

Exo Diels–Alder adducts between *ortho*- and *para*-*N*-acetoxyphenylmaleimides and furan

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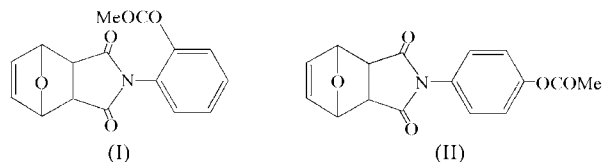
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In *exo*-2-(3,5-dioxo-10-oxa-4-azatricyclo[5.2.1.0^{2,6}]dec-8-en-4-yl)phenyl acetate, C₁₆H₁₃NO₅, the plane of the acetoxy group lies almost perpendicular to that of the phenyl ring [dihedral angle = 89.8 (1)°], in contrast with the smaller deviations found in the *para* isomer *exo*-4-(3,5-dioxo-10-oxa-4-azatricyclo[5.2.1.0^{2,6}]dec-8-en-4-yl)phenyl acetate, C₁₆H₁₃NO₅, these being 63.6 (1) and 37.0 (1)° for the two crystallographically independent molecules. Irrespective of the position of the acetoxy group, both compounds pack through soft C–H⋯X (X is O or phenyl) interactions, forming interlinked centrosymmetric tetramers in the *bc* plane.

Comment

Diels–Alder reactions between furan and maleimide have been well studied (Kwart & King, 1968). However, there are few examples of their crystal structures, particularly of those derived from *N*-arylmaleimides (Cambridge Structural Database, Version of November 2003; Allen, 2002). Diels–Alder reactions produce the *endo* and *exo* isomers, depending on the relative orientation of the approach of the diene and dienophile. Usually, the *exo* adduct is the thermodynamic product



and the *endo* the kinetic one (Kwart & Burchuk, 1951). Control of the adduct stereochemistry has been achieved using chiral *N*-arylmaleimides (Kitagawa *et al.*, 1998). Recently, several efforts have been made to accelerate the rate (Howell *et al.*, 2002) or control the stereochemistry (Philip &

Robertson, 1998) of Diels–Alder cycloadditions, on the basis of molecular recognition or supramolecular chemistry. Here, the molecular and supramolecular structures of the Diels–Alder adducts *exo*-2-(3,5-dioxo-10-oxa-4-azatricyclo[5.2.1.0^{2,6}]dec-8-en-4-yl)phenyl acetate, (I), and its *para* isomer, (II), are reported.

Compounds (I) and (II) crystallize in the monoclinic space groups *P*₂₁/*c* and *P*₂₁/*n*, respectively, and their molecular structures are depicted in Figs. 1 and 2, respectively. The two independent molecules found in the asymmetric unit of (II) are labelled (IIA) and (IIB). The common geometric features of both molecules (Tables 1 and 3) show that the N–C_{aryl} and N–C(=O) bond lengths are in the mean ranges reported for free *N*-arylmaleimides (Miller *et al.*, 2000).

The phenyl ring is twisted relative to the succinimide ring in both adducts, with mean interplanar angles of 65.8 (1), 61.8 (1) and 66.8 (1)° for (I), (IIA) and (IIB), respectively. The torsion angle between these two rings is very similar to the value reported for both a furan–*N*-phenylmaleimide Diels–Alder adduct (Jarosz *et al.*, 2001) and several free *N*-arylmaleimides (Miller *et al.*, 2000). The acetoxy group points towards the oxo bridge and is almost perpendicular to the phenyl ring both in the *ortho* isomer (I) and in the *para* isomer (IIA), whereas in the *para* isomer (IIB) it is close to planarity; the angles between the phenyl and acetoxy planes are 89.8 (1), 63.6 (1) and 37.0 (1)° for (I), (IIA) and (IIB), respectively.

The conformational differences determine the supramolecular structure exhibited by each compound. In the absence of strong hydrogen-bonding donors, the intermolecular geom-

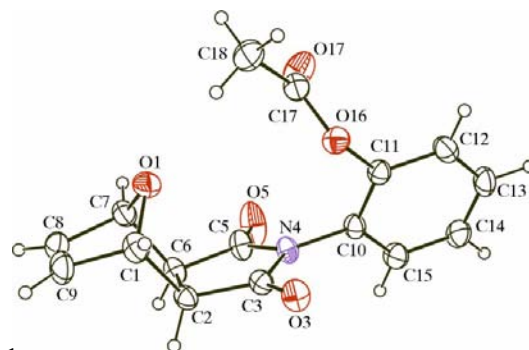


Figure 1

The molecular structure of the *ortho* Diels–Alder adduct, (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

