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Crystal and Molecular Structures of Tetraisopropyl 1,1′-(1,8-Naphthylene)-bis[1*H*-1,2,3-triazole-4,5-dicarboxylate] and Diisopropyl 1-(1-Naphthyl)-1*H*-1,2,3-triazole-4,5-dicarboxylate

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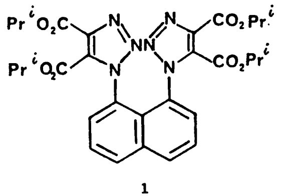
Molecular structures of the title compounds, which belong to the class of peri-substituted naphthalenes, have been determined by means of X-ray diffraction. Tetraisopropyl 1,1′-(1,8-naphthylene)bis[1*H*-1,2,3-triazole-4,5-dicarboxylate] (**1**), monoclinic, space group $C2/c$, a=35.034(3), b=9.717(1), c=18.504(1) Å, β=96.43(1)°, Z=8; Diisopropyl 1-(1-naphthyl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (**2**), monoclinic, space group $P2_1/n$, a=16.246(2), b=10.115(1), c=12.390(1) Å, β=96.67(1)°, Z=4. The dihedral angles between the mean plane of the naphthalene ring and that of the triazole ring are 88° and 85° for **1** and **2**, respectively. The naphthalene framework of **1** is much more distorted than that of **2** because of a steric repulsion between the two triazole rings with bulky ester groups of **1**.

Owing to a steric repulsion between two substituents, the naphthalene framework is distorted to some degree from a planar structure in 1,8-disubstituted naphthalenes. Among them, such a distorsion of 1,8-bis(trimethylelement) naphthalenes has been shown to be very large by an X-ray diffraction study.[1] However, there have been few X-ray analyses of compounds having aromatic rings in the 1 and 8 positions of the naphthalene moiety.[2]

Recently, we synthesized novel 1,8-bis(heteroaromatic substituted)naphthalenes, 1,1′-(1,8-naphthylene)bis[1*H*-1,2,3-triazoles], and investigated their spectroscopic properties.[3] A structure determination of the parent compound, 1,1′-(1,8-naphthylene)bis[1*H*-1,2,3-triazole], showed that the naphthalene framework in this molecule is very distorted because of a steric repulsion between the two triazole rings.[4]

In this paper, we report on the crystal and molecular structures of more overcrowding compounds, tetraisopropyl 1,1′-(1,8-naphthylene)bis[1*H*-1,2,3-triazole-4,5-dicarboxylate] (**1**), and the corresponding monotriazolylnaphthalene, diisopropyl 1-(1-naphthyl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (**2**).



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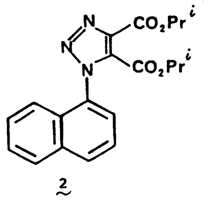

2

Experimental

Compounds **1** and **2** were prepared and purified according to a previous report.[3] Colorless needle-like crystals of **1** and **2** were obtained by a slow evaporation from diethyl ether-hexane solution at room temperature.

Crystal Data. **1**; $C_{30}H_{34}N_6O_8$, M=606.6, monoclinic, space group $C2/c$, a=35.034(3), b=9.717(1), c=18.504(1) Å, β=96.43(1)°, V=6259.7(6) Å^3, D_m=1.28 g cm^{-3} (by floatation in carbon tetrachloride and hexane), D_c=1.29 g cm^{-3} for Z=8, μ(Mo $K\alpha$)=1.02 cm^{-1}. **2**; $C_{20}H_{21}N_3O_4$, M=367.4, monoclinic, space group $P2_1/n$, a=16.246(2), b=10.115(1), c=12.390(1) Å, β=96.67(1)°, V=2022.2(3) Å^3, D_m=1.20 g cm^{-3} (by floatation in carbon tetrachloride and hexane), D_c=1.21 g cm^{-3} for Z=4, μ(Mo $K\alpha$)=0.92 cm^{-1}.

