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ORIGINAL PAPER

Synthesis, spectral analysis, and thermodynamic parameters of gold(III) complex in the presence of 4-bromo-2,6-bis(hydroxymethyl)phenol and *m*-nitroaniline

Özlen Altun[1] · **Zeliha Yoruç**[1]

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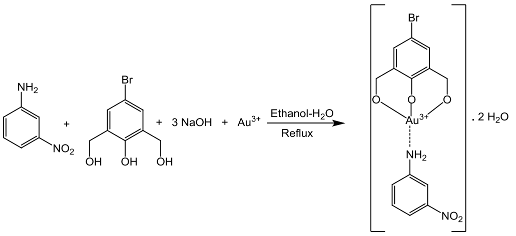
Abstract

In the present study, we synthesized 4-bromo-2,6-bis(hydroxymethyl)phenol (BBHMP), and a gold(III) complex was synthesized in the presence of BBHMP and *m*-nitroaniline (*m*-NA). The synthesized complex was characterized using spectral (mass, UV–Vis, FT-IR, ^{1}H NMR, ^{13}C NMR) and thermal measurements (TG–DTA). The results showed that the optimum conditions for the reactions are pH 7, $\lambda = 412$ nm and the mole ratio of BBHMP:*m*-NA:Au^{3+} is 1:1:1. The reaction of BBHMP and *m*-NA with Au(III) was found to be a complexation reaction and one BBHMP molecule and one *m*-NA molecule react with one Au(III) molecule. Additionally, the equilibrium constant (*K*), Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) were calculated at 298.16 and 313.16 K, respectively. According to the thermodynamic parameters, the reaction was exothermic because of the decrease in entropy and it was a non-spontaneous process.

Graphic abstract



Keywords Ligand · Au(III) · Structural analysis · Molar composition · Equilibrium constant

Introduction

4-Bromo-2,6-bis(hydroxymethyl)phenol (BBHMP) is a halogenated phenol derivative used for proteomic research and is obtained from 4-bromophenol, formaldehyde, and NaOH [1, 2]. Studies on BBHMP date back early 1990s with some performed more recently [3–10]. For example, a multidentate ligand was synthesized by Chokkanathan and Geetha [2] using BBHMP and diethylenetriamine with copper(II) precursors to form copper(II) complexes. In another study [11], the colorimetric method was defined for naked-eye detection of fluorine ion in the presence BBHMP which showed a selective and sensitive fluorescence quenching response towards fluorine ion among chlorine, bromine and iodine ions.

m-Nitroaniline (*m*-NA) [12] is a non-volatile stable solid with a functional nitro group in meta-position and is used as a raw material in dyes and as a chemical intermediate in many synthesis processes [13]. *m*-NA can enter into the environment where it undergoes complex transformations, making the compound a great concern in the field

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✉ Özlen Altun
ozlenaltun@yahoo.com

[1] Department of General and Inorganic Chemistry, Trakya University, Edirne 22030, Turkey

of environmental science because of its high toxicity and suspected carcinogenic role [14, 15].

There exist a large number of studies involving some mixed ligand complexes of Au(III) [16–22], but no study has been performed so far about the complex of Au(III) with BBHMP and *m*-NA. In the present study, we synthesized gold(III) complex in the presence of BBHMP and *m*-NA, and determined structural characterization with spectral and thermal analysis. We also determined reaction conditions, the composition of the complex and the thermodynamic parameters using spectral measurements.

Results and discussion

Determination of the reaction conditions

To determine the wavelength for the reactions, various solutions with pH values ranging from 1 to 10 of the molar composition (BBHMP + *m*-NA) with Au(III) were arranged in the mole ratio of 1:1 (Au^{3+}:BBHMP + *m*-NA) and the spectra were recorded at room temperature. The product produced absorption peaks at $\lambda = 322$ and 412 nm. Since the absorbance was fixed with pH values above 7 (Fig. 1), the absorbance value $\lambda = 412$ nm and the pH value of 7 were chosen as the working medium [23–27].

