, was observed 95 times amongst the 204 phase sets. The peak list from SHELXS-86 revealed all the phenyl rings and the side chains were revealed partially. The difference fourier map showed the positions of all missing non-hydrogen atoms. The structure was refined by full matrix least-squares using SHELXL-93 [3] using 6191 unique reflections. The hydrogen atoms were generated at chemically acceptable positions and refined with isotropic thermal parameters assigned to them. 920 parameters were refined using 1578 observed reflections with $I > 2\sigma(I)$ to $R1 = 0.0456$ and $wR2 = 0.1265$. In the final difference map $(\Delta/\sigma)_{max} = 0.034$, $(\Delta\rho)_{min} = -0.179$, $(\Delta\rho)_{max} = 0.196$ e.Å$^{-3}$ and goodness of fit is 0.955. After the completion of the structure, the coordinates were checked for missing symmetry using BUNYIP [4]. No missing symmetry could be detected.

## RESULTS AND DISCUSSIONS

The positional parameters and equivalent temperature factors for non-hydrogen atoms are given in Table I. Anisotropic parameters ($U_{ij}$) are listed in Table II. Table III gives the bond distances and angles. Figure 1 represents the ORTEP [5] diagram of the molecule with thermal ellipsoids at 50% probability. Figures 2–4 show packing of molecules in the unit cell down *a*, *b* and *c* axes respectively. Two molecules are bound into dimers through well defined OH···O hydrogen bonds, as observed in the case of crystalline aggregates of fatty acids [6]. Intermolecular and intramolecular

TABLE I Atomic coordinates and $U_{eq}$ of non-hydrogen atoms

| *Atom* | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| O1A | 0.1480(13) | 0.4715(8) | 0.6077(20) | 0.072(5) |
| O2A | 0.0383(13) | 0.4259(8) | 0.7789(19) | 0.073(5) |
| C1A | 0.1169(20) | 0.4627(13) | 0.7543(33) | 0.067(8) |
| C2A | 0.1891(20) | 0.5085(12) | 0.9136(30) | 0.068(7) |
| C3A | 0.1643(17) | 0.4962(11) | 1.0782(33) | 0.066(7) |
| C4A | 0.2242(16) | 0.5356(11) | 1.2202(29) | 0.066(6) |
| C5A | 0.3130(22) | 0.5879(14) | 1.2012(35) | 0.073(8) |
| C6A | 0.3372(20) | 0.5976(12) | 1.0386(38) | 0.084(9) |
| C7A | 0.2805(22) | 0.5594(12) | 0.8964(33) | 0.082(8) |
| C8A | 0.3814(20) | 0.6313(13) | 1.3601(34) | 0.083(8) |
| C9A | 0.3322(16) | 0.6659(12) | 1.4679(29) | 0.066(7) |
| C10A | 0.4063(22) | 0.7074(15) | 1.6280(33) | 0.093(9) |
| C11A | 0.3603(20) | 0.7412(13) | 1.7380(30) | 0.068(7) |
| C12A | 0.4425(19) | 0.7795(12) | 1.9030(35) | 0.079(8) |
| C13A | 0.3941(24) | 0.8052(14) | 2.0298(36) | 0.097(10) |
| C14A | 0.4731(25) | 0.8440(14) | 2.1956(46) | 0.122(12) |
| C15A | 0.4350(35) | 0.8682(21) | 2.3349(53) | 0.157(15) |
| O1B | 0.0152(14) | 0.3982(9) | 0.3342(23) | 0.083(5) |
| O2B | −0.1018(15) | 0.3589(9) | 0.4941(24) | 0.090(6) |
| C1B | −0.0787(23) | 0.3629(13) | 0.3400(33) | 0.072(8) |
| C2B | −0.1554(22) | 0.3231(13) | 0.1920(37) | 0.068(8) |
| C3B | −0.1276(22) | 0.3260(12) | 0.0283(32) | 0.071(7) |
| C4B | −0.1994(21) | 0.2936(12) | −0.1156(35) | 0.077(8) |
| C5B | −0.3056(19) | 0.2597(11) | −0.0971(40) | 0.069(8) |
| C6B | −0.3236(21) | 0.2605(14) | 0.0723(39) | 0.079(9) |
| C7B | −0.2540(19) | 0.2917(11) | 0.2120(35) | 0.064(7) |
| C8B | −0.3875(21) | 0.2220(15) | −0.2651(41) | 0.120(12) |
| C9B | −0.3646(22) | 0.1773(15) | −0.3697(40) | 0.132(12) |
| C10B | −0.4538(24) | 0.1481(14) | −0.5373(35) | 0.095(10) |
| C11B | −0.4208(19) | 0.1091(13) | −0.6540(39) | 0.096(9) |
| C12B | −0.5124(24) | 0.0773(15) | −0.8238(38) | 0.101(10) |
| C13B | −0.4740(19) | 0.0417(16) | −0.9482(40) | 0.120(12) |
| C14B | −0.5499(23) | 0.0141(18) | −1.1169(43) | 0.127(12) |
| C15B | −0.5150(30) | −0.0280(19) | −1.2347(45) | 0.168(17) |
| O1C | 0.5329(16) | 0.3422(10) | 0.3475(26) | 0.103(6) |
| O2C | 0.4189(16) | 0.2971(10) | 0.5169(26) | 0.095(6) |
| C1C | 0.4401(23) | 0.2966(14) | 0.3641(32) | 0.076(8) |
| C2C | 0.3739(17) | 0.2565(10) | 0.2126(30) | 0.050(6) |
| C3C | 0.2891(20) | 0.2080(13) | 0.2325(31) | 0.070(8) |
| C4C | 0.2260(21) | 0.1669(11) | 0.0879(36) | 0.077(8) |
| C5C | 0.2466(20) | 0.1747(14) | −0.0886(31) | 0.069(8) |
| C6C | 0.3325(21) | 0.2212(13) | −0.0997(33) | 0.082(9) |
| C7C | 0.3961(23) | 0.2618(14) | 0.0442(32) | 0.085(8) |
| C8C | 0.1725(21) | 0.1304(13) | −0.2460(35) | 0.084(8) |
| C9C | 0.2271(23) | 0.0938(14) | −0.3397(34) | 0.094(9) |
| C10C | 0.1539(22) | 0.0550(13) | −0.4990(35) | 0.082(8) |
| C11C | 0.2003(27) | 0.0227(14) | −0.6259(39) | 0.114(11) |
| C12C | 0.1251(26) | −0.0170(15) | −0.7783(38) | 0.122(12) |
| C13C | 0.1635(30) | −0.0465(17) | −0.9197(49) | 0.138(13) |
| C14C | 0.0834(29) | −0.0777(18) | −1.0740(40) | 0.139(15) |
| C15C | 0.1336(36) | −0.1108(21) | −1.2013(52) | 0.159(15) |
| O1D | 0.6604(16) | 0.4027(9) | 0.6136(22) | 0.090(6) |
| O2D | 0.5463(15) | 0.3673(9) | 0.7975(21) | 0.083(5) |
| C1D | 0.6346(20) | 0.4017(10) | 0.7656(37) | 0.066(7) |

