2(*b*). They indicate a good planarity of the hexyl group which is twisted 15·6 (1)° from the planar amidic group. The angle between the twofold axis and the normal to the hexyl plane is 51·7 (3)°.

From the above results and those recently reported for the analogous diamide member (Rueda & Fayos, 1982), we note that the inclusion of the ketonic group in the middle of the dicarboxylic acid drastically changes the shape and packing of the molecules. Nevertheless, the two hydrogen bonds per molecule are completely preserved. Indeed a shorter N···O distance is present for the ketonic member.

The authors thank Professor S. Garcia-Blanco for his support and the Centro de Cálculo of JEN of Madrid for the use of the 1100/80 Univac computer. We are also indebted to Dr F. J. Baltá Calleja and Dr F. Ania for their comments during the work.

References

ANIA, F., BALTÁ CALLEJA, F. J. & DE ABAJO, J. (1979). *J. Macromol. Sci. Phys.* **16**, 377–388.
ANIA, F., RUEDA, D. R. & BALTÁ CALLEJA, F. J. (1981). *Makromol. Chem.* **183**, 235–239.
MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England and Louvain, Belgium.
MARTÍNEZ-RIPOLL, M. & CANO, F. H. (1975). *PESOS.* Instituto Rocasolano, CSIC, Madrid-6, Spain.
OBESO, R. M. (1978). PhD Thesis, Univ. of Madrid, Spain.
OBESO, R. M., DE ABAJO, J. & FONTÁN, J. (1977). *Rev. Plast. Mod.* **257**, 679–686.
RUEDA, D. R. & FAYOS, J. (1982). *Eur. Polym. J.* **18**, 1–4.
STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

12-Phenyl-5,12-dihydrodibenz[*b,g*]azocin-5-one, C21H15NO

BY HANS PREUT AND MICHAEL THIMME

Abteilung Chemie der Universität Dortmund, Postfach 500500, D-4600 Dortmund 50, Federal Republic of Germany

THEOPHIL EICHER

Fachbereich 14 Organische Chemie, Universität des Saarlandes, D-6600 Saarbrücken 11, Federal Republic of Germany

AND CARL KRÜGER

Max-Planck-Institut für Kohlenforschung, Lembkestrasse 5, D-4330 Mülheim/Ruhr, Federal Republic of Germany

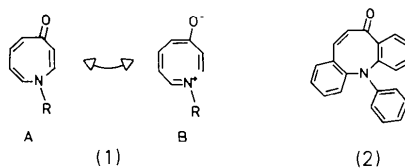(*Received* 17 *December* 1982; *accepted* 4 *February* 1983)

Abstract. $M_r = 297\cdot4$, monoclinic, $C2/c$, $a = 15\cdot724$ (12), $b = 9\cdot222$ (6), $c = 21\cdot504$ (16) Å, $\beta = 95\cdot91$ (8)°, $U = 3102$ Å^3, $Z = 8$, $D_x = 1\cdot274$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot41$ mm^{-1}, $F(000) = 1248$, $T = 294$ (1) K. Final $R = 0\cdot055$ for 1170 unique X-ray diffractometer data. The molecular structure has been elucidated.

Introduction. Our former results in studying synthesis, structural properties and chemical behaviour of cyclocyanines (Eicher, Abdesaken, Franke & Weber, 1975; Eicher & Franke, 1981) prompted us to direct synthetic efforts to the class of eight-membered cyclic merocyanines (1), especially with regard to the aspect of a probable participation of a resonance hybrid $A \longleftrightarrow B$ in the ground-state description of the 1,4-dihydroazocin-4-one system (1).

(1)   (2)

The title compound (2) was synthesized recently (Thimme, 1982) as the first member of the hitherto unknown class of azocin-4-ones. The above aspects of a probable bond delocalization in the eight-membered ring and the necessity of gaining rigorous proof of structure for the product of a multi-step synthesis (its spectroscopic data lacked specific information on detailed structural and conformational properties) made it desirable to determine the molecular structure of the dibenzazocinone (2).

