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Crystal and Molecular Structures of Diethyl 6-[4-(4′-Nitrophenylazo)phenoxy]-hexylmalonate and its 2′-Nitrophenyl Isomer

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Crystal and Molecular Structures of Diethyl 6-[4-(4′-Nitrophenylazo)phenoxy]-hexylmalonate and its 2′-Nitrophenyl Isomer

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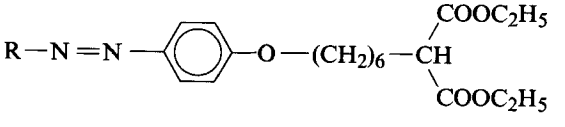
The crystal and molecular structures of diethyl 6-[4-(4′-nitrophenylazo)phenoxy]hexylmalonate (**1**) as a comonomer in the synthesis of liquid-crystalline polymers and its 2′-nitrophenyl isomer (**2**) have been determined by X-ray analysis. Both compounds crystallize in space group $P\bar{1}$ with two molecules per unit cell and the following lattice parameters. **1**: $a = 7.995(2)$, $b = 10.699(2)$, $c = 15.021(4)$ Å, $\alpha = 95.04(2)$, $\beta = 96.54(2)$, $\gamma = 94.72(3)°$. **2**: $a = 8.912(2)$, $b = 9.664(2)$, $c = 16.064(4)$ Å, $\alpha = 98.68(2)$, $\beta = 97.54(1)$, $\gamma = 105.11(1)°$. The structures were solved by direct methods and refined to R (wR) values of 0.082 (0.068) and 0.071 (0.076), respectively.

The molecules have an optimum stretched shape excepted one of their two ethoxycarbonyl groups which is perpendicularly oriented to the molecular long axis. The molecular packing in the crystal can be described in terms of a sheet structure in which the sheets are penetrated by the laterally branched ethoxycarbonyl groups of neighboring sheets.

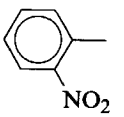
Keywords: Liquid-crystalline polymers, side group mesogens, molecular structure, crystal packing.

INTRODUCTION

As part of a program of structural investigations on mesogenic and related compounds with an unconventional molecular shape X-ray structure analyses of diethyl 6-[4-(4′-nitrophenylazo)phenoxy]hexylmalonate (**1**) and its 2′-nitrophenyl isomer (**2**) have been performed.



1: R = O_2N—



2: R =

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According to the survey given by Demus[1] **1** and **2** can be classified as polar swallow-tailed polycatenar compounds. Recently, **1** was subject of dielectric studies about the competition between dipolar and steric interactions in binary mixtures of swallow-tailed substances.[2] Moreover, it was used as a comonomer in the synthesis of liquid-crystalline polymers. Using a method of Reck and Ringsdorf[3] enabling the synthesis of side group polyesters with definite lengths of main chain spacers for the polycondensation of mesogenic diethyl malonates with aliphatic α,ω-diols HO—$(CH_2)_x$—OH Böhme *et al.*[4] prepared a series of polymalonates carrying a bisazobenzene mesogen. In continuation of these investigations, a homologous series of such liquid-crystalline polymers with **1** as mesogenic side group but with a different number x of methylene groups, i.e. a different distance between the contact points of two neighboring side groups, were synthesized. Though **1** itself has no liquid-crystalline properties, the corresponding polyesters exhibit mesophases of different kind and number.[5]

For the discussion and modelling of the structures of the above-mentioned liquid-crystalline polymers, a detailed knowledge of the molecular structure and packing arrangement within the crystal for **1** seemed to be useful. The X-ray analysis of **2** has been carried out for structural comparison.

EXPERIMENTAL

Synthesis

The preparation and characterization of compound **1** were performed as described previously.[5]

Compound **2** was synthesized in an analogous way: in a first step *o*-nitroaniline was diazotized and coupled with phenol to 4-(2′-nitrophenylazo)phenol. The following etherification with diethyl bromo-hexylmalonate gave **2**.

