Strained Molecules: Crystal Structure of 2,2,3-Triphenyl-1-azabicyclo[1.1.0]butane

Romuald Bartnik,[a] Zofia Cebulska[a] and René Faure*[b]

[a] Department of Applied Organic Chemistry, University of Lodz, Narutowicza 65, 90-136 Lodz, Poland
[b] Laboratoire de Chimie Analytique II, Université Claude Bernard Lyon I, 43 boulevard du 11 Novembre 1918, 69622 Villeurbanne Cedex, France

The first X-ray crystal structure determination of a 1-azabicyclo[1.1.0]butane derivative is reported.

Highly strained bicyclic molecules have attracted considerable attention during the last decades.[1] Among them the 1-azabicyclo[1.1.0]butane system is of great interest because of its unique chemical reactivity[2–5] and supposedly rigid structure. No crystal study of this system has been published in contrast with the bicyclo[1.1.0]butane[6–8] and azabicyclo[2.1.0]pentane[9,10] systems.

We now present the first X-ray crystal structure of a 1-azabicyclo[1.1.0]butane derivative. 2,2,3-Triphenyl-1-azabicyclo[1.1.0]butane was prepared using a photochemical

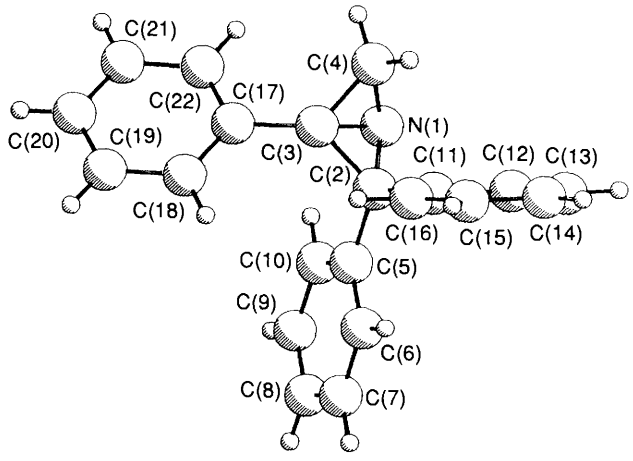

Fig. 1 PLUTO[13] drawing showing the atom numbering scheme

method described by us several years ago.[11] It was difficult to obtain suitable crystals for X-ray analysis because of relatively easy polymerization. Nevertheless we obtained monocrystals from hexane; freshly prepared crystals melt at 100–101 °C.

The structure analysis† shows that the interflap angle (the dihedral angle between the planes of the two aziridine rings) is 118.7(4)°. The phenyls at C(2) are almost perpendicular to one another [82.4(2)°]. Bond distances and angles of the 1-azabicyclo[1.1.0]butane skeleton are very close to those of bicyclo[1.1.0]butane.[6–8] Both C–N and C–C bond distances vary from 1.467(8) to 1.521(6) Å and bond angles from 58.3(3) to 61.5(3)°.

Received, 5th October 1992; Com. 2/05348I

† *Crystal data*: $C_{21}H_{17}N$, M = 283.4, monoclinic, space group $P2_1/c$, a = 28.71(1), b = 8.633(2), c = 33.96(1) Å, β = 158.17(4)°, V = 3130(3) Å^3, Z = 8, D_c = 1.07 g cm^{-3}. Data were collected on a Nonius CAD4 diffractometer. Of the 5896 unique reflections measured [$2\theta_{max}$ = 146°, μ(Cu-Kα) = 4.8 cm^{-1}], 3109 had $I > 3\sigma(I)$ and were used for all calculations with the Standard Diffraction Package.[12] The two molecules of the asymmetric unit were found from MULTAN. The hydrogen atoms were fixed at idealized positions. The final refinement gave R = 0.087. The two independent molecules of the asymmetric unit show nearly the same conformation. Atomic coordinates, bond lengths and angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. See Notice to Authors, Issue No. 1.

References

1 W. E. Billups, M. M. Haley and G. A. Lee, *Chem. Rev.*, 1989, **89**, 1147.
2 A. G. Hortmann and D. A. Robertson, *J. Am. Chem. Soc.*, 1972, **94**, 2758.
3 W. Funke, *Angew. Chem., Int. Ed. Engl.*, 1969, **8**, 70.
4 G. Alvernhe, A. Laurent, K. Touhami, R. Bartnik and G. Mloston, *J. Fluorine Chem.*, 1985, **29**, 363.
5 A. Galindo, Thesis, University of Lodz, 1990.
6 P. G. Gassman, M. L. Greenlee, D. A. Dixon, S. Richtsmeier and J. Z. Gougoutas, *J. Am. Chem. Soc.*, 1983, **105**, 5865.
7 P. L. Johnson and J. P. Schaefer, *J. Org. Chem.*, 1972, **37**, 2762.
8 P. G. Gassman and M. J. Mullins, *Tetrahedron Lett.*, 1979, 4457.
9 B. Fouchet, M. Joucla and L. Toupet, *Acta Crystallogr., Sect. C*, 1983, **39**, 497.
10 Y. Kodama and T. Ito, *Agric. Biol. Chem.*, 1980, **44**, 73.
11 R. Bartnik, Z. Cebulska, A. Laurent and B. Orlowska, *J. Chem. Res. (S)*, 1986, 5.
12 B. A. Frenz and Associates inc., *SDP Structure Determination Package*, College Station, Texas, USA, 1982.
13 W. D. S. Motherwell and W. Clegg, PLUTO: *Program for plotting molecular and crystal structures*, University of Cambridge, England, 1978.