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The crystal structure of a compartmental heptadentate ligand

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Abstract

2-(2-hydroxyphenyl)-1,3-bis[4-(2-hydroxyphenyl)-3-azabut-3-enyl]-1,3-imidazolidine (H_3L) was obtained with high purity by Schiff's condensation between 5-bromosalicyaldehyde and triethylenetetramine. Its recrystallisation in methanol yields single crystals suitable for X-ray diffraction studies. H_3L crystallises in the monoclinic system, space group $P2_1/c$, with a = 21.641(3) Å, b = 11.1217(14) Å, c = 11.4156(15) Å and β = 90.016 º.

Introduction

The design and synthesis of multidentate ligands is an essential step in the search for new polynuclear complexes of predefined architectures, an area of growing significance in the field of molecular materials. Following with our work in this topic, we describe herein the crystal structure of the potential heptadentate compartmental donor H_3L.

Results and discussion

A perspective view of the molecule is shown in Fig. 1. All the angles and distances (Table 1) are in the range of those expected for this kind of Schiff base and do not deserve further consideration [1]. The crystal data show that the central phenol oxygen atom is disordered over two sites (O103 and O13') at 0.5 partial occupancies.

The ligand adopts a conformation which is determined by the formation of a five-membered imidazolidine ring and by the presence of strong intramolecular hydrogen bonds between the phenol functions and the nitrogen atoms. Accordingly, the terminal phenol oxygen atoms interact with the imine nitrogen atoms (distance O101···N101 = 2.607(4) Å and O102···N102 = 2.598(4) Å). Besides, the disordered central phenol function is hydrogen bonded to both imidazolidine nitrogen atoms (distance O103···N102 = 2.790(6) Å and O13'···N104 = 2.784(6) Å).

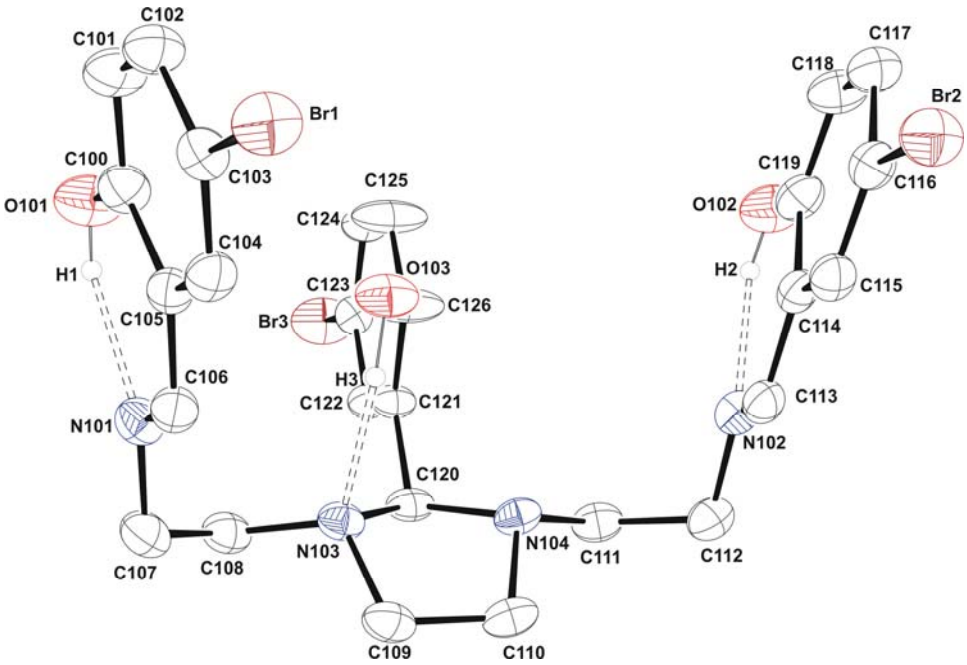

Fig.1

Table 1. Selected distances (Å) and angles (º) for H_3L

O101-C100	1.343(4)	O101-C100-C101	118.9(3)
C103-Br1	1.900(3)	O101-C100-C105	121.3(3)
N101-C106	1.274(4)	C104-C103-Br1	119.9(3)
N101-C107	1.454(5)	C102-C103-Br1	119.1(3)
C108-N103	1.454(4)	C106-N101-C107	117.9(3)
N103-C120	1.464(4)	C108-N103-C120	113.3(3)
N103-C109	1.476(4)	C108-N103-C109	113.8(3)
C110-N104	1.476(4)	C120-N103-C109	103.5(3)
N104-C111	1.455(4)	C111-N104-C120	113.2(3)
N104-C120	1.467(4)	C111-N104-C110	114.1(3)
N102-C113	1.273(4)	C120-N104-C110	103.5(3)
C116-Br2	1.903(3)	C113-N102-C112	118.2(3)
C119-O102	1.340(4)	C115-C116-Br2	120.0(3)
C123-Br3	1.888(3)	C117-C116-Br2	118.7(3)
C126-O103	1.463(7)	O102-C119-C118	119.4(3)
C126-O13'	1.474(7)	O102-C119-C114	121.4(4)
O101-C100	1.343(4)	N103-C120-N104	100.5(2)
C103-Br1	1.900(3)	C124-C123-Br3	118.6(2)
C106-N101	1.274(4)	C122-C123-Br3	120.6(2)
N101-C107	1.454(5)	C121-C126-O103	117.8(4)
N103-C120	1.464(4)	C125-C126-O103	116.1(4)
N103-C109	1.476(4)	C121-C126-O13'	117.2(4)
		C125-C126-O13'	116.1(4)

Experimental

H_4L was isolated as follows: 5-bromo-salicylaldehyde (0.87 g, 4.32 mmol) was added to a methanol (10 mL) solution of triethylentetramine (0.21 g, 1.44 mmol). The solution was stirred in air for 4 h. and a yellow solid precipitated, being subsequently filtered off. (0.800 g, 80 %), mp = 156-158 ºC.

References

[1] L-W. Yang, S. Liu, E. Wong, S. J. Rettig, C. Orvig, *Inorg. Chem.* **1995**, *34*, 2164.