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# Modular Crystalline Hosts Derived from Mandelic Acid. Host Synthesis, Inclusion Formation, and Xray Crystal Structures of Two Free Hosts and One Inclusion Complex with Methanol

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Six new host compounds 2-5 (a, b) in optically resolved and racemic forms derived from mandelic acid and having particular lateral substituents were synthesized. Their properties in crystalline inclusion formation were studied and discussed relative to the unsubstituted parent molecules 1 (a, b). Crystal structures of two optically resolved free host compounds (2a, 3a) and of a respective methanol inclusion complex [2a MeOH (1:1)] have been determined by Xray analysis [2a: a = 6.1641(2), b =19.932(1), c = 14.3469(7) Å, orthorhombic,  $P2_12_12_1$ ,  $D_c$ = 1.200 g  $\cdot$  cm<sup>-3</sup>, Z = 4, R = 0.051 for 1650 observed reflexions; 3a: a = 6.2828(3), b = 24.686(4), c = 15.000(1)Å, orthorhombic,  $P2_12_12_1$ ,  $D_c = 1.149 \text{ g} \cdot \text{cm}^{-3}$ , Z = 4, R = 0.048 for 2154 observed reflexions; 2a MeOH (1:1): a = 13.7316(7), b = 5.8722(2), c = 12.7330(6) Å,  $\beta =$ 99.278(4) °, monoclinic,  $P2_1$ ,  $D_c = 1.149 \text{ g} \cdot \text{cm}^{-3}$ , Z = 2, R = 0.046 for 1823 observed reflexions]. In all compounds, the molecules are arranged in columns via OH…O hydrogen bonds. In the free hosts only one hydroxyl group acts as donor and the other one is involved in OH ... phenyl interactions. However, in the complex, both hydroxyl groups of the host are connected through the methanol molecule by hydrogen bonds being responsible for the formation of the chains. Organic structures with R < 0.030retrieved from the Cambridge Structural Database

revealed the absence of correlation between the C— O bond length and the strength of the hydrogen bond (length of the O $\cdots$ O distance) in the C(sp<sup>3</sup>)OH $\cdots$ OC(sp<sup>3</sup>) fragments.

#### INTRODUCTION

Crystal engineering [1] involving organic molecules has emerged a major challenge in supramolecular science [2]. Both homomolecular and cocrystalline systems that selfassemble to give particular properties are in demand [3]. Selective inclusion crystallization of molecular guests into an organic lattice structure (host) is one important use [4]. These hosts may be designed according to some basic principles including a rigid framework and bulky substituents that make the compound difficult to crystallize without a suitable space filling guest thus forming a clathrate (crystalline hostguest com-

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plex) [5]. The clever installment of chirality is another point to control the inclusion property of the host lattice giving rise to high enantioselective clathrate formation [6]. Supramolecular synthons to create specific aggregate structures have been defined and proven successful [7]. Nevertheless, dealing with weak intermolecular interactions other than Hbonds [8] is still a problem considering potential relevance for a multitude of possible packing structures [7, 9].

Previously we have shown that mandelic acid when modified by addition of two phenyl rings to give **1a** and **1b** (optically resolved and racemic forms) yield crystalline host compounds displaying particular inclusion behaviour [10] (Tab. I). Binding schemes that characterize the packing structures have been discussed illustrating dimers and chains and the contribution of  $OH \cdots$  phenyl contacts [10]. Lateral substituents attached to the phenyl rings of **1a** and **1b** are expected to have considerable consequences on both the packing structure and the host behaviour. Following this line, we became interested to study the compounds **25** (**a**, **b**) that are derivatives of **1a** and **1b** containing substituents of different sizes and polarities.

Here we describe the preparation of the new compounds, discuss the properties of crystalline inclusion formation and report Xray structural studies of unsolvated hosts **2a** and **3a** and of the 1:1 (host:guest) methanol inclusion complex of **2a** considering the previous results [10].

#### **RESULTS AND DISCUSSION**

#### Synthesis

Compounds 2a, 2b, 3a, 3b, 4a and 5a were synthesized in a twostep process from (S), (R)

 TABLE I
 Crystalline Inclusion Compounds (host:guest stoichiometric ratio)

Guest solvent <sup>a</sup>	Host compound								
	1a <sup>b</sup>	1b	2a	2b	3a	3b	4a	5a	
MeOH	1:1		1:1						_
1PrOH	2:1								
1BuOH	1:1								
tBuOH	2:1						1:1		
<i>c</i> PentOH	1:1	1:1							
<i>c</i> HeptOH	1:2	1:2							
3MecHexOH	1:1								
iBuNH2	2:1		1:1		1:1		1:1		
2BuNH <sub>2</sub>	2:1		1:1		1:1				
cPentNH <sub>2</sub>	1:1		1:1		1:2				
cHexNH <sub>2</sub>	1:1		1:1		1:1				
2MecHexNH <sub>2</sub>	1:1		1:2				1:1		
3MecHexNH <sub>2</sub>	2:3		1:3				с		
Cyclopentanone	1:1						4:3		
Cyclohexanone	1:1	1:1			1:2	1:2	с	1:1	
3Methylcyclohexanone	1:1	1:1	1:1	1:1	1:1	1:1	с	1:1	
Dimethylformamide	1:3	1:3	1:1	1:1	1:1	1:1		2:1	
Dimethyl sulfoxide	1:1	1:1	1:1	1:1	1:1	1:1		1:1	
1,4Dioxane	2:1						1:1		
Morpholine	1:1	2:3	1:1	1:2	1:1	1:2			
Piperidine	1:1		1:1		1:2		с		
3Methylpiperidine	3:2		1:1	1:2	1:2	1:2	с		
Pyridine	2:1	2:1							
3Picoline	2:1		1:1	1:1				1:1	

<sup>a</sup> The following solvents yielded no crystalline inclusions: EtOH, 2PrOH, 2BuOH, *i*BuOH, *i*BuOH, *i*ChexOH, 2MecHexOH, acetone, 2methylcyclohexanone, 4methylcyclohexanone,  $\beta$ butyrolactone,  $\gamma$ valerolactone, benzaldehyde, acetonitrile, propionitrile, butyronitrile, nitromethane, nitroethane, propylene oxide, tetrahydrofuran, 2methyltetrahydrofuran, 3methyltetrahydrofuran, toluene, xylene.

<sup>b</sup> Compound 1a also yields crystalline inclusions with  $\gamma$ valerolactone and cycloheptanone.

<sup>c</sup> Difficult to crystallize.

and (R, S)mandelic acid, respectively, by conversion of the acid into the methyl ester followed by reaction with the corresponding aryllithium reagent to give the products in moderate to good overall yields. Inclusion compounds were obtained by recrystallization of the hosts from the respective guest solvent.



#### **Inclusion Properties**

In order to make a comparative study possible, the new potential host compounds **25** (**a** and **b**) were tested with the same variety of solvents used for **1a** and **1b** [10]. These include alcohols and amines of different molecular sizes and degrees of ramification, dipolar aprotic compounds of different polarities, heterocycles with different numbers and types of heteroatoms, as well as aromatic hydrocarbons (Tab. I).

The ability of the new compounds to form crystalline inclusions is evident. However, the range of these inclusions is in no case equal to 1a, the optically resolved parent compound for the reported modifications. Generally, the inclusion ability of the optically resolved form is always higher compared to the respective racemic host, also relating to 1b. There is no case of an inclusion only yielded by the racemic form; in effect inclusion only occurs with solvents that are also enclathrated by the optically resolved host. Aside from this general facts specific differences and conformity in the inclusion ability of the new compounds 25 (a and b) relative to 1 (a, b) are as follows.

Alcohols are found a very inefficient class of guests for the new compounds, with the inclusions between 2a and MeOH or 4a and tBuOH being the rare exceptions. By way of contrast, 1a gave crystalline inclusions with alcohols of different size and shape while 1b seems to prefer cyclic alcohols. Another remarkable finding is for the amine class of guests. Among the new compounds only 2a, 3a and 4a, decreasing in this order, are capable of forming inclusions with amines, whereas 5a, just as 1b, failed and 1a is again superior. On the other hand, there is a block in Table I including aprotic polar solvents such as 3methylcyclohexanone, dimethylformamide and dimethyl sulfoxide that form inclusions with nearly all hosts irrespective of substituents and optical specification. 1,4Dioxane has proved a common guest molecule in lattice inclusion [11]. But not here since inclusion compounds of 1,4dioxane are only formed with 1a and 4a though with different stoichiometric ratios. And one last remark, apolar hydrocarbon molecules totally refuse to be accommodated.

#### **Structural Studies**

A reasonable way of understanding the consequences coming from a modification of substituents is to compare related species. Thus structural studies of free host lattices **2a** and **3a** and of the methanol inclusion complex of **2a** are quite obvious including the previous studies [10].

The main characteristics of the molecular and crystal structures of compounds 2a,  $2a \cdot MeOH$  (1:1) and 3a are shown in Table II according to the numbering scheme depicted in Figure 1. The OH groups are in a *gauche* conformation in all compounds, however, significant differences between the lengths of both C—O bonds in the free host with respect to those in the complex

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TABLE II Selected geometrical parameters (Å, °)<sup>a</sup>

