Cryst.Struct.Comm. (1982). *11*, 999.

1-(2,4-DIBROMOPHENYLAMINO)-5-PHENYL-TETRAZOLE, $C_{13}H_9N_5Br_2$

Z Dauter*, S A Chawdhury & M A Hamid†
Physics Department, University of York, York YO1 5DD, UK

PRELIMINARY INFORMATION

The crystal structure of the title compound was undertaken to determine the position of the dibromophenyl substituent in the molecule and establish the conformation of 5 or 6 membered ring containing N atoms as there was an ambiguity in the molecular configuration of the related compounds (Gibson, 1962). The conventional spectroscopic and chemical methods failed to establish the proposed structure unambiguously.

CRYSTAL DATA

(From Weissenberg photographs and single crystal diffractometry CuK_α = 1.5418 Å) Monoclinic, a = 8.413(3), b = 21.495(5), c = 8.003(3)Å, β = 92.84(5)°, Dx = 1.81 g cm^{-3} for Z = 4; systematic absences for 0k0 reflexions with k = 2n+1 and for h0l reflexions with l = 2n+1, space group $P2_1/c$.

*Permanent address: Department of Pharmaceutical Technology, Technical University, 80 952 Gdansk, Poland.

†Present address : Department of Physics, Alfateh University, Tripoli Libya.

INTENSITY DATA, STRUCTURE DETERMINATION AND REFINEMENT

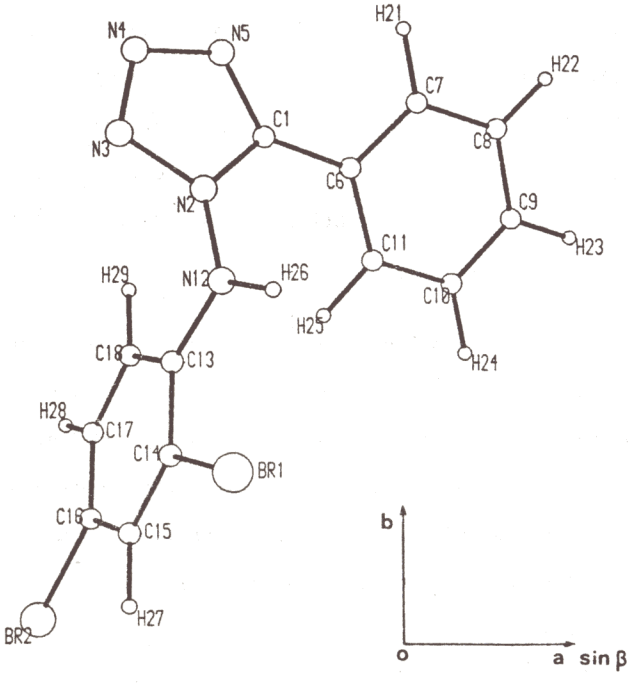
The intensities of 1495 unique reflexions were measured on a PDP8 computer controlled Hilger and Watts four-circle diffractometer. The E-map corresponding to the phase set with highest combined figure of merit produced by the MULTAN program (Main, 1980) showed two bromine atoms and five membered ring. The subsequent atoms were located in the difference map. The structure was refined by full-matrix least squares, using anisotropic temperature factors for non-hydrogen atoms and isotropic temperature factors for all the hydrogen atoms. The positions of all the hydrogen atoms were calculated geometrically. The final conventional R-factor was 6.8%. The final atomic coordinates, some intra-molecular bond distances and angles are listed in the tables.

ATOMIC CO-ORDINATES ($\times 10^4$)

	x/a(σ)	y/b(σ)	z/c(σ)		x/a(σ)	y/b(σ)	z/c(σ)
C(1)	4423(15)	2387(5)	8751(14)	C(11)	6203(16)	1599(6)	7560(15)
N(2)	3470(12)	2028(4)	9704(11)	N(12)	3763(11)	1444(4)	10421(11)
N(3)	2177(12)	2367(5)	10143(11)	C(13)	2946(14)	934(5)	9690(14)
N(4)	2383(14)	2917(5)	9481(13)	C(14)	2959(13)	362(5)	10521(13)
N(5)	3733(12)	2939(4)	8617(12)	C(15)	2267(14)	-151(5)	9806(16)
C(6)	5837(14)	2204(6)	7989(13)	C(16)	1509(14)	-104(6)	8244(16)
C(7)	6966(18)	2652(5)	7634(14)	C(17)	1472(15)	448(6)	7350(16)
C(8)	8360(17)	2503(7)	6903(16)	C(18)	2161(15)	971(6)	8123(16)
C(9)	8635(16)	1896(7)	6468(16)	Br(19)	4003(2)	307(1)	12659(2)
C(10)	7570(18)	1447(6)	6811(17)	Br(20)	528(2)	-826(1)	7254(2)