TABLE I (Continued)

| *Atom* | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| C2D | 0.7102(19) | 0.4329(11) | 0.9273(36) | 0.062(7) |
| C3D | 0.8132(26) | 0.4684(16) | 0.9095(37) | 0.094(10) |
| C4D | 0.8937(24) | 0.5045(14) | 1.0647(44) | 0.095(9) |
| C5D | 0.8625(26) | 0.5076(15) | 1.2213(37) | 0.088(9) |
| C6D | 0.7687(24) | 0.4738(13) | 1.2416(29) | 0.073(8) |
| C7D | 0.6855(22) | 0.4392(12) | 1.0946(34) | 0.076(7) |
| C8D | 0.9486(23) | 0.5374(14) | 1.3736(37) | 0.097(10) |
| C9D | 0.9257(21) | 0.5829(13) | 1.4958(27) | 0.079(8) |
| C10D | 1.0109(23) | 0.6162(16) | 1.6440(39) | 0.106(11) |
| C11D | 0.9852(24) | 0.6560(16) | 1.7783(33) | 0.111(11) |
| C12D | 1.0613(22) | 0.6856(16) | 1.9325(40) | 0.100(10) |
| C13D | 1.0296(27) | 0.7202(15) | 2.0745(39) | 0.121(13) |
| C14D | 1.1087(34) | 0.7528(21) | 2.2191(48) | 0.178(18) |
| C15D | 1.0786(33) | 0.7808(20) | 2.3662(42) | 0.157(15) |
| O1E | 0.3995(14) | 0.4428(8) | 0.1058(21) | 0.069(5) |
| O2E | 0.2953(16) | 0.3979(9) | 0.2866(21) | 0.078(5) |
| C1E | 0.3748(22) | 0.4327(14) | 0.2560(40) | 0.074(8) |
| C2E | 0.4463(18) | 0.4746(12) | 0.4095(34) | 0.065(7) |
| C3E | 0.4307(20) | 0.4744(14) | 0.5830(35) | 0.077(8) |
| C4E | 0.4997(23) | 0.5123(13) | 0.7192(31) | 0.080(8) |
| C5E | 0.5946(22) | 0.5536(14) | 0.7046(32) | 0.078(8) |
| C6E | 0.6079(17) | 0.5549(11) | 0.5310(33) | 0.062(7) |
| C7E | 0.5417(18) | 0.5175(11) | 0.3896(26) | 0.068(8) |
| C8E | 0.6698(20) | 0.5932(13) | 0.8515(35) | 0.081(8) |
| C9E | 0.6337(21) | 0.6332(12) | 0.9675(32) | 0.071(8) |
| C10E | 0.7155(18) | 0.6692(13) | 1.1241(31) | 0.069(7) |
| C11E | 0.6802(18) | 0.7053(13) | 1.2600(33) | 0.075(8) |
| C12E | 0.7593(20) | 0.7375(12) | 1.4231(36) | 0.087(9) |
| C13E | 0.7207(25) | 0.7695(16) | 1.5626(38) | 0.112(11) |
| C14E | 0.8027(24) | 0.8034(17) | 1.7164(40) | 0.120(12) |
| C15E | 0.7567(30) | 0.8273(16) | 1.8537(40) | 0.126(11) |
| O1F | 0.2719(16) | 0.3708(9) | −0.1583(24) | 0.093(6) |
| O2F | 0.1628(16) | 0.3240(10) | 0.0182(27) | 0.105(7) |
| C1F | 0.1820(19) | 0.3258(12) | −0.1391(32) | 0.060(7) |
| C2F | 0.1080(21) | 0.2849(12) | −0.2890(30) | 0.060(7) |
| C3F | 0.1358(20) | 0.2903(12) | −0.4552(28) | 0.070(8) |
| C4F | 0.0651(20) | 0.2510(12) | −0.6010(33) | 0.074(8) |
| C5F | −0.0319(16) | 0.2090(10) | −0.5703(31) | 0.053(6) |
| C6F | −0.0631(20) | 0.2044(13) | −0.4049(38) | 0.092(8) |
| C7F | 0.0149(20) | 0.2437(12) | −0.2645(33) | 0.086(9) |
| C8F | −0.1154(20) | 0.1669(13) | −0.7383(31) | 0.079(8) |
| C9F | −0.0738(19) | 0.1305(13) | −0.8550(33) | 0.081(8) |
| C10F | −0.1593(22) | 0.0929(12) | −1.0123(35) | 0.085(8) |
| C11F | −0.1155(23) | 0.0594(14) | −1.1300(34) | 0.090(9) |
| C12F | −0.2033(21) | 0.0232(13) | −1.2903(29) | 0.084(9) |
| C13F | −0.1607(22) | −0.0052(14) | −1.4207(38) | 0.085(8) |
| C14F | −0.2416(28) | −0.0376(15) | −1.5880(42) | 0.119(12) |
| C15F | −0.2040(33) | −0.0718(18) | −1.7230(47) | 0.168(17) |

