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Crystal Structure of Liquid-Crystalline Compounds Possessing a 1,4-Diaryl-1-buten-3-yne Rigid Core

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In the studies of new mesogen compounds featuring an enyne rigid core, three molecular structures were analyzed by X-ray. Two of them are presented here: 1) 1,4-di(octaoxyaryl)-1-buten-3-yne, [(C_8H_{17})O(C_6H_4)CHCHCC(C_6H_4)O(C_8H_{17})], monoclinic crystal system, Pa space group, a = 6.193(2) Å, b = 7.381(2) Å, c = 31.783(10) Å, β = 94.87(3)°, Z = 2, R1 = 0.0659, wR2 = 0.1738; and 2) 1,4-di(decaoxyaryl)-1-buten-3-yne [($C_{10}H_{21}$)O(C_6H_4)CHCHCC(C_6H_4)O($C_{10}H_{21}$)], monoclinic crystal system, Pa space group, a = 6.108(2) Å, b = 7.385(4) Å, c = 36.391(16) Å, β = 91.64(3)°, Z = 2, R1 = 0.0752, and wR2 = 0.1832.

Keywords: enyne rigid core; liquid crystal; mesogen; smectogen

INTRODUCTION

A series of liquid-crystalline molecules recently prepared by Soldera *et al*. designed from an enyne (1,4-diaryl-1-buten-3-yne) rigid core inserted between alkoxy chains was found to exhibit a rich thermal polymorphism; some compounds revealed up to four distinct mesophases [1]. The mesogens differed one from the other by the length of the alkoxy chains bound up with the core. Chains of n = 6, 8, 10, or 12 carbon atoms were used. The notation employed to designate the molecules is pDTn, where p is the number of carbon atoms in the chain linked to the double-bound (D) moiety of the enyne core

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TABLE 1 Crystallographic Data for 8DT8 and 10DT10

Compound	8DT8	10DT10
CCDC deposition number	283246	283247
Empirical formula	$C_{32}H_{44}O_2$	$C_{36}H_{52}O_2$
Formula weight (g)	460.67	516.78
Temperature	293(2)	
Wavelength (Å)	1.54176	1.54056
Crystal system	Monoclinic	
Space group	Pa	Pa
Unit cell dimensions	a = 6.139(2) Å	a = 6.108(2) Å
	b = 7.381(2) Å	b = 7.385(4) Å
	c = 31.783(10) Å	c = 36.391(16) Å
	$\alpha = 90^\circ$	$\alpha = 90^\circ$
	$\beta = 94.87(3)^\circ$	$\beta = 91.64(3)^\circ$
	$\gamma = 90^\circ$	$\gamma = 90^\circ$
Volume ($Å^3$)	1447.5(8)	1640.6(14)
Z	2	2
Density (calculated) (Mg/m^3)	1.057	1.046
Absorption coefficient (mm^{-1})	0.485	0.473
F(000)	504	568

Crystal size	$0.5 \times 0.5 \times 0.15\,mm^3$	$0.8 \times 0.5 \times 0.1\,mm^3$
θ range for data collection	1.39 to 70.03°	1.21 to 69.62°
Index ranges	$-7 \le h \le 7,\ 0 \le k \le 8,\ 0 \le l \le 38$	$-7 \le h \le 7,\ 0 \le k \le 8,\ 0 \le l \le 44$
Reflections collected	2717	3007
Independent reflections	2717 [R(int) = 0.0000]	3007 [R(int) = 0.0000]
Completeness to θ_{max}	98.8%	97%
Absorption correction	Empirical	Empirical
Max. and min. transmission	0.8052 and 0.6487	0.8722 and 0.4340
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	2712/2/329	3007/116/318
Goodness of fit on F^2	0.904	0.878
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0659, wR2 = 0.1738	R1 = 0.0752, wR2 = 0.1832
R indices (all data)	R1 = 0.1334, wR2 = 0.2170	R1 = 0.2228, wR2 = 0.2455
Absolute structure parameter	−0.5(8)	0.2(9)
Extinction coefficient	0.0019(7)	0.0004(3)
Largest diff. peak and hole	0.204 and −0.184 e.Å^{-3}	0.182 and −0.120 e.Å^{-3}

TABLE 2 Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 8DT8 (U_{eq} is Defined as One Third of the Trace of the Orthogonalized U_{ij} Tensor)

