poorly resolved singlet are recorded; the latter could be a result of the overlap of the spectra of acyl radicals $^{\bullet}C_{60}C(O)CF(CF_3)_2$ and $^{\bullet}C_{60}C(O)CF_2CF_3$.

Thus, fullerene instead of thermally unstable nitroso compounds[9] can be used as a radical trap for studying the thermolysis and photolysis of fluoroorganic compounds

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Reaction of 1-chloro-2-phenylethane-2,2-dithiol with divalent metal (Cu, Hg, Pb, Fe, Co, and Ni) salts

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Reactions of 1-chloro-2-phenylethane-2,2-dithiol with lead(II) acetate and mercury(II) chloride lead to 1-chloro-2-phenylethane-2,2-dithiolates of lead and mercury. The reactions with copper(II) chloride, iron(II) sulfate hydrate, cobalt (II) chloride hydrate, and nickel(II) acetate give metal-containing compounds $C_{16}H_{18}S_4M$. The ^1H NMR, IR, and ESR spectra of the obtained compounds were recorded; their specific dark electroconductivities and activation energies of dark conductivity were determined, and their film-forming ability was studied.

Key words: *gem*-dithiols, metallic derivatives; specific dark electroconductivity, organic semiconductors.

Metallic derivatives of 1-halo-2-organylethane-2,2-dithiols [$R{-}C(SH)_2{-}CH_2Cl$, R = Me, Ph, 5-chlorothienyl][1] have not yet been described. We studied the interaction of 1-chloro-2-phenylethane-2,2-dithiol (**1**) with salts of divalent metals (Cu, Hg, Pb, Fe, Co, Ni).

Dithiol **1** reacts with either lead(II) acetate or mercury(II) chloride in methanol to form 1-chloro-2-phenylethane-2,2-dithiolates of lead (**2b**) or mercury (**2a**):

$$\mathrm{Ph{-}C(SH)_2{-}CH_2Cl + MX_2 \longrightarrow Ph{-}C(S_2M){-}CH_2Cl + 2\,HX}$$

1 **2a, b**

M = Hg (**2a**); Pb (**2b**)

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The structures of compounds **2** may be confirmed by the reaction of lead 2-phenyl-1-chloro-2,2-dithiolate (**2b**) with elementary sulfur giving exclusively bis(2-phenyl-2-epidithioethyl) sulfide.[2]

Under similar conditions, in the reactions with copper(II) chloride, iron(II) sulfate hydrate, cobalt(II) chloride hydrate, and nickel (II) acetate, dithiol **1** unexpectedly gives compounds $C_{16}H_{18}S_4M$ (**3a—d**) [M = Cu (**a**), Fe (**b**), Co (**c**), Ni (**d**)], whose structures are being studied at present.

Compounds **3a—d** are paramagnetic black powders that are very soluble in chloroform and acetone. Their solutions form high-quality films on glass and quartz supports that exhibit the properties of typical organic semiconductors. The specific dark electric conductivities of compounds **3a—d** at 20 °C are $6.7 \cdot 10^{-10}$, $2.8 \cdot 10^{-13}$, $2.0 \cdot 10^{-11}$, and $4.3 \cdot 10^{-9}$ Ohm cm^{-1}, and the activation energies of dark conductivity are 0.76, 1.94, 2.40, and 0.95 eV, respectively.

It should be noted that dithiol **1** does not react with Zn^{II}, Mn^{II}, or lanthanide salts under the conditions studied.

Experimental

^{1}H NMR spectra were recorded on a Jeol FX-90Q spectrometer (90 MHz) in $CDCl_3$. HMDS was used as the internal standard. IR spectra were obtained on a Specord IR-75 spectrometer (resolution 1 cm^{-1}) for Nujol mulls and in KBr tablets. Spectra were recorded for 11 min at 100 cm^{-1} per 1 s. ESR spectra of solid samples were recorded at 20 °C on a SE/X-2547 Radiopan spectrometer equipped with a magnetometer and a detector of microwave frequency.

For studying specific dark electroconductivity, 5—10 μm thick layers were prepared by deposition of a chloroform solution of a sample on quartz supports on which Al or Ag electrodes of the raster or sandwich type had been preliminarily placed. The specific electroconductivity was measured using an electrometric VK-2-16 amplifier; voltage was applied to a sample using a BS-50 constant current source.

Reaction of 1-chloro-2-phenylethane-2,2-dithiol with salts (general procedure). A solution of the corresponding metal salt (0.05 mmol) in 5 mL of methanol was added to a solution of 1-chloro-2-phenylethane-2,2-dithiol **1** (1 mmol) in 10 mL of anhydrous methanol at 0 °C under argon. The mixture was warmed to 20 °C and kept at this temperature for 12 h. The precipitate that formed was filtered off and dried *in vacuo*. Compounds **3a—d** were dissolved in chloroform and reprecipitated with hexane.

