A complex of gold(1) benzenethiolate with isocyanide: synthesis and crystal and molecular structures

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2,6-Dimethylphenyl isocyanide forms complexes with gold(1) chloride (complex 4) and gold(1) benzenethiolate (complex 5) but forms no stable complexes with gold alkanethiolates. A reaction of complex 5 with tetramethylthiuram disulfide yields gold(1) dimethyldithiocarbamate. Stable gold(11) derivatives cannot be obtained in such a way. Shortened intermolecular Au...Au contacts in complexes 4 and 5 (X-ray diffraction) suggest the presence of "aurophilic" interactions in them.

Key words: 2,6-dimethylphenyl isocyanide, complex of gold benzenethiolate with isocyanide, complex of gold chloride with isocyanide, gold benzenethiolate, gold decanethiolate, gold 2-methylpropane-2-thiolate, gold dimethyldithiocarbamate, "aurophilic" interactions.

Among isonitrile complexes of gold(1), thiolate complexes of the type X-Au \cdot CN-R' (X = RS (1)) still remain poorly studied. Structural data for these complexes are scarce in the literature. At the same time, isocyanide complexes of gold thiolates are promising for the preparation of calamitic (containing rodlike fragments in molecules) liquid crystals, by analogy with isocyanide complexes of polyfluorophenyl derivatives of gold.¹

Some complexes of substituted gold benzenethiolates with isocyanides are known to be stable^{2,3} and so are similar complexes of pyridine-2-thiol and quinoline-2-thiol and binuclear complexes of 1,3,4-thiadiazole-2,5-dithiol.³ However, complexes of unsubstituted gold benzenethiolate with alkyl isocyanides of the type PhSAu · CN- C_nH_{2n+1} are unstable. These complexes tend to decompose easily, giving polymeric gold benzenethiolate through the loss of the isocyanide ligand.⁴ At the same time, gold(1) chloride is known to form the complexes RNCAuCl with both aryl and alkyl isocyanides.⁵⁻⁷

These data suggest that the stability of complexes of the type **1** depends, at least, on two factors: the electronegativity of the acido ligand (X) and the π -withdrawing properties of the isocyanide ligand.

We studied reactions of gold thiolates with 2,6-dimethylphenyl isocyanide* (2). It turned out that this ligand forms a complex with gold benzenethiolate but its complexes with gold alkanethiolates are unstable. At the same time, it yields complexes of the formula RNCAuCl. Thus,

* The presence of two methyl groups in the ligand should make the complexes more soluble.

here we compared the tendencies of various acido ligands to form isocyanide complexes with gold.

Results and Discussion

Earlier,⁸ a complex of gold chloride has been obtained from isocyanide 2 and the arsine complex $Ph_3AsAuCl$. We found that the metathesis reaction of isocyanide 2 with a tetrahydrothiophene complex of gold(1) chloride (3) is a more convenient route to gold chloride complex 4 (Scheme 1).



It should be noted that the reverse transformation $4\rightarrow 3$ did not take place even in the presence of a twofold molar excess of tetrahydrothiophene.

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Chloride complex 4 reacts with lithium benzenethiolate to give isonitrile complex 5 (Scheme 2). This is a colorless crystalline solid (m.p. 117-119 °C, without decomp.) soluble in organic solvents.

Scheme 2



Alternatively, isonitrile complex 5 can be obtained by the action of a solution of ligand 2 in THF on polymeric gold benzenethiolate 6. The reaction is accompanied by dissolution of compound 6 and the yield of complex 5 is high (Scheme 3). This method of the synthesis of complex 5 is more convenient than the aforementioned one.

Scheme 3



The stability of complex 5, in contrast to unstable complexes of gold benzenethiolate with alkyl isocyanides (see above), is probably due to the stronger π -withdrawing properties of ligand 2 compared to alkyl isocyanides, which stabilizes the bond of this ligand with a post-transition metal, which is gold.

Our attempted synthesis of complexes of ligand 2 with gold alkanethiolates $(C_{10}H_{21}SAu)_x$ (7) and $(Bu^tSAu)_x$ (8), which are analogs of compound 6, was unsuccessful. Polymeric gold thiolate 7 or 8 remained inert in these reactions, regardless of the synthetic pathway. As mentioned above, the difference between compound 6 and compounds 7 and 8 can be associated with the weaker acid character of the ligands $C_{10}H_{21}S^-$ and Bu^tS^- compared to PhS⁻.