Experimental. Crystal size $0.3 \times 0.2 \times 0.3$ mm, $\omega/2\theta$ scan, scan speed $0.16–0.02°$ s^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit with 46 reflexions in range $2.7 < \theta < 9.6°$; three standard reflexions recorded every 67 min, only random deviations; 4971 reflexions, $2\theta \le 60°$, $\pm h$, $+k$, $+l$; after averaging 4746 unique reflexions, 1170 with $I > 3\sigma(I)$; Lorentz and polarization correction, no absorption correction; systematic absences: (hkl) $h + k = 2n+1$ and $(h0l)$ $h = 2n+1$ and $l = 2n+1$, space group $C2/c$ or Cc; crystal structure solved with $MULTAN$ (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and subsequent difference Fourier syntheses in space group $C2/c$; refined by full-matrix least-squares with $SHELX$ (Sheldrick, 1976) with anisotropic temperature factors for all non-H atoms, common isotropic temperature factor for H; refinement based on F^2 and 197 parameters, $R = 0.055$, unit weights; maximum shift over error for all parameters was 0.032, average 0.01; H atoms placed in geometrically calculated positions (C—H 1.08 Å, equal C—C—H) and allowed to ride on the C atom to which they are attached; phenyl group was refined as a rigid body (C—C 1.395 Å, C—C—C $120°$); complex neutral-atom scattering factors taken from Cromer & Mann (1968), Cromer & Liberman (1970) and for H from Cromer (personal communication to G. M. Sheldrick); a final ΔF synthesis did not reveal any significant features;

figures drawn with $ORTEP$ (Johnson, 1976) and POP 1 (van de Waal, 1976).

Discussion. The structure of the title compound is shown in Fig. 1 and in a stereoview in Fig. 2. Positional parameters and equivalent values U_{eq} of the anisotropic temperature factors U_{ij} are given in Table 1.* Bond lengths and bond angles are given in Table 2. The unit cell contains one independent molecule. The calculation of the least-squares planes through the positions C(11), C(12), C(13), C(14), C(15) and C(16) (I: $0.457x - 0.864y - 0.211z = 5.119$ Å; xyz are orthogonalized coordinates) and the positions C(21), C(22), C(23), C(24), C(25) and C(26) (II: $-0.912x - 0.072y - 0.404z = -0.283$ Å) shows that no atom deviates more than 0.014 (6) Å from planarity. The atoms N(1) and C(1) have distances of -0.029 (4) and -0.007 (7) Å, respectively, from plane (I) and the atoms N(1), C(2) and C(3) have distances of 0.092 (4), -0.152 (7) and 0.113 (7) Å, respectively, from plane (II). The dihedral angle between these planes is 74.3 (2)°.

There are no intermolecular distances indicating interactions exceeding van der Waals interactions.

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38389 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. General view of the molecule, showing atom numbering.
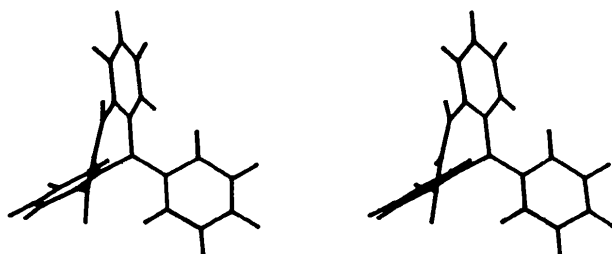
Fig. 2. Stereoscopic view of the molecule.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic U values* (Å$^2 \times 10^3$)

