1259 measured reflections
1190 independent reflections

3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.062$
$S = 1.120$
1181 reflections
55 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0267P)^2 + 0.1264P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.310$ e Å^{-3}
$\Delta\rho_{min} = -0.171$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.058 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Si1—C1	1.8469 (15)	Si1—Cl2	2.0283 (6)
C1—C1^{i}	1.536 (3)	Si1—Cl3	2.0225 (6)
Si1—Cl1	2.0229 (6)		
C1—Si1—Cl1	111.44 (6)	Cl1—Si1—Cl3	107.17 (3)
C1—Si1—Cl2	109.80 (5)	Cl2—Si1—Cl3	108.50 (3)
C1—Si1—Cl3	111.27 (6)	C1^{i}—C1—Si1	113.93 (13)
Cl1—Si1—Cl2	108.55 (3)		
Cl1—Si1—C1—C1^{i}	−61.7 (2)	Cl3—Si1—C1—C1^{i}	57.8 (2)
Cl2—Si1—C1—C1^{i}	177.96 (14)		

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

Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1992). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1006). Services for accessing these data are described at the back of the journal.

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1-Aryl-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole Derivatives

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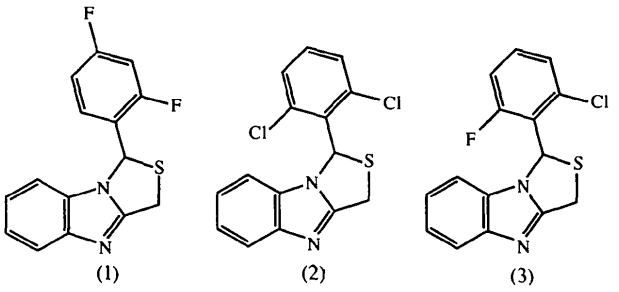
Abstract

The structural features of some 1-aryl-1*H*,3*H*-thiazolo-[3,4-*a*]benzimidazoles active against human immunodefiency virus (HIV) are reported. The diffractometric analysis reveals that the title compounds, 1-(2,4-difluorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $C_{15}H_{10}F_2N_2S$, 1-(2,6-dichlorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $C_{15}H_{10}Cl_2N_2S$, and 1-(2-chloro-6-fluorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, $C_{15}H_{10}ClFN_2S$, all adopt a butterfly-like conformation which appears to be particularly important for the inhibition of HIV-1 reverse transcriptase (RT), an enzyme which plays a key role in the viral life-cycle.

Comment

In previous papers, we reported the synthesis and evaluation of anti-HIV activity of a series of 1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole derivatives (Chimirri, Grasso, Monforte, Monforte & Zappalá, 1991; Monforte *et al.*, 1993; Bruno, Chimirri *et al.*, 1996; Chimirri *et al.*, 1996; Bruno, Monforte, Nicoló & Scopelliti, 1996). Some of the studied compounds exhibited significant activity (Chimirri *et al.*, 1991, 1996) and in particular the 1-(2,6-difluorophenyl)- derivative (TBZ) was found to be a highly potent non-nucleoside HIV-1 reverse transciptase (RT) inhibitor (NNRTI) (Schultz *et al.*, 1992; Buckheit *et al.*, 1993; Bruno, Chimirri *et al.*, 1996). It was recently ascertained (Scafer *et al.*, 1993; Ding *et al.*, 1995) that some structural features, such as two systems arranged in a butterfly-like conformation and a hydrogen-bond acceptor in an appropriate position, are necessary for the inhibition of the RT enzyme. On this basis, it is very important to determine the geometry of thiazolobenzimidazole derivatives, a new class of anti-HIV agents, in order to clarify which molecular requirements are common to NNRTIs. As part of a broader structural and pharmacological study of these compounds, we report here the results of the X-ray



structure determination of 1-(2,4-difluorophenyl)-, (1), 1-(2,6-dichlorophenyl)-, (2), and 1-(2-chloro-6-fluorophenyl)-1*H*,3*H*-thiazolo[3,4-*a*]benzimidazole, (3).

