

C4	0.2707 (1)	-0.0812 (1)	-0.1874 (1)	0.060 (1)
C5	0.2714 (1)	-0.1584 (1)	-0.0851 (2)	0.053 (1)
C6	0.2541 (1)	-0.1414 (1)	0.0840 (1)	0.042 (1)
C7	0.1934 (1)	-0.0655 (1)	0.1140 (1)	0.039 (1)
C8	0.1405 (1)	-0.0746 (1)	0.2637 (2)	0.051 (1)
C9	0.2041 (1)	-0.0820 (1)	0.3912 (2)	0.053 (1)
C10	0.2409 (1)	-0.0058 (1)	0.4079 (1)	0.052 (1)
C11	0.2016 (1)	0.0545 (1)	0.2922 (2)	0.046 (1)
C12	0.2333 (1)	0.0272 (1)	0.1313 (1)	0.035 (1)
C13	0.1096 (1)	0.0188 (1)	0.2872 (2)	0.058 (1)
O1	0.3826 (1)	0.0394 (1)	0.2321 (1)	0.044 (1)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.539 (2)	C1—C12	1.542 (1)
C1—O1	1.423 (1)	C2—C3	1.499 (2)
C3—C4	1.315 (3)	C4—C5	1.503 (2)
C5—C6	1.544 (2)	C6—C7	1.531 (2)
C7—C8	1.565 (2)	C7—C12	1.574 (2)
C8—C9	1.504 (2)	C8—C13	1.540 (2)
C9—C10	1.323 (2)	C10—C11	1.515 (2)
C11—C12	1.566 (2)	C11—C13	1.533 (2)
O1—O1	2.736 (1)		
O1—C1—C12	114.3 (1)	O1—C1—C2	108.9 (1)
C12—C1—C2	116.6 (1)	C3—C2—C1	114.7 (1)
C4—C3—C2	123.5 (1)	C5—C4—C3	125.7 (1)
C6—C5—C4	116.5 (1)	C7—C6—C5	114.0 (1)
C12—C7—C8	101.9 (1)	C12—C7—C6	118.5 (1)
C8—C7—C6	113.6 (1)	C13—C8—C9	100.1 (1)
C13—C8—C7	101.2 (1)	C9—C8—C7	107.4 (1)
C10—C9—C8	107.4 (1)	C11—C10—C9	107.6 (1)
C13—C11—C10	99.7 (1)	C13—C11—C12	99.9 (1)
C12—C11—C10	108.8 (1)	C11—C12—C7	102.3 (1)
C11—C12—C1	112.9 (1)	C7—C12—C1	124.5 (1)
C11—C13—C8	93.1 (1)		

Table 3. Comparison of selected bond lengths (Å) and bond angles (°) of known crystal structures

	C7—C12	C9—C10	C1—C12—C11	C6—C7—C8
(I)	1.574 (2)	1.323 (2)	112.9 (1)	113.6 (1)
(II)	1.571 (2)	1.318 (2)	110.8 (1)	113.6 (1)
(III) (molecule 1)	1.564 (3)	1.307 (4)	112.4 (2)	112.9 (2)
(molecule 2)	1.561 (4)	1.313 (5)	113.1 (2)	112.2 (3)
(IV)	1.569 (2)	1.312 (2)	112.3 (2)	111.8 (1)

Table 4. Comparison of r.m.s. deviations (Å) of the C<sub>13</sub> skeleton of tricyclo[8.2.1.0<sup>2,9</sup>]trideca-5,11-diene in known crystal structures

	(I)	(II)	(III)	(IV)
R.m.s. deviation	0.09	0.17	0.09	0.21

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovitch, 1965). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993), *GFMLX* (Flack, 1983). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989), *XANADU* (Roberts & Sheldrick, 1975), *SYBIL* (Tripos Associates Inc., 1994). Software used to prepare material for publication: *DAESD* (Davis & Harris, 1970).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1181). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## N-Benzoyl-2-phenyldecahydroquinolin-4-one, C<sub>22</sub>H<sub>23</sub>NO<sub>2</sub>

