

C(6)	0.7412 (2)	0.0132 (1)	0.0465 (2)	0.056 (1)
C(7)	0.6917 (2)	0.0391 (1)	-0.1298 (2)	0.057 (1)
C(8)	0.8729 (1)	0.1133 (1)	-0.0010 (2)	0.039 (1)
C(9)	0.7718 (2)	0.1369 (1)	-0.3022 (2)	0.062 (1)
C(10)	0.9391 (2)	0.0546 (1)	0.2760 (2)	0.065 (1)
O	0.9376 (1)	0.1834 (1)	0.0509 (1)	0.043 (1)
C(11)	1.0404 (1)	0.2042 (1)	-0.0145 (1)	0.038 (1)
O(11)	1.0740 (1)	0.1720 (1)	-0.1236 (1)	0.053 (1)
N	1.0973 (1)	0.2662 (1)	0.0584 (1)	0.044 (1)
C(12)	1.2050 (1)	0.3071 (1)	0.0221 (1)	0.038 (1)
C(13)	1.2259 (2)	0.3773 (1)	0.0869 (2)	0.048 (1)
C(14)	1.3258 (2)	0.4218 (1)	0.0524 (2)	0.057 (1)
C(15)	1.4055 (2)	0.3972 (1)	-0.0465 (2)	0.058 (1)
C(16)	1.3870 (2)	0.3270 (1)	-0.1073 (2)	0.061 (1)
C(17)	1.2882 (1)	0.2812 (1)	-0.0730 (2)	0.050 (1)

Table 2. Selected geometric parameters (Å, °)

C(1)—C(8)	1.527 (2)	C(5)—C(8)	1.530 (2)
C(1)—C(9)	1.527 (2)	C(5)—C(6)	1.546 (2)
C(1)—C(2)	1.530 (2)	C(6)—C(7)	1.541 (2)
C(1)—C(7)	1.546 (2)	C(8)—O	1.452 (2)
C(2)—C(3)	1.519 (2)	O—C(11)	1.345 (2)
C(3)—C(4)	1.518 (2)	C(11)—O(11)	1.211 (2)
C(4)—C(5)	1.534 (2)	C(11)—N	1.348 (2)
C(5)—C(10)	1.518 (2)	N—C(12)	1.413 (2)
O—C(8)—C(1)	114.92 (11)	C(10)—C(5)—C(8)	112.49 (12)
C(1)—C(8)—C(5)	103.94 (11)	C(10)—C(5)—C(4)	110.84 (13)
O(11)—C(11)—O	124.40 (12)	C(8)—C(5)—C(4)	108.95 (11)
O—C(11)—N	109.43 (10)	C(10)—C(5)—C(6)	113.85 (13)
C(11)—N—C(12)	127.38 (11)	C(8)—C(5)—C(6)	99.35 (10)
C(8)—C(1)—C(9)	114.40 (12)	C(4)—C(5)—C(6)	110.79 (12)
C(8)—C(1)—C(2)	109.12 (11)	C(7)—C(6)—C(5)	105.93 (11)
C(9)—C(1)—C(2)	110.47 (13)	C(6)—C(7)—C(1)	106.63 (11)
C(8)—C(1)—C(7)	98.93 (11)	O—C(8)—C(5)	109.60 (10)
C(9)—C(1)—C(7)	112.39 (12)	C(11)—O—C(8)	117.26 (10)
C(2)—C(1)—C(7)	111.02 (12)	O(11)—C(11)—N	126.16 (12)
C(3)—C(2)—C(1)	112.88 (13)	C(17)—C(12)—N	123.89 (12)
C(4)—C(3)—C(2)	112.18 (13)	C(13)—C(12)—N	116.79 (12)
C(3)—C(4)—C(5)	112.97 (13)		

The positions of the H atoms of (1) could be found from difference Fourier maps, but refinement resulted in slightly deformed arrangements. Thus, for final refinement, H atoms were set at calculated positions [except for H(N)].

Data collection and cell refinement were carried out with CAD-4 EXPRESS (Enraf-Nonius, 1993). All calculations were performed with SHELXS86 (Sheldrick, 1985) and SHELXL93 (Sheldrick, 1993). Molecular graphics were obtained with SCHAKAL92 (Keller, 1992).

