

Electronic Supplementary Information (ESI)

A novel carbazole-based gold(I) complex with interesting solid-state multi-stimuli-responsive characteristics

Zhao Chen, Jinhua Liang, Yuting Nie, Xuqingfeng Xu, Guang-Ao Yu, Jun Yin,* and Sheng Hua Liu*

Key Laboratory of Pesticide and Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, P. R. China

Tel: +86-27-67867725 Fax: +86-27-67867725

Corresponding author E-mail: yinj@mail.ccnu.edu.cn, chshliu@mail.ccnu.edu.cn

Supporting Information.pdf

Movie S1.3gp

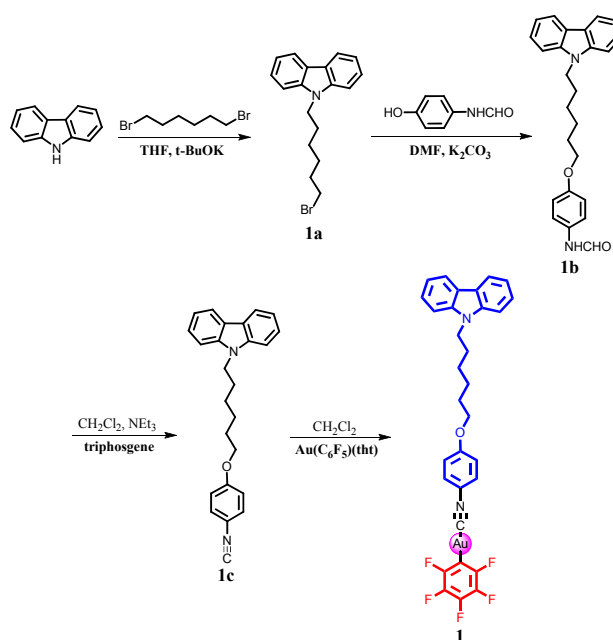
TABLE OF CONTENTS

1. Experimental Section.....	S3
2. References.....	S6
3. Figs. S1-S7.....	S6
4. Tables S1-S4.....	S10
5. Copies of NMR spectra and Mass spectra.....	S16

1. Experimental Section

Materials and measurements

The starting material carbazole purchased from Alfa Aesar was used as received. CH_2Cl_2 was dried with CaH_2 then distilled, and ultra-pure water was used in the experiments. All other starting materials and reagents were purchased as analytical-grade from Shen Shi Hua Gong Company (China) and used without further purification. Compounds **1a**^[1], N-(4-hydroxyphenyl)formamide^[2], $\text{Au}(\text{C}_6\text{F}_5)(\text{tht})$ ^[3] (tht = tetrahydrothiophene) were prepared by procedures described in the corresponding literatures. ^1H NMR (400 MHz) and ^{13}C NMR (100.6 MHz) spectra were collected on American Varian Mercury Plus 400 spectrometer (400 MHz). ^1H NMR spectra are reported as followed: chemical shift in ppm (δ) relative to the chemical shift of TMS at 0.00 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, m = multiplet), and coupling constant (Hz). ^{13}C NMR chemical shifts reported in ppm (δ) relative to the central line of triplet for CDCl_3 at 77 ppm. ^{19}F NMR chemical shifts are relative to C_6F_6 ($\delta = -163.00$). EI-MS was obtained using Thermo scientific DSQ II. Elemental analyses (C, H, N) were performed by the Microanalytical Services, College of Chemistry, CCNU. Fluorescence spectra were recorded on a Hitachi-F-4500 fluorescence spectrophotometer and Fluoromax-P luminescence spectrometer (HORIBA JOBIN YVON INC.). XRD studies were recorded on a Shimadzu XRD-6000 diffractometer using Ni-filtered and graphite-monochromated Cu K α radiation ($\lambda = 1.54 \text{ \AA}$, 40 kV, 30 mA). The DMF/water mixtures with various water fractions were prepared by slowly adding ultra-pure water into the DMF solution of samples. The X-ray crystal-structure determinations of complex **1** were obtained on a Bruker APEX DUO CCD system.



Scheme S1. Synthesis of the complex **1**.

General procedure for the synthesis

Synthesis of **1b**: A mixture of compounds **1a** (12.44 mmol, 4.11 g), N-(4-hydroxyphenyl)formamide (13.69 mmol), K₂CO₃ (54.76 mmol) were stirred in DMF (50 ml) for overnight under an argon atmosphere at 60 °C. After completion of present reaction, DMF was removed from reaction system by vacuum distillation. the residual mixture was extracted with dichloromethane (3 × 20 mL). The combined organic layers were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residues were purified by column chromatography, affording the expected white solid product in a yield of 73 %. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.49-8.30 (m, 1H), 8.10 (d, J = 8 Hz, 2H), 7.58-7.39 (m, 5H), 7.22 (d, J = 8 Hz, 2H), 7.07-6.97 (m, 2H), 6.83-6.80 (m, 2H), 4.32 (t, J = 6 Hz, 2H), 3.88 (t, J = 6 Hz, 2H), 1.91 (s, 2H), 1.72 (s, 2H), 1.47 (s, 4H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 163.03, 158.82, 156.92, 156.02, 140.30, 129.71, 129.29, 125.54, 122.70, 121.62, 121.46, 120.28, 118.69, 115.32, 114.67, 108.57, 67.87, 42.82, 28.95, 28.85, 26.97, 25.81. EI-MS: m/z= 386.37[M]⁺. Anal. Calcd. for C₂₅H₂₆N₂O₂: C, 77.69; H, 6.78; N, 7.25. Found: C, 77.65; H, 6.76; N, 7.28.

