Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



## Electronic Supplementary Information (ESI) for CrystEngComm

# A novel arenedisulfonate-templated 1D silver ladder constructed from 4-aminobenzonitrile ligand

**Di Sun,‡ Fu-Jing Liu,‡ Hong-Jun Hao, Yun-Hua Li, Na Zhang, Rong-Bin Huang,* and Lan-Sun Zheng**

*Department of chemistry, Xiamen University, Xiamen 361005, People's Republic of China and State key Laboratory for Physical Chemistry of Solid Surfaces, Xiamen University, Xiamen 361005, People's Republic of China*

‡*These authors contributed equally to this work*

## *Content*



*Correspondence e-mail: rbhuang@xmu.edu.cn. Fax: 86-592-2183074

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



## (1) Experiment details

Materials and General Methods.

All chemicals and solvents used in the syntheses were of analytical grade and used without further purification. IR spectra were measured on a Nicolet Avatar 330 FTIR Spectrometer at the range of 4000-400 $cm^{-1}$. Elemental analyses were carried out on a CE instruments EA 1110 elemental analyzer. Photoluminescence spectra were measured on a Hitachi F-7000 Fluorescence Spectrophotometer (slit width: 5 nm; sensitivity: high). X-ray powder diffractions were measured on a Panalytical X-Pert pro diffractometer with Cu-Kα radiation. TG curves were measured from 30 to 600 $^{o}C$ on a SDT Q600 instrument at a heating rate 10 $^{o}C$/min under the $N_2$ atmosphere (100 ml/min).

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



## (2) Synthesis of 1-5

**Synthesis of [nds ⊂ $Ag_2(abn)_4]_n$. (1)**

Reaction of $Ag_2O$ (12 mg, 0.05 mmol), $H_2nds·4H_2O$ (36 mg, 0.1 mmol) and abn (12 mg, 0.1 mmol) in methanol-ethanol mixed solvent (5 mL, *v*/*v*: 3/2), then aqueous $NH_3$ solution (25%, 3 mL) was dropped into the mixture to give a clear solution under ultrasonic treatment. The resultant colorless solution was allowed slowly to evaporate at room temperature for two week to give colorless plate crystals of **1**. The crystals were isolated by filtration and washed by ethanol and dried in air. Yield: *Ca*. 57% based on Ag. Elemental analysis: Anal. Calc. for $AgC_{19}H_{15}N_4O_3S$: C 46.83, H 3.10, N 11.50%. Found: C 44.71, H 3.20, N 10.75%. Selected IR peaks ($cm^{-1}$): 3477 (s), 3371 (s), 3213 (m), 2214 (m), 1628 (s), 1603 (s), 1515 (m), 1320(w), 1235 (w), 1205 (s), 1176 (m), 1161 (m), 1045 (m), 830 (w), 792 (w), 609 (m), 546 (m).

**Synthesis of $[Ag(abn)_2·NO_3]_n$. (2):**

Reaction of $AgNO_3$ (17 mg, 0.1 mmol) and abn (12 mg, 0.1 mmol) in methanol-water mixed solvent (6 mL, *v*/*v*: 4/2), then aqueous $NH_3$ solution (25%, 1 mL) was dropped into the mixture to give a clear solution under ultrasonic treatment. The resultant colorless solution was allowed slowly to evaporate at room temperature for two week to give colorless plate crystals of **2**. The crystals were isolated by filtration and washed by ethanol and dried in air. Elemental analysis: Anal. Calc. for $AgC_{14}H_{12}N_5O_3$: C 41.40, H 2.98, N 17.24%. Found: C 40.99, H 2.84, N 17.37%. Selected IR peaks ($cm^{-1}$): 3477 (s), 3372 (s), 3213 (m), 2214 (s), 1628 (s), 1602 (s), 1515 (s), 1384(s), 1320 (m), 1176 (m), 834 (m), 830 (m), 698 (w), 547 (m).