Atom	*x*	*y*	*z*	U_{eq}
C(1)	18170(30)	7690(30)	1920(5)	220(9)
C(2)	17970(30)	6991(19)	2351(4)	173(6)
C(3)	16243(18)	7787(17)	2585(3)	127(3)
C(4)	16031(17)	7113(14)	3022(3)	106(3)
C(5)	14161(17)	7783(13)	3246(3)	112(3)
C(6)	13930(16)	7067(15)	3679(3)	109(3)
C(7)	12014(17)	7842(12)	3869(3)	104(2)
C(8)	11631(16)	7112(15)	4295(3)	120(3)
C(10)	9022(14)	7564(10)	4803(3)	96(2)
C(11)	10285(13)	6665(11)	5137(2)	94(2)
C(12)	9540(13)	6680(11)	5532(3)	91(2)
C(13)	7597(12)	7501(11)	5632(3)	97(2)
C(14)	6372(12)	8403(9)	5284(2)	101(3)
C(15)	7117(11)	8373(10)	4886(2)	88(2)
C(16)	6891(14)	7374(12)	6037(3)	96(2)
C(17)	6250(30)	7260(20)	6386(6)	96(7)
C(18)	5380(40)	7050(20)	6780(7)	106(7)
C(17′)	4300(30)	7800(30)	6580(8)	104(7)
C(18′)	5030(40)	7920(30)	6177(6)	106(8)
C(16′)	3548(15)	7602(12)	6903(3)	101(3)
C(20)	2772(12)	7519(9)	7316(3)	86(2)
C(21)	3962(12)	6687(10)	7651(2)	92(2)
C(22)	3289(12)	6603(10)	8039(3)	97(2)
C(23)	1349(13)	7414(10)	8126(2)	80(2)
C(24)	127(13)	8270(11)	7807(2)	95(2)
C(25)	840(14)	8295(12)	7405(3)	102(2)
C(27)	−1247(10)	7942(13)	8643(2)	87(2)
C(28)	−1518(15)	7228(16)	9068(3)	112(3)
C(29)	−3542(14)	7847(15)	9262(3)	102(2)
C(30)	−3730(16)	7086(14)	9709(3)	109(3)
C(31)	−5590(20)	7899(18)	9920(4)	137(4)
C(32)	−5670(19)	7203(18)	10370(4)	137(4)
C(33)	−7580(20)	7960(20)	10582(5)	186(6)
C(34)	−7590(40)	7330(30)	11033(6)	261(10)
O(9)	9554(9)	7721(8)	4398(2)	102(2)
O(26)	795(8)	7264(9)	8525(2)	102(2)

and n is the number of carbon atoms attached to the triple-bound (T) moiety. The occurrence of thermal mesomorphism in organic compounds is largely influenced by the molecular arrangement in the crystalline state. As part of the structure–property relationship investigation of these liquid crystals, X-ray studies were

TABLE 3 Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 8DT8 (U_{eq} is Defined as One Third of the Trace of the Orthogonalized U_{ij} Tensor)

Atom	*x*	*y*	*z*	U_{eq}
C(1)	−10090(40)	7270(20)	3171(5)	303(13)
C(2)	−9840(40)	8010(30)	3570(4)	237(9)
C(3)	−7940(20)	7070(20)	3776(3)	164(6)
C(4)	−7640(20)	7909(18)	4150(3)	141(4)
C(5)	−5710(30)	7103(18)	4342(3)	157(5)
C(6)	−5290(20)	7899(15)	4726(3)	135(4)
C(7)	−3260(20)	7170(16)	4925(3)	126(4)
C(8)	−2770(20)	7896(15)	5313(3)	126(4)
C(9)	−810(30)	7118(18)	5492(3)	143(4)
C(10)	−270(30)	7959(17)	5879(3)	143(4)
C(12)	2620(15)	7407(8)	6315(1)	106(3)
C(13)	1544(14)	8306(8)	6593(2)	122(3)
C(14)	2476(15)	8377(8)	6946(2)	126(3)
C(15)	4484(15)	7550(9)	7021(2)	121(3)
C(16)	5560(14)	6652(8)	6743(2)	131(3)
C(17)	4628(15)	6581(8)	6390(2)	125(3)
C(18)	5400(13)	7668(13)	7398(3)	119(4)
C(19)	7500	7017(15)	7492(3)	166(4)
C(20)	8300	7300	7871(3)	148(4)
C(21)	9100	7473(16)	8158(3)	154(5)
C(22)	10250(15)	7554(10)	8520(1)	118(3)
C(23)	9157(14)	8407(8)	8801(2)	110(3)
C(24)	10053(16)	8399(8)	9157(2)	114(3)
C(25)	12042(15)	7539(9)	9231(2)	124(3)
C(26)	13135(14)	6686(8)	8949(2)	123(3)
C(27)	12239(15)	6694(9)	8593(2)	126(3)
C(29)	14970(20)	7043(14)	9698(2)	112(3)
C(30)	15510(20)	7751(16)	10054(3)	121(4)
C(31)	17760(20)	7125(16)	10225(3)	127(4)
C(32)	18130(20)	7885(17)	10606(3)	137(4)
C(33)	20210(20)	7125(17)	10803(3)	139(4)
C(34)	20450(20)	7850(15)	11193(3)	124(4)
C(35)	22510(30)	7160(20)	11410(4)	178(6)
C(36)	22830(30)	7769(19)	11804(4)	201(8)
C(37)	24760(30)	7030(20)	12007(4)	213(8)
C(38)	25110(30)	7630(20)	12381(5)	264(11)
O(11)	1822(17)	7302(11)	5963(2)	130(3)
O(28)	12800(16)	7739(10)	9587(2)	129(2)