Synthesis of **1c**: A CH₂Cl₂ suspension (15 ml) of **1b** (0.90 g, 2.33 mmol) and

triethylamine (5 ml) was cooled to 0 °C. To the mixture was added dropwise a CH₂Cl₂ solution (10 ml) of triphosgene (0.76 g, 2.56 mmol). The mixture was refluxed under an argon atmosphere for 3 h, then 10 % aq. Na₂CO₃ (50 ml) was added dropwise at room temperature. the mixture was extracted with dichloromethane (3 × 20 mL). The combined organic layers were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residues were purified by column chromatography, affording the expected earth yellow solid product in a yield of 83 %. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.11 (d, J = 4 Hz, 2H), 7.45-7.39 (m, 4H), 7.28-7.23 (m, 4H), 6.78 (d, J = 8 Hz, 2H), 4.33 (t, J = 6 Hz, 2H), 3.89-3.84 (m, 2H), 1.91 (d, J = 8 Hz, 2H), 1.72 (d, J = 8 Hz, 2H), 1.45 (s, 4H). ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 162.36, 159.11, 140.21, 127.48, 125.46, 122.64, 120.19, 118.63, 114.77, 108.48, 67.88, 42.67, 28.74, 28.67, 26.82, 25.68. EI-MS: m/z= 368.35[M]⁺. Anal. Calcd. for C₂₅H₂₄N₂O: C, 81.49; H, 6.57; N, 7.60. Found: C, 81.46; H, 6.55; N, 7.64.

Synthesis of **1**: A mixture of C₆F₅Au(tht) (0.18 g, 0.40 mmol) and **1c** (0.14 g, 0.38 mmol) was stirred in CH₂Cl₂ (20 ml) over night under an argon atmosphere at room temperature. After completion of present reaction, the solvent was evaporated. A small amount of CH₂Cl₂ was added, and then a lot of n-hexane was added. Collecting the white solid product by suction filtration. Yield = 92 %. ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.11 (d, J = 8 Hz, 2H), 7.48-7.40 (m, 6H), 7.23 (d, J = 8 Hz, 2H), 6.86 (d, J = 8 Hz, 2H), 4.34 (t, J = 8 Hz, 2H), 3.92 (t, J = 6 Hz, 2H), 1.94 (d, J = 8 Hz, 2H), 1.76 (s, 2H), 1.47 (s, 4H). ¹⁹F NMR (CDCl₃): δ (ppm) = -116.98, -158.52, -163.35. Anal. Calcd. for C₃₁H₂₄AuF₅N₂O: C, 50.83; H, 3.30; N, 3.82. Found: C, 50.76; H, 3.35; N, 3.85.

Crystallographic Details

Two types of single crystals of complex **1** suitable for X-ray analysis were obtained by slow diffusion of *n*-hexane into a solution of dichloromethane. A white-emitting crystal of **1** with approximate dimensions of 0.10 × 0.10 × 0.10 mm³ for **1** was mounted on a glass fiber for diffraction experiment. A yellow-emitting crystal of **1** with approximate dimensions of 0.20 × 0.15 × 0.10 mm³ for **1** was also mounted on a

glass fiber for diffraction experiment. Intensity data were collected on a Nonius Kappa CCD diffractometer with Mo K α radiation (0.71073 Å) at room temperature. The structures were solved by a combination of direct methods (SHELXS-97)^[4] and Fourier difference techniques and refined by full-matrix least-squares (SHELXL-97)^[5]. All non-H atoms were refined anisotropically. The hydrogen atoms were placed in the ideal positions and refined as riding atoms. Further crystal datas are provided in Table S1 and S3. Bond distances and angles are given in Table S2 and Table S4. Crystallographic data for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplemental publication CCDC 1044227 and 1044242.

2. References

- [1] W. Huang, L. Su, Z. Bo, *J. Am. Chem. Soc.*, 2009, **131**, 10348.
- [2] M. Hosseini-Sarvari, H. Sharghi, *J. Org. Chem.*, 2006, **71**, 6652.
- [3] R. Uson, A. Laguna, M. Laguna, *Inorg. Synth.*, 1989, **26**, 85.
- [4] G. M. Sheldrick, SHELXS-97: Program for crystal structure solution, University of Göttingen, Göttingen, Germany, **1997**.
- [5] G. M. Sheldrick, SHELXL-97: Program for crystal structure refinement, University of Göttingen, Göttingen, Germany, **1997**.

3. Figs. S1-S7

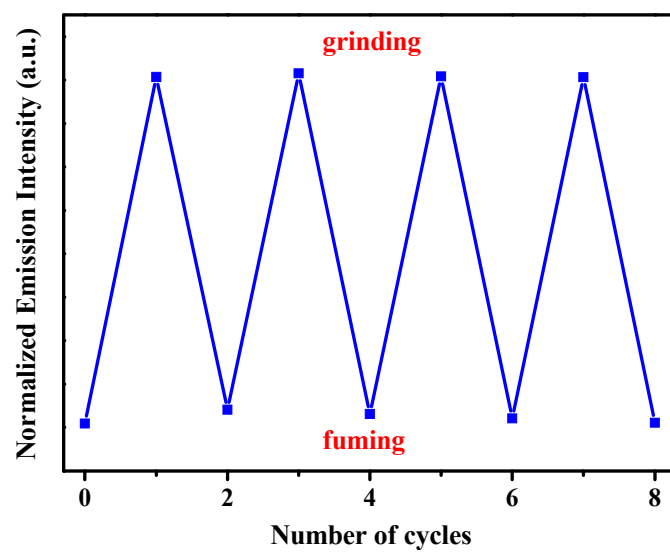


Figure S1. Invertible grinding-fuming processes of the photoluminescence of complex **1** at 513 nm.

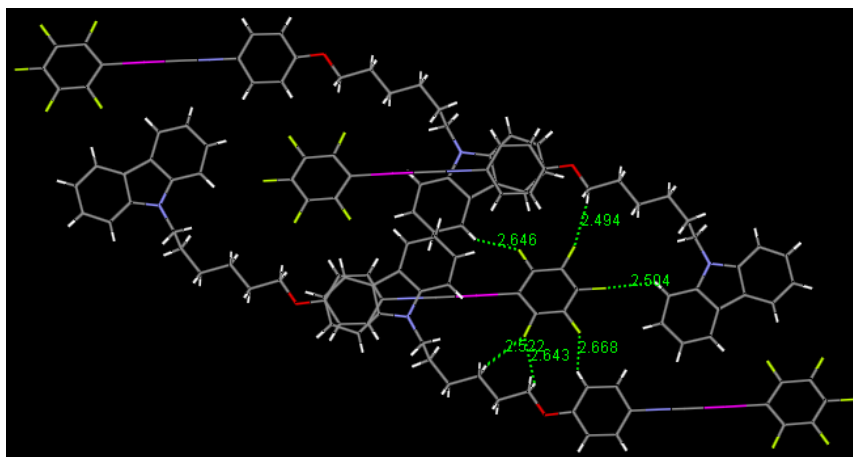


Figure S2. Crystal packing diagram of complex **1**. It shows weak interactions of intermolecular C-H...F.