SP thanks the Studienstiftung des deutschen Volkes for a doctoral fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1075). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Enraf-Nonius (1993). CAD-4 EXPRESS. Enraf-Nonius, Delft, The Netherlands.
- Haufe, G., Wolf, A. & Schulze, K. (1986). *Tetrahedron*, **42**, 4719–4728.
- Keller, E. (1992). SCHAKAL92. Fortran Program for the Graphic Representation of Molecular and Crystallographic Models. University of Freiburg, Germany.
- Pietz, S. & Haufe, G. (1995). 14te Vortragsstagung der Fachgruppe Biochemie der Gesellschaft Deutscher Chemiker, Kaiserslautern, Germany, 15–17 March. Abstracts of papers, p. 26.

- Sheldrick, G. M. (1985). SHELXS86. Program for the Solution of Crystal Structures. University of Göttingen, Germany.
- Sheldrick, G. M. (1993). SHELXL93. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.
- Vigne, B., Archelas, A., Fourmeron, J. D. & Furstoss, R. (1986). *Tetrahedron*, **42**, 3863–3869.
- Vigne, B., Archelas, A., Fourmeron, J. D. & Furstoss, R. (1987). *Nouv. J. Chem.* **11**, 297–320.
- Vigne, B., Archelas, A. & Furstoss, R. (1988). *Tetrahedron*, **44**, 1447–1458.
- Whitesell, J. K., Matthews, R. S. & Solomon, P. A. (1976). *Tetrahedron Lett.* pp. 1549–1552.

Acta Cryst. (1996). **C52**, 470–472

A 1:1 Molecular Complex of 1,5-Diphenylbiuret and Phenyl Carbamidonitrile

QING-CHUANG YANG,* DAN-MEI HUANG, HUI-YING CHEN AND YOU-QI TANG

Department of Chemistry, Peking University, Beijing 100871, China

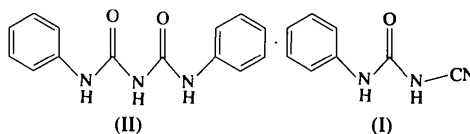
(Received 14 February 1995; accepted 7 August 1995)

Abstract

Crystals of the title compound, 1,5-diphenylbiuret-*N*-cyano-*N'*-phenylurea (1/1), C₁₄H₁₃N₃O₂·C₈H₇N₃O, were obtained from both ethanol and ethyl acetate solutions of phenyl carbamidonitrile. The structure of the complex features pairs of diphenylbiuret and phenyl carbamidonitrile molecules linked by hydrogen bonds. Excluding a tilted phenyl group, the molecule of 1,5-diphenylbiuret has a large, approximately coplanar, fragment in which all C—N bonds are partial double bonds.

Comment

In the course of our research on the relationship of the structure and properties of conjugated polynitriles, the monomeric structure of phenyl carbamidonitrile, (I), has been reported (Yang, Huang, Chen & Tang, 1995). While growing the crystals of (I) from both ethanol and ethyl acetate solutions of (I), we accidentally obtained crystals of a 1:1 complex of (I) with 1,5-diphenylbiuret, (II). It appears that a condensation-like reaction occurred in the solution. The melting point of the 1:1 complex (439–442 K) is much higher than the melting point of (I) (405–407 K).



The structure of the 1:1 complex contains pairs of molecules of (I) and (II) linked by hydrogen bonds, as shown in Fig. 1. The conformation of the molecule of (I) is similar to that found in pure phenyl carbamidonitrile (Yang, Huang, Chen & Tang, 1995), with a nearly planar carbamidonitrile group (mean deviation 0.08 Å). The dihedral angle between the phenyl ring and the plane of the carbamidonitrile group is 30.5°. Except for the C(11)—N(12) nitrile bond, the C—N bonds in the molecule of (I) are partial double bonds (Gilli, Bertolasi, Bellucci, Fabrizio & Ferretti, 1986) ranging from 1.286 (3) to 1.408 (3) Å. Both N(7) and N(10) are sp^2 -hybridized.