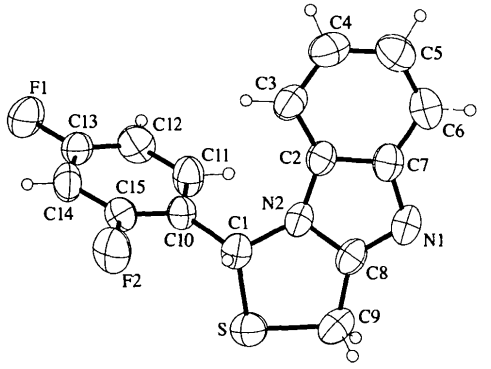


Fig. 1. A view of compound (1) showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level for non-H atoms.

The structure analyses were performed in order to provide information on the effects of the substituent at the aromatic ring bound to C1 on the stereochemistry of the molecules and consequently on anti-HIV activity. All compounds crystallize in centrosymmetric space groups, (1) in $P2_1/c$ with one molecule in the asymmetric unit, (2) in $P\bar{1}$ and (3) in the non-standard space group *I2/a*. Compounds (2) and (3) have two independent molecules in the asymmetric unit (*A* and *B*) (Figs. 2 and 3, respectively), which display opposite absolute configuations with respect to the chiral C atom (the choice is arbitrary). This occurs in compound (2) as a result of close packing, while in compound (3) it is due to molecules *A* and *B* being conformers. All three compounds adopt the already mentioned (Bruno, Monforte *et al.*, 1996) butterfly-like conformation as exhibited by the dihedral angles between the three-fused-ring system and the phenyl substitutent at the chiral C1 atom: 74.22 (5)° in (1), and 78.45 (7) and 76.36 (6), and 85.8 (1) and 84.7 (1)° for molecules *A* and *B* in compounds (2) and (3), respectively. The molecules all show the same geometric features: (*a*) planarity of the benzimidazole system: C9—C8—N1—C7 = −178.4 (2)° for (1), C9*A*—C8*A*—N1*A*—C7*A* = −175.7 (2) and 178.6 (6)°, and C9*B*—C8*B*—N1*B*—C7*B* = 176.9 (2) and 178.8 (6)° for (2) and (3), respectively; (*b*) steric hindrance of the phenyl substitutent at the chiral C atom, which can be seen by comparing the S—C9 and S—C1 bond distances: 1.814 (2) and 1.842 (2) Å for (1), 1.820 (3) and 1.840 (3), and 1.832 (5) and 1.854 (5) Å for molecule *A*

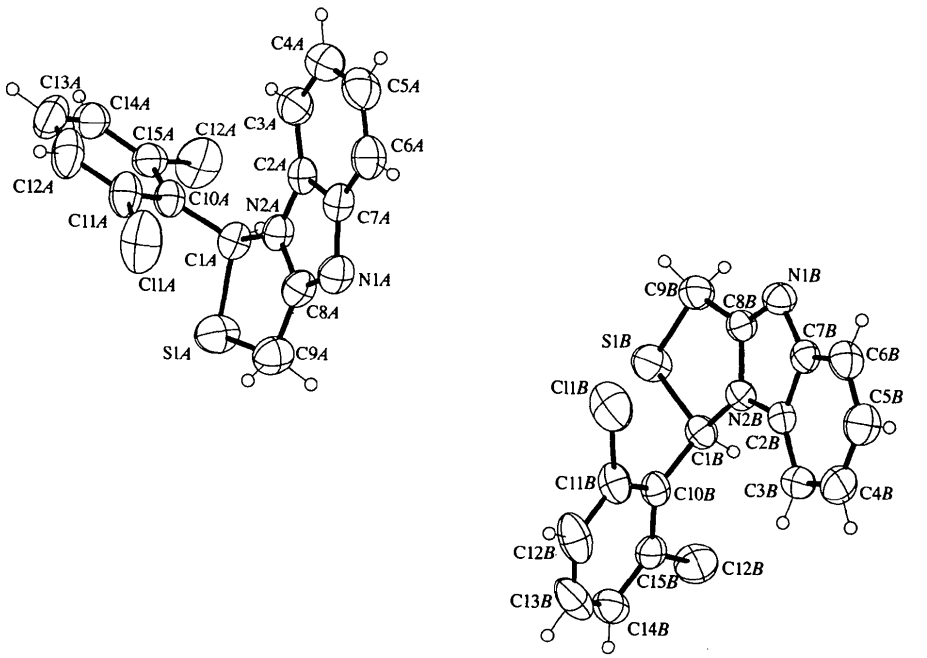


Fig. 2. A view of the asymmetric unit of (2) showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level for non-H atoms.