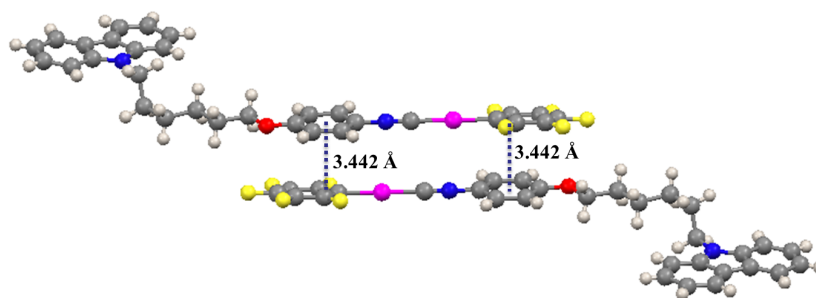


Figure S3. Crystal packing diagram of complex **1**. It shows the intermolecular $\pi - \pi$ interactions.

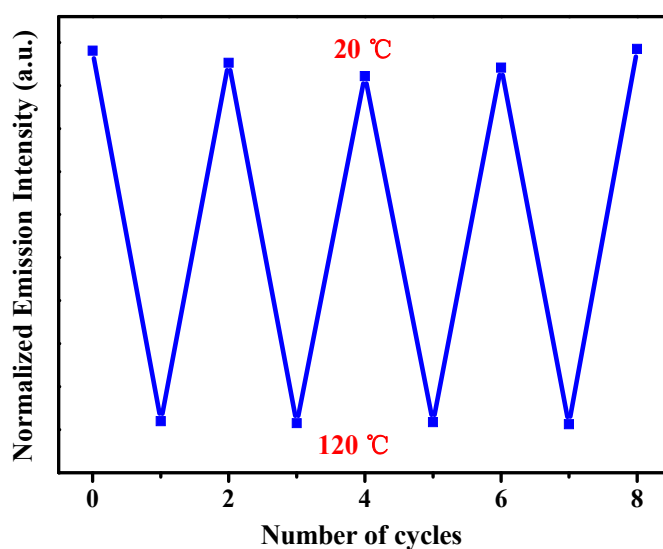


Figure S4. Invertible heating-cooling processes (20 °C - 120 °C) of the photoluminescence of complex **1** at 423 nm.

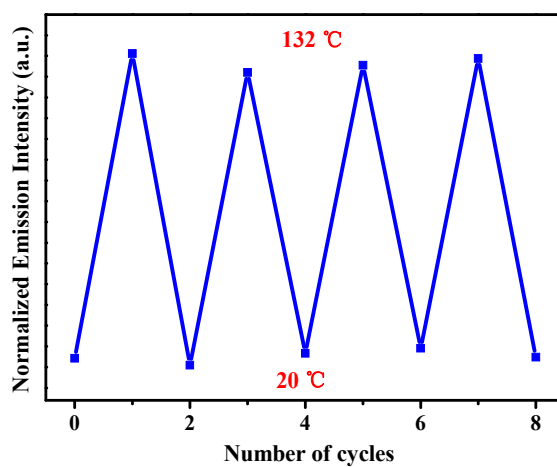


Figure S5. Invertible heating-cooling processes (20 °C - 132 °C) of the

photoluminescence of complex **1** at 550 nm.

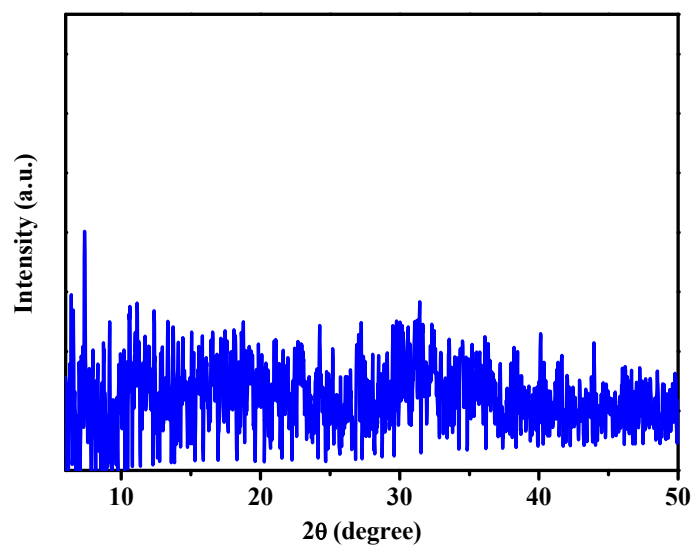


Figure S6. The XRD pattern of the green-emitting solid sample **1**.

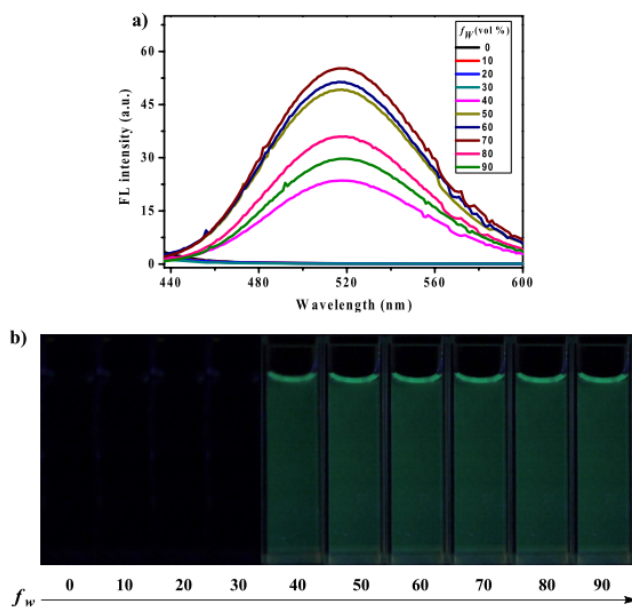


Figure S7. a) PL spectra of the dilute solutions of luminogen **1** ($1.0 \times 10^{-5} \text{ mol L}^{-1}$) in DMF-H₂O mixtures with different f_w . Excitation wavelength = 330 nm; b) The fluorescence images of **1** (concentration: $1.0 \times 10^{-5} \text{ mol L}^{-1}$) in diverse DMF-H₂O mixtures with various f_w values (0-90 %) under 365 nm UV irradiation.

