and 0·091 (1) Å from the relevant benzene planes. The steric repulsion is also apparent from the bond angles about the thioacetal C atom C7 where the S—C—S angle of 108·10 (9)° is smaller than the C—C—S angle, mean value 113·8 (1)°. The mean S—C_{sp^3} distance [1·828 (2) Å] and the mean S—$C_{\rm arom}$ distance [1·777 (2) Å] are in agreement with those reported previously, *e.g.* 1·826 (3) and 1·776 (3) Å for these two bonds in tetraphenyl orthothiocarbonate (Kato, 1972). The S—$C_{\rm arom}$ bond length of 1·777 (2) Å is also in accord with the average value of 1·773 Å reported for phenyl sulfides (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987).

The bond angles at sulfur [average 102·9 (1)°] are less than tetrahedral as is usually found in simple sulfides. The various C—C bond lengths are in accord with the anticipated values. There are no untoward intermolecular contacts.

We are indebted to the NSERC Canada for the award of an operating grant (to GF) and a Visiting Professorship (to BK). We thank the Irish Department of Education and UCC for a postgraduate studentship (to BMD).

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Structure of (*E*)-2-Methyl-1,3-bis(2,4,5-trimethoxyphenyl)-1-pentene and 1-(2,4,5-Trimethoxyphenyl)-2-methyl-3-ethyl-4,6,7-trimethoxyindan* $C_{24}H_{32}O_6$: Two Asarone Dimers†

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(*Received* 24 *April* 1989; *accepted* 7 *November* 1989)

Abstract. (*E*)-2-Methyl-1,3-bis(2,4,5-trimethoxyphenyl)-1-pentene, $C_{24}H_{32}O_6$, isomer (1), $M_r = 416·2$, monoclinic, $P2_1/n$, $a = 7·082$ (3), $b = 11·954$ (7), $c = 27·136$ (17) Å, $\beta = 94·14$ (4)°, $V = 2291$ (2) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·8$ cm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0·056$, $wR = 0·063$ for 2901 (71·4%) reflections with $F > 3\sigma(F)$. 1-(2,4,5-Trimethoxyphenyl)-2-methyl-3-ethyl-4,6,7-trimethoxyindan, isomer (2), $C_{24}H_{32}O_6$, $M_r = 416·2$, monoclinic, $P2_1/a$, $a = 17·281$ (5), $b = 7·701$ (1), $c = 18·057$ (6) Å, $\beta = 108·23$ (2)°, $V = 2282$ (1) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 6·7$ cm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0·045$, $wR = 0·061$ for 2601 (90·3%) reflections with $F > 3\sigma(F)$. The X-ray structures of (1) and (2) confirm the structures previously assigned on the basis of chemical and NMR spectral evidence. Isomer (1) is non-planar. In isomer (2) the five-membered ring adopts an envelope conformation and the substituents at C(1) and C(3) are antiperiplanar to the methyl group at C(2). In both isomers the orientation of the trimethoxyphenyl substituent is determined by C—H···O intramolecular interactions. The packing in the crystal is entirely due to van der Waals forces.

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* IUPAC name: 1-ethyl-4,5,7-trimethoxy-2-methyl-3-(2,4,5-trimethoxyphenyl)indan.

† Contribution No. 978 of the Instituto de Química, UNAM.

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0108-2701/90/081542-04$03.00

Table 1. *Experimental details*

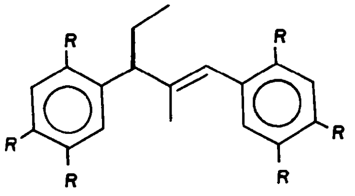
	Isomer (1) (...pentene)	Isomer (2) (...indan)
Reflections for cell determination, number/2θ range (°)	$25/6{\cdot}91 \leq 2\theta \leq 18{\cdot}67$	$25/13{\cdot}34 \leq 2\theta \leq 49{\cdot}19$
Radiation	Mo $K\alpha$ Graphite-monochromated	Cu $K\alpha$ Ni-filtered
Max. 2θ(°)/octants	$50/h, k, \pm l$	$110/h, k \pm l$
Number of data collected* (including standards)/unique intensities	4861/4063 ($h_{\max} = 8$, $k_{\max} = 14$, $l_{\max} = 32$)	3481/2879 ($h_{\max} = 18$, $k_{\max} = 10$, $l_{\max} = 20$)
Structure solution	Direct methods (*SHELXTL*)	Direct methods (*SHELXTL*)
Variables/number of data in least squares	271/2901 [$F > 3\sigma(F)$]	271/2601 [$F > 3\sigma(F)$]
Final R (wR)	0·0562 (0·0629)	0·0451 (0·0608)
Max. Δ/σ in final cycle	0·044	0·061
Factor used in weighting scheme (g)†	0·002	0·00239
Max./min. $\Delta\rho$ (e Å^{-3})	0·28/−0·25	0·17/−0·23