It is known that the acidity of benzenethiol is 4-5 orders of magnitude higher than the acidity of alkanethiols.⁹

Another factor that can be crucial for the above difference is the low solubilities of thiolates 7 and 8 in THF under the conditions of the synthesis. This assumption is confirmed by X-ray powder diffraction data for the gold thiolates under discussion. Indeed, the diffraction patterns of gold alkanethiolates 7 and 8 both prior to and after the treatment with a solution of ligand 2 show intense (though broadened) reflections from the crystalline phases. At the same time, freshly prepared gold benzenethiolate 6 is virtually X-ray amorphous. In addition, the gold atoms in compounds 7 and 8 are shielded by bulky alkyl ligands, which makes gold less accessible for isonitrile molecules.

It should be taken into account that the bond between ligand 2 and the Au^I atom in complex 5 is sufficiently labile. For instance, complex 5 undergoes partial dissociation in THF, forming a small amount of insoluble benzenethiolate 6. However, the precipitate dissolves upon addition of an excess of isocyanide 2. It becomes clear therefrom why isocyanide 2, which forms a complex with X-ray amorphous benzenethiolate 6, cannot break the crystal structures of alkanethiolates 7 and 8 and cannot form complexes with these gold derivatives.

We studied a reaction of complex 5 with tetramethylthiuram disulfide (9). Earlier,¹⁰ we have found that tetraalkylthiuram disulfides add to organic derivatives of gold(1), yielding gold(11) dimethyldithiocarbamates. Complex 5 also reacts with compound 9 to give an intensely colored red solution characteristic of gold(11) derivatives. However, we failed to isolate gold(111) derivatives in the individual state under these conditions. When stored, the solution gradually produces a bright yellow precipitate of gold(1) dimethyldithiocarbamate (10) (Scheme 4).

Scheme 4



We determined the crystal and molecular structure of complex **5** and, for comparison, the structure of gold chloride complex with isonitrile **4**.

X-ray diffraction study of complexes **4** and **5** clearly revealed shortened intermolecular Au···Au contacts giving rise to dimers (Table 1; Figs 1, 2). Both structures contain two crystallographically independent molecules; dimers consists of nonidentical molecules. Previous X-ray diffraction data for complex **4** were ambiguous in whether its structure is built from discrete dimers¹¹ or forms a united polynuclear complex.⁹

Shortened intermolecular Au···Au contacts, which are intermediate between nonbonding metal—metal distances (>4 Å) and the shortest interatomic spacing in metallic gold (2.884 Å), are characteristic of gold(1) complexes. Such contacts are attributed to additional "aurophilic" interactions. The shortest contacts have been found in gold complexes with tertiary phosphines (2.50–3.24 Å).¹² In complexes **4** and **5** with the isocyanide ligand, the distance between adjacent Au atoms is longer (see Table 1); therefore, the corresponding Au···Au interactions are weaker.

Nevertheless, the dimer of benzenethiol complex **5** shows short nonbonding contacts between the S atom of benzenethiolate and the C atom of the isocyanide ligand (3.69 Å). The coordination of the isocyanide C atom is nonlinear (see Table 1).

At the same time, in chloride complex **4** with the crossed coordination "dumbbells" C—Au—Cl, the coordination of the isocyanide C atom is nearly linear. The Au atoms in the dimer approach each other (3.54 Å).

Crystal structure 5 is similar to that of the pyridine-2thiol complex Au(2-SPy)(2,6-Me₂C₆H₃NC) (see Ref. 3). Although the space groups of these complexes are different, their molecular structures and packing are very much the same. In both complexes, the aromatic rings are virtually coplanar. The angles between the mean-square planes of the rings in structure 5 are about 6° or 7° for independent molecules; in Au(2-SPy)(2,6-Me₂C₆H₃NC), these angles are approximately 10°. Because of this, the mole-

Table 1. Selected structural characteristics ofcomplexes 4 and 5

Parameter	4	5
Bond	d/Å	
Au–C Au–S	1.88, 1.96 —	1.91, 2.01 2.26, 2.27
Angle	ω/deg	
C—Au—X	177, 178 (X = Cl)	177, 177
N—C—Au	169, 176 (X = S)	166, 171
Distance	<i>e*/</i> Å	
NC…S	_	3.69
Au…Au	3.54	3.65

* *e* is the shortest distance.



Fig. 1. Dimers in the crystal structure **4**. The Au(1)—Au(2) distance is 3.54 Å.