performed on three target compounds: 8DT8, 10DT10, and 12DT6. In this article, we present the crystal structure of 8DT8 and 10DT10. 12DT6 is absent because of difficulties in getting crystals of sufficient quality.

TABLE 4 Bond Length and Angles for 8DT8

Bond	Length (Å)	Bond	Angle (°)
C(1)–C(2)	1.477(18)	C(1)–C(2)–C(3)	116.8(14)
C(2)–C(3)	1.478(16)	C(2)–C(3)–C(4)	117.5(10)
C(3)–C(4)	1.491(14)	C(3)–C(4)–C(5)	117.7(9)
C(4)–C(5)	1.494(14)	C(4)–C(5)–C(6)	117.4(9)
C(5)–C(6)	1.494(13)	C(7)–C(6)–C(5)	112.2(9)
C(6)–C(7)	1.490(14)	C(6)–C(7)–C(8)	114.9(8)
C(7)–C(8)	1.495(14)	O(9)–C(8)–C(7)	108.1(9)
C(8)–O(9)	1.426(12)	O(9)–C(10)–C(15)	115.3(7)
C(10)–O(9)	1.361(11)	O(9)–C(10)–C(11)	125.5(8)
C(10)–C(15)	1.368(12)	C(15)–C(10)–C(11)	119.2(9)
C(10)–C(11)	1.428(12)	C(12)–C(11)–C(10)	118.0(8)
C(11)–C(12)	1.375(12)	C(11)–C(12)–C(13)	124.7(8)
C(12)–C(13)	1.407(11)	C(16)–C(13)–C(12)	121.3(9)
C(13)–C(16)	1.397(12)	C(16)–C(13)–C(14)	123.3(8)
C(13)–C(14)	1.450(12)	C(12)–C(13)–C(14)	115.4(9)
C(14)–C(15)	1.383(11)	C(15)–C(14)–C(13)	119.8(7)
C(16)–C(17)	1.211(19)	C(10)–C(15)–C(14)	122.9(7)
C(16)–C(18′)	1.33(2)	C(17)–C(16)–C(18′)	50.0(11)
C(17)–C(18)	1.42(3)	C(17)–C(16)–C(13)	179.2(12)
C(18)–C(16′)	1.30(2)	C(18′)–C(16)–C(13)	129.4(13)
C(17′)–C(16′)	1.17(2)	C(16)–C(17)–C(18)	175.8(18)
C(17′)–C(18′)	1.40(3)	C(16′)–C(18)–C(17)	129.9(19)
C(16′)–C(20)	1.436(12)	C(16′)–C(17′)–C(18′)	174.4(18)
C(20)–C(25)	1.377(12)	C(16)–C(18′)–C(17′)	130(2)
C(20)–C(21)	1.385(11)	C(17′)–C(16′)–C(18)	49.7(12)
C(21)–C(22)	1.336(11)	C(17′)–C(16′)–C(20)	173.9(12)
C(22)–C(23)	1.391(11)	C(18)–C(16′)–C(20)	129.1(14)
C(23)–O(26)	1.344(9)	C(25)–C(20)–C(21)	116.3(8)
C(23)–C(24)	1.367(11)	C(25)–C(20)–C(16′)	122.2(8)
C(24)–C(25)	1.390(12)	C(21)–C(20)–C(16′)	121.5(8)
C(27)–O(26)	1.439(10)	C(22)–C(21)–C(20)	122.8(7)
C(27)–C(28)	1.474(12)	C(21)–C(22)–C(23)	120.5(7)
C(28)–C(29)	1.514(14)	O(26)–C(23)–C(24)	124.5(7)
C(29)–C(30)	1.540(13)	O(26)–C(23)–C(22)	116.5(6)
C(30)–C(31)	1.507(16)	C(24)–C(23)–C(22)	119.0(7)
C(31)–C(32)	1.523(15)	C(23)–C(24)–C(25)	119.3(7)
C(32)–C(33)	1.518(17)	C(20)–C(25)–C(24)	122.2(8)
C(33)–C(34)	1.51(2)	O(26)–C(27)–C(28)	106.6(7)
		C(27)–C(28)–C(29)	115.4(9)
		C(28)–C(29)–C(30)	113.3(8)
		C(31)–C(30)–C(29)	112.7(8)
		C(30)–C(31)–C(32)	111.8(10)
		C(33)–C(32)–C(31)	112.4(11)
		C(34)–C(33)–C(32)	112.0(13)
		C(10)–O(9)–C(8)	119.1(7)
		C(23)–O(26)–C(27)	121.1(6)

