Asian Journal of Chemistry; Vol. 25, No. 14 (2013), 8229-8230

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.14673

NOTE

Study on Novel Structure of Potassium Piperazine Complex: $C_4H_{10}N_2K_3O_2$

HAI-XING LIU[1,*], JING-ZHONG XIAO[2], HUAN-MEI GUO[1], KAI-QI YE[3], GUANG ZENG[3] and XI-SHI TAI[1]

[1]Chemistry and Chemical and Environmental Engineering College, Weifang University, Weifang 261061, P.R. China
[2]CEMDRX, Department of Physics, University of Coimbra, Coimbra 3004-516, Portugal
[3]State Key Laboratory of Inorganic Synthesis and Preparative Chemistry, College of Chemistry, Jilin University, Changchun 130012, P.R. China

*Corresponding author: E-mail: haixingliu@tom.com

(*Received*: 12 October 2012; *Accepted*: 12 August 2013) AJC-13917

A novel potassium piperazine complex $C_4H_{10}N_2O_2K_3$ has been synthesized from a hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. The K atom is coordinated by three, four, five atoms, respectively. The crystal is stabilized by N-H···O and N-H···N hydrogen bonds interaction.

Key Words: Potassium piperazine complex, Structure analysis.

The polymers containing ligand groups can bind metal ions forming polymer-metal complexes[1]. The metal-complexes, which have a free coordination site or a weak ligand, can also coordinate with the polymers. The ionic complexes can also form ionic compounds with the polymer acting as counter ion. The interaction of the piperazine ring and its derivatives with metal ions have been extensively studied in solid state[2-5]. In these compounds the piperazine group takes the chair conformation which is the most stable. *N,N'*-disubstituted piperazines have found application as ligands in metal complexes and form the basis of various natural products that exhibit favourable pharmacological properties. Piperazine is conveniently substituted at the *N* and *N'* positions *via* reductive amination and nucleophilic substitution reactions[6]. Piperazine and substituted piperazine derivatives have been found to possess diverse biological activities like antimicrobial, anti-plasmodial, anti-inflammatory, antioxidant and antipsychotic activity[7-9]. In this paper, the novel potassium piperazine complex is reported.

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of Ho_2O_3 (0.01 mmol, 0.004 g), dilute HNO_3, KCl (0.1 mmol, 0.008 g), piperazine (0.1 mmol, 0.016 g) and boric acid (0.1 mmol, 0.009 g) were added into 20 mL water with 10 % (v/v) ethanol and heated for 5 h at 413 K. The solution was obtained by filtration after cooling the reaction to room temperature. Colourless block single crystals suitable for X-ray measurements were obtained after a few weeks.

The crystal structure of potassium piperazine complex (Fig. 1) is built up of K atom, O atom and piperazine. The crystal data and structure refinement is shown in Table-1. The K1 atom is coordinated by three O atoms (two O1, one O2). The K2 atom is coordinated by two O1 atoms and two N1 atoms from piperazine. The K3 atom is coordinated by two O1 atoms, two N1 atoms and one O2 atom. The four atoms (K1, O1, K3, O2) are in the same plane. The distance between two parallel planes is 6.429 Å. The distances d(K-O) are in the range of 3.04-3.43 Å. The distances d(K-N) are in the range of 3.192-3.397 Å. The O1 atom, O2 atom, K1 atom and K3 atom are located in the equatorial plane. The bond angles of O1-K2-O1, N1-K3-N1 are 80.27 and 89.82º, respectively. Selected bond lengths and bond angles are shown in Table-2.

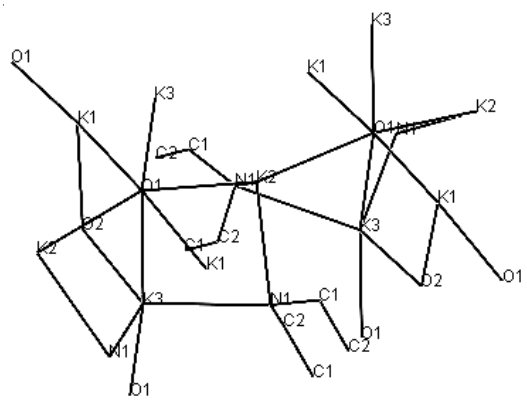

Fig. 1. Molecular structure of the title complex

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT FOR THE POTASSIUM PIPERAZINE COMPLEX

