

## Packing considerations of bis-dimedone derivatives

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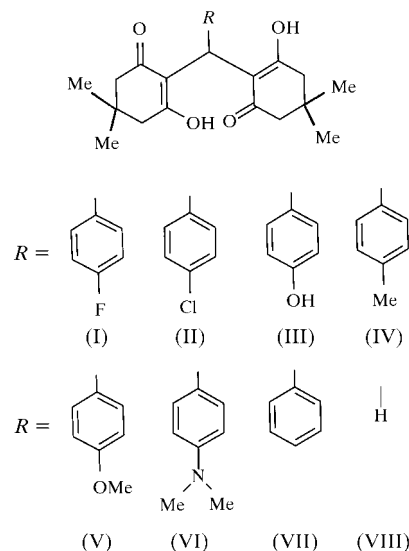
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We have determined the crystal structures of 2,2'-(4-fluorophenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{23}H_{27}FO_4$ , (I), 2,2'-(4-chlorophenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{23}H_{27}ClO_4$ , (II), 2,2'-(4-hydroxyphenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{23}H_{28}O_5$ , (III), 2,2'-(4-methylphenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{24}H_{30}O_4$ , (IV), 2,2'-(4-methoxyphenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{24}H_{30}O_5$ , (V), and 2,2'-(4-*N,N'*-dimethylaminophenyl)methylenebis(3-hydroxy-5,5-dimethyl-2-cyclohexen-1-one),  $C_{25}H_{33}NO_4$ , (VI). Structures (III) to (VI) of these bis-dimedone derivatives show nearly the same packing pattern irrespective of the different substituent in the *para* position of the aromatic ring. However, (II) does not fit into this scheme, although the Cl atom is a substituent not too different from the others. The different packing of the fluoro compound, (I), can be explained by the fact that it crystallizes with two molecules in the asymmetric unit, which show a different conformation of the dimedone ring. On the other hand, (I) shows a similar packing pattern to bis(2-hydroxy-4,4-dimethyl-6-oxo-1-cyclohexenyl)phenylmethane, a compound containing an aromatic ring without any substituent and with  $Z' = 2$ .

### Comment

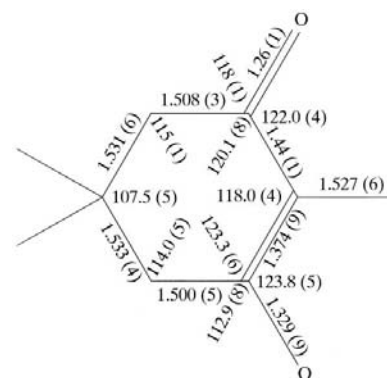
The introduction of area detectors has led to a significant reduction in the time needed for data collection and, as a result, to an increase in the number of determined structures. In the course of this development, problem structures, especially twins, have attracted the interest of crystallographers (Herbst-Irmer & Sheldrick, 1998). Some years ago, we accidentally encountered the twinned crystal structures of two similar compounds bearing the bis-dimedone moiety, namely bis(2-hydroxy-4,4-dimethyl-6-oxo-1-cyclohexenyl)phenylmethane, (VII) (Bolte *et al.*, 1997), and 2,2'-methylenebis(3-hydroxy-5,5-dimethylcyclohex-2-en-1-one), (VIII) (Bolte & Scholtysik, 1997). Since these compounds can be prepared easily (Hünig *et al.*, 1979), we decided to synthesize various derivatives with the objective of finding out if these are also

twinned. Unfortunately, none of these structures turned out to be twinned. On the other hand, a closer inspection of the determined structures revealed that their packing patterns are characterized by striking similarities. Thus, we present in this paper a comparison of compounds containing the bis-dimedone moiety, (I)–(VI).