Crystallographic Measurements. The cell dimensions and diffraction intensities of both crystals were measured on a Rigaku AFC-4 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation (λ=0.71069 Å) at room temperature. The crystals, approximate dimensions of 0.8×0.3×0.1 and 0.7×0.3×0.2 mm for **1** and **2**, were used for intensity data measurements. Unit-cell dimensions of both crystals were determined by least-squares fits using 25 reflections in the range 20°<2θ<30°. Intensity data were collected by the ω–2θ scan mode at a rate of 2°(2θ) min^{-1}. The scan range (2θ) was from 2° to 55° for both crystals. Three standard reflections were measured after every 50 reflections, showing no significant intensity decay throughout the data collection for both crystals. Totals of 7793 and 5080 (including systematically absent) reflections were measured, of which 3024 and 2303 were observed ($|F_o| > 3\sigma(|F_o|)$) for **1** and **2**, respectively. The usual Lorentz and polarization corrections were applied, but no absorption corrections were made.

Structure Determination of 1. The structure was solved by direct methods using MULTAN 78.[5] An E-map using 276 ($|E| > 1.2$) revealed all the non-hydrogen atoms. Hydrogen atoms were placed at the calculated positions. The structure was refined by a block-diagonal least-squares method, using the UNICS III system.[6] Anisotropic thermal parameters were assumed for the non-hydrogen atoms, and isotropic parameters, for the hydrogen atoms. The final refinement, employing a weighting scheme of w=$(0.0024|F_o|^2-0.0156|F_o|+6.413)^{-1}$, led to R and R_w values of 0.058 and 0.056, respectively. The final atomic parameters are listed in Table 1.

Strucure Determination of 2. The solution and refinement of the structure of **2** was accomplished in the same way as that of **1**. The weighting scheme used in the final cycle of the refinement was w=$(0.0024|F_o|^2+0.054|F_o|+0.313)^{-1}$. The final R and R_w value was 0.069 and 0.067, respectively; the final atomic parameters are listed in Table 2.

Atomic scattering factors were taken from International Tables for X-Ray Crystallography.[7] All the calculations were carried out on a FACOM M-380 computer at Tsukuba Research Center.

Table 1. Final Atomic Coordinates (×10^4) and Equivalent Isotropic Temperature Factors of the Compound **1** with Estimated Standard Deviations in Parentheses $B_{eq}=4/3\sum_i\sum_j\beta_{ij}a_ia_j$