**Synthesis of $[Ag(abn)_2·ClO_4]_n$. (3):**

Reaction of $Ag_2O$ (23 mg, 0.1 mmol), $NaClO_4$ (25 mg, 0.2 mmol) and abn (24 mg, 0.2 mmol) in methanol-ethanol-water mixed solvent (7 mL, *v*/*v*/*v*: 3/3/1), then aqueous $NH_3$ solution (25%, 0.5 mL) was dropped into the mixture to give a clear solution under ultrasonic treatment. The resultant colorless solution was allowed slowly to evaporate at room temperature for two week to give colorless plate crystals of **3**. The crystals were isolated by filtration and washed by ethanol and dried in air. Elemental analysis: Anal. Calc. for $AgC_{14}H_{12}ClN_4O_4$: C 37.91, H 2.73, N 12.63%. Found: C 37.62, H 2.86, N 12.67%. Selected IR peaks ($cm^{-1}$): 3477 (s), 3372 (s), 2214 (m), 1628 (s), 1603 (m), 1515 (m), 1320 (w), 1177 (m), 1143 (m), 1109 (m), 1189 (m), 839 (m), 830 (m), 697 (w), 626 (m), 546 (m).

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



**Synthesis of $[Ag(abn)_2 \cdot PF_6]_n$. (4):**

Reaction of $Ag_2O$ (23 mg, 0.1 mmol), $KPF_6$ (37 mg, 0.2 mmol) and abn (24 mg, 0.2 mmol) in methanol-ethanol mixed solvent (5 mL, *v*/*v*: 3/2), then aqueous $NH_3$ solution (25%, 1 mL) was dropped into the mixture to give a clear solution under ultrasonic treatment. The resultant colorless solution was allowed slowly to evaporate at room temperature for two week to give colorless plate crystals of **4**. The crystals were isolated by filtration and washed by ethanol and dried in air. Elemental analysis: Anal. Calc. for $AgC_{14}H_{12}F_6N_4P$: C 34.38, H 2.47, N 11.46%. Found: C 33.43, H 2.42, N 11.26%. Selected IR peaks ($cm^{-1}$): 3478 (s), 3372 (s), 3213 (w), 2214 (s), 1628 (s), 1603 (s), 1516 (s), 1320 (m), 1176 (s), 839 (s), 697 (w), 561 (m), 546 (m).

**Synthesis of $[Ag(abn)_2 \cdot CF_3COO]_n$. (5):**

Reaction of $AgOOCCF_3$ (22 mg, 0.1 mmol) and abn (24 mg, 0.2 mmol) in methanol-ethanol mixed solvent (5 mL, *v*/*v*: 3/2), then aqueous $NH_3$ solution (25%, 1 mL) was dropped into the mixture to give a clear solution under ultrasonic treatment. The resultant colorless solution was allowed slowly to evaporate at room temperature for two week to give colorless plate crystals of **5**. The crystals were isolated by filtration and washed by ethanol and dried in air. Elemental analysis: Anal. Calc. for $AgC_{16}H_{12}F_3N_4O_2$: C 42.04, H 2.65, N 12.26%. Found: C 41.05, H 2.44, N 11.82%. Selected IR peaks ($cm^{-1}$): 3477 (s), 3372 (s), 2214 (m), 1686 (m), 1628 (s), 1603 (s), 1515 (m), 1320 (w), 1208 (m), 1176 (m), 1137 (m), 833 (m), 830 (m), 802 (w), 724 (w), 697 (w), 547 (m).

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



## (3) X-ray Crystallography

Single crystals of the complexes **1-5** with appropriate dimensions were chosen under an optical microscope and mounted on a glass fiber for data collection. Data were collected on a Rigaku R–AXIS RAPID Image Plate single–crystal diffractometer with graphite–monochromated Mo Kα radiation source (λ = 0.71073 Å) operating at 50 kV and 90 mA in $\omega$ scan mode for **1**, **3** and **5**, and a Bruker-AXS CCD diffractometer equipped with a graphite-monochromated Mo Kα radiation source (λ = 0.71073 Å) operating at 50 kV and 30 mA in $\omega$ scan mode for **2** and **4**. In all cases, the highest possible space group was chosen. All structures were solved by direct methods using SHELXS–97[1] and refined on $F^2$ by full-matrix least–squares procedures with SHELXL–97.[2] All structures were examined using the Addsym subroutine of PLATON[3] to assure that no additional symmetry could be applied to the models.

(1) G. M. Sheldrick, *SHELXS-97, Program for X-ray Crystal Structure Determination*, University of Göttingen, Germany, 1997.

(2) G. M. Sheldrick, *SHELXL-97, Program for X-ray Crystal Structure Refinement,* University of Göttingen, Germany, 1997.

(3) A. L. Spek, *Implemented as the PLATON Procedure, a Multipurpose Crystallographic Tool,* Utrecht University, Utrecht, The Netherlands, 1998.