The main geometric feature of all eight compounds (see Scheme above) is that two strong intramolecular hydrogen bonds are formed (Table 1) connecting the two cyclohexenone rings and forming two new eight-membered rings. Average values for bond lengths and angles of the dimedone moiety of the six structures in this paper and the two already published show very small standard deviations (Fig. 1). The C–C single bond between the carbonyl bond and the C=C double bond is significantly shortened, while the other bonds show normal values. Comparing these structures with 2,2'-ethylenebis(1,3-cyclohexanedione) (Bertolasi *et al.*, 1996) reveals the influence of the two methyl groups on the geometry of the ring. In the latter, the two C–C bonds adjacent to the methyl groups are shortened to a mean value of 1.498 (5) Å, whereas the angle between these two bonds is increased to 110.0 (6)°.

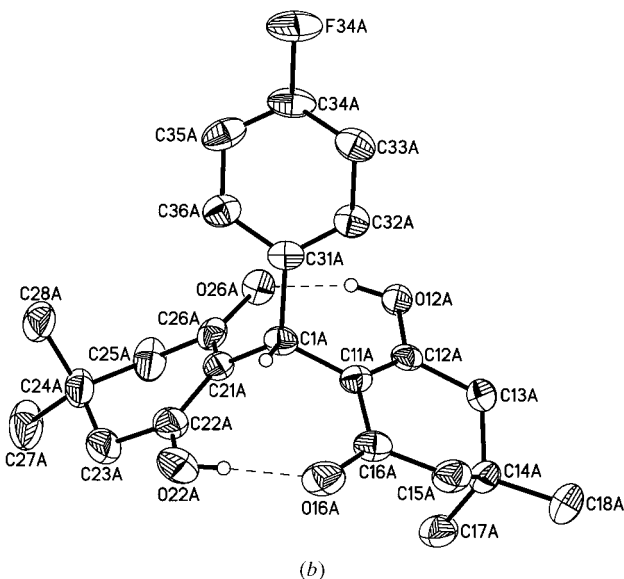
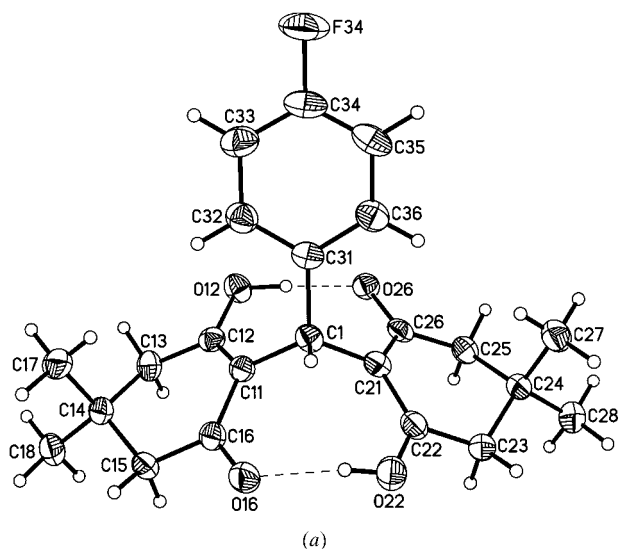
Compounds (I) (Figs. 2a and 2b) and (VII) are very similar. The only difference is the F atom in the *para* position of the



**Figure 1**  
Mean bond lengths (Å) and angles (°) in the dimedone moiety with s.u.'s in parentheses.

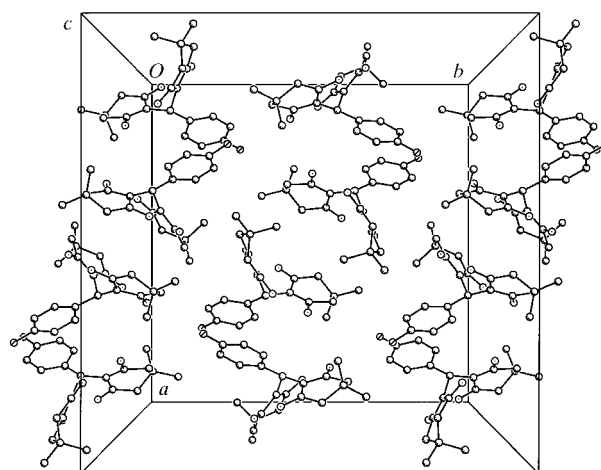
aromatic ring. Both compounds crystallize with two molecules in the asymmetric unit. All four cyclohexenone rings in (I) and (VII) display envelope conformations, but the difference between them is that in three of the four rings, the C atom carrying two methyl groups is directed towards the aromatic ring, but in one case it is bent away from the aromatic residue. A least-squares fit of all non-H atoms excluding the fluorine substituents of the two corresponding molecules in the asymmetric unit of (I) and (VII) (r.m.s. deviation = 0.092 and 0.132 Å, respectively) reveals that the substitution by an F atom does not cause any major difference in the conformation of the molecule. The cell parameters of both crystal structures look rather similar and both compounds crystallize in an I-centred tetragonal space group, but whereas for (VII) the centrosymmetric space group  $I4_1/a$  was found, (I) shows the non-centrosymmetric space group  $I4_1cd$ , thus, not only the