A. THIRUVALLUVAR\*

Department of Physics, Bharathidasan University, Tiruchirappalli 620 024, India

K. SANKAR RAJA RAJ AND M. KRISHNA PILLAY

Department of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, India

K. VENKATASUBRAMANIAN†

Central Salt & Marine Chemical Research Institute, Bhavnagar 364 002, India

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## Abstract

The title molecule consists of a piperidin-4-one ring fused to a cyclohexane moiety. The piperidine ring adopts a twisted conformation and the cyclohexane ring adopts a chair conformation. The crystal structure determination confirms the *trans* fusion of the two rings.

† Deceased.

### Comment

The title compound, (I), has been analysed as part of our crystallographic studies on substituted decahydroquinolines.

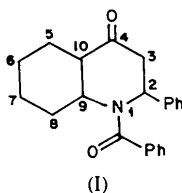


Fig. 1 shows a perspective view of the molecule with the atomic numbering (ORTEPII; Johnson, 1976). The H9—C9—C10—H10 torsion angle is  $-173.3(3)^\circ$ . This confirms the *trans* configuration of the title compound. The departures of angles C2—N1—C11 [ $121.1(3)^\circ$ ], C2—N1—C9 [ $117.8(3)^\circ$ ] and C9—N1—C11 [ $116.9(2)^\circ$ ] from tetrahedral values indicate a flattening of the piperidine ring at N1. The N1—C11 bond length is  $1.371(4) \text{ \AA}$ . This shows partial double-bond character due to the delocalization of the N-atom lone pair of electrons over the —N—C=O moiety. The displacement of N1 from the plane defined by C2, C9 and C11 is  $-0.172(3) \text{ \AA}$ . As evident from the torsion angles (Table 2), the piperidine ring adopts a flexible twist conformation. The benzoyl group substituted on N1 is in an equatorial orientation; according to Aroney & Le Fèvre (1960), bulky substituents on the N atom of piperidine exist mainly in the equatorial position. The phenyl ring is substituted on C2 in an axial position, the torsion angle C9—N1—C2—C21 being  $-86.2(3)^\circ$ . The torsion angles C12—C11—N1—C9 and O11—C11—N1—C9 are  $-179.1(3)$  and  $0.5(5)^\circ$ , respectively, which indicate that the peptide-like group formed by C9, N1, C11, O11 and C12 is planar. The fused cyclohexane ring adopts a chair conformation.

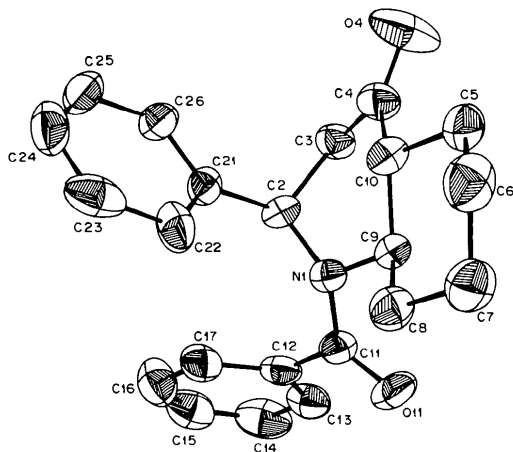


Fig. 1. Molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

The angle between the least-squares planes through the piperidine and cyclohexane rings is  $7.9(1)^\circ$ . The angle between the least-squares planes of the two phenyl rings is  $69.2(1)^\circ$ .