4. Tables S1-S4

Table S1. Structure determination summary of the white-emitting single crystal of **1**.

Empirical formula	C ₃₁ H ₂₄ Au F ₅ N ₂ O
Formula weight	732.49
Temperature (K)	295(2)
Crystal system	Triclinic
Space group	P-1
<i>a</i> (Å)	7.6240(11)
<i>b</i> (Å)	13.6244(18)
<i>c</i> (Å)	14.516(2)
α (deg)	102.723(2)
β (deg)	100.056(2)
γ (deg)	105.814(2)
<i>V</i> (Å ³)	1370.2(3)
<i>Z</i>	2
Absorption coefficient (mm ⁻¹)	5.430
<i>F</i> (000)	712
Theta range for data collection (deg)	1.49 to 26.00
Index ranges	-9 ≤ <i>h</i> ≤ 9, -16 ≤ <i>k</i> ≤ 16, -17 ≤ <i>l</i> ≤ 17
Reflections collected/unique	10466/5355 (<i>R</i> _{int} = 0.0328)
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0366, <i>wR</i> ₂ = 0.1131
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0414, <i>wR</i> ₂ = 0.1207
Goodness-of-fit on <i>F</i> ²	1.103
Largest difference peak and hole (e Å ⁻³)	0.851, -1.692

Table S2. Bond lengths [Å] and angles [°] of the white-emitting single crystal of **1**.

Au(1)-C(25)	1.962(7)	C(1)-N(1)	1.408(8)
Au(1)-C(26)	2.017(6)	C(2)-C(3)	1.364(9)
C(1)-C(2)	1.365(10)	C(2)-H(2)	0.9300
C(1)-C(6)	1.397(9)	C(3)-C(4)	1.403(8)

C(3)-H(3)	0.9300	C(19)-C(24)	1.402(7)
C(4)-O(1)	1.361(7)	C(20)-C(21)	1.372(9)
C(4)-C(5)	1.371(9)	C(20)-H(20)	0.9300
C(5)-C(6)	1.389(8)	C(21)-C(22)	1.371(10)
C(5)-H(5)	0.9300	C(21)-H(21)	0.9300
C(6)-H(6)	0.9300	C(22)-C(23)	1.361(9)
C(7)-O(1)	1.427(7)	C(22)-H(22)	0.9300
C(7)-C(8)	1.498(8)	C(23)-C(24)	1.380(8)
C(7)-H(7A)	0.9700	C(23)-H(23)	0.9300
C(7)-H(7B)	0.9700	C(24)-N(2)	1.395(6)
C(8)-C(9)	1.507(8)	C(25)-N(1)	1.151(9)
C(8)-H(8A)	0.9700	C(26)-C(31)	1.372(9)
C(8)-H(8B)	0.9700	C(26)-C(27)	1.395(9)
C(9)-C(10)	1.506(7)	C(27)-F(1)	1.333(8)
C(9)-H(9A)	0.9700	C(27)-C(28)	1.355(9)
C(9)-H(9B)	0.9700	C(28)-C(29)	1.321(12)
C(10)-C(11)	1.528(6)	C(28)-F(2)	1.387(8)
C(10)-H(10A)	0.9700	C(29)-F(3)	1.367(7)
C(10)-H(10B)	0.9700	C(29)-C(30)	1.374(11)
C(11)-C(12)	1.509(7)	C(30)-F(4)	1.353(8)
C(11)-H(11A)	0.9700	C(30)-C(31)	1.356(9)
C(11)-H(11B)	0.9700	C(31)-F(5)	1.354(7)
C(12)-N(2)	1.463(6)		
C(12)-H(12A)	0.9700	C(25)-Au(1)-C(26)	178.7(2)
C(12)-H(12B)	0.9700	C(2)-C(1)-C(6)	121.5(6)
C(13)-C(14)	1.369(8)	C(2)-C(1)-N(1)	120.2(6)
C(13)-N(2)	1.387(7)	C(6)-C(1)-N(1)	118.2(6)
C(13)-C(18)	1.424(7)	C(3)-C(2)-C(1)	119.2(6)
C(14)-C(15)	1.374(9)	C(3)-C(2)-H(2)	120.4
C(14)-H(14)	0.9300	C(1)-C(2)-H(2)	120.4
C(15)-C(16)	1.401(10)	C(2)-C(3)-C(4)	120.5(6)
C(15)-H(15)	0.9300	C(2)-C(3)-H(3)	119.7
C(16)-C(17)	1.358(9)	C(4)-C(3)-H(3)	119.7
C(16)-H(16)	0.9300	O(1)-C(4)-C(5)	124.8(5)
C(17)-C(18)	1.400(8)	O(1)-C(4)-C(3)	114.9(5)
C(17)-H(17)	0.9300	C(5)-C(4)-C(3)	120.3(6)
C(18)-C(19)	1.445(8)	C(4)-C(5)-C(6)	119.3(6)
C(19)-C(20)	1.390(8)	C(4)-C(5)-H(5)	120.3