hydrogen bonds are given in Table IV. These dimers are stacked in layers (see Fig. 2). The two benzene rings of the dimer are coplanar. Alkyl chains are in extended conformation. The dimer formation clearly explains the small dipole moment of this mesogen.

TABLE II Anisotropic displacement parameters ($Å^2$) for non-hydrogen atoms

| *Atom* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| O1A | 0.059(10) | 0.097(12) | 0.048(9) | 0.012(8) | 0.019(8) | 0.004(9) |
| O2A | 0.070(11) | 0.080(11) | 0.053(10) | 0.007(8) | 0.008(9) | 0.004(9) |
| C1A | 0.050(16) | 0.096(20) | 0.077(19) | 0.053(16) | 0.042(14) | 0.036(15) |
| C2A | 0.074(17) | 0.090(18) | 0.038(14) | 0.034(13) | 0.025(13) | 0.014(15) |
| C3A | 0.041(13) | 0.068(15) | 0.069(17) | −0.006(13) | 0.002(13) | −0.007(12) |
| C4A | 0.049(13) | 0.084(16) | 0.065(15) | 0.041(13) | 0.022(12) | 0.015(13) |
| C5A | 0.083(19) | 0.088(21) | 0.063(19) | 0.006(16) | 0.024(16) | 0.041(17) |
| C6A | 0.062(18) | 0.079(20) | 0.097(22) | −0.024(17) | −0.007(16) | 0.018(15) |
| C7A | 0.104(21) | 0.075(17) | 0.078(19) | 0.035(14) | 0.049(17) | 0.023(16) |
| C8A | 0.070(18) | 0.098(21) | 0.090(20) | 0.001(16) | 0.030(16) | 0.032(16) |
| C9A | 0.039(12) | 0.091(17) | 0.068(15) | −0.028(13) | −0.011(11) | 0.032(12) |
| C10A | 0.089(20) | 0.117(24) | 0.071(18) | −0.032(16) | −0.014(16) | 0.049(19) |
| C11A | 0.069(17) | 0.088(21) | 0.053(14) | −0.003(13) | 0.016(13) | 0.034(16) |
| C12A | 0.068(16) | 0.066(17) | 0.111(21) | −0.022(15) | 0.003(15) | 0.042(14) |
| C13A | 0.093(21) | 0.087(21) | 0.091(19) | −0.052(16) | 0.007(16) | 0.009(17) |
| C14A | 0.099(24) | 0.100(20) | 0.155(32) | −0.042(19) | −0.006(22) | 0.034(18) |
| C15A | 0.176(35) | 0.146(30) | 0.154(33) | −0.053(25) | 0.051(27) | 0.047(27) |
| O1B | 0.059(12) | 0.087(14) | 0.095(13) | 0.005(10) | 0.011(10) | 0.016(11) |
| O2B | 0.079(13) | 0.094(13) | 0.079(13) | −0.016(10) | 0.010(10) | 0.004(11) |
| C1B | 0.083(20) | 0.088(20) | 0.046(16) | 0.022(14) | 0.024(15) | 0.018(17) |
| C2B | 0.062(17) | 0.063(18) | 0.085(20) | −0.005(14) | 0.013(15) | 0.029(15) |
| C3B | 0.072(19) | 0.075(19) | 0.057(16) | 0.000(13) | 0.029(15) | 0.003(15) |
| C4B | 0.081(19) | 0.071(19) | 0.073(18) | 0.006(14) | 0.019(15) | 0.012(16) |
| C5B | 0.044(16) | 0.037(14) | 0.110(23) | 0.010(14) | −0.002(15) | 0.001(12) |
| C6B | 0.055(17) | 0.107(24) | 0.093(20) | 0.042(18) | 0.035(16) | 0.038(17) |
| C7B | 0.049(16) | 0.057(15) | 0.076(18) | 0.013(13) | 0.004(14) | 0.007(12) |
| C8B | 0.072(18) | 0.127(25) | 0.154(27) | −0.042(21) | −0.043(17) | 0.062(18) |
| C9B | 0.095(21) | 0.134(26) | 0.156(25) | −0.073(20) | −0.047(18) | 0.066(19) |
| C10B | 0.127(25) | 0.101(21) | 0.076(19) | −0.032(16) | −0.002(18) | 0.080(20) |
| C11B | 0.056(16) | 0.058(18) | 0.141(23) | 0.002(16) | −0.008(16) | −0.011(13) |
| C12B | 0.111(22) | 0.101(22) | 0.098(22) | 0.001(17) | 0.041(19) | 0.030(18) |
| C13B | 0.059(15) | 0.165(30) | 0.136(25) | −0.047(21) | 0.003(16) | 0.046(18) |
| C14B | 0.087(21) | 0.147(32) | 0.121(24) | −0.040(21) | −0.021(19) | 0.025(22) |
| C15B | 0.178(34) | 0.189(33) | 0.141(30) | −0.070(25) | 0.059(26) | 0.051(26) |
| O1C | 0.098(14) | 0.104(15) | 0.115(15) | 0.033(11) | 0.055(12) | 0.022(12) |
| O2C | 0.107(15) | 0.102(15) | 0.083(14) | 0.021(11) | 0.030(12) | 0.034(12) |
| C1C | 0.101(22) | 0.086(20) | 0.040(16) | 0.000(14) | 0.000(16) | 0.036(18) |
| C2C | 0.039(13) | 0.048(14) | 0.062(15) | −0.011(11) | −0.007(12) | 0.021(12) |
| C3C | 0.062(17) | 0.095(22) | 0.046(15) | −0.010(15) | 0.012(13) | 0.015(17) |
| C4C | 0.080(19) | 0.053(16) | 0.096(21) | 0.024(14) | 0.044(17) | 0.003(14) |
| C5C | 0.053(17) | 0.099(22) | 0.056(16) | 0.000(15) | 0.006(13) | 0.029(17) |
| C6C | 0.075(19) | 0.102(22) | 0.068(18) | −0.022(15) | 0.038(16) | 0.010(17) |
| C7C | 0.118(22) | 0.109(22) | 0.054(15) | 0.031(15) | 0.044(15) | 0.057(19) |
| C8C | 0.085(19) | 0.081(19) | 0.085(20) | −0.002(15) | 0.003(16) | 0.035(16) |
| C9C | 0.114(22) | 0.086(21) | 0.075(19) | 0.025(16) | 0.023(17) | 0.021(18) |
| C10C | 0.090(20) | 0.075(18) | 0.082(20) | 0.023(15) | 0.040(17) | 0.014(15) |
| C11C | 0.142(28) | 0.100(23) | 0.096(23) | −0.002(18) | 0.035(22) | 0.028(21) |
| C12C | 0.141(27) | 0.106(24) | 0.084(20) | −0.003(17) | 0.012(20) | −0.004(21) |
| C13C | 0.142(32) | 0.148(31) | 0.135(33) | 0.029(25) | 0.044(27) | 0.050(26) |
| C14C | 0.157(33) | 0.136(33) | 0.095(23) | −0.056(22) | 0.031(23) | 0.005(25) |
| C15C | 0.155(32) | 0.184(37) | 0.120(26) | −0.011(23) | 0.000(23) | 0.042(28) |
| O1D | 0.125(16) | 0.113(16) | 0.048(10) | 0.027(10) | 0.039(11) | 0.048(13) |
| O2D | 0.096(14) | 0.090(13) | 0.049(11) | 0.005(9) | 0.017(10) | 0.009(12) |
| C1D | 0.065(15) | 0.032(11) | 0.112(23) | 0.018(13) | 0.014(16) | 0.034(11) |

