Asian Journal of Chemistry; *Vol. 25, No. 17 (2013), 10004-10006*

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.16062

Synthesis and Crystal Structure of 1,4-*bis*{[(2'-Thenylaminoformyl)phenoxyl]methyl}benzene

LI WANG, XIAO-RUN WANG, GUO-QUAN CHENG, YUN-QIAO PENG and XUE-QIN SONG*

School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China

*Corresponding author: Fax: +86 931 4938755; Tel: +86 931 8843385; E-mail: songxq@mail.lzjtu.cn; songxueqin001@163.com

(*Received*: 18 July 2013; *Accepted*: 30 October 2013) AJC-14325

A novel salicylamide derivative *i.e.*, 1,4-*bis*{[(2'-thenylaminoformyl)phenoxyl]methyl}benzene with the m.f. $C_{32}H_{28}N_2O_4S_2$ has been synthesized and the crystal structure was determined by single crystal X-ray diffraction. There are strong intramolecular hydrogen bonding between the amide nitrogen atoms and the ethereal oxygen atoms which stabilized the structure to form a *trans* configuration. Furthermore the title compound are linked by intermolecular C9-H9…O1 hydrogen bonds into an infinite 1D chain parallel to the *bc* plane .

Key Words: Salicylamide derivative, Synthesis, *trans*-Configuration, Crystal structure.

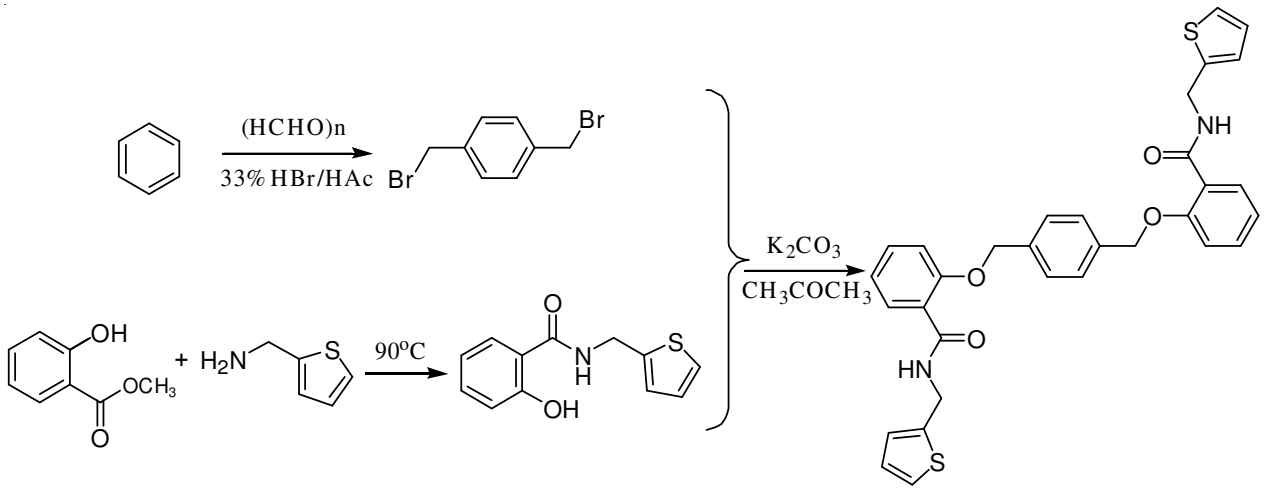
INTRODUCTION

In the last decade, synthesis of salicylamide-derived ligands have been carried out because of their attractive sensitization of lanthanide luminescence as well as construction of novel metal-organic frameworks[1]. So far a series of salicylamide ligands which incorporated different backbones and terminal groups have been synthesized[2-5]. We are systematically studying the structure and the photophysical properties of complexing salicylamide ligands of capable of sensitizing Eu(III) and Tb(III) emission recently[6-8]. Herein, we report the synthesis and crystal structure of 1,4-*bis*{[(2'-thenylaminoformyl)-phenoxyl]methyl}benzene.