space groups, but also the Laue groups are different. But in spite of the different space groups, the packing pattern of both compounds is almost identical (Figs. 3 and 4).



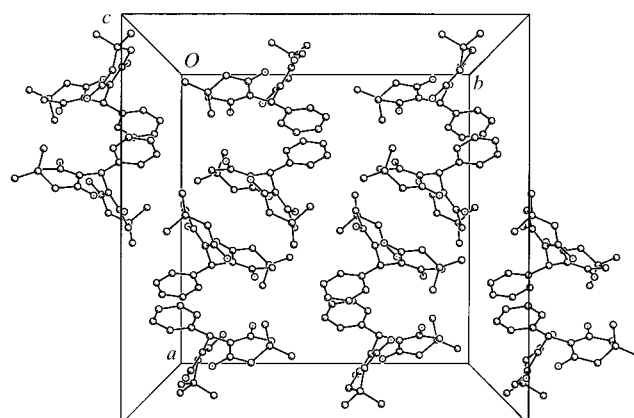
**Figure 2**

Perspective view of (a) molecule 1 and (b) molecule 2 of (I) with the atomic numbering; displacement ellipsoids are at the 50% probability level.



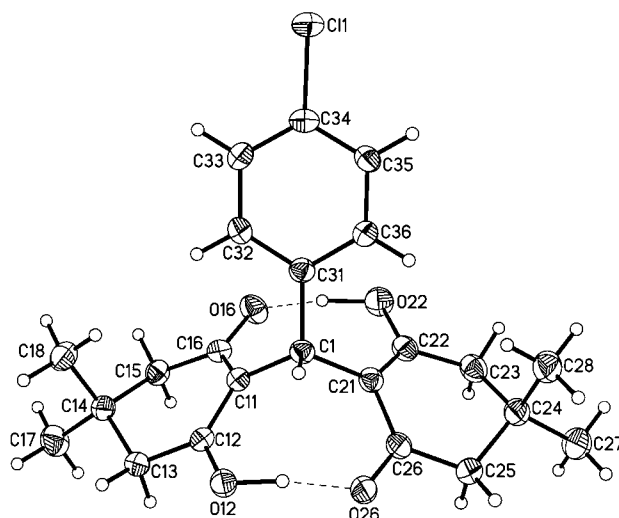
**Figure 3**

Packing diagram of (I); the view is onto the  $bc$  plane.



**Figure 4**

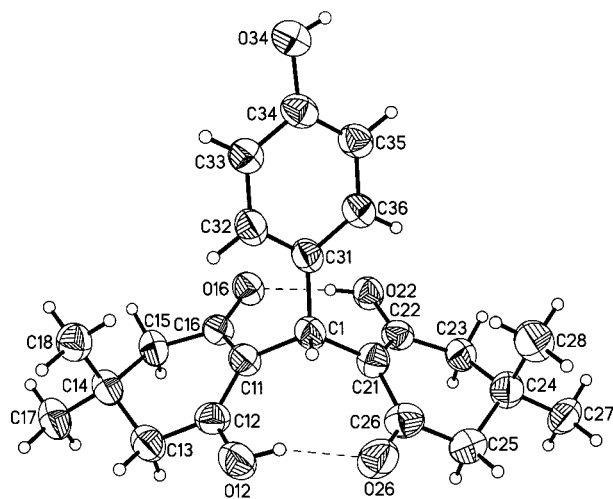
Packing diagram of (VII); the view is onto the  $bc$  plane.



