$\Sigma \Delta F$(obs.)ΔF(calc.) = 10025(29), enantiomorph $\Sigma \Delta F$(obs.) $\times \Delta F$(calc.) = $-9742(29)$ on 1104 Bijvoet pairs. The results indicate that the model corresponds to the correct configuration, with the same $2R,3R$ configuration for the tartaric moiety as that of the tartaric acid used in the synthesis.

Througout this paper, the averaged values are means weighted according to the reciprocals of the largest 'external' and internal standard deviations (Topping, 1960).

The calculations were carried out on the ENCORE-GOULD-POWERNODE 6040 computer of the 'Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma)'. In addition to the quoted programs, *LQPARM* (Nardelli & Mangia, 1984), *PARST* (Nardelli, 1983a), and *ORTEP* (Johnson, 1965) were also used.

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Structure and Conformation of 1'-Phenyl-indan-2-spiro-4'-piperidin-1-one

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Abstract

The title compound was obtained, amongst other products, by the condensation of quinuclidinone enolate with bromobenzene in the presence of the $NaNH_2$.'BuONa complex base. X-ray analysis proved the only way of successfully identifying its structure. Its formation suggests a new mechanism of arynic condensation. The spiro addition of piperidine to the indanone system does not modify the geometry of the two moieties significantly. It is possible that some degree of conjugation between the N lone pair and the phenyl π system determines the conformation about the N—Ph bond, which itself seems mainly influenced by packing interactions.

Comment

The condensation of ketone enolates with aryl halides in the presence of $NaNH_2.R$ONa complex bases is now a well established procedure which allows the synthesis of a wide variety of compounds (Caubère, 1974; Carré, Jamart-Grégoire, Geoffroy, Caubère, Ianelli & Nardelli, 1988, and references cited therein). In order to develop research into this kind of reaction, we have been studying the condensation of ketones containing functional groups for some time. As part of this work, we carried out the condensation of the enolate of quinuclidinone with bromobenzene.

Amongst the products isolated was one that we were unable to identify by the usual spectroscopic methods and

so X-ray analysis was used to determine its crystal structure. This compound was found to be a spiro derivative, indicating that the condensation reaction should be formulated as:
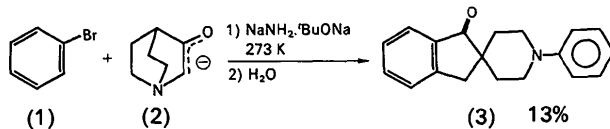
Although the yield was low (13%), the formation of the compound is very interesting as it reveals a new mechanism of arynic condensation, the details of which have yet to be elucidated. This paper presents the results of the crystal structure analysis of the title compound and discusses its molecular conformation.

The molecule consists of a nearly planar indanone system [$\Sigma(\Delta/\sigma)^2 = 212.5$], spiro-fused to a piperidine ring such that the planes through them form a dihedral angle of 88.7(1)°, and a phenyl group bound to N with a dihedral angle of 68.6(1)° to the mean plane through the piperidine ring. The ORTEP diagram of the molecule is shown in Fig. 1, whilst bond distances and angles are given in Table 2. The bond distances and angles of the indanone system compare well with those found by us in a previous study (Ianelli, Nardelli, Belletti, Jamart-Grégoire, Carré & Caubère, 1989) even though the five-membered ring was then fused with a cycloalkane ring, whereas it is now spiro-fused to a piperidine ring. The different situation influences the puckering (Cremer & Pople, 1975) of the cyclopentene ring which was more pronounced in the previous compounds (Q_T = 0.159–0.260 Å) than it is now [Q_T = 0.051(2) Å]. The values of the endocyclic angles in the benzo moiety indicate that the deformation of the ring is such as to maintain equal opposite angles. The contraction of the endocyclic angles at the C atoms ortho with respect to the junction [C5—C4—C9 118.5(2), C6—C7—C8 118.6(2)°] corresponds quite well to that observed for fusion of benzene with a cyclopentene ring [Benassi, Ianelli, Nardelli & Taddei (1991); average value 118.6(2)°]. In addition, the bond distances and angles agree with the averaged values from Benassi et al..