	x	y	z	B_{eq}/Å^2
O(1)	4807(1)	2494(4)	1382(2)	8.1(1)
O(2)	4283(1)	1207(4)	1190(2)	5.9(1)
O(3)	4523(1)	5128(3)	1966(2)	8.3(1)
O(4)	4358(1)	5604(3)	3063(1)	6.1(1)
O(5)	3060(1)	1532(4)	807(2)	8.8(1)
O(6)	3310(1)	−125(3)	1552(2)	5.3(1)
O(7)	3367(1)	1202(4)	3898(2)	7.2(1)
O(8)	2995(1)	222(3)	2978(1)	5.3(1)
N(1)	4267(1)	2859(3)	3371(2)	4.0(1)
N(2)	4240(1)	1470(3)	3360(2)	5.0(1)
N(3)	4326(1)	1051(3)	2728(2)	5.1(1)
N(4)	3478(1)	3411(3)	2921(1)	4.0(1)
N(5)	3535(1)	4180(4)	2336(2)	5.1(1)
N(6)	3451(1)	3413(4)	1763(2)	5.0(1)
C(1)	4201(1)	3599(4)	4025(2)	4.0(1)
C(2)	4508(1)	3713(5)	4540(2)	5.3(1)
C(3)	4471(1)	4425(5)	5196(2)	6.0(1)
C(4)	4134(2)	4968(5)	5307(2)	5.5(1)
C(5)	3456(2)	5477(5)	4917(2)	6.0(1)
C(6)	3141(2)	5404(6)	4432(3)	6.9(1)
C(7)	3162(1)	4692(6)	3767(2)	6.0(1)
C(8)	3494(1)	4090(4)	3629(2)	4.3(1)
C(9)	3837(1)	4144(4)	4117(2)	3.8(1)
C(10)	3808(1)	4873(4)	4784(2)	4.5(1)
C(11)	4417(1)	2151(4)	2334(2)	4.2(1)
C(12)	4386(1)	3322(4)	2733(2)	4.0(1)
C(13)	4531(1)	1980(4)	1586(2)	4.7(1)
C(14)	4367(2)	871(6)	441(3)	6.7(1)
C(15)	3998(2)	728(9)	−1(3)	10.6(1)
C(16)	4608(3)	−334(11)	483(4)	15.7(1)
C(17)	4433(1)	4788(4)	2540(2)	4.8(1)
C(18)	4353(2)	7112(4)	2938(3)	6.4(2)
C(19)	3969(2)	7604(5)	3157(4)	9.1(1)
C(20)	4689(2)	7668(6)	3412(4)	10.3(2)
C(21)	3337(1)	2163(4)	1970(2)	4.1(2)
C(22)	3349(1)	2139(4)	2719(2)	3.9(2)
C(23)	3215(1)	1151(5)	1386(2)	5.2(2)
C(24)	3240(2)	−1156(5)	968(3)	6.9(2)
C(25)	3510(3)	−2310(8)	1201(5)	13.1(3)
C(26)	2821(2)	−1558(8)	888(4)	10.8(3)
C(27)	3243(1)	1143(4)	3270(2)	4.7(3)
C(28)	2868(1)	−841(5)	3463(3)	6.1(3)
C(29)	2733(2)	−2045(6)	2962(4)	8.4(3)
C(30)	2554(2)	−289(7)	3848(4)	10.4(4)

Table 2. Final Atomic Coordinates (×10^4) and Equivalent Isotropic Temperature Factors of the Compound **2** with Estimated Standard Deviations in Parentheses $B_{eq}=4/3\sum_i\sum_j\beta_{ij}a_ia_j$

	x	y	z	B_{eq}/Å^2
O(1)	−2016(2)	440(3)	5826(3)	7.5(1)
O(2)	−1803(2)	2549(3)	6264(3)	6.1(1)
O(3)	−702(2)	−1507(3)	5208(2)	6.3(1)
O(4)	−185(2)	−2284(2)	6840(2)	5.9(1)
N(1)	490(2)	291(3)	7225(2)	3.7(1)
N(2)	486(2)	1582(3)	7522(3)	4.4(1)
N(3)	−235(2)	2072(3)	7161(3)	4.4(1)
C(1)	1218(2)	−497(3)	7558(3)	3.9(1)
C(2)	1855(2)	−458(4)	6945(3)	5.0(1)
C(3)	2582(3)	−1175(5)	7308(4)	6.5(1)
C(4)	2634(2)	−1883(4)	8232(4)	6.2(1)
C(5)	2003(3)	−2704(5)	9836(4)	6.9(1)
C(6)	1366(4)	−2732(5)	10429(4)	7.7(2)
C(7)	637(3)	−2014(5)	10098(4)	6.6(1)
C(8)	582(2)	−1276(4)	9174(3)	4.9(1)
C(9)	1233(2)	−1229(3)	8534(3)	4.0(1)
C(10)	1975(2)	−1954(4)	8870(3)	5.0(1)
C(11)	−699(2)	1112(3)	6618(3)	3.8(1)
C(12)	−238(2)	−29(3)	6641(3)	3.5(1)
C(13)	−1575(2)	1308(4)	6184(3)	4.4(1)
C(14)	−2683(3)	2860(5)	5943(4)	7.2(2)
C(15)	−2740(5)	3613(10)	4941(7)	14.0(1)
C(16)	−2963(5)	3667(13)	6839(7)	16.4(2)
C(17)	−408(2)	−1360(3)	6126(3)	4.1(3)
C(18)	−226(4)	−3673(4)	6467(5)	8.1(5)
C(19)	587(5)	−4056(8)	6232(10)	15.1(4)
C(20)	−410(10)	−4390(7)	7472(10)	19.9(6)