C(6)-C(5)-H(5)	120.3	C(11)-C(12)-H(12B)	108.7
C(5)-C(6)-C(1)	119.1(6)	H(12A)-C(12)-H(12B)	107.6
C(5)-C(6)-H(6)	120.4	C(14)-C(13)-N(2)	129.5(5)
C(1)-C(6)-H(6)	120.4	C(14)-C(13)-C(18)	122.8(5)
O(1)-C(7)-C(8)	109.1(5)	N(2)-C(13)-C(18)	107.6(4)
O(1)-C(7)-H(7A)	109.9	C(13)-C(14)-C(15)	117.5(5)
C(8)-C(7)-H(7A)	109.9	C(13)-C(14)-H(14)	121.3
O(1)-C(7)-H(7B)	109.9	C(15)-C(14)-H(14)	121.3
C(8)-C(7)-H(7B)	109.9	C(14)-C(15)-C(16)	121.0(6)
H(7A)-C(7)-H(7B)	108.3	C(14)-C(15)-H(15)	119.5
C(7)-C(8)-C(9)	113.1(5)	C(16)-C(15)-H(15)	119.5
C(7)-C(8)-H(8A)	109.0	C(17)-C(16)-C(15)	121.7(6)
C(9)-C(8)-H(8A)	109.0	C(17)-C(16)-H(16)	119.1
C(7)-C(8)-H(8B)	109.0	C(15)-C(16)-H(16)	119.1
C(9)-C(8)-H(8B)	109.0	C(16)-C(17)-C(18)	119.1(5)
H(8A)-C(8)-H(8B)	107.8	C(16)-C(17)-H(17)	120.5
C(10)-C(9)-C(8)	114.4(5)	C(18)-C(17)-H(17)	120.5
C(10)-C(9)-H(9A)	108.7	C(17)-C(18)-C(13)	117.8(5)
C(8)-C(9)-H(9A)	108.7	C(17)-C(18)-C(19)	134.7(5)
C(10)-C(9)-H(9B)	108.7	C(13)-C(18)-C(19)	107.4(4)
C(8)-C(9)-H(9B)	108.7	C(20)-C(19)-C(24)	118.2(5)
H(9A)-C(9)-H(9B)	107.6	C(20)-C(19)-C(18)	135.2(5)
C(9)-C(10)-C(11)	113.7(4)	C(24)-C(19)-C(18)	106.6(4)
C(9)-C(10)-H(10A)	108.8	C(21)-C(20)-C(19)	119.5(6)
C(11)-C(10)-H(10A)	108.8	C(21)-C(20)-H(20)	120.3
C(9)-C(10)-H(10B)	108.8	C(19)-C(20)-H(20)	120.3
C(11)-C(10)-H(10B)	108.8	C(22)-C(21)-C(20)	120.2(6)
H(10A)-C(10)-H(10B)	107.7	C(22)-C(21)-H(21)	119.9
C(12)-C(11)-C(10)	111.5(4)	C(20)-C(21)-H(21)	119.9
C(12)-C(11)-H(11A)	109.3	C(23)-C(22)-C(21)	122.7(6)
C(10)-C(11)-H(11A)	109.3	C(23)-C(22)-H(22)	118.6
C(12)-C(11)-H(11B)	109.3	C(21)-C(22)-H(22)	118.6
C(10)-C(11)-H(11B)	109.3	C(22)-C(23)-C(24)	117.0(5)
H(11A)-C(11)-H(11B)	108.0	C(22)-C(23)-H(23)	121.5
N(2)-C(12)-C(11)	114.4(4)	C(24)-C(23)-H(23)	121.5
N(2)-C(12)-H(12A)	108.7	C(23)-C(24)-N(2)	128.7(5)
C(11)-C(12)-H(12A)	108.7	C(23)-C(24)-C(19)	122.3(5)
N(2)-C(12)-H(12B)	108.7	N(2)-C(24)-C(19)	109.0(4)

N(1)-C(25)-Au(1)	179.1(6)	F(3)-C(29)-C(30)	119.4(7)
C(31)-C(26)-C(27)	112.9(6)	F(4)-C(30)-C(31)	120.5(7)
C(31)-C(26)-Au(1)	122.9(5)	F(4)-C(30)-C(29)	121.0(6)
C(27)-C(26)-Au(1)	124.2(5)	C(31)-C(30)-C(29)	118.5(6)
F(1)-C(27)-C(28)	117.3(6)	F(5)-C(31)-C(30)	116.5(6)
F(1)-C(27)-C(26)	120.1(6)	F(5)-C(31)-C(26)	118.4(6)
C(28)-C(27)-C(26)	122.7(6)	C(30)-C(31)-C(26)	125.1(6)
C(29)-C(28)-C(27)	121.7(6)	C(25)-N(1)-C(1)	179.4(8)
C(29)-C(28)-F(2)	118.5(6)	C(13)-N(2)-C(24)	109.4(4)
C(27)-C(28)-F(2)	119.7(7)	C(13)-N(2)-C(12)	124.7(4)
C(28)-C(29)-F(3)	121.5(7)	C(24)-N(2)-C(12)	125.8(4)
C(28)-C(29)-C(30)	119.1(6)	C(4)-O(1)-C(7)	117.8(5)

Table S3. Structure determination summary of the yellow-emitting single crystal of **1**.

Empirical formula	C ₃₂ H ₂₆ Au Cl ₂ F ₅ N ₂ O
Formula weight	817.41
Temperature (K)	100(2)
Crystal system	Monoclinic
Space group	P2(1)/c
<i>a</i> (Å)	18.712(4)
<i>b</i> (Å)	8.2180(16)
<i>c</i> (Å)	21.103(4)
α (deg)	90
β (deg)	114.175(2)
γ (deg)	90
<i>V</i> (Å ³)	2960.5(10)
<i>Z</i>	4
Absorption coefficient (mm ⁻¹)	5.211
F (000)	1592
Theta range for data collection (deg)	1.19 to 30.00
Index ranges	-26 ≤ <i>h</i> ≤ 26, -11 ≤ <i>k</i> ≤ 11, -29 ≤ <i>l</i> ≤ 29
Reflections collected/unique	28688/8617 (<i>R</i> _{int} = 0.0335)
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0260, <i>wR</i> ₂ = 0.0681
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0323, <i>wR</i> ₂ = 0.0710

Goodness-of-fit on F^2	1.097
Largest difference peak and hole(e \AA^{-3})	2.061, -1.887

