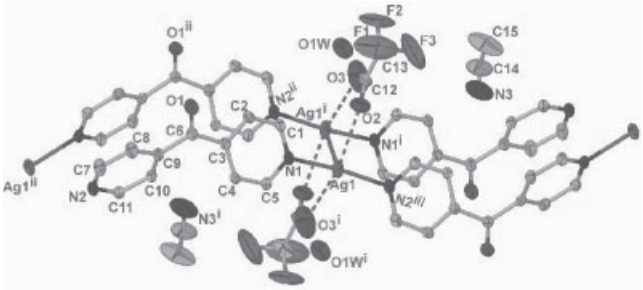
Crystal structure of poly[μ_2-di-4-pyridinylmethanone-κ^2*N:N'*-silver(I)] trifluoroacetate-water-acetonitrile (2/1/1), $C_{14}H_{10.5}AgF_3N_{2.50}O_{3.50}$

Xin-Zhan Sun, Hao-Jie Yan and Chong-Qing Wan*

Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China

Received April 06, 2013, accepted June 18, 2013, available online August 19, 2013, CCDC no. 1267/4013



Abstract

$C_{14}H_{10.5}AgF_3N_{2.50}O_{3.50}$, monoclinic, *C*2/*c* (no. 15), a = 16.155(2) Å, b = 10.670(2) Å, c = 18.098(3) Å, β = 95.251(3)°, V = 3106.3 Å^3, Z = 8, $R_{gt}(F)$ = 0.0466, $wR_{ref}(F^2)$ = 0.1429, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.30×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.51 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD area detector, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8214, 2744
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1666
$N(param)_{refined}$:	255
Programs:	SHELX [8]

Source of material

All reagents and solvents from commercial sources were used without further purification. Di-4-pyridinylmethanone was obtained via the reaction of 4-lithiopyridine and ethyl isonicotinate in a procedure according to the literature [1]. $AgCF_3CO_2$ (22 mg, 0.1 mmol) and di-4-pyridinylmethanone (19 mg, 0.1 mmol) were dissolved in a mixed solvent of 1 ml methanol and 3 ml acetonitrile with stirring at room temperature. The clear solution was filtrated and left stand in air. Colourless crystals of the title complex (yield 23.1 mg, 53% based on Ag) were deposited after 2 weeks.

Discussion

The coordination chemistry of di-pyridyl ketone has been well explored in the past decades [2]. Di-2-pyridinylmethanone (di-2-pyridyl ketone), $(2\text{-}C_5H_4N)_2CO$, is the typical member of this family, which has attracted much interest as it is able to have various conformations to stabilize metal complexes, including its neat ketone form, singly and doubly deprotonated *gem*-diol forms [3]. The metal complexes of the positional isomers, such as 2-pyridinyl-3-pyridinylmethanone [4], as well as di-3-pyridinylmethanone [5] have also been reported. Excepting sporadic reports, the coordination chemistry of the di-4-pyridinylmethanone remains largely un-explored [6]. Herein, we report one new silver(I) complex with di-4-pyridinylmethanone. In the dinuclear title complex, each of the two silver(I) centers are surrounded by two N atoms from two different di-4-pyridinylmethanone ligands with Ag–N distances 2.199(4) Å and of 2.202(5) Å (Figure). Furthermore, each Ag1 ion is weakly connected to O2 and O3^i atoms from two symmetry related trifluoroacetates with Ag···O distances of 2.612(3) Å and 2.836(3) Å, respectively. Ag1 links to one symmetry related Ag atom through argentophilic interaction [7] (Ag1···Ag1^i = 3.205(3) Å) to form the afore mentioned dinuclear complex. Considering the weak Ag···O($CF_3CO_2^-$) contact, the trifluoroacetate can be viewed as a *syn*-η^1:η^1:μ_2 ligation mode to bridge and stabilize the dinuclear Ag atoms with an inversion center between them. The dinuclear Ag(I) and a pair of the μ_2-bridging di-4-pyridinylmethanone ligands are alternately arranged and link together, forming a double-bridged undulant chain structure along the [101] direction. The formed chains are stacked along the [-101] direction and interconnect through $\pi\cdots\pi$ interactions (Cg1···Cg1^i = 3.718(4) Å) to form a three-dimensional framework with the lattice water and acetonitrile molecules embedded among the interstices (Cg1 represents N1-C1-C2-C3-C4-C5 ring).