Compound	2a	2a · MeOH (1:1)		3a
C(1)-C(2) C(1)-O(4) C(1)-C(11) C(1)-C(21) C(2)-C(21) C(2)-C(31) C(2)-O(5)	1.553(5) 1.425(4) 1.536(5) 1.532(4) 1.509(5) 1.444(4)	$\begin{array}{c} 1.551(4) \\ 1.445(4) \\ 1.525(4) \\ 1.525(4) \\ 1.516(4) \\ 1.419(4) \end{array}$	1. 1. 1. 1. 1. 1. 1.	556(3) 425(3) 543(4) 532(3) 513(3) 444(3)
C(12)-C(11)-C(16) C(22)-C(21)-C(26) C(32)-C(31)-C(36) C(2)-C(1)-C(11) C(1)-C(11)-C(12) C(2)-C(1)-C(21) C(1)-C(21)-C(22)	117.8(3) 118.5(3) 118.1(4) 113.1(3) 124.8(3) 108.0(3) 119.8(3)	117.3(3) 118.1(3) 118.9(3) 112.9(3) 123.7(3) 108.5(2) 120.2(3)	1 1 1 1 1 1 1 1 1 1	17.3(2) 16.7(2) 19.7(2) 07.9(2) 21.9(2) 11.4(2) 22.7(2)
O(4)-C(1)-C(2)-O(5) C(21)-C(1)-C(2)-C(31) C(11)-C(1)-C(2)-C(31) C(1)-C(2)-C(31)-C(32) C(2)-C(1)-C(11)-C(12) C(2)-C(1)-C(21)-C(22)	61.0(3) 56.3(4) 178.3(3) -116.2(4) -20.1(5) 64.9(4)	62.9(3) 56.3(3) 178.4(3) -107.1(3) -16.6(4) 76.4(4)	1 -1 -	58.9(2) 57.3(3) 78.3(3) 08.3(3) 59.3(3) 46.9(3)
2a	X-H	$\mathbf{X}\cdots\mathbf{Y}$	$H\cdots Y$	$X-H\cdots Y$
$\begin{array}{l} O(4)-H(4)\cdots O(5)(-1/2+x,1/2-y,-z)\\ O(5)-H(5)\cdots C(1116)(1/2+x,1/2-y,-z)\\ C(17)-H(171)\cdots C(3136)(-1/2+x,1/2-y,-z)\\ C(27)-H(271)\cdots C(2126)(1/2+x,1/2-y,1-z) \end{array}$	0.76(6) 0.75(5) 0.93(8) 0.80(7)	3.077(4) 3.631(3) 3.973(7) 3.910(7)	2.40(6) 2.92(5) 3.27(8) 3.21(7)	150(5) 161(5) 133(6) 148(6)
2a · MeOH (1:1)				
$\begin{array}{l} O(4)-H(4)\cdots O(6)\\ O(5)-H(5)\cdots O(6)(1-x,1/2+y,1-z)\\ O(6)-H(6)\cdots O(5)\\ C(7)-H(72)\cdots C(3136)(1-x,-1/2+y,1-z)\\ C(33)-H(33)\cdots C(3136)(1-x,1/2+y,2-z)\\ C(15)-H(15)\cdots C(1116)(-x,-1/2+y,1-z)\\ C(34)-H(34)\cdots C(2126)(1-x,-1/2+y,2-z)\\ C(17)-H(173)\cdots C(2126)(-x,1/2+y,1-z)\\ \end{array}$	0.95(4) 0.83(6) 0.97(7) 0.93(8) 0.96(5) 0.98(5) 0.97(4) 0.91(15)	3.072(4) 2.713(4) 2.657(4) 3.625(5) 3.882(4) 4.106(4) 4.176(4) 3.618(5)	2.14(4) 1.89(6) 1.70(7) 2.90(8) 3.03(5) 3.31(5) 3.32(4) 3.22(11)	166(3) 167(5) 172(6) 136(5) 148(3) 140(3) 147(3) 109(8)
3a				
$\begin{array}{l} O(4)-H(4)\cdots O(5)(-1/2+x,1/2-y,-z)\\ O(5)-H(5)\cdots C(1116)\\ O(5)-H(5)\cdots C(3136)(-1/2+x,1/2-y,-z)\\ C(301)-H(3013)\cdots C(1116)(1/2+x,1/2-y,1-z)\\ C(292)-H(2921)\cdots C(1116)(1/2+x,1/2-y,1-z)\\ C(301)-H(3012)\cdots C(2126)(1/2+x,1/2-y,1-z)\\ C(292)-H(2923)\cdots C(2126)(1/2+x,1/2-y,1-z)\\ \end{array}$	0.80(3) 0.90(3) 0.90(3) 0.97(-) 0.93(-) 0.82(13) 0.92(16)	2.848(2) 3.801(2) 4.076(2) 3.882(7) 3.904(8) 3.748(9) 4.338(12)	2.12(3) 3.08(3) 3.36(2) 3.11(-) 2.97(-) 3.05(13) 3.45(10)	151(3) 138(3) 138(3) 140(-) 177(-) 144(10) 141(6)

<sup>a</sup>C(i1i6), i = 1, 2, 3 represent the centroids of the C(11)  $\cdots$  C(16), C(21)  $\cdots$  C(26) and C(31)  $\cdots$  C(36) phenyl rings.

have been detected. For each compound, these differences [C(1)-O(4) vs. C(2)-O(5)], seem to be correlated with the strength of the hydrogen bond in which they are involved as measured by their  $O \cdots O$  distances. The larger the C—O distance the weaker the interaction (larger  $O \cdots X$ ). This effect has also been observed in

previous studies of mandelic and lactic acid derivatives [10, 12, 13]. In spite of the broad distribution, a linear correlation can be observed in Figure 2 (there are two points which present an anomalous behaviour).

Angular distortions at C(1), C(11) and C(21) are closely related to the spatial disposition of



FIGURE 1 Molecular structures of (a) compound 2a, (b) the asymmetric unit of  $2a \cdot MeOH$  (1:1) (dotted lines specify hydrogen bonds) and (c) compound 3a showing the disorder of one *t*butyl group. Displacement ellipsoids were drawn at 30 % probability level.



FIGURE 2 Scatterplot of the C—O (donor) vs.  $O \cdots O$  distances for mandelic and lactic acid related structures (see text for references).

the corresponding phenyl rings with respect to C(2). The compounds **2a** and **2a** · **MeOH** (1:1), where the C(11) · · · C(16) and C(21) · · · C(26) rings are more and less coplanar with the C(2) atom, present the same pattern of bond angles. The opposite situation is observed in **3a**. In all phenyl rings, the *ipso* angle [C(i2)C(i1)C(i6); i = 1, 2, 3] reflects the influence of the  $\sigma$ electron withdrawing character of the substituent [14] (Tab. II).

Compounds 2a and 3a roughly display the same crystal packing. The hydroxy group at C(1) links molecules forming chains that are reinforced by OH ... phenyl interactions [15] (Figs. 3a,b and Fig. 4). The hydrogen bond network around the twofold screw axes form chains of molecules along *a* that are quite similar in both crystals. The small differences are due to the conformation of the phenyl rings (Tab. II and Fig. 4) and also the disposition of the hydrogen at  $O(5)[H(5)O(5)C(2)C(1) = -165(5), 37(2)^\circ$ , respectively]. The OH orientation in 3a allows the formation of a three center hydrogen interaction, while in 2a this interaction appears to be stronger and more linear. Besides that, the H · · · centroid distances and the O-H · · · centroid





FIGURE 3 (Continued).





FIGURE 4 Projection along the *b* axis of one of the chains of molecules in compound (a) **2a** and (b) **3a**.

FIGURE 3 Crystal packing of (a) compound 2a projected along the *a* axis; (b) 3a projected along the *a* axis; (c) and (d)  $1a \cdot MeOH$  (phenyl) (in ref 10) and  $2a \cdot MeOH$  (ptolyl), respectively, projected along the *b* axis, together with a schematic representation of the crystal packing for both complexes.

angle place these interactions among those belonging to the small group of short and linear interactions observed in a statistical study for these type of contacts [10]. When comparing the crystal structures of the free hosts, the influence of the bulky substituent in *para* position is mainly reflected by the elongation of the *b* axis. The distances between the C(2126) planes related by a twofold screw axis along *a* and between the corresponding centroids increase [from 1.856(2) to 4.068(1) Å and from 5.527(3) to 6.335(2) Å, respectively] to accommodate a disordered *f*butyl group. However, similar C—H ··· phenyl contacts are pre-

sent (Tab. II) forming sheets of chains in the *ac* planes.

Host molecules related by twofold screw axes in 2a · MeOH (1:1) are joined through hydrogen bonds that involve the methanol guest to from chains parallel to the b axis (Fig. 3d). The crystal packing of this complex can be related with that formed by the same guest and the parent host molecule 1a without the lateral methyl groups [10] (Fig. 3c). The unit cell parameters used for the present crystal structure determination (see experimental part) could be transformed, for comparison purpose, according to a = -a, b = b and c = a + c. The new values are a = 13.732, b = 5.872, c = 17.155 A and  $\beta =$ 124.65°. For both complexes, the previously described chains are almost identical and pack in a similar fashion to form sheets in the ab plane. A schematic representation of the packing modes are shown in Figures 3c and 3d. The presence of the methyl groups changes the surface topology of the sheets, hindering the way they interdigitate in the previously described compound [10] (Fig. 3). This change causes an expansion of the interplanar space between sheets (with the elongation of the *c* axis) and also a glide displacement along a to optimize the contact of the hydrophobic surface between sheets (with the opening of the  $\beta$  angle). In this new arrangement, the phenyl...phenyl, Ttype, interactions (Tab. II) are conserved inside each sheet but new contacts replace those ones that are lost in the interface between sheets. In all compounds described here, this kind of weak aromatic interactions (Tab. II) join together chains giving rise to the whole crystal.

There are no voids in the structures when using a model of interpenetrating spheres of van der Waals radii [16]. The total packing coefficients are 0.66 for both free hosts and 0.64 for  $2a \cdot MeOH$  (1:1) while the local coefficient for the guest is 0.54. The methanol molecules were allocated in cavities that almost form channels. They are prolate in shape [16] with the maximum dimension along the *a* axis.

The O-H···O hydrogen interactions have been the subject of several studies [17]. It is well known that the O---H bond length increases with the shortening of the  $O \cdots O$  distance. Besides, the C--O<sup>-</sup> bond appears to be shorter than the corresponding C-O single bond if the proton is completely transferred. For example, that is the case in the picrate anion vs. the picric acid [18] or in phenolate compounds vs. phenol derivatives [19]. The differences in length observed between both C-O bonds in the present compounds lead us to perform a statistical study using the Cambridge Structural Database [20] in order to find out if there is any correlation between them and the  $O \cdots O$  intermolecular distances. Organic structures with  $C(sp^3)$ -OH···O-C(sp^3) fragments and a good degree of accuracy (R < 0.030), neither disorder nor errors and O—H $\cdots$ O angles larger than 130° have been retained (198 structures and 457 hits). Figure 5 shows the scatterplot of both distances, C—O(donor) vs.  $O \cdots O$  (a), C—O(acceptor) vs.  $O \cdots O$  (b) and their corresponding distributions together with that of the parameter [C-O(donor)C—O(acceptor)] and the O— $H \cdots O$  angle (c, d, e, f). In spite of the lack of correlation between the parameters in Figure 5a, there are two empty regions at the top left and the bottom right in the scatterplot.  $\Delta$  values up to 0.06 Å have been observed. The values C--O(donor/acceptor) and O····O ranges are 1.3841.455Å/ 1.3741.476 Å and 2.6173.026 Å (3.04 Å being the sum of van der Waals radii) [21] with average values of 1.423(11)/ 1.427(11) and 2.786(83) Å, respectively (the standard deviation of the sample being in parentheses). If the search (same fragment) is restricted to donor OH groups in which the O atom is not involved in other hydrogen bonds as acceptor, similar results are obtained for 97 structures and 110 interactions. In both cases, the shortest O···O distance (2.617 Å) [22] corresponds to similar C—O bonds (1.427 and 1.425 Å; CSD refcode: LIJPEZ). The histogram is quite symmetrical (skew parameter = -0.026) while the ones corresponding to C-O(donor/accep-



FIGURE 5 Statistical study of the  $C(sp^3)$ —OH···O— $C(sp^3)$  fragment: (a) Scatterplot of the C—O(donor) vs. O···O distances, (b) same for C—O(acceptor) vs. O···O, (c) histogram of the C—O(donor) distance, (d) histogram of the O···O contacts, (e) histogram of ( = difference values from the mean C—O distance) and (f) histogram of O—H···O angle.

tor),  $O \cdots O$  distances and the O—H $\cdots$ O angle are not (skew = -0.251/-0.241, 0.729 and -0.862, respectively). Skewness and nonGaussian distribution of the values concerned here might indicate that some of the contacts in this ensemble derive from nonproductive interactions. Consequently this may smear correlation of C—O and O $\cdots$ O values in the data set assembled. The most probable values for the O $\cdots$ O and O—H $\cdots$ O parameters are 2.72 Å and 170°.

