

SHORT COMMUNICATIONS

Reaction of Si-Si Bond in Hexaisopropoxydisilane with Sodium Methylate

By Ukiko TAKAHASHI

(Received March 10, 1955)

Recently, Ōkawara and his co-workers¹⁾ reported on the preparation of alkoxydisilanes in pure state, and they pointed out the uncertainty of volumetric determination of hydrogen evolved by the cleavage of the Si-Si bond with basic reagents. I wish to report on the possibility of determining the Si-Si bond by volumetric method when the bond is cloven by sodium methylate.

Isopropoxydisilane is the only reliable alkoxydisilane to be taken as a standard sample, for it is obtained in crystalline state and can be purified by recrystallization from the contaminating liquid materials, especially, hexaisopropoxydisiloxane which has the boiling point close to that of disilane. In this experiment, the recrystallized isopropoxydisilane having the melting point 86.0°C (reported¹⁾, 84.7°C) was used. Calcd. for $(C_3H_7O)_2Si_2$: Si, 13.68%. Found: Si, 13.73%. Sodium methylate solution was prepared by edding sodium metal to the completely dehydrated methanol. Methanol was dehydrated with magnesium and distilled through a Stedman column of about 20 theoretical plates, any contamination of moisture being carefully prevented.

With 2.8N CH_3ONa solution and isopropoxydisilane the reaction proceeded rapidly in Zerewitinoff apparatus at 13°C, but with 0.6 N- CH_3ONa the reaction was slow and completed in forty hours at the same temperature.

Anal. H_2 evolved from 0.3554 g. of $(C_3H_7O)_2Si_2$ and 6 cc. of 2.8 N- CH_3ONa solution: Calcd. vol., 19.39 cc. Found vol. 19.40 cc. (corrected), 21.97 cc. (uncorrected).

Since the hydrogen evolved was replaced by methanol, the vapor pressure of methanol was taken account of in the volume correction.

In this experiment, care should be taken to use completely dehydrated methanol in preparing sodium methylate solution. For if an incompletely dehydrated methanol is used, the solution becomes turbid at the early

stage of the reaction with disilane and the volume of hydrogen can not attain the calculated value. It is to be considered that, for the same reason, the volumetric determination of alkoxydisilanes will give inconsistent results¹⁾ with the basic reagents containing water which will cause flocculent precipitate or gelation of disilanes.

1) R. Ōkawara, T. Tanaka and K. Maruo, This Bulletin 28, 189 (1955).

Osaka Prefectural Kozu Senior High School,
Higashi-Kozu Kitanocho, Tennoji-ku,
Osaka