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

I. V. Magedov,[a]* *A. Yu. Polykarpov,*[b] *T. O. Zotova,*[a] *Yu. I. Smushkevich,*[b] *and V. N. Drozd*[a]

[a]*K. A. Timiryazev Agricultural Academy, Department of Organic Chemistry,*
49 ul. Timiryasevskaya, 127550 Moscow, Russian Federation.
Fax: +7 (095) 976 1820
[b]*D. I. Mendeleev Chemico-Technological University, Department of Organic Chemistry,*
9 Miusskaya pl., 125820 Moscow, Russian Federation

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.

1066-5285/95/4411-2197$12.50 ©1996 Plenum Publishing Corporation

This study was financially supported by the Russian Foundation for Basic Research (Grant No. 93-03-04290) and the State Committee for Higher Education of the Russian Federation (scientific and technical program "Fine organic synthesis", Grant No. FT-12).

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Received June 1, 1995

Synthesis of tetranuclear butterfly clusters $RuM_3(\mu\text{-}H)(CO)_{12}(\mu_4\text{-}\eta^1\text{-}\eta^1\text{-}\eta^1\text{-}\eta^2\text{-}C_2Fc)$ (M = Ru, Os; Fc = ferrocenyl) and $RuOs_3(CO)_{12}(\mu_4\text{-}\eta^1\text{-}\eta^1\text{-}\eta^2\text{-}\eta^2\text{-}HC_2Me)$ from trinuclear acetylide precursors. Fluctuation of μ_4-bonded acetylide ligand

A. A. Koridze, V. I. Zdanovich, V. Yu. Lagunova, A. M. Sheloumov, F. M. Dolgushin, A. I. Yanovsky, Yu. T. Struchkov,† M. G. Ezernitckaya, E. V. Vorontsov, and P. V. Petrovskii*

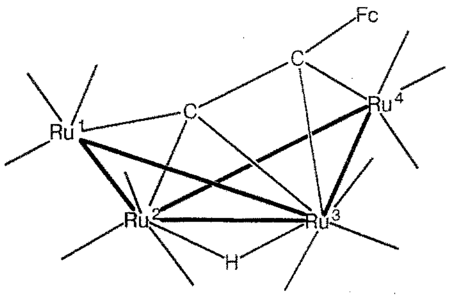
A. N. Nesmeyanov Institute of Organometallic Compounds, Russian Academy of Sciences,
28 ul. Vavilova, 117813 Moscow, Russian Federation.
Fax: +7 (095) 135 5085

Being highly unsaturated and able to donate from one to five electrons, acetylide ligands show a diversity of bonding possibilities in transition metal clusters.[1] The study of molecular clusters with these ligands allows the processes of hydrocarbon C_2 group transformation on the polymetal centers of a catalytic surface to be clarified.

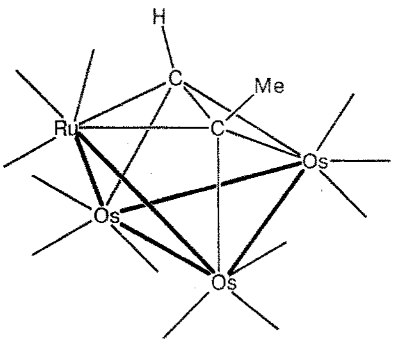
Previously, for the synthesis of acetylide derivatives of tri- and tetranuclear clusters we used an approach based on the interaction of mononuclear acetylide complexes with metal carbonyl clusters.[2–4] In this paper, the synthesis of tetranuclear butterfly clusters from trinuclear acetylide clusters is described.

Treatment of $Ru_3H(CO)_9(C{\equiv}CFc)$ (**1**, Fc = ferrocenyl) with dodecacarbonyltriruthenium in refluxing hexane gave the dark green $Ru_4(\mu\text{-}H)(CO)_{12}(\mu_4\text{-}\eta^1\text{-}\eta^1\text{-}\eta^1\text{-}\eta^2\text{-}C_2Fc)$ complex (**2**) in 40 % yield. Cluster **2** can also be obtained by the interaction of ferrocenylacetylene with $Ru_3(CO)_{12}$, but the yield is lower.

The IR spectrum of **2** revealed the presence only of terminal CO ligands (νCO/cm^{-1}, hexane): 2090 w, 2062 vs, 2052 s, 2038 w, 2019 s, 1996 w (br), 1979 m (br). The ^{1}H NMR spectrum ($C_6D_5CD_3$, 25 °C) contains signals of the hydride ligand at δ −22.18 (1 H) and the ferrocenyl group at 4.31 (5 H), 4.76 (2 H), and 5.55 (2 H), the later being broadened.



2



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Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 11, pp. 2292—2293, November, 1995.