Mercury 1-chloro-2-phenylethane-2,2-dithiolate (2a), yield 78%, decomp.p. 230—232 °C (chloroform—diethyl ether). ^{1}H NMR, δ: 3.80 (s, 2 H, CH_2); 7.55—7.88 (m, 5 H, C_6H_5). IR, ν/cm^{-1}: 1645 v.s (M—π arom.); 1380 v.w (C—C); 1070 w (δ CH); 685, 745 (π-CH). ESR spectrum: singlet g = 2.0036, ΔH = 1.6 mT. Found (%): C, 23.50; H, 1.68; Cl, 8.70; Hg, 50.01; S, 16.06. $C_8H_7ClHgS_2$. Calculated (%): C, 23.88; H, 1.74; Cl, 8.70; Hg, 49.75; S, 15.90.

Lead 1-chloro-2-phenylethane-2,2-dithiolate (2b), yield 80%, decomp.p. 180—182 °C (methanol). ^{1}H NMR, δ: 3.46 (s, 2 H, CH_2); 7.19—7.68 (m, 5 H, C_6H_5). IR, ν/cm^{-1}: 1620 v.s; 1660 w (M—π arom.); 1400 (C—C); 1100 (C—C); 1025 (δ CH); 745, 685 (π-CH). ESR spectrum: singlet g = 2.0036, ΔH = 0.1 mT. Found (%): C, 22.98; H, 2.00; Cl, 8.95; S, 16.18; $C_8H_7ClPbS_2$. Calculated (%): C, 23.44; H, 1.71; Cl, 8.67; S, 15.63.

Compound 3a, yield 74%, decomp.p. 178—180 °C. ^{1}H NMR, δ: 1.56 (s, 2 H, SH); 4.21 (s, 4 H, CH_2); 7.30—7.96 (m, 10 H, C_6H_5). IR, ν/cm^{-1}: 1360 (C—C); 1185 (C—C); 1030 (δ CH); 850, 750, 685 (π-CH). ESR spectrum: $g_{\parallel}$ = 2.2206; $g_{\perp}$ = 2.0177. Found (%): C, 48.36; H, 4.24; Cu, 16.00; S, 32.54. $C_{16}H_{18}CuS_4$. Calculated (%): C, 48.06; H, 4.00; Cu, 15.89; S, 32.04.

Compound 3b, yield 68%, decomp.p. 125—126 °C. ^{1}H NMR, δ: 1.26 (s, 2 H, SH); 4.26 (s, 4 H, CH_2); 7.25—8.19 (m, 10 H, C_6H_5). IR, ν/cm^{-1}: 1625, 1660 w (M—π arom.); 1440 (C—C); 1120 (C—C); 1000 (δ CH); 745, 685 (π-CH). ESR spectrum: $g_{\parallel}$ = 2.1562; $g_{\perp}$ = 2.0240. Found (%): C, 49.07; H, 4.00; Fe, 14.39; S, 32.55. $C_{16}H_{18}FeS_4$. Calculated (%): C, 48.97; H, 4.08; Fe, 14.28; S, 32.65.

Compound 3c, yield 78%, decomp.p. 290—292 °C. ^{1}H NMR, δ: 1.44 (s, 2 H, SH); 3.55 (s, 4 H, CH_2); 7.38—8.36 (m, 10 H, C_6H_5). IR, ν/cm^{-1}: 1615, 1660 w (M—π arom.); 1420 (C—C); 1100 (C—C); 1000 (δ CH); 745, 685 (π-CH). ESR spectrum: g = 2.0142. Found (%): C, 49.00; H, 4.08; Co, 14.76; S, 32.83. $C_{16}H_{18}CoS_4$. Calculated (%): C, 48.60; H, 4.05; Co, 14.93; S, 32.40.

Compound 3d, yield 79%, decomp.p. 270—272 °C. ^{1}H NMR, δ: 1.50 (s, 2 H, SH); 3.74 (s, 4 H, CH_2); 7.49—7.96 (m, 10 H, C_6H_5). IR, ν/cm^{-1}: 1360 (C—C); 1185 (C—C); 1030 (δ CH); 855, 750, 685 (π-CH). ESR spectrum: $g_{\parallel}$ = 2.2267, $g_{\perp}$ = 2.0177. Found (%): C, 48.81; H, 4.04; Ni, 14.93; S, 32.72. $C_{16}H_{18}NiS_4$. Calculated (%): C, 48.60; H, 4.05; Ni, 14.93; S, 32.40.

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