Diethyl 6-[4-(2′-nitrophenylazo)phenoxy]hexylmalonate (**2**):

$C_{25}H_{31}N_3O_7$, m.p.: 73°C, Calc. C 61.85 H 6.39 N 8.66
Found C 61.82 H 6.45 N 8.78

^{1}H-NMR spectra (200.13 MHz in $CDCl_3$): δ (in ppm) = 7.92 – 7.85 (m, 3H, arom.), 7.65 – 7.62 (m, 2H, arom.), 7.53 – 7.46 (m, 1H, arom.), 6.99 – 6.94 (d, 2H, arom.), 4.23 – 4.12 (q, 4H, OCH_2), 4.05 – 3.98 (t, 2H, OCH_2), 3.34 – 3.26 (t, 1H, CH), 2 – 1 (m, H, CH_2 and CH_3).

Crystal Structure Determination

Optically clear orange crystals of **1** and **2**, obtained by recrystallization from ethanol, were cut to give specimens suitable for X-ray diffraction measurements. They were mounted on a Stoe STADI4 four-circle diffractometer and investigated using graphite monochromatized MoKα radiation. Lattice parameters were derived by least-squares

treatment of the setting angles for 34 (for **1**) and 30 (for **2**) reflections, respectively. Intensity data were measured by ω/θ-scanning mode. Data reduction was carried out applying Lorentz and polarization correction but neglecting absorption and extinction effects. Both structures were solved by direct methods and refined by full-matrix least-squares refinement on F. Non-H atom positions were refined with anisotropic displacement parameters. The positions of the H atoms were geometrically calculated and refined isotropically using the riding model.

Relevant crystal data and parameters of structure solution and refinement are summarized in Table I, final atomic parameters in Table II. All calculations were done on an IBM RISC/6000-320 work station using the program packages SHELXS-86,[6] SHELX-76,[7] and EDIT.[8] Figures of molecular structures and packing were plotted using programs ORTEP[9] and PLUTO.[10]

TABLE I

Crystal Data and Details of Intensity Measurements and Structure Refinements

Compound	1		2
Chemical formula		$C_{25}H_{31}N_3O_7$	
T_F (°C)	116		73
Molecular weight ($gmol^{-1}$)		485.54	
Crystal description		prisms	
Measuring temperature (°C)	20		20
Crystal system	triclinic		triclinic
Space group	$P\bar{1}$		$P\bar{1}$
a (Å)	7.995(2)		8.912(2)
b (Å)	10.699(2)		9.664(2)
c (Å)	15.021(4)		16.064(4)
α (°)	95.04(2)		98.68(2)
β (°)	96.54(2)		97.54(1)
γ (°)	94.72(3)		105.11(1)
Unit cell volume ($Å^3$)	1266.0(5)		1299.1(5)
Z	2		2
D_x (Mgm^{-3})	1.2740		1.2412
Crystal size (mm)	0.13 × 0.27 × 0.44		0.08 × 0.34 × 0.99
Radiation		Mo K(α), $\lambda = 0.71073$Å	
Absorption coefficient (mm^{-1})		0.090	
F(000)		516	
No. of reflections measured	10516		8162
No. of independent reflections	5258		4082
No. of observed reflections ($F_0 > 3.92\sigma(F_0)$)	2020		1989
Collection method		ω/θ-scan	
R_{int}	0.035		0.035
θ_{max} (°)	26.6		24.0
Variation of standards (%)	3.4		2.1
h, k, l(min/max)	$\bar{10}, \bar{13}, 0/10, 13, 18$		$\bar{10}, \bar{11}, 0/10, 11, 18$
No. of reflections/parameter	7.1		6.7
R	0.0816		0.0714
wR	0.0680		0.0763
S	4.69		4.14
Weighting scheme	$w = 4.56/(\sigma^2(F_0) + 0.0003F_0^2))$		$w = 3.32/(\sigma^2(F_0) + 0.0005F_0^2))$
$(\Delta/\sigma)_{max}$	0.001		0.001
min/max $\Delta\rho$ ($eÅ^{-3}$)	−0.385/0.345		−0.274/0.233

TABLE II

Final Fractional Atomic Coordinates and Equivalent Thermal Parameters (Å^2) $U_{eq} = (1/3)\sum i \sum j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$