#### CONCLUSIONS

Laterally substituted derivatives of a previously introduced host compound 1 [10] derived from mandelic acid and produced in optically resolved and racemic forms 25 are shown to form crystalline inclusion complexes, thus establishing the framework of a new robust host family allowing for structural modification. Depending on the particular substituent (Me, tBu, Ph, F) and on the optical species, the property of 25 to form hostguest inclusion complexes is rather different. However, in all cases the optically resolved compound is much more efficient compared to the racemic analogue, and none of the new compounds is able to compete with the unsubstituted parent host 1a as far as the number of inclusion compounds is concerned. This illustrates superiority of 1a, but from the selectivity point of view the substituted derivatives are more effective.

The superority of the optically resolved compounds may be interpreted from the structural results as follows. In the optically resolved free hosts (**1a**, [10] **2a**, **3a**), the molecules are arranged in columns via  $OH \cdots O$  hydrogen bonds. However, only one hydroxyl group acts as donor and the other one is involved in  $OH \cdots$  phenyl interactions. Thus, these structures do not meet the optimum hydrogen bonding rule in crystals recently defined by Etter [23] and others, [8] but exhibit a compromise between close packing and hydrogen bonding. In the alcohol complexes  $[1a \cdot MeOH (1:1) [10] and 2a$ Me · OH (1:1)], the situation improves since both hydroxyl groups of the hosts are connected through the alcohol molecules by hydrogen bonds being responsible for the formation of the supramolecular chains.

By way of contrast, in the structure of the free racemic host 1b, previously reported, [10] hydrogen bonded dimers of enantiomeric host molecules are formed. It seems that this particular pattern is fairly stable allowing a relative densely packed crystal without having recourse to solvent molecules, thus explaining low inclusion behaviour of 1b relative to 1a and perhaps also of 2b and 3b relative to 2a and 3a. This is probably the situation in case of the racemic hosts and most of the strong hydrogen donor solvents (Tab. I), not competing with the dimer units, unlike the strong hydrogen acceptor solvents presumably overruling the dimer formation of hosts to give inclusion compounds. These findings suggest that other optically differentiated host compounds may have very distinct inclusion properties, such as here, stimulating further studies in this field.

#### **EXPERIMENTAL**

#### Synthesis

#### General

Melting points were taken with a Kofler hot stage apparatus. IR spectra were recorded on a Perkin-Elmer 1600 FTIR spectrometer; spectral bonds are reported in cm<sup>-1</sup>. Proton NMR spectra were measured with a Varian T60A (60 MHz) spectrometer and <sup>13</sup>C NMR spectra with a Bruker MSL 300 (300 MHz) instrument; chemical shifts are reported in ppm downfield from tetramethylsilane as internal reference. Microanalyses were carried out by the Microanalytical Laboratory of the Technical University Bergakademie Freiberg.

Compounds **1a** and **1b** were prepared as described previously [10].

#### Compounds 2a, 2b, 3a, 3b, 4a and 5a

They were synthesized from optically resolved or racemic methyl mandelates [24] and aryllithium reagents analogous to 1a/1b [10]. Specific details are given for each compound.

### (R)2Phenyl1,1di(ptolyl)ethane1,2diol (2a)

(*R*)Methyl mandelate and 4bromotoluene were used; colourless crystals [from benzenecyclohexane, (1:1)], 34%, m.p. 150 °C,  $[\alpha]^{21}_{D}$  + 205° (c1, CHCl<sub>3</sub>). IR (KBr): 3510, 3476 (OH), 2922 (CH<sub>3</sub>), 744, 700 (Ar). <sup>1</sup>HNMR (60 MHz, CDCl<sub>3</sub>): 7.457.0 (m, 13H, Ar), 5.37 (s, 1H, CH), 3.02 (s, 1H, OH), 2.75 (s, 1H, OH), 2.21 (2s, 6H), MeH). <sup>13</sup>CNMR (75 MHz, CDCl<sub>3</sub>): 20.9, 21, 77.8, 80.5, 126129.8, 136142.3 (19 signals). GCMS (m/z): 284 (5), 211 (100), 119 (99), 91 (40). Anal.: found: C, 83.03; H, 6.64; C<sub>22</sub>H<sub>22</sub>O<sub>2</sub> requires: C, 82,99; H, 6.96.

## (R,S)2Phenyl1,1di(ptolyl)ethane 1,2diol (2b)

(R,S)Methyl mandelate and 4bromotoluene were used; colourless crystals, 55 %, m.p. 178°C. Spectroscopic data as given for the optically resolved species **2a**.

## (R)1,1Bis(4tertbutylphenyl)2phenylethane1,2diol (3a)

(*R*)Methyl mandelate and 4*tert*butylbromobenzene were used; colourless crystals [from benzenenhexane, (1:1)], 62 %, m.p. 172 °C,  $[\alpha]^{21}_{D}$  + 143° (c1, CHCl<sub>3</sub>). IR (KBr): 3514, 3430 (OH), 1394, 1362 (*t*Bu), 749, 715 (Ar). <sup>1</sup>HNMR (60 MHz, CDCl<sub>3</sub>): 7.467.0 (m, 13H, Ar), 5.53 (s, 1H, CH), 2.98 (s, 1H, OH), 2.45 (s, 1H, OH), 1.21 (d, 18H, *t*BuH). <sup>13</sup>CNMR (75 MHz, CDCl<sub>3</sub>): 31.3, 31.4, 34.4, 34.4, 78.1, 80.6, 124128.1, 138149.9 (18 signals). GCMS (m/z): 355 (5), 295 (100), 297 (13), 161 (36). Anal.: found: C, 83.50; H, 8.04; C<sub>28</sub>H<sub>34</sub>O<sub>2</sub> requires: C, 83.54; H, 8.51.

# (R,S)1,1Bis(4tertbutylphenyl) 2phenylethan1,2diol (3b)

(*R*,*S*)Methyl mandelate and 4*tert*butylbromobenzen were used; colourless crystals, 75 %, m.p. 196°C. Spectroscopic data as given for the optically resolved species **3a**.

# (S)1,1Di(4biphenylyl)2phenylethane 1,2diol (4a)

(S)Methyl mandelate and 4bromobiphenyl were used; colourless crystals (from toluene), 40 %, m.p. 190 °C,  $[\alpha]^{21}{}_{D}$  –194° (c1, CHCl<sub>3</sub>). IR (KBr): 3570, 3456 (OH), 3030 (CH), 1486, 831702 (Ar). <sup>1</sup>HNMR (60 MHz, CDCl<sub>3</sub>): 7.267.1 (m, 14H, BiPhH), 7.0 (s, 5H, ArH), 5.53 (s, 1H, CH), 2.43 (s, 1H, OH), 2.01 (s, 1H, OH). <sup>13</sup>CNMR (75 MHz, CDCl<sub>3</sub>): 77.9, 80.6, 126131.8, 138144.1 (26 signals). GCMS (m/z): 334 (83), 257 (8), 181 (100), 152 (67). Anal.: found: C, 86.85; H 5.92; C<sub>32</sub>H<sub>26</sub>O<sub>2</sub> requires: C; 87.01; H, 6.23.

## (S)1,1Bis(4fluorophenyl)2phenylethane 1,2diol (5a)

(S)Methyl mandelate and 4fluorobrombenzene were used; colourless crystals [from toluenecyclohexane (1:1)], 68 %, m.p. 133 °C,  $[\alpha]^{21}_{D}$  –189° (c1, CHCl<sub>3</sub>). IR (KBr): 3456 (OH), 3030 (CH), 1486, 831702 (Ar). <sup>1</sup>HNMR (60 MHz, CDCl<sub>3</sub>): 7.686.75 (m, 13H, ArH), 5.45 (s, 1H, CH), 3.22 (s, 1H, OH), 2.44 (s, 1H, OH). <sup>13</sup>CNMR (75 MHz, CDCl<sub>3</sub>): 77.9, 80.0, 114.2140.8, 163.2, 163.6 (signals). GCMS (m/z): 218 (28), 123 (100), 95 (63), 75 (21). Anal.: found: C, 73.27, H, 5.09; C<sub>20</sub>H<sub>16</sub>O<sub>2</sub>F<sub>2</sub> requires: C, 73.16; H, 5.53.

#### **Crystalline Inclusion Compounds**

These were obtained by recrystallization of the corresponding host compound from a minimum amount of the respective guest solvent. The crystals formed were collected by suction filtration, washed with an inert solvent (hexane) and dried (0.5 h, 15 Torr, room temperature). The

hostguest stoichiometric ratio was determined by <sup>1</sup>HNMR integration. Data for each compound are given in Table I.

# Xray Data Collection, Structure Determination, and Refinement

Single crystals of **2a**, **2a** · **MeOH** (1:1) and **3a** were prepared by slow evaporation of solvent from solutions of nitromethane and MeOH in the latter cases.