### Experimental

A mixture of 2-phenyldecahydroquinalin-4-one (2.29 g, 0.01 mol) in 20 ml of dry benzene, benzoyl chloride (1.4 g, 0.01 mol) and 3 drops of triethylamine was refluxed for 2 h. After the completion of the reaction, the reaction mixture was washed with portions of water ( $4 \times 10 \text{ ml}$ ) and dried over anhydrous sodium sulfate. The excess benzene was distilled off under reduced pressure. Recrystallization from ethanol afforded colourless crystals, m.p. 506 K. The yield was 2.7 g, 80%. The density  $D_m$  was measured by flotation in CCl<sub>4</sub> and petroleum ether.

#### Crystal data

C<sub>22</sub>H<sub>23</sub>NO<sub>2</sub>  
 $M_r = 333.4$   
 Orthorhombic  
 $P2_12_1$   
 $a = 10.001(1) \text{ \AA}$   
 $b = 20.603(2) \text{ \AA}$   
 $c = 8.601(1) \text{ \AA}$   
 $V = 1772.2(3) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.250 \text{ Mg m}^{-3}$   
 $D_m = 1.260 \text{ Mg m}^{-3}$

Cu K $\alpha$  radiation  
 $\lambda = 1.5418 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 14-30^\circ$   
 $\mu = 0.59 \text{ mm}^{-1}$   
 $T = 298 \text{ K}$   
 Plate  
 $0.22 \times 0.18 \times 0.14 \text{ mm}$   
 Colourless

#### Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1746 measured reflections  
 1317 independent reflections  
 1288 observed reflections  
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.03$   
 $\theta_{\text{max}} = 65^\circ$   
 $h = 0 \rightarrow 11$   
 $k = 0 \rightarrow 24$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: negligible

#### Refinement

Refinement on  $F$   
 $R = 0.037$   
 $wR = 0.038$   
 $S = 1.18$   
 1288 reflections  
 226 parameters  
 H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.001F^2]$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from SHELX76 (Sheldrick, 1976)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O4	-0.0487 (4)	0.1669 (1)	0.6543 (4)	0.091 (1)
O11	0.3057 (2)	-0.0037 (1)	1.0196 (4)	0.065 (1)
N1	0.1024 (2)	0.0435 (1)	0.9901 (3)	0.036 (1)
C2	-0.0356 (3)	0.0349 (2)	0.9333 (4)	0.037 (1)

C3	-0.0451 (4)	0.0625 (2)	0.7684 (4)	0.041 (1)
C4	-0.0102 (4)	0.1326 (2)	0.7594 (4)	0.046 (1)
C5	0.1505 (3)	0.2192 (2)	0.8507 (5)	0.049 (1)
C6	0.2263 (4)	0.2457 (2)	0.9897 (6)	0.062 (1)
C7	0.3162 (4)	0.1954 (2)	1.0602 (5)	0.062 (2)
C8	0.2403 (3)	0.1336 (2)	1.0990 (4)	0.048 (1)
C9	0.1703 (3)	0.1059 (2)	0.9554 (4)	0.038 (1)
C10	0.0747 (3)	0.1575 (2)	0.8909 (4)	0.040 (1)
C11	0.1827 (3)	-0.0089 (2)	1.0204 (4)	0.045 (1)
C12	0.1204 (3)	-0.0736 (2)	1.0540 (4)	0.043 (1)
C13	0.1805 (4)	-0.1281 (2)	0.9907 (4)	0.054 (1)
C14	0.1260 (5)	-0.1890 (2)	1.0121 (5)	0.068 (2)
C15	0.0131 (5)	-0.1963 (2)	1.1009 (6)	0.070 (2)
C16	-0.0436 (5)	-0.1433 (2)	1.1711 (5)	0.068 (2)
C17	0.0093 (4)	-0.0822 (2)	1.1475 (5)	0.055 (1)
C21	-0.1413 (3)	0.0614 (1)	1.0438 (4)	0.038 (1)
C22	-0.1116 (4)	0.0849 (2)	1.1913 (4)	0.047 (1)
C23	-0.2121 (5)	0.1085 (2)	1.2875 (5)	0.063 (2)
C24	-0.3427 (4)	0.1079 (2)	1.2391 (5)	0.069 (2)
C25	-0.3738 (4)	0.0835 (2)	1.0951 (6)	0.066 (2)
C26	-0.2747 (3)	0.0603 (2)	0.9979 (5)	0.050 (1)