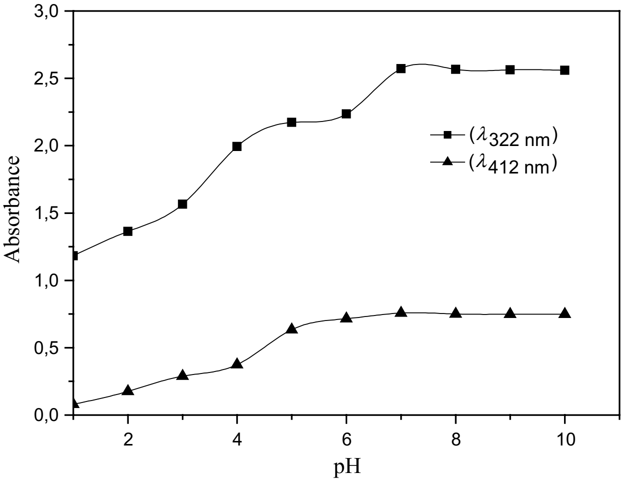


Fig. 1 The effect of pH on absorbance

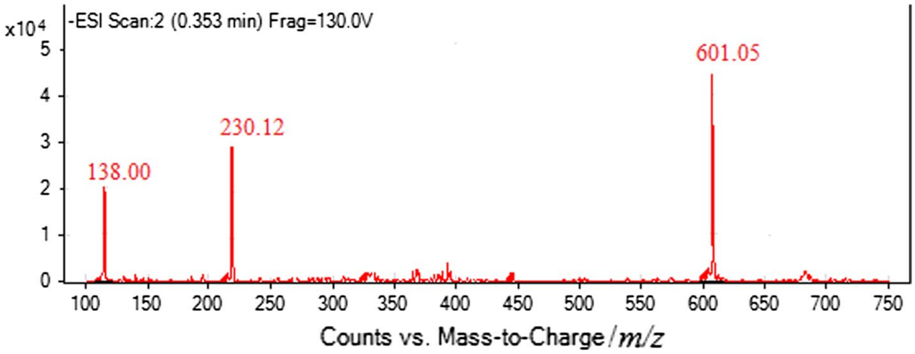
Structural analysis of the complex

Elemental analysis of C, H, N, O, and Au is compatible with the suggested general formula given for BBHMP and the complex. The molar conductivity of the complex was calculated with the equation $\Lambda m = K/C$, where C is the molar concentration of the complex solution. The Au(III) complex was dissolved in DMF solvent with a molar concentration of 10^{-3} M at 25 °C and used for measurements. The molar conductivity value of the complex was found as 49.6 Ω^{-1} mol^{-1} cm^2. Therefore, this complex can be regarded as a non-electrolyte complex [28–30]. The magnetic moment of the complex was calculated from the corrected magnetic susceptibilities determined at room temperature and was found as 0.00 BM. This result indicates diamagnetic nature, as expected for low spin d^8 complexes, suggesting square–planar geometry for the obtained Au(III) complex.

The electron impact mass spectrum of gold(III) complex was used to determine its molecular weight and fragments. In the mass spectrum of the complex (Fig. 2), the peaks at $m/z = 138.00$ (found: 138.14) and 230.12 (found: 230.06) were assigned to $[M_{m\text{-NA}}]^+$ and $[M_{BBHMP}]^+$, respectively. The peak at $m/z = 601.05$ (found: 601) is also compatible with $[M]^+$ for the molecular weight of the complex. The overall results of the mass spectrum indicate the presence of a complexation.