* Nicolet $R3$ diffractometer, $2\theta:\theta$ scans, scan rate variable (3·89–29·3° min^{-1}), 2 standard reflections every 50 measurements (no significant variation with time). Lorentz and polarization corrections applied to all data collected.

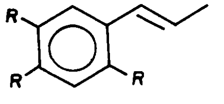
† $w = [\sigma^2(F) + gF^2]^{-1}$.

Introduction. As part of our studies directed towards the total synthesis of asarone-like hypocholestoremic agents, we became interested in the work of Szeki (Szeki, 1906; Haraszti & Szeki, 1933), who claimed the isolation of an asarone dimer possessing the cyclobutane structure (4), from the direct hydrogen chloride treatment of an ether solution of asarone (3). Kovacs (1950) subsequently proposed the indan-derived structure (2) for a new asarone dimer. No spectral evidence was given to support structure (2). Later, we reported (Lemini, Cruz & Sánchez, 1981) the synthesis of a novel asarone dimer and the dimer described by Kovacs. On the basis of spectral and analytical evidence we proposed structure (1) for the new dimer. Furthermore, we found that dimer (1) isomerizes to (2).

X-ray analyses of (1) and (2) were undertaken to confirm structural assignments made mainly by NMR and to establish unambiguously the stereochemical relationships.

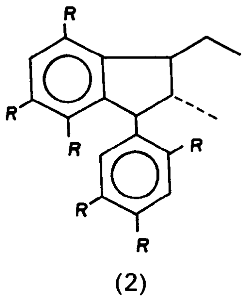


(1)

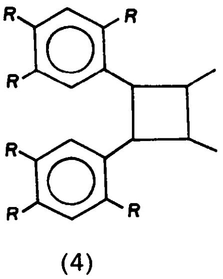


(3)

R =-OMe



(2)



(4)

Experimental. Isomer (1) was prepared as described elsewhere (Lemini, Cruz & Sánchez, 1981). Crystallization from methanol gave prismatic colorless crystals, m.p. 369–370 K. Crystal of overall dimensions 0·39 × 0·40 × 0·5 mm used throughout. Isomer (2) was prepared by prolonged treatment of isomer (1) with PBr_3 (Lemini *et al.*, 1981). Crystallization from methanol gave colorless plate-like crystals, m.p. 373–374 K. Crystal of overall dimensions 0·64 × 0·66 × 0·16 mm used throughout. The work on the diffractometer gave the experimental results summarized in Table 1. The space group in both cases was uniquely determined from systematic absences [isomer (1): $R_{\rm merge} = 0{\cdot}028$, $R_{\rm sigma} = 0{\cdot}034$; isomer (2): $R_{\rm merge} = 0{\cdot}014$, $R_{\rm sigma} = 0{\cdot}015$]. Both structures were solved by direct methods and refined by block-cascade least-squares methods. H atoms were first located in ΔF maps before the positions of the CH, CH_2 and CH_3 H atoms were idealized. In both isomers methyl groups were refined as rigid groups. Least-squares refinement minimizing $\sum w\Delta F^2$ [all non-H atoms anisotropic and fixed isotropic ($U = 0{\cdot}06$ Å^2) for H] converged to the indicated agreement indices. Calculations on Nova 4S computer using programs in *SHELXTL* (Sheldrick, 1978). Scattering factors stored in programs were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters, bond lengths and bond angles for both isomers are given in Tables 2–5, respectively.*

Discussion. In all essential details, the molecular geometry of both isomers in terms of the bond lengths and angles show normal values. Isomer (1):