TABLE 5 Bond Length and Angles for 10DT10

Bond	Length (Å)	Bond	Angle (°)
C(1)–C(2)	1.55(2)	C(3)–C(2)–C(1)	110.6(17)
C(2)–C(3)	1.53(2)	C(4)–C(3)–C(2)	109.3(13)
C(3)–C(4)	1.500(15)	C(5)–C(4)–C(3)	109.9(11)
C(4)–C(5)	1.476(15)	C(4)–C(5)–C(6)	112.9(11)
C(5)–C(6)	1.530(15)	C(7)–C(6)–C(5)	114.5(11)
C(6)–C(7)	1.516(15)	C(6)–C(7)–C(8)	117.0(10)
C(7)–C(8)	1.532(14)	C(9)–C(8)–C(7)	114.3(11)
C(8)–C(9)	1.462(14)	C(8)–C(9)–C(10)	113.3(11)
C(9)–C(10)	1.567(15)	O(11)–C(10)–C(9)	103.2(10)
C(10)–O(11)	1.392(12)	O(11)–C(12)–C(13)	123.3(6)
C(12)–O(11)	1.360(9)	O(11)–C(12)–C(17)	116.7(6)
C(12)–C(13)	1.39	C(13)–C(12)–C(17)	120
C(12)–C(17)	1.39	C(14)–C(13)–C(12)	120
C(13)–C(14)	1.39	C(13)–C(14)–C(15)	120
C(14)–C(15)	1.39	C(16)–C(15)–C(14)	120
C(15)–C(16)	1.39	C(16)–C(15)–C(18)	122.2(6)
C(15)–C(18)	1.471(11)	C(14)–C(15)–C(18)	117.8(6)
C(16)–C(17)	1.39	C(15)–C(16)–C(17)	120
C(18)–C(19)	1.403(8)	C(16)–C(17)–C(12)	120
C(19)–C(20)	1.467(10)	C(19)–C(18)–C(15)	121.9(9)
C(20)–C(21)	1.145(10)	C(18)–C(19)–C(20)	117.3(8)
C(21)–C(22)	1.476(10)	C(21)–C(20)–C(19)	174.0(3)
C(22)–C(23)	1.39	C(20)–C(21)–C(22)	174.9(9)
C(22)–C(27)	1.39	C(23)–C(22)–C(27)	120
C(23)–C(24)	1.39	C(23)–C(22)–C(21)	116.7(6)
C(24)–C(25)	1.39	C(27)–C(22)–C(21)	123.0(6)
C(25)–O(28)	1.374(8)	C(24)–C(23)–C(22)	120
C(25)–C(26)	1.39	C(23)–C(24)–C(25)	120
C(26)–C(27)	1.39	O(28)–C(25)–C(26)	126.1(5)
C(29)–C(30)	1.425(13)	O(28)–C(25)–C(24)	113.7(5)
C(29)–O(28)	1.467(12)	C(26)–C(25)–C(24)	120
C(30)–C(31)	1.562(13)	C(25)–C(26)–C(27)	120
C(31)–C(32)	1.507(14)	C(26)–C(27)–C(22)	120
C(32)–C(33)	1.549(16)	C(30)–C(29)–O(28)	107.7(8)
C(33)–C(34)	1.521(15)	C(29)–C(30)–C(31)	115.5(10)
C(34)–C(35)	1.549(15)	C(32)–C(31)–C(30)	111.3(9)
C(35)–C(36)	1.514(19)	C(31)–C(32)–C(33)	113.1(10)
C(36)–C(37)	1.476(19)	C(34)–C(33)–C(32)	111.2(11)
C(37)–C(38)	1.441(17)	C(33)–C(34)–C(35)	114.4(12)
		C(36)–C(35)–C(34)	117.8(15)
		C(37)–C(36)–C(35)	116.3(15)
		C(38)–C(37)–C(36)	116.7(15)
		C(12)–O(11)–C(10)	119.2(8)
		C(25)–O(28)–C(29)	119.9(6)

