

Refinement

Refinement on *F*Final *R* = 0.0376*wR* = 0.0410*S* = 1.448

1041 reflections

206 parameters

All H-atom parameters refined

Weighting scheme based on measured e.s.d.'s

 $(\Delta/\sigma)_{\max} = 0.0119$ $\Delta\rho_{\max} = 0.10781 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.22802 \text{ e } \text{\AA}^{-3}$ Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN*; *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *TEXSAN FINISH*. Literature survey: *CSSR* (1984).

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
N1	0.3332 (2)	0.0345 (2)	0.1326 (2)	0.0451
C2	0.3523 (3)	0.0062 (3)	0.2462 (3)	0.0550
N3	0.2755 (2)	-0.0239 (2)	0.2654 (2)	0.0560
C4	0.2023 (2)	-0.0136 (2)	0.1587 (3)	0.0446
C5	0.2366 (2)	0.0229 (2)	0.0749 (3)	0.0406
N6	0.3978 (2)	0.0794 (2)	0.0880 (2)	0.0472
C7	0.1046 (3)	-0.0365 (3)	0.1438 (3)	0.0523
N8	0.0256 (2)	-0.0534 (3)	0.1297 (3)	0.0756
N9	0.1951 (2)	0.0493 (2)	-0.0370 (2)	0.0523
C11	0.5176 (2)	0.1769 (3)	-0.0213 (3)	0.0544
C12	0.4595 (3)	0.2186 (3)	0.0404 (3)	0.0608
C13	0.4037 (2)	0.1724 (2)	0.0908 (3)	0.0468
C14	0.3464 (3)	0.2219 (3)	0.1560 (4)	0.0665
O15	0.5637 (2)	0.2409 (2)	-0.0645 (2)	0.0821
O16	0.5260 (2)	0.0955 (2)	-0.0354 (2)	0.0572
C17	0.6245 (4)	0.2079 (3)	-0.1305 (5)	0.0937
C18	0.6557 (7)	0.2923 (6)	-0.1811 (9)	0.1384

Table 2. Geometric parameters (\AA , $^\circ$)

N1—C2	1.368 (4)	C5—N9	1.343 (4)
N1—C5	1.370 (3)	N6—C13	1.360 (4)
N1—N6	1.390 (3)	C11—C12	1.430 (5)
C2—N3	1.294 (4)	C11—O15	1.349 (4)
N3—C4	1.391 (4)	C11—O16	1.211 (4)
C4—C5	1.369 (4)	C12—C13	1.343 (4)
C2—N1—C5	108.4 (2)	N1—N6—C13	120.4 (3)
C2—N1—N6	125.5 (3)	C12—C11—O15	111.0 (3)
C5—N1—N6	125.5 (2)	C11—C12—C13	124.6 (3)
N3—C4—C7	122.5 (3)	N6—C13—C12	122.1 (3)
N1—C5—C4	103.7 (2)		

Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> ... <i>H</i>	<i>H</i> — <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
N6—H6...O16	0.86 (3)	2.21 (3)	2.747 (4)	121 (3)
N6—H6...O16 ⁱ	0.86 (3)	2.19 (3)	2.932 (4)	144 (3)
N9—H9A...N3 ⁱⁱ	0.88 (3)	2.14 (3)	2.992 (4)	164 (3)
N9—H9B...N8 ⁱⁱⁱ	0.92 (4)	2.17 (3)	3.060 (4)	161 (3)

Symmetry codes: (i) 1 - *x*, -*y*, -*z*; (ii) *x*, -*y*, *z* - ½; (iii) -*x*, -*y*, -*x*.