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



## (4) Table S1: Crystal data for 1-5

| Compound | **1** | **2** | **3** | **4** | **5** |
|---|---|---|---|---|---|
| Empirical formula | $AgC_{19}H_{15}N_4O_3S$ | $AgC_{14}H_{12}N_5O_3$ | $AgC_{14}H_{12}ClN_4O_4$ | $AgC_{14}H_{12}F_6N_4P_2$ | $AgC_{16}H_{12}F_3N_4O_2$ |
| Formula weight | 487.29 | 406.16 | 443.60 | 489.12 | 457.17 |
| Crystal system | triclinic | monoclinic | monoclinic | monoclinic | triclinic |
| Space group | *P*-1 | *P*2(1)/n | *P*2(1)/c | *P*2(1)/n | *P*-1 |
| *a* (Å) | 9.768(2) | 9.2284(18) | 9.1859(15) | 9.4587(19) | 9.4187(19) |
| *b* (Å) | 10.475(2) | 9.4398(19) | 9.6910(16) | 9.989(2) | 10.098(2) |
| *c* (Å) | 10.640(2) | 17.845(4) | 20.348(3) | 18.523(4) | 10.271(2) |
| $\alpha$ (deg) | 74.74(3) | 90.00 | 90.00 | 90.00 | 111.21(3) |
| $\beta$ (deg) | 80.82(3) | 92.018(4) | 115.309(6) | 90.07(3) | 106.08(3) |
| $\gamma$ (deg) | 69.50(3) | 90.00 | 90.00 | 90.00 | 91.41(3) |
| $V$(Å$^3$) | 981.0(3) | 1553.6(5) | 1637.5(5) | 1750.1(6) | 866.5(3) |
| $T$(K) | 173(2) | 173(2) | 173(2) | 173(2) | 173(2) |
| $Z$, $D_{calcd}$ (Mg/m$^3$) | 2, 1.650 | 4, 1.737 | 4, 1.799 | 4, 1.856 | 2, 1.752 |
| $F$(000) | 488 | 808 | 880 | 960 | 452 |
| $\mu$(mm$^{-1}$) | 1.161 | 1.319 | 1.420 | 1.309 | 1.212 |
| Ref. collected/unique | 8509 / 3835 | 6204 / 2616 | 7992 / 2865 | 9767 / 3078 | 6827 / 3038 |
| $R_{int}$ | 0.0246 | 0.0193 | 0.0225 | 0.0253 | 0.0260 |
| Parameters | 253 | 208 | 217 | 235 | 262 |
| Final $R$ indices[$I > 2\sigma(I)$] | $R_1 = 0.0255$ $wR_2 = 0.0624$ | $R_1 = 0.0379$ $wR_2 = 0.0929$ | $R_1 = 0.0320$ $wR_2 = 0.0769$ | $R_1 = 0.0308$ $wR_2 = 0.0826$ | $R_1 = 0.0339$ $wR_2 = 0.0849$ |
| $R$ indices (all data) | $R_1 = 0.0287$ $wR_2 = 0.0640$ | $R_1 = 0.0414$ $wR_2 = 0.0954$ | $R_1 = 0.0338$ $wR_2 = 0.0779$ | $R_1 = 0.0364$ $wR_2 = 0.0857$ | $R_1 = 0.0385$ $wR_2 = 0.0908$ |
| GOF | 1.047 | 1.143 | 1.159 | 1.079 | 1.115 |
| Max./ min., $\Delta\rho$ (e·Å$^{-3}$) | 0.856/-0.679 | 0.689/-0.360 | 0.690/-0.386 | 0.933/-0.670 | 0.725/-0.705 |

$R_1 = \Sigma| |F_o|-|F_c| | / \Sigma|F_o|$, $wR_2 = [\Sigma w(F_o^2-F_c^2)^2]/ \Sigma w(F_o^2)^2]^{1/2}$

