Electronic supplementary materials *Mendeleev Commun.*, 2027, **37**, just accepted

Silver wheel incorporating acetone oxalohydrazide dihydrazone: structure, thermal stability and luminescent properties

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Experimental section

Synthesis of $[Ag_2L]_n \cdot 0.167nCH_3CN \cdot 0.5nH_2O$ (**1**). The ligand H_2L was prepared in situ by condensation of oxalyl dihydrazide (0.059 g, 0.5 mmol) with acetone (10 mL) under reflux for 30 min. A solution of $AgNO_3$ (0.170 g, 1.0 mmol) in a 1:1 acetonitrile–water mixture (10 mL) was then added, and the resulting mixture was stirred for 30 min at room temperature. Subsequently, a 25% aqueous ammonia solution (2 mL) was added, and stirring was continued for another 15 min. The solution was filtered, and the filtrate was left for slow evaporation at room temperature. Colourless block-shaped crystals suitable for X-ray diffraction were obtained after 3 days. The crystals were filtered off, washed with a small amount of cold acetonitrile, and dried in air. Yield **1**: 0.127 g (59%, based on Ag. Anal. Calc. for $C_{8.3}H_{13.5}Ag_2N_{4.2}O_{2.5}$: C, 23.40; H, 3.18; N, 13.64. Found: C, 22.95; H, 3.13; N, 13.19. FT-IR (ATR), ν/cm^{-1}: 3224 w (H_2O), 3047 w (C–H), 2854 w (C–H), 2083 w (CH_3CN), 1994 w (CH_3CN), 1917 w (CH_3CN), 1662 m (C=O), 1581 w (C=N), 1504 w (C=N), 1300 s (C–N), 1246 v.s (C–N/N–N), 1107 s (N–N), 1038 m (C–H), 980 m (N–N/C–C), 818 s (δ N–C=O), 779 m (C–H), 644 s (δ N–C=O).

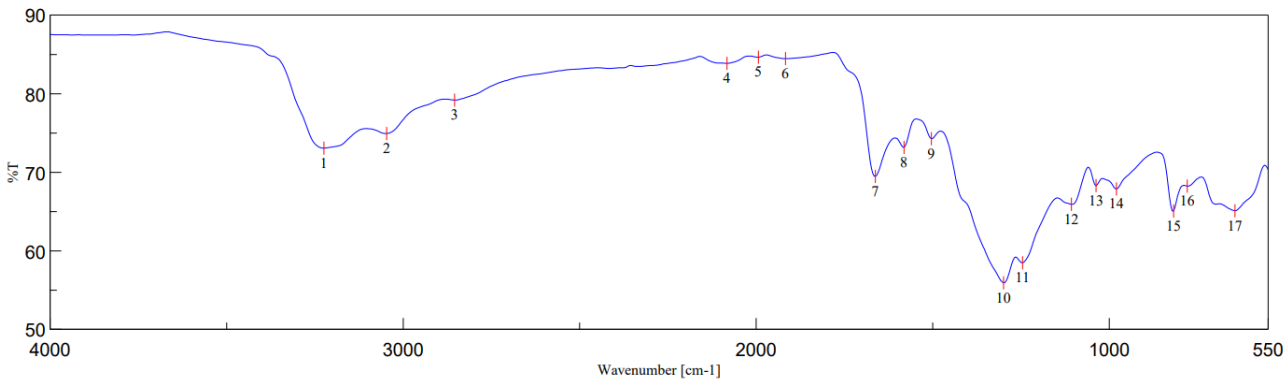
Commercial reagents (silver nitrate (99.85%, Acros Organics), oxalyl dihydrazide (>96%, TCI)) and solvents (acetonitrile (≥99%), acetone (≥99%), aqueous ammonia solution (25%)) were used for the synthesis.

IR spectrum was recorded a JASCO Fourier transform IR spectrophotometer using the attenuated total internal reflection (ATR) method in the frequency range of 550–4000 cm^{-1}. Microprobe analyses were carried out using a Carlo Erba EA 1108 Series CHNS Elemental Analyzer.

Thermal behavior **1** was studied using the simultaneous thermal analysis (STA) technique for parallel recording of thermogravimetry (TG) and differential scanning calorimetry (DSC) curves. The study was performed on an STA 449 F1 Jupiter® instrument («NETZSCH») in Al-crucibles under a lid with a hole to ensure a vapor pressure of 1 atm during the thermal decomposition of the samples. The rate of heating to 500 °C was 10 °C min^{-1} under an argon atmosphere (content Ar > 99.998%, O_2 < 0.0002%, N_2 < 0.001%, water vapor < 0.0003%, CH_4 < 0.0001%). The sample mass was in the range of 3.34 mg. The accuracy of temperature measurements was ±0.7 °C, and that of mass changes was ±1·10^{-2} mg.