Fig. 2. Dimers in the crystal structure **5**. The Au(1)-Au(2) distance is 3.64 Å.

cules in both crystals form dimers with parallel head-totail arrangement of their planar fragments. The dimer in $Au(2-SPy)(2,6-Me_2C_6H_3NC)$ is centrosymmetric, while that in complex 5 is pseudocentrosymmetric. The meansquare deviation of the corresponding non-hydrogen atoms for the most closely spaced independent molecules (the overlay procedure in the Mercury program¹³) is 0.19 Å. Dimers are united into a chain with alternating Au--Au distances inside a dimer and between dimers. In the complex Au(2-SPy)(2,6-Me₂C₆H₃NC), the alternating distances are 3.59 and 4.04 Å. In structure 5, these distances are 3.65 and 3.88 Å. These values substantially exceed twice the van der Waals radius of Au (1.66 Å according to Bondi¹⁴). Otherwise, these structures should be regarded as topologically identical. For the closest possible overlay of the first (Au(1)) molecule of structure 5 with a Au $(2-SPy)(2,6-Me_2C_6H_3NC)$ molecule (mean-square deviation is 0.15 Å), the corresponding deviation for the molecule nearest to the second (Au(2)) molecule is only 0.91 Å.

To sum up, we developed convenient methods for the synthesis of complexes of gold(1) chloride and gold(1) benzenethiolate with 2,6-dimethylphenyl isocyanide. We demonstrated that gold alkanethiolates do not form stable complexes with isocyanide. A reaction of an isocyanide complex of gold benzenethiolate with tetramethylthiuram disulfide yields unstable gold(III) complexes. X-ray diffraction for the gold complexes obtained reveals the presence of nonbonding intermolecular Au $\cdot\cdot\cdot$ Au contacts (3.5–4.0 Å).

Experimental

2,6-Dimethylformanilide. A mixture of 2,6-dimethylaniline (10.00 g, 0.083 mol) and 93% formic acid (9.30 g, 0.188 mol) was refluxed in toluene (30 mL) with a Dean–Stark trap until water ceased to evolve (2.5 h). Then the solvent was removed *in vacuo* and the residue was recrystallized from aqueous ethanol. The yield of 2,6-dimethylformanilide was 6.8 g (60%), colorless crystals, m.p. 163–164 °C (*cf.* Ref. 15: m.p. 164–165 °C).

2,6-Dimethylphenyl isocyanide (2). A solution of diphosgene (0.5 mL) in CH₂Cl₂ (4 mL) was added dropwise to a boiling suspension of 2,6-dimethylformanilide (1.00 g, 6.7 mmol) in a mixture of anhydrous triethylamine (4.7 mL) and CH₂Cl₂ (17 mL). The reaction mixture was refluxed with stirring for 15 min and then washed with 10% aqueous Na₂CO₃. The organic layer was separated, dried with anhydrous Na₂SO₄, and concentrated *in vacuo*. The residue was chromatographed on silica gel with chloroform—light petroleum (1 : 1) as the eluent. The yield of isocyanide **2** was 0.75 g (85%), colorless powder, m.p. 72.5–74 °C (*cf.* Ref. 16: m.p. 74–75 °C). ¹H NMR (CDCl₃), δ : 2.41 (s, 6 H, 2 CH₃); 7.15 (m, 3 H, C₆H₄). IR (Nujol): 2130 cm⁻¹ (v_{NC}).

Complex of gold(1) chloride with tetrahydrothiophene (3). The synthesis was carried out in a shaded room. A solution of bis(2-hydroxyethyl) sulfide (1.2 mL, 11.6 mmol) in ethanol (7 mL) was added at 0 °C to a stirred solution of HAuCl₄ · $3H_2O$ (2.20 g, 2.8 mmol) in water (10 mL). The reaction mixture was stirred for 10 min. Then a solution of tetrahydrothiophene (0.56 mL, 0.28 g, 6.4 mmol) in ethanol (6 mL) was added and stirring was continued for an additional 10 min. The precipitate of complex **3** that formed was filtered off, washed with water (2×10 mL) to a neutral reaction and then successively with ethanol (10 mL), acetone (10 mL), and diethyl ether (10 mL), and dried in air. The yield of complex **3** was 1.50 g (84%), beige powder (decomp. > 140 °C). Found (%): C, 14.98; H, 2.50; S, 9.19. C₄H₈ClSAu. Calculated (%): C, 14.99; H, 2.52; S, 10.00.

Gold benzenethiolate (6). A solution of benzenethiol (2.26 g, 20.5 mmol) in methanol (8 mL) was added for 5 min to a stirred solution of HAuCl₄· 3H₂O (2.10 g, 5.33 mmol) in a mixture of water (12 mL) and methanol (61 mL). The reaction mixture was stirred for 10 min. The precipitate of gold benzenethiolate that formed was filtered off, washed with methanol (60 mL), acetone (60 mL), and diethyl ether (60 mL), and dried in air. The yield of compound **6** was 1.60 g (98%), yellowish powder (decomp. > >178 °C). Found (%): C, 23.71; H, 1.50. C₆H₅SAu. Calculated (%): C, 23.54; H, 1.65.