EXPERIMENTAL

The 8DT8 crystal was grown by slow evaporation of a solution mixture of 2:1 methylethylketone–toluene at room temperature. The 10DT10 crystal was grown from a different solution mixture: 1:2 methylethylketone–toluene. In both cases, a single crystal was mounted using a glass fiber

TABLE 6 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 8DT8 (the Anisotropic Displacement Factor Exponent Takes the Form $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*b^*U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	247(18)	290(20)	129(10)	−23(11)	67(11)	−73(15)
C(2)	218(13)	200(12)	110(7)	−31(7)	73(8)	−33(10)
C(3)	131(7)	144(8)	102(6)	−15(5)	−12(5)	−15(6)
C(4)	120(6)	119(6)	80(4)	−22(4)	15(4)	−9(5)
C(5)	132(7)	100(5)	103(6)	−7(5)	−2(5)	−15(6)
C(6)	123(7)	103(5)	96(5)	−12(4)	−12(4)	−4(5)
C(7)	119(6)	87(5)	105(6)	8(4)	4(4)	6(5)
C(8)	135(8)	103(6)	120(7)	−26(5)	0(6)	−13(6)
C(10)	84(5)	75(5)	127(6)	−13(4)	−13(4)	−20(4)
C(11)	87(4)	87(5)	105(5)	−7(4)	−17(4)	−6(3)
C(12)	97(5)	82(5)	94(5)	−2(4)	2(4)	−1(4)
C(13)	88(5)	92(5)	112(6)	−20(4)	8(4)	−22(4)
C(14)	98(5)	77(4)	120(5)	3(4)	−44(4)	14(3)
C(15)	80(4)	85(5)	95(4)	13(4)	−6(3)	−6(4)
C(16)	99(6)	91(6)	98(6)	2(4)	3(5)	−3(4)
C(17)	123(14)	79(9)	84(11)	−6(7)	−1(9)	4(9)
C(18)	114(15)	78(9)	128(14)	16(9)	21(11)	12(10)
C(17′)	82(11)	89(12)	136(16)	−6(11)	−15(10)	11(9)
C(18′)	129(17)	114(14)	75(11)	−16(9)	8(10)	−24(12)
C(16′)	108(7)	86(6)	110(7)	−12(4)	13(5)	−4(5)
C(20)	92(5)	62(4)	103(5)	14(3)	−6(4)	11(3)
C(21)	83(4)	79(4)	118(5)	−3(4)	34(4)	−9(3)
C(22)	89(5)	83(5)	118(6)	−12(4)	−2(4)	0(4)
C(23)	84(4)	80(4)	77(4)	5(3)	8(3)	6(3)
C(24)	95(5)	78(4)	116(6)	10(4)	29(4)	23(4)
C(25)	94(5)	92(6)	117(6)	20(5)	−3(4)	13(4)
C(27)	63(3)	107(5)	89(4)	4(4)	0(3)	2(4)
C(28)	91(5)	140(7)	103(6)	−18(5)	−2(4)	−9(5)
C(29)	95(5)	111(6)	101(5)	9(5)	7(4)	3(5)
C(30)	107(6)	108(6)	108(6)	4(5)	−7(4)	11(5)
C(31)	120(7)	146(9)	141(8)	23(6)	−9(6)	9(7)
C(32)	156(9)	141(9)	118(7)	13(6)	40(6)	8(7)
C(33)	155(11)	234(14)	171(12)	24(10)	20(8)	45(11)
C(34)	250(20)	390(30)	148(12)	41(14)	63(12)	80(20)
O(9)	100(4)	109(4)	97(4)	−5(3)	4(3)	1(3)
O(26)	88(3)	112(4)	103(4)	17(3)	−5(3)	6(3)

on the goniometer. Data were collected on an Enraf-Nonius CAD-4 automatic diffractometer using $\omega/2\theta$ scans at 293(2) K. The DIFRAC [2] program was used for centering, indexing, and data collection. Two standard reflections were measured every 100 reflections; an intensity