Results and Discussion

Molecular Structure. The molecular structures and the atom labelings of **1** and **2** are presented by an ORTEP[8] drawing in Fig. 1. The selected bond distances and angles of **1** and **2** are given in Tables 3 and 4, respectively.[9]

The deviations of the each atom of the triazole rings from the mean plane are within 0.01 Å in both **1** and **2**, which suggests that they are almost planar due to an aromatic ring system. The dihedral angles between the mean plane of the naphthalene ring and those of two triazole rings in **1** are 87.1(1)° for the one triazole ring and 89.3(1)° for the other. In the case of **2**, the angle is 85.1(1)°, which is slightly smaller than that in **1**. The angles are nearly ninety degrees while in the parent compounds, 1,1′-(1,8-naphthylene)bis[1*H*-1,2,3-

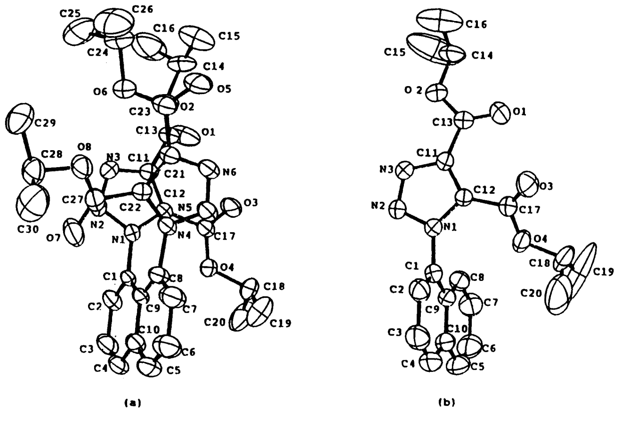

Fig. 1. Molecular structures. Non-hydrogen atoms are expressed as thermal ellipsoids with 40% probability level. Hydrogen atoms are omitted for clarity. (a) Compound **1**. (b) Compound **2**.

Table 3. Selected Bond Lengths and Bond Angles in **1** with Estimated Standard Deviations in Parentheses