TABLE II (Continued)

| *Atom* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C2D | 0.041(15) | 0.056(14) | 0.073(19) | 0.009(12) | −0.004(14) | 0.002(12) |
| C3D | 0.128(28) | 0.125(27) | 0.066(18) | 0.014(17) | 0.031(18) | 0.086(25) |
| C4D | 0.075(20) | 0.095(22) | 0.114(25) | −0.023(18) | 0.010(19) | 0.031(17) |
| C5D | 0.104(22) | 0.108(22) | 0.071(19) | −0.011(16) | 0.022(17) | 0.059(19) |
| C6D | 0.095(23) | 0.085(19) | 0.029(13) | −0.021(12) | 0.009(14) | 0.015(18) |
| C7D | 0.081(18) | 0.096(18) | 0.063(18) | −0.006(14) | 0.013(15) | 0.046(15) |
| C8D | 0.106(23) | 0.095(22) | 0.082(20) | −0.027(17) | 0.015(18) | 0.025(18) |
| C9D | 0.094(21) | 0.103(21) | 0.039(13) | 0.028(13) | 0.018(14) | 0.024(17) |
| C10D | 0.079(20) | 0.124(27) | 0.091(19) | −0.023(18) | −0.016(17) | 0.017(19) |
| C11D | 0.133(27) | 0.175(27) | 0.047(16) | −0.009(17) | 0.005(17) | 0.087(22) |
| C12D | 0.067(17) | 0.108(25) | 0.109(23) | −0.017(19) | −0.015(17) | 0.023(17) |
| C13D | 0.186(36) | 0.108(25) | 0.068(21) | −0.003(17) | 0.029(22) | 0.042(24) |
| C14D | 0.226(45) | 0.222(43) | 0.119(27) | −0.054(27) | 0.040(29) | 0.116(37) |
| C15D | 0.197(39) | 0.192(35) | 0.109(26) | −0.005(23) | 0.003(25) | 0.120(32) |
| O1E | 0.080(12) | 0.080(12) | 0.042(10) | 0.002(8) | 0.010(9) | 0.019(10) |
| O2E | 0.073(12) | 0.093(13) | 0.050(10) | −0.009(9) | −0.005(9) | 0.007(11) |
| C1E | 0.058(17) | 0.079(19) | 0.088(24) | −0.006(17) | 0.000(18) | 0.036(15) |
| C2E | 0.039(14) | 0.077(17) | 0.076(17) | 0.025(13) | 0.011(13) | 0.011(13) |
| C3E | 0.063(17) | 0.107(22) | 0.067(18) | 0.021(16) | 0.025(15) | 0.025(17) |
| C4E | 0.104(22) | 0.110(21) | 0.046(15) | −0.001(14) | 0.044(16) | 0.046(18) |
| C5E | 0.080(20) | 0.105(21) | 0.057(16) | 0.034(15) | 0.027(15) | 0.034(17) |
| C6E | 0.046(14) | 0.058(16) | 0.077(18) | 0.028(14) | 0.028(13) | 0.001(12) |
| C7E | 0.070(16) | 0.100(17) | 0.045(13) | −0.043(12) | −0.002(12) | 0.053(14) |
| C8E | 0.058(17) | 0.065(18) | 0.108(21) | −0.013(15) | 0.017(16) | 0.003(14) |
| C9E | 0.096(21) | 0.047(16) | 0.073(17) | −0.021(13) | 0.010(15) | 0.029(15) |
| C10E | 0.043(14) | 0.108(20) | 0.061(16) | 0.003(14) | 0.003(12) | 0.035(14) |
| C11E | 0.056(15) | 0.071(20) | 0.089(19) | 0.001(15) | −0.009(14) | 0.020(14) |
| C12E | 0.069(17) | 0.062(15) | 0.125(23) | 0.014(14) | −0.008(16) | 0.030(13) |
| C13E | 0.126(26) | 0.121(24) | 0.070(20) | −0.036(17) | 0.006(18) | 0.023(20) |
| C14E | 0.091(24) | 0.156(32) | 0.098(21) | −0.002(21) | 0.003(19) | 0.026(23) |
| C15E | 0.176(31) | 0.133(25) | 0.088(20) | −0.004(17) | 0.037(21) | 0.071(23) |
| O1F | 0.106(14) | 0.080(13) | 0.091(13) | 0.025(10) | 0.034(11) | 0.016(11) |
| O2F | 0.095(15) | 0.137(17) | 0.096(15) | 0.023(12) | 0.049(12) | 0.036(13) |
| C1F | 0.067(18) | 0.061(17) | 0.062(17) | 0.023(13) | 0.033(15) | 0.020(15) |
| C2F | 0.084(19) | 0.061(17) | 0.035(14) | −0.006(12) | 0.008(14) | 0.026(15) |
| C3F | 0.068(17) | 0.075(17) | 0.045(15) | 0.001(13) | 0.004(13) | −0.006(14) |
| C4F | 0.070(18) | 0.063(17) | 0.073(17) | 0.013(13) | −0.002(14) | 0.010(15) |
| C5F | 0.032(12) | 0.047(14) | 0.076(17) | −0.013(12) | −0.008(12) | 0.017(11) |
| C6F | 0.063(14) | 0.108(19) | 0.120(21) | −0.013(16) | 0.032(14) | 0.040(14) |