The UV–Vis spectra of the free BBHMP, *m*-NA and their Au(III) complex in $CHCl_3$ revealed absorption peaks between 200 and 500 nm (Fig. 3). The electronic spectra of the free ligands have two peaks in the UV region for each ligand [2, 12]. The first two peaks at 246 and 247 nm are assigned to $\pi \rightarrow \pi^*$ (phenyl rings) transitions [31], and the second two peaks at 353 and 358 nm are ascribed to $n \rightarrow \pi^*$ because of the long pair electrons of the nitrogen and oxygen atoms [32]. In the UV–Vis spectrum of the complex, these transitions are shifted to higher values. The complex has

Fig. 2 The mass spectra of the Au(III) complex



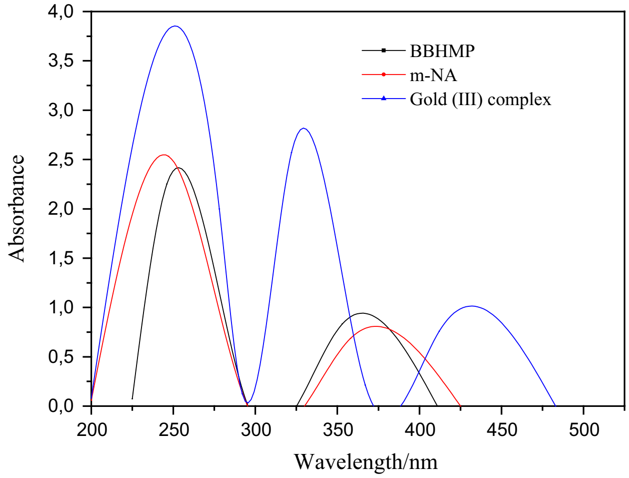


Fig. 3 UV–Vis spectra of the BBHMP, *m*-NA, and the Au(III) complex

three absorption peaks at 412, 322, and 298 nm which are assigned to $^1A_{1g} \rightarrow {}^1A_{2g}$, $^1A_{1g} \rightarrow {}^1B_{1g}$, and $^1A_{1g} \rightarrow {}^1E_g$ transitions, respectively. The peak at 298 nm, corresponding to the transitional charge transfer from the ligand to the Au(III) ion. These transitions and assignments show that the complex is a low spin square–planar complex [33–37].

In the FT-IR spectrum of *m*-NA [12, 32, 38, 39], the peak at 3400 cm^{-1} due to $\nu(NH_2)$ stretching vibration was obtained at 3434 cm^{-1} in the complex. The NH_2 stretching peak appears to shift to the higher frequencies in the FT-IR spectrum of complex. The reason for this is that the nitrogen atom gives its electrons to Au^{3+} and makes a coordinated covalent bond. The complex exhibits a broad peak at 3400 cm^{-1}, which was attributed to ν(OH) stretching frequencies of coordinated water molecules [40]. The stretching vibrations at 1581, 1247, 2954, and 1347 cm^{-1} due to ν(C=C), ν(C–N), $\nu(CH_2)$, and $\nu(Ar–NO_2)$, respectively, shift to higher wave numbers compared with the free ligands. The oxygen atoms in the BBHMP structure that bond with Au^{3+} attract electrons of neighboring atoms [2]. This causes the bonds between neighboring atoms to be more polar. Thus, functional groups such as ν(C=C), ν(C–N), $\nu(–CH_2)$, and $\nu(–NO_2)$ occur at higher frequencies. The occurrence of new peaks at 553 and 644 cm^{-1} in the spectrum of the complex which may be assigned to ν(Au–N) and ν(Au–O) supports coordination with the nitrogen and oxygen atoms in the ν(C–N) and ν(C–O) groups, respectively [41].

