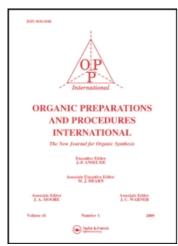
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Publisher Taylor & Francis

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Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t902189982

A FACILE SYNTHESIS OF DIARYLACETONITRILE FROM DIARYKETONES AND TRIMETHYLSILYL CYANIDE

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To cite this Article Saito, Katsuhiro , Kagabu, Shinzo , Horie, Yoichi and Takahashi, Kensuke(1989) 'A FACILE SYNTHESIS OF DIARYLACETONITRILE FROM DIARYKETONES AND TRIMETHYLSILYL CYANIDE', Organic Preparations and Procedures International, 21:3,354-355

To link to this Article: DOI: 10.1080/00304948909356395 URL: http://dx.doi.org/10.1080/00304948909356395

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A FACILE SYNTHESIS OF DIARYLACETONITRILE FROM DIARYKETONES AND TRIMETHYLSILYL CYANIDE

Submitted by (07/13/88) Katsuhiro Saito,* Shinzo Kagabu[†], Yoichi Horie and Kensuke Takahashi

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Introduction of the nitrile group into organic compounds is a very valuable technique in organic synthesis. Conversion of carbonyl compounds into nitrile compounds has been investigated actively and a wide variety of methods have been developed. Trimethylsilyl cyanide proved to be very effective to transform carbonyl compounds into α -cyanohydrins. However, reduction of the hydroxy group of the cyanohydrins requires conversion of the cyanohydrins to α -halonitriles, which are then reduced with zinc. α

We now report that diarylketones (1) may be converted into diarylacetonitriles (3) in moderate yields, $\frac{3}{2}$ via the reaction of trimethylsilyl cyanide with the sodium salts of diarylketone

tosylhydrazones ($\underline{2}$), very easily prepared in quantitative yields from $\underline{1}$ and tosylhydrazide. The procedure is a simple one-pot reaction which requires only addition of sodium hydride and trimethylsilyl cyanide to a diglyme solution of the tosylhydrazone.

TABLE. Yields of Diarylacetonitriles (3)⁵

 2	X	Y	Yields of <u>3</u> ^a (%)	IR (CN) (cm ⁻¹)
<u>2a</u>	Н	Н	49	2222
<u>2b</u>	Н	Cl	43	2221
<u>2c</u>	Н	OMe	52	2220
<u>2d</u>	OMe	ОМе	51	2223

a) All the yields are isolated yields based on 2.

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EXPERIMENTAL SECTION

Reaction of Sodium Salt of Benzophenonetosylhydrazone (2a) with Trimethylsilyl Cyanide. Representative Procedure. To a solution of 2a (7.36 g, 20 mmol) in anhydrous diglyme (50 ml) was added sodium hydride (50% in oil, 0.72 g, 30 mmol). After evolution of hydrogen gas had ceased, trimethylsilyl cyanide (9.90 g, 100 mmol) was added and the mixture was heated at 125° for 5 min. to evolve a quantitative amount of nitrogen gas. After filtration the filtrate was diluted with ether (200 ml), washed with water (200 ml x 5), dried over anhydrous sodium sulfate, and finally the solvent was evaporated on a rotary evaporator. The oily residue was chromatographed on silica gel (hexane-benzene 6:4) to give 3a (1.87 g, 49%).

Acknowledgement.- The authors are indebted to Professor Paul G. Gassman of the University of Minnesota for fruitful suggestions.

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AN IMPROVED PROCEDURE FOR THE DICHLOROACETYLATION OF PRIMARY AND SECONDARY AMINES[†]

Submitted by (04/18/88)

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The dichloroacetamido moiety (-NHCOCHCl₂) is prevalent in number of biologically