EXPERIMENTAL

Phenylamine was obtained from Alfa Aesar Co. Other commercially available chemicals were of analytical grade and were used without further purification. X-ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector. Melting points were determined on a Kofler apparatus and the thermometer was uncorrected.

Synthesis of the title compound: The synthetic route for the title compound is shown in **Scheme-I**. 2-Thenylsalicylamide was prepared according to the literature procedure with minor modifications[9] and 1,4-*bis*-bromomethyl-benzene were prepared according to the literature[10].



Scheme-I: Synthetic route of the title compound

To a solution of 2-thenylsalicylamide (2.33g, 10.5 mmol) in dry acetone was added 1.52 g (11 mmol) dried K_2CO_3 and the mixture was stirred for 0.5 h at room temperature, then 1.32 g (5 mmol) 1,4-*bis*-bromomethyl-benzene in 20 mL of dry acetone was added dropwise in 0.5 h and the resulting solution stirred and heated to reflux for 24 h. After cooling down, inorganic salts were separated by filtration and the solvent removed from the filtrate under reduced pressure. The crude product was recrystallized with ethyl acetate to give a white solid 2.43 g, Yield 86 %, m.p. 175-176 °C.

The title compound (0.057 g, 0. 1 mmol) was dissolved in ethyl acetate (10 mL) and sealed for crystallization at room temperature. After about 2 weeks colourless crystals suitable for analysis were obtained.

X-Ray structure determination: The single crystal of the title compound with the approximate dimensions of 0.15 mm × 0.10 mm × 0.08 mm was placed on a Bruker Smart 1 000 CCD area detector. The reflections were collected using a graphite monochromated MoK_α radition (λ = 0.71073 Å) at 296 (2) K. The structure was solved by using the program SHELXL-97 and Fourier difference techniques and refined by the full-matrix least-squares method on F^2. All non-hydrogen atoms were subjected to anisotropic refinement and all hydrogen atoms were added in idealized positions and refined isotropically. Crystal data and details of the refinement are summarized in Table-1, representative bond lengths (Å) and angles (°) are presented in Table-2. CCDC reference numbers 950108.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT FOR THE TITLE COMPOUND

Empirical formula	$C_{32}H_{28}N_2O_4S_2$
Formula weight	564.91
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	P 2_1/c
Cell dimensions, (Å, deg)	a = 10.0826(17), b = 17.546(3), c = 8.2262(14), α = 90, β = 107.244(3), γ = 90
Volume (Å^3)	1389.9(4)
Z	2
Density (calculated) (mg/m^3)	1.3498
Absorption coefficient (mm^{-1})	0.218
$F_{(000)}$	593.1083
Index ranges	-13 ≤ h ≤ 7, -23 ≤ k ≤ 22, -10 ≤ l ≤ 10
θ (°)	2.11-25.00
Reflections collected/unique	2425/1740
Data/restraints/parameters	2425/1/250
Independent reflections (R_{int})	0.0352
Goodness of fit indicator	1.0726
Final R indices [I > 2σ(I)]	R1 = 0.0405, wR^2 = 0.0988
R indices (all data)	R1 = 0.0601, wR^2 = 0.0988

RESULTS AND DISCUSSION

X-ray crystallographic analysis revealed the crystal structure of the title compound. The structure is shown in Fig. 1. It is worth noting that there are strong intramolecular hydrogen bonding between the amide nitrogen atoms and the etheric oxygen atoms with O2…N1 separations ca. 2.815 Å and N1-H1…O2 angles averaging 134.3° which stabilized the structure to form a *trans* configuration (Fig. 2). Furthermore, the title compound are linked by intermolecular C9-H9…O1 hydrogen bonds into an infinite 1D chain parallel to the *bc* plane, as illustrated in Fig. 3 and hydrogen bonds data are summarized in Table-3.