	x	y	z	U_{eq}*
N(1)	1980 (3)	−1964 (5)	−3808 (2)	41 (4)
C(1)	1108 (4)	−3417 (7)	−2886 (3)	51 (4)
C(2)	1543 (4)	−762 (8)	−2613 (3)	59 (5)
H(2)	1620 (4)	−129 (8)	−2189 (3)	†
C(3)	1372 (4)	−2144 (8)	−2502 (3)	63 (5)
H(3)	1442 (4)	−2391 (8)	−2009 (3)	†
O(1)	1106 (3)	−4600 (5)	−2636 (2)	81 (4)
C(11)	1161 (3)	−2604 (6)	−4001 (3)	38 (4)
C(12)	730 (3)	−3298 (6)	−3553 (3)	41 (4)
C(13)	−62 (4)	−3912 (7)	−3728 (3)	52 (5)
H(13)	−393 (4)	−4483 (7)	−3386 (3)	†
C(14)	−428 (4)	−3801 (7)	−4332 (3)	60 (5)
H(14)	−1055 (4)	−4253 (7)	−4461 (3)	†
C(15)	3 (4)	−3107 (7)	−4781 (3)	62 (5)
H(15)	−285 (4)	−3031 (7)	−5258 (3)	†
C(16)	804 (4)	−2512 (7)	−4612 (3)	54 (5)
H(16)	1147 (4)	−1981 (7)	−4958 (3)	†
C(21)	1923 (3)	−419 (6)	−3720 (3)	39 (4)
C(22)	1655 (4)	110 (6)	−3161 (3)	42 (4)
C(23)	1565 (4)	1625 (7)	−3120 (3)	53 (5)
H(23)	1357 (4)	2085 (7)	−2700 (3)	†
C(24)	1732 (4)	2531 (8)	−3595 (3)	61 (5)
H(24)	1645 (4)	3686 (8)	−3549 (3)	†
C(25)	2011 (4)	1979 (7)	−4133 (3)	58 (5)
H(25)	2146 (4)	2698 (7)	−4507 (3)	†
C(26)	2118 (4)	495 (7)	−4193 (3)	51 (5)
H(26)	2355 (4)	56 (7)	−4607 (3)	†
C(31)	2749 (2)	−2702 (3)	−3829 (2)	34 (4)
C(32)	3508 (2)	−2054 (3)	−3576 (2)	46 (4)
C(33)	4280 (2)	−2799 (3)	−3573 (2)	54 (5)
C(34)	4293 (2)	−4193 (3)	−3824 (2)	59 (5)
C(35)	3534 (2)	−4841 (3)	−4078 (2)	55 (5)
C(36)	2762 (2)	−4096 (3)	−4081 (2)	41 (4)
H(32)	3498 (2)	−975 (3)	−3381 (2)	†
H(33)	4868 (2)	−2297 (3)	−3377 (2)	†
H(34)	4892 (2)	−4770 (3)	−3822 (2)	†
H(35)	3545 (2)	−5920 (3)	−4272 (2)	†
H(36)	2174 (2)	−4598 (3)	−4277 (2)	†

* $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$.
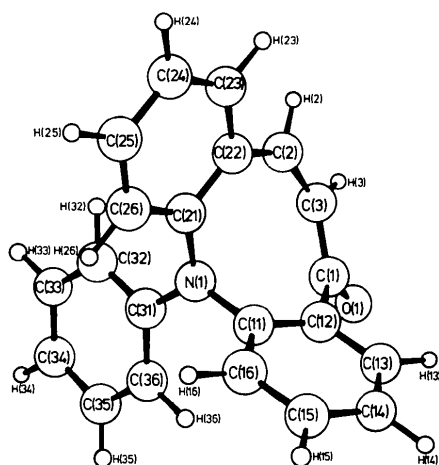† Common isotropic temperature factor of 0.083 (5) Å^2.

Table 2. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

N(1)–C(31)	1·392 (6)	C(1)–O(1)	1·217 (7)
N(1)–C(21)	1·441 (7)	C(1)–C(3)	1·471 (9)
N(1)–C(11)	1·439 (6)	C(3)–C(2)	1·329 (9)
C(11)–C(12)	1·390 (7)	C(2)–C(22)	1·453 (8)
C(12)–C(13)	1·384 (7)	C(21)–C(22)	1·401 (7)
C(13)–C(14)	1·371 (8)	C(22)–C(23)	1·408 (8)
C(14)–C(15)	1·390 (9)	C(23)–C(24)	1·367 (9)
C(15)–C(16)	1·388 (8)	C(24)–C(25)	1·375 (8)
C(16)–C(11)	1·377 (7)	C(25)–C(26)	1·387 (8)
C(12)–C(1)	1·499 (8)	C(26)–C(21)	1·379 (8)
N(1)–C(31)–C(32)	119·2 (2)	C(15)–C(16)–C(11)	119·6 (6)
N(1)–C(31)–C(36)	120·7 (2)	C(12)–C(1)–O(1)	118·1 (6)
C(31)–N(1)–C(21)	123·7 (4)	C(12)–C(1)–C(3)	122·7 (6)
C(31)–N(1)–C(11)	123·0 (4)	O(1)–C(1)–C(3)	118·8 (6)
C(11)–N(1)–C(21)	112·2 (5)	C(1)–C(3)–C(2)	135·7 (6)
N(1)–C(11)–C(16)	121·0 (5)	C(3)–C(2)–C(22)	136·1 (6)
N(1)–C(11)–C(12)	118·4 (5)	C(2)–C(22)–C(21)	125·1 (6)
C(12)–C(11)–C(16)	120·7 (5)	C(2)–C(22)–C(23)	118·5 (6)
C(11)–C(12)–C(13)	119·3 (5)	C(21)–C(22)–C(23)	116·2 (6)
C(11)–C(12)–C(1)	121·6 (5)	C(22)–C(23)–C(24)	122·1 (6)
C(1)–C(12)–C(13)	119·1 (5)	C(23)–C(24)–C(25)	120·3 (7)
C(12)–C(13)–C(14)	120·3 (6)	C(24)–C(25)–C(26)	119·7 (6)
C(13)–C(14)–C(15)	120·4 (6)	C(25)–C(26)–C(21)	119·8 (6)
C(14)–C(15)–C(16)	119·6 (6)	C(26)–C(21)–C(22)	121·9 (6)