**Figure 5**

Perspective view of (II) with the atomic numbering; displacement ellipsoids are at the 50% probability level.

Compound (II) (Fig. 5), whose structure has been determined recently at room temperature (Shi *et al.*, 1998), contains a Cl atom in the *para* position of the phenyl ring. In contrast to (I) and (VII), where one apex of a cyclohexenone ring is bent away from the aromatic ring, in (II) and all remaining structures, all apices are directed towards the aromatic ring. Furthermore, all compounds except (I) and (VII) crystallize with just one molecule in the asymmetric unit. A least-squares fit of all C and O atoms of (II) and molecule 1 of (I) (r.m.s. deviation = 0.146 Å) shows that the exchange of fluorine for chlorine does not change the molecular conformation markedly. On the other hand, the crystal packing of (II) and (I) is completely different. This might be due to the fact that in (I) and (VII) there are two molecules in the asymmetric unit (in which the cyclohexenone rings display a different conformation), whereas  $Z' = 1$  in (II).



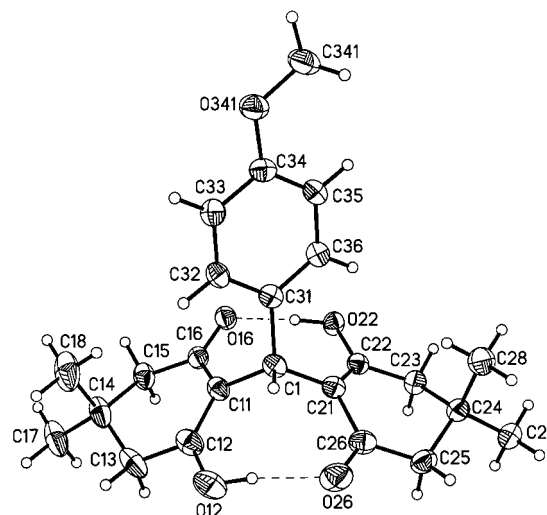
**Figure 6**  
Perspective view of (III) with the atomic numbering; displacement ellipsoids are at the 50% probability level.

In (III) (Fig. 6), the phenyl ring bears a hydroxyl group in the *para* position, which forms an intermolecular hydrogen bond to the hydroxyl O22 atom. As a result, two molecules form a centrosymmetric hydrogen-bonded dimer.

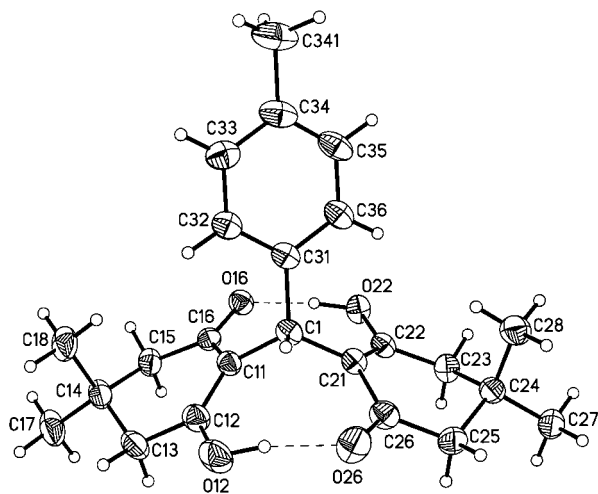
Compound (IV) (Fig. 7) carries a methyl group in the *para* position of the phenyl ring.

Substituting the methyl group in (IV) with a methoxy group leads to (V) (Fig. 8). The torsion angles  $C35-C34-O341-C341$  [ $0.4(2)^\circ$ ] and  $C33-C34-O341-C341$  [ $180.0(2)^\circ$ ] show that the methoxy group lies in the plane of the phenyl ring.