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011



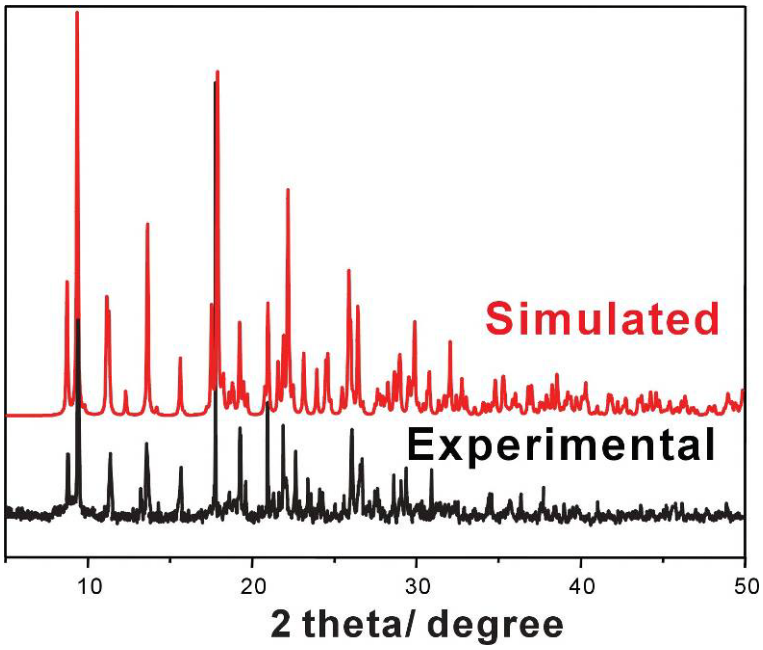
## (5) Table S2: The selected bond distances and angles for 1-5

| | | | |
|---|---|---|---|
| Complex **1** | | | |
| Ag1—N1 | 2.253 (2) | Ag1—N3$^{i}$ | 2.415 (2) |
| Ag1—N4 | 2.263 (2) | Ag1—N2$^{ii}$ | 2.447 (2) |
| N1—Ag1—N4 | 138.70 (8) | N1—Ag1—N2$^{ii}$ | 99.42 (8) |
| N1—Ag1—N3$^{i}$ | 90.64 (8) | N4—Ag1—N2$^{ii}$ | 111.66 (7) |
| N4—Ag1—N3$^{i}$ | 114.26 (7) | N3$^{i}$—Ag1—N2$^{ii}$ | 91.34 (8) |
| Symmetry codes: (i) $x$, $y$+1, $z$; (ii) −$x$, −$y$+1, −$z$. | | | |
| Complex **2** | | | |
| Ag1—N1 | 2.211 (4) | Ag1—N3 | 2.325 (3) |
| Ag1—N2$^{i}$ | 2.319 (3) | Ag1—N4$^{ii}$ | 2.404 (3) |
| N1—Ag1—N2$^{i}$ | 122.71 (12) | N1—Ag1—N4$^{ii}$ | 92.45 (13) |
| N1—Ag1—N3 | 124.07 (13) | N2$^{i}$—Ag1—N4$^{ii}$ | 124.11 (12) |
| N2$^{i}$—Ag1—N3 | 94.72 (12) | N3—Ag1—N4$^{ii}$ | 98.91 (11) |
| Symmetry codes: (i) $x$+1/2, −$y$+1/2, $z$−1/2; (ii) −$x$+1, −$y$+1, −$z$+1. | | | |
| Complex **3** | | | |
| Ag1—N3 | 2.212 (3) | Ag1—N4$^{i}$ | 2.362 (3) |
| Ag1—N1 | 2.326 (3) | Ag1—N2$^{ii}$ | 2.417 (3) |
| N3—Ag1—N1 | 123.95 (11) | N3—Ag1—N2$^{ii}$ | 102.23 (11) |
| N3—Ag1—N4$^{i}$ | 121.02 (11) | N1—Ag1—N2$^{ii}$ | 94.27 (10) |
| N1—Ag1—N4$^{i}$ | 95.54 (10) | N4$^{i}$—Ag1—N2$^{ii}$ | 117.88 (10) |
| Symmetry codes: (i) $x$, −$y$+3/2, $z$−1/2; (ii) −$x$+1, −$y$+1, −$z$+1. | | | |
| Complex **4** | | | |
| Ag1—N1 | 2.257 (3) | Ag1—N2$^{ii}$ | 2.377 (3) |
| Ag1—N3$^{i}$ | 2.320 (3) | Ag1—N4 | 2.440 (3) |
| N1—Ag1—N3$^{i}$ | 124.08 (11) | N1—Ag1—N4 | 95.24 (11) |
| N1—Ag1—N2$^{ii}$ | 125.06 (10) | N3$^{i}$—Ag1—N4 | 95.09 (11) |
| N3$^{i}$—Ag1—N2$^{ii}$ | 96.37 (11) | N2$^{ii}$—Ag1—N4 | 118.53 (10) |
| Symmetry codes: (i) −$x$+1, −$y$+1, −$z$+1; (ii) $x$−1/2, −$y$+3/2, $z$−1/2. | | | |
| Complex **5** | | | |
| Ag1—N3 | 2.201 (3) | Ag1—N2$^{i}$ | 2.436 (3) |
| Ag1—N1 | 2.221 (3) | Ag1—N4$^{ii}$ | 2.496 (3) |
| N3—Ag1—N1 | 134.33 (13) | N3—Ag1—N4$^{ii}$ | 98.64 (12) |
| N3—Ag1—N2$^{i}$ | 121.28 (12) | N1—Ag1—N4$^{ii}$ | 97.52 (14) |
| N1—Ag1—N2$^{i}$ | 96.85 (12) | N2$^{i}$—Ag1—N4$^{ii}$ | 100.93 (12) |
| Symmetry codes: (i) −$x$+2, −$y$+2, −$z$+3; (ii) −$x$+1, −$y$+1, −$z$+1. | | | |