^{1}H NMR (300 MHz, $CDCl_3$ for BBHMP, *m*-NA and complex) spectra of free ligands and their Au(III) complex were recorded to verify the binding of the BBHMP and *m*-NA molecules to the Au^{3+} ion. For BBHMP, the characteristic ^{1}H NMR signal at $\delta = 7.13$ ppm (s, 1H, C_2–H and s, 1H, C_6–H) is assigned to aromatic ring protons. The peaks at 4.20 and 5.64 ppm are attributed to aliphatic $–CH_2$ (s, 1H, C_7–H and s, 1H, C_8–H) and –OH (s, 1H, OH) protons, respectively [2]. The ^{1}H NMR spectrum of *m*-NA has proton signals at 7.52 (s, 1H, C_2–H), 8.54 (d, 1H, C_4–H), 7.67 (m, 1H, C_5–H), 8.43 (d, 1H, C_6–H), and 4.43 ppm (s, 2H, NH_2) [12]. In the ^{1}H NMR spectra of the Au(III) complex, the peak observed at 2.41 ppm may be attributed to coordinated water molecules of the complex. The position of the CH_2 molecules bound to oxygen atoms in the complex is shifted in comparison with that of the free ligand, suggesting deshielding of the protons of the CH_2 molecules due to its coordination to Au^{3+} ion through the ligand oxygen atoms. Additionally, OH peaks are missing and little shifts are seen in other values.

Table 1 Thermal analysis data of the Au(III) complex

Compound	Steps	*Tb*–*Tc*/°C	Weight loss/%	Assignments
Au(III) complex	1st	25–200	6.7	2 H_2O
	2nd	200–600	45.3	Organic comp.
	3rd	600–900	48.0	Au metal

The ^{13}C NMR (75 MHz, $CDCl_3$) spectrum of BBHMP shows signals at $\delta = 114.8$ (C_1), 127.6 (C_2 and C_6), 133.3 (C_3 and C_5), 156.4 (C_4), and 63.2 (C_7 and C_8) ppm [2]. For *m*-NA, characteristic ^{13}C NMR signals at 149.3, 121.1, 130.4, 113.9, 148.7, and 108.1 ppm are assigned to C_1, C_2, C_3, C_4, C_5, and C_6, respectively [12]. The ^{13}C NMR data showed that the complex was shifted in comparison to BBHMP and *m*-NA.

The decomposition temperature, percentage mass loss, and assignments of the complex are presented in Table 1. In the thermal decomposition of the complex, three decomposition steps were observed in the temperature range of 25–900 °C. The first decomposition in the range of 25–200 °C corresponds to the loss of two water molecules with a mass loss of 6.7%, the second decomposition in the range 200–600 °C is the decomposition of the organic component (45.3%). The remaining compound over 600 °C is Au (48%). These results show that the complex is in good agreement with the proposed structure.

Composition and thermodynamic parameters of the complex

One maximum for the complex was obtained at different two temperatures from perpetual change graphs using Job's method (Fig. 4) [42, 43]. According to Job's method, the composition of 1:1 complexes in solution is 0.5. But in our study, the composition of complex is calculated by extrapolation as $[Au^{3+}]/([Au^{3+}] + [BBHMP + m\text{-}NA]) \sim 0.471$ at 25 °C and ~ 0.492 at 40 °C, respectively. The potential experimental error may have been caused by laboratory conditions, environmental conditions or UV–Vis device. These

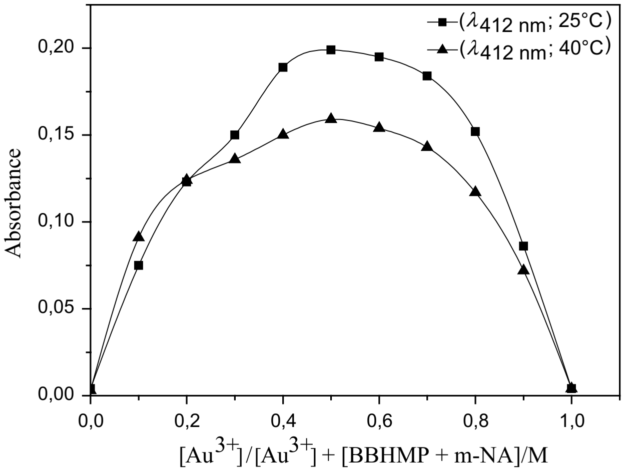


Fig. 4 The composition of the Au(III) complex

results show that one BBHMP molecule and one *m*-NA molecule react with one molecule of Au^{3+} ion.