BOND DISTANCES (Å)

C(1) - N(2)	1.371(13)	C(9) - C(10)	1.354(17)
C(1) - N(5)	1.324(13)	C(10) - C(11)	1.363(16)
C(1) - C(6)	1.419(15)	N(12) - C(13)	1.406(13)
N(2) - N(3)	1.370(11)	C(13) - C(14)	1.398(14)
N(2) - N(12)	1.397(12)	C(13) - C(18)	1.391(15)
N(3) - N(4)	1.312(12)	C(14) - C(15)	1.360(14)
N(4) - N(5)	1.359(12)	C(14) - Br(19)	1.888(11)
C(6) - C(7)	1.392(15)	C(15) - C(16)	1.379(16)
C(6) - C(11)	1.384(15)	C(16) - C(17)	1.386(16)
C(7) - C(8)	1.374(15)	C(16) - Br(20)	1.910(12)
C(8) - C(9)	1.372(16)	C(17) - C(18)	1.394(16)



The molecule viewed along the [001] direction and numbering scheme of the atoms

BOND ANGLES (^{o})

(Standard deviations are about 0.9^{o})

N(2) - C(1) - N(5)	106.5	C(9) - C(10) - C(11)	120.0
N(2) - C(1) - C(6)	127.5	C(6) - C(11) - C(10)	122.5
N(5) - C(1) - C(6)	126.0	N(2) - N(12) - C(13)	117.2
C(1) - N(2) - N(3)	109.7	N(12)- N(13) - C(14)	119.7
C(1) - N(2) - N(12)	129.6	N(12)- C(13) - C(18)	122.1
N(3) - N(2) - N(12)	119.9	C(14)- C(13) - C(18)	118.2
N(2) - N(3) - N(4)	104.7	C(13)- C(14) - C(15)	121.3
N(3) - N(4) - N(5)	111.5	C(13)- C(14) - Br(19)	118.6
N(4) - N(5) - C(1)	107.6	C(15)- C(14) - Br(19)	120.1
C(1) - C(6) - C(7)	119.4	C(14)- C(15) - C(16)	119.4
C(1) - C(6) - C(11)	124.6	C(15)- C(16) - C(17)	121.9
C(7) - C(6) - C(11)	116.0	C(15)- C(16) - Br(20)	119.2
C(6) - C(7) - C(8)	122.0	C(17)- C(16) - Br(20)	118.9
C(7) - C(8) - C(9)	119.3	C(16)- C(17) - C(18)	117.9
C(8) - C(9) - C(10)	120.0	C(13)- C(18) - C(17)	121.5

COMMENTS

The results serve to confirm the structure proposed on the basis of the chemical information. The ring containing N atoms is five-membered. A projection of the molecule when viewed along [001] direction is shown in the figure where the details of the molecular geometry are shown. The two phenyl rings and the five-membered rings are planar within limits of accuracy. Some calculation of planes and the displacement of atoms are shown in the table.

DISTANCES FROM PLANES (Å)

Plane 1		Plane 2		Plane 3	
C(1)*	-0.016	C(6)*	0.005	C(13)*	0.008
N(2)*	-0.011	C(7)*	0.002	C(14)*	-0.002
N(3)*	0.013	C(8)*	-0.012	C(15)*	0.003
N(4)*	0.002	C(9)*	0.011	C(16)*	-0.009
N(5)*	-0.005	C(10)*	-0.001	C(17)*	0.017
C(6)*	0.017	C(11)*	-0.007	C(18)*	-0.017
N(12)	-0.215	C(1)	0.015	N(12)	0.091
				Br(19)	0.014
				Br(20)	-0.013

*Atoms included in calculation of the plane

Angles between planes 1 & 2 = 26°, 1 & 3 = 90° and 2 & 3 = 89°.

ACKNOWLEDGEMENT

The authors are indebted to Professor M M Woolfson for his keen interest in the work.

REFERENCES

Gibson, M S (1962) Tetrahedron, 1377

Main, P (1980) MULTAN'80 - A system of computer program for the automatic solution of crystal structures from X-ray diffraction data, University of York, England.

Received: 19 February 1982