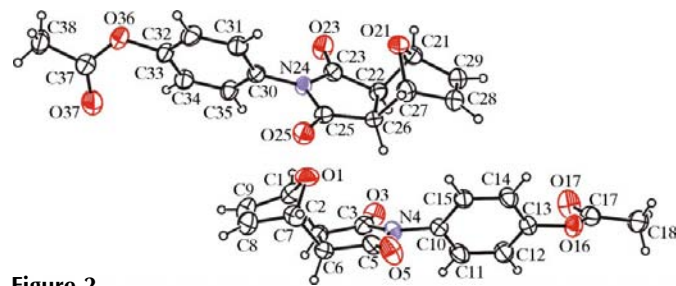


Figure 2

The structure of the two independent molecules in the asymmetric unit of the *para* Diels–Alder adduct, (II), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. Molecule (IIB) is at the top and molecule (IIA) is at the bottom.

etry features weak C—H···X (X is O or phenyl) interactions (Steiner, 2002; Umezawa *et al.*, 1998), which are summarized in Tables 2 and 4 for (I) and (II), respectively. Graph-set notation (Bernstein *et al.*, 1995) is used throughout this paper to describe the hydrogen-bonding patterns.

In compound (I), the interlinked $C_2^2(14)[D_aD_b]$ antiparallel chains zigzagging along the *b* axis are formed through C6—H6A···O16ⁱ (D_a) and C12—H12A···O5ⁱⁱ (D_b) interactions [symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$]. Thus, tetrameric units exhibiting an $R_4^4(28)[D_aD_c]$ ring pattern (Fig. 3) appear *via* interlinking C15—H15A···O17ⁱⁱⁱ (D_c) interactions [symmetry code: (iii) $x, \frac{1}{2} - y, z - \frac{1}{2}$].

The *para* isomer presents a different supramolecular structure, in which sheets of molecules (IIA) and sheets of molecules (IIB) lie in an alternating family of parallel ($\bar{1}0\bar{1}$) and ($\bar{2}02$) planes, respectively. Centrosymmetric dimers of (IIA) are formed through C1—H1A···Ph_A^{ix} [D_i ; C1···Ph_A^{ix} = 3.899 (4) Å and C1—H1A···Ph_A^{ix} = 175°; symmetry code: (ix)

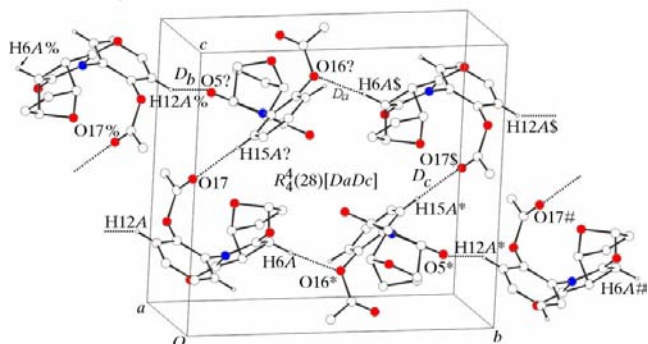


Figure 3
The supramolecular structure of (I), viewed along the *a* axis. The marked atoms are at the symmetry positions: (*) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (#) $x - 1, 1 + y, z$; (\$) $1 - x, 1 - y, 1 - z$; (?) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (%) $2 - x, -y, 1 - z$.

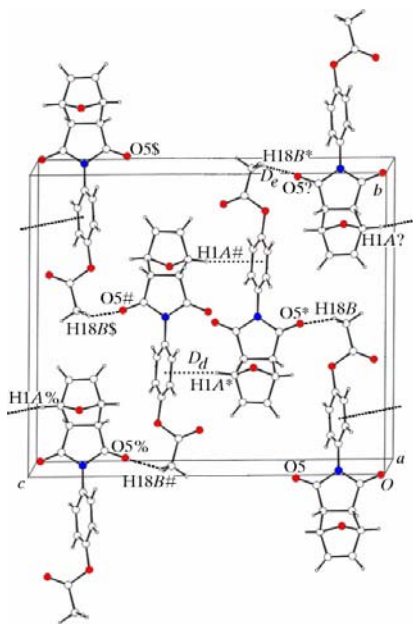


Figure 4
The supramolecular structure of (IIA), viewed along the *a* axis. The marked atoms are at the symmetry positions: (*) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (#) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (\$) $1 - x, 1 - y, 1 - z$; (?) $x, 1 + y, z$; (%) $1 - x, -y, 1 - z$.