Atom	1				2			
	x/a	*y/b*	*z/c*	U_{eq}	*x/a*	*y/b*	*z/c*	U_{eq}
C1	0.0057(6)	−0.4110(4)	0.7769(3)	0.063(5)	0.5446(5)	0.3782(5)	0.2141(3)	0.055(4)
C2	−0.1095(6)	−0.3292(4)	0.7509(3)	0.068(5)	0.6366(5)	0.4202(5)	0.2953(3)	0.052(4)
C3	−0.0795(5)	−0.2056(4)	0.7873(3)	0.067(5)	0.5995(5)	0.3281(5)	0.3523(3)	0.068(5)
C4	0.0590(5)	−0.1665(4)	0.8482(3)	0.053(4)	0.4788(6)	0.2007(6)	0.3285(4)	0.076(5)
C5	0.1711(5)	−0.2520(4)	0.8754(3)	0.066(5)	0.3900(6)	0.1611(5)	0.2476(4)	0.074(5)
C6	0.1446(6)	−0.3747(4)	0.8387(3)	0.071(5)	0.4203(5)	0.2524(5)	0.1892(3)	0.065(5)
C7	0.2126(5)	0.1289(4)	0.9682(3)	0.054(4)	0.9569(5)	0.7130(5)	0.4117(3)	0.052(4)
C8	0.0941(5)	0.2113(4)	0.9398(3)	0.062(4)	1.0477(5)	0.7390(5)	0.4917(3)	0.059(5)
C9	0.1153(5)	0.3342(4)	0.9738(3)	0.061(4)	1.1627(5)	0.8689(5)	0.5238(3)	0.058(4)
C10	0.2528(5)	0.3789(4)	1.0367(3)	0.053(4)	1.1899(5)	0.9734(5)	0.4738(3)	0.052(4)
C11	0.3696(5)	0.2986(4)	1.0663(3)	0.060(4)	1.0980(5)	0.9495(5)	0.3926(3)	0.060(5)
C12	0.3468(5)	0.1735(4)	1.0308(3)	0.061(4)	0.9819(5)	0.8197(5)	0.3615(3)	0.056(5)
C13	0.3997(5)	0.5608(4)	1.1277(3)	0.060(4)	1.4037(5)	1.1404(5)	0.5782(3)	0.063(5)
C14	0.3731(5)	0.6979(4)	1.1431(3)	0.061(4)	1.5222(5)	1.2844(5)	0.5811(3)	0.062(4)
C15	0.5061(5)	0.7735(4)	1.2117(3)	0.059(4)	1.6432(5)	1.3393(5)	0.6635(3)	0.064(5)
C16	0.4791(5)	0.9120(4)	1.2184(3)	0.060(4)	1.7563(5)	1.4853(5)	0.6632(3)	0.059(4)
C17	0.6013(5)	0.9944(4)	1.2894(3)	0.060(4)	1.8775(5)	1.5499(5)	0.7441(3)	0.060(4)
C18	0.5583(5)	1.1299(4)	1.2948(3)	0.063(4)	1.9868(5)	1.6979(5)	0.7398(3)	0.061(4)
C19	0.6748(5)	1.2191(4)	1.3647(3)	0.055(4)	2.1066(5)	1.7705(5)	0.8214(3)	0.053(4)
C20	0.6333(6)	1.3539(4)	1.3600(3)	0.067(5)	2.2076(6)	1.9164(6)	0.8110(4)	0.073(5)
C21	0.6954(7)	1.5644(4)	1.4272(4)	0.092(5)	2.4145(8)	2.1288(6)	0.8799(4)	0.099(6)
C22	0.8200(8)	1.6331(5)	1.4929(4)	0.127(6)	2.5181(8)	2.1806(7)	0.9559(5)	0.132(7)
C23	0.6546(6)	1.1827(4)	1.4591(3)	0.061(4)	2.0203(6)	1.7988(5)	0.8943(3)	0.060(5)
C24	0.7783(6)	1.1090(5)	1.5886(3)	0.091(5)	1.9670(9)	1.7406(9)	1.0260(4)	0.125(7)
C25	0.9524(7)	1.0754(6)	1.6259(4)	0.115(6)	2.051(1)	1.727(1)	1.1005(4)	0.151(8)
N1	−0.0235(7)	−0.5443(4)	0.7383(3)	0.085(5)	0.5759(6)	0.4731(5)	0.1517(3)	0.073(5)
N2	0.0737(4)	−0.0368(3)	0.8806(2)	0.058(4)	0.7518(4)	0.5590(4)	0.3175(2)	0.056(4)
N3	0.2007(4)	−0.0009(3)	0.9370(2)	0.058(4)	0.8433(4)	0.5748(4)	0.3862(2)	0.060(4)
O1	0.0814(6)	−0.6149(4)	0.7607(3)	0.118(5)	0.4636(6)	0.5064(5)	0.1172(3)	0.123(5)
O2	−0.1510(6)	−0.5750(4)	0.6869(3)	0.122(5)	0.7060(5)	0.5101(5)	0.1364(3)	0.106(5)
O3	0.2597(3)	0.5030(3)	1.0654(2)	0.067(3)	1.3021(4)	1.1041(3)	0.4961(2)	0.065(4)
O4	0.5277(5)	1.3869(3)	1.3074(3)	0.105(4)	2.2040(5)	1.9648(5)	0.7468(3)	0.120(5)
O5	0.7303(4)	1.4315(3)	1.4208(2)	0.086(4)	2.3103(4)	1.9840(4)	0.8808(2)	0.081(4)
O6	0.5251(5)	1.1829(3)	1.4908(2)	0.093(4)	1.9328(4)	1.8751(4)	0.8964(2)	0.088(4)
O7	0.7959(3)	1.1470(3)	1.4994(2)	0.067(4)	2.0493(4)	1.7267(4)	0.9547(2)	0.088(4)