Complex of gold chloride with 2,6-dimethylphenyl isocyanide (4). A solution of 2,6-dimethylphenyl isocyanide (2) (0.131 g, 1 mmol) in THF (20 mL) was added to a stirred solution of a complex of gold(1) chloride with tetrahydrothiophene (3) (0.320 g, 1 mmol) in THF (30 mL). The reaction mixture was stirred for 10 min and concentrated *in vacuo* to a volume of \sim 10 mL. The solution was filtered to remove traces of metallic gold. Complex **4** was precipitated with light petroleum (20 mL). The yield was 0.250 g (70%), colorless crystals, m.p. 142–144 °C. Found (%): C, 29.55; H, 2.35; N, 3.72. C₉H₉AuClN. Calculated (%): C, 29.73; H, 2.50; N, 3.85. ¹H NMR (CDCl₃), δ : 2.43 (s, 6 H, 2 CH₃); 7.18 (m, 2 H, C₆H₃); 7.34 (m, 1 H, C₆H₃). IR (Nujol): 2228 cm⁻¹ (v_{NC}).

Complex of gold benzenethiolate with 2,6-dimethylphenyl isocyanide (5). A. Synthesis from a gold chloride complex with tetrahydrothiophene. A Schlenk apparatus filled with argon was charged with a solution of benzenethiol (0.086 g, 0.78 mmol) in THF (8 mL) and cooled to -40 °C. A 1.93 *M* solution of *n*-butyllithium (0.4 mL, 0.78 mmol) in hexane was added. The reaction mixture was stirred for 10 min. At -30 °C, complex **3** (0.250 g, 0.78 mmol) and, after 5 min, a solution of 2,6-dimethylphenyl isocyanide (2) (0.100 g, 0.78 mmol) in THF (20 mL) were added. The resulting mixture was stirred for 30 min without forced cooling. The precipitate that formed was filtered off and the filtrate was concentrated *in vacuo*. The residue was dissolved in a minimum amount of benzene and complex **5** was precipitated with an excess of light petroleum. The yield of complex **5** was 0.130 g (38%), m.p. 117–119 °C.

B. Synthesis from gold benzenethiolate. A solution of 2,6-dimethylphenyl isocyanide (2) (0.30 g, 2.29 mmol) in THF (10 mL) was added to a suspension of gold benzenethiolate (6) (0.50 g, 1.63 mmol) in THF (20 mL). The reaction mixture was stirred for 25 min and concentrated in vacuo to a volume of 10 mL. The resulting light yellow solution was carefully diluted with light petroleum (4 mL) and kept in a refrigerator for 3 h. Then light petroleum (4 mL) was added again and the solution was kept in a refrigerator for 16 h. Then another portion of light petroleum (4 mL) was added. After several hours, the precipitate that formed was filtered off, washed with light petroleum, and dried in air. The yield of complex 5 was 0.67 g (94%), yellowish crystals, m.p. 117-119 °C. Found (%): C, 41.42; H, 3.37; N, 3.22. C₁₅H₁₄NSAu. Calculated (%): C, 41.20; H, 3.23; N, 3.20. ¹H NMR (CDCl₃), δ: 2.45 (s, 6 H, 2 CH₃); 7.01 (1 H); 7.09–7.17 (m, 4 H, C₆H₄-S); 7.56–7.58 (2 H); 7.33 (m, 1 H). IR (Nujol): 2228 cm⁻¹ (v_{NC}).

Gold decanethiolate (7). Methanol (30 mL) and decanethiol (1.76 g, 10.1 mmol) were added to a stirred solution of HAuCl₄·3H₂O (1.00 g, 2.54 mmol) in water (5 mL). The reaction mixture was stirred for 10 min. The yellowish precipitate of gold decanethiolate that formed was filtered off, washed with acetone (10 mL) and diethyl ether (10 mL), and dried on the filter. The yield of compound 7 was 0.63 g (67%), yellowish powder (decomp. > 120 °C). Found (%): C, 32.23; H, 5.70. C₁₀H₂₁ClSAu. Calculated (%): C, 32.43; H, 5.72.