Bond length l/Å				Bond angle ϕ/°			
C(1)-C(2)	1.361(5)	C(1)-C(9)	1.407(6)	C(2)-C(1)-C(9)	122.8(3)	C(2)-C(1)-N(1)	115.9(4)
C(1)-N(1)	1.449(5)	C(2)-C(3)	1.415(6)	C(9)-C(1)-N(1)	121.3(3)	C(1)-C(2)-C(3)	119.9(4)
C(3)-C(4)	1.332(7)	C(4)-C(10)	1.416(6)	C(2)-C(3)-C(4)	119.6(4)	C(3)-C(4)-C(10)	122.1(4)
C(5)-C(6)	1.345(7)	C(5)-C(10)	1.410(7)	C(6)-C(5)-C(10)	122.0(4)	C(5)-C(6)-C(7)	119.0(5)
C(6)-C(7)	1.421(7)	C(7)-C(8)	1.350(7)	C(6)-C(7)-C(8)	120.3(4)	C(7)-C(8)-C(9)	123.2(3)
C(8)-C(9)	1.423(5)	C(8)-N(4)	1.462(4)	C(7)-C(8)-N(4)	114.7(3)	C(9)-C(8)-N(4)	122.0(4)
C(9)-C(10)	1.437(5)	C(11)-C(12)	1.367(5)	C(1)-C(9)-C(8)	128.5(3)	C(1)-C(9)-C(10)	116.3(3)
C(11)-C(13)	1.491(5)	C(11)-N(3)	1.352(5)	C(8)-C(9)-C(10)	115.2(4)	C(4)-C(10)-C(5)	120.5(4)
C(12)-C(17)	1.482(5)	C(12)-N(1)	1.371(5)	C(4)-C(10)-C(9)	119.3(4)	C(5)-C(10)-C(9)	120.2(4)
C(13)-O(1)	1.188(6)	C(13)-O(2)	1.310(5)	C(12)-C(11)-C(13)	129.8(4)	C(12)-C(11)-N(3)	109.2(3)
C(17)-O(3)	1.190(5)	C(17)-O(4)	1.300(5)	C(13)-C(11)-N(3)	121.0(3)	C(11)-C(12)-C(17)	130.7(3)
C(21)-C(22)	1.382(5)	C(21)-C(23)	1.488(6)	C(11)-C(12)-N(1)	104.0(3)	C(17)-C(12)-N(1)	125.1(3)
C(21)-N(6)	1.348(5)	C(22)-C(27)	1.483(5)	C(11)-C(13)-O(1)	124.1(4)	C(11)-C(13)-O(2)	110.8(4)
C(22)-N(4)	1.355(5)	C(23)-O(5)	1.204(5)	O(1)-C(13)-O(2)	125.1(4)	C(12)-C(17)-O(3)	122.0(4)
C(23)-O(6)	1.312(5)	C(27)-O(7)	1.197(5)	C(12)-C(17)-O(4)	111.8(3)	O(3)-C(17)-O(4)	126.2(4)
C(27)-O(8)	1.320(5)	N(1)-N(2)	1.353(4)	C(22)-C(21)-C(23)	133.8(4)	C(22)-C(21)-N(6)	108.9(3)
N(2)-N(3)	1.306(5)	N(4)-N(5)	1.349(4)	C(23)-C(21)-N(6)	117.3(3)	C(21)-C(22)-C(27)	135.9(4)
N(5)-N(6)	1.303(4)			C(21)-C(22)-N(4)	103.5(3)	C(27)-C(22)-N(4)	120.5(3)
				C(21)-C(23)-O(5)	120.5(4)	C(21)-C(23)-O(6)	114.3(3)
				O(5)-C(23)-O(6)	125.1(4)	C(22)-C(27)-O(7)	123.0(4)
				C(22)-C(27)-O(8)	111.5(3)	O(7)-C(27)-O(8)	125.6(4)

Table 4. Selected Bond Lengths and Bond Angles in **2** with Estimated Standard Deviations in Parentheses

Bond length l/Å				Bond angle ϕ/°			
C(1)-C(2)	1.355(5)	C(1)-C(9)	1.416(5)	C(2)-C(1)-C(9)	123.8(3)	C(2)-C(1)-N(1)	118.5(3)
C(1)-N(1)	1.445(4)	C(2)-C(3)	1.415(6)	C(9)-C(1)-N(1)	117.8(3)	C(1)-C(2)-C(3)	118.2(4)
C(3)-C(4)	1.345(7)	C(4)-C(10)	1.405(6)	C(2)-C(3)-C(4)	120.7(4)	C(3)-C(4)-C(10)	122.1(4)
C(5)-C(6)	1.338(8)	C(5)-C(10)	1.413(6)	C(6)-C(5)-C(10)	121.8(4)	C(5)-C(6)-C(7)	120.5(5)
C(6)-C(7)	1.409(8)	C(7)-C(8)	1.361(6)	C(6)-C(7)-C(8)	119.9(5)	C(7)-C(8)-C(9)	121.1(4)
C(8)-C(9)	1.394(5)	C(9)-C(10)	1.431(5)	C(1)-C(9)-C(8)	124.3(3)	C(1)-C(9)-C(10)	116.6(3)
C(11)-C(12)	1.374(4)	C(11)-C(13)	1.475(5)	C(8)-C(9)-C(10)	119.1(3)	C(4)-C(10)-C(5)	123.6(4)
C(11)-N(3)	1.358(4)	C(12)-C(17)	1.502(5)	C(4)-C(10)-C(9)	118.8(4)	C(5)-C(10)-C(9)	117.7(4)
C(12)-N(1)	1.352(4)	C(13)-O(1)	1.186(5)	C(12)-C(11)-C(13)	128.4(3)	C(12)-C(11)-N(3)	108.7(3)
C(13)-O(2)	1.316(5)	C(17)-O(3)	1.191(4)	C(13)-C(11)-N(3)	122.8(3)	C(11)-C(12)-C(17)	132.2(3)
C(17)-O(4)	1.309(4)			C(11)-C(12)-N(1)	104.6(3)	C(17)-C(12)-N(1)	123.2(3)
				C(11)-C(13)-O(1)	123.5(3)	C(11)-C(13)-O(2)	111.6(3)
				O(1)-C(13)-O(2)	124.8(3)	C(12)-C(17)-O(3)	123.4(3)
				C(12)-C(17)-O(4)	109.3(3)	O(3)-C(17)-O(4)	127.4(3)