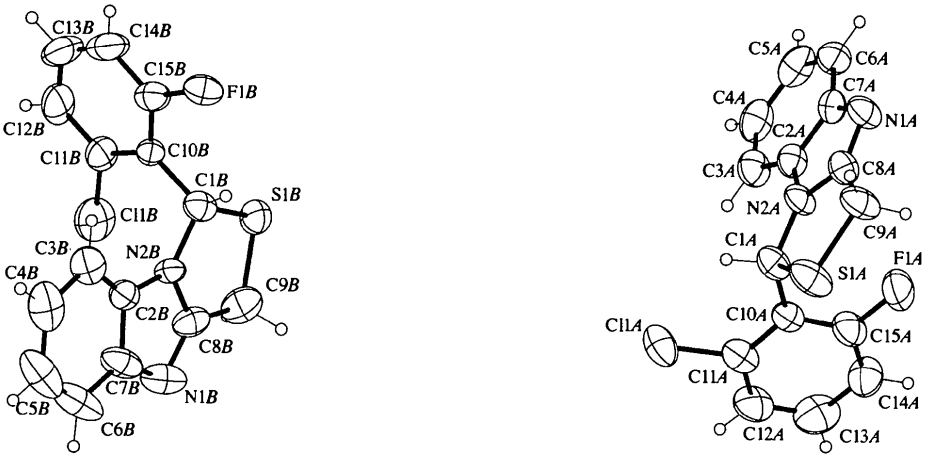


Fig. 3. A view of the asymmetric unit of (3) showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level for non-H atoms.

of compounds (2) and (3), and 1.811 (2) and 1.846 (2), and 1.810 (6) and 1.836 (6) Å for molecule *B* of compounds (2) and (3), respectively. The bond angle C1—S—C9 varies within a small range [from 94.60 (8) for (1) to 96.4 (2)° for molecule *A* of compound (3)] and the five-membered ring (S, C9, C8, N2, C1) tends to assume an intermediate conformation between an envelope and twisted with a pseudo-twofold axis through C8 and the midpoint of S—C1 [Q_T = 0.235 (2), 0.055 (2), 0.120 (2), 0.031 (5) and 0.062 (4), and φ = −12.0 (4), 180 (2), −7 (1), 166 (10) and 161 (5)° for molecules (1), (2*A*), (2*B*), (3*A*) and (3*B*), respectively; Cremer & Pople, 1975], which is more evident in thiazoles (1) and (2*B*).

In the solid state, the molecules display intermolecular and intramolecular interactions involving F or Cl and H atoms. Compound (1) shows only one intramolecular interaction [F2···H1 = 2.39 Å, F2···H1—C1 = 103.3° and F2···C1 = 2.788 (2) Å], compound (2) shows intramolecular [Cl2*A*···H1*A* = 2.47 Å, Cl2*A*···H1*A*—C1*A* = 116° and Cl2*A*···C1*A* = 3.036 (3) Å; Cl2*B*···H1*B* = 2.48 Å, Cl2*B*···H1*B*—C1*B* = 115° and C1*B*···Cl2*B* = 3.026 (3) Å] and intermolecular interactions [N1*A*···H12*B*^{i} = 2.54 Å, N1*A*···H12*B*^{i}—C12*B*^{i} = 176° and N1*A*···C12*B*^{i} = 3.467 (4) Å; N1*B*···H12*A*^{ii} = 2.49 Å, N1*B*···H12*A*^{ii}—C12*A*^{ii} = 170° and N1*B*···C12*A*^{ii} = 3.409 (4) Å; symmetry codes (i) $-x, -y+1, -z+2$; (ii) $-x+1, -y+1, -z+2$], and compound (3) shows intramolecular interactions [Cl1*A*···H1*A* = 2.54 Å, Cl1*A*···H1*A*—C1*A* = 115° and Cl1*A*···C1*A* = 3.080 (5) Å; F1*B*···H1*B* = 2.35 Å, F1*B*···H1*B*—C1*B* = 111° and F1*B*···C1*B* = 2.859 (6) Å] and intermolecular interactions [F1*A*···H3*B*^{iii} = 2.44 Å, F1*A*···H3*B*^{iii}—C3*B*^{iii} = 162° and F1*A*···C3*B*^{iii} = 3.334 (7) Å; symmetry code: (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$]. The results of tests of biological activity of compounds (1), (2) and (3) have been reported elsewhere (Chimirri *et al.*, 1991).