Further details of the crystal structure determinations are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, on quoting the depository numbers CSD-400 396 (**1**) and CSD-400 397 (**2**), the names of the authors, and the journal citation.

RESULTS AND DISCUSSION

Molecular Structure

The molecular structures of **1** and **2** included the applied labelling are shown in Figures 1 and 3. Essential parameters of the molecular geometry are summarized in Tables III and IV. A realistic picture of the molecular shape of **1** can be visualized by the space-filling PLUTO model[10] using the van der Waals radii given by Bondi[11] shown in Figure 2.

As can be seen from Figures 1 and 2, the molecules of **1** have a perfectly stretched shape excepted the C19... C25 fragment (see below). Moreover, the planar structural moieties are largely parallel oriented to each other. So the nitro group in **1** is tilted to the benzene ring to which it is attached by only 2.6(2)°. A reasonable number of X-ray structure determinations have been shown that azobenzene and its 4,4′-disubstituted derivatives are completely or essentially planar in the solid state.[12–14] These results are confirmed by the observed conformation of the azobenzene moiety in **1**. Its C—N=N—C central part is exactly planar within experimental error (cf. the

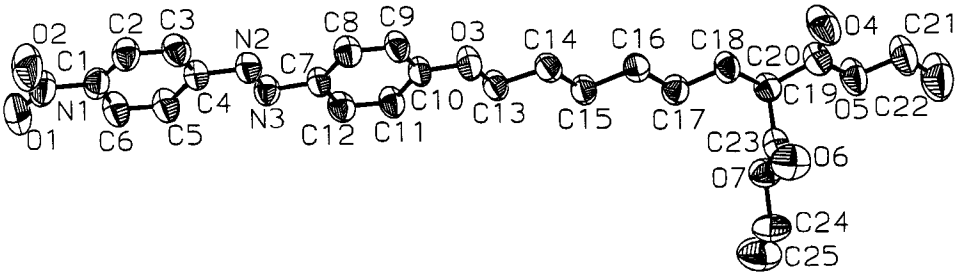

FIGURE 1 ORTEP plot[9] of the molecular structure of compound **1** with the atom labelling used in the X-ray analysis. Thermal ellipsoids are shown at the 50% probability level; H atoms are omitted for clarity.

