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Methods for Obtaining Aromatic 1, 3-Diketones Based on Fluoralkyl Substituents

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Abstract:

The article presents the results of studying the cis-enol structure of 1,3-diketones using NMR and PMR spectroscopy.

Keywords: Aroyltrifluoroacetylmethanes, NMR, PMR, cis-enol structures, tautomeric forms, 1,3-diketones, β-diketones, polyketones, fluoroalkyl-containing compounds.

INTRODUCTION

Polyfluorinated unsymmetrical 1,3-diketones as objects These studies seem to be unique [1–11] and previously systematically a scientific study of the properties of which has not been undertaken. As examples The following compounds were synthesized by the reaction:

LITERATURE REVIEW. The literature review summarizes the state of the art in the study of methods for the synthesis of fluorinated β-dicarbonyl compounds, various derivatives of 1,3-ketoesters with both linear and cyclic structures, as well as complex compounds of mono- and dinuclear structure with transition metals based on them, such as diamagnetic and paramagnetic nature. For homobinuclear complex compounds, possible antiferromagnetic exchange interactions between paramagnetic ions and an approximate estimate of the exchange integral 2 J within the framework of the theory are discussed Heisenberg–Dirac–Van Vleck (HDVF) diketones with a fluoroalkyl substituent in the cis-enol tautomeric structure synthesized in the article have been proven based on their corresponding formulas.

DISCUSSION OF RESULTS. Polyfluorinated unsymmetrical 1,3-diketones as objects of study seem to be unique [1] and the properties of which have not previously been systematically studied. As examples, the following compounds were synthesized by the reaction diketones with a fluoroalkyl substituent in the cis-enol tautomeric structure synthesized in the article have been proven based on their corresponding formulas.

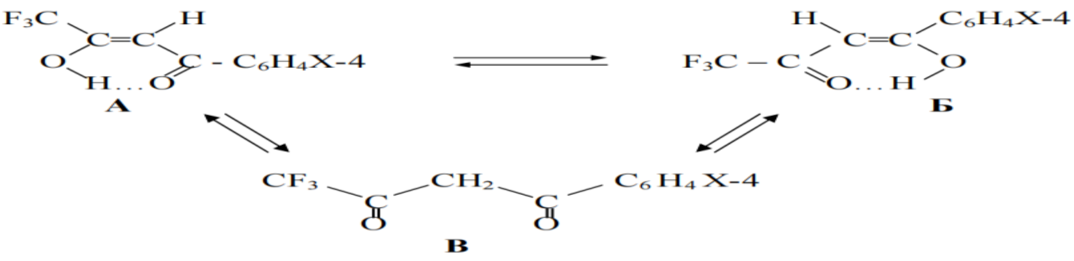
DISCUSSION OF RESULTS. Polyfluorinated unsymmetrical 1,3-diketones as objects of study seem to be unique [1] and the properties of which have not previously been systematically studied. As examples, the following compounds were synthesized by the reaction:

$$R^FCOOC_2H_5 \quad + \quad CH_3COC_6H_4X\text{-}4 \xrightarrow{CH_3ONa} R^FCOCH_2COC_6H_4X\text{-}4$$

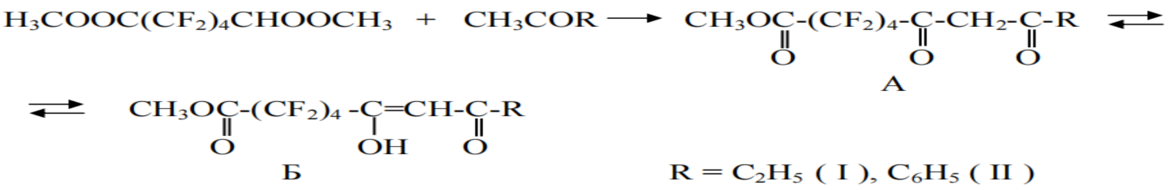
$$R^F = CF_3,\ C_2F_5,\ C_3F_7; \quad X = H,\ CH_3,\ CH_3O,\ Cl,\ NO_2$$

Aroyltrifluoroacetylmethanes exist in solutions as conjugated enols, which are clearly detected by NMR [3]. In specially filmed PMR spectra of these compounds do not contain signals corresponding to diketone form [2].

