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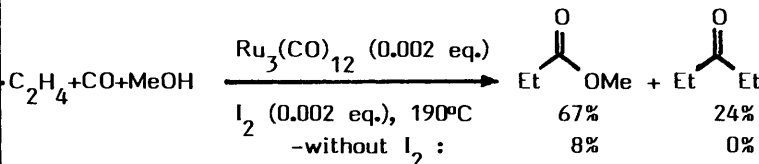
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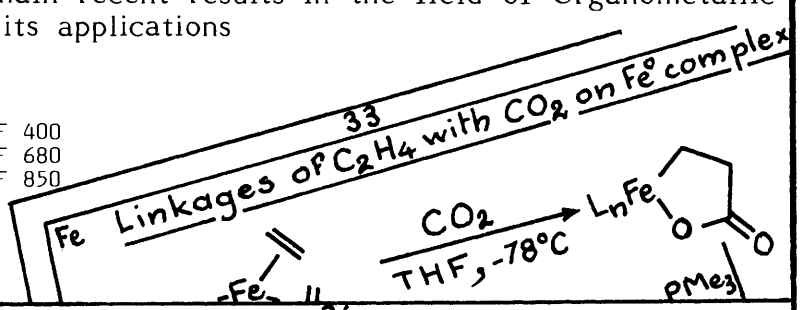
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J. Mol. Catal., 1987, 40 (2), 243-254.

Ru Cationic metallocyclophanes.



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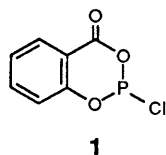
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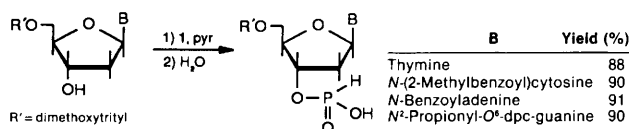
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van Boom's Reagent

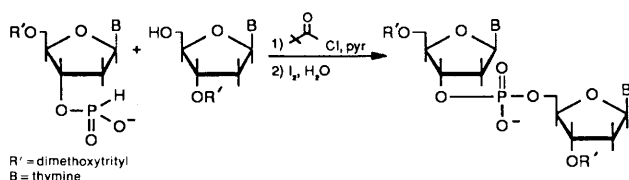


Prof. J.H. van Boom of Leiden University in Holland has recently introduced¹ a new monofunctional phosphitylating reagent, **2-chloro-4H-1,3,2-benzodioxaphosphorin-4-one (1)**, (salicylchlorophosphite).

This low-melting, crystalline substance² is highly effective for the preparation of 5'-protected-2'-deoxynucleoside-3'-hydrogen phosphates.



These nucleoside hydrogen phosphates are useful intermediates for the chemical syntheses of nucleic acids because the required internucleotide 3', 5'-phosphodiester linkage can be conveniently constructed.³



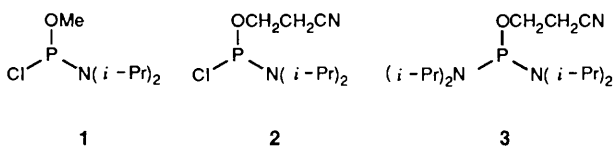
van Boom's Reagent has also been employed in the preparation of glycosyl phosphates⁴ and interglycosidic phosphodiester linkages,⁵ as well as in the preparation of modified and non-modified phosphate derivatives of biologically important oligopeptides.⁶

References:

(1) Marugg, J.E.; Tromp, M.; Kuyl-Yeheskiely, E.; van der Marel, G.A.; van Boom, J.H. *Tetrahedron Lett.* **1986**, *27*, 2661. (2) This material was first prepared by Anschutz and Emery (Anschutz, R.; Emery, W.O. *Ann.* **1887**, *239*, 301) and later by Young (Young, R.W. *J. Am. Chem. Soc.* **1952**, *74*, 1672). (3) Froehler, B.C.; Mateucci, M.D. *Tetrahedron Lett.* **1986**, *27*, 469. (4) Hermans, J.P.G.; de Vroom, E.; Elie, C.J.J.; van der-Marel, G.A.; van Boom, J.H. *Recl. Trav. Chim. Pays-Bas* **1986**, *105*, 510. (5) Westerduin, P.; Veeneman, G.H.; van der Marel, G.A.; van Boom, J.H. *Tetrahedron Lett.* **1986**, *27*, 6271. (6) Kuyl-Yeheskiely, E.; Tromp, M.; van der Marel, G.A.; van Boom, J.H. *ibid.*, in press.