The piperidine ring has a chair conformation with a puckering amplitude Q_T = 0.575(2) Å and a geometry which is in quite good agreement with that found for this
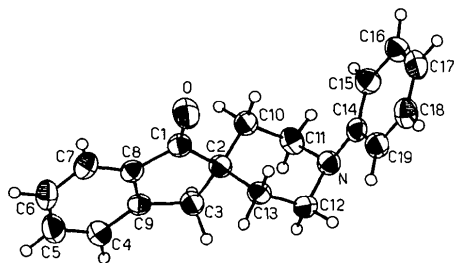
system in the literature. The N atom is 0.334(2) Å out of the plane through the C atoms to which it is bonded, in agreement with its sp^3 character. The N—C(phenyl) distance is 1.409(2) Å; this is not significantly different from the length of a Csp^2—Nsp^3 single bond, indicating that the degree of conjugation of the N lone pair with the phenyl π system should be quite low. On the other hand, the lone pair is orientated approximately parallel to the direction of the $2p_z$ C orbitals, the angle between these two directions being only 12°. In order to determine whether this arrangement of the phenyl substituent occurs systematically in the N-phenylpiperidine fragments of molecules, a search of the Cambridge Structural Database (January 1992 release; Allen et al., 1991) was undertaken. Table 3 gives the averaged data for the eight systems of this kind with $R<0.07$ which did not have any substituents that could influence the nature of bonds at N. The data show that N is pyramidal in all eight systems and that the N—C(ar) and N—Csp^3 distances are in agreement with those found in the present analysis. Moreover, the orientation of the lone pair at N is similar to the one we found which implies that a conjugative effect should be present, even if it is weak and not manifested in a significant variation of the bond distance.

In order to obtain further information about the factors influencing the conformation about the N—Ph bond, the variation of the non-bonding energy was calculated for rotation of the phenyl group about that bond; the curves obtained have been deposited. The curve for the isolated molecule shows four small peaks of ca 10 and 20 kJ mol⁻¹ which are due to the steric hindrance encountered by the ortho H atoms of the two rings. It should be noted that the conformation found in the crystal does not correspond to a minimum, but to a small peak of ca 3 kJ mol⁻¹ which is indicative that other effects are responsible for the conformation. The curve for the molecule packed in the crystal shows merely a deep minimum displaced by only $-10°$ with respect to the position corresponding to the conformation found experimentally; this indicates the importance of packing effects.

The deformation of the phenyl ring induced by the piperidine substituent is such as to narrow the angles at the ipso and para C atoms and widen those at the ortho and meta C atoms (Table 3), in agreement with the findings of Domenicano, Vaciago & Coulson (1975). No particular trends are observed for the variations of bond distances in the ring.

The results of the analysis of the anisotropic atomic displacements are given in Table 4. [The analysis was carried out in terms of the LST rigid-body approximation according to Schomaker & Trueblood (1968) and Trueblood (1978) and considering also the internal motions according to Dunitz & White (1973).] The data indicate the presence of internal motions affecting particularly the O atom which also exhibits the maximum anisotropy [as indicated by the atomic ellipsoids of Fig. 1 and the values of the ratios of their principal axes (deposited)].

Experimental

Crystal data

$C_{19}H_{19}NO$

M_r = 277.36

Orthorhombic

Pbca

a = 29.018 (3) Å

b = 8.530 (1) Å

c = 11.762 (1) Å

V = 2911 (5) Å^3

Z = 8

D_x = 1.266 Mg m^{-3}

Cu $K\alpha_1$ radiation

λ = 1.540562 Å

Cell parameters from 30 reflections

θ = 15–37°

μ = 0.569 mm^{-1}

T = 293 (2) K

Thin prisms

0.77 × 0.37 × 0.13 mm

Colourless

Data collection

Siemens–AED diffractometer

θ–2θ scans

Absorption correction:
    not applied

3205 measured reflections

1829 observed reflections
    $[I>3\sigma(I)]$

θ_{max} = 70°

$h = 0 \rightarrow 35$

$k = 0 \rightarrow 14$

$l = 0 \rightarrow 10$

1 standard reflection
    monitored every 50
    reflections
    intensity variation: none

Refinement

Refinement on F

Final R = 0.0395

wR = 0.0566

S = 0.8064

1829 reflections

266 parameters

All H-atom parameters refined

$w=1/[\sigma^2(F_o)+0.00833F_o^2]$

$(\Delta/\sigma)_{max}$ = 0.09

$\Delta\rho_{max}$ = 0.09 e Å^{-3}

$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A, 2.2C and 2.3.1)