To obtain the equilibrium constant with the help of solutions that give equal absorption, dilution curves were drawn at the same temperatures. Molar composition (BBHMP + *m*-NA) and Au(III) solutions prepared with equal concentrations at 25 and 40 °C were diluted at regular intervals and their absorbances were measured at $\lambda = 412$ nm. The obtained absorbance values of the solutions at different concentrations are shown in Table 2 and Fig. 5. The plots of absorbance (nm) against $[Au^{3+}] = [BBHMP + m\text{-}NA]$ are linear.

The equations and the correlation coefficients for 25 and 40 °C from Fig. 5 were found as $y = 0.0744\,x$; $R^2 = 0.9981$ and $y = 0.0762\,x$; $R^2 = 0.9994$, respectively. In the 1:1 stoichiometric reactions, the equilibrium constant (*K*) formula [44] is

$$K = \frac{(a_1 - x)(b_1 - x)}{x} = \frac{(a_2 - x)(b_2 - x)}{x}, \tag{1}$$

where a_1 is the concentration of metal ion ($[Au^{3+}]$) which is obtained from $[Au^{3+}]/([Au^{3+}] + [BBHMP + m\text{-}NA])$, b_1 is the concentration of the ligand ($[BBHMP + m\text{-}NA]$) which is calculated from $([Au^{3+}] + [BBHMP + m\text{-}NA]) = 1 \times 10^{-3}$ M and *x* shows the concentration of complex formed. The values of *K* for the reactions of the mixed ligand with Au(III) are calculated from the above equations.

Table 2 The values of dilution curves of the Au(III) complex

$[Au^{3+}] = [BBHMP + m\text{-}NA] \times 10^{-3}$	ABS	
	λ_{412}, 25 °C	λ_{412}, 40 °C
1.66	0.070	0.075
2.66	0.140	0.147
3.66	0.222	0.228
4.66	0.300	0.307
5.66	0.375	0.382

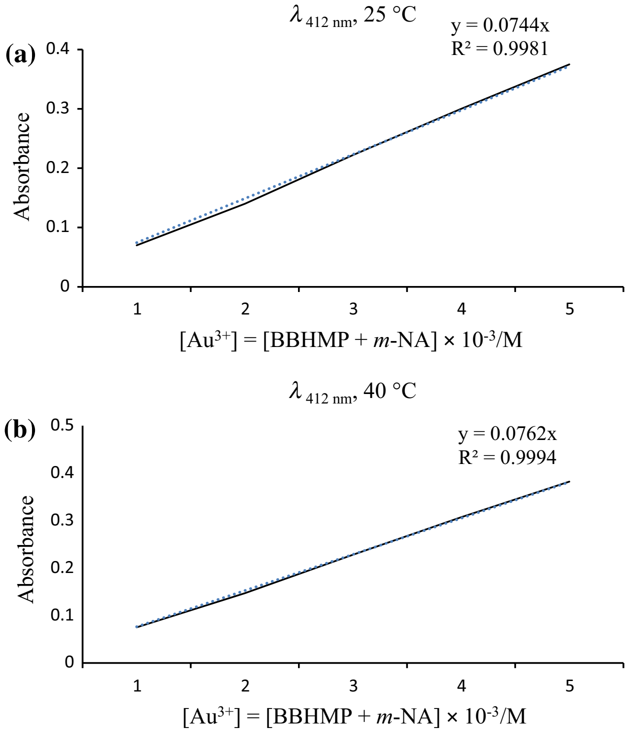


Fig. 5 The dilution curves for $\lambda_{412\,nm}$, 25 °C (**a**) and $\lambda_{412\,nm}$, 40 °C (**b**)