TABLE 7 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 10DT10 (the Anisotropic Displacement Factor Exponent Takes the Form $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*b^*U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	380(30)	360(30)	164(14)	−21(18)	−60(16)	−70(30)
C(2)	290(20)	239(18)	184(15)	−32(13)	24(16)	−65(19)
C(3)	122(9)	219(14)	148(11)	23(9)	−30(7)	−24(9)
C(4)	125(9)	165(11)	132(10)	−10(8)	−4(7)	10(9)
C(5)	157(11)	197(12)	117(8)	−49(8)	0(7)	12(10)
C(6)	134(9)	147(9)	126(9)	28(7)	21(6)	25(8)
C(7)	155(10)	124(9)	103(7)	−9(6)	33(6)	−16(8)
C(8)	114(8)	116(8)	149(10)	39(7)	24(6)	0(7)
C(9)	157(11)	131(10)	142(10)	−21(8)	1(8)	2(9)
C(10)	145(11)	120(9)	162(11)	19(8)	−16(7)	21(8)
C(12)	106(6)	84(6)	129(7)	30(5)	36(5)	−15(5)
C(13)	132(8)	98(7)	136(7)	−11(6)	−3(5)	19(6)
C(14)	125(7)	112(8)	139(6)	6(6)	−9(5)	−1(6)
C(15)	120(7)	101(9)	141(8)	6(6)	1(6)	−22(5)
C(16)	107(7)	121(9)	164(8)	−19(7)	2(5)	−16(5)
C(17)	108(6)	119(8)	147(7)	3(6)	15(5)	2(5)
C(18)	83(6)	115(9)	160(9)	20(7)	19(6)	35(6)
C(19)	168(10)	176(10)	155(8)	21(7)	1(7)	−33(8)
C(20)	140(9)	154(9)	150(7)	7(9)	0(6)	−28(8)
C(21)	202(13)	130(10)	129(7)	−16(7)	14(7)	−54(9)
C(22)	130(7)	105(8)	119(6)	−14(5)	18(5)	−7(6)
C(23)	109(6)	95(7)	128(6)	9(5)	25(4)	9(5)
C(24)	102(6)	117(8)	125(5)	−5(6)	30(5)	−10(5)
C(25)	124(7)	117(9)	131(7)	−36(6)	−2(6)	1(6)
C(26)	101(6)	127(9)	142(7)	5(6)	31(5)	6(5)
C(27)	119(7)	124(9)	136(6)	−24(7)	30(5)	−2(6)
C(29)	122(8)	123(7)	95(6)	−15(6)	44(5)	−18(7)
C(30)	115(8)	137(9)	112(8)	25(7)	20(6)	17(7)
C(31)	116(8)	158(10)	108(8)	−23(6)	14(5)	8(7)
C(32)	103(7)	137(10)	171(12)	23(8)	−11(7)	12(7)
C(33)	154(11)	117(9)	145(10)	−25(7)	−11(7)	−17(8)
C(34)	126(8)	106(8)	139(9)	31(7)	13(7)	−25(7)
C(35)	210(15)	180(14)	144(10)	20(10)	0(10)	−9(12)
C(36)	310(20)	153(12)	138(12)	−12(9)	31(13)	24(14)
C(37)	207(15)	264(16)	161(13)	−2(11)	−98(12)	70(13)
C(38)	230(19)	370(30)	185(16)	20(18)	−55(14)	50(20)
O(11)	123(6)	138(7)	129(5)	12(5)	14(4)	−20(5)
O(28)	110(5)	150(7)	128(5)	−16(4)	11(4)	29(5)

