

## Crystal structure of H<sub>4</sub>L (N-N'-Bis(o-hydroxybenzoyl) propylenediamine

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### ABSTRACT

One bis-amide bis-phenoxi N<sub>2</sub>O<sub>2</sub> ligand was obtained from the 2:1 molar reaction of phenyl salicylate and the diamine, 1,3-Diaminopropane, to yield H<sub>4</sub>L. The ligand has been characterised by elemental analysis, IR, and <sup>1</sup>H <sup>13</sup>C NMR spectroscopies, mass spectrometry (ES) and X-ray diffraction.

### INTRODUCTION

Our interest in this kind of ligands derives from the known ability of ligands containing amide groups to stabilise high oxidation states of metal ions when coordinated with the deprotonated nitrogen atom. We have thought that the utilization of tetranionic bis-amide bis-phenoxi ligands should favour the aggregation of neighbouring complexes through these donor atoms<sup>1</sup> (amide and phenoxo oxygen). In previous papers we reported on the synthesis, structures and properties of Mn(III) complexes with asymmetrical trianionic amido-imino-phenoxo ligands. At this goal we have designed the tetranionic ligand, H<sub>4</sub>L<sup>b</sup> that contain six potential donor atoms: two amide nitrogen, two phenoxo and two amide oxygen atoms

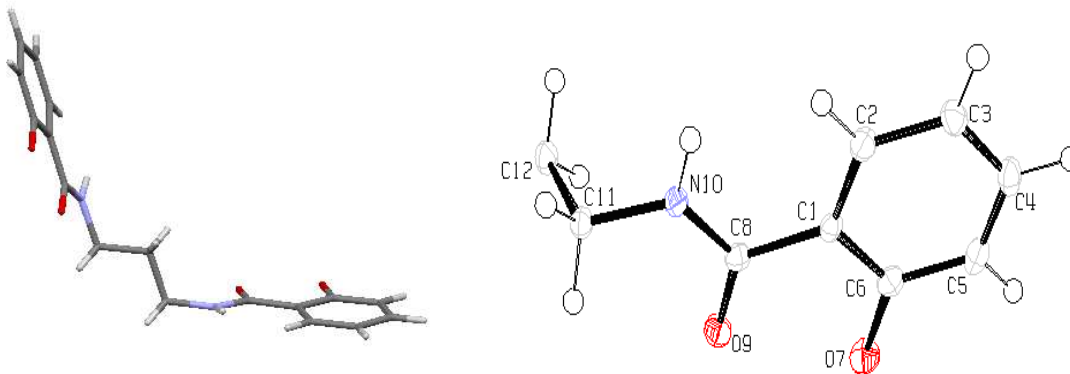
### RESULTS AND DISCUSSION

H<sub>4</sub>L were prepared according to the literature<sup>2</sup>, in this case by reaction in a 2:1 molar reaction of phenyl salicylate and the diamine at 180-190 ° for 1 h. The product was treated with diethyl ether to obtain a white powder at almost quantitative yield. White crystals of H<sub>4</sub>L, suitable for single crystal X-ray diffraction studies, were obtained by slow evaporation of methanol solution of the ligand. The characteristics of the ligand is:

Ligand H<sub>4</sub>L: M.p.177 °C. Anal Calc. for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub>: C,64.9; H, 5.7; N, 8.9. Found: C,63.8; H, 6.1; N,9.1%. MS ES (m/z): 315; IR (KBr, cm<sup>-1</sup>): ν(N-H) 3377, ν(O-H) 3077, ν(amido I) 1645, ν(amido II) 1545, ν(C-O) 1246. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, ppm): δ 8.81 (s: singlet, 2H), 6.83-6.88 (m: multiplet, 4H), 7.37 (t: triplet, 2H), 7.80 (d: doublet, 2H), 3.34-3.48 (m, 4H), 1.82 (q, 2H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, ppm): δ 161.3 (C-OH), 169.7 (C=O), 37.4 (CH<sub>2</sub>), 29.6 (CH<sub>2</sub>).

Crystal structure, with the numbering scheme, is shown in figure 1. Selected bond lengths and angles, as well as potential hydrogen bonds and bonding scheme, are listed in table 1. In the crystal structure, the title ligand C<sub>17</sub>H<sub>16</sub>N<sub>2</sub>O<sub>4</sub> exhibits crystallographic twofold rotation symmetry. In the ligand H<sub>4</sub>L exists two identical parts and so indicate carbon atoms to the number 12. The imine group is coplanar with the aromatic ring with an N-C-C-C torsion angle of -177.48(15) °.

The C(8)-O(9) and C(8)- distance of 1,253, are consistent with C=O double bonding of amide groups. The oxygen O(7) atom is forming phenolic group, and they present C-O distances are forming and O(7)-C(6) of 1.357 (3) Å, corresponding to the expected single bonds. C-C distances chain amine C (11) - C (12) 1.522 (1) Å are longer than the distances C-C aromatic ring C (2) - C (3) 1.383 Å, which is consistent with a single bond and double bond parameters respectively.



This molecule has intramolecular hydrogen bond O (7) - H (7) ... O (9) between the phenolic oxygen O (7) and amide oxygen O(9). It also presents an intermolecular hydrogen bond between the nitrogen N (10) - H (10) and the phenolic oxygen O (7) of another molecule.  $\pi$  interactions is further appreciated - stacking between the benzene rings (Table 2, Figure 2)

**Table1:** Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) for  $\text{H}_4\text{L}$ .

H(10) - N(10)	0,89(2)	C(11) - C(12)	
C(6) - O(7)	1,357(3)	C(11) - C(12)	
C(8) - O(9)	1,253(2)	C(12) - H(12A)	
C(8) - N(10)	1,331(3)	C(12) - H(12A)	
N(10) - C(11)	1,458(3)	C(12) - H(12B)	
C(11) - H(11A)	0,990(2)	C(12) - H(12B)	
C(11) - H(11B)	0,990(2)		
C(2)-C(1)-C(8)	123,8(2)	H(11A)-C(11)-C(12)	109,2(2)
C(6)-C(1)-C(8)	118,2(2)	C(11)-C(12)-C(11)	111,2(2)
C(1)-C(6)-O(7)	120,9(2)	C(11)-C(12)-H(12A)	109,4(1)
C(5)-C(6)-O(7)	118,6(2)	H(12A)-C(12)-H(12A)	108,0(3)
C(1)-C(8)-O(9)	120,6(2)	H(12A)-C(12)-H(12B)	0,0
C(1)-C(8)-N(10)	118,2(2)	H(11A)-C(11)-C(12)	109,2(2)
O(9)-C(8)-N(10)	121,2(2)	N(10)-C(11)-H(11A)	109,2(2)
H(10)-N(10)-C(8)	120,4(13)	N(10)-C(11)-C(12)	112,3(2)
H(10)-N(10)-C(11)	115,9(13)	H(11A)-C(11)-H(11B)	107,9(2)
C(8)-N(10)-C(11)	123,1(2)		

**Table2.-** Hydrogen bonds

D-H...A	d(D-H)	d(H...A)	d(D...A)	(DHA)
O(7)-H(7)-----O(9)	0,93(3)	1,63(3)	2,483(13)	154 (13)
N(10) -H(10) O(7)*	0,89(2)	2,03(2)	2,8509(18)	153,0(19)

symmetry operations \*=  $x, -y, 1/2+z$