triazole] and 1-(1-naphthyl)-1*H*-1,2,3-triazole, the corresponding angles are 50.6(2)° and 56.8(2)°, respectively.[10] These results indicate that the repulsion between the isopropoxycarbonyl group in the 5-position of the triazole ring and the hydrogen in the 2′-position of the naphthalene ring is very large. The two triazole rings of **1** face each other with a dihedral angle of 33.1(2)°, and assume a trans conformation.

The fusion angle, C1-C9-C8, is 129.0(4)° in **1**, while the angle in **2** is 124.3(3)°. The enlargement of this angle in **1** indicates that a severe steric strain is caused by a repulsion between two triazole rings with bulky ester groups. The angles C9-C1-N1 and C9-C8-N4 in **1**, which are related to the pivot bond between the triazole ring and the naphthalene ring, are 121.3(3)° and 122.0(4)°, respectively. These are large compared with that in **2** (117.8(3)°), also suggesting a large repulsion between the two triazole rings of **1**. Similar relationships have been observed for the parent compounds.[10]

Table 5. The Equations of the Naphthalene Least Squares Planes and the Deviations of the Atoms for the Compounds **1** and **2** (l/Å)

Compound **1**	$-0.3093X-0.8509Y+0.4566Z+4.1135=0$
Compound **2**	$-0.3158X-0.7927Y-0.4814Z+4.7353=0$

	Compound **1**	Compound **2**
C(1)	−0.012	0.002
C(2)	−0.005	0.009
C(3)	0.000	−0.005
C(4)	0.011	−0.015
C(5)	−0.005	0.012
C(6)	−0.013	0.007
C(7)	−0.009	0.002
C(8)	0.013	−0.010
C(9)	0.009	0.000
C(10)	0.001	−0.001

The equations of the least-squares planes of the naphthalene ring and the deviations of the atoms are listed in Table 5. The planarity of the naphthalene ring in **1** is as good as that in **2**.

In **2**, the torsion angles C12-C11-C13-O1, 3.6(6)°, and C11-C12-C17-O3, 44.5(6)°, show that the carbonyl

Table 6. Distortions of the Naphthalene Framework in 1,8-Disubstituted Naphthalenes

	Distance (Å)		Bond angle (°)		Torsion angle (°)		Ref.
	C1-C8	Z1-Z8	C1-C9-C8	C9-C1-Z1	C10-C9-C1-Z1	C1-C9-C10-C4	
1	2.55	2.85	128.5	121.7	179.0	0.9	a)
1,1′-(1,8-Naphthylene)-bis[1*H*-1,2,3-triazole]	2.56	2.91	129.0	120.6	165.9	7.7	b)
1,8-Diphenylnaphthalene	2.56	2.99	126.2	125.2	178.0	6.3	c)
1,8-Dipiperidinonaphthalene	2.60	2.89	126.8	120.6	167.5	9.9	d)
1,8-Bis(dimethylamino)naphthalene	2.56	2.79	125.8	120.5	168.2	9.7	e)
1,8-Di-*t*-butylnaphthalene	2.62	3.86	129.9	125.7	138.1	19.3	f)

a) This work. b) Ref. 4. c) Calculated with Ref. 11. d) Calculated with Ref. 12. e) Calculated with Ref. 13. f) Calculated with Ref. 14.