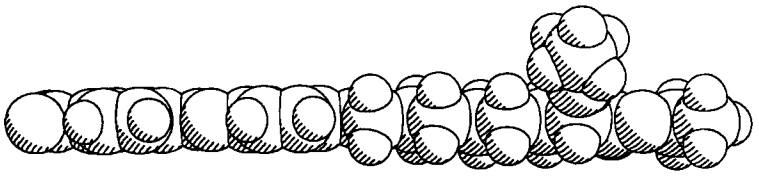

FIGURE 2 Space-filling PLUTO plot[10] of the molecular structure of **1** (atom spheres according to van der Waals radii[11]).

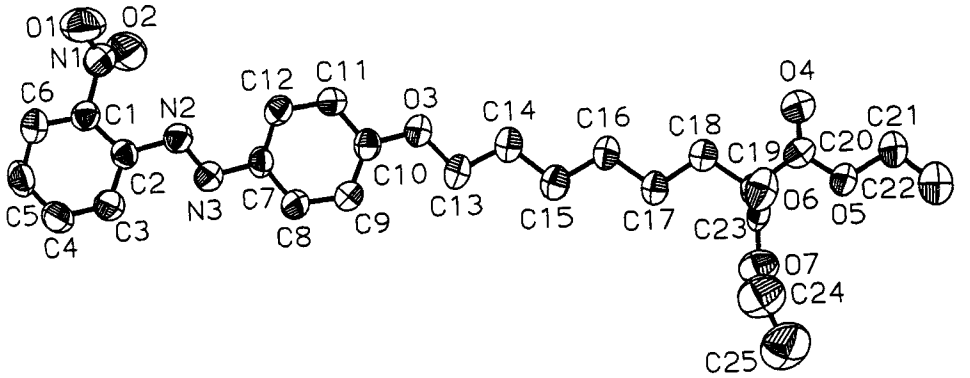

FIGURE 3 ORTEP plot[9] of the molecular structure of **2**.

TABLE III

Selected Bond Lengths and Angles for Non-Hydrogen Atoms in **1** and **2** (esd's in Parantheses)

Atoms	Distance (Å)		Atoms	Angle (°)	
	1	**2**		**1**	**2**
			Nitro group		
C1–N1	1.482(6)	1.463(7)	C1–N1–O1	117.8(4)	116.7(4)
N1–O1	1.213(6)	1.220(7)	C1–N1–O2	117.5(4)	119.0(4)
N1–O2	1.210(7)	1.191(7)	O1–N1–O2	124.7(4)	124.3(4)
			Azo group		
C4–N2	1.421(5)	–	C4–N2–N3	115.1(3)	–
C2–N2	–	1.422(6)	C2–N2–N3	–	113.0(3)
N2–N3	1.256(5)	1.246(5)	N2–N3–C7	114.0(3)	114.7(3)
N3–C7	1.418(5)	1.413(6)			
			Diethyl malonate group		
C18–C19	1.528(6)	1.523(6)	C18–C19–C20	111.0(3)	109.9(4)
C19–C20	1.512(6)	1.509(7)	C18–C19–C23	109.9(3)	109.3(4)
C19–C23	1.526(6)	1.511(7)	C19–C20–O4	124.7(4)	125.9(4)
C20–O4	1.186(6)	1.195(8)	C19–C20–O5	111.8(3)	111.8(4)
C20–O5	1.314(6)	1.311(7)	C20–O5–C21	117.2(3)	117.5(4)
O5–C21	1.468(5)	1.467(7)	C19–C23–O6	125.6(4)	124.7(4)
C23–O6	1.188(6)	1.206(7)	C19–C23–O7	112.0(3)	115.6(4)
C23–O7	1.322(5)	1.315(7)	C23–O7–C24	112.8(3)	116.3(4)
O7–C24	1.452(6)	1.447(8)			
			Hexyloxy group		
C10–O3	1.354(5)	1.354(5)	C10–O3–C13	119.9(3)	119.1(3)
O3–C13	1.435(5)	1.437(6)	O3–C13–C14	106.8(3)	106.5(3)
C–C (mean)	1.514(9)	1.513(9)	C–C–C (mean)	113.1(1.9)	113.2(1.4)
			Mean values for benzene rings		
			Ring I (C1 ... C6)		
C–C	1.373(8)	1.380(8)	C–C–C	120.0(1.6)	120.0(2.3)
			Ring II (C7 ... C12)		
C–C	1.381(16)	1.382(11)	C–C–C	120.0(1.1)	120.0(8)