Table 2. Selected geometric parameters (Å, °)

O4—C4	1.210 (5)	N1—C2	1.475 (4)
O11—C11	1.235 (4)	N1—C9	1.484 (4)
O4—C4—C3	122.1 (3)	C13—C12—C17	118.2 (4)
C3—C4—C10	115.1 (3)	C2—C21—C26	118.9 (3)
O4—C4—C10	122.8 (4)	C2—C21—C22	123.1 (3)
C11—C12—C17	124.0 (4)	C22—C21—C26	118.0 (3)
C11—C12—C13	117.7 (3)		
C2—N1—C11—C12	-22.8 (5)	O4—C4—C10—C9	-148.9 (4)
C2—N1—C9—C10	11.0 (4)	C6—C5—C10—C9	-56.7 (4)
C11—N1—C9—C8	-69.1 (4)	C10—C5—C6—C7	54.0 (5)
C2—N1—C9—C8	133.7 (3)	C5—C6—C7—C8	-53.5 (5)
C9—N1—C2—C3	41.4 (4)	C6—C7—C8—C9	56.6 (4)
N1—C2—C21—C22	-6.7 (5)	C7—C8—C9—C10	-58.2 (4)
N1—C2—C3—C4	-59.2 (4)	N1—C9—C10—C4	-49.4 (4)
C3—C2—C21—C22	-131.7 (4)	C8—C9—C10—C5	58.2 (4)
C2—C3—C4—C10	21.9 (5)	N1—C11—C12—C13	139.8 (3)
C3—C4—C10—C5	158.3 (3)	O11—C11—C12—C13	-39.8 (5)
C3—C4—C10—C9	31.8 (4)		

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). All non-H atoms were refined with anisotropic displacement parameters. All H atoms were obtained from difference Fourier maps and were included in the structure-factor calculations; they were given displacement parameters equal to  $1.1U_{eq}$  of their respective carrier atom, but their parameters were not refined (Sheldrick, 1976). The geometrical calculations were performed using *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: VJ1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *o*-Phenylenediammonium Bis(hydrogen-sulfide)

O. V. SHISHKIN, N. N. KOLOS AND V. D. ORLOV

*Department of Organic Chemistry, Kharkov University, Svoboda Square 4, 310077 Kharkov, Ukraine*

V. P. KUZNETSOV AND E. E. LAKIN

*Institute for Single Crystals, Academy of Sciences of the Ukraine, Lenin Avenue 50, 310001 Kharkov, Ukraine*

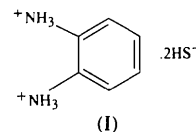
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## Abstract

The title compound,  $C_6H_{10}N_2^{2+} \cdot 2HS^-$ , forms crystals with *C2/c* symmetry. The anionic  $HS^-$  groups are located near the  $NH_3^+$  substituents and are oriented practically perpendicular to the benzene ring.

## Comment

The molecule in the unit cell of the title compound, (I), is situated on the twofold axis, which passes through the midpoints of the C(4A)—C(4B) and C(1A)—C(1B) bonds.



The presence of H atoms at every N atom and the C(4A)—N(1A) bond length of 1.457 (2) Å indicate protonation of both amino groups. The anionic  $HS^-$  groups are oriented practically perpendicular to the benzene ring [ $104(1)^\circ$ ]. The short interatomic distances  $N(1A) \cdots S(A)$  [3.11 (1) Å] and  $H(1N1A) \cdots S(A)$  [2.13 (1) Å] confirm the existence of a strong electrostatic interaction between the dication and the anions.