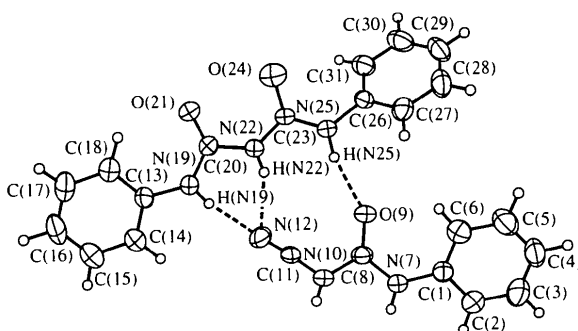


Fig. 1. The structure of the title complex with the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level for non-H atoms; H atoms are shown as spheres of arbitrary radii.

The molecule of (II) adopts an extended conformation and contains an approximately coplanar fragment consisting of 13 atoms from C(13) to N(25) [mean deviation from best plane 0.08 Å; maximum deviation for N(25) 0.23 Å]. The C—N bond lengths in the fragment [1.324 (3)–1.414 (3) Å] are also in the range expected for partial double C—N bonds. This indicates that there is a large delocalized π -electron system through the 13 atoms. The second phenyl ring in the molecule of (II) is tilted by 62.4° with respect to the large planar fragment, to which it is connected by a single C(26)—N(25) bond of length 1.431 (3) Å.

Intermolecular hydrogen bonds connect the nitrile N atoms and the carbonyl O atoms of each molecule of (I) (as acceptors) with the N atoms of the nearest molecule of (II) (as donors), forming 1:1 molecular pairs. These, in turn, pack with van der Waals' interactions in the crystal. It is thought that the *cis* conformation of the two carbonyl groups in the molecule of (II) is due to the intermolecular hydrogen bond shown in Fig. 1. The dihedral angle between the phenyl rings C(1)—C(6) and C(13)—C(18) in each pair is only 13.5°.

Experimental

The title crystals can be obtained from ethanol or ethyl acetate solutions of phenyl carbamidonitrile by the vapour-diffusion method with a pentane precipitant.

Crystal data

$C_{14}H_{13}N_3O_2 \cdot C_8H_7N_3O$
 $M_r = 416.4$
 Triclinic
 $P\bar{1}$
 $a = 7.638 (2) \text{ \AA}$
 $b = 10.603 (3) \text{ \AA}$
 $c = 14.364 (5) \text{ \AA}$
 $\alpha = 107.63 (2)^\circ$
 $\beta = 94.57 (2)^\circ$
 $\gamma = 107.12 (2)^\circ$
 $V = 1041.2 (5) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.328 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 7.06\text{--}10.88^\circ$
 $\mu = 0.086 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Block prism
 $0.5 \times 0.5 \times 0.5 \text{ mm}$
 Colourless

Data collection

Siemens R3m/V diffractometer
 θ/θ scans
 Absorption correction: none
 3668 measured reflections
 3668 independent reflections
 3133 observed reflections
 $[F > 2.0\sigma(F)]$

$\theta_{\max} = 26.0^\circ$
 $h = 0 \rightarrow 9$
 $k = -12 \rightarrow 12$
 $l = -17 \rightarrow 16$
 2 standard reflections monitored every 100 reflections
 intensity decay: 1.0%

Refinement

Refinement on I
 $R[F > 2.0\sigma(F)] = 0.0578$
 $wR = 0.0637$
 $S = 2.45$
 3133 reflections
 281 parameters
 H-atom parameters not refined
 $w = 1/[\sigma^2(F) + 0.0002F^2]$
 $(\Delta/\sigma)_{\max} = 0.033$