According to the thermodynamic stability constants, the values of the changes in Δ*G*, Δ*H*, and Δ*S* were determined using the following equations:

$$\Delta G = -RT \ln K, \tag{2}$$

$$\ln \frac{K_2}{K_1} = \frac{\Delta H}{R}\left[\frac{T_2 - T_1}{T_1 \cdot T_2}\right], \tag{3}$$

$$\Delta S = \frac{\Delta H - \Delta G}{T}, \tag{4}$$

where *R* is the gas constant (8.314 J K^{-1} mol^{-1}), *T* is temperature (K), and *K* is the equilibrium constant. Thermodynamic parameters for the reaction of the mixed ligand with Au(III) were recorded using the above equations (Table 3) [25, 45]. According to Table 3, *K* and Δ*G* values were calculated as 3.11×10^{-4}, 3.10×10^{-4}, and 20.019, 21.034 kJ mol^{-1} at 298.16 and 313.16 K, respectively. Δ*H* and Δ*S* values of the complex are −0.167 kJ mol^{-1}, −66.58 and −66.63 J mol^{-1} K^{-1} at same temperatures, respectively.

Table 3 The thermodynamic parameters of the Au(III) complex

T/K	K	ΔG/kJ mol^{-1}	ΔH/kJ mol^{-1}	ΔS/J mol^{-1} K^{-1}
298.16	3.11×10^{-4}	20.019	−0.167	−66.58
313.16	3.10×10^{-4}	21.034	−0.167	−66.63

Conclusion

In the present paper, a gold(III) complex was synthesized in the presence of 4-bromo-2,6-bis(hydroxymethyl)phenol (BBHMP) and *m*-nitroaniline (*m*-NA) and characterized with elemental analysis, magnetic susceptibility, conductivity, mass, UV–Vis, FT-IR, ^{1}H NMR, ^{13}C NMR, and TG–DTA. The results of the physical, spectral, and thermal analysis showed that the reaction of BBHMP and *m*-NA with Au(III) is a complex reaction. One molecule of BBHMP and one molecule of *m*-NA react with one molecule of Au^{3+} ion. The formula of the complex is predicted to be in the form of [Au(BBHMP)(*m*-NA)]·2H$_2$O. Considering the thermodynamic parameters, the negative value of Δ*S* and the positive value of Δ*G* mean that the process is non-spontaneous and the negative value of Δ*H* shows that a certain amount of energy is released during the reaction. As a result, the enthalpy contribution changes slightly with temperature and the reaction is entropy controlled.

Experimental

Physical measurements

Elemental analysis for C, H, O, and N were carried out with a Costech ECS 4010 CHNSO elemental analyzer and an ICP-MS 7700 X (Agilent) analyzer was used for Au metal. The magnetic moment measurements were recorded using a MK-1 Sherwood scientific magnetic susceptibility balance. Conductivity experiments were completed in DMF with an Inolab Thermal 740P. pH measurements were made with a calibrated Metrohm 654 digital pH meter with a Sensorex combination pH glass electrode assembly. Mass spectra were investigated on an AB-SCIEX Triple TOF 4600 System. UV–Vis spectra were determined using a Shimadzu UV-1700 Pharma spectrophotometer in the wavelength range of 200–800 nm. FT-IR spectra were measured in transmission mode using a Shimadzu FT-IR-470 spectrometer in the wavenumber range of 4000–400 cm^{-1}. KBr was used as the matrix material for pellets. ^{1}H NMR and ^{13}C NMR spectra were determined in CDCl$_3$ on a Bruker DPX-400 spectrometer. TG/DTA curves were obtained with a Seiko Exstar TG/DTA 6200 thermal analyzer in the temperature range of 25–1200 °C in static atmosphere of dry air with a heating rate of 10 °C min^{-1}. Platinum crucibles with a sample size of 5–10 mg were used in the analysis.