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52765 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for isomer* (1)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	*y*	*z*	U_{eq}
C(1)	−1499 (3)	2976 (2)	2083 (1)	34 (1)
C(2)	−1959 (3)	2300 (2)	2445 (1)	30 (1)
C(3)	−2853 (3)	2774 (2)	2898 (1)	32 (1)
C(4)	−4880 (3)	2316 (2)	2933 (1)	43 (1)
C(5)	−6242 (3)	2731 (2)	2513 (1)	57 (2)
C(6)	−1602 (4)	1057 (2)	2455 (1)	44 (1)
C(7)	−573 (3)	2665 (2)	1629 (1)	36 (1)
C(8)	824 (3)	3356 (2)	1459 (1)	38 (1)
C(9)	1859 (3)	3024 (2)	1065 (1)	43 (1)
C(10)	1476 (3)	2015 (2)	828 (1)	43 (1)
C(11)	−15 (3)	1352 (2)	966 (1)	44 (1)
C(12)	−1007 (3)	1687 (2)	1364 (1)	40 (1)
C(13)	−1560 (3)	2579 (2)	3366 (1)	32 (1)
C(14)	−1804 (3)	1677 (2)	3684 (1)	37 (1)
C(15)	−568 (3)	1475 (2)	4094 (1)	40 (1)
C(16)	968 (3)	2195 (2)	4194 (1)	39 (1)
C(17)	1210 (3)	3116 (2)	3895 (1)	39 (1)
C(18)	−46 (3)	3301 (2)	3482 (1)	37 (1)
O(1)	1154 (2)	4346 (1)	1706 (1)	50 (1)
C(19)	2642 (4)	5035 (2)	1552 (1)	63 (1)
O(2)	2470 (3)	1606 (2)	452 (1)	61 (1)
C(20)	4083 (4)	2215 (3)	324 (1)	75 (1)
O(3)	−338 (3)	390 (2)	701 (1)	67 (1)
C(21)	−1970 (4)	−223 (3)	774 (2)	81 (1)
O(4)	−697 (3)	601 (2)	4418 (1)	59 (1)
C(22)	−2284 (4)	−106 (3)	4358 (1)	73 (1)
O(5)	2164 (2)	1924 (2)	4596 (1)	56 (1)
C(23)	3830 (4)	2562 (3)	4689 (1)	74 (1)
O(6)	105 (2)	4199 (1)	3172 (1)	51 (1)
C(24)	1578 (4)	4979 (2)	3282 (1)	71 (1)

Table 3. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for isomer* (2)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	*y*	*z*	U_{eq}
C(1)	9004 (1)	5448 (2)	2570 (1)	43 (1)
C(2)	9943 (1)	5476 (3)	2726 (1)	47 (1)
C(3)	10100 (1)	4054 (3)	2183 (1)	47 (1)
C(3a)	9313 (1)	4083 (3)	1512 (1)	44 (1)
C(4)	9162 (1)	3551 (3)	752 (1)	50 (1)
C(5)	8386 (1)	3696 (3)	223 (1)	52 (1)
C(6)	7760 (1)	4387 (2)	463 (1)	47 (1)
C(7)	7898 (1)	4925 (2)	1224 (1)	44 (1)
C(7a)	8685 (1)	4786 (2)	1747 (1)	41 (1)
C(8)	10216 (1)	7246 (3)	2529 (1)	64 (1)
C(9)	10275 (1)	2253 (3)	2553 (1)	54 (1)
C(10)	11053 (2)	2120 (4)	3227 (1)	72 (1)
O(1)	9822 (1)	2878 (2)	572 (1)	69 (1)
C(11)	9747 (2)	2553 (4)	−213 (1)	84 (1)
O(2)	6972 (1)	4575 (2)	−21 (1)	65 (1)
C(12)	6834 (2)	4518 (4)	−832 (1)	79 (1)
O(3)	7302 (1)	5742 (2)	1455 (1)	56 (1)
C(13)	6711 (2)	4624 (4)	1595 (2)	82 (1)
C(14)	8740 (1)	4455 (2)	3175 (1)	41 (1)
C(15)	8850 (1)	5183 (3)	3899 (1)	47 (1)
C(16)	8613 (1)	4316 (3)	4469 (1)	49 (1)
C(17)	8270 (1)	2677 (3)	4318 (1)	46 (1)
C(18)	8169 (1)	1906 (2)	3601 (1)	41 (1)
C(19)	8396 (1)	2793 (2)	3037 (1)	41 (1)
O(4)	9215 (1)	6797 (2)	4009 (1)	66 (1)
C(20)	9300 (2)	7689 (3)	4718 (1)	70 (1)
O(5)	8008 (1)	1737 (2)	4838 (1)	60 (1)
C(21)	8108 (2)	2474 (4)	5573 (1)	71 (1)
O(6)	7818 (1)	275 (2)	3502 (1)	55 (1)
C(22)	7702 (2)	−548 (3)	2782 (1)	64 (1)