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (º) FOR THE TITLE COMPOUND

Bond	Lengths	Bond	Lengths	Bond	Lengths
O(2)-C(5)	1.365(2)	C(11)-O(1)	1.228(2)	C(6)-C(7)	1.382(3)
O(2)- C(4)	1.439(2)	C(11)-C(10)	1.498(3)	C(8)-C(7)	1.371(3)
N(1)-C(11)	1.334(2)	C(9)-C(10)	1.395(3)	C(13)-C(12)	1.490(3)
N(1)-C(12)	1.447(3)	C(9)-C(8)	1.375(3)	C(13)-C(14)	1.362(15)
C(5)- C(10)	1.402(2)	C(2)-C(4)	1.498(3)	C(13)-S(1)	1.685(4)
C(5)-C(6)	1.384(3)	C(2)-C(1)	1.368(3)	C(16)-C(15)	1.320(3)
C(1)-C(3)	1.381(3)	C(2)-C(3)	1.365(3)	C(16)-S(1)	1.647(5)
C(15)-C(14)	1.493(17)				

Bond	Angles	Bond	Angles
C(4)-O(2)-C(5)	121.26(15)	C(7)-C(6)-C(5)	120.2(2)
C(12)-N(1)-C(11)	120.64(18)	C(7)-C(8)-C(9)	119.6(2)
C(10)-C(5)-O(2)	116.32(16)	C(8)-C(7)-C(6)	120.3(2)
C(6)-C(5)-O(2)	123.09(17)	C(14)-C(13)-C(12)	128.6(7)
C(6)-C(5)-O(10)	120.58(17)	S(1)-C(13)-C(12)	120.9(2)
O(1)-C(11)-N(1)	120.60(19)	S(1)-C(13)-C(14)	110.4(7)
C(10)-C(11)-N(1)	118.73(16)	S(1)-C(16)-C(15)	119.5(3)
C(10)-C(11)-O(1)	120.66(17)	C(14)-C(15)-C(16)	104.6(6)
C(8)-C(9)-C(10)	122.0(2)	C(13)-C(12)-N(1)	111.38(18)
C(11)-C(10)-C(5)	126.28(16)	C(2)-C(4)-O(2)	105.40(16)
C(9)-C(10)-C(5)	117.36(18)	C(3)-C(1)-C(2)	121.4(2)
C(9)-C(10)-C(11)	116.35(17)	C(1)-C(3)-C(2)	121.0(2)
C(1)-C(2)-C(4)	120.5(2)	C(15)-C(14)-C(13)	114.1(10)
C(3)-C(2)-C(4)	121.9(2)	C(16)-S(1)-C(13)	91.3(2)
C(3)-C(2)-C(1)	117.57(19)		

TABLE-3
DATA FOR HYDROGEN-BONDING INTERACTIONS (Å, º)

D-H···A	d(D-H)	d(H···A)	d(D···A)	∠D-H···A	Symmetry code
N1-H1···O2	0.91(2)	1.91(2)	2.625(2)	134.3(19)	
C9-H9···O1	0.95(2)	2.48(2)	3.301(3)	144.7(16)	2-x, -y,2z

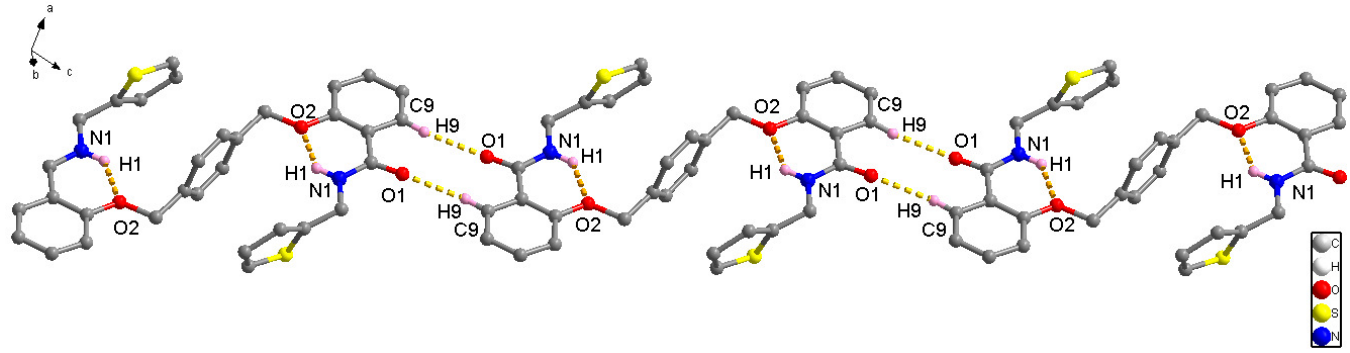


Fig. 3. View of the infinite 1D chain motif of the title compound (hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity)