Crystal data, experimental details and refinement parameters are displayed in Table III. A crystal of  $2a \cdot MeOH$  (1:1) was mounted in a glass capillary with solvent to prevent decomposition. Initial structure models for 2a,  $2a \cdot MeOH$  (1:1), and 3a obtained by direct methods (SIR 92 [25]) were consecutively refined by fullmatrixleastsquares. One of the *t*butyl groups is statistically disordered in two positions with occupancy factors of 0.55(2) and 0.45(2). Hydrogen atoms were located in the

	2a	2a · MeOH (1:1)	
Crystal data			
Formula Crystal habit Crystal size (mm)	$C_{22}H_{22}O_2$ Colourless prism $0.50 \times 0.40 \times 0.47$ Orthorbombic P2 2 2	$C_{22}H_{22}O_2 \cdot CH_3OH$ Colourless prism $0.50 \times 0.40 \times 0.17$ Monoclinic, P2	$C_{28}H_{34}O_2$ Colourless prism $0.20 \times 0.20 \times 0.40$ Orthornhig, P2-2-2.
Unit cell determination	Leastsquares fit from 59 reflexions ( $\theta < 45^{\circ}$ )	Leastsquares fit from 84 reflexions ( $\theta < 45^{\circ}$ )	Leastsquares fit from 81 reflexions ( $\theta < 45^{\circ}$ )
Unit cell dimensions (Å, °)	a = 6.1641(2) b = 19.932(1) c = 14.3469(7) 90, 90, 90	a = 13.7316(7) b = 5.8722(2) c = 12.7330(6) 90 = 9278(4) = 90	a = 6.2828(3) b = 24.686(4) c = 15.000(1) 90 90 90
Packing: V(Å), Ζ Dc(g/cm <sup>3</sup> ), Μ, F(000) μ(cm <sup>-1</sup> ) T(K)	1762.8(1), 4 1.200, 318.42, 680 5.56 295	1013.3(1), 2 1.149, 350.46, 376 5.59 225	2326.6(4), 4 1.149, 402.58, 872 5.10 150
Experimental data			
Technique	Four circle diffractometer: Graphite monochromator: Detector apertures 1×1°.	Philips PW1 100, Bisecting geometry CuK $\alpha$ , $\omega/2\theta$ scans	
Scan width Scan speed	1.5° 1 min./reflex.	1.5° 0.5 min./reflex.	1.5° 1.0 min./reflex.
Independent Observed Standard reflexions	1733 1650 (3σ (I) criterion) 2 reflexions every 90 minutes No variation	1887 1823 (3σ (I) criterion) 8.3%	2290 2154 (3σ (I) criterion) 2.5%
Solution and refinement			
Solution Refinement		Direct Methods: SIR92	
LeastSquares on Fo	full matrix	full matrix	full matrix
Parameters: Number of variables Degrees of freedom Ratio of freedom	305 1345 5.4	338 1485 5.4	442 1712 4.9
H atoms		From difference synthesis*	
Weightingscheme	Empirical as to	give no trends in $\langle \omega^2 F \rangle$ vs. $\langle  Fe $	$ \rangle$ and $\langle \sin \theta / \lambda \rangle$
Max. thermal value (Å <sup>2</sup> ) Final ρ peaks (eÅ <sup>-3</sup> ) Final R and Rw	U11(C(17)) = 0.200(9) $\pm 0.20$ 0.051, 0.068	U22(C(17)) = 0.101(3) $\pm 0.16$ 0.046, 0.053	U11(C(302)) = 0.168(14) ±0.23 0.048, 0.046

TABLE III Crystal analysis parameters

\*See experimetal section.