Table 4. *Bond lengths* (Å) *and angles* (°) *for isomer* (1); *e.s.d.'s are given in parentheses*

C(1)—C(2)	1·330 (3)	C(1)—C(7)	1·484 (3)
C(2)—C(3)	1·531 (3)	C(2)—C(6)	1·507 (3)
C(3)—C(4)	1·546 (3)	C(3)—C(13)	1·530 (3)
C(4)—C(5)	1·522 (4)	C(7)—C(8)	1·393 (3)
C(7)—C(12)	1·396 (3)	C(8)—C(9)	1·398 (3)
C(8)—O(1)	1·371 (3)	C(9)—C(10)	1·384 (3)
C(10)—C(11)	1·393 (3)	C(10)—O(2)	1·372 (3)
C(11)—C(12)	1·388 (4)	C(11)—O(3)	1·368 (3)
C(13)—C(14)	1·399 (3)	C(13)—C(18)	1·395 (3)
C(14)—C(15)	1·386 (3)	C(15)—C(16)	1·398 (3)
C(15)—O(4)	1·373 (3)	C(16)—C(17)	1·387 (3)
C(16)—O(5)	1·369 (3)	C(17)—C(18)	1·397 (3)
C(18)—O(6)	1·373 (3)	O(1)—C(19)	1·423 (3)
O(2)—C(20)	1·419 (4)	O(3)—C(21)	1·395 (4)
O(4)—C(22)	1·406 (4)	O(5)—C(23)	1·413 (4)
O(6)—C(24)	1·415 (3)		

C(2)—C(1)—C(7)	127·3 (2)	C(1)—C(2)—C(3)	120·3 (2)
C(1)—C(2)—C(6)	124·3 (2)	C(3)—C(2)—C(6)	115·4 (2)
C(2)—C(3)—C(4)	111·0 (2)	C(2)—C(3)—C(13)	110·7 (2)
C(4)—C(3)—C(13)	113·4 (2)	C(3)—C(4)—C(5)	112·3 (2)
C(1)—C(7)—C(8)	119·6 (2)	C(1)—C(7)—C(12)	123·0 (2)
C(8)—C(7)—C(12)	117·5 (2)	C(7)—C(8)—C(9)	120·6 (2)
C(7)—C(8)—O(1)	116·7 (2)	C(9)—C(8)—O(1)	122·6 (2)
C(8)—C(9)—C(10)	120·4 (2)	C(9)—C(10)—C(11)	119·8 (2)
C(9)—C(10)—O(2)	124·2 (2)	C(11)—C(10)—O(2)	115·9 (2)
C(10)—C(11)—C(12)	118·8 (2)	C(10)—C(11)—O(3)	115·9 (2)
C(12)—C(11)—O(3)	125·3 (2)	C(7)—C(12)—C(11)	122·5 (2)
C(3)—C(13)—C(14)	122·4 (2)	C(3)—C(13)—C(18)	119·8 (2)
C(14)—C(13)—C(18)	117·7 (2)	C(13)—C(14)—C(15)	122·0 (2)
C(14)—C(15)—C(16)	119·1 (2)	C(14)—C(15)—O(4)	125·4 (2)
C(16)—C(15)—O(4)	115·5 (2)	C(15)—C(16)—C(17)	120·1 (2)
C(15)—C(16)—O(5)	115·9 (2)	C(17)—C(16)—O(5)	123·9 (2)
C(16)—C(17)—C(18)	119·8 (2)	C(13)—C(18)—C(17)	121·2 (2)
C(13)—C(18)—O(6)	116·0 (2)	C(17)—C(18)—O(6)	122·8 (2)
C(8)—O(1)—C(19)	117·4 (2)	C(10)—O(2)—C(20)	117·7 (2)
C(11)—O(3)—C(21)	118·4 (2)	C(15)—O(4)—C(22)	118·2 (2)
C(16)—O(5)—C(23)	118·4 (2)	C(18)—O(6)—C(24)	118·4 (2)