Cell refinement: *LQPARM* (Nardelli & Mangia, 1984). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O	0.42628 (6)	0.3651 (2)	0.9935 (1)	0.0682 (6)
N	0.31783 (5)	0.0629 (2)	0.7997 (1)	0.0430 (5)
C1	0.41793 (6)	0.4060 (2)	0.8967 (2)	0.0436 (6)
C2	0.38472 (6)	0.3223 (2)	0.8159 (2)	0.0401 (5)
C3	0.38866 (7)	0.4148 (2)	0.7023 (2)	0.0467 (6)
C4	0.43274 (7)	0.6737 (2)	0.6578 (2)	0.0501 (7)
C5	0.46151 (8)	0.7885 (3)	0.7009 (2)	0.0583 (7)
C6	0.47866 (8)	0.7807 (3)	0.8107 (2)	0.0572 (7)
C7	0.46661 (7)	0.6581 (2)	0.8805 (2)	0.0481 (7)
C8	0.43686 (6)	0.5446 (2)	0.8388 (2)	0.0381 (6)
C9	0.41973 (6)	0.5511 (2)	0.7285 (2)	0.0391 (5)
C10	0.39885 (7)	0.1499 (2)	0.8030 (2)	0.0455 (6)
C11	0.36234 (8)	0.0561 (3)	0.7406 (2)	0.0481 (7)
C12	0.30227 (7)	0.2264 (2)	0.8024 (2)	0.0458 (6)
C13	0.33579 (7)	0.3307 (2)	0.8666 (2)	0.0439 (6)
C14	0.31252 (6)	−0.0279 (2)	0.8989 (2)	0.0395 (5)
C15	0.33727 (7)	−0.1663 (2)	0.9146 (2)	0.0491 (7)
C16	0.33050 (8)	−0.2581 (3)	1.0107 (2)	0.0530 (7)
C17	0.29947 (8)	−0.2162 (3)	1.0936 (2)	0.0552 (7)
C18	0.27443 (8)	−0.0810 (3)	1.0784 (2)	0.0567 (7)
C19	0.28044 (7)	0.0121 (3)	0.9832 (2)	0.0505 (6)

Table 2. *Geometric parameters* (Å, °)

O—C1	1.215 (2)	C5—C6	1.386 (3)
N—C11	1.467 (2)	C6—C7	1.375 (3)
N—C12	1.467 (2)	C7—C8	1.387 (2)
N—C14	1.409 (2)	C8—C9	1.390 (2)
C1—C2	1.531 (2)	C10—C11	1.516 (3)
C1—C8	1.471 (2)	C12—C13	1.519 (2)
C2—C3	1.556 (2)	C14—C15	1.395 (2)
C2—C10	1.534 (2)	C14—C19	1.402 (2)
C2—C13	1.542 (2)	C15—C16	1.390 (3)
C3—C9	1.504 (2)	C16—C17	1.375 (3)
C4—C5	1.383 (3)	C17—C18	1.375 (3)
C4—C9	1.388 (2)	C18—C19	1.384 (3)
C12—N—C14	118.1 (2)	C7—C8—C9	121.7 (2)
C11—N—C14	117.8 (2)	C1—C8—C9	109.3 (2)
C11—N—C12	108.6 (2)	C4—C9—C8	119.5 (2)
O—C1—C8	126.2 (2)	C3—C9—C8	112.0 (2)
O—C1—C2	125.0 (2)	C3—C9—C4	128.5 (2)
C2—C1—C8	108.8 (2)	C2—C10—C11	111.5 (2)
C1—C2—C13	108.5 (2)	N—C11—C10	111.4 (2)
C1—C2—C10	109.9 (2)	N—C12—C13	111.8 (2)
C1—C2—C3	104.5 (2)	C2—C13—C12	111.7 (2)
C10—C2—C13	109.2 (2)	N—C14—C19	121.7 (2)
C3—C2—C13	112.1 (2)	N—C14—C15	121.2 (2)
C3—C2—C10	112.4 (2)	C15—C14—C19	117.1 (2)
C2—C3—C9	105.1 (2)	C14—C15—C16	120.8 (2)
C5—C4—C9	118.5 (2)	C15—C16—C17	121.5 (2)
C4—C5—C6	121.6 (2)	C16—C17—C18	118.2 (2)
C5—C6—C7	120.1 (2)	C17—C18—C19	121.3 (2)
C6—C7—C8	118.6 (2)	C14—C19—C18	121.1 (2)
C1—C8—C7	129.0 (2)		

Table 3. *Relevant geometrical descriptors of the N-phenyl moiety* (Å, °)

Square brackets denote mean planes; Lp = lone pair; *ipso, ortho, meta, para* refer to the endocyclic angles of the phenyl ring.