Materials

4-Bromophenol, formaldehyde, acetic acid, diethyl ether, *m*-nitroaniline, and Na[AuCl$_4$]·2H$_2$O were obtained from Sigma-Aldrich. NaOH, CHCl$_3$, and DMF were obtained from Merck and used as supplied.

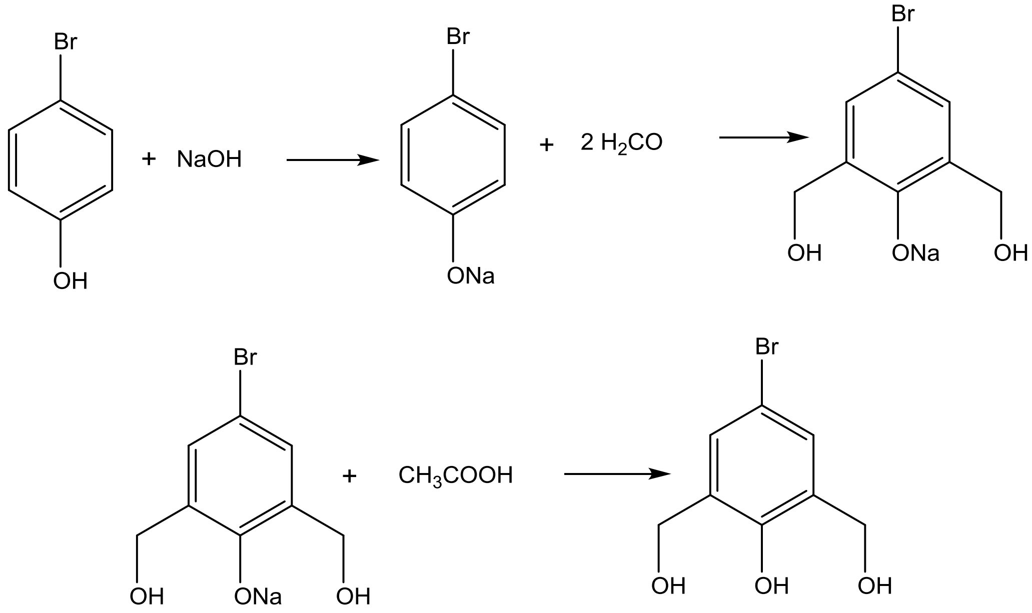
4-Bromo-2,6-bis(hydroxymethyl)phenol (1) The reaction mixture, including an aquatic solution (15 cm^3) of 0.04 g NaOH (1 mmol) and 0.173 g 4-bromophenol (1 mmol) was stirred for 30 min. Then 30.15 cm^3 formaldehyde (7%, 2 mmol) was added and stirred for 1 week. At the end of 1 week, the product was dissolved in water and concentrated acetic acid was added dropwise to pH 5 on the magnetic stirrer. The obtained white solid precipitate was washed with water and diethyl ether and dried in vacuum [2] (Scheme 1). White solid; yield: 0.198 g (85%); m.p.: 152 °C; NMR spectra agree with the ones described in Ref. [2].