The molecule has three planar regions, the alkene group and the trimethoxyphenyl groups (Fig. 1). The conformation of these groups around C(1)—C(7) and C(3)—C(13) is stabilized by two intramolecular C—H···O interactions involving C(1) and C(3) which are at 2·795 (8) and 2·761 (8) Å from O(1) and O(6) [*D*—H···*A* angles 92 (1) and 101 (1)°], respectively. The dihedral angles between these phenyl rings and the C(1)—C(2) double bond are 41·0 (7) and 79·6 (7)°, and 74·7 (7)° between the two rings. Isomer (2) (Fig. 2): The five-membered ring adopts a distorted envelope conformation with C(2) 0·47 Å from the best mean plane and puckering constants Δ = 16 (1)° and φ_m = 30° (Altona, Geise & Romers, 1968) and asymmetry parameter ΔC_s = 6·22 (Duax, Weeks & Rohrer, 1976). The ethyl group at C(3), τ_1 [C(8)—C(2)—C(3)—C(9)] = 149·8 (2)°, and the trimethoxyphenyl group at C(1), τ_2 [C(14)—C(1)—C(2)—C(8)] = −140·8 (2)° have an antiperiplanar relation with the methyl group at C(2). An intramolecular C—H···O interaction involving C(1) and O(4) [2·715 (8) Å; 99 (1)°] stabilizes the orientation of the trimethoxyphenyl group around the C(1)—C(14) bond. The angle to the plane of the indan moiety is 100·9 (7)°. The methoxy group attached to C(7) is

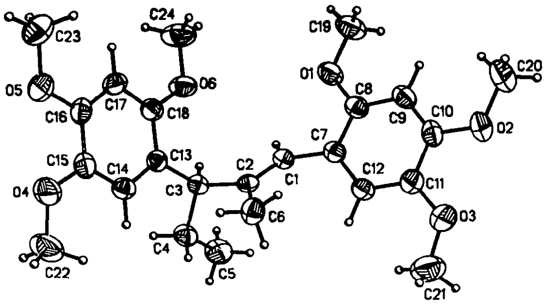

Fig. 1. The molecular structure of isomer (1) showing the atom labelling. The thermal ellipsoids are drawn at 50% probability level.

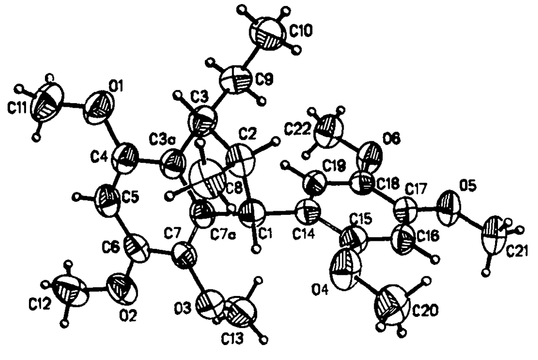

Fig. 2. View of isomer (2).

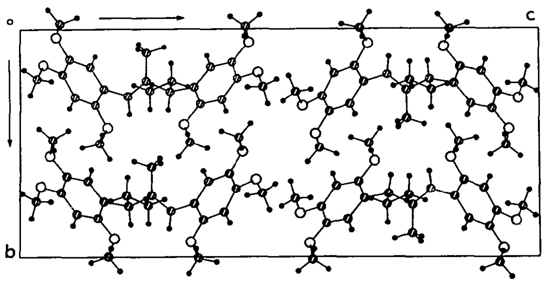

Fig. 3. The crystal packing in isomer (1) viewed along **a**.

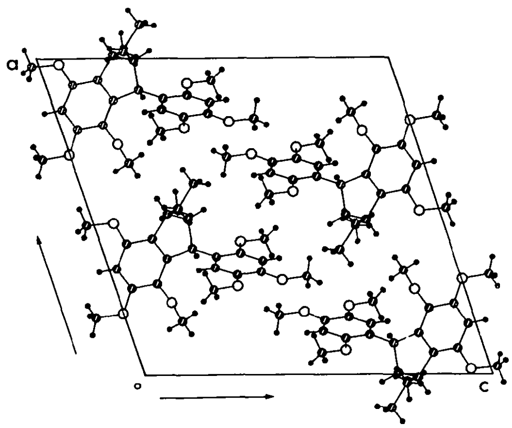

Fig. 4. The crystal packing in isomer (2) viewed along **b**.