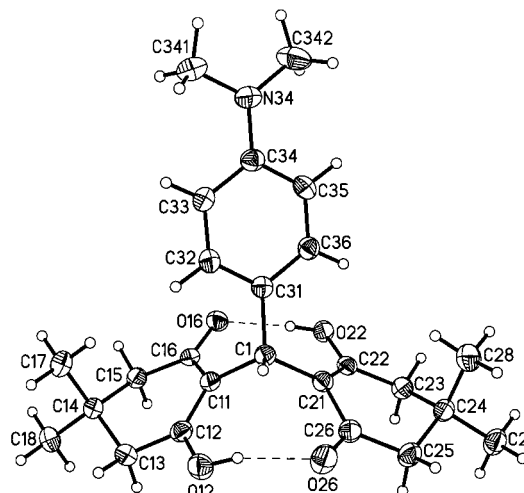
In (VI) (Fig. 9), the phenyl ring carries a dimethylamino group in the *para* position. The environment around the N atom can be described as trigonal planar [ $C34-N34-C342$   $120.3(2)^\circ$ ,  $C34-N34-C341$   $120.2(2)^\circ$  and  $C342-N34-C341$   $118.9(2)^\circ$ ]. Whereas one of the methyl groups lies nearly exactly in the plane of the aromatic ring [ $C33-C34-N34-C341$   $-0.9(3)^\circ$  and  $C35-C34-N34-C341$   $-179.1(2)^\circ$ ], the



**Figure 8**  
Perspective view of (V) with the atomic numbering; displacement ellipsoids are at the 50% probability level.



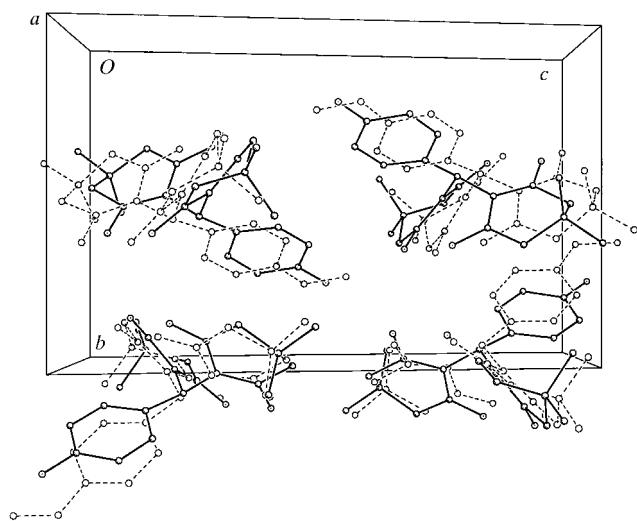
**Figure 7**  
Perspective view of (IV) with the atomic numbering; displacement ellipsoids are at the 50% probability level.



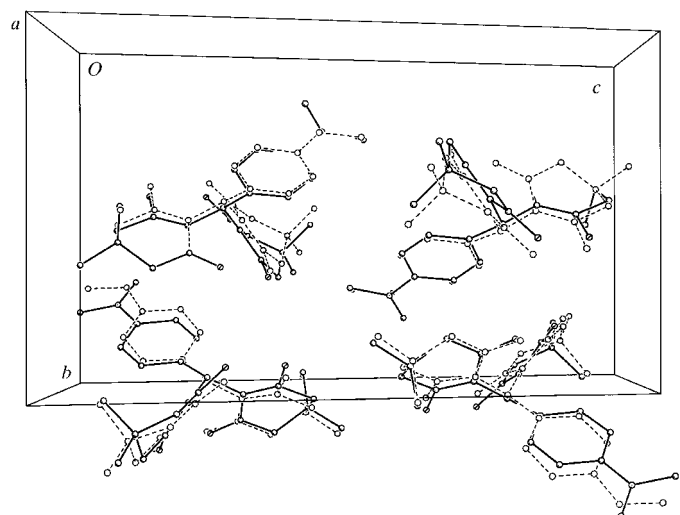
**Figure 9**  
Perspective view of (VI) with the atomic numbering; displacement ellipsoids are at the 50% probability level.

other one is slightly displaced from this plane [C35–C34–N34–C342 10.3 (3)° and C33–C34–N34–C342 –171.5 (2)°].