The title compound was prepared by the addition of a catalytic amount of perchloric acid (2 drops) to a suspension of 1,5-

diamino-4-imidazolecarbonitrile (4.07 mmol) in ethyl acetate (1 ml, 7.85 mmol) and ethanol (15 ml). The mixture was stirred for 7 d at room temperature to give the product as a white crystalline solid in 85% (3.48 mmol) yield. The crystals were purified by washing with diethyl ether.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71203 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1047]

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1,3,5-Triacetyl-2,4,6-triphenylbenzene: a Sterically Hindered Molecule

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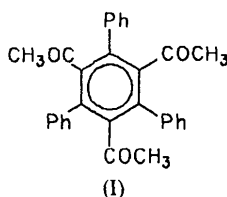
Abstract

Steric congestion has twisted the acetyl and phenyl substituents out of conjugation with the central benzene ring with interplanar angles ranging from 59.5 to 80.2°. The molecular conformation is thus locked in the form of a rotor with two acetyl-O atoms projecting below and one above the benzene plane.

Comment

In the IR spectrum of the title 1,3,5 isomer (I) a single carbonyl stretching vibration at 1700 cm^{-1} is ob-

served. Aromatic ketones (e.g. acetophenone) normally exhibit a band at 1683 cm^{-1} , and observation of the $\nu(\text{CO})$ vibration at the higher frequency suggested that the acetyl groups in the 1,3,5-triacetyltriphenylbenzene were severely twisted out of the plane of the central benzene ring and could not be in conjugation with this ring. Support for this hypothesis came from an examination of the ^{13}C NMR spectrum, which showed the carbonyl-C atom at a chemical shift of 204.3 p.p.m. This is more typical of an aliphatic ketone (cf. acetophenone δ 196.9 p.p.m.).



This structure investigation shows that the title molecule (Fig. 1) is shaped like a rotor with substituent $\text{C}_{\text{benz}}-\text{C}_{\text{benz}}-\text{C}-\text{C}$ torsion angles ranging from $60.0(3)$ to $79.8(3)^\circ$. A similar situation exists in hexaphenylbenzene (Bart, 1968); however, the torsion angles in hexamethyl benzenecarboxylate (Romero, Vegas, Santos & Lopez de Lerma, 1987) show, on average, a smaller deviation from planarity [$\text{C}-\text{C}-\text{C}=\text{O}$ range $46(1)-66.5(8)^\circ$]. Further evidence of the greater twist needed to accommodate phenyl substituents comes from 1,3,5-triphenylbenzene (Lin & Williams, 1975) and 1,3,5-triacetylbenzene (O'Connor, 1973; O'Connor & Moore, 1973), where an average interplanar angle of 38° in the former contrasts with an average torsion angle of 8.8° in the latter.

An acetyl group would, therefore, be expected to experience less overlap and shielding from a neighbouring acetyl than a phenyl substituent, which could explain the marked difference in chemical reactivity between the 1,2,4 and the 1,3,5 isomers. For example, attempts to re-

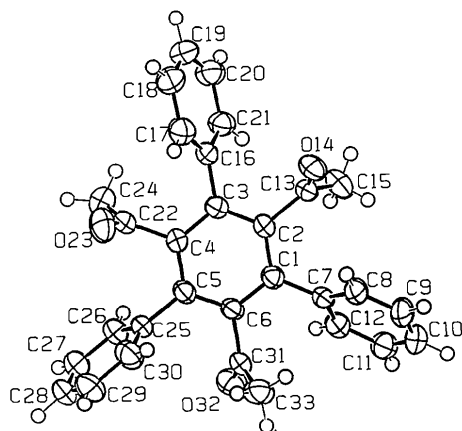


Fig. 1. The title molecule, including atomic numbering scheme, drawn using ORTEPII (Johnson, 1976).

duce a 3:1 mixture of the 1,2,4 and 1,3,5 isomers to the corresponding triols by reaction with lithium aluminium hydride in refluxing tetrahydrofuran over two days gave only recovered starting material, indicating that the carbonyl groups are shielded towards nucleophilic attack. When a similar mixture was heated with *n*-butyllithium in tetrahydrofuran only the 1,2,4 isomer reacted, presumably to form an enolate. The 1,3,5 isomer was unaffected and was recovered free from the 1,2,4 isomer after the reaction.

