

STABLE SOLUTIONS OF METHYLSULFINYL CARBANION

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The great synthetic value of methylsulfinyl carbanion, sodium methylsulfinylmethanide, has recently been demonstrated by Corey and Chaykovsky (1).

Solutions containing this anion have generally been prepared by heating a suspension of sodium hydride in dimethyl sulfoxide at 70° for one hour (2). Such solutions are sensitive to heat and air (3) and decompose rapidly above 85°. At 20° they lose some eight per cent of their activity per week (3). The decomposition is indicated by the appearance of a grey precipitate and by darkening of the solution.

For synthetic purposes it would be convenient to have a stable, standardized stock solution of methylsulfinyl carbanion at hand. Such solutions can be prepared according to the following procedure.

A 50% dispersion of sodium hydride in mineral oil (15g) was stirred into dry dimethyl sulfoxide (200ml) and with continuous stirring treated with ultrasound. The ultrasound source used was a Lehfeldt GT 200a apparatus of 0.8 Mc/s operated at 200 W. The temperature rose to 50° and a fine, very reactive dispersion resulted, which in one hour yielded a clear solution of methylsulfinyl carbanion of much

weaker colour than when no ultrasound was applied.

The solution was protected against air by a one centimeter surface layer of mineral oil, added after the ultrasound was switched off. For synthetic work the use of anaerobic burettes and hypodermic needles (3) is unnecessary. The required amount of reagent can be withdrawn from the stock solution simply by means of a pipette.

Stocks of this reagent prepared as described above solidify at about 10^o and in this state they can be stored for at least two months.

REFERENCES

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3. G. G. Price and M. C. Whiting, Chem. Ind. (London), 775 (1963).