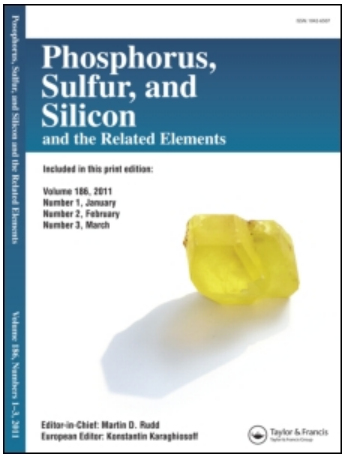

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Synthesis and NMR-Spectroscopical Studies of Fluorinated Arylmethyl-Phosphinic Acid Derivatives

Nils P. Osthaus[a]; Gerhard Hägele[a]
[a] Institute of Inorganic Chemistry and Structural Chemistry I; Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany

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SYNTHESIS AND NMR-SPECTROSCOPICAL STUDIES OF FLUORINATED ARYLMETHYL-PHOSPHINIC ACID DERIVATIVES

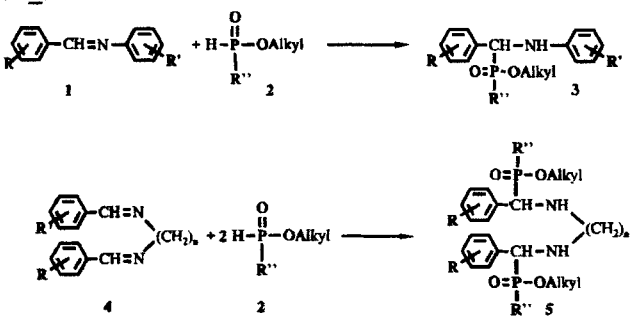
NILS PATRIK OSTHAUS and GERHARD HÄGELE
Institute of Inorganic Chemistry and Structural Chemistry I; Heinrich-Heine-Universität Düsseldorf, Universitätsstraße 1, 40225 Düsseldorf (Germany)

INTRODUCTION

Since Horiguchi in 1959 isolated CILIATIN from rumen protozoa the class of amino phosphonic and phosphinic acids has generated widespread interests directed towards synthetical problems and biological applications such as insecticides, fungicides, virostatica and enzyme inhibitors. Recent efforts have been devoted to more convenient preparations and stereospecific aspects. Here we wish to report on a facile method for the synthesis of C-fluorinated aminoalkane-mono- and bis-phosphinic acids and corresponding derivatives.

RESULTS AND DISCUSSIONS:

C-fluorinated imines react readily with aryl- or alkyl phosphonous acid esters without any catalyst at temperatures between 90 and 110°C to yield phosphinic acid esters of type 1 or 2.



3: R = H, 3,4-F_2, 2-F, 3-F, 4- F, 4-CF_3; R' =H, 2-F, 3-F, 4-F, 4-CF_3; R" = CH_3, C_2H_5, C_5H_6; Alkyl = CH_3, C_2H_5, n-C_4H_9;

5: R = 3,4-F_2, 2-F, 3-F, 4-F; n = 2, 3; R" = CH_3, C_2H_5; Alkyl = C_2H_5, n-C_4H_9

Ortho substituents on the aromatic residues give rise to extended reaction times and a decrease of the yields. The crude products are crystallised from unpolar solvents. Compounds **3** exist as pairs of diastereomers, which in fortunate cases are separated by crystallisation. Cleavage of 1 by the Bromosilane method lead to the free acids. NMR deduced the existence of 2-4 stereoisomers of esters **5**. Extensive ^{1}H-, ^{13}C-, ^{19}F- and ^{31}P-NMR in 1D- and 2D-techniques deduced purity and structures of **3** and **5**.