TABLE IV Final atomic coordinates and Ueq<sup>a</sup>

Compound 2a C1 0.2296(2) 0.1297(2) 44(1) C2 0.42816(6) 0.1955(2) 0.1024(2) 44(1) C31 0.4451(6) 0.10297(2) 0.1149(2) 44(1) C32 0.6579(8) 0.09292(2) 0.1497(3) 62(1) C33 0.6596(11) 0.0232(2) 0.1550(4) 77(2) C34 0.4689(12) -0.0185(2) 0.1353(4) 82(2) C35 0.2295(11) 0.0088(2) 0.1054(5) 91(2) C36 0.2741(9) 0.0777(2) 0.0933(4) 77(2) C4 0.0514(4) 0.2036(1) 0.1599(2) 48(1) C1 0.2534(6) 0.3065(2) 0.1565(2) 46(1) C1 0.2534(6) 0.3065(2) 0.1565(2) 46(1) C1 0.2534(6) 0.3065(2) 0.1565(2) 46(1) C1 0.2534(6) 0.3065(2) 0.1565(2) 46(1) C1 0.0455(10) 0.4449(2) 0.1363(3) 74(2) C1 0.0455(10) 0.4449(2) 0.1363(3) 74(2) C1 0.0455(10) 0.4449(2) 0.1363(3) 74(2) C1 0.0455(10) 0.4449(2) 0.1365(3) 74(2) C1 0.055(10) 0.0239(2) 0.0256(3) 74(1) C1 0.055(10) 0.0239(2) 0.0256(3) 74(1) C1 0.055(10) 0.0239(2) 0.0256(3) 74(1) C1 0.055(10) 0.0239(2) 0.0256(3) 74(1) C1 0.055(10) 0.0239(2) 0.0256(3) 74(1) C2 0.5959(8) 0.2166(3) 0.3946(3) 71(1) C2 0.5959(8) 0.2129(2) 0.4449(3) 71(1) C2 0.5959(8) 0.2129(2) 0.4449(3) 71(1) C2 0.5959(8) 0.2129(2) 0.4449(3) 71(1) C2 0.5959(8) 0.2129(2) 0.4449(3) 71(1) C2 0.2964(2) 0.4677(8) 0.9358(3) 87(1) C3 0.05959(2) 0.46677(8) 0.9358(3) 87(1) C3 0.05959(2) 0.46677(8) 0.9358(3) 87(1) C3 0.05959(2) 0.46677(8) 0.9358(3) 87(1) C3 0.05959(2) 0.4497(2) 0.5305(6) 0.576(2) 56(1) C3 0.05959(3) 0.741(7) 0.4770(3) 58(1) C3 0.05959(3) 0.741(7) 0.4770(3) 58(1) C3 0.05959(3) 0.741(7) 0.4770(3) 58(1) C3 0.05959(3) 0.741(7) 0.4770(3) 58(1) C3 0.05959(3) 0.741(7) 0.5752(2) 40(1) C3 0.02959(3) 0.756(2) 0.9667(13) 28(1) C3 0.02959(3) 0.255(3) 22(1) 0.55697(1) 0.5753(1) 27(1) C3 0.03969(2) 0.2449(1) 0.17	Atom	<i>x</i>	y	<i>z</i>	Ueq	
C1         0.2626(6)         0.2298(2)         0.1687(2)         44(1)           C2         0.4281(6)         0.1955(2)         0.1124(2)         46(1)           C3         0.4570(8)         0.0222(2)         0.1590(4)         77(2)           C3         0.4550(1)         0.0232(2)         0.1590(4)         77(2)           C3         0.4590(1)         0.0088(2)         0.1054(1)         92(2)           C4         0.4990(1)         0.0099(2)         73(1)           C4         0.9514(4)         0.0096(1)         0.1999(2)         73(1)           C1         0.3915(11)         0.4138(2)         0.1568(2)         46(1)           C12         0.446(8)         0.3497(2)         0.1568(2)         74(1)           C14         0.2297(1)         0.4694(2)         0.1393(3)         74(2)           C14         0.2297(1)         0.4694(2)         0.1757(3)         71(1)           C15         0.0455(10)         0.4694(2)         0.1757(3)         71(1)           C16         0.074(8)         0.3393(2)         0.1844(3)         113(3)           C1         0.3558(2)         0.4597(2)         6(3)         6(3)           C2         0.2696(8)         0	Compound 2a					
C2         0.4281(6)         0.1959(2)         0.1024(2)         44(1)           C31         0.4351(6)         0.1205(2)         0.1149(2)         48(1)           C32         0.6359(6)         0.0225(2)         0.1550(4)         77(2)           C34         0.4380(12)         -0.0185(2)         0.1550(4)         77(2)           C35         0.2355(11)         0.0088(2)         0.155(5)         91(2)           C36         0.2741(9)         0.777(2)         0.933(4)         73(2)           C4         0.355(5)         0.2118(1)         0.0088(2)         35(5)           C1         0.2534(6)         0.306(2)         0.1565(2)         46(1)           C12         0.4146(8)         0.3447(2)         0.1156(3)         79(1)           C14         0.229(11)         0.4459(2)         0.1336(3)         74(2)           C15         0.4657(10)         0.4459(2)         0.1364(3)         71(1)           C16         0.674(8)         0.3393(2)         0.184(3)         71(1)           C17         0.1812(2)         0.2394(3)         0.7(1)           C16         0.5535(1)         0.1512(2)         0.4905(6)         0.544(3)           C17         0.5535(2)	C1	0.2626(6)	0 2298(2)	0 1687(2)	44(1)	
C1         0.4451(6)         0.1205(2)         0.1149(2)         48(1)           C32         0.6576(11)         0.0232(2)         0.1556(4)         77(2)           C33         0.6596(11)         0.0088(2)         0.1556(4)         77(2)           C34         0.4880(12)         0.1054(5)         91(2)         0.353(4)         73(2)           C36         0.2241(9)         0.0077(2)         0.0933(4)         73(2)           C4         0.0514(4)         0.2066(1)         0.1056(2)         43(1)           C5         0.3606(5)         0.2118(1)         0.0088(2)         53(1)           C11         0.2334(6)         0.3063(2)         0.1565(2)         46(1)           C12         0.4146(8)         0.3472(2)         0.1158(3)         74(2)           C14         0.229(11)         0.4484(2)         0.1757(3)         71(1)           C16         0.067(8)         0.3392(2)         0.1884(3)         9(1)           C21         0.3558(3)         0.216(4)         0.317(3)         71(1)           C1         0.3558(3)         0.216(3)         0.346(3)         71(1)           C22         0.558(8)         0.216(3)         0.346(3)         71(1)	$\tilde{C}^2$	0.4281(6)	0 1955(2)	0.1024(2)	46(1)	
C22         0.6370(8)         0.0925(2)         0.1447(3)         C2(1)           C33         0.6596(11)         0.0032(2)         0.1550(4)         77(2)           C34         0.4880(12)         -0.0185(2)         0.1353(4)         82(2)           C35         0.2595(11)         0.0093(4)         73(2)         0.430(1)         0.1599(2)         48(1)           C36         0.2741(9)         0.0777(2)         0.0933(4)         73(2)         0.43(1)           C4         0.0514(4)         0.2363(2)         0.1565(2)         46(1)         0.15(1)         0.155(2)         46(1)           C11         0.2354(6)         0.3447(2)         0.136(3)         74(2)         74(2)           C13         0.3931(2)         0.1584(3)         97(1)         71(1)           C14         0.2029(11)         0.4449(2)         0.1757(3)         71(1)           C15         0.0457(10)         0.1589(2)         0.184(3)         97(1)           C17         0.3323(6)         0.2124(2)         0.2699(2)         44(1)           C2         0.538(1)         0.2169(3)         0.354(6)         71(1)           C3         0.558(8)         0.2129(2)         0.4497(2)         91(1) <tr< td=""><td>C31</td><td>0.4451(6)</td><td>0.1205(2)</td><td>0.1149(2)</td><td>48(1)</td><td></td></tr<>	C31	0.4451(6)	0.1205(2)	0.1149(2)	48(1)	
C33         0.6596(T1)         0.0232(2)         0.1550(4)         77(2)           C34         0.4890(12)         -0.0185(2)         0.1353(4)         72(2)           C35         0.2395(11)         0.0098(2)         0.1054(3)         73(2)           C36         0.2741(9)         0.077(2)         0.933(4)         73(2)           C36         0.3964(5)         0.2118(1)         0.0088(2)         53(1)           C11         0.2334(6)         0.3963(2)         0.1556(2)         6(1)           C12         0.4146(8)         0.3472(2)         0.1156(3)         74(2)           C14         0.2292(11)         0.4489(2)         0.1357(3)         71(1)           C16         0.0574(8)         0.3393(2)         0.1884(3)         56(1)           C11         0.2535(6)         0.2124(2)         0.2599(2)         41(1)           C12         0.3598(3)         0.2146(3)         0.391(3)         71(1)           C14         0.2598(6)         0.2175(2)         0.416(4)         111(3)           C1         0.3598(3)         0.2164(3)         0.61(1)         C2           C2         0.3598(3)         0.2156(2)         0.416(2)         6(1)           C2	C32	0.6370(8)	0.0925(2)	0.1447(3)	62(1)	
C34         0.4880(12)         -0.0185(2)         0.1353(4)         922)           C35         0.2595(11)         0.0088(2)         0.1584(5)         912)           C36         0.2741(9)         0.0777(2)         0.0933(4)         732)           C36         0.2544(9)         0.0777(2)         0.0933(4)         732)           C31         0.3546(6)         0.3663(2)         0.1556(2)         46(1)           C11         0.2334(6)         0.3664(2)         0.1556(2)         46(1)           C13         0.3915(11)         0.4489(2)         0.1356(3)         74(2)           C15         0.0455(10)         0.4494(2)         0.1757(3)         71(1)           C16         0.0674(8)         0.3393(2)         0.1884(3)         59(1)           C17         0.312(20)         0.2269(6)         0.1168(4)         111(3)           C14         0.3253(6)         0.2124(2)         0.2699(2)         4(1)           C22         0.5198(8)         0.2166(3)         0.354(63)         7(1)           C33         0.5550(8)         0.2126(2)         0.4169(3)         7(1)           C34         0.4587(8)         0.354(6)         57(1)           C34         0.4587(8)	C33	0.6596(11)	0.0232(2)	0 1550(4)	77(2)	
C35         0.2955(1)         0.0088(2)         0.1054(6)         91(2)           C36         0.2741(9)         0.0777(2)         0.0933(4)         73(2)           O4         0.0514(4)         0.2036(1)         0.1597(2)         48(1)           O5         0.366(5)         0.2118(1)         0.0088(2)         53(1)           C11         0.2534(6)         0.3063(2)         0.156(3)         74(2)           C12         0.446(8)         0.347(2)         0.136(3)         74(2)           C13         0.3915(1)         0.4459(2)         0.1356(3)         74(2)           C14         0.2292(1)         0.4459(2)         0.1356(3)         74(2)           C15         0.0455(10)         0.4084(2)         0.1757(3)         71(1)           C16         0.674(8)         0.3393(2)         0.1184(3)         59(1)           C11         0.3235(6)         0.2209(3)         0.3022(5)         67(1)           C22         0.5398(8)         0.2164(3)         0.3022(5)         67(1)           C36         0.4933(6)         0.4129(3)         97(1)           C4         0.4988(2)         0.1757(2)         0.4149(3)         97(1)           C47         0.3956(2)	C34	0.4880(12)	-0.0185(2)	0.1353(4)	82(2)	
C36         0.2741(0)         0.0777(2)         0.0733(4)         73(2)           O4         0.0514(4)         0.2036(1)         0.159(2)         48(1)           O5         0.3606(5)         0.2118(1)         0.098(2)         53(1)           C11         0.2534(6)         0.366(2)         0.155(2)         46(1)           C12         0.4146(8)         0.347(2)         0.115(3)         65(1)           C13         0.3915(1)         0.4459(2)         0.1336(3)         74(2)           C14         0.2029(1)         0.4459(2)         0.1393(3)         59(1)           C15         0.0657(18)         0.3393(2)         0.1884(3)         59(1)           C16         0.0674(8)         0.2394(3)         0.352(3)         67(1)           C21         0.2356(8)         0.2464(3)         0.352(3)         67(1)           C23         0.5560(8)         0.2166(3)         0.352(3)         67(1)           C24         0.4533(9)         0.175(2)         0.4597(2)         40(1)           C32         0.556(8)         0.2494(3)         71(1)           C4         0.4598(2)         0.4597(2)         42(1)           C4         0.1596(2)         0.4597(2)	C35	0.2955(11)	0.0088(2)	0.1054(5)	91(2)	
O4         0.0514(4)         0.2036(1)         0.1509(2)         48(1)           O5         0.3064(5)         0.2118(1)         0.0088(2)         53(1)           C11         0.2534(6)         0.3063(2)         0.156(2)         64(1)           C12         0.4146(8)         0.3497(2)         0.113(6)         67(1)           C13         0.3915(1)         0.4459(2)         0.1336(3)         74(2)           C15         0.0455(10)         0.4469(2)         0.1757(3)         71(1)           C16         0.0674(8)         0.3393(2)         0.1848(3)         99(1)           C17         0.1812(20)         0.5206(3)         0.71(1)         C12           C22         0.5598(8)         0.2124(2)         0.2690(2)         44(1)           C33         0.5853(9)         0.1767(2)         0.4197(3)         77(1)           C34         0.4583(9)         0.1767(2)         0.4197(3)         87(2)           C32         0.2594(1)         0.1559(2)         0.4197(3)         39(1)           C35         0.2694(1)         0.1559(2)         0.4197(3)         39(1)           C37         0.3348(2)         0.5658(3)         0.57(1)         234(1)           C36	C36	0.2741(9)	0.0777(2)	0.0933(4)	73(2)	
05         0.3606(5)         0.2118(1)         0.008(2)         35(1)           C11         0.2534(6)         0.365(2)         0.155(2)         46(1)           C12         0.4146(8)         0.3447(2)         0.115(3)         65(1)           C13         0.3915(1)         0.4459(2)         0.1356(3)         74(2)           C14         0.2029(11)         0.4459(2)         0.1376(3)         74(1)           C15         0.0657(4)         0.2123(2)         0.390(2)         44(1)           C17         0.1812(20)         0.5290(3)         0.1188(4)         111(3)           C12         0.3253(6)         0.2124(2)         0.3290(2)         44(1)           C23         0.5850(8)         0.2166(3)         0.3924(3)         7(11)           C24         0.4533(9)         0.1767(2)         0.4190(3)         7(1)           C34         0.4533(9)         0.1757(2)         0.4149(3)         87(1)           C47         0.3254(1)         0.1557(2)         0.4149(3)         87(1)           C37         0.2568(2)         0.4677(8)         0.3931(3)         57(1)           C47         0.2968(2)         0.4677(8)         0.3318(3)         57(1)           C31	04	0.0514(4)	0.2036(1)	0.1509(2)	48(1)	
C11         0.2334(6)         0.3063(2)         0.1565(2)         46(1)           C12         0.4146(8)         0.3447(2)         0.116(13)         65(1)           C13         0.3915(11)         0.4459(2)         0.1336(3)         74(2)           C15         0.0455(10)         0.4044(2)         0.1757(3)         57(1)           C16         0.0674(8)         0.2393(2)         0.168(4)         111(3)           C21         0.3253(6)         0.2123(2)         0.2690(2)         44(1)           C22         0.5198(8)         0.2194(3)         0.3092(3)         67(1)           C32         0.5550(8)         0.216(3)         0.3946(3)         71(1)           C34         0.4558(9)         0.1767(2)         0.4407(2)         60(1)           C34         0.4558(9)         0.176(2)         0.420(3)         59(1)           C34         0.4568(8)         0.712(2)         0.420(3)         59(1)           C34         0.4568(8)         0.420(3)         59(1)           C34         0.4568(8)         0.420(3)         57(1)           C34         0.4568(2)         0.5631(6)         0.5543(3)         57(1)           C33         0.5558(2)         0.6677(8)	O5	0.3606(5)	0.2118(1)	0.0088(2)	53(1)	