$\Delta\rho_{\max} = 0.49 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$
 Extinction correction: empirical, isotropic
 Extinction coefficient: $x = 0.005 (2)$
 Atomic scattering factors from SHELXTL-Plus (Sheldrick, 1990)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms, $U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$ for all others.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}/U_{\text{eq}}$
C(1)	0.3220 (3)	0.4783 (2)	0.6635 (2)	0.042 (1)
C(2)	0.2574 (4)	0.5210 (3)	0.7511 (2)	0.059 (1)
C(3)	0.3572 (5)	0.5355 (3)	0.8405 (2)	0.074 (2)
C(4)	0.5223 (5)	0.5089 (3)	0.8441 (2)	0.070 (1)
C(5)	0.5878 (4)	0.4693 (3)	0.7580 (2)	0.071 (1)
C(6)	0.4901 (3)	0.4542 (3)	0.6680 (2)	0.058 (1)
N(7)	0.2156 (3)	0.4658 (2)	0.5745 (1)	0.044 (1)
C(8)	0.2074 (3)	0.3801 (2)	0.4812 (2)	0.040 (1)
O(9)	0.3025 (2)	0.3029 (2)	0.4617 (1)	0.053 (1)
N(10)	0.0819 (3)	0.3899 (2)	0.4128 (1)	0.045 (1)
C(11)	0.0378 (3)	0.2944 (3)	0.3263 (2)	0.043 (1)
N(12)	-0.0141 (3)	0.2099 (2)	0.2468 (1)	0.056 (1)
C(13)	0.0576 (3)	0.1889 (2)	-0.0233 (2)	0.042 (1)
C(14)	-0.1009 (4)	0.2274 (3)	-0.0198 (2)	0.059 (1)
C(15)	-0.1808 (4)	0.2472 (3)	-0.1018 (2)	0.072 (1)
C(16)	-0.1062 (4)	0.2277 (3)	-0.1863 (2)	0.074 (2)
C(17)	0.0487 (4)	0.1891 (3)	-0.1895 (2)	0.074 (2)
C(18)	0.1335 (4)	0.1696 (3)	-0.1083 (2)	0.057 (1)
N(19)	0.1293 (3)	0.1709 (2)	0.0638 (1)	0.044 (1)
C(20)	0.2793 (3)	0.1346 (2)	0.0818 (2)	0.040 (1)
O(21)	0.3789 (2)	0.1028 (2)	0.0233 (1)	0.054 (1)
N(22)	0.3085 (2)	0.1363 (2)	0.1798 (1)	0.044 (1)
C(23)	0.4562 (3)	0.1202 (2)	0.2267 (2)	0.037 (1)

O(24)	0.5769 (3)	0.0753 (2)	0.1765 (1)	0.068 (1)
N(25)	0.4719 (3)	0.1497 (2)	0.3240 (1)	0.044 (1)
C(26)	0.5938 (3)	0.1142 (2)	0.3850 (2)	0.038 (1)
C(27)	0.6982 (3)	0.2160 (3)	0.4721 (2)	0.047 (1)
C(28)	0.8042 (3)	0.1816 (3)	0.5362 (2)	0.058 (1)
C(29)	0.8050 (4)	0.0471 (3)	0.5142 (2)	0.063 (1)
C(30)	0.7025 (4)	-0.0546 (3)	0.4266 (2)	0.061 (1)
C(31)	0.5965 (3)	-0.0211 (3)	0.3615 (2)	0.051 (1)
H(N19)	0.0774	0.1860	0.1156	0.080
H(N22)	0.2358	0.1603	0.2150	0.080
H(N25)	0.4099	0.1987	0.3575	0.080

Table 2. Selected geometric parameters (Å, °)