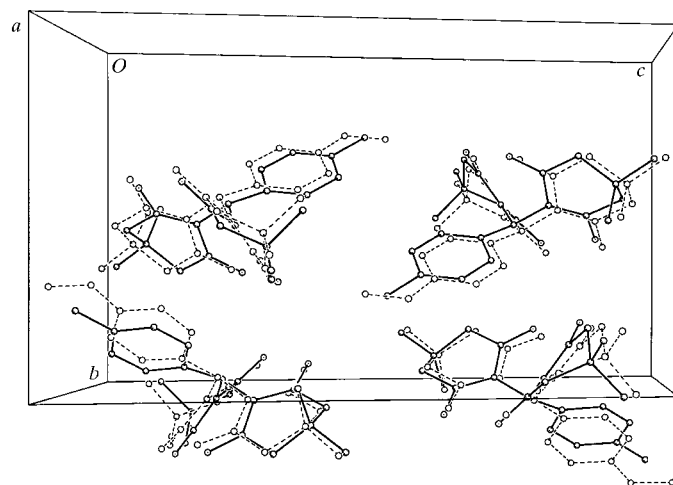
The most remarkable feature of compounds (III) to (VI) is that they display almost the same crystal packing (Figs. 10–12). All molecules crystallize in space group  $P2_1/n$  and their cell parameters are very similar. Even the coordinates of corresponding atoms reveal striking similarities, e.g. the coordinates of (V) and (VI) are nearly equal. The mean and maximum differences between the fractional coordinates are: 0.04 (2), 0.02 (1) and 0.009 (5), and 0.084, 0.042 and 0.019 for  $x$ ,  $y$  and  $z$ , respectively. Figs. 10–12 show a superimposition of the different structures. These packing diagrams demonstrate clearly that the molecules pack in a very similar way. Compound (II), on the other hand, crystallizes in a different way. The only similarity with compounds (III) to (VI) is the



**Figure 10**  
Packing diagram showing the superimposition of the structure of (III) (solid lines) on that of (IV) (broken lines).



**Figure 11**  
Packing diagram showing the superimposition of the structure of (VI) (solid lines) on that of (V) (broken lines).



**Figure 12**  
Packing diagram showing the superimposition of the structure of (IV) (solid lines) on that of (V) (broken lines).

length of the  $b$  axis. Summarizing, it can be said that for compounds (III) to (VI) the main part of the molecules determines the packing pattern, whereas the nature of the substituent in the *para* position of the phenyl ring is only of minor importance. However, compound (II) does not fit into this scheme.

## Experimental

Compounds (I)–(VI) were synthesized according to the methods of Nagarajan & Shenoy (1992) and Cremlyn & Saunders (1993).

### Compound (I)

#### Crystal data

$C_{23}H_{27}FO_4$   
 $M_r = 386.45$   
Tetragonal,  $I4_1cd$   
 $a = 21.186$  (4) Å  
 $c = 36.633$  (1) Å  
 $V = 16443$  (4) Å<sup>3</sup>  
 $Z = 32$   
 $D_x = 1.249$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
Cell parameters from 8192 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.090$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
Block, colourless  
0.55 × 0.40 × 0.25 mm

#### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.952$ ,  $T_{\max} = 0.978$   
97 175 measured reflections  
4615 independent reflections  
3818 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.080$   
 $\theta_{\text{max}} = 27.10^\circ$   
 $h = -27 \rightarrow 27$   
 $k = -27 \rightarrow 27$   
 $l = -46 \rightarrow 46$   
543 standard reflections  
frequency: 1200 min  
intensity decay: none

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.083$   
 $S = 1.046$   
4615 reflections  
521 parameters  
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 9.1605P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.15$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