Empirical formula	$C_4H_{10}N_2O_2K_3$
Formula weight	235.44
Temperature	273(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbcm
Unit cell dimensions	a = 6.5081(5) Å α = 90° b = 12.8580(8) Å β = 90° c = 12.8580(9) Å γ = 90°
Volume	1075.97(13) $Å^3$
Z, Calculated density	4, 1.453 Mg/m^3
Absorption coefficient	1.229 mm^{-1}
F(000)	484
Crystal size	0.45 × 0.44 × 0.41 mm
Theta range for data collection	3.13 to 24.99°
Limiting indices	-7 <= h <= 7, -14 <= k <= 15, -15 <= l <= 15
Reflections collected/unique	10484/998 [R(int) = 0.0354]
Completeness to θ = 26	100 %
Absorption correction	Semi-empirical from equivalents
Max and min transmission	0.6327 and 0.6077
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	998/0/58
Goodness-of-fit on F^2	1.192
Final R indices [I > 2σ (I)]	R1 = 0.0518, wR2 = 0.1839
R indices (all data)	R1 = 0.0550, wR2 = 0.1873
Extinction coefficient	0.010(3)
Largest diff. peak and hole	0.874 and -0.718 e.$Å^{-3}$

TABLE-2
SELECT BOND LENGTHS [Å] AND ANGLES [°] FOR THE POTASSIUM PIPERAZINE COMPLEX

K(1)-O(2)	3.070(4)	O(2)-K(3)-N(1)	131.00(7)
K(1)-O(1)	3.201(5)	N(1)-K(3)-O(1)	82.66(9)
K(2)-N(1)	3.192(3)	O(2)-K(3)-K(2)	85.65(6)
K(2)-O(1)	3.2931(12)	N(1)-K(3)-K(2)	48.57(6)
K(3)-O(2)	3.040(4)	O(1)-K(3)-K(2)	50.28(2)
K(3)-N(1)	3.340(3)	O(2)-K(3)-K(1)	37.41(8)
K(3)-O(1)	3.354(6)	N(1)-K(3)-K(1)	111.05(6)
N(1)-C(2)	1.484(5)	O(1)-K(3)-K(1)	40.87(9)
N(1)-C(1)	1.488(5)	K(2)-K(3)-K(1)	63.44(2)
O(2)-H(2)	0.8500	C(2)-N(1)-C(1)	110.7(3)
C(1)-H(1A)	0.9700	C(2)-N(1)-K(2)	99.0(2)
O(2)-K(1)-O(1)	80.27(12)	C(1)-N(1)-K(2)	97.8(2)
O(2)-K(1)-K(2)	75.11(6)	C(2)-N(1)-K(3)	87.9(2)
O(1)-K(1)-K(2)	43.14(2)	C(1)-N(1)-K(3)	161.5(2)
N(1)-K(2)-O(1)	85.93(11)	K(2)-N(1)-K(3)	79.76(7)
N(1)-K(2)-K(3)	51.67(6)	K(1)-O(1)-K(2)	95.20(10)
O(1)-K(2)-K(3)	51.58(9)	K(2)-O(1)-K(3)	78.13(9)
N(1)-K(2)-K(1)	115.80(6)	K(1)-O(2)-H(2)	110.6
O(1)-K(2)-K(1)	41.66(9)	N(1)-C(1)-H(1A)	109.3
K(3)-K(2)-K(1)	65.18(2)	H(1A)-C(1)-H(1B)	108.0

Crystal is stabilized by N-H···O and N-H···N hydrogen bonds interaction

ACKNOWLEDGEMENTS

This study were supported by the Natural Science Foundation of Shandong Province (No. ZR2010BL025), State Key Laboratory of Inorganic Synthesis and Preparative Chemistry (Jilin University) (No. 2011-13), MOE Key Laboratory of Analytical Chemistry for Life Science (Nanjing University) (No. KLACLS1002) and the National Science Foundation of China (No. 21171132) and Open Project of State Key Laboratory of Supramolecular Structure and Materials (No. sklssm201323) (Jilin University).

REFERENCES

1. B.L. Rivas and G.Y. Seguel, *J. Appl. Po1ym. Sci.*, **85**, 2546 (2002).
2. D.A. Cleinente, A. Marrotto, G.C. Valle and C.J. Visona, *Polyhedron*, **18**, 2749 (1999).
3. A. Marrotto, D.A. Clemente, F. Benetollo and G.C. Valle, *Polyhedron*, **20**, 171 (2001).
4. A. Ciccarese, D.A. Clemente, F.P. Fanizzi, A. Marrotto and G. Valle, *Inorg. Chim. Acta*, **419**, 275 (1998).
5. G.V. Seguel and B.L. Rivas, *Polym Bull.*, **51**, 39 (2003).
6. J.P. Safko and R.D. Pike, *J. Chem. Crystallogr.*, **42**, 981(2012).
7. S.G. Shingade and S.B. Bari, *Med. Chem. Res.*, 22, 699 (2012).
8. E. Yoo, J. Yoon, A.N. Pae, H. Rhim, W. Park, J.Y. Kong and H.P. Choo, *Biol. Med. Chem.*, **18**, 1665 (2010).
9. S.-R. Chen, A.-J. Li and M.-M. Chen, *Asian J. Chem.*, **24**, 1680 (2012).