COMMUNICATIONS TO THE EDITOR

GRIGNARD REAGENTS FROM DIALKYL SULFATES

Sir:

In a study of some of the reactions of di-*n*-butyl sulfate it was found that this compound reacts with *n*-butylmagnesium bromide according to the equation: $(n\text{-}C_4H_9)_2SO_4 + n\text{-}C_4H_9MgBr \longrightarrow n\text{-}C_4H_9MgOSO_2OC_4H_9 + n\text{-}C_4H_9Br$. This indicated that dialkyl sulfates might form Grignard reagents directly and this has been found to be the case. Diethyl sulfate reacts readily with magnesium in dry ether to give a slightly soluble Grignard reagent as follows: $(C_2H_5)_2SO_4 + Mg \longrightarrow C_2H_5Mg(C_2H_5)SO_4$. This with benzaldehyde gave a good yield of phenylethylcarbinol. Di-*n*-butyl sulfate similarly formed a compound which hydrolyzed readily to give *n*-butane. So far as we are at present aware this type of Grignard reagent has not been studied heretofore.

The investigation upon the preparation and properties of di-*n*-butyl sulfate will be published shortly. Work upon the Grignard reagents from dialkyl sulfates is still in progress.

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CONVERSION OF ORTHO TO PARA HYDROGEN OVER PROMOTED IRON SYNTHETIC AMMONIA CATALYSTS AT −190°

Sir:

The catalytic conversion of ortho to para hydrogen at about −190° has now been reported for a number of catalysts. It is generally recognized that the mechanism of the low temperature conversion, whatever be its nature, is quite different from that involved in the high temperature conversion (from 0 to 500°). The experiments on a promoted iron catalyst to be summarized briefly in the present letter afford several additional contrasts between the characteristics of the high and the low temperature conversion.

The adsorption of hydrogen by a promoted iron synthetic ammonia catalyst was found to inhibit markedly the activity of the latter toward the ortho-para conversion at −190°. Thus at a space velocity of 12,000 practically 100% conversion of a 25:75 to a 47:53 para-ortho mixture could be obtained if the freshly reduced sample was degassed at 450° and cooled to −190° in helium; whereas if the sample was cooled in