Previously performed spectroscopic studies these β-diketones also indicate an equilibrium between the two cis-enol structures A and B, and these tautomeric forms are flax in comparable quantities [4]:



The interest in the synthesis of such compounds is explained by the fact that they are starting materials for the production of fluorinated polyketonovo [7], where the authors studied the interaction of perfluoro-dimethyl ether adipic acid with



$$H_3COOC(CF_2)_4CHOOCH_3 + CH_3COR \longrightarrow CH_3O\underset{\underset{O}{\|}}{C}\text{-}(CF_2)_4\text{-}\underset{\underset{O}{\|}}{C}\text{-}CH_2\text{-}\underset{\underset{O}{\|}}{C}\text{-}R \rightleftarrows$$

$$\rightleftarrows CH_3O\underset{\underset{O}{\|}}{C}\text{-}(CF_2)_4\text{-}\underset{\underset{OH}{|}}{C}{=}CH\text{-}\underset{\underset{O}{\|}}{C}\text{-}R$$

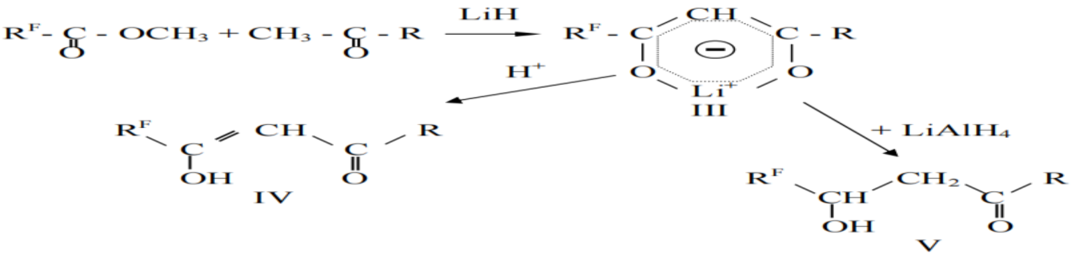
A

Б

$R = C_2H_5$ (I), C_6H_5 (II)

Study of the structure of the obtained compounds by PMR spectroscopy troscopy showed that the content of the enol form for compound I cois 90%, and for II – 84%.

The authors [8] propose original methods for obtaining fluoroacid-containing compounds. Unlike conventional condensation reactions Claisen [3,10] authors use lithium hydride as a catalyst instead of sodium alcoholate . The resulting lithium salts of diketones in the form of freecolored powders (III) in high yields, easily when exposed to acids transform into β-diketones (IV), and when processing the reaction mass, tetralithium hydridealuminate is reduced to β-hydroxyketones (V):



Compounds V can be synthesized in high yields when R = C_6H_5. At R = Alk, the formation of a large number of side reaction products [8, 10], in particular, regioisomeric β- hydroxyketones, α,β-enones, β-aminovinyl ketones.

Selection of a deputy (alkyl, aryl, fluoroalkyl), solvent and reaction conditions are suitable The reduction can be directed towards the formation of oxyketone V [8].

Condensation reaction of 1,3-dicarbonyl compounds with aldehydes (Knoevenagel condensation) is widely used in organic synthesis for obtaining various substances [6, 7, 9].

CONCLUSION.

1. Due to the preparative simplicity, high yields and availability of starting compounds, the most acceptable known method for the preparation of 2-polyfluoroacylcycloalkanones is Claisen condensation;
2. The structure of these 1,3-dicarbonyl compounds was studied in to some extent only on the example of 2-trifluoroacetyl-substituted representatives leaders of this series.

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