Journal of Organometallic Chemistry, 463 (1993) C1–C2

JOM 24055PC

Preliminary Communication

Insertion of tellurium(0) into yttrium–carbon bonds

Irina P. Beletskaya [a], Alexander Z. Voskoboynikov [b], Alla K. Shestakova [b] and Herbert Schumann [c]

[a] *Department of Chemistry, Moscow State University, 119899 Moscow GSP-3 (Russian Federation)*

[b] *State Scientific-Research Institute of Chemistry and Technology of Organoelement Compounds, sh. Entuziastov 38, 111123 Moscow (Russian Federation)*

[c] *Institut für Anorganische und Analytische Chemie der Technische Universität Berlin, Straße des 17. Juni 135, D-10623 Berlin (Germany)*

(Received July 7, 1993)

Abstract

The dimer $\{(^t\mathrm{BuC_5H_4})_2\mathrm{Y}(\mu\text{-Me})\}_2$ reacts with metallic tellurium to form $\{(^t\mathrm{BuC_5H_4})_2\mathrm{Y}(\mu\text{-TeMe})\}_2$ with a high yield.

Organotellurium derivatives of lanthanides can play an important role as precursors for the preparation of very pure and homogeneous materials for electronics. However, synthesis of such compounds in analogy to the corresponding sulfur and selenium derivatives by cleavage of Ln–C, Ln–H or Ln–N bonds with RTeH is rather difficult because of low stability of organotellurium hydrides RTeH [1–7]. Also metathesis reactions between lanthanide halides and salts such as NaTeR suffer from very low yields of products. We have now succeeded in the preparation of one representative of this interesting class of compounds by a very simple route. Metallic tellurium reacts readily with dimeric bis(tert-butylcyclopentadienyl)methyl yttrium by insertion of tellurium(0) into the yttrium–carbon bonds, thus substituting the bridging methyl groups:

$$\left\{\left(^t\mathrm{BuC_5H_4}\right)_2\mathrm{Y}(\mu\text{-Me})\right\}_2 + 2\mathrm{Te} \xrightarrow[20°\mathrm{C},10\,\mathrm{h}]{\text{toluene}} \{(^t\mathrm{BuC_5H_4})_2\mathrm{Y}(\mu\text{-TeMe})\}_2$$

As determined by nuclear magnetic resonance (NMR), it is the main product of this transformation [8*]. The compound crystallizes from toluene as colourless thin needles, which are sensitive to both air and moisture. They also decompose slowly under light with deposition of tellurium.

The ^{1}H NMR spectrum shows some interesting features. The spectrum displays spin-coupling $^1\mathrm{H}$–$^{89}\mathrm{Y}$ for the μ-TeMe protons ($J = 0.6$ Hz), and surprisingly also long-range coupling for the four cyclopentadienyl ring protons ($J = 0.5$ Hz). Up to now, the last coupling could be observed using special NMR procedures. The fact that the methyl protons give rise to a triplet signal proves the dimeric state of the compound even in benzene solution.

According to preliminary NMR results the organolutetium compounds $\{\mathrm{Cp'_2Lu}(\mu\text{-Me})\}_2$ ($\mathrm{Cp'} = \mathrm{C_5H_5}$, $^t\mathrm{BuC_5H_5}$ or $\mathrm{Me_3SiC_5H_4}$) react with metallic tellurium in an analogous manner to form the complexes $\{\mathrm{Cp'_2Lu}(\mu\text{-TeMe})\}_2$.

Acknowledgment

At the Technische Universität Berlin this research was supported by the Deutsche Forschungsgemeinschaft (Sonderforschungsbereich "Anisotrope Fluide" by a scholarship to A.Z.V.)

References and notes

1 H. Schumann, I. Albrecht and E. Hahn, *Angew. Chem., 97* (1985) 991; *Angew. Chem., Int. Ed. Engl., 24* (1985) 985.
2 H. Schumann, I. Albrecht, M. Gallagher, E. Hahn, C. Murchmori and J. Pickardt, *J. Organomet. Chem., 349* (1988) 103.
3 S.D. Stults, R.A. Andersen and A. Zalkin, *Organometallics, 9* (1990) 1623.
4 B. Cetinkaya, P.B. Hitchcock, M.F. Lappert and R.G. Smith, *J. Chem. Soc., Chem. Commun.*, (1992) 932.
5 H.C. Aspinall, D.C. Bradley, M.B. Hursthouse, K.P. Sales and N.P.C. Walker, *J. Chem. Soc., Chem. Commun.*, (1985) 1585.
6 S.P. Nolan, D. Stern and T.J. Marks, *J. Am. Chem. Soc., 111* (1989) 7844.
7 Ju.F. Rad'kov, E.A. Fjodorova, S.Ja. Horoshev, G.S. Kalinina, M.N. Bochkarjov and G.A. Razuvaev, *Zh. Obsch. Khim., 55* (1985) 2153.
8 (a) All manipulations have been done on the high vacuum line and in an atmosphere of thoroughly purified argon using standard Schlenk technique. ^{1}H and ^{13}C NMR spectra were recorded with a Bruker AM 360 instrument. Electron probe X-ray analysis were carried out using a Philips SEM-505 scanning electron microscope equipped with a unit for X-ray energy dispersive analysis. (b) $\{(^t\mathrm{BuC_5H_4})_2\mathrm{Y}(\mu\text{-TeMe})\}_2$: to a solution of 1.16 g (1.68 mol) of $\{(^t\mathrm{BuC_5H_4})_2\mathrm{Y}(\mu\text{-Me})\}_2$ in 40 ml of toluene was added 1.0 g (7.8

Correspondence to: Dr. I.P. Beletskaya.

* A reference number with an asterisk indicates a note in the list of references.

0022-328X/93/$6.00

© 1993 – Elsevier Sequoia S.A. All rights reserved

$\{(^tBuC_5H_4)_2Y(\mu\text{-}Me)\}_2$ in 40 ml of toluene was added 1.0 g (7.8 mmol) of metallic tellurium powder. After stirring at room temperature for 10 h the yellow solution was decanted from the precipitate with a tellurium excess and evaporated *in vacuo* to about 5 ml. The product precipitated on cooling to 0°C and was filtered off. The crude product was recrystallized from a minimal amount of toluene and dried *in vacuo* to give 1.21 g (76%) of colourless microcrystalline powder. Anal. Found: C, 48.27; H, 6.20. $C_{38}H_{58}Te_2Y_2$ calc.: C, 48.10; H, 6.12%. Electron probe microanalysis: Y:Te = 1:1. ^1H NMR (C_6D_6, Me_4Si (external)): 1.40 (s, 36 H, ^tBu); 2.04 (t, 6 H, $J(^1H\text{–}^{89}Y) = 0.6$ Hz, μ-TeMe); 6.28 (d-t, 8 H, $J(^1H\text{–}^{89}Y) = 0.5$ Hz, $J = 2.7$ Hz, 2,5-H_2 in C_5H_4); 6.62 (d-t, 8H, $J(^1H\text{–}^{89}Y) = 0.5$ Hz, $J = 2.7$ Hz, 3,4–H_2 in C_5H_4) ppm. ^{13}C NMR (C_6D_6, Me_4Si (external)): 7.2 (μ-TeMe); 32.3 (CMe_3); 41.7 (CMe_3); 109.5 (2,5-C_2 in C_5H_4); 109.8 (3,4-C_2 in C_5H_4); 140.0 (1-C in C_5H_4) ppm.