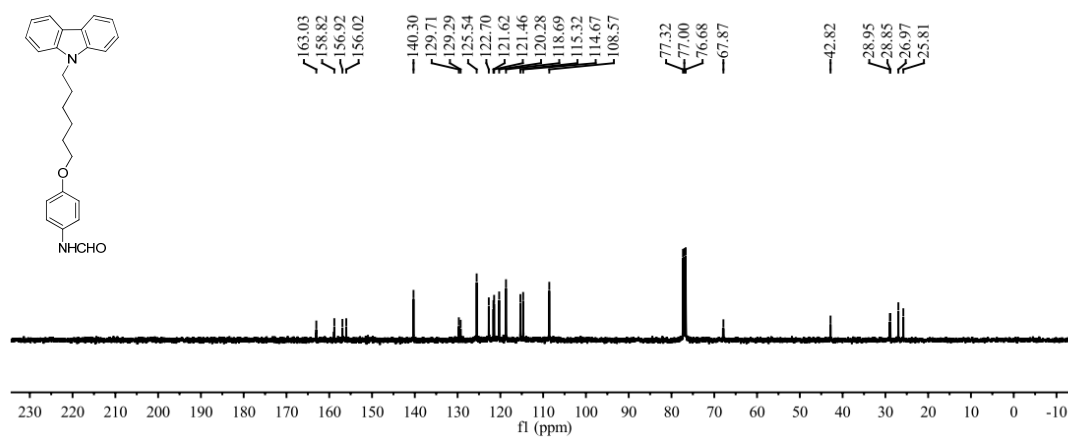
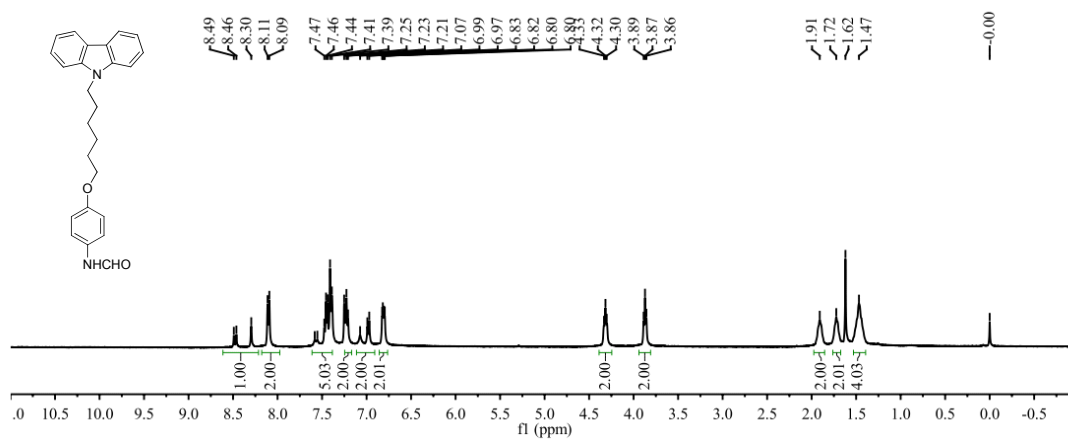
Table S4. Bond lengths [\AA] and angles [$^\circ$] of the yellow-emitting single crystal of **1**.

Au(1)-C(25)	1.967(3)	C(15)-C(16)	1.526(3)
Au(1)-C(26)	2.023(2)	C(15)-H(15A)	0.9900
C(1)-N(1)	1.382(3)	C(15)-H(15B)	0.9900
C(1)-C(2)	1.394(3)	C(16)-C(17)	1.525(3)
C(1)-C(6)	1.407(3)	C(16)-H(16A)	0.9900
C(2)-C(3)	1.390(4)	C(16)-H(16B)	0.9900
C(2)-H(2)	0.9500	C(17)-C(18)	1.502(3)
C(3)-C(4)	1.399(4)	C(17)-H(17A)	0.9900
C(3)-H(3)	0.9500	C(17)-H(17B)	0.9900
C(4)-C(5)	1.382(4)	C(18)-O(1)	1.442(3)
C(4)-H(4)	0.9500	C(18)-H(18A)	0.9900
C(5)-C(6)	1.399(4)	C(18)-H(18B)	0.9900
C(5)-H(5)	0.9500	C(19)-O(1)	1.358(3)
C(6)-C(7)	1.443(4)	C(19)-C(20)	1.395(4)
C(7)-C(8)	1.398(3)	C(19)-C(24)	1.397(3)
C(7)-C(12)	1.411(4)	C(20)-C(21)	1.386(4)
C(8)-C(9)	1.388(5)	C(20)-H(20)	0.9500
C(8)-H(8)	0.9500	C(21)-C(22)	1.382(4)
C(9)-C(10)	1.393(5)	C(21)-H(21)	0.9500
C(9)-H(9)	0.9500	C(22)-C(23)	1.395(3)
C(10)-C(11)	1.385(4)	C(22)-N(2)	1.401(3)
C(10)-H(10)	0.9500	C(23)-C(24)	1.375(4)
C(11)-C(12)	1.396(4)	C(23)-H(23)	0.9500
C(11)-H(11)	0.9500	C(24)-H(24)	0.9500
C(12)-N(1)	1.383(3)	C(25)-N(2)	1.146(4)
C(13)-N(1)	1.456(3)	C(26)-C(27)	1.385(3)
C(13)-C(14)	1.521(4)	C(26)-C(31)	1.388(3)
C(13)-H(13A)	0.9900	C(27)-F(1)	1.349(3)
C(13)-H(13B)	0.9900	C(27)-C(28)	1.375(4)
C(14)-C(15)	1.525(4)	C(28)-F(2)	1.345(3)
C(14)-H(14A)	0.9900	C(28)-C(29)	1.372(4)
C(14)-H(14B)	0.9900	C(29)-F(3)	1.343(3)