Experimental

Crystal data

$\text{C}_{30}\text{H}_{24}\text{O}_3$
 $M_r = 432.52$
 Triclinic
 $P\bar{1}$
 $a = 10.368(2)\text{ \AA}$
 $b = 15.595(3)\text{ \AA}$
 $c = 7.470(2)\text{ \AA}$
 $\alpha = 101.65(1)^\circ$
 $\beta = 97.74(2)^\circ$
 $\gamma = 78.94(2)^\circ$
 $V = 1155.1(4)\text{ \AA}^3$
 $Z = 2$

$D_x = 1.243\text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\lambda = 0.71069\text{ \AA}$
 Cell parameters from 21 reflections
 $\theta = 31.23-39.33^\circ$
 $\mu = 0.0738\text{ mm}^{-1}$
 $T = 296\text{ K}$
 Cube
 $0.40 \times 0.40 \times 0.40\text{ mm}$
 Colourless

Data collection

AFC-6S diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 empirical
 $T_{\text{max}} = 0.92$
 4319 measured reflections
 4074 independent reflections
 2749 observed reflections
 $[F > 3\sigma(F)]$

$R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 25.04^\circ$
 $h = 0 \rightarrow 12$
 $k = -18 \rightarrow 18$
 $l = -8 \rightarrow 8$
 3 standard reflections monitored every 150 reflections
 intensity variation: 0.4%

Refinement

Refinement on F
 Final $R = 0.0473$
 $wR = 0.0495$
 $S = 1.688$
 2749 reflections
 395 parameters
 All H-atom parameters refined
 Weighting scheme based on measured e.s.d.'s
 $(\Delta/\sigma)_{\text{max}} = 0.0553$

$\Delta\rho_{\text{max}} = 0.18675\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18675\text{ e \AA}^{-3}$
 Extinction correction:
 Zachariasen type 2 Gaussian isotropic
 Extinction coefficient:
 $32(5) \times 10^{-7}$
 Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structural Corporation, 1988). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: TEXSAN PROCESS (Molecular Structural Corporation, 1985). Program(s) used to solve structure: TEXSAN; MITHRIL (Gilmore, 1984). Program(s) used to refine structure: TEXSAN LS. Molecular graphics: TEXSAN; ORTEPII (Johnson, 1976). Software used to prepare material for publication: TEXSAN FINISH.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
O14	0.5472 (2)	0.3763 (1)	-0.0494 (2)	0.0538
O23	0.9604 (2)	0.2414 (1)	0.5088 (3)	0.0641
O32	0.4332 (2)	0.1238 (1)	0.5279 (3)	0.0610
C1	0.4883 (2)	0.2469 (2)	0.2384 (3)	0.0384
C2	0.5495 (2)	0.3166 (2)	0.2174 (3)	0.0382
C3	0.6685 (2)	0.3317 (2)	0.3205 (3)	0.0399
C4	0.7292 (2)	0.2741 (2)	0.4408 (3)	0.0390
C5	0.6698 (2)	0.2042 (2)	0.4636 (3)	0.0386
C6	0.5491 (2)	0.1918 (2)	0.3625 (3)	0.0375
C7	0.3608 (2)	0.2295 (1)	0.1289 (3)	0.0376
C8	0.3516 (3)	0.2097 (2)	-0.0617 (3)	0.0460
C9	0.2343 (3)	0.1900 (2)	-0.1608 (4)	0.0564
C10	0.1279 (3)	0.1890 (2)	-0.0721 (4)	0.0578
C11	0.1348 (3)	0.2088 (2)	0.1157 (4)	0.0543
C12	0.2513 (3)	0.2291 (2)	0.2167 (4)	0.0461
C13	0.4881 (2)	0.3757 (2)	0.0790 (3)	0.0410
C15	0.3582 (3)	0.4334 (2)	0.1113 (5)	0.0592
C16	0.7280 (2)	0.4112 (2)	0.3133 (3)	0.0416
C17	0.8535 (3)	0.4033 (2)	0.2621 (4)	0.0578
C18	0.9087 (3)	0.4787 (3)	0.2698 (5)	0.0736
C19	0.8391 (4)	0.5618 (3)	0.3277 (5)	0.0724
C20	0.7147 (4)	0.5706 (2)	0.3733 (4)	0.0617
C21	0.6584 (3)	0.4957 (2)	0.3655 (3)	0.0498
C22	0.8589 (3)	0.2873 (2)	0.5547 (3)	0.0434
C24	0.8527 (3)	0.3564 (2)	0.7238 (4)	0.0598
C25	0.7336 (2)	0.1439 (2)	0.5959 (3)	0.0409
C26	0.7225 (3)	0.1713 (2)	0.7814 (4)	0.0519
C27	0.7799 (3)	0.1152 (3)	0.9038 (5)	0.0675
C28	0.8457 (3)	0.0331 (3)	0.8424 (6)	0.0752
C29	0.8578 (3)	0.0046 (2)	0.6587 (6)	0.0745
C30	0.8012 (3)	0.0606 (2)	0.5348 (5)	0.0602
C31	0.4845 (2)	0.1168 (2)	0.3896 (3)	0.0420
C33	0.4893 (4)	0.0370 (2)	0.2421 (5)	0.0628