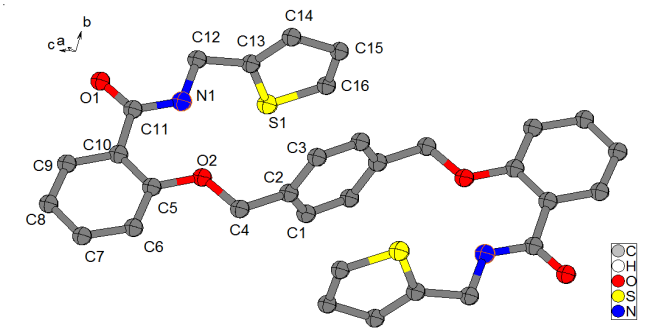


Fig. 1. Molecule structure of the title compound (hydrogen atoms are omitted for clarity)

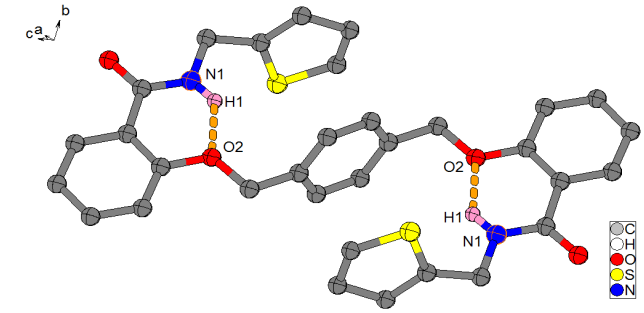


Fig. 2. View of the intramolecular hydrogen-bonding interactions of the title compound (hydrogen atoms, except those forming hydrogen bonds are omitted for clarity)

ACKNOWLEDGEMENTS

This work was supported by the National Natural Science Foundation of China (Grant No. 21161011) and Gansu Natural Science Foundation of China (Grant No. 1212RJZA038).

REFERENCES

1. J. Zhang, Y. Tang, N. Tang, M.Y. Tan, W.S. Liu and K.B. Yu, *J. Chem. Soc. Dalton Trans*., 832 (2002).
2. T. Terai, H. Ito, K. Kikuchi and T. Nagano, *Chem. Eur. J.,* **18**, 7377 (2012).
3. Y.W. Wang, Y.L. Zhang, W. Dou, A.J. Zhang, W.W. Qin and W.S. Liu, *J. Chem. Soc. Dalton Trans*., **39**, 9013 (2010).
4. P.K. Mohapatra, M. Iqbal, D.R. Raut, W. Verboom, J. Huskens and S.V. Godbole, *J. Chem. Soc. Dalton Trans*., **41**, 360 (2012).
5. S.V. Eliseeva and J.C.G. Bünzli, *Chem. Soc. Rev*., **39**, 189 (2010).
6. X.Q. Song, X.Y. Zhou, W.S. Liu, W. Dou, J.X. Ma, X.L. Tang and J.R. Zheng, *Inorg. Chem*., **47**, 11501 (2008).
7. X.Q. Song, X.G. Wen, W.S. Liu and D.Q. Wang, *J. Solid State Chem*., **183**, 1 (2010).
8. X.Q. Song, L. Wang, M.M. Zhao, G,Q. Cheng, X.R. Wang and Y.Q. Peng, *Inorg. Chim. Acta*, **402**, 156 (2013).
9. K. Michio, *Bull. Chem. Soc. Jpn,* **49**, 2679 (1976).
10. Z. Jiri, P. Magdalena, H. Petr and T. Milos, *Synthesis*, 1132 (1994).