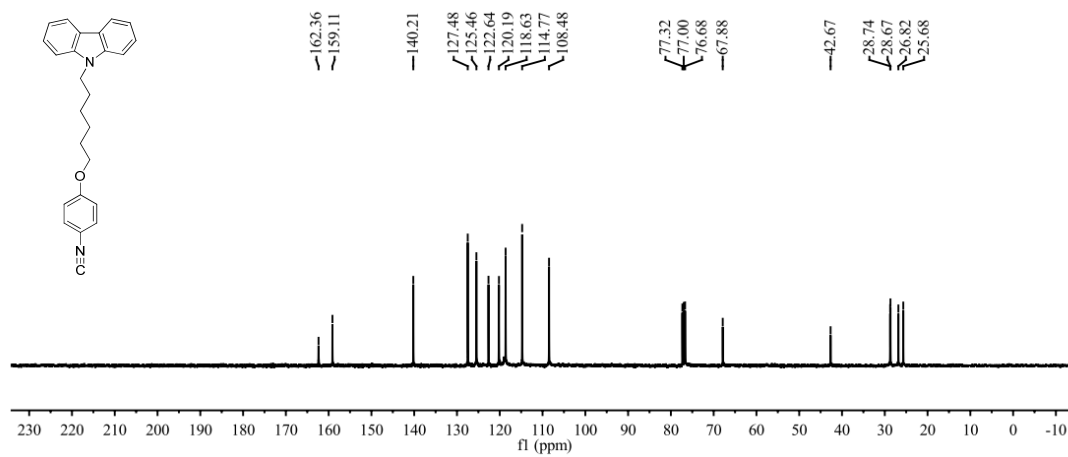
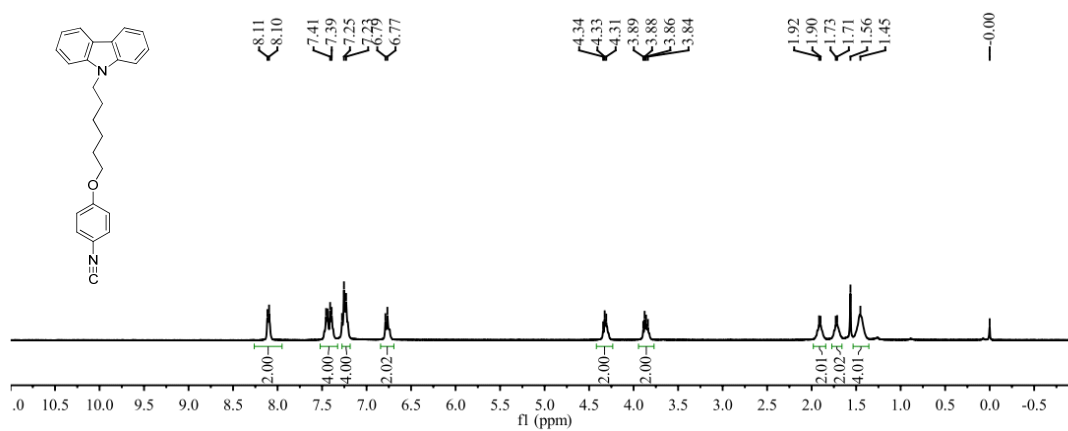
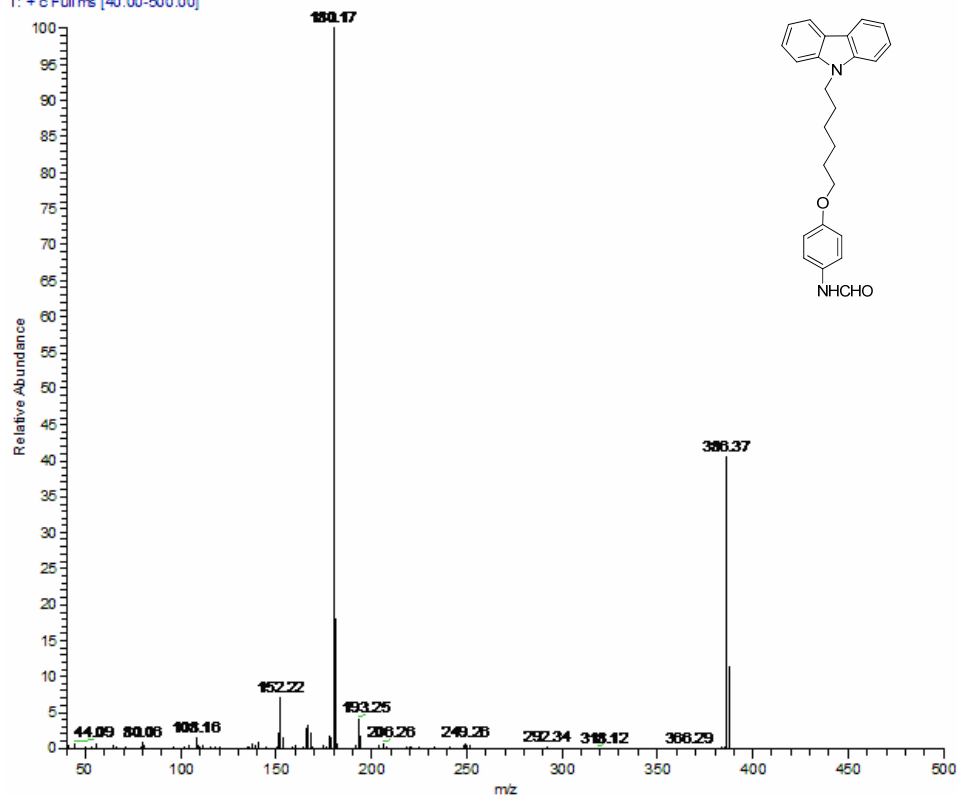
C(29)-C(30)	1.375(4)	C(11)-C(10)-H(10)	119.1
C(30)-F(4)	1.342(3)	C(9)-C(10)-H(10)	119.1
C(30)-C(31)	1.373(4)	C(10)-C(11)-C(12)	117.3(3)
C(31)-F(5)	1.351(3)	C(10)-C(11)-H(11)	121.3
C(32)-Cl(1)	1.732(4)	C(12)-C(11)-H(11)	121.3
C(32)-Cl(2)	1.754(5)	N(1)-C(12)-C(11)	129.3(2)
C(32)-H(32A)	0.9900	N(1)-C(12)-C(7)	108.9(2)
C(32)-H(32B)	0.9900	C(11)-C(12)-C(7)	121.8(2)
		N(1)-C(13)-C(14)	114.1(2)
C(25)-Au(1)-C(26)	177.32(11)	N(1)-C(13)-H(13A)	108.7
N(1)-C(1)-C(2)	128.4(2)	C(14)-C(13)-H(13A)	108.7
N(1)-C(1)-C(6)	109.9(2)	N(1)-C(13)-H(13B)	108.7
C(2)-C(1)-C(6)	121.7(2)	C(14)-C(13)-H(13B)	108.7
C(3)-C(2)-C(1)	117.3(2)	H(13A)-C(13)-H(13B)	107.6
C(3)-C(2)-H(2)	121.4	C(13)-C(14)-C(15)	113.2(2)
C(1)-C(2)-H(2)	121.4	C(13)-C(14)-H(14A)	108.9
C(2)-C(3)-C(4)	121.6(2)	C(15)-C(14)-H(14A)	108.9
C(2)-C(3)-H(3)	119.2	C(13)-C(14)-H(14B)	108.9
C(4)-C(3)-H(3)	119.2	C(15)-C(14)-H(14B)	108.9
C(5)-C(4)-C(3)	120.8(2)	H(14A)-C(14)-H(14B)	107.8
C(5)-C(4)-H(4)	119.6	C(14)-C(15)-C(16)	113.1(2)
C(3)-C(4)-H(4)	119.6	C(14)-C(15)-H(15A)	109.0
C(4)-C(5)-C(6)	118.7(2)	C(16)-C(15)-H(15A)	109.0
C(4)-C(5)-H(5)	120.6	C(14)-C(15)-H(15B)	109.0
C(6)-C(5)-H(5)	120.6	C(16)-C(15)-H(15B)	109.0
C(5)-C(6)-C(1)	119.8(2)	H(15A)-C(15)-H(15B)	107.8
C(5)-C(6)-C(7)	134.3(2)	C(17)-C(16)-C(15)	111.4(2)
C(1)-C(6)-C(7)	105.9(2)	C(17)-C(16)-H(16A)	109.3
C(8)-C(7)-C(12)	119.5(3)	C(15)-C(16)-H(16A)	109.3
C(8)-C(7)-C(6)	133.5(3)	C(17)-C(16)-H(16B)	109.3
C(12)-C(7)-C(6)	107.1(2)	C(15)-C(16)-H(16B)	109.3
C(9)-C(8)-C(7)	118.7(3)	H(16A)-C(16)-H(16B)	108.0
C(9)-C(8)-H(8)	120.6	C(18)-C(17)-C(16)	112.6(2)
C(7)-C(8)-H(8)	120.6	C(18)-C(17)-H(17A)	109.1
C(8)-C(9)-C(10)	120.9(3)	C(16)-C(17)-H(17A)	109.1
C(8)-C(9)-H(9)	119.6	C(18)-C(17)-H(17B)	109.1
C(10)-C(9)-H(9)	119.5	C(16)-C(17)-H(17B)	109.1
C(11)-C(10)-C(9)	121.7(3)	H(17A)-C(17)-H(17B)	107.8