Table 2. Geometric parameters (\AA , $^\circ$)

C1—C2	1.402 (3)	C3—C16	1.501 (3)
C1—C6	1.397 (3)	C4—C5	1.398 (3)
C1—C7	1.498 (3)	C4—C22	1.517 (3)
C2—C3	1.398 (3)	C5—C6	1.398 (3)
C2—C13	1.519 (3)	C5—C25	1.504 (3)
C3—C4	1.403 (3)	C6—C31	1.514 (3)
C2—C1—C6	118.9 (2)	C3—C4—C5	120.9 (2)
C1—C2—C3	120.8 (2)	C4—C5—C6	118.9 (2)
C2—C3—C4	119.1 (2)	C1—C6—C5	121.3 (2)

Sample preparation involved refluxing a hexane solution (25 cm^{-3}) of 4-phenylbut-3-yn-2-one (0.13 mol) and a catalytic amount of (η^5 -indenyl)bis(ethylene)rhodium (0.58 mmol) for 6 h under an atmosphere of argon. The product, which was a mixture containing 1,2,4-triacetyltriphenylbenzene and 1,3,5-triacetyltriphenylbenzene (3:1 ratio), precipitated from solution as a yellow solid in 76% yield. Repeated washing with diethyl ether removed the 1,2,4 isomer, and repeated recrystallization of the residual 1,3,5 isomer from dry acetone, followed by dry flash chromatography (silica GF₂₅₄ Fluka, eluents hexane/ethyl acetate) gave crystals of the pure 1,3,5 isomer.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71209 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1046]

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Structure of 3-{2-[4-(6-Fluoro-1,2-benzisoxazol-3-yl)piperidino]ethyl}-6,7,8,9-tetrahydro-2-methyl-4H-pyrido[1,2-a]pyrimidin-4-one (Risperidone)

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Abstract

The benzisoxazole and pyrimidine moieties are essentially planar and the dihedral angle between these planes is $5.6 (1)^\circ$. The piperidine ring is in a slightly distorted chair conformation, while the tetrahydropyridine moiety shows a half-chair conformation. The crystal structure is stabilized by a hydrogen bond between the H atom on position 4 of the benzisoxazole and the O atom of the pyrimidinone of a translated ($x, y-1, z$) molecule [$C \cdots O = 3.372 (7)$, $H \cdots O = 2.327 \text{ \AA}$, $C-H \cdots O = 161.6^\circ$].

Comment

Risperidone is an antipsychotic drug with serotonin- S_2 and dopamine- D_2 antagonistic properties (Janssen *et al.*, 1988). The determination was undertaken to compare the structural features with other known serotonin and dopamine antagonists in the hope of obtaining a better in-