corresponding torsion angles in Table IV) and not significantly inclined to the neighboring phenyl rings I (C1 ... C6) and II (C7 ... C12). The hexyloxy group adopts a fully staggered conformation and is largely coplanar with the neighboring structural fragments benzene ring II (interplanar angle 2.7(2)°) and acetyloxy group C19, C20, O4, O5 (9.0(2)°). In relation to the molecular long axis the two branches of the diethyl malonate fragment have not the expected Y orientation. Whereas, as already discussed, the C19 ... C22 chain lies in the direction of the long axis, the C19 ... C25 branch is perpendicular oriented (tilt angle: 86°) to it.

According to our expectation the molecular structures of the isomers **1** and **2** are in nearly complete agreement with only one relevant exception. As already above mentioned, the para-positioned nitro group in **1** is essentially coplanar with the benzene ring. But in **2** the nitro group as a result of its ortho-position is markedly twisted about the C1—N1 bond and inclined to the benzene ring by 52.7(2)°. It should be worth mentioning that also the planarity of the azobenzene fragment is somewhat affected. The interplanar angles between the C—N=N—C central part and the phenyl rings I and II amount to 14.5(3) and 7.0(3)°, respectively, and between the phenyl rings to 8.2(2)°.

All individual values for the bond lengths and angles in both structures are quite normal, they agree well with standard values[15] and require no comment. A significant shortening of the bond lengths for both terminal ethyl groups in **2** is correlated with above-average high atomic displacement parameters and due to a slight disorder effect.

Crystal Packing

The molecular arrangements in the crystals of **1** and **2** are illustrated in Figures 4 and 5. According to the space group symmetry $P\bar{1}$ the crystal packing is characterized by a perfectly parallel alignment of the molecular long axes and an antiparallel orientation

TABLE IV

Selected Torsion Angles (°)

	1	**2**
C2–C1–N1–O1	178.6(5)	−127.4(5)
C2–C1–N1–O2	−1.8(6)	+54.5(6)
C3–C4–N2–N3	179.7(4)	–
C1–C2–N2–N3	–	−169.5(5)
C4–N2–N3–C7	179.9(4)	–
C2–N2–N3–C7	–	−178.6(4)
N2–N3–C7–C8	0.3(5)	−173.3(5)
C9–C10–O3–C13	−177.6(4)	0.1(5)
C10–O3–C13–C14	177.9(4)	176.3(4)
C17–C18–C19–C20	175.0(4)	−179.5(4)
C17–C18–C19–C23	−65.1(4)	−61.7(4)
C18–C19–C20–O4	−2.1(5)	−4.9(6)
C18–C19–C20–O5	178.8(4)	+178.7(5)
C18–C19–C23–O6	−61.0(5)	−74.5(6)
C18–C19–C23–O7	116.8(4)	+103.6(4)
C19–C20–O5–C21	−176.2(4)	−179.1(5)
C19–C23–O7–C24	−178.6(4)	−176.7(6)