group at the 4-position exists in the triazole plane, but that at the 5-position it twists from the triazole plane. Because the latter group is situated between the ester group at the 4-position and the naphthalene ring, coplanarity cannot be maintained owing to severe steric overcrowding. In the case of **1**, the arrangements of four carbonyl groups and two triazole rings are more complicated than those of **2**. The torsion angles C12-C11-C13-O1, C11-C12-C17-O3, C22-C21-C23-O5, and C21-C22-C27-O7 are 49.1(7)°, −2.4(7)°, 144.8(4)°, and 160.5(5)°, respectively. Although the results reflect the requirements of intramolecular contacts, the influence of intermolecular packing can not be ignored.

Comparison with Other Peri-Substituted Naphthalenes. Typical data regarding the effects of the distorsion on the naphthalene ring in **1** are listed in Table 6 along with data for the other peri-substituted naphthalenes. In this Table, Z1 and Z8 are the atoms in substituents and there bound to C1 and C8, respectively. In order to avoid any steric overcrowding of two bulky peri-substituents, the nonbonding distance between C1 and C8 as well as that between Z1 and Z8 is enlarged. Accordingly, fused angle C1-C9-C8 expands more than 120°, and C1 and C8 are displaced in opposite directions from the naphthalene ring with torsion angle C1-C9-C10-C4 not equal to 0°. In order to lengthen the distance between Z1 and Z8, angle C9-C1-Z1 expands more than 120°, and torsion angle C10-C9-C1-Z1 becomes less than 180°.

Considering the data in Table 6, there are various modes in which the strain of bulky substituents affect the naphthalene framework. The distorsion of the naphthalene ring in 1,3,6,8-tetra-*t*-butylnaphthalene is remarkably large because of severe steric overcrowding. Compared with other peri-substituted naphthalenes, the displacement of C1 and C8 from the naphthalene ring of **1** is very small. However, the two angles, C1-C9-C8 and C9-C1-Z1, are significantly large. Therefore, it may be pointed out that the above-described distorsions in **1** relieve the severe steric repulsion between the two triazole rings with bulky ester groups.

Crystal Structure. The packing modes of molecules in both crystals are shown in Fig. 2. The crystal structures are stabilized mainly by van der Waals forces; the shortest intermolecular distances are 3.209(5) Å for O(1) (x,y,z)-C(12) $(1-x,y,0.5-z)$ and 3.286(4) Å for O(3) (x,y,z)-C(12) $(-x,-y,1-z)$ in **1** and **2**, respectively.

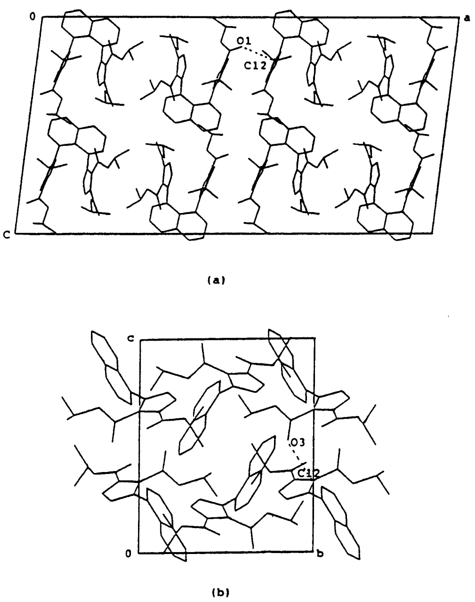

Fig. 2. Crystal structures. Hydrogen atoms are omitted for clarity. (a) Compound **1**, projected along the b axis. (b) Compound **2**, projected along the a* axis.

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