32,412-4	2-Chloro-4H-1,3,2-benzodioxaphosphorin-4-one (1, van Boom's Reagent)	25g \$18.00
10,001-3	4,4'-Dimethoxytrityl chloride, 98%	5g \$20.05; 25g \$65.40
85,500-6	Thymidine, 99 + %	1g \$5.80; 5g \$23.10 25g \$79.25
T7,260-5	Trimethylacetyl chloride, 99% (pivaloyl chloride)	100g \$8.60; 500g \$26.60

Phosphoramidite Reagents for solid-phase DNA synthesis



Currently, the most useful method for the synthesis of deoxyoligonucleotides¹ employs a coupling process which utilizes the phosphite-triester protocol² modified through the use of phosphoramidite reagents (e.g., 1-3)³ and a silica-based support to anchor the nascent polymer. The major advantage of the use of phosphoramidite reagent **1**, first introduced by Caruthers,³ over the previously used chlorophosphites is stability, hence selectivity, as well as ready activation for coupling by tetrazole.⁴ The reagent produces oligonucleotides with methyl-protected phosphotriester groups.

Of growing importance is a new technology recently described by Köster⁵ based on 2-cyanoethyl protection using nucleoside phosphoramidites prepared with reagent **2**. The cyanoethyl protecting group is removed during the extremely mild reaction (aqueous ammonia, 50°C) used for deprotection of the heterocyclic bases, and cleavage of the deoxyoligonucleotide from the inert support.⁵ Its use thus eliminates the need for the harsher conditions normally used in the removal of methyl protecting groups. It has been pointed out that under prolonged reaction times in basic media methyl-protected deoxyoligonucleotides in the phosphotriester form can methylate thymine at the N-1 position.⁶

Very recently, **2** was employed successfully in the phosphitylation of the anomeric hydroxyl function of sugar derivatives in the preparation of biologically important glycosyl derivatives.⁷

Cyanoethyl phosphoramidite **3** was used to prepare solutions of deoxynucleoside-3'-phosphoramidites *in situ*.⁸ The solutions were then applied directly to a solid-phase synthesizer affording oligonucleotides containing 16-25 bases.⁸

References:

(1) Caruthers, M.H. *Synthesis and Applications of DNA and RNA*; Narang, S.A., Ed.; Academic Press, Inc.: Orlando, 1987; pp 47-94. (2) Letsinger, R.L.; Lunsford, W.B. *J. Am. Chem. Soc.* **1976**, *98*, 3655. (3) McBride, L.J.; Caruthers, M.H. *Tetrahedron Lett.* **1983**, *24*, 245. (4) Caruthers, M.H. *Science* **1985**, *230*, 281. (5) Köster, H. *et al. Tetrahedron Lett.* **1983**, *24*, 5843. (6) Jones, R.A. *et al. Nucleic Acids Res.* **1985**, *13*, 573. (7) van Boom, J.H. *et al. Tetrahedron Lett.* **1986**, *27*, 1211. (8) van Boom, J.H. *et al. Nucleic Acids Res.* **1986**, *14*, 7391.

26,252-8	N,N-Diisopropylmethylphosphoramidic chloride (1, methyl N,N-diisopropylchlorophosphoramidite)	1g \$11.45; 5g \$37.40
30,230-9	2-Cyanoethyl N,N-diisopropylchlorophosphoramidite (2)	1g \$18.50; 5g \$66.00
30,599-5	2-Cyanoethyl N,N,N',N'-tetraisopropylphosphorodiamidite, 97% (3)	500mg \$18.00; 1g \$30.00
24,395-7	1H-Tetrazole, 99 + % , GOLD LABEL	1g \$14.75 5g \$60.70
15,569-1	1H-Tetrazole, 98%	1g \$9.30; 5g \$40.90 25g \$128.05



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