This article was downloaded by: On: *27 January 2011* Access details: *Access Details: Free Access* Publisher *Taylor & Francis* Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## **Organic Preparations and Procedures International**

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

### DIETHYLVINYLPHOSPHATE

H. Gross<sup>a</sup>; B. Costisella<sup>a</sup> <sup>a</sup> Institut fur Organische Chemle der Deutschen Akademie der Wissenschaften, Berlin-Adlershof

**To cite this Article** Gross, H. and Costisella, B.(1969) 'DIETHYLVINYLPHOSPHATE', Organic Preparations and Procedures International, 1: 2, 97 – 98 **To link to this Article: DOI:** 10.1080/00304946909458359

URL: http://dx.doi.org/10.1080/00304946909458359

# PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

#### DIETHYLVINYLPHOSPHATE

H. Gross and B. Costisella Institut fur Organische Chemie der Deutschen Akademie der Wissenschaften, Berlin-Adlershof



This paper describes a simple synthesis of diethylvinylphosphate from chloroethylenecarbonate. The procedure corresponds essentially to the method which has been reported recently.<sup>1,2</sup> But the yield has been increased to 68% by the use of the following modifications. Chloroethylenecarbonate was converted to chloroacetaldehyde by heating with catalytic amounts of triethylamine. The chloroacetaldehyde formed was collected into a receiver containing triethylphosphite. Diethylvinylphosphate is obtained pure without by-products.

#### Experimental

<u>Diethylvinylphosphate</u>. A mixture of 12.25 g (0.10 mole) of freshly distilled chloroethylenecarbonate<sup>3</sup> and two drops of

#### H. GROSS AND B. COSTISELLA

triethylamine is heated in a distillation apparatus to an inner temperature of 165-170°C. Chloroacetaldehyde<sup>4</sup> which distills at 85-88°C is collected into a receiver heated to 75°C containing 16.6 g (0.10 mole) of triethylphosphite. The inner temperature of the receiver is maintained between 75° and 80°C during the distillation. After 60 minutes, the decomposition of the chloroethylenecarbonate is completed. The reaction mixture is then heated to 110° for 15 minutes and distilled in vacuo to give 12.3 g (68%) diethylvinylphosphate, b.p.<sub>11</sub><sub>mm.</sub> 94-95°C,  $n_D^{35°} = 1.4100$ , lit.<sup>5</sup> b.p.<sub>6mm</sub> 69°,  $n_D^{35°} = 1.4100$ .

#### References

- Roy C. De Selms and Tan-Wan-Lin, J. Org. Chem., <u>32</u>, 2023 (1967).
- H. Gross, G. Engelhardt, J. Freiberg, W. Burger and B. Costisella, Ann., <u>707</u>, 35 (1967).
- M. S. Newman and R. W. Addor, J. Am. Chem. Soc., <u>77</u>, 3791 (1955); <u>75</u>, 1264 (1953).
- 4. H. Gross, J. prakt. Chem., <u>41</u>, 99 (1963).
- 5. J. F. Allen and O. H. Johnson, J. Am. Chem. Soc., <u>77</u>, 2871 (1955).

(Received November 18, 1968)