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	8*f*		0.6331	0.5069	0.4610	0.073
H(2A)	8*f*		0.5384	0.6002	0.3781	0.070
H(4A)	8*f*		0.4005	0.2907	0.3779	0.071
H(5A)	8*f*		0.4984	0.2053	0.4615	0.073
H(7A)	8*f*		0.1842	0.4887	0.1395	0.098
H(8A)	8*f*		0.2623	0.5892	0.2321	0.090
H(10A)	8*f*		0.4102	0.2890	0.2517	0.064
H(11A)	8*f*		0.3286	0.1977	0.1568	0.071
H(15A)	8*f*	0.5	0.5256	0.8245	0.2309	0.260
H(15B)	8*f*	0.5	0.5002	0.8488	0.3112	0.260
H(15C)	8*f*	0.5	0.4323	0.8488	0.2436	0.260

* Correspondence author (e-mail: wancq@cnu.edu.cn)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	8*f*		0.67130(3)	0.25951(6)	0.54759(3)	0.0783(4)	0.1144(6)	0.0669(4)	0.0174(3)	–0.0235(3)	0.0078(3)
C(1)	8*f*		0.5857(4)	0.4623(6)	0.4438(3)	0.061(4)	0.062(4)	0.058(3)	–0.003(3)	–0.007(3)	–0.016(3)
C(2)	8*f*		0.5293(4)	0.5188(5)	0.3937(3)	0.069(4)	0.050(3)	0.055(3)	–0.006(3)	–0.003(3)	–0.009(3)
C(3)	8*f*		0.4590(3)	0.4551(5)	0.3663(3)	0.060(3)	0.050(3)	0.046(3)	0.004(3)	–0.001(3)	–0.007(3)
C(4)	8*f*		0.4479(3)	0.3363(6)	0.3936(3)	0.061(4)	0.062(4)	0.052(3)	–0.011(3)	–0.011(3)	–0.003(3)
C(5)	8*f*		0.5069(4)	0.2858(6)	0.4440(3)	0.069(4)	0.061(4)	0.050(3)	–0.006(3)	–0.005(3)	0.002(3)
N(1)	8*f*		0.5761(3)	0.3464(5)	0.4692(2)	0.059(3)	0.066(3)	0.053(3)	0.006(2)	–0.008(2)	–0.007(2)
C(6)	8*f*		0.3985(4)	0.5202(6)	0.3118(3)	0.065(4)	0.056(4)	0.056(3)	0.006(3)	–0.007(3)	–0.004(3)
O(1)	8*f*		0.3949(3)	0.6344(4)	0.3121(3)	0.124(4)	0.054(3)	0.101(4)	0.013(3)	–0.036(3)	–0.007(3)
C(7)	8*f*		0.2306(4)	0.4479(7)	0.1623(4)	0.072(4)	0.092(5)	0.076(4)	0.021(4)	–0.024(4)	–0.004(4)
C(8)	8*f*		0.2767(4)	0.5088(6)	0.2181(4)	0.077(4)	0.072(4)	0.071(4)	0.025(3)	–0.013(3)	–0.009(3)
C(9)	8*f*		0.3454(3)	0.4491(5)	0.2535(3)	0.056(3)	0.058(4)	0.049(3)	0.004(3)	–0.004(3)	–0.002(3)
C(10)	8*f*		0.3643(3)	0.3317(5)	0.2296(3)	0.056(3)	0.058(4)	0.045(3)	0.003(3)	–0.004(2)	0.003(3)
C(11)	8*f*		0.3147(4)	0.2774(5)	0.1725(3)	0.069(4)	0.056(4)	0.051(3)	–0.002(3)	–0.003(3)	0.001(3)
N(2)	8*f*		0.2480(3)	0.3333(5)	0.1385(3)	0.063(3)	0.079(4)	0.060(3)	–0.005(3)	–0.013(2)	0.002(3)
C(12)	8*f*		0.8039(7)	0.5218(8)	0.5587(6)	0.150(9)	0.063(5)	0.138(8)	–0.043(6)	–0.081(7)	0.025(5)
C(13)	8*f*		0.8234(8)	0.647(1)	0.5877(7)	0.13(1)	0.16(1)	0.15(1)	–0.031(9)	0.006(8)	0.002(9)
O(2)	8*f*		0.7397(6)	0.4791(9)	0.5730(6)	0.194(8)	0.175(8)	0.27(1)	–0.112(7)	–0.131(8)	0.114(8)
O(3)	8*f*		0.8523(8)	0.478(1)	0.5218(6)	0.31(2)	0.177(9)	0.21(1)	0.10(1)	–0.07(1)	–0.094(8)
F(3)	8*f*	0.609	0.875(1)	0.718(1)	0.5631(9)	0.17(1)	0.112(9)	0.20(2)	–0.090(9)	0.05(1)	–0.020(9)
F(2)	8*f*	0.609	0.844(1)	0.635(2)	0.6615(7)	0.22(2)	0.29(2)	0.14(1)	–0.07(1)	–0.00(1)	–0.05(1)
F(1)	8*f*	0.609	0.751(1)	0.710(2)	0.582(1)	0.19(1)	0.21(1)	0.29(2)	0.01(1)	0.06(1)	–0.04(2)
F(3')	8*f*	0.391	0.828(2)	0.719(3)	0.527(2)	0.21(2)	0.15(2)	0.27(2)	–0.07(2)	0.04(2)	0.04(2)
F(2')	8*f*	0.391	0.907(1)	0.652(3)	0.601(2)	0.18(2)	0.20(2)	0.19(2)	–0.12(1)	0.00(1)	–0.01(2)
F(1')	8*f*	0.391	0.781(2)	0.705(3)	0.633(2)	0.20(2)	0.20(2)	0.20(2)	–0.02(2)	0.06(2)	–0.08(2)
N(3)	4*e*		$\frac{1}{2}$	1.096(1)	$\frac{1}{4}$	0.23(2)	0.063(8)	0.38(3)	0	–0.07(2)	0
C(14)	4*e*		$\frac{1}{2}$	0.997(1)	$\frac{1}{4}$	0.16(2)	0.063(9)	0.32(3)	0	–0.01(2)	0
C(15)	8*f*	0.5	0.488(2)	0.870(2)	0.261(1)	0.34(3)	0.28(2)	0.37(3)	0.00(2)	–0.02(2)	0.01(2)
O(1W)	8*f*	0.5	0.954(1)	0.472(2)	0.4420(9)	0.17(1)	0.16(1)	0.18(2)	–0.01(1)	–0.03(1)	–0.00(1)