Gold 2-methylpropane-2-thiolate (8). Methanol (30 mL) and 2-methylpropane-2-thiol (0.92 g, 10.16 mmol) were added to a stirred solution of HAuCl₄· 3H₂O (1.00 g, 2.54 mmol) in water (5 mL). The reaction mixture was stirred for 1 h. The pinkish precipitate of gold 2-methylpropane-2-thiolate that formed was filtered off, washed with acetone (10 mL) and diethyl ether (10 mL), and dried on the filter. The yield of compound **8** was 0.67 g (92%), pinkish powder (decomp. > 216 °C). Found (%): C, 16.92; H, 3.13. C₄H₉SAu. Calculated (%): C, 16.79; H, 3.17.

Reaction of gold decanethiolate (7) with isocyanide 2. A solution of isocyanide (2) (0.262 g, 2 mmol) was added to a suspension of decanethiolate 7 (0.740 g, 2 mmol) in THF (10 mL). The mixture was stirred for 30 min. The precipitate that formed was filtered off, thoroughly washed with THF (in two portions of

5 and 10 mL), and dried in air to a constant weight. Decanethiolate 7 (0.706 g, 95%) was recovered. Its X-ray diffraction pattern was identical with that of the starting reagent. The filtrate was combined with the rinsing portions of THF and concentrated *in vacuo*. The residue was dissolved in diethyl ether; the resulting slightly turbid solution was filtered and concentrated *in vacuo*. The residue was dried to a constant weight. Isocyanide 2 (0.25 g, 95%) was recovered, m.p. 72–74 °C.

Reaction of gold 2-methylpropane-2-thiolate (8) with isocyanide 2 was carried out as described above for decanethiolate 7. The starting reagent 8 (93%) was recovered.

Reaction of complex 5 with tetramethylthiuram disulfide (9). Isocyanide **2** (0.01 g, 0.08 mmol) and a solution of compound **9** (0.12 g, 0.5 mmol) were added to a solution of complex **5** (0.219 g, 0.5 mmol) in THF (5 mL). The reaction mixture was stirred for 2 h. The precipitate of gold dimethyldithiocarbamate (**10**) that formed was filtered off and washed with THF. The filtrate was concentrated *in vacuo*. The product from the residue was extracted with THF and combined with the main crop of compound **10**. The yield of gold dimethyldithiocarbamate (**10**) was 0.122 g (77%). Found (%): C, 11.70; H, 1.91; N, 4.45. $C_{15}H_{14}NSAu$. Calculated (%): C, 11.36; H, 1.91; N, 4.42.

X-ray diffraction analysis. X-ray powder diffraction data for polycrystalline samples were collected at room temperature in the Bragg—Brentano geometries on a Rigaku DMAX 2500 diffractometer (Science Materials Department of the Moscow State University) equipped with a vertical goniometer and a rotating copper anode X-ray tube (generator operating at 50 kV, 250 mA, $\lambda = 1.5418$ Å (Cu-K α), diffracted beam monochromator, scintillation point detector, continuous scan with a step of 0.02°, $4^{\circ} < 20 < 64^{\circ}$, scan rate 5 deg min⁻¹).

Single crystals of the complexes were obtained by slow precipitation with light petroleum from their solutions in THF. X-ray diffraction study of complexes **4** and **5** was carried out at the Crystallographic Center of the A. N. Nesmeyanov Institute of Organoelement Compounds (Russian Academy of Sciences) on an Enraf-Nonius CAD 4 diffractometer (graphite monochromator, λ (Mo-K α) = 0.71073 Å, $\theta/2\theta$ scan mode) at room temperature. The structures were solved by the direct methods with the SHELXTL program package.¹⁷ The hydrogen atoms were located from difference electron-density maps. The structures were refined anisotropically (isotropically for H atoms) on F^2 by the full-matrix least-squares method. The structural parameters of complexes **4** and **5** have been deposited with the Cambridge Crystallographic Data Center (CCDC Nos. 739 392 and 739 391, respectively).

Complex 4. Monoclinic crystals, space group $P2_1/n$, Z = 8, a = 10.651(2) Å, b = 17.289(4) Å, c = 11.860(2) Å, $\beta = 97.51(3)^\circ$, V = 2165.1(8) Å³, $\theta_{\text{max}} = 27.96^\circ$. The number of independent reflections is 5078; the number of reflections with $F > 4\sigma(F)$ is 2658, $R_1 = 0.0419$.

Complex 5. Triclinic crystals, space group *P*1, *Z* = 2, a = 7.381(3) Å, b = 8.274(3) Å, c = 12.889(3) Å, $\alpha = 100.35(2)^{\circ}$, $\beta = 92.14(2)^\circ$, $\gamma = 112.56(3)^\circ$, V = 710.2(4) Å³, $\theta_{max} = 29.96^\circ$. The number of independent reflections is 4198; the number of reflections with $F > 4\sigma(F)$ is 3173, $R_1 = 0.0283$.

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