| C7F | 0.071(18) | 0.085(19) | 0.081(18) | 0.048(15) | 0.010(15) | −0.005(15) |
| C8F | 0.086(18) | 0.111(21) | 0.046(16) | −0.002(14) | 0.000(13) | 0.046(16) |
| C9F | 0.061(17) | 0.104(21) | 0.072(17) | 0.021(15) | 0.013(14) | 0.018(15) |
| C10F | 0.106(20) | 0.054(18) | 0.080(19) | −0.014(14) | −0.007(15) | 0.020(16) |
| C11F | 0.108(23) | 0.105(20) | 0.068(18) | −0.009(15) | 0.029(17) | 0.045(18) |
| C12F | 0.095(20) | 0.088(21) | 0.065(15) | −0.007(14) | 0.039(14) | 0.008(17) |
| C13F | 0.083(19) | 0.076(20) | 0.106(22) | 0.004(16) | 0.023(17) | 0.035(16) |
| C14F | 0.165(32) | 0.096(22) | 0.096(25) | −0.024(18) | 0.036(24) | 0.036(22) |
| C15F | 0.205(40) | 0.162(33) | 0.139(28) | −0.035(23) | 0.108(29) | 0.016(28) |

TABLE III Bond lengths (Å)

| *Atoms* | *Length (Å)* | *Atoms* | *Length (Å)* | *Atoms* | *Length (Å)* |
|---|---|---|---|---|---|
| O1A — C1A | 1.27(2) | O1C — C1C | 1.39(3) | O1E — C1E | 1.27(3) |
| O2A — C1A | 1.18(3) | O2C — C1C | 1.26(3) | O2E — C1E | 1.19(3) |
| C1A — C2A | 1.55(4) | C1C — C2C | 1.43(3) | C1E — C2E | 1.48(4) |
| C2A — C3A | 1.38(3) | C2C — C3C | 1.35(3) | C2E — C3E | 1.39(3) |
| C2A — C7A | 1.43(3) | C2C — C7C | 1.38(3) | C2E — C7E | 1.39(3) |
| C3A — C4A | 1.35(3) | C3C — C4C | 1.39(3) | C3E — C4E | 1.34(3) |
| C4A — C5A | 1.43(3) | C4C — C5C | 1.44(3) | C4E — C5E | 1.36(3) |
| C5A — C6A | 1.36(3) | C5C — C6C | 1.32(3) | C5E — C8E | 1.44(3) |
| C5A — C8A | 1.51(4) | C5C — C8C | 1.53(3) | C5E — C6E | 1.38(3) |
| C6A — C7A | 1.33(3) | C6C — C7C | 1.38(3) | C6E — C7E | 1.35(3) |
| C8A — C9A | 1.50(3) | C8C — C9C | 1.51(4) | C8E — C9E | 1.50(3) |
| C9A — C10A | 1.52(3) | C9C — C10C | 1.49(3) | C9E — C10E | 1.50(3) |
| C10A — C11A | 1.47(3) | C10C — C11C | 1.54(3) | C10E — C11E | 1.54(3) |
| C11A — C12A | 1.56(3) | C11C — C12C | 1.47(4) | C11E — C12E | 1.49(3) |
| C12A — C13A | 1.47(3) | C12C — C13C | 1.52(4) | C12E — C13E | 1.54(3) |
| C13A — C14A | 1.55(4) | C13C — C14C | 1.45(5) | C13E — C14E | 1.47(4) |
| C14A — C15A | 1.44(4) | C14C — C15C | 1.59(5) | C14E — C15E | 1.49(4) |
| O1B — C1B | 1.29(3) | O1D — C1D | 1.28(3) | O1F — C1F | 1.36(3) |
| O2B — C1B | 1.28(3) | O2D — C1D | 1.28(3) | O2F — C1F | 1.28(3) |
| C1B — C2B | 1.46(4) | C1D — C2D | 1.46(3) | C1F — C2F | 1.46(3) |
| C2B — C7B | 1.35(3) | C2D — C7D | 1.41(3) | C2F — C7F | 1.37(3) |
| C2B — C3B | 1.38(3) | C2D — C3D | 1.42(4) | C2F — C3F | 1.40(3) |
| C3B — C4B | 1.35(4) | C3D — C4D | 1.49(4) | C3F — C4F | 1.41(3) |
| C4B — C5B | 1.45(3) | C4D — C5D | 1.36(4) | C4F — C5F | 1.43(3) |
| C5B — C6B | 1.37(3) | C5D — C6D | 1.31(4) | C5F — C6F | 1.41(3) |
| C5B — C8B | 1.57(4) | C5D — C8D | 1.47(4) | C5F — C8F | 1.61(3) |
| C6B — C7B | 1.31(3) | C6D — C7D | 1.45(4) | C6F — C7F | 1.42(3) |
| C8B — C9B | 1.41(4) | C8D — C9D | 1.51(3) | C8F — C9F | 1.50(3) |
| C9B — C10B | 1.57(3) | C9D — C10D | 1.46(3) | C9F — C10F | 1.53(3) |
| C10B — C11B | 1.46(4) | C10D — C11D | 1.51(4) | C10F — C11F | 1.48(3) |
| C11B — C12B | 1.60(4) | C11D — C12D | 1.42(4) | C11F — C12F | 1.55(3) |
| C12B — C13B | 1.50(4) | C12D — C13D | 1.53(4) | C12F — C13F | 1.47(3) |
| C13B — C14B | 1.48(4) | C13D — C14D | 1.40(5) | C13F — C14F | 1.53(4) |
| C14B — C15B | 1.53(4) | C14D — C15D | 1.47(4) | C14F — C15F | 1.53(4) |