Acknowledgments. This work was supported in part by the Science and Technology program, Beijing Municipal Education Commission.

References

1. Braverman, M. A.; Nettleman, J. H.; Supkomski, R. M.; LaDuca, R. L.: Copper Phthalate Coordination Polymers Incorporating Kinked Dipyridyl Ligands: An Unprecedented 8 Connected Network and One-Dimensional Chiral Nanobarrels with Hydrophobic Channels Constructed from Septuple Helical Motifs. Inorg. Chem. **48** (2009) 4918-4926.
2. Papaefstathiou, G. S.; Perlepes, S. P.: Families of Polynuclear Manganese, Cobalt, Nickel and Copper Complexes Stabilized by Various Forms of Di-2-pyridyl Ketone. Comments Inorg. Chem. **23** (2002) 249-321.
3. Abrahams, B. F.; Hudson, T. A.; Robson, R.: In Situ Synthesis of Tri-substituted Methanol Ligands and Their Potential as One-Pot Generators of Cubane-like Metal Complexes. Chem. Eur. J. **12** (2006) 7095-7102.
4. Chen, X.-D.; Du, M.; Mak, T. C. W.: Controlled generation of heterochiral or homochiral coordination polymer: helical conformational polymorphs and argentophilicity-induced spontaneous resolution. Chem. Commun. (2005) 4417-4419.
5. Chen, X.-D.; Mak, T. C. W.: Helical and zigzag coordination polymeric chains derived from di-3-pyridyl ketone and silver(I) salts. J. Mol. Struct. **743** (2005) 1-6.
6. Farnum, G. A.; Knapp, W. R.; LaDuca, R. L.: Silver Nitrate Molecular Species and Coordination Polymers with Divergent Supramolecular Morphology Constructed from Hydrogen-bonding Capable Dipyridyl Ligands. Polyhedron **28** (2009) 291-299.
7. Bondi, A.: van der Waals Volumes and Radii. J. Phys. Chem. **68** (1964) 441-456.
8. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.