$-x, -y, -z$; Ph_A is the phenyl group of molecule (IIA)], which are interlinked through weak C18—H18B···O5^{iv} hydrogen-bond interactions [D_e ; symmetry code: (iv) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$] (Fig. 4). Sheets of (IIB) molecules are composed of interlinked tetrameric units formed *via* C28—H28A···Ph_B^v [D_f ; C28···Ph_B^v = 3.694 (4) Å and C28—H28A···Ph_B^v = 146°; symmetry code: (v) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; Ph_B is the phenyl group of molecule (IIB)], C38—H38B···Ph_B^x [D_g ; C38···Ph_B^x = 3.658 (4) Å and C38—H38B···Ph_B^x = 151°; symmetry code: (x) $2 - x, -y, 1 - z$] and C27—H27A···O37^v (D_h) interactions, which define an $R_6^6(18)$ ring pattern (Fig. 5).

Finally, a three-dimensional network is achieved by the association between molecules (IIA) and (IIB), the former molecule providing most of the acceptors and the latter most of the donors. Thus, zigzag tapes are formed through C14—H14A···O25^{vi} (D_i), C18—H18C···O37^{iv} (D_j), C32—H32A···O5^{vii} (D_k), and C22—H22···O3^{viii} (D_l) interactions [symmetry codes: (vi) $x - 1, y, z$; (vii) $1 + x, y, z$; (viii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$], to conform with the tapes described by the $C_4^4(23)[R_2^2(14)R_2^2(22)]$ hydrogen-bonding pattern (Fig. 6).

In summary, the *exo* Diels–Alder adducts between furan and *ortho*- and *para*-*N*-acetoxyphenylmaleimides, *viz.* (I) and (II), respectively, are characterized by hydrogen-bonded tetrameric structures, which form sheets in the *bc* plane

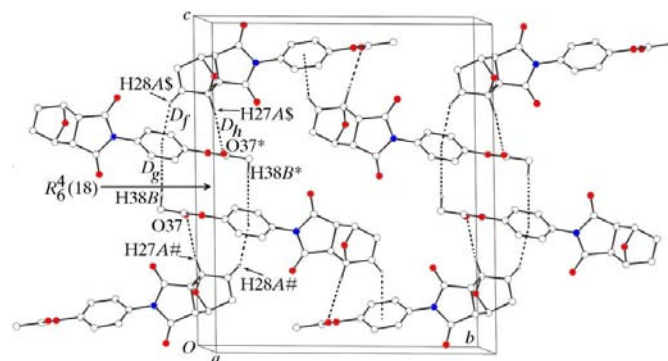


Figure 5
The supramolecular structure of (IIB), viewed along the *a* axis. The marked atoms are at the symmetry positions: (*) $2 - x, -y, 1 - z$; (#) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (\$) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

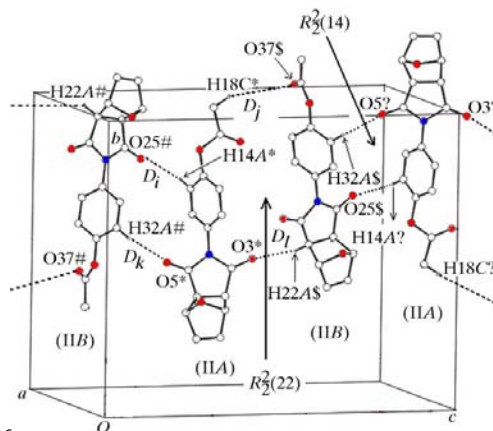


Figure 6
A view along the *a* axis of the molecular tapes formed between the *para* Diels–Alder adducts (IIA) and (IIB). The marked atoms are at the symmetry positions: (*) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (#) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (\$) $1 - x, 1 - y, 1 - z$; (?) $-x, 1 - y, 1 - z$.

through soft C—H...X (X is O or phenyl) interactions. Notably, the interlinked tetrameric units persist when the position of the acetoxy group is altered.