TABLE III (Continued) Bond Angles

| *Atoms* | *Angle* (°) | *Atoms* | *Angle* (°) | *Atoms* | *Angle* (°) |
|---|---|---|---|---|---|
| O2A — C1A — O1A | 127.4(27) | O2C — C1C — C2C | 125.2(28) | O2E — C1E — O1E | 127.6(25) |
| O2A — C1A — C2A | 117.6(21) | O2C — C1C — O1C | 114.5(23) | O2E — C1E — C2E | 115.2(27) |
| O1A — C1A — C2A | 114.8(22) | C2C — C1C — O1C | 120.2(24) | O1E — C1E — C2E | 116.6(26) |
| C3A — C2A — C7A | 120.5(23) | C3C — C2C — C7C | 116.9(22) | C3E — C2E — C7E | 113.6(23) |
| C3A — C2A — C1A | 116.5(21) | C3C — C2C — C1C | 120.0(24) | C3E — C2E — C1E | 126.0(23) |
| C7A — C2A — C1A | 122.9(21) | C7C — C2C — C1C | 123.0(25) | C7E — C2E — C1E | 120.3(24) |
| C4A — C3A — C2A | 118.5(22) | C2C — C3C — C4C | 120.9(22) | C2E — C3E — C4E | 123.7(25) |
| C3A — C4A — C5A | 120.7(21) | C5C — C4C — C3C | 121.9(22) | C3E — C4E — C5E | 124.0(23) |
| C4A — C5A — C6A | 119.3(25) | C6C — C5C — C4C | 114.8(23) | C4E — C5E — C8E | 124.0(24) |
| C4A — C5A — C8A | 120.9(24) | C6C — C5C — C8C | 124.8(25) | C4E — C5E — C6E | 111.8(24) |
| C6A — C5A — C8A | 119.7(25) | C4C — C5C — C8C | 120.4(25) | C8E — C5E — C6E | 124.1(25) |
| C7A — C6A — C5A | 121.5(26) | C5C — C6C — C7C | 123.4(25) | C7E — C6E — C5E | 126.5(22) |
| C6A — C7A — C2A | 119.3(24) | C9C — C8C — C5C | 112.9(21) | C6E — C7E — C2E | 120.2(21) |
| C5A — C8A — C9A | 118.9(21) | C10C — C9C — C8C | 110.9(24) | C5E — C8E — C9E | 119.0(23) |
| C8A — C9A — C10A | 115.4(19) | C9C — C10C — C11C | 117.9(25) | C8E — C9E — C10E | 115.2(21) |
| C11A — C10A — C9A | 116.8(21) | C10C — C11C — C12C | 116.3(29) | C11E — C10E — C9E | 117.6(18) |
| C10A — C11A — C12A | 112.3(20) | C11C — C12C — C13C | 120.6(30) | C10E — C11E — C12E | 118.1(19) |
| C13A — C12A — C11A | 112.8(19) | C14C — C13C — C12C | 115.3(31) | C11E — C12E — C13E | 117.2(21) |
| C13A — C14A — C15A | 119.9(31) | C15C — C14C — C13C | 109.1(33) | C14E — C13E — C12E | 115.2(26) |
| O2B — C1B — O1B | 115.9(24) | O2D — C1D — O1D | 124.6(24) | C13E — C14E — C15E | 111.6(27) |
| O2B — C1B — C2B | 117.9(24) | O2D — C1D — C2D | 112.6(24) | O2F — C1F — C2F | 121.8(23) |
| O1B — C1B — C2B | 125.9(23) | O1D — C1D — C2D | 122.2(23) | O2F — C1F — O1F | 115.5(23) |
| C7B — C2B — C1B | 120.1(26) | C7D — C2D — C3D | 116.4(24) | C2F — C1F — O1F | 122.6(21) |
| C7B — C2B — C3B | 122.1(25) | C7D — C2D — C1D | 125.3(23) | C7F — C2F — C1F | 120.2(22) |
| C1B — C2B — C3B | 117.2(25) | C3D — C2D — C1D | 117.5(25) | C7F — C2F — C3F | 122.2(22) |
| C4B — C3B — C2B | 119.1(25) | C2D — C3D — C4D | 121.4(26) | C1F — C2F — C3F | 117.6(23) |
| C3B — C4B — C5B | 119.6(25) | C3D — C4D — C5D | 117.5(28) | C4F — C3F — C2F | 118.2(23) |
| C6B — C5B — C4B | 115.6(24) | C6D — C5D — C8D | 122.2(26) | C3F — C4F — C5F | 117.9(23) |
| C6B — C5B — C8B | 125.4(25) | C6D — C5D — C4D | 121.5(27) | C6F — C5F — C4F | 124.8(21) |
| C4B — C5B — C8B | 118.8(27) | C8D — C5D — C4D | 114.4(28) | C6F — C5F — C8F | 117.0(21) |
| C5B — C6B — C7B | 124.8(25) | C5D — C6D — C7D | 123.1(23) | C4F — C5F — C8F | 118.0(22) |
| C6B — C7B — C2B | 118.6(26) | C2D — C7D — C6D | 118.9(25) | C5F — C6F — C7F | 113.3(21) |