## Compound (II)

### Crystal data

$C_{23}H_{27}ClO_4$   
 $M_r = 402.90$   
 Monoclinic,  $P2_1/c$   
 $a = 14.892(1) \text{ \AA}$   
 $b = 11.813(1) \text{ \AA}$   
 $c = 13.351(1) \text{ \AA}$   
 $\beta = 115.78(1)^\circ$   
 $V = 2114.9(3) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.265 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 4736 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.206 \text{ mm}^{-1}$   
 $T = 173(2) \text{ K}$   
 Block, colourless  
 $0.54 \times 0.42 \times 0.36 \text{ mm}$

### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.897$ ,  $T_{\max} = 0.930$   
 39 320 measured reflections  
 4667 independent reflections  
 3419 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$   
 $\theta_{\text{max}} = 27.10^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -15 \rightarrow 15$   
 $l = -17 \rightarrow 17$   
 321 standard reflections  
 frequency: 1200 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.100$   
 $S = 1.007$   
 4667 reflections  
 262 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.8901P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.0041 (8)

## Compound (III)

### Crystal data

$C_{23}H_{28}O_5$   
 $M_r = 384.45$   
 Monoclinic,  $P2_1/c$   
 $a = 9.736(2) \text{ \AA}$   
 $b = 11.370(3) \text{ \AA}$   
 $c = 18.263(3) \text{ \AA}$   
 $\beta = 99.54(1)^\circ$   
 $V = 1993.7(7) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.281 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 3615 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.089 \text{ mm}^{-1}$   
 $T = 173(2) \text{ K}$   
 Plate, colourless  
 $0.50 \times 0.20 \times 0.10 \text{ mm}$

### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.957$ ,  $T_{\max} = 0.991$   
 16 535 measured reflections  
 4076 independent reflections  
 1722 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.090$   
 $\theta_{\text{max}} = 26.37^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -14 \rightarrow 12$   
 $l = -22 \rightarrow 22$   
 419 standard reflections  
 frequency: 1000 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.079$   
 $wR(F^2) = 0.226$   
 $S = 1.223$   
 4076 reflections  
 266 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0852P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.009 (2)

## Compound (IV)

### Crystal data

$C_{24}H_{30}O_4$   
 $M_r = 382.48$   
 Monoclinic,  $P2_1/c$   
 $a = 9.339(1) \text{ \AA}$   
 $b = 11.638(1) \text{ \AA}$   
 $c = 20.052(1) \text{ \AA}$   
 $\beta = 102.65(1)^\circ$   
 $V = 2126.5(3) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.195 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 8192 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.080 \text{ mm}^{-1}$   
 $T = 173(2) \text{ K}$   
 Block, colourless  
 $0.70 \times 0.45 \times 0.30 \text{ mm}$

### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.946$ ,  $T_{\max} = 0.976$   
 40 291 measured reflections  
 4868 independent reflections  
 3895 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$   
 $\theta_{\text{max}} = 27.48^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -15 \rightarrow 15$   
 $l = -26 \rightarrow 26$   
 503 standard reflections  
 frequency: 1200 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.111$   
 $S = 1.038$   
 4868 reflections  
 263 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.6993P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.0092 (10)

## Compound (V)

### Crystal data

$C_{24}H_{30}O_5$   
 $M_r = 398.48$   
 Monoclinic,  $P2_1/n$   
 $a = 8.916(2) \text{ \AA}$   
 $b = 11.573(2) \text{ \AA}$   
 $c = 21.106(5) \text{ \AA}$   
 $\beta = 98.15(1)^\circ$   
 $V = 2155.8(8) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.228 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 5837 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.085 \text{ mm}^{-1}$   
 $T = 173(2) \text{ K}$   
 Block, colourless  
 $0.70 \times 0.40 \times 0.30 \text{ mm}$

### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.943$ ,  $T_{\max} = 0.975$   
 40 510 measured reflections  
 4937 independent reflections  
 3429 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$   
 $\theta_{\text{max}} = 27.48^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -15 \rightarrow 15$   
 $l = -27 \rightarrow 27$   
 266 standard reflections  
 frequency: 1200 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.046$   
 $wR(F^2) = 0.114$   
 $S = 1.000$   
 4937 reflections  
 271 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 1.1616P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.0055 (7)