Experimental

The syntheses of title compounds (1), (2) and (3) were carried out according to a previously reported procedure (Chimirri *et al.*, 1991) by a condensation–cyclization reaction of 1,2-phenylenediamine with a suitable aldehyde and 2-mercaptoacetic acid. Crystals suitable for X-ray analysis were grown by slow evaporation of an ethanol solution at room temperature.

Compound (1)

Crystal data

$C_{15}H_{10}F_2N_2S$
M_r = 288.31
Monoclinic
$P2_1/c$
a = 11.507 (2) Å
b = 7.399 (1) Å
c = 15.802 (2) Å
β = 106.83 (1)°
V = 1287.8 (3) Å^3
Z = 4
D_x = 1.487 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 44 reflections
θ = 7.84–18.31°
μ = 0.265 mm^{-1}
T = 293 (2) K
Irregular
0.50 × 0.30 × 0.27 mm
Colourless

Data collection

Siemens *R3m/V* diffractometer
ω–2θ scans
Absorption correction:
ψ scan (Kopfmann & Huber, 1968)
T_{min} = 0.816, T_{max} = 0.893
3074 measured reflections
2274 independent reflections

1905 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.06°
$h = -1 \rightarrow 13$
$k = -1 \rightarrow 8$
$l = -18 \rightarrow 18$
3 standard reflections
every 97 reflections
intensity decay: 1.21%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.090

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.157 e Å^{-3}
$\Delta\rho_{min}$ = −0.214 e Å^{-3}

$S = 1.077$
2274 reflections
182 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.2764P]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0039 (12)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Compound (2)

Crystal data

$C_{15}H_{10}Cl_2N_2S$
$M_r = 321.21$
Triclinic
$P\bar{1}$
$a = 7.740$ (2) Å
$b = 13.030$ (5) Å
$c = 14.694$ (5) Å
$\alpha = 83.30$ (3)°
$\beta = 79.20$ (3)°
$\gamma = 76.72$ (3)°
$V = 1412.5$ (8) Å^3
$Z = 4$
$D_x = 1.510$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 7.17$–$12.99°$
$\mu = 0.596$ mm^{-1}
$T = 293$ (2) K
Irregular
0.36 × 0.18 × 0.14 mm
Yellow

Data collection

Siemens *R3m/V* diffractometer
ω–2θ scans
Absorption correction: none
6205 measured reflections
5000 independent reflections
3709 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 25.05°$
$h = -1 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$
3 standard reflections
every 197 reflections
intensity decay: 1.75%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.074$
4999 reflections
363 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.0608P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.373$ e Å^{-3}
$\Delta\rho_{min} = -0.399$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0031 (11)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Compound (3)

Crystal data

$C_{15}H_{10}ClFN_2S$
$M_r = 304.76$
Monoclinic
$I2/a$
$a = 22.093$ (5) Å
$b = 7.878$ (1) Å
$c = 31.473$ (5) Å
$\beta = 100.28$ (3)°
$V = 5389.9$ (16) Å^3
$Z = 16$
$D_x = 1.502$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 47 reflections
$\theta = 5.73$–$13.74°$
$\mu = 0.439$ mm^{-1}
$T = 293$ (2) K
Prismatic
0.26 × 0.11 × 0.06 mm
Colourless

