Notes A department for short papers of immediate interest.

Direct Preparation of Vinyllithium[1]

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Received October 4, 1960

The useful intermediate vinyllithium can be prepared by cleavage of tetravinyltin with phenyllithium.[2] The direct preparation of vinyllithium from vinyl halides and lithium metal at very low temperatures has been reported,[3] but the yields are low and erratic.[4]

In these laboratories we have been unsuccessful in all attempts to prepare vinyllithium from vinyl chloride or bromide and ordinary lithium metal containing about 0.02% sodium, in various solvents. Recently the presence of sodium in the lithium metal used has been found to have a pronounced catalytic effect on the ease of preparation of certain organolithium compounds.[5] This observation prompted us to study the effect of sodium on the preparation of vinyllithium. We find that vinyllithium can be readily prepared in tetrahydrofuran from vinyl chloride and a lithium dispersion containing about 2% sodium.[6] This procedure should provide a highly convenient method for the preparation of vinyllithium reagent solutions. Satisfactory yields are obtained at 25°, but the preferred temperature for the reaction is 0-10°.

EXPERIMENTAL

Vinyl chloride (Matheson Co.) from a cylinder was condensed into a calibrated trap. Six grams (0.10 mole) of the chloride was entrained in a stream of argon and bubbled into a stirred flask containing 1.5 g. of lithium −2% sodium dispersion in 250 ml. of tetrahydrofuran under an argon atmosphere. A reaction commenced after about one-fourth of the vinyl chloride had been added, and the remainder of the addition was carried out at 0–10°. After addition the mixture was stirred for 2 hr. at 0° and then allowed to warm to room temperature with stirring. Filtration in an argon atmosphere gave a clear colorless solution of vinyllithium in tetrahydrofuran. The yield of vinyllithium was 60–65% as estimated by titration with base and by reaction with vanadium pentoxide followed by titration with standard permanganate solution.[7] A derivative from acetone yielded approximately 20% of dimethylvinylcarbinol, b.p. 97–99°, n_D^{25} 1.4178.[8]

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(1) This research was supported by a grant from the Atomic Energy Commission.

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(6) Supplied by the Lithium Corp. of America, Inc., through the courtesy of Dr. Donald L. Esmay.

(7) Technical Bulletin No. 312-360, Lithium Corp. of America, Inc., Minneapolis, Minn., 1960.

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Relative Stabilities of *cis* and *trans* Isomers. XI. The 9-Methyldecalins[1]

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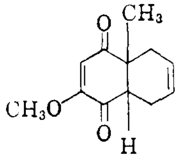
Received August 9, 1960

The 9-methyldecalin system occurs widely in nature, and the relative stabilities of the *cis* and *trans* isomers have been of considerable interest. The thermodynamic quantities for the reaction *cis*⇌*trans* 9-methyldecalin are reported in this paper.

The *cis*- and *trans*-9-methyldecalins are known compounds, and the assignment of their stereochemistry is based on the fact that I (*cis*-form) results from the Diels-Alder addition of butadiene to the appropriate quinone,[2] while the *trans* form is obtained by treating the Diels-Alder adduct with base.[3] That the energies (enthalpies) of the isomeric 9-methyldecalins would be more similar to one another than is found with the decalins themselves was recognized some years ago by Turner[4] who predicted that the *trans* would have a lower enthalpy by 0.8 kcal./mole. Recently[5]



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