Scanning electron microscopy (SEM) images were taken using a Tescan Amber GMH scanning electron microscope. Images were obtained using an Everhart–Thornley SE detector at ×3.000–100.000 magnifications and at an accelerating voltage of 20 kV to show silver nanoparticles covered by the organic matrix. SEM images were processed using SEM_scale_bar software (version 4.2; https://oxide.ru/?q=content/%D0%BF%D0%BE). Energy-dispersive X-ray spectra (EDX) were recorded using an Ultim MAX EDS detector with a 100 mm^2 active area (Oxford Instruments) and at an accelerating voltage of 20 kV using AZtec 5.0 SP1 software.

Luminescent spectrum was measured with a Perkin Elmer LS-55 spectrofluorimeter.



[Result of Peak Picking]

No.	Position	Intensity	No.	Position	Intensity	No.	Position	Intensity
1	3224.4	73.064	2	3046.98	74.9116	3	2854.13	79.1809
4	2082.74	83.8568	5	1994.03	84.6359	6	1916.9	84.4556
7	1662.34	69.4436	8	1581.34	73.1561	9	1504.2	74.2693
10	1299.79	55.9147	11	1245.79	58.4661	12	1106.94	65.9042
13	1037.52	68.2829	14	979.661	67.8429	15	817.67	65.0472
16	779.101	68.2169	17	644.108	65.0852			

Figure S1 FT-IR spectrum of complex **1**.

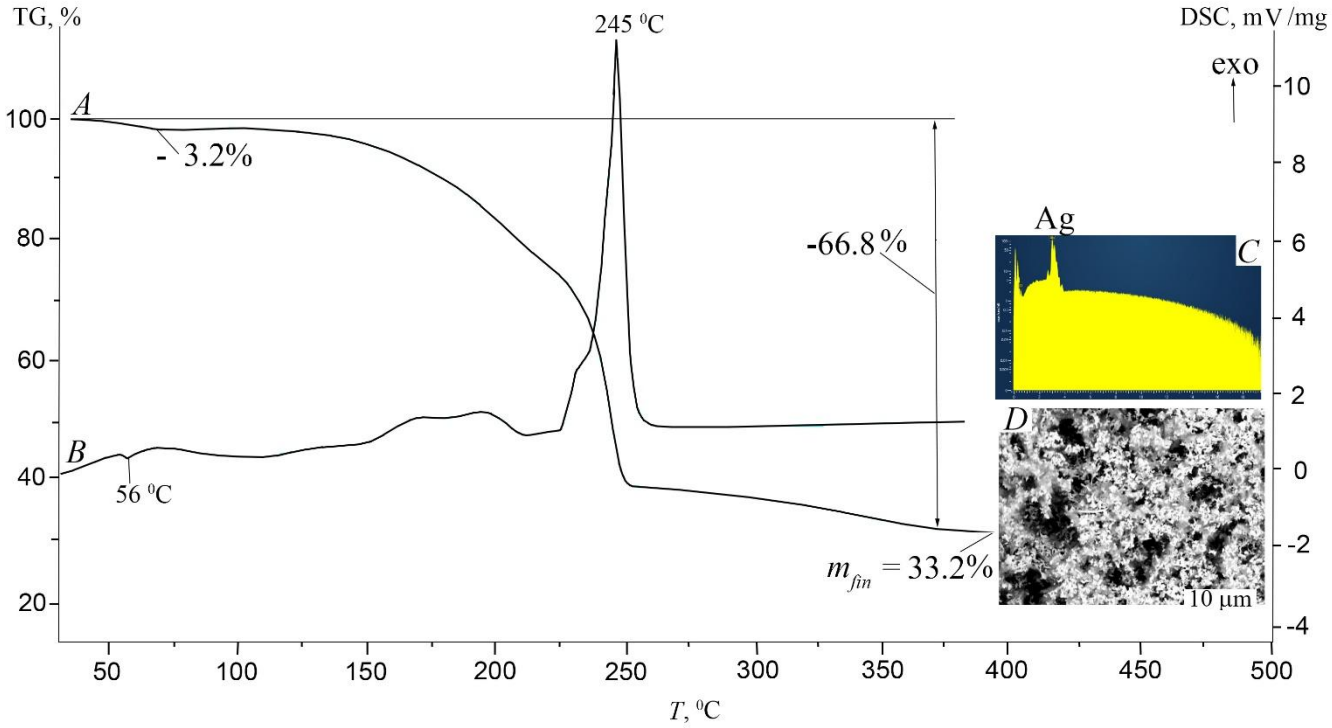


Figure S2 TG (*A*), DSC (*B*) curves and residue after thermolysis according to EDX data (*C*) and photo SEM (*D*) of complex **1**.