C, 65.67; H, 4.23; F, 14.18. ^{1}H NMR (δ, J/Hz): 7.50 (s, 1 H, OH); 7.30 (s, 1 H, OH); 7.01 (d, 2 H, H-2, H-6, ^3J = 8.1); 6.85 (s, 2 H, H-2′, H-6′); 6.81 (d, 2 H, H-3, H-5, ^3J = 8.1); 3.75 (s, 3 H, OMe); 2.20 (s, 6 H, 2 Me).

The work was supported by the Russian Foundation for Basic Research (Project No. 93-07-22696).

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Received April 18, 1995;
in revised form June 15, 1995

Synthesis of fluoroalkyl-containing bis-β-diketones of aromatic and heterocyclic series

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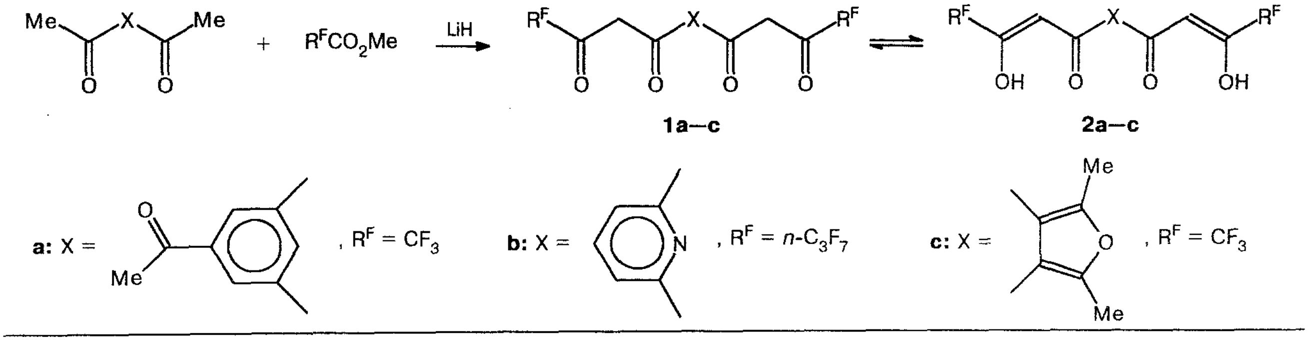
A huge number of fluoroalkyl-containing β-diketones, widely used in both organic synthesis and practice,[1] are presently known. At the same time there is only one example of fluoro-containing bis-β-diketones in which two β-diketone fragments are bound by the saturated fluoroaliphatic chain,[2] whereas aliphatic bis-β-diketones have for a long time drawn the attention of investigators.[3]

We have synthesized for the first time fluoroalkyl-containing bis-β-diketones (**1**), in which diketone fragments are separated by aromatic cycle or heterocycle with a consequent considerable extension of p—π-conjugation in the chain (Scheme 1).

Claisen condensation of a methyl polyfluorocarboxylate with a diacetyl derivative was applied for this synthesis. Compounds **1a—c** exist as bis-enols **2a—c**, which is typical for fluoro-containing diketones.

5-Acetyl-1,3-di-(4,4,4-trifluoro-1,3-dioxobut-1-yl)benzene (1a). 1,3,5-Triacetylbezene (2.04 g, 0.01 mol), LiH (0.3 g, 0.036 mol), and dry methyltrifluoroacetate (25 mL) were placed in a 100-ml three-neck flask. The reaction mixture was stirred and refluxed for 20 h. The solvent was distilled off, water (20 mL) was added to the reaction mixture, and the pH was brought to 3—4 by dilute HCl. The aqueous solution was extracted with ether (4×30 mL), and the extracts were washed with water and shaken with an aqueous solution of Cu(OAc)$_2$ (3×50 ml). Then ether was distilled off, and the residue was

Scheme 1



Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 11, pp. 2289—2290, November, 1995.

