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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>

### Convenient One-Step Dialkylation of Diethyl Malonate With Sodium Hydride

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**To cite this Article** Kaiser, Edwin M. , Fries, Janet A. and Simonsen Jr., Walter J.(1971) 'Convenient One-Step Dialkylation of Diethyl Malonate With Sodium Hydride', *Organic Preparations and Procedures International*, 3: 6, 305 – 306

**To link to this Article:** DOI: 10.1080/00304947109356081

**URL:** <http://dx.doi.org/10.1080/00304947109356081>

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3. For representative examples see, (a) C. L. Arcus and E. A. Lucken, J. Chem. Soc., 1634 (1955); (b) Y. Sprinzak, J. Am. Chem. Soc., 74, 2116 (1952); (c) W. Schlenk, Chem. Ber., 64, 742 (1931).
4. G. Wittig, H. Doser and I. Lorenz, Ann., 562, 192 (1949) have reported the isolation of certain carbinols of type 3 by rearrangement of 9-fluorenyl ethers with phenyllithium. This method, which is less convenient than either the Grignard procedure or the present approach has apparently seen little synthetic use.
5. All chemicals were commercial reagent grade and were used without purification. Melting points were taken on a Thomas-Hoover apparatus in open capillaries and are corrected.

#### ACKNOWLEDGMENT

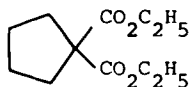
We are pleased to acknowledge The National Aeronautics and Space Administration and The National Science Foundation for support of JAC in the form of a NASA Traineeship and an NSF Summer Fellowship.

#### Convenient One-Step Dialkylation of Diethyl Malonate With Sodium Hydride

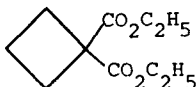
Submitted by: Edwin M. Kaiser, Janet A. Fries  
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The authors describe the title procedure using sodium hydride in tetrahydrofuran (THF). Thus, the di-n-butyl (86%), di-n-octyl (66%), di-benzyl (98%) and di-2-phenethyl(49%) diethyl malonates were obtained in the given yields from the respective halides.<sup>1</sup> Cyclizations using 1,4-dibromobutane and 1,3-dibromopropane afforded the cyclopentyl (I 63%) and

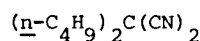
JOHN L. FERRARI



I



II



III

cyclobutyl (II 45%) derivatives. Significantly, the procedure may be applicable to related compounds; for example, di-n-butylmalononitrile III was similarly obtained from malononitrile (76%).

#### EXPERIMENTAL

General Procedure. To a refluxing slurry of four equivalents of sodium hydride<sup>2</sup> in 200 ml of anhydrous THF was added dropwise, during one hour, a solution of 0.1 m of diethyl malonate and 0.4 m of the alkyl halide (or 0.2 m of the alkyl dihalide) in 100 ml of THF. The mixture was refluxed for three more hours, cooled to 0° and hydrolyzed by cautious dropwise addition of 100 ml of water. Work up of the organic layers followed by vacuum distillation produced the desired compounds which exhibited correct boiling points<sup>1a</sup> and consistent nmr spectra.

#### ACKNOWLEDGMENT

This research was supported by the Petroleum Research Fund, administered by the American Chemical Society, on Grant 959-G.

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2. Purchased from Metal Hydrides, Inc.