decay of approximately 2% was observed during data collection for 8DT8 whereas no intensity decay occurred for 10DT10. The data were corrected for absorption by empirical methods based on Ψ scans and reduced with the NRCVAX [3] programs. They were solved using SHELXS-97 [4] and refined by full-matrix least squares on F^2 with SHELXL-97 [5]. The nonhydrogen atoms were refined anisotropically. The hydrogen atoms were placed at idealized calculated geometric position and refined isotropically using a riding model. The absolute structure [6] could not be determined with the anomalous dispersion effects because no heavy atoms were present. In the case of 8DT8, the C16 to C18 carbons were refined as the actual and inverted absolute structure positions with an additive total occupation of 1 to better fit the observed data. A population of approximately 50% for one or the other is consistent with the absence of heavy atoms to determine the real absolute structure. In the case of 10DT10, the two phenyls were refined with ideal six-member rings and SIMU (similar thermal) DELU (equal bond) restraints. C18 to C22 carbons were refined with EADP (equal thermal) and positional restraints to avoid observed positional and thermal distortion caused by the inverted structure. Crystallographic data for 8DT8 and 10DT10 are summarized in Table 1. Fully completed crystallographic information files (CIF) have been deposited with the Cambridge Crystallographic Data Centre bearing the deposition numbers CCDC 283246 and 283247 for 8DT8 and 10DT10, respectively.

RESULTS AND DISCUSSION

Atomic coordinates and equivalent isotropic displacement parameters for 8DT8 are listed in Table 2, and the ones for 10DT10 are given in

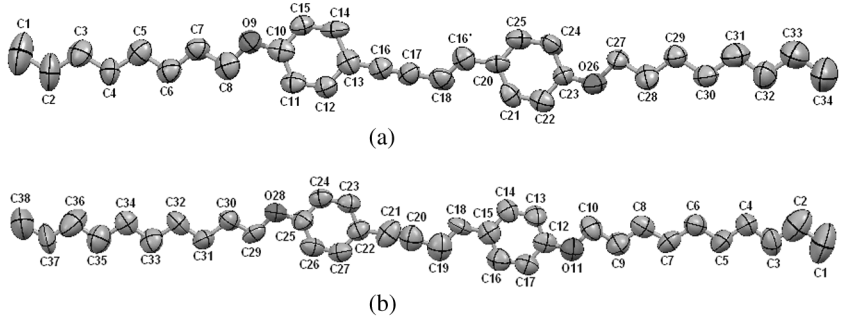

FIGURE 1 Molecular conformation, atom labeling, and ellipsoid representation (50% probability) of (a) 8DT8 and (b) 10DT10.

Table 3. Tables 4 and 5 present the bond lengths and angles for 8DT8 and 10DT10, respectively. Anisotropic displacement parameters for nonhydrogen atoms are given in Tables 6 and 7 for 8DT8 and 10DT10 respectively. Figure 1 shows the molecular conformation, the atomic labeling, and the ellipsoid representation (50% probability) of both compounds. The phenyl rings are nearly coplanar. We drew a mean plane through each phenyl ring and calculated the angle between