Experimental

Diels–Alder adducts were obtained by the reaction between 1-(2-acetoxyphenyl)-1*H*-pyrrole-2,5-dione [for (I)] or 1-(4-acetoxyphenyl)-1*H*-pyrrole-2,5-dione [for (II)] (1.0 mmol) and furan (2.2 mmol) dissolved in xylene (3 ml) in a sealed ampoule at 413 K for 8 h. The products crystallized as white solids in 73 and 56% yield, respectively. In both cases, crystals suitable for X-ray analysis were obtained from chloroform solutions by slow hexane diffusion. Analysis for (I): colourless crystals, m.p. 448–452 K; IR (KBr, ν , cm^{-1}): 1728, 1766 (C=O), 1592 (C=C), 1384 (OAc), 1310 (C–N); ^1H NMR (270.17 MHz, CDCl_3 , δ): 7.44 (*m*, 1H, H13), 7.32 (*m*, 1H, H14), 7.27 (*m*, 1H, H12), 7.26 (*m*, 1H, H15), 6.56 (*s*, 2H, H8,9), 5.37 (*s*, 2H, H1,7), 3.00 (*s*, 2H, H2,6), 2.19 (*s*, 3H, Me); ^{13}C NMR (67.94 MHz, CDCl_3 , δ): 174.6 (C3,5), 168.4 (C17), 146.3 (C11), 136.7 (C8,9), 130.5 (C13), 129.1 (C15), 126.5 (C14), 124.4 (C10), 123.8 (C12), 81.3 (C1,7), 47.7 (C2,6), 20.6 (Me); MS, *m/z* (%): 299 (2) (M^+), 257 (14), 231 (13), 189 (100), 161 (3), 145 (62). Analysis for (II): colourless crystals, m.p. 438–443 K; IR (KBr, ν , cm^{-1}): 1710, 1758 (C=O), 1600 (C=C), 1366 (OAc), 1310 (C–N); ^1H NMR (270.17 MHz, CDCl_3 , δ): 7.31 (*d*, 2H, H11,15), 7.19 (*d*, 2H, H12,14), 6.53 (*s*, 2H, H8,9), 5.36 (*s*, 2H, H1,7), 2.97 (*s*, 2H, H2,6), 2.29 (*s*, 3H, Me); ^{13}C NMR (67.94 MHz, CDCl_3 , δ): 175.2 (C3,5), 168.8 (C17), 150.4 (C13), 136.6 (C8,9), 129.0 (C11), 127.5 (C11,15), 122.2 (C12,14), 81.3 (C1,7), 47.4 (C2,6), 21.0 (Me); MS, *m/z* (%): 299 (4) (M^+), 257 (6), 231 (13), 189 (100), 161 (5), 145 (7).

Compound (I)

Crystal data

$\text{C}_{16}\text{H}_{13}\text{NO}_5$	$D_x = 1.423 \text{ Mg m}^{-3}$
$M_r = 299.27$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 24 reflections
$a = 8.859 (1) \text{ \AA}$	$\theta = 10\text{--}11^\circ$
$b = 12.830 (2) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 12.615 (1) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 102.971 (2)^\circ$	Square prism, colourless
$V = 1397.2 (3) \text{ \AA}^3$	$0.5 \times 0.3 \times 0.1 \text{ mm}$
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 27.0^\circ$
$\omega/2\theta$ scans	$h = -11 \rightarrow 11$
3024 measured reflections	$k = 0 \rightarrow 16$
3024 independent reflections	$l = 0 \rightarrow 16$
1767 reflections with $I > 2\sigma(I)$	3 standard reflections every 200 reflections
$R_{\text{int}} = 0.012$	intensity decay: 3%

Table 1

Selected geometric parameters (\AA , $^\circ$) for (I).

O16–C11	1.401 (2)	N4–C5	1.392 (2)
O16–C17	1.360 (2)	N4–C10	1.425 (2)
O17–C17	1.189 (2)	C2–C6	1.538 (2)
N4–C3	1.401 (2)	C8–C9	1.309 (3)
C1–O1–C7	96.20 (13)	N4–C10–C11	120.25 (14)
C11–O16–C17	116.68 (13)	N4–C10–C15	120.60 (15)
C3–N4–C5	112.64 (14)	O16–C11–C10	119.28 (14)
C3–N4–C10	123.44 (13)	O16–C11–C12	119.46 (15)
C5–N4–C10	123.92 (14)	O16–C17–O17	122.51 (17)
O1–C7–C6	100.45 (13)	O16–C17–C18	110.80 (15)
O1–C7–C8	101.64 (14)	O17–C17–C18	126.69 (17)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$) for (I).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C6–H6A...O16 ⁱ	0.98	2.58	3.478 (2)	153
C12–H12A...O5 ⁱⁱ	0.93	2.43	3.113 (2)	130
C15–H15A...O17 ⁱⁱⁱ	0.93	2.56	3.243 (2)	131

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

Table 3

Selected geometric parameters (\AA , $^\circ$) for (II).