C12         0.4146(8)         0.3447(2)         0.116(3)         65(1)           C13         0.3195(11)         0.4138(2)         0.1336(3)         74(2)           C14         0.2029(11)         0.4459(2)         0.1336(3)         74(2)           C15         0.06574(8)         0.3393(2)         0.1884(3)         59(1)           C17         0.1812(20)         0.5206(3)         0.1168(4)         111(3)           C21         0.2325(6)         0.2124(2)         0.2306(3)         7(1)           C23         0.556(08)         0.216(2)         0.449(3)         7(1)           C24         0.4583(9)         0.1767(2)         0.4507(2)         60(1)           C25         0.2504(10)         0.159(2)         0.449(3)         7(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         57(1)           C27         0.5325(14)         0.180(3)         0.55(1)         10(1)           C2         0.3948(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.7313(3)         55(1)           C41         0.6998(2)         0.4667(8)         0.9313(3)         57(1)           C33         0.5736(6)	C11	0.2534(6)	0.3063(2)	0.1565(2)	46(1)	
C13         0.3915(11)         0.4138(2)         0.1056(4)         79(2)           C14         0.2029(11)         0.4459(2)         0.1757(3)         71(1)           C15         0.0455(10)         0.4084(2)         0.1757(3)         71(1)           C16         0.0674(8)         0.339(2)         0.1864(3)         111(3)           C17         0.1812(20)         0.2596(3)         0.1464(3)         95(1)           C22         0.5198(8)         0.2249(3)         0.3052(3)         67(1)           C23         0.5550(3)         0.1166(3)         0.3946(3)         71(1)           C24         0.4583(9)         0.1767(2)         0.4007(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.4449(3)         71(1)           C26         0.2604(2)         0.1767(2)         0.4203(3)         57(1)           C26         0.2968(2)         0.4203(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5631(6)         0.7581(2)         41(1)           C32         0.4488(2)         0.7387(2)         47(1)           C33         0.5558(2)         0.6677(8)         0.93313(3)         55(1)           C34         0.6698(	C12	0.4146(8)	0.3447(2)	0.1161(3)	65(1)	
C14         0.2029(11)         0.4459(2)         0.1336(3)         74(2)           C15         0.0674(8)         0.3393(2)         0.1884(3)         59(1)           C17         0.1812(20)         0.500(3)         0.11684(3)         71(1)           C21         0.3253(6)         0.2124(2)         0.2690(2)         44(1)           C22         0.5198(8)         0.2166(3)         0.3052(3)         67(1)           C23         0.5850(8)         0.2166(3)         0.3463(3)         71(1)           C24         0.4583(9)         0.1767(2)         0.4597(2)         60(1)           C25         0.2604(10)         0.1595(2)         0.4194(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         57(1)           C31         0.4688(2)         0.6531(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.531(6)         0.551(2)         41(1)           C32         0.4458(2)         0.531(6)         0.551(2)         41(1)           C33         0.558(2)         0.6607(2)         42(1)         0.531(6)         0.556(2)         45(1)           C33         0.5936(3)         0.511(1)         0.333(3)         57(1)	C13	0.3915(11)	0.4138(2)	0.1056(4)	79(2)	
C15         0.4455(10)         0.4084(2)         0.1757(3)         71(1)           C16         0.0674(8)         0.3393(2)         0.1884(3)         59(1)           C17         0.1812(20)         0.5206(3)         0.1168(4)         111(3)           C21         0.32535(6)         0.2124(2)         0.2909(2)         44(1)           C22         0.5198(8)         0.2166(3)         0.394(3)         71(1)           C23         0.5850(8)         0.2166(3)         0.394(3)         71(1)           C24         0.4583(9)         0.1757(2)         0.4149(3)         71(1)           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         59(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           C31         0.4688(2)         0.6431(6)         0.548(3)         87(1)           C31         0.4688(2)         0.5631(6)         0.7581(2)         41(1)           C32         0.44688(2)         0.5631(6)         0.558(3)         57(1)           C33         0.5598(3)         0.5159(7)         0.813(3)         61(1)           C34 </td <td>C14</td> <td>0.2029(11)</td> <td>0.4459(2)</td> <td>0.1336(3)</td> <td>74(2)</td> <td></td>	C14	0.2029(11)	0.4459(2)	0.1336(3)	74(2)	
C16         0.0674(8)         0.3393(2)         0.1884(3)         59(1)           C17         0.1812(20)         0.5206(3)         0.1168(4)         111(3)           C21         0.3253(6)         0.2124(2)         0.2690(2)         44(1)           C22         0.5198(8)         0.2126(3)         0.3052(3)         67(1)           C23         0.3850(8)         0.2166(3)         0.304(3)         71(1)           C24         0.4583(9)         0.1767(2)         0.44907(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.44903(2)         57(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         57(1)           C27         0.3525(14)         0.1580(3)         0.546(3)         87(2)           Campound 2a · MeOH (1:)          10         0.2968(2)         0.6529(6)         0.66607(2)         42(1)           C31         0.4688(2)         0.5531(6)         0.7581(2)         41(1)           C32         0.4858(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5598(2)         0.6657(8)         0.9313(3)         57(1)           C34         0.6098(2)         0.6457(8)         0.9313(3) <td>C15</td> <td>0.0455(10)</td> <td>0.4084(2)</td> <td>0.1757(3)</td> <td>71(1)</td> <td></td>	C15	0.0455(10)	0.4084(2)	0.1757(3)	71(1)	
C17         0.1812(20)         0.5206(3)         0.1168(4)         111(3)           C21         0.23235(6)         0.2149(3)         0.3052(3)         67(1)           C22         0.5198(8)         0.2149(3)         0.3052(3)         67(1)           C24         0.4583(9)         0.1767(2)         0.4507(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.240(3)         59(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:)           71(1)         71(1)           C1         0.2968(2)         0.4903(6)         0.6697(2)         42(1)           C31         0.4688(2)         0.5511(6)         0.7581(2)         41(1)           C32         0.4488(2)         0.5531(6)         0.5781(2)         41(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         57(1)           C34         0.6998(2)         0.4677(8)         0.9313(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.818(8)         61(1)           C36 <td>C16</td> <td>0.0674(8)</td> <td>0.3393(2)</td> <td>0.1884(3)</td> <td>59(1)</td> <td></td>	C16	0.0674(8)	0.3393(2)	0.1884(3)	59(1)	
C21         0.3253(6)         0.223(2)         0.2690(2)         44(1)           C22         0.5198(8)         0.2249(3)         0.352(3)         67(1)           C23         0.5850(8)         0.2166(3)         0.3946(3)         71(1)           C24         0.4883(9)         0.1767(2)         0.4507(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound <b>2a</b> · MeOH (1:1)         0.2968(2)         0.4903(6)         0.6494(2)         39(1)           C31         0.4688(2)         0.5531(6)         0.7581(2)         41(1)           C32         0.44858(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.64677(8)         0.9358(3)         67(1)           C35         0.533(3)         0.3159(7)         0.8518(3)         61(1)           C35         0.533(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.5618(6)         0.5526(2)         45(1)      <	C17	0.1812(20)	0.5206(3)	0.1168(4)	111(3)	
C22         0.5198(8)         0.2349(3)         0.3052(3)         67(1)           C33         0.5580(8)         0.2166(3)         0.3946(3)         71(1)           C24         0.4583(9)         0.1767(2)         0.4507(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         59(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:)           71(1)         66607(2)         42(1)           C1         0.2968(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5631(6)         0.7581(2)         41(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.6677(8)         0.9358(3)         61(1)           C35         0.5935(3)         0.3159(7)         0.8430(2)         42(1)           C35         0.5935(3)         0.511         0.4562(2)         45(1)           C36         0.5230(2)         0.5654(2)         56(1)           C37	C21	0.3253(6)	0.2123(2)	0.2690(2)	44(1)	
C23         0.5850(8)         0.2166(3)         0.3946(3)         71(1)           C24         0.4853(9)         0.1767(2)         0.4507(2)         60(1)           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         59(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound <b>2a</b> · MeOH (1:1)         0.2968(2)         0.459(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5531(6)         0.7531(2)         41(1)           C32         0.4885(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.4677(8)         0.9313(3)         51(1)           C35         0.5936(3)         0.3159(7)         0.8430(2)         56(1)           C35         0.5936(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.5618(6)         0.5552(2)         41(1)           C11         0.2228(2)         0.5618(6)         0.5552(2)         41(1)	C22	0.5198(8)	0.2349(3)	0.3052(3)	67(1)	
C24         0.4583(9)         0.1767(2)         0.4507(2)         6011           C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         59(1)           Carpound 2a · MeOH (1:)         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:)         0.2968(2)         0.4903(6)         0.6607(2)         42(1)           C1         0.2968(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5313(7)         0.8430(2)         47(1)           C32         0.44588(2)         0.6657(8)         0.9313(3)         55(1)           C33         0.5558(2)         0.6657(8)         0.9358(3)         57(1)           C34         0.6098(2)         0.4677(8)         0.9358(3)         57(1)           C35         0.5230(2)         0.3581(6)         0.5526(2)         41(1)           C36         0.3230(2)         0.2500         0.6422(2)         45(1)           C36         0.3230(2)         0.5618(6)         0.5526(2)         41(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)	C23	0.5850(8)	0.2166(3)	0.3946(3)	71(1)	
C25         0.2604(10)         0.1559(2)         0.4149(3)         71(1)           C26         0.1960(8)         0.1720(2)         0.3240(3)         59(1)           C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:1)         0.2968(2)         0.4903(6)         0.6494(2)         39(1)           C1         0.2968(2)         0.6259(6)         0.6607(2)         41(1)           C31         0.4688(2)         0.5531(6)         0.7581(2)         41(1)           C32         0.4858(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5558(2)         0.6677(8)         0.9338(3)         57(1)           C34         0.6098(2)         0.4677(8)         0.9338(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.7628(3)         61(1)           C36         0.5320(2)         0.3632(7)         0.7628(3)         53(1)           C44         0.4352(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C14         0.0721(2)         0.4786(8)         0.4310(2)         52(1) <t< td=""><td>C24</td><td>0.4583(9)</td><td>0.1767(2)</td><td>0.4507(2)</td><td>60(1)</td><td></td></t<>	C24	0.4583(9)	0.1767(2)	0.4507(2)	60(1)	
C26         0.1960(8)         0.1720(2)         0.324(3)         99(1)           C37         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:1)          1         0.2968(2)         0.4903(6)         0.6494(2)         39(1)           C1         0.2968(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4888(2)         0.5631(6)         0.7581(2)         41(1)           C32         0.4858(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.46677(8)         0.9358(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           C44         0.3174(1)         0.2500         0.6422(2)         45(1)           C5         0.4352(2)         0.5638(6)         0.5556(2)         56(1)           C11         0.2295(3)         0.7641(7)         0.470(3)         58(1)           C12         0.2295(3)         0.7641(7)         0.470(2)         52(1)           C14         0.078(2)         0.428(8)         0.4310(2) <td< td=""><td>C25</td><td>0.2604(10)</td><td>0.1559(2)</td><td>0.4149(3)</td><td>71(1)</td><td></td></td<>	C25	0.2604(10)	0.1559(2)	0.4149(3)	71(1)	
C27         0.5325(14)         0.1580(3)         0.5468(3)         87(2)           Compound 2a · MeOH (1:1)	C26	0.1960(8)	0.1720(2)	0.3240(3)	59(1)	
Compound 2a · MeOH (1:1)         C1       0.2968(2)       0.4903(6)       0.6494(2)       39(1)         C2       0.3948(2)       0.6629(6)       0.6607(2)       42(1)         C31       0.4688(2)       0.733(7)       0.8430(2)       47(1)         C32       0.44588(2)       0.7133(7)       0.8430(2)       47(1)         C33       0.5558(2)       0.6657(8)       0.9313(3)       55(1)         C34       0.6098(2)       0.4667(8)       0.9358(3)       57(1)         C35       0.5935(3)       0.3159(7)       0.818(3)       61(1)         C36       0.5230(2)       0.3632(7)       0.7628(3)       53(1)         C44       0.3174(1)       0.2500       0.6422(2)       45(1)         C5       0.43352(2)       0.5618(6)       0.5556(2)       56(1)         C11       0.2229(2)       0.5618(6)       0.5556(2)       56(1)         C14       0.0782(2)       0.6802(7)       0.3760(2)       52(1)         C15       0.0721(2)       0.4788(8)       0.4310(2)       52(1)         C14       0.1699(2)       0.7249(1)       0.5172(2)       47(1)         C15       0.0721(2)       0.4788(8)       0.4310(2)	C27	0.5325(14)	0.1580(3)	0.5468(3)	87(2)	
C1         0.2968(2)         0.4903(6)         0.6494(2)         39(1)           C2         0.3948(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5531(6)         0.7881(2)         41(1)           C32         0.4488(2)         0.657(8)         0.9313(3)         55(1)           C33         0.5558(2)         0.6657(8)         0.9338(3)         57(1)           C34         0.6098(2)         0.4677(8)         0.9338(3)         53(1)           C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           C44         0.3174(1)         0.2500         0.6422(2)         45(1)           C5         0.4332(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(2)         52(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.4498(2)         0.4217(7)         0.5172(2)         47(1)	Compound 2a · MeO	PH (1:1)				