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precipitated with ethanol. The resulting product was treated with an excess of dilute H_2SO_4, extracted with ether (3×20 mL), and dried over $MgSO_4$. After that the solvent was distilled off, and the residue was twice recrystallized from ether. Yield: 0.82 g (21%) of compound **1a**, m.p. 141—142 °C. Found (%): C, 48.29; H, 2.27; F, 28.78. $C_{16}H_{10}F_6O_5$. Calculated (%): C, 48.50; H, 2.54; F, 28.77. IR spectrum (in Nujol), ν/cm^{-1}: 1675 (C=O), 1625 (C=O...H). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 2.76 (s, 3 H, CH_3CO), 6.71 (s, 2 H, 2[=CH—]), 8.68 (s, 3 H, C_6H_3), 14.19 (s, 2 H, 2[OH]).

2,6-Di-(4,4,5,5,6,6,6-heptafluoro-1,3-dioxohex-1-yl)pyridine (1b). This product was obtained in the same manner. Yield: 3%, m.p. 128—130 °C. Found (%): C, 37.17; H, 1.70; N, 2.54; F, 47.49. $C_{17}H_7F_{14}NO_4$. Calculated (%): C, 36.78; H, 1.27; N, 2.52; F, 47.90. IR spectrum (in Nujol), ν/cm^{-1}: 1625 (C=O...H). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 7.34 (s, 2 H, 2[=CH—]), 8.04—8.39 (m, 3 H, C_5H_3N), 11.71 (s, 2 H, 2[OH]).

2,5-Dimethyl-3,4-di-(4,4,4-trifluoro-1,3-dioxobut-1-yl)furan (1c). Yield: 46%, m.p. 62—63 °C. Found (%): C, 45.68; H, 2.77; F, 30.98. $C_{14}H_{10}O_5F_6$. Calculated (%): C, 45.17; H, 2.71; F, 30.62. IR spectrum (in Nujol), ν/cm^{-1}: 1600 (C=O...H). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm): 2.52 (s, 6 H, 2[CH_3]), 6.05 (s, 2 H, 2[=CH—]), 14.36 (s, 2 H, 2[OH]).

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Received May 30, 1995;
in revised form July 12, 1995

Convenient method for the preparation of a solution of potassium bis(trimethylsilyl)amide

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Potassium bis(trimethylsilyl)amide is an important non-nucleophilic base that is widely used in organic synthesis. The main methods for its preparation are interaction of hexamethyldisilazane with potassium amide,[1] potassium hydride,[2] potassium metal, and styrene,[3] isoprene,[3] or naphthalene[4] as electron acceptors. However, these methods have several disadvantages: one has to work with pyrophoric potassium hydride, and products of reduction of electron acceptors are present in the reaction mixture. The reaction of hexamethyldisilazane with potassium and bromobenzene is similar to that described below but proceeds in a low yield, 61 %.[5]

We developed a convenient one-step method for preparation of a solution of potassium bis(trimethylsilyl)amide which does not have the above-mentioned disadvantages. The method is based on the reaction of a dispersion of potassium with hexamethyldisilazane in tetrahydrofuran in the presence of butyl chloride and involves *in situ* formation of butylpotassium.

$$HN(SiMe_3)_2 + 2K + BuCl \xrightarrow[-KCl,\ -BuH]{THF} KN(SiMe_3)_2$$

A mixture of hexamethyldisilazane (20 mmol), potassium metal (50 mmol), and tetrahydrofuran (40 mL) was heated in argon atmosphere with vigorous stirring until melting of the potassium, and the mixture was then very quickly cooled to 20 °C. A solution of butyl chloride (20 mmol) in THF (5 mL) was added with stirring to the resulting dispersion of potassium over a period of 30 min. The mixture was stirred for an additional 20 min, and then the precipitate of KCl that formed was filtered off under argon to afford a light-yellow solution of potassium bis(trimethylsilyl)amide (0.3 *M*) in 92 % yield. The yield was determined by titration using 1′,4′-dihydro-2,3′-biquinoline as the indicator.[6]

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 11, pp. 2290—2291, November, 1995.
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