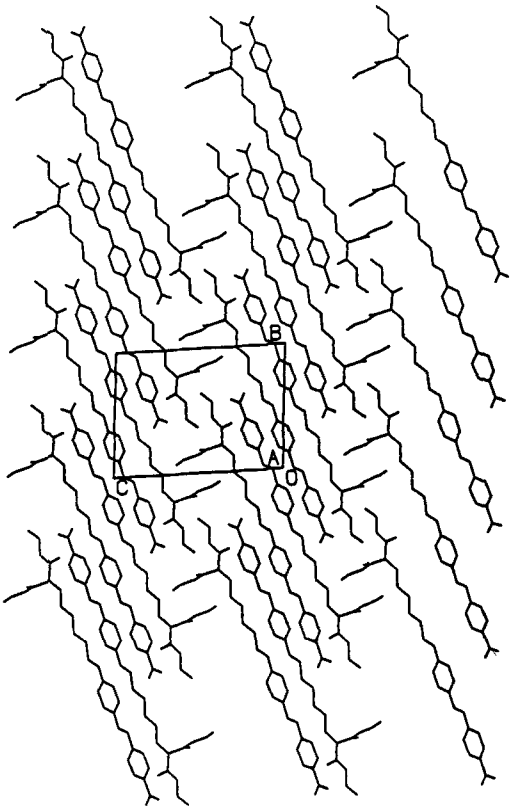

FIGURE 4 PLUTO plot[10] of the crystal packing of **1** projected along [100].

of neighbored molecules. This antiparallelism is a well known phenomenon for crystalline mesogens in general and for swallow-tailed compounds in particular.

For compound **1** the molecular packing can be described in detail as follows. As can be seen in Figure 4, two neighboring molecules related by the inversion centre in (1/2, 1/2, 0) are arranged side by side without a noticeable parallel shift and form a "non-shifted" molecular pair. The shortest distance between the long axes (the long axis of a molecule is definded as the least-squares line through all non-H atoms excepted those of the nitro group, the C23 ... C25 branch and atom O4) of such a pair amounts to 5.48 Å. In contrast to that, two molecules related by the inversion centre in (0, 0, 0) are shifted by about one half of their lengths and form a "shifted" molecular pair with a considerably greater distance of 7.43 Å. Considering these molecular neighborly relations, it seems to be justified to see the non-shifted pairs as a kind of structural units within the crystal structure. By their translations in direction of their long axes as well as along the lattice vector *a* "double sheets" are built up. These sheets on their part are stacked one upon the other in such a way that neighboring double sheets are shifted against each other in the above described manner. Thus the double sheets form an imbricated packing. The large gaps between the non-shifted molecular pairs of one double sheet in their arrangement one behind the other are filled with the laterally

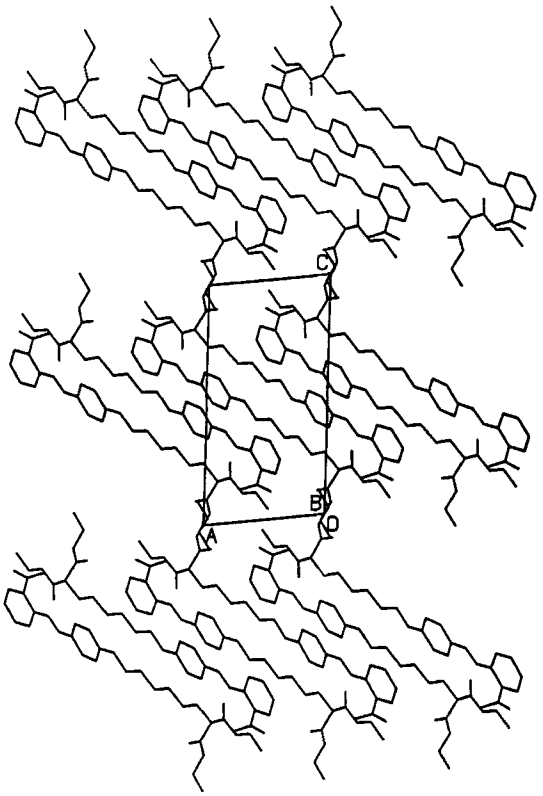

FIGURE 5 PLUTO plot[10] of the crystal packing of **2** projected along [010].

branched ethoxycarbonyl groups of molecules located in the two adjacent sheets (cf. Figure 4) thus forming an interlocked packing.

The crystal packing of **2** (Figure 5) is rather similar to that of **1**. It is also characterized by double sheets built up of pairs of non-shifted molecules, stacking of double sheets in imbricated manner and penetration of sheets by the lateral branches of neighboring sheets. The laterally substituted nitro group is not participated in this penetration because it is directed towards the inside of the double sheets.

In both structures all non-H intermolecular distances are greater than the sum of the van der Waals radii. There is no indication of other than normal van der Waals forces within the crystal lattice.

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