Data collection

Siemens *R3m/V* diffractometer
ω–2θ scans
Absorption correction: none
9506 measured reflections
4807 independent reflections
2510 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 25.60°$
$h = -25 \rightarrow 19$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 36$
3 standard reflections
every 97 reflections
intensity decay: 3.26%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.214$
$S = 0.905$
4806 reflections
361 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1312P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.411$ e Å^{-3}
$\Delta\rho_{min} = -0.561$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Reflection intensities were evaluated by profile fitting of a 96-steps peak scan with the 2θ shells procedure (Diamond, 1969) and then corrected for Lorentz and polarization effects. Standard deviations $\sigma(I)$ were estimated from counting statistics. An absorption correction was applied only to the structure of (1). All three structures were solved by direct methods and completed by a combination of full-matrix least-squares techniques and difference Fourier maps. All non-H atoms were refined anisotropically. H atoms were placed in idealized positions and allowed to ride on their parent C atoms, with a common isotropic displacement parameter ($U_{iso} = 0.07$ Å^2). The choice of the non-standard *I* lattice type for compound (3) was required in order to erase correlations showed by the standard C lattice type, where the angle β was 118.01 (1)°. All calculations were performed on a μ-VAX 3400 and on a AXP DecStation 3000/400.

For all compounds, data collection: *P3/V Software* (Siemens, 1989); cell refinement: *P3/V Software*; data reduction: *SHELXTL-Plus* (Sheldrick, 1991); program(s) used to solve structures: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *PARST*95 (Nardelli, 1995) and *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1293). Services for accessing these data are described at the back of the journal.

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A Highly Strained Tertiary Alcohol: *anti*-Di(1-adamantyl)[2,5-di(isopropyl)phenyl]-methanol

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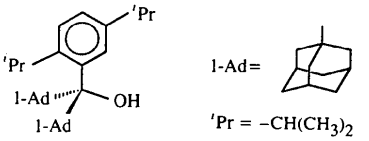
Abstract

Steric interactions between the *ortho* isopropyl group and the adamantyl groups in the title compound, $C_{33}H_{48}O$, result in very large in-plane deformations of the benzene ring.

Comment

Though other aryldi(*tert*-alkyl)methanols have been described, notably 3,4,5-trimethoxyphenyldi(*tert*-butyl)-methanol (van Koningsveld & van Meurs, 1977), *syn*-4-methoxy-2-methylphenyldi(*tert*-butyl)methanol (Hough & Lomas, 1984) and, more recently, the *anti*/*syn* rotamer pair of 3-(*tert*-butyl)phenyldi(1-adamantyl)-methanol (Lomas & Vaissermann, 1996*a*), no *anti* rotamer bearing a substituent in the *ortho* position has been investigated. Though they are substantially less thermodynamically stable than the *syn* isomers, the *anti* isomers are the major product of the addition of *ortho*-alkyl-substituted phenyllithiums to bulky ketones (Lomas, Luong & Dubois, 1977). Rotamer interconversion is, moreover, very slow when large *tert*-alkyl substituents are present (Lomas & Dubois, 1981). We now report an X-ray study of the title compound, (I), the most sterically congested *anti-ortho*-substituted aryldi(*tert*-alkyl)methanol yet synthesized (Lomas & Vaissermann, 1996*b*).



(I)

The main features of this molecule are consistent with data from previous studies of aryldi(1-adamantyl)-methyl derivatives, notably the very large C101—C10—C201 angle subtended by the adamantyl (Ad) groups at the C—OH carbon and the long bonds to this C atom. In a di-1-adamantylmethane derivative, the corresponding bonds are substantially shorter (1.554 Å) but non-bonded interactions are reduced by opening of the Ad—C—Ad angle to 125.0° (Ermer & Bödecker, 1981). Though the C10—O11 bond is not far from the plane of the benzene ring, the two adamantyl groups are quite distinct; that which is further from the benzene plane, in terms of the torsion angles with respect to *ortho* C atoms, is approximately staggered with respect to the C1—C10 bond. The other is about 30° out of the staggered position. Both adamantyl groups are somewhat compressed, the mean of the internal angles at the quaternary C atoms, C101 and C201, being 106.6 (10)°.

The benzene ring is normal as far as planarity is concerned (greatest deviation of any one C atom from the mean plane: 0.012 Å) but shows considerable deformation within the plane, notably, the rather long C1—C2 bond and internal angles ranging from 116.1 (3) to 125.4 (3)°. The external angles subtended by the pair of *ortho* substituents to C1 and C2 are also remarkably large and are comparable to those found in compounds bearing *ortho*-related pairs of *tert*-butyl groups (Stam, 1972; Watanabe, Kawashima, Tokitoh & Okazaki, 1995). Further deformation to reduce steric interaction between the *ortho*-isopropyl and the adamantyl groups includes lengthening of the C1—C10 bond. The