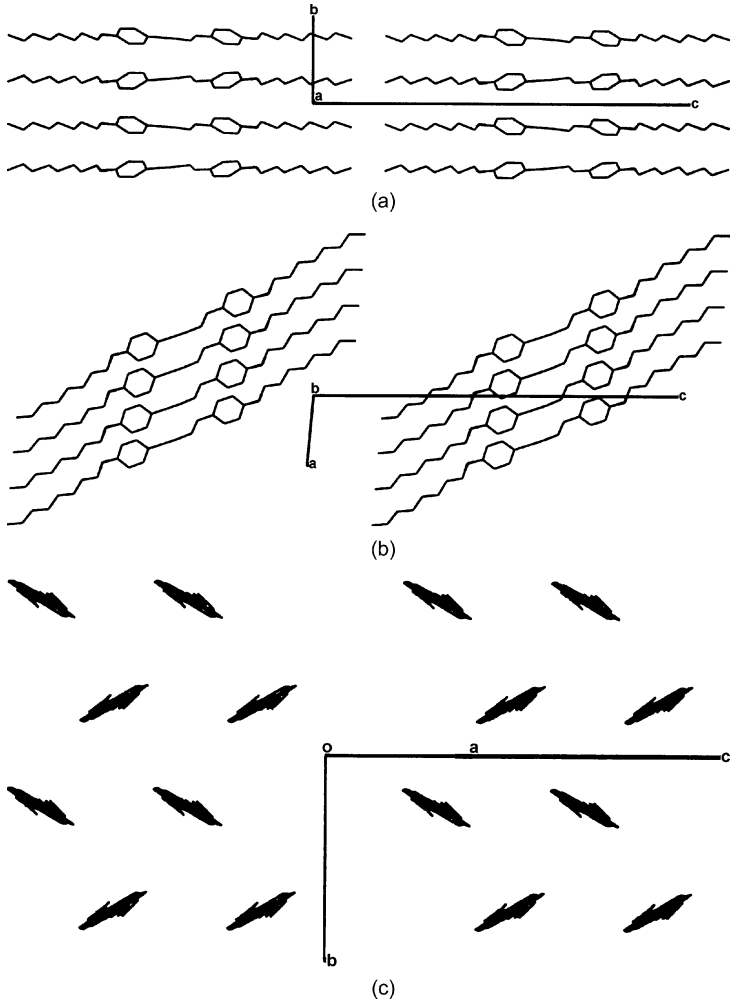

FIGURE 2 Crystal packing of 8DT8 viewed along (a) *a*-axis, (b) *b*-axis, and (c) the molecular axis.

these planes to be 1.29° for 8DT8 and 2.58° for 10DT10. The enyne moiety is very slightly bent, as can be observed from the torsion angles C(16)–C(17)–C(18)–C(16′) = –176.46° in 8DT8 and C(18)–C(19)–C(20)–C(21) = 177.32° in 10DT10. The molecules are fully extended with lengths of 34.214 and 39.384 Å for 8DT8 and 10DT10 respectively.

Figures 2 and 3 show the packing structures of 8DT8 and 10DT10, respectively. Different projections are presented. In Figs. 2b and 3b the molecules are viewed along the *b*-axis. The molecular arrangement

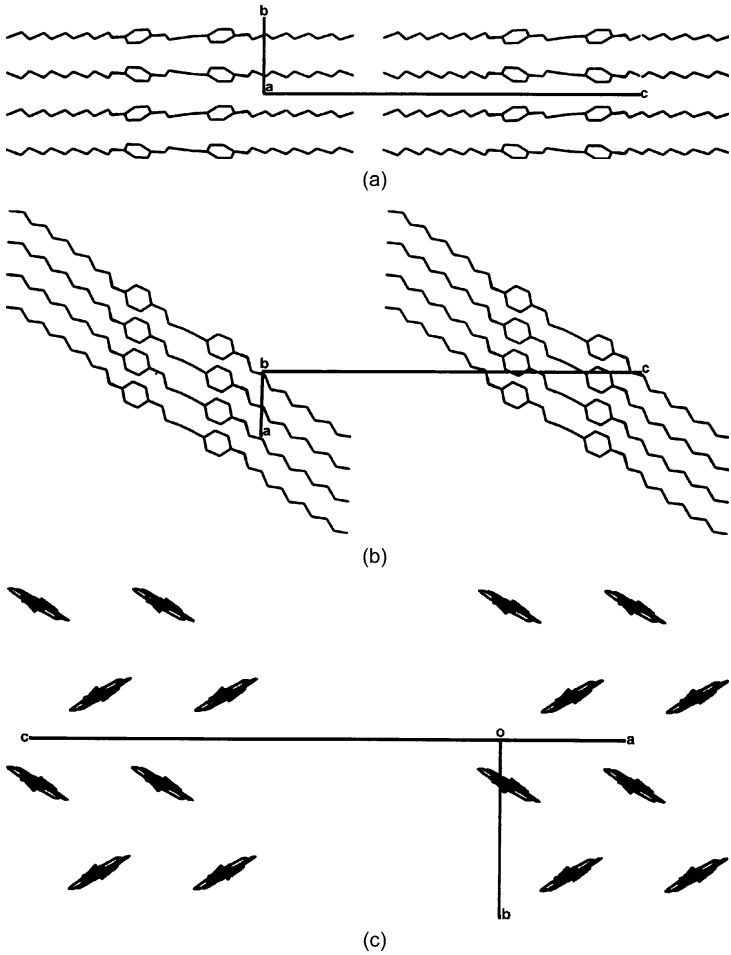

FIGURE 3 Crystal packing of 10DT10 viewed along (a) *a*-axis, (b) *b*-axis, and (c) the molecular axis.

appears as a tilted layer structure, which is clearly a precursor of the tilted smectic phases observed for both compounds [1]. Viewing the structure along the molecular axis (Figs. 2c and 3c) reveals the herringbone-type packing. This arrangement precludes π-stacking of the phenyl groups between neighboring molecules. The lateral intermolecular attractions are then limited to weak van der Waals forces, which could explain the relatively low transition temperature for the appearance of the isotropic liquid phase (133.6°C for 8DT8, 134.4 for 10DT10).

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