	Found	Literature
N—Csp^3	1.467 (2)	1.465 (3)
N—C(ar)	1.409 (2)	1.401 (4)
N···[C11,C12,C14]	0.334 (2)	0.292 (22)
N···[phenyl]	0.046 (2)	0.096 (16)
Lp [phenyl]	11.7 (3)	15.0 (37)
ipso	117.1 (2)	117.8 (4)
ortho	121.0 (2)	120.5 (2)
meta	121.4 (1)	121.3 (2)
para	118.2 (2)	118.6 (3)

Table 4. *Analysis of the anisotropic atomic displacements in terms of LST rigid-body motion and internal motions*

CPN is the normal at the centroid of the mean plane through the cyclopentene ring, BPN is the normal to the benzene plane at the midpoint of the C8—C9 bond, Δ is the mean difference of the mean-square vibrational amplitudes along the interatomic directions for all pairs of atoms, $\Delta U = U_{ij}$(obs.) $-$ U_{ij}(calc.), $R_{wU} = [\Sigma(w\Delta U)^2/\Sigma(wU_o)^2]^{1/2}$, $\sigma(w\Delta U) = [\Sigma(w\Delta U)^2/\Sigma w^2]^{1/2}$, $\sigma(U_o)$ is the mean e.s.d of U_o's.

Treatment	$\Delta\times10^4$ (Å)	$\sigma(w\Delta U)\times10^4$	$\sigma(U_o)\times10^4$	R_{wU}
Rigid-body		44		0.146
	33 (49)		11	
Internal motions		35		0.116

Group librating	Libration along	Libration amplitude (°)
O	CPN	3.0 (5)
C15,...C19	N—C14	3.2 (15)
C4,...C7	BPN	3.4 (15)

The integrated intensities were measured using a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure. Corrections were applied for Lorentz and polarization effects but not for absorption. Extinction was considered according to Zachariasen (1963) [$g = 4.06(1) \times 10^{-8}$].

The structure was determined by direct methods using SHELXS86 and refined by anisotropic full-matrix least squares on F using SHELX76. The H atoms were localized from a final difference Fourier synthesis and refined isotropically.

The atom-atom non-bonded potential-energy calculations were carried out using ROTENER (Nardelli, 1988) and the atomic charges calculated by the iterative partial equalization of orbital electronegativity method of Gasteiger & Marsili (1980) using ATOMCHAR (Nardelli, 1991).

Throughout this paper, the averaged values are means weighted according to the reciprocals of the variances and the corresponding e.s.d.'s are the largest values of the 'external' and 'internal' standard deviations (Topping, 1960).

The calculations were performed using the ENCORE-GOULD-POWERNODE 6040 computer at the 'Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma)'.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, together with the results of the Cambridge Structural Database search and difference van der Waals energy profiles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55178 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1005]

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Structure and Conformation of cis,anti,cis-4b,4c,8b,9,10,10a-Hexahydro-4b-hydroxy-benzo[3,4]cyclobuta[1,2-a]biphenylene-4-carbaldehyde

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Abstract

The title compound was prepared as part of a study of polyphenylene derivatives obtained by arynic condensation in the presence of complex bases. X-ray analysis shows that the two benzocyclobutene moieties have an *anti* arrangement. The hydroxyl group and the H atoms are *cis* at each junction and *trans* for one junction with respect to the other. The two pseudosymmetrical parts of the core of the molecule show the same geometry in spite of having different substitution. Previously observed deformations for the benzene moiety fused with the cyclobutene ring and lengthening of the cyclobutane Csp^3—Csp^3 bond are confirmed. The orientations of the formyl and hydroxyl groups are discussed.