Electronic Supplementary Material (ESI) for CrystEngComm
This journal is © The Royal Society of Chemistry 2011

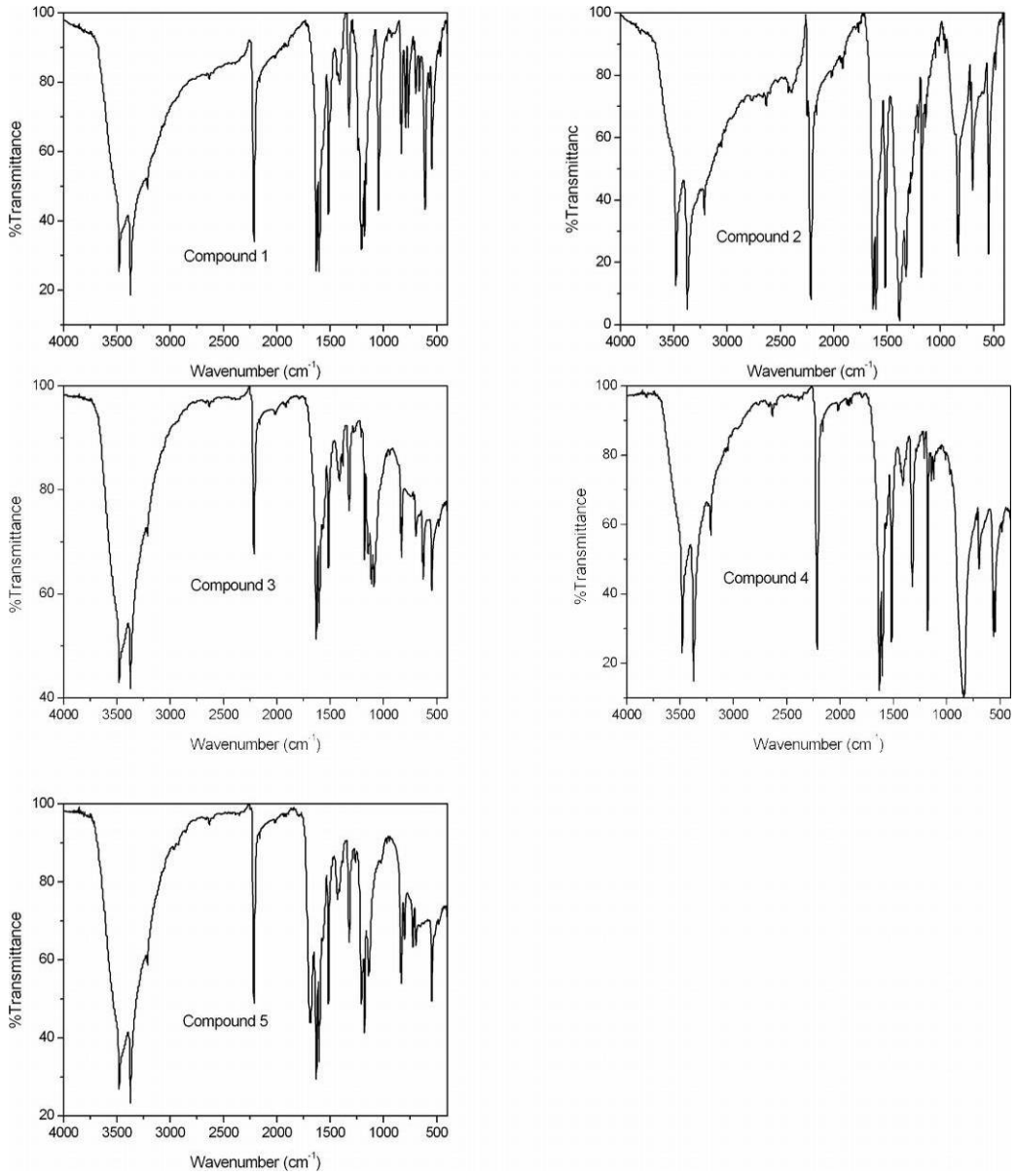


**(6) Figure S1: XRD spectra of 1**



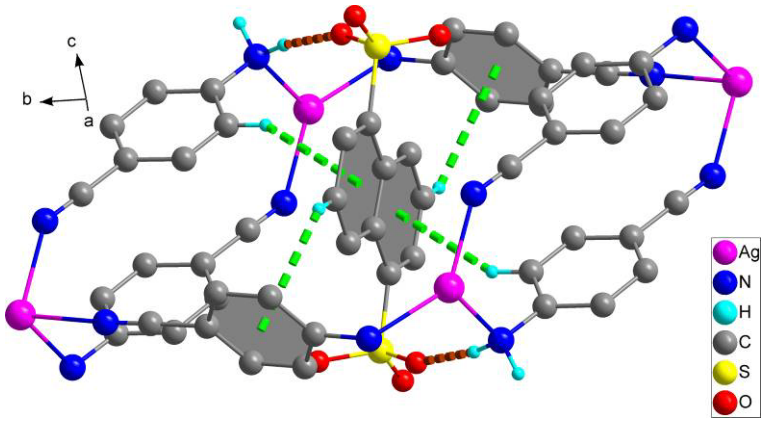
This journal is © The Royal Society of Chemistry 2011

## (7) Figure S2: IR spectra of 1-5



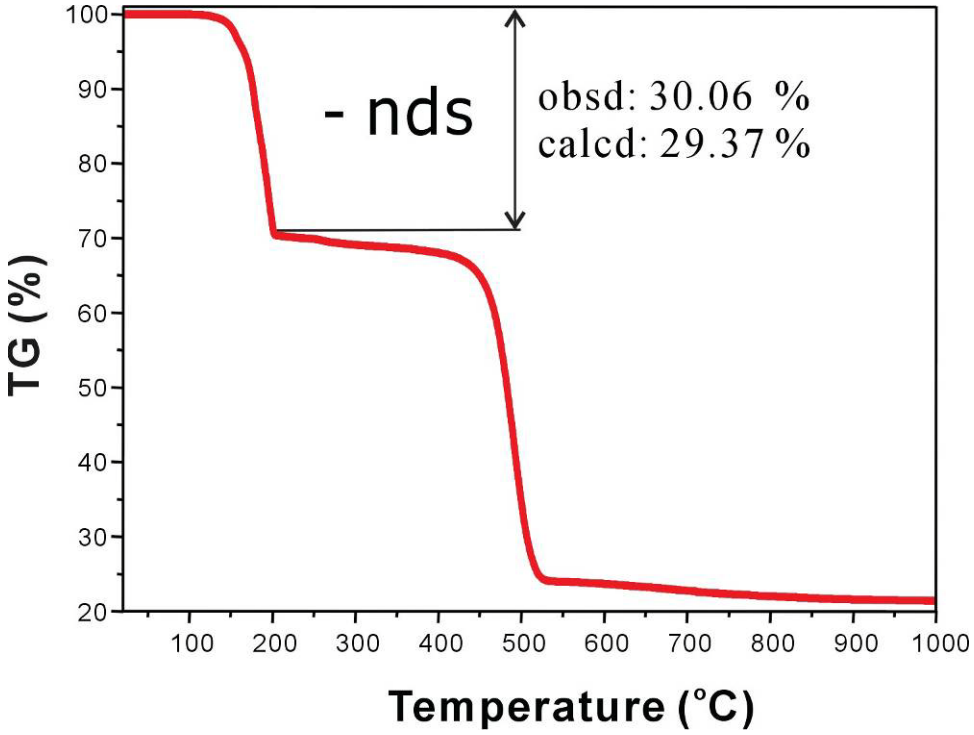
This journal is © The Royal Society of Chemistry 2011

**(8) Figure S3: The noncovalent interaction between nds and the gird.**



This journal is © The Royal Society of Chemistry 2011

**(9) Figure S4: The TG curve of complex 1**



**(10) Figure S5: The photoluminescent properties of complexes 2-5**