O(1)-C(18)-C(17)	107.22(19)	C(31)-C(26)-Au(1)	125.08(18)
O(1)-C(18)-H(18A)	110.3	F(1)-C(27)-C(28)	117.1(2)
C(17)-C(18)-H(18A)	110.3	F(1)-C(27)-C(26)	119.1(2)
O(1)-C(18)-H(18B)	110.3	C(28)-C(27)-C(26)	123.8(2)
C(17)-C(18)-H(18B)	110.3	F(2)-C(28)-C(29)	119.6(3)
H(18A)-C(18)-H(18B)	108.5	F(2)-C(28)-C(27)	120.8(2)
O(1)-C(19)-C(20)	124.0(2)	C(29)-C(28)-C(27)	119.6(2)
O(1)-C(19)-C(24)	115.9(2)	F(3)-C(29)-C(28)	120.1(3)
C(20)-C(19)-C(24)	120.0(2)	F(3)-C(29)-C(30)	120.6(3)
C(21)-C(20)-C(19)	119.9(2)	C(28)-C(29)-C(30)	119.2(2)
C(21)-C(20)-H(20)	120.1	F(4)-C(30)-C(31)	121.6(3)
C(19)-C(20)-H(20)	120.1	F(4)-C(30)-C(29)	119.0(2)
C(22)-C(21)-C(20)	119.3(2)	C(31)-C(30)-C(29)	119.4(2)
C(22)-C(21)-H(21)	120.3	F(5)-C(31)-C(30)	116.4(2)
C(20)-C(21)-H(21)	120.3	F(5)-C(31)-C(26)	119.7(2)
C(21)-C(22)-C(23)	121.4(2)	C(30)-C(31)-C(26)	123.9(2)
C(21)-C(22)-N(2)	118.9(2)	Cl(1)-C(32)-Cl(2)	114.3(2)
C(23)-C(22)-N(2)	119.8(2)	Cl(1)-C(32)-H(32A)	108.7
C(24)-C(23)-C(22)	119.2(2)	Cl(2)-C(32)-H(32A)	108.7
C(24)-C(23)-H(23)	120.4	Cl(1)-C(32)-H(32B)	108.7
C(22)-C(23)-H(23)	120.4	Cl(2)-C(32)-H(32B)	108.7
C(23)-C(24)-C(19)	120.2(2)	H(32A)-C(32)-H(32B)	107.6
C(23)-C(24)-H(24)	119.9	C(1)-N(1)-C(12)	108.3(2)
C(19)-C(24)-H(24)	119.9	C(1)-N(1)-C(13)	125.0(2)
N(2)-C(25)-Au(1)	177.9(3)	C(12)-N(1)-C(13)	126.6(2)
C(27)-C(26)-C(31)	114.1(2)	C(25)-N(2)-C(22)	178.8(3)
C(27)-C(26)-Au(1)	120.79(18)	C(19)-O(1)-C(18)	118.20(19)

5. Copies of NMR spectra and Mass spectra



CZ235 #677 RT: 3.15 AV: 1 SB: 749 0.04-2.92, 3.44-4.00 NL: 3.45E5
T: + c Full ms [40.00-500.00]



CZ237 #556 RT: 2.60 AV: 1 SB: 584 0.11-2.33 , 2.84-3.30 NL: 1.23E6
T: + c Full ms [40.00-500.00]

