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Preliminary communication

Fixation of the carbon monoxide in the system $TiCl_3 \cdot 3THF + Mg + THF$

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Yamamoto et al.[1,2] demonstrated the molecular nitrogen fixation in the system $TiCl_3 \cdot 3\ THF + Mg + THF$ (A), with formation of the nitrogen-titanium compound $TiNMg_2Cl_2 \cdot THF$. In that process 0.5 N_2 molecule and 2.5 g-atoms magnesium react per one titanium g-atom.

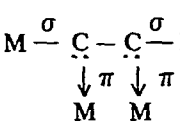
We have now shown that in the system (A) under mild conditions, there is also carbon monoxide fixation. In such a system, when there is a tenfold magnesium excess, 3 CO molecules per titanium atom are fixed. As happens during the nitrogen fixation, the solution changes its colour from blue to black. During the reaction, liberation of gases was observed, the principal component of which was methane.

The black product (B) obtained after the separation of the magnesium excess and after evaporation of tetrahydrofuran under vacuum, was treated with water. The gas products were chromatographically examined. After hydrolysis it was found, that besides hydrocarbons, which are liberated in the CO fixation, a new gas product, namely acetylene, was formed.

$$TiCl_3 \cdot 3\ THF + Mg + THF + CO \rightarrow (B) + CH_4 \xrightarrow{H_2O} C_2H_2 + CH_4$$

In the IR spectrum of the product (B) no band to the fixed carbon monoxide was observed.

Formation of acetylene after hydrolysis of the product (B) indicates the reduction of carbon monoxide to carbide MC_2 (where M = Mg, or Mg and Ti) or the formation of a carbenoide in system A:



Liberation of hydrocarbons from system (A) and during the N_2 and CO fixation is most probably caused by breakdown of the organometallic compounds due to the dehydration of tetrahydrofuran under influence of titanium at the low oxidation state[3].

REFERENCES

1 A. Yamamoto, M. Ookawa and S. Ikeda, *Chem. Commun.* (1969) 841
2 A. Yamamoto, S. Go, M. Ookawa, M. Tahahashi, S. Ikeda and T. Keii, *Bull. Chem. Soc. Jap.*, 45 (1972) 3110
3 B. Jeżowska-Trzebiatowska and P. Sobota, *J. Organometal. Chem.*, 46 (1972) 333