| | | | | | |
|---|---|---|---|---|---|
| C9B — C8B — C5B | 121.1(22) | C5D — C8D — C9D | 115.0(24) | C2F — C7F — C6F | 123.5(23) |
| C8B — C9B — C10B | 113.0(22) | C10D — C9D — C8D | 117.0(24) | C9F — C8F — C5F | 116.4(19) |
| C11B — C10B — C9B | 111.0(22) | C11D — C10D — C9D | 117.4(24) | C8F — C9F — C10F | 112.2(20) |
| C10B — C11B — C12B | 111.7(22) | C12D — C11D — C10D | 120.6(26) | C9F — C10F — C11F | 111.3(22) |
| C13B — C12B — C11B | 110.7(22) | C11D — C12D — C13D | 119.3(26) | C10F — C11F — C12F | 110.0(21) |
| C14B — C13B — C12B | 115.4(23) | C14D — C13D — C12D | 117.6(31) | C13F — C12F — C11F | 111.7(21) |
| C13B — C14B — C15B | 115.2(27) | C13D — C14D — C15D | 118.6(38) | C12F — C13F — C14F | 114.3(23) |
| | | | | C13F — C14F — C15F | 116.9(30) |

TABLE IV Hydrogen bond lengths (Å) and angles (°)

| *Atoms* | *Length (Å)* | *Angle (°)* |
|---|---|---|
| 02A — H2A···O2B | 2.689 | 82.92 |
| C3A — H3A···O2A | 2.726 | 98.59 |
| C7A — H7A···O1A | 2.862 | 93.96 |
| C3B — H3B···O1B | 2.851 | 99.84 |
| C7B — H7B···O2B | 2.744 | 98.40 |
| C3C — H3C···O2C | 2.844 | 98.40 |
| C7C — H7C···O1C | 2.879 | 100.18 |
| 02D — H2D···O1C | 3.457 | 133.13 |
| 02D — H2D···O2C | 2.631 | 164.42 |
| C3D — H3D···O1D | 2.807 | 100.61 |
| C7D — H7D···O2D | 2.785 | 94.72 |
| 02E — H2E···O2C | 3.480 | 148.13 |
| 02E — H2E···O2F | 2.624 | 82.45 |
| C3E — H3E···O2E | 2.803 | 95.61 |
| C7E — H7E···O1E | 2.754 | 98.52 |
| C3F — H3F···O1F | 2.844 | 100.79 |
| C7F — H7F···O2F | 2.826 | 100.80 |

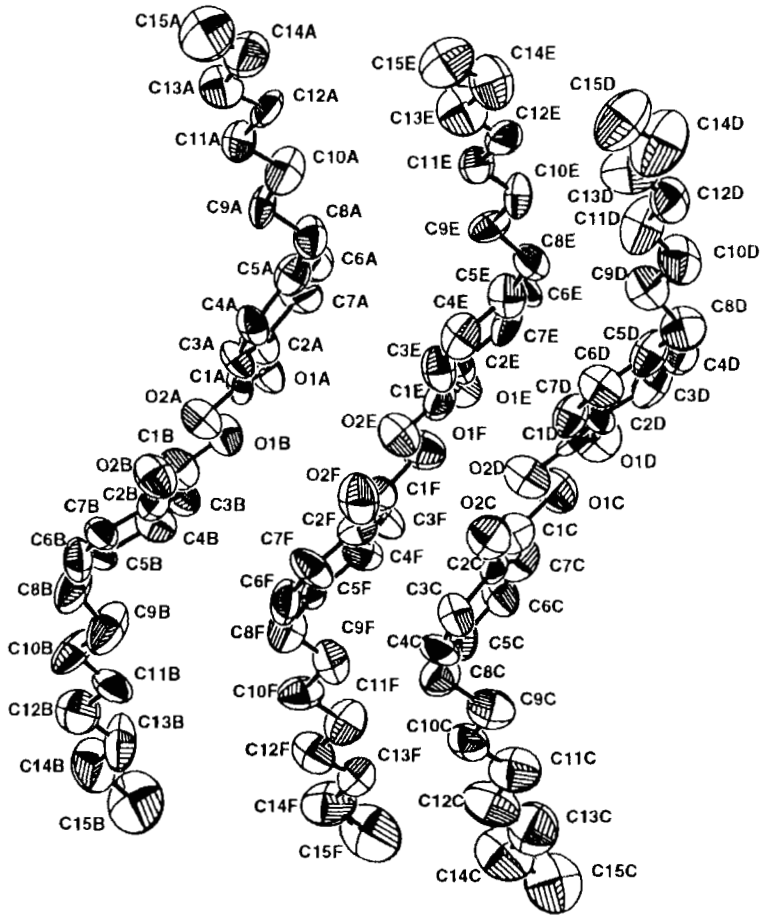


FIGURE 1 ORTEP of the molecule at 50% probability.