4-Bromo-2,6-bis(hydroxymethyl)phenol *m*-nitroaniline gold(III) dihydrate ([Au(BBHMP)(*m*-NA)]·2H$_2$O, 2, C$_{14}$H$_{16}$AuBrN$_2$O$_7$) 0233 g BBHMP (1 mmol), 0.12 g NaOH (3 mmol), and 0.126 g *m*-NA (1 mmol) were dissolved in 25 cm^3 ethanol. Then 0.398 g Na[AuCl$_4$]·2H$_2$O (1 mmol) was added and refluxed for 5 h at 75–80 °C. A solid, dark yellow product was obtained, washed with water, and dried. The complex was soluble in certain organic solvents such as CHCl$_3$ and DMF. Dark yellow solid; yield: 0.450 g (75%); m.p.: 141 °C; UV–Vis: $\lambda = 298$, 322, 412 nm; FT-IR (KBr): = 3400 (OH), 3434 (NH$_2$), 1581 (C=C), 1247 (C–N), 2954 (CH$_2$), 1347 (Ar–NO$_2$), 553 (AuN), 644 (AuO) cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$): $\delta = 6.77$ (s, 1H), 7.42 (s, 1H), 7.32 (d, 1H), 6.90 (m, 1H), 6.77 (s, 1H), 7.24 (d, 1H), 4.36 (s, 2H), 2.41 (s, 2H) ppm; ^{13}C NMR (75 MHz, CDCl$_3$): $\delta = 115.9$ and 152.3 (C$_1$), 131.1 and 122.7 (C$_2$), 135.7 and 132.8 (C$_3$), 159.7 and 114.2 (C$_4$), 135.7 and 150.1 (C$_5$), 131.1 and 107.5 (C$_6$), 69.1 (C$_7$), 69.1 (C$_7$) ppm; ESI–MS: $m/z = 138.00$ (found: 138.14) [M$_{\text{m-NA}}$]$^+$, 230.12 (found: 230.06) [M$_{\text{BBHMP}}$]$^+$, 601.05 (found: 601) [M]$^+$; magnetic moment: diamagnetic; molar conductivity: 49.6 Ω^{-1} cm^2 mol^{-1}). Accordingly, the reaction as shown in Scheme 2 was determined.

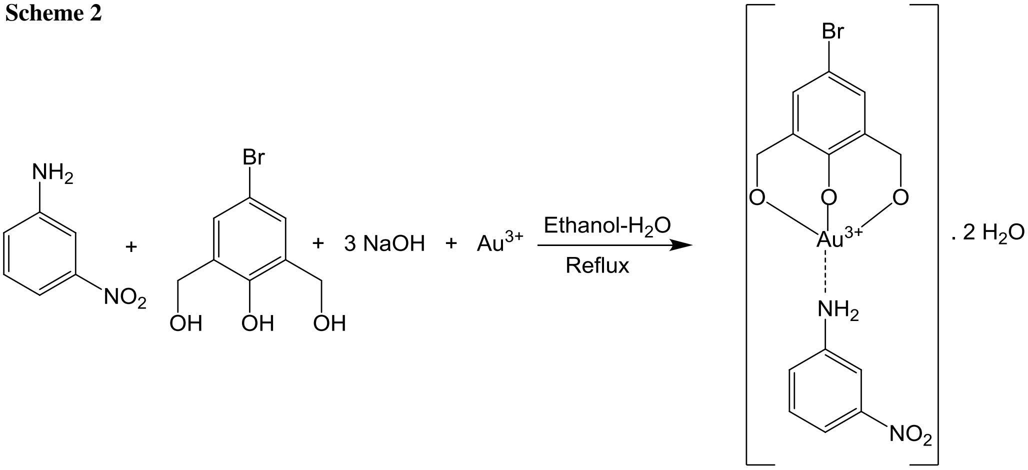
Composition of the complex

For the investigation of the composition and equilibrium constant of the complex, the methods of Job [42, 43] and Turner & Anderson [44] were used. Various solutions were prepared with total concentrations of 1×10^{-3} M, containing the molar composition (BBHMP + *m*-NA)

Scheme 1



Scheme 2



and Au(III) at room temperature and 40 °C. [Au^{3+}]/([Au^{3+}] + [BBHMP + *m*-NA]) values were calculated and the absorbances were measured at $\lambda = 412$ nm. Then, the perpetual change graphs for absorbance measurements against [Au^{3+}]/([Au^{3+}] + [BBHMP + *m*-NA]) were plotted. Composition of the complex was found from the maximum values of these graphs.

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