C(1)—N(7)	1.408 (3)	N(19)—C(20)	1.342 (3)
N(7)—C(8)	1.357 (3)	C(20)—O(21)	1.216 (3)
C(8)—O(9)	1.233 (3)	C(20)—N(22)	1.402 (3)
C(8)—N(10)	1.366 (3)	N(22)—C(23)	1.349 (3)
N(10)—C(11)	1.286 (3)	C(23)—O(24)	1.325 (3)
C(11)—N(12)	1.167 (3)	C(23)—N(25)	1.324 (3)
C(13)—N(19)	1.414 (3)	N(25)—C(26)	1.431 (3)
C(1)—N(7)—C(8)	127.6 (2)	N(19)—C(20)—N(22)	110.6 (2)
N(7)—C(8)—O(9)	123.4 (2)	O(21)—C(20)—N(22)	122.9 (2)
N(7)—C(8)—N(10)	111.9 (2)	C(20)—N(22)—C(23)	126.8 (2)
O(9)—C(8)—N(10)	124.7 (2)	N(22)—C(23)—O(24)	120.8 (2)
C(8)—N(10)—C(11)	116.4 (2)	N(22)—C(23)—N(25)	116.7 (2)
N(10)—C(11)—N(12)	175.0 (3)	O(24)—C(23)—N(25)	122.5 (2)
C(13)—N(19)—C(20)	128.7 (2)	C(23)—N(25)—C(26)	125.8 (2)
N(19)—C(20)—O(21)	126.6 (2)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	D...A	D—H...A
N(19)—H(N19)...N(12)	0.866 (2)	2.889 (3)	170.7 (2)
N(22)—H(N22)...N(12)	0.823 (2)	2.942 (3)	156.1 (1)
N(25)—H(N25)...O(9)	0.868 (2)	2.814 (3)	165.5 (1)

H atoms bonded to N atoms were located from a difference Fourier map, while those bonded to C atoms were introduced in calculated positions and refined using a riding model with fixed isotropic U 's.

Data collection: *P3/V Data Collection Program* (Siemens, 1989). Cell refinement: *P3/V Data Collection Program*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

The project was supported by the National Natural Science Foundation of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Gilli, G., Bertolasi, V., Bellucci, F., Fabrizio, B. & Ferretti, V. (1986). *J. Am. Chem. Soc.* **108**, 2420–2424.
- Sheldrick, G. M. (1990). *SHELXTL-Plus. Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1989). *P3/V Data Collection Program*. Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Yang, Q.-C., Huang, D.-M., Chen H.-Y. & Tang, Y.-Q. (1995). *Acta Cryst.* **C51**, 1412–1414.

Acta Cryst. (1996). **C52**, 472–474

N-[2-(1-3*H*-Indenyl)ethyl]-*p*-toluenesulfonamide

GRAEME J. GAINSFORD* AND CORNELIS LENSINK

The New Zealand Institute for Industrial Research and Development, Materials Chemistry, PO Box 31-310, Lower Hutt, New Zealand. E-mail: g.gainsford@irl.cri.nz

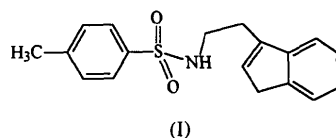
(Received 23 May 1995; accepted 19 September 1995)

Abstract

Lithium indene reacts with *N-p*-toluenesulfonylaziridene to yield *N*-[2-(1-3*H*-indenyl)ethyl]-*p*-toluenesulfonamide, C₁₈H₁₉NO₂S. The structure consists of independent molecules with the indene group substituted at the expected 1 position of the five-membered ring. The sulfonamido moiety has a planar arrangement about the N atom.

Comment

The current interest in bidentate cyclopentadiene alkoxides and amides as ligands (Hughes, Meetsma & Teuben, 1993) for early transition metals prompted us to investigate the synthesis of molecules of this type *via* the reaction of cyclopentadienyls with aziridenes. The title compound, (I), was prepared by the reaction of the indenyl anion with *N-p*-toluenesulfonylaziridene. An analogous reaction has been described for the substitution of epoxides with fluorenyl or cyclopentadienyl to yield cyclopentadiene alkoxides (Rieger, Steimann & Fawzi, 1992).



The structure of (I) (Fig. 1, Table 1) shows it to be a 1,2-disubstituted ethane with a regular indenyl moiety substituted at the C atom in position 1 of the five-membered ring and having the double bond in the 1 position [C1=C2 1.340 (4) Å], as expected from NMR evidence. The sulfonamido moiety displays a basically planar N atom, with the bound H atom 0.25 (3) Å from the plane through atoms S1, N1, C10; the closest intermolecular contact is 2.23 Å for O2...H1(N1). Only C2 is significantly out of the indenyl plane [0.023 (4) Å]; there is an angle of 40.2 (1)° between this and the plane of the toluene moiety. There are no significant deviations from expected bond lengths and angles in the molecule (*International Tables for Crystallography*, 1992, Vol. C, Table 9.5.1.1).