Table 5. *Bond lengths* (Å) *and angles* (°) *for isomer* (2); *e.s.d.'s are given in parentheses*

C(1)—C(2)	1·558 (3)	C(1)—C(7a)	1·502 (3)
C(1)—C(14)	1·517 (3)	C(2)—C(3)	1·550 (3)
C(2)—C(8)	1·521 (3)	C(3)—C(3a)	1·512 (2)
C(3)—C(9)	1·528 (3)	C(3a)—C(4)	1·376 (3)
C(3a)—C(7a)	1·392 (3)	C(4)—C(5)	1·386 (3)
C(4)—O(1)	1·381 (3)	C(5)—C(6)	1·391 (3)
C(6)—C(7)	1·383 (3)	C(6)—O(2)	1·377 (2)
C(7)—C(7a)	1·395 (2)	C(7)—O(3)	1·379 (3)
C(9)—C(10)	1·508 (3)	O(1)—C(11)	1·405 (3)
O(2)—C(12)	1·408 (3)	O(3)—C(13)	1·418 (3)
C(14)—C(15)	1·379 (3)	C(14)—C(19)	1·401 (3)
C(15)—C(16)	1·391 (3)	C(15)—O(4)	1·380 (2)
C(16)—C(17)	1·384 (3)	C(17)—C(18)	1·386 (3)
C(17)—O(5)	1·370 (3)	C(18)—C(19)	1·381 (3)
C(18)—O(6)	1·381 (2)	O(4)—C(20)	1·420 (3)
O(5)—C(21)	1·402 (3)	O(6)—C(22)	1·404 (3)

C(2)—C(1)—C(7a)	102·9 (2)	C(2)—C(1)—C(14)	114·0 (1)
C(7a)—C(1)—C(14)	116·2 (2)	C(1)—C(2)—C(3)	104·8 (1)
C(1)—C(2)—C(8)	110·6 (2)	C(3)—C(2)—C(8)	110·9 (2)
C(2)—C(3)—C(3a)	101·8 (2)	C(2)—C(3)—C(9)	114·5 (2)
C(3a)—C(3)—C(9)	111·7 (2)	C(3)—C(3a)—C(4)	129·5 (2)
C(3)—C(3a)—C(7a)	110·7 (2)	C(4)—C(3a)—C(7a)	119·8 (2)
C(3a)—C(4)—C(5)	120·3 (2)	C(3a)—C(4)—O(1)	115·7 (2)
C(5)—C(4)—O(1)	124·0 (2)	C(4)—C(5)—C(6)	119·6 (2)
C(5)—C(6)—C(7)	121·0 (2)	C(5)—C(6)—O(2)	123·7 (2)
C(7)—C(6)—O(2)	115·4 (2)	C(6)—C(7)—C(7a)	118·6 (2)
C(6)—C(7)—O(3)	121·6 (2)	C(7a)—C(7)—O(3)	119·5 (2)
C(1)—C(7a)—C(3a)	110·7 (2)	C(1)—C(7a)—C(7)	128·4 (2)
C(3a)—C(7a)—C(7)	120·7 (2)	C(3)—C(9)—C(10)	114·8 (2)
C(4)—O(1)—C(11)	118·8 (2)	C(6)—O(2)—C(12)	117·9 (2)
C(7)—O(3)—C(13)	115·2 (2)	C(1)—C(14)—C(15)	119·6 (2)
C(1)—C(14)—C(19)	122·6 (2)	C(15)—C(14)—C(19)	117·8 (2)
C(14)—C(15)—C(16)	121·3 (2)	C(14)—C(15)—O(4)	115·1 (2)
C(16)—C(15)—O(4)	123·6 (2)	C(15)—C(16)—C(17)	120·0 (2)
C(16)—C(17)—C(18)	119·7 (2)	C(16)—C(17)—O(5)	124·1 (2)
C(18)—C(17)—O(5)	116·2 (2)	C(17)—C(18)—C(19)	119·7 (2)
C(17)—C(18)—O(6)	115·6 (2)	C(19)—C(18)—O(6)	124·7 (2)
C(14)—C(19)—C(18)	121·5 (2)	C(15)—O(4)—C(20)	118·6 (2)
C(17)—O(5)—C(21)	117·7 (2)	C(18)—O(16)—C(22)	117·4 (2)

oriented out of the plane in order to minimize repulsions.

For both isomers no other short molecular contacts are found and molecules are mainly packed by van der Waals forces (Figs. 3 and 4).

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