C2         0.3948(2)         0.6259(6)         0.6607(2)         42(1)           C31         0.4688(2)         0.5631(6)         0.7581(2)         41(1)           C32         0.4858(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.4677(8)         0.9358(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           O4         0.3174(1)         0.2500         0.6422(2)         45(1)           O5         0.4352(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.478(8)         0.4310(2)         52(1)           C16         0.1428(2)         0.4278(8)         0.4310(2)         55(1)           C21	C1	0.2968(2)	0.4903(6)	0.6494(2)	39(1)	
C31       0.4688(2)       0.5631(6)       0.7581(2)       41(1)         C32       0.4858(2)       0.7133(7)       0.8430(2)       47(1)         C33       0.5558(2)       0.6657(8)       0.9313(3)       55(1)         C34       0.6098(2)       0.4677(8)       0.9358(3)       57(1)         C35       0.5935(3)       0.3159(7)       0.8518(3)       61(1)         C36       0.5230(2)       0.3632(7)       0.7628(3)       53(1)         C44       0.3174(1)       0.2500       0.6422(2)       45(1)         C5       0.4352(2)       0.5618(6)       0.5526(2)       41(1)         C12       0.2295(3)       0.7641(7)       0.4970(3)       58(1)         C13       0.1576(3)       0.8207(7)       0.4109(3)       62(1)         C14       0.0782(2)       0.6802(7)       0.3760(2)       52(1)         C15       0.0721(2)       0.4788(8)       0.4310(2)       52(1)         C16       0.1428(2)       0.4201(7)       0.5172(2)       47(1)         C17       0.003(3)       0.7459(12)       0.2812(3)       71(2)         C21       0.2048(2)       0.7270(6)       0.7675(3)       50(1)         C22	C2	0.3948(2)	0.6259(6)	0.6607(2)	42(1)	
C32         0.4858(2)         0.7133(7)         0.8430(2)         47(1)           C33         0.5558(2)         0.6657(8)         0.9313(3)         55(1)           C34         0.6098(2)         0.4677(8)         0.9338(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           O4         0.3174(1)         0.2500         0.6422(2)         45(1)           C5         0.4352(2)         0.5678(6)         0.5556(2)         56(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.47878(8)         0.4310(2)         52(1)           C16         0.1428(2)         0.4201(7)         0.5172(2)         47(1)           C17         0.0033(3)         0.7459(12)         0.2812(3)         71(2)           C21	C31	0.4688(2)	0.5631(6)	0.7581(2)	41(1)	
C33       0.5558(2)       0.6657(8)       0.9313(3)       55(1)         C34       0.6098(2)       0.4677(8)       0.9358(3)       57(1)         C35       0.5535(3)       0.3159(7)       0.8518(3)       61(1)         C36       0.5230(2)       0.3632(7)       0.7628(3)       53(1)         O4       0.3174(1)       0.2500       0.6422(2)       45(1)         O5       0.4352(2)       0.5618(6)       0.5556(2)       41(1)         C12       0.2295(3)       0.7641(7)       0.4970(3)       58(1)         C13       0.1576(3)       0.8207(7)       0.4109(3)       62(1)         C14       0.0721(2)       0.6802(7)       0.3760(2)       52(1)         C15       0.0721(2)       0.4788(8)       0.4310(2)       52(1)         C16       0.1428(2)       0.4201(7)       0.5172(2)       47(1)         C17       0.0033(3)       0.7459(12)       0.2812(3)       71(2)         C21       0.2482(2)       0.5237(6)       0.7505(2)       40(1)         C22       0.2048(2)       0.7270(6)       0.7675(3)       50(1)         C23       0.1649(2)       0.5942(8)       0.9372(2)       55(1)         C25	C32	0.4858(2)	0.7133(7)	0.8430(2)	47(1)	
C34         0.6098(2)         0.4677(8)         0.9358(3)         57(1)           C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           O4         0.3174(1)         0.2500         0.6422(2)         45(1)           O5         0.4352(2)         0.5873(6)         0.5526(2)         56(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.4788(8)         0.4310(2)         52(1)           C16         0.1428(2)         0.4201(7)         0.5172(2)         47(1)           C17         0.0033(3)         0.7459(12)         0.2812(3)         71(2)           C21         0.2522(2)         0.5237(6)         0.7505(2)         40(1)           C22         0.2048(2)         0.7270(6)         0.7675(3)         50(1)           C23         0.1649(2)         0.5494(8)         0.9372(2)         55(1)           C24	C33	0.5558(2)	0.6657(8)	0.9313(3)	55(1)	
C35         0.5935(3)         0.3159(7)         0.8518(3)         61(1)           C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           O4         0.3174(1)         0.2500         0.6422(2)         45(1)           O5         0.4352(2)         0.5873(6)         0.5656(2)         56(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.4201(7)         0.5172(2)         47(1)           C17         0.0033(3)         0.7459(12)         0.2812(3)         71(2)           C21         0.2522(2)         0.5237(6)         0.7505(2)         40(1)           C22         0.2048(2)         0.7270(6)         0.7675(3)         50(1)           C23         0.1649(2)         0.5942(8)         0.9372(2)         55(1)           C25         0.2176(2)         0.3569(7)         0.8286(2)         48(1)           C27	C34	0.6098(2)	0.4677(8)	0.9358(3)	57(1)	
C36         0.5230(2)         0.3632(7)         0.7628(3)         53(1)           O4         0.3174(1)         0.2500         0.6422(2)         45(1)           O5         0.4352(2)         0.5873(6)         0.5656(2)         56(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.2295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.4788(8)         0.4310(2)         52(1)           C16         0.1428(2)         0.4201(7)         0.5172(2)         47(1)           C17         0.0033(3)         0.7459(12)         0.2812(3)         71(2)           C21         0.2048(2)         0.7270(6)         0.7675(3)         50(1)           C22         0.2048(2)         0.7201(6)         0.7675(3)         56(1)           C24         0.1699(2)         0.594(8)         0.9372(2)         55(1)           C25         0.2176(2)         0.3922(8)         0.9202(3)         56(1)           C27	C35	0.5935(3)	0.3159(7)	0.8518(3)	61(1)	
O4 $0.31/4(1)$ $0.2500$ $0.6422(2)$ $45(1)$ O5 $0.4352(2)$ $0.5873(6)$ $0.5656(2)$ $56(1)$ C11 $0.2228(2)$ $0.5618(6)$ $0.5552(2)$ $41(1)$ C12 $0.2295(3)$ $0.7641(7)$ $0.4970(3)$ $58(1)$ C13 $0.1576(3)$ $0.8207(7)$ $0.4199(3)$ $62(1)$ C14 $0.0782(2)$ $0.6602(7)$ $0.3760(2)$ $52(1)$ C15 $0.0721(2)$ $0.4788(8)$ $0.4310(2)$ $52(1)$ C16 $0.1428(2)$ $0.4201(7)$ $0.5172(2)$ $47(1)$ C17 $0.0033(3)$ $0.7459(12)$ $0.2812(3)$ $71(2)$ C21 $0.2522(2)$ $0.5237(6)$ $0.7505(2)$ $40(1)$ C22 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C33 $0.1649(2)$ $0.7610(8)$ $0.8393(3)$ $56(1)$ C44 $0.1699(2)$ $0.5942(8)$ $0.9202(3)$ $56(1)$ C25 $0.2176(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ CCompound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C3 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C36	0.5230(2)	0.3632(7)	0.7628(3)	53(1)	
OS         0.4352(2)         0.3873(6)         0.5656(2)         56(1)           C11         0.2228(2)         0.5618(6)         0.5526(2)         41(1)           C12         0.22295(3)         0.7641(7)         0.4970(3)         58(1)           C13         0.1576(3)         0.8207(7)         0.4109(3)         62(1)           C14         0.0782(2)         0.6802(7)         0.3760(2)         52(1)           C15         0.0721(2)         0.44788(8)         0.4310(2)         52(1)           C16         0.1428(2)         0.4201(7)         0.5172(2)         47(1)           C17         0.0033(3)         0.7459(12)         0.2812(3)         71(2)           C21         0.2522(2)         0.5237(6)         0.7505(2)         40(1)           C22         0.2048(2)         0.7270(6)         0.7675(3)         50(1)           C23         0.1649(2)         0.5942(8)         0.9372(2)         55(1)           C24         0.1699(2)         0.3922(8)         0.9202(3)         56(1)           C25         0.2176(2)         0.3924(8)         0.9202(3)         56(1)           C26         0.2585(2)         0.3669(7)         0.82286(2)         48(1)           C27<	04	0.3174(1)	0.2500	0.6422(2)	45(1)	
C11 $0.2228(2)$ $0.3618(6)$ $0.3520(2)$ $41(1)$ C12 $0.2295(3)$ $0.7641(7)$ $0.4970(3)$ $58(1)$ C13 $0.1576(3)$ $0.8207(7)$ $0.4109(3)$ $62(1)$ C14 $0.0782(2)$ $0.6802(7)$ $0.3760(2)$ $52(1)$ C15 $0.0721(2)$ $0.4788(8)$ $0.4310(2)$ $52(1)$ C16 $0.1428(2)$ $0.4201(7)$ $0.5172(2)$ $47(1)$ C17 $0.0033(3)$ $0.7459(12)$ $0.2812(3)$ $71(2)$ C21 $0.2522(2)$ $0.5237(6)$ $0.7505(2)$ $40(1)$ C22 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C23 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3322(8)$ $0.9202(3)$ $56(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ Compound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.3948(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	05	0.4352(2)	0.5873(6)	0.5656(2)	55(1)	
C12 $0.2295(3)$ $0.7641(7)$ $0.4370(3)$ $36(1)$ C13 $0.1576(3)$ $0.8207(7)$ $0.4109(3)$ $62(1)$ C14 $0.0782(2)$ $0.6802(7)$ $0.3760(2)$ $52(1)$ C15 $0.0721(2)$ $0.4788(8)$ $0.4310(2)$ $52(1)$ C16 $0.1428(2)$ $0.4201(7)$ $0.5172(2)$ $47(1)$ C17 $0.0033(3)$ $0.7459(12)$ $0.2812(3)$ $71(2)$ C21 $0.2522(2)$ $0.5237(6)$ $0.7505(2)$ $40(1)$ C22 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C23 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ Compound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	CII	0.2228(2)	0.7641(7)	0.5526(2)	41(1)	
C13 $0.1376(3)$ $0.5207(7)$ $0.4709(3)$ $0.217$ C14 $0.0782(2)$ $0.6802(7)$ $0.3760(2)$ $52(1)$ C15 $0.0721(2)$ $0.4788(8)$ $0.4310(2)$ $52(1)$ C16 $0.1428(2)$ $0.4201(7)$ $0.5172(2)$ $47(1)$ C17 $0.0033(3)$ $0.7459(12)$ $0.2812(3)$ $71(2)$ C21 $0.2522(2)$ $0.5237(6)$ $0.7505(2)$ $40(1)$ C22 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C23 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ CC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.2101(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C12 C12	0.2295(3)	0.207(7)	0.4970(3)	50(1) (2(1)	
C14 $0.0782(2)$ $0.000(2)$ $0.370(2)$ $0.371(2)$ C15 $0.0721(2)$ $0.4788(8)$ $0.4310(2)$ $52(1)$ C16 $0.1428(2)$ $0.4201(7)$ $0.5172(2)$ $47(1)$ C17 $0.0033(3)$ $0.7459(12)$ $0.2812(3)$ $71(2)$ C21 $0.2522(2)$ $0.5237(6)$ $0.7505(2)$ $40(1)$ C22 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C23 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ CC10.03665(36) $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C13	0.1576(3)	0.6207(7)	0.4109(3)	52(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C14 C15	0.0782(2)	0.0002(7)	0.3700(2)	52(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C15 C16	0.0721(2) 0.1428(2)	0.4700(0)	0.5172(2)	47(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C10 C17	0.0033(3)	0.4201(7) 0.7459(12)	0.3172(2) 0.2812(3)	71(7)	
C11 $0.2048(2)$ $0.7270(6)$ $0.7675(3)$ $50(1)$ C23 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ Compound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C21	0.2522(2)	0.5237(6)	0.7505(2)	40(1)	
CL2 $0.1649(2)$ $0.7610(8)$ $0.8593(3)$ $56(1)$ C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ CCC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C21 C22	0.2048(2)	0.7270(6)	0.7675(3)	50(1)	
C24 $0.1699(2)$ $0.5942(8)$ $0.9372(2)$ $55(1)$ C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ Compound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C23	0.1649(2)	0.7610(8)	0.8593(3)	56(1)	
C25 $0.2176(2)$ $0.3922(8)$ $0.9202(3)$ $56(1)$ C26 $0.2585(2)$ $0.3569(7)$ $0.8286(2)$ $48(1)$ C27 $0.1251(4)$ $0.6323(11)$ $1.0369(3)$ $79(1)$ O6 $0.3809(2)$ $0.2648(6)$ $0.4217(2)$ $65(1)$ C7 $0.3177(3)$ $0.3134(10)$ $0.3254(3)$ $72(2)$ Compound 3aC1 $0.03665(36)$ $0.24701(10)$ $0.17363(13)$ $28(1)$ C2 $0.19295(37)$ $0.23220(9)$ $0.09697(13)$ $27(1)$ C31 $0.21001(40)$ $0.17239(10)$ $0.07602(14)$ $30(1)$ C32 $0.39448(48)$ $0.14452(12)$ $0.09922(18)$ $41(1)$	C24	0.1699(2)	0.5942(8)	0.9372(2)	55(1)	
$\begin{array}{cccccc} \hline C26 & 0.2585(2) & 0.3569(7) & 0.8286(2) & 48(1) \\ C27 & 0.1251(4) & 0.6323(11) & 1.0369(3) & 79(1) \\ O6 & 0.3809(2) & 0.2648(6) & 0.4217(2) & 65(1) \\ C7 & 0.3177(3) & 0.3134(10) & 0.3254(3) & 72(2) \\ \hline \\ Compound 3a \\ \hline \\ C1 & 0.03665(36) & 0.24701(10) & 0.17363(13) & 28(1) \\ C2 & 0.19295(37) & 0.23220(9) & 0.09697(13) & 27(1) \\ C31 & 0.21001(40) & 0.17239(10) & 0.07602(14) & 30(1) \\ C32 & 0.39448(48) & 0.14452(12) & 0.09922(18) & 41(1) \\ \hline \end{array}$	C25	0.2176(2)	0.3922(8)	0.9202(3)	56(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C26	0.2585(2)	0.3569(7)	0.8286(2)	48(1)	
O6         0.3809(2)         0.2648(6)         0.4217(2)         65(1)           C7         0.3177(3)         0.3134(10)         0.3254(3)         72(2)           Compound 3a         C1         0.03665(36)         0.24701(10)         0.17363(13)         28(1)           C2         0.19295(37)         0.23220(9)         0.09697(13)         27(1)           C31         0.21001(40)         0.17239(10)         0.07602(14)         30(1)           C32         0.39448(48)         0.14452(12)         0.09922(18)         41(1)	C27	0.1251(4)	0.6323(11)	1.0369(3)	79(1)	
C70.3177(3)0.3134(10)0.3254(3)72(2)Compound 3aC10.03665(36)0.24701(10)0.17363(13)28(1)C20.19295(37)0.23220(9)0.09697(13)27(1)C310.21001(40)0.17239(10)0.07602(14)30(1)C320.39448(48)0.14452(12)0.09922(18)41(1)	O6	0.3809(2)	0.2648(6)	0.4217(2)	65(1)	
Compound 3aC10.03665(36)0.24701(10)0.17363(13)28(1)C20.19295(37)0.23220(9)0.09697(13)27(1)C310.21001(40)0.17239(10)0.07602(14)30(1)C320.39448(48)0.14452(12)0.09922(18)41(1)	C7	0.3177(3)	0.3134(10)	0.3254(3)	72(2)	
C10.03665(36)0.24701(10)0.17363(13)28(1)C20.19295(37)0.23220(9)0.09697(13)27(1)C310.21001(40)0.17239(10)0.07602(14)30(1)C320.39448(48)0.14452(12)0.09922(18)41(1)	Compound <b>3a</b>					
C2         0.19295(37)         0.23220(9)         0.09697(13)         27(1)           C31         0.21001(40)         0.17239(10)         0.07602(14)         30(1)           C32         0.39448(48)         0.14452(12)         0.09922(18)         41(1)	C1	0.03665(36)	0.24701(10)	0.17363(13)	28(1)	
$ \begin{array}{cccc} C31 & 0.21001(40) & 0.17239(10) & 0.07602(14) & 30(1) \\ C32 & 0.39448(48) & 0.14452(12) & 0.09922(18) & 41(1) \\ \end{array} $	C2	0.19295(37)	0.23220(9)	0.09697(13)	27(1)	
C32 0.39448(48) 0.14452(12) 0.09922(18) 41(1)	C31	0.21001(40)	0.17239(10)	0.07602(14)	30(1)	
	C32	0.39448(48)	0.14452(12)	0.09922(18)	41(1)	