## Compound (VI)

## Crystal data

$C_{25}H_{33}NO_4$   
 $M_r = 411.52$   
 Monoclinic,  $P2_1/n$   
 $a = 9.223$  (1) Å  
 $b = 12.012$  (1) Å  
 $c = 20.661$  (1) Å  
 $\beta = 101.42$  (1)°  
 $V = 2243.6$  (3) Å<sup>3</sup>  
 $Z = 4$

## Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.972$ ,  $T_{\max} = 0.984$   
 35 071 measured reflections  
 4957 independent reflections  
 3598 reflections with  $I > 2\sigma(I)$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.131$   
 $S = 1.029$   
 4957 reflections  
 282 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$D_x = 1.218$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 8192 reflections  
 $\theta = 0-25^\circ$   
 $\mu = 0.082$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Block, colourless  
 $0.35 \times 0.25 \times 0.20$  mm

$R_{\text{int}} = 0.059$   
 $\theta_{\text{max}} = 27.10^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -15 \rightarrow 15$   
 $l = -26 \rightarrow 26$   
 296 standard reflections  
 frequency: 3600 min  
 intensity decay: none

$w = 1/[\sigma^2(F_o^2) + (0.0450P)^2 + 1.8561P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.35$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 0.0043 (8)

All H atoms were located by difference Fourier synthesis and were refined with fixed individual displacement parameters [ $U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$  or  $1.2U_{\text{eq}}(\text{C})$ ] using a riding model with C—H(aromatic) = 0.95, C—H(methyl) = 0.98, C—H(secondary) = 0.99 or C—H(tertiary) = 1.00 Å. The hydroxyl H atoms were refined isotropically. The methyl group attached to the aromatic ring in (IV) and the two methyl groups attached to the N atom in (VI) were allowed to rotate about their local threefold axis. The absolute structure of (I) could not be determined. Friedel opposites were merged and the Flack parameter refined to 0.1 (8).

For all compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP in SHELXTL-Plus* (Sheldrick, 1991).

Table 1

Hydrogen-bonding geometry (Å, °) for compounds (I)–(VI).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
(I)				
O12—H12 $\cdots$ O26	0.95 (4)	1.68 (4)	2.590 (3)	161 (4)
O22—H22 $\cdots$ O16	0.85 (4)	1.81 (4)	2.636 (3)	164 (4)
O12A—H12A $\cdots$ O26A	0.85 (4)	1.77 (4)	2.564 (3)	154 (3)
O22A—H22A $\cdots$ O16A	0.86 (5)	1.85 (5)	2.701 (4)	174 (5)
(II)				
O12—H12 $\cdots$ O26	1.09 (3)	1.50 (3)	2.5845 (16)	169 (3)
O22—H22 $\cdots$ O16	1.17 (4)	1.44 (4)	2.6018 (18)	169 (3)
(III)				
O12—H12 $\cdots$ O26	1.06 (6)	1.66 (6)	2.667 (4)	157 (5)
O22—H22 $\cdots$ O16	0.96 (7)	1.62 (7)	2.561 (4)	165 (6)
O34—H34 $\cdots$ O22 <sup>i</sup>	1.09 (6)	1.84 (5)	2.876 (4)	156 (4)
(IV)				
O12—H12 $\cdots$ O26	0.98 (2)	1.73 (2)	2.6946 (16)	168 (2)
O22—H22 $\cdots$ O16	0.99 (2)	1.60 (2)	2.5714 (13)	168.0 (18)
(V)				
O12—H12 $\cdots$ O26	0.94 (3)	1.79 (3)	2.714 (2)	166 (3)
O22—H22 $\cdots$ O16	0.94 (3)	1.63 (3)	2.5558 (17)	167 (2)
(VI)				
O12—H12 $\cdots$ O26	1.03 (4)	1.67 (4)	2.686 (2)	168 (3)
O22—H22 $\cdots$ O16	1.05 (3)	1.55 (3)	2.5821 (19)	166 (3)

Symmetry code: (i)  $2 - x, 1 - y, 1 - z$ .

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1446). Services for accessing these data are described at the back of the journal.

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