O1–C1	1.436 (4)	O25–C25	1.208 (3)
O1–C7	1.436 (4)	O36–C33	1.404 (3)
O3–C3	1.207 (4)	O36–C37	1.333 (4)
O5–C5	1.204 (4)	O37–C37	1.185 (5)
O16–C13	1.402 (3)	N4–C5	1.388 (4)
O16–C17	1.365 (4)	N4–C10	1.430 (3)
O17–C17	1.178 (4)	N4–C3	1.386 (4)
O21–C21	1.434 (3)	N24–C23	1.390 (3)
O21–C27	1.445 (3)	N24–C25	1.386 (3)
O23–C23	1.210 (3)	N24–C30	1.429 (3)
C1–O1–C7	95.7 (2)	O16–C13–C14	118.6 (3)
C13–O16–C17	117.3 (2)	O16–C13–C12	120.8 (3)
C21–O21–C27	95.6 (2)	O16–C17–C18	111.3 (3)
C33–O36–C37	122.4 (2)	O16–C17–O17	122.3 (3)
C5–N4–C10	123.0 (2)	O17–C17–C18	126.4 (3)
C3–N4–C5	111.9 (2)	O21–C21–C22	100.3 (2)
C3–N4–C10	124.6 (2)	O21–C21–C29	102.3 (2)
C23–N24–C25	112.3 (2)	O23–C23–N24	123.6 (2)
C23–N24–C30	124.0 (2)	O23–C23–C22	127.2 (2)
C25–N24–C30	123.6 (2)	N24–C23–C22	109.2 (2)
O1–C1–C2	101.0 (2)	O25–C25–N24	124.2 (2)
O1–C1–C9	102.1 (3)	O25–C25–C26	127.1 (2)
N4–C3–C2	108.5 (2)	N24–C25–C26	108.8 (2)
O3–C3–C2	127.0 (3)	O21–C27–C26	100.9 (2)
O3–C3–N4	124.4 (3)	O21–C27–C28	101.5 (2)
O5–C5–N4	123.9 (3)	N24–C30–C31	120.7 (2)
N4–C5–C6	109.4 (2)	N24–C30–C35	119.0 (2)
O5–C5–C6	126.7 (3)	O36–C33–C32	114.8 (2)
O1–C7–C8	102.2 (3)	O36–C33–C34	123.6 (2)
O1–C7–C6	99.0 (2)	O36–C37–O37	122.4 (3)
N4–C10–C15	120.8 (2)	O36–C37–C38	112.9 (3)
N4–C10–C11	119.4 (3)	O37–C37–C38	124.5 (3)

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.127$
 $S = 1.02$
 3024 reflections
 199 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$$

Compound (II)

Crystal data

$\text{C}_{16}\text{H}_{13}\text{NO}_5$	Mo $K\alpha$ radiation
$M_r = 299.27$	Cell parameters from 24 reflections
Monoclinic, $P2_1/n$	$\theta = 11\text{--}12^\circ$
$a = 9.7030 (10) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$b = 15.8560 (10) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 18.8450 (19) \text{ \AA}$	Square prism, colourless
$\beta = 101.962 (5)^\circ$	$0.30 \times 0.25 \times 0.20 \text{ mm}$
$V = 2836.4 (4) \text{ \AA}^3$	
$Z = 8$	
$D_x = 1.402 \text{ Mg m}^{-3}$	

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
C18—H18B...O5 ^{iv}	0.96	2.36	3.277 (4)	159
C27—H27A...O37 ^v	0.98	2.62	3.518 (4)	153
C14—H14A...O25 ^{vi}	0.93	2.61	3.503 (4)	162
C32—H32A...O5 ^{vii}	0.93	2.41	3.289 (4)	158
C22—H22A...O3 ^{viii}	0.98	2.36	3.245 (3)	150
C18—H18C...O37 ^{iv}	0.96	2.54	3.166 (5)	123

Symmetry codes: (iv) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (v) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vi) $x - 1, y, z$; (vii) $1 + x, y, z$; (viii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

Data collection

Enraf–Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
5538 measured reflections
5538 independent reflections
2253 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

$\theta_{\text{max}} = 26.0^\circ$
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 23$
3 standard reflections
every 200 reflections
intensity decay: 3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.147$
 $S = 0.93$
5538 reflections
400 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0646P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.020$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{Å}^{-3}$

H atoms were treated as riding, with C—H distances in the range 0.93–0.98 Å, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for the other H atoms.

For both compounds, data collection: CAD-4 EXPRESS (Enraf–Nonius, 1995); cell refinement: CAD-4 EXPRESS; data reduction: JANA98 (Petříček & Dušek, 1998); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine struc-

ture: SHELXL97 (Sheldrick, 1997); molecular graphics: WinGX (Farrugia, 1999); software used to prepare material for publication: SHELXL97 and RESVIEW (Schwenk-Kircher, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1565). Services for accessing these data are described at the back of the journal.

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