Atom	<i>x</i>	у	Z	Ueq	
C33	0.41223(61)	0.08937(13)	0.07962(23)	53(1)	
C34	0.24695(64)	0.06216(12)	0.03862(23)	55(1)	
C35	0.06423(54)	0.09005(13)	0.01555(21)	50(1)	
C36	0.04574(43)	0.14514(11)	0.03260(17)	39(1)	
O4	-0.17337(23)	0.23008(7)	0.15104(10)	28(1)	
O5	0.12004(24)	0.25889(7)	0.01696(9)	28(1)	
C11	0.04352(38)	0.30903(10)	0.18632(13)	29(1)	
C12	0.23104(42)	0.33608(12)	0.20779(21)	45(1)	
C13	0.23456(43)	0.39198(13)	0.21771(22)	47(1)	
C14	0.05353(41)	0.42364(11)	0.20606(15)	35(1)	
C15	-0.13189(44)	0.39657(11)	0.18444(18)	40(1)	
C16	0.13764(39)	0.34027(11)	0.17440(16)	35(1)	
C17	0.06486(49)	0.48574(12)	0.21547(21)	45(1)	
C18	0.19841(69)	0.50821(13)	0.13790(30)	67(1)	
C19	0.16994(65)	0.50068(15)	0.30365(27)	65(1)	
C20	-0.15691(53)	0.51193(13)	0.21313(26)	56(1)	
C21	0.09567(36)	0.21783(10)	0.26034(14)	28(1)	
C22	0.30297(41)	0.21594(14)	0.29293(18)	46(1)	
C23	0.34942(45)	0.19214(15)	0.37496(18)	47(1)	
C24	0.19382(41)	0.16920(10)	0.42738(15)	31(1)	
C25	-0.01112(41)	0.16816(11)	0.39156(18)	37(1)	
C26	0.05810(38)	0.19192(10)	0.31030(16)	33(1)	
C27	0.24564(46)	0.14574(12)	0.51919(17)	41(1)	
C281	0.06436(138)	0.11693(65)	0.56206(62)	85(4)	
C291	0.41653(136)	0.09668(26)	0.49947(44)	64(2)	
C301	0.35443(202)	0.18513(34)	0.57322(40)	80(4)	
C282#	0.12269(158)	0.18443(39)	0.58999(38)	63(3)	
C292#	0.47251(115)	0.15102(51)	0.54720(50)	72(3)	
C302#	0.15322(344)	0.09219(39)	0.53218(73)	88(5)	

TABLE IV (Continued)

<sup>a</sup>Ueq =  $(1/3)\Sigma$  [Uij · a<sub>i</sub>\* · a<sub>j</sub> · a<sub>i</sub> · a<sub>j</sub> · cos(a<sub>i</sub>, a<sub>j</sub>)] × 10<sup>3</sup>.

pp = 0.55(2), #pp = 0.45(2).

corresponding difference Fourier maps and were included isotropically in the last cycles of refinement. Some hydrogen atoms of the disordered *t*butyl group were kept fixed during the last cycles of refinement. Most of the calculations were carried out with the XRAY80 System [26] on a Vax 6410 computer. The final atomic coordinates are reported in Table IV. The atomic scattering factors were taken from the International Tables for XRay Crystallography [27].

#### Supplementary Material

Lists of the structure factors, atomic coordinates and thermal components for the nonhydrogen atoms and hydrogen atom parameters are available from the authors (CF).

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