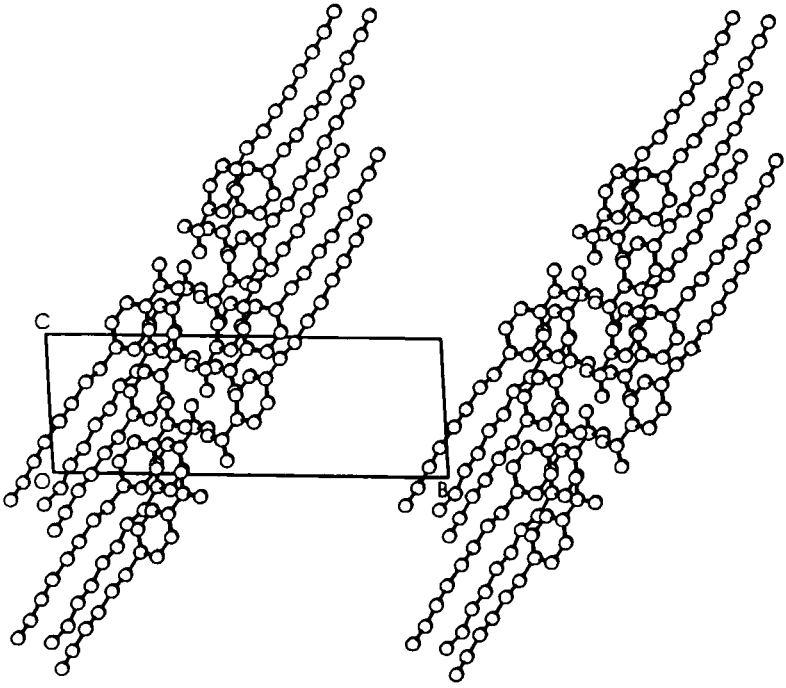


FIGURE 2 Packing of the molecules down *a*.

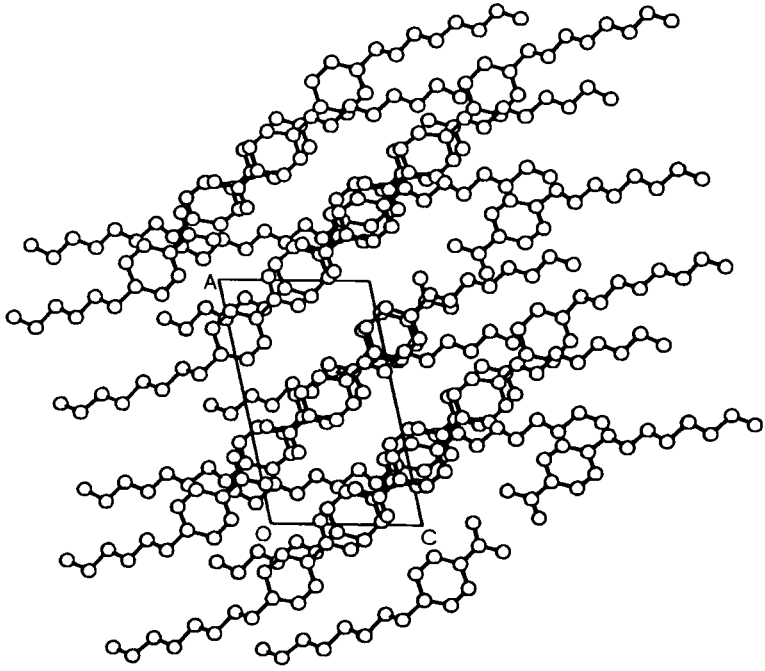


FIGURE 3 Packing of the molecules down *b*.

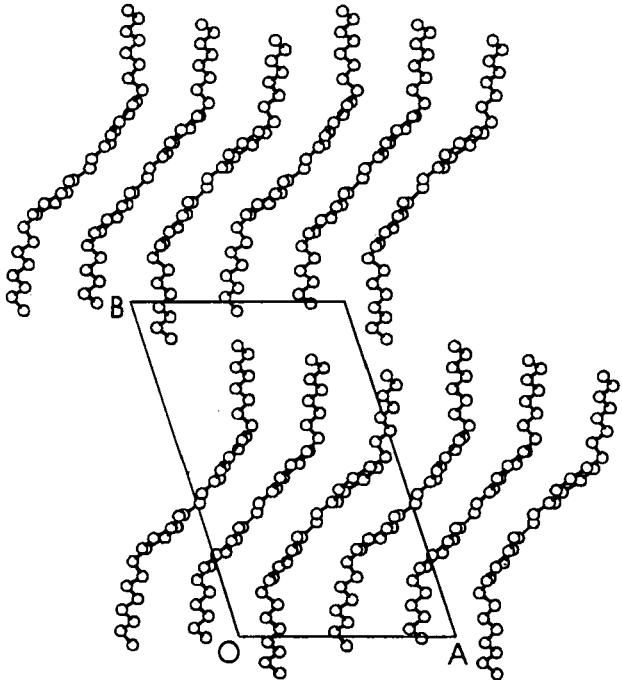


FIGURE 4 Packing of the molecules down *c*.



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