References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
EICHER, TH., ABDESAKEN, F., FRANKE, G. & WEBER, J. L. (1975). *Tetrahedron Lett.* pp. 3915–3918.
EICHER, TH. & FRANKE, G. (1981). *Justus Liebigs Ann. Chem.* pp. 1337–1353.
JOHNSON, C. K. (1976). *ORTEP* II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1980). *MULTAN* 80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
SHELDRICK, G. M. (1976). *SHELX.* A program for crystal structure determination. Univ. of Cambridge, England.
THIMME, M. (1982). Dissertation. *Versuche zur Synthese von Dibenzannelierten Azocin-4-onen.* Univ. of Dortmund, Federal Republic of Germany.
WAAL, B. W. VAN DE (1976). *POP* 1. Plot of packing program. Twente Univ. of Technology, The Netherlands.

Structure of *syn*-1,6:8,13-Bismethano-2-aza[14]annulene,* C₁₅H₁₃N

BY J. LEX AND EMANUEL VOGEL

Institut für Organische Chemie der Universität, D-5000 Köln 41, Federal Republic of Germany

AND MASSIMO SIMONETTA

Istituto di Chimica Fisica dell'Università e Centro CNR, Via Golgi 19, I 20133 Milano, Italy

Abstract. $M_r = 207·28$, monoclinic, $P2_1/c$, $a = 9·473$ (2), $b = 8·959$ (3), $c = 13·617$ (4) Å, $\beta = 109·34$ (2)°, $V = 1090·4$ (6) Å³, $Z = 4$, $D_m = 1·26$ (by flotation in dilute K_2HgI_4), $D_x = 1·262$ Mg m⁻³, $F(000) = 440$, Mo Kα, $\lambda = 0·71069$ Å, $\mu = 0·038$ mm⁻¹, room temperature, $R = 0·043$ for 1269 independent observed reflections. There are no unusual geometrical features.

Introduction. In recent years 1,6-methano-2-aza[10]annulene, the first example of an aza derivative of bridged annulenes, was synthesized (Schaefer-Ridder, Wagner, Schwanborn, Schreiner, Devrout & Vogel, 1978) and the molecular geometry of a bromo derivative has been determined by X-ray diffraction (Destro, Simonetta & Vogel, 1981). One homolog of this compound, namely *syn*-1,6:8,13-bismethano-2-aza[14]annulene (I), has now become available (Vogel, 1983) and the results of the study of its crystal structure are presented here.

---

* IUPAC name: 4-azatricyclo[8.4.1.1³·⁸]hexadeca-2,4,6,8,10,12-14-heptaene.

(I)   (II)
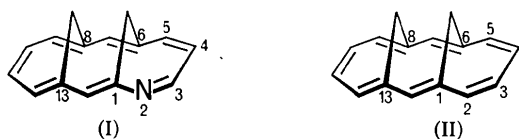
Experimental. Crystals of the title compound were obtained from ethyl acetate/hexane; single crystal (yellow-orange prism) 0·3 × 0·5 × 0·3 mm, Enraf–Nonius CAD-4 diffractometer, 2681 independent reflexions measured, $\theta \le 30°$, 1269 considered observed ($I \ge 2·5\sigma_i$) and used in crystal analysis; each reflection was assigned a variance $\sigma(I) = |\sigma(I_{raw})^2 + (pI_{raw})^2|^{\frac{1}{2}}$/Lp with $\sigma(I_{raw})$ based on counting statistics and $p = 0·011$; intensities and their standard deviations were corrected for Lorentz and polarization effects but not for absorption. Structure solved by direct methods and refined by least squares using the program *SHELX* (Sheldrick, 1976), non-H atoms anisotropic and H atoms isotropic; scattering factors for C and N atoms from Cromer & Mann (1968), those of Stewart, Davidson & Simpson (1965) used for H; refinement based on minimization of $\sum w(|F| - k|F_c|)^2$ with