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DEVELOPMENT OF A TECHNOLOGICAL SCHEME FOR THE SYNTHESIS OF PHENYL-3-METHYLPHENOXYPROPIONATE BY REACTION OF PHENYL-2-CHLOROPROPIONATE WITH 3-METHYLPHENOL

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Abstract

The article presents nucleophilic substitution reactions of phenyl-2-chloropropionate with 3-methylphenol under various conditions and a method for preparing phenyl-3-methylphenoxypropionate. Nucleophilic substitution reactions were carried out in the solvents benzene, acetone, dioxane, DMF (Dimethylformamide), and DMSO (Dimethyl sulfoxide). It was found that these solvents affect the duration and yield of the reaction. In the reaction, phenyl-3-methylphenoxypropionate was obtained in a DMSO solution with a yield of 83%. A technological scheme for the synthesis of phenyl-3-methylphenoxypropionate in acetone was developed. The reason is that acetone is a cheap solvent and is convenient for the industrial production of organic substances. The structure of the synthesized new organic substance, phenyl-3-methylphenoxypropionate, was confirmed using modern IR, ^{1}HyAMR and 13CYaMR spectra.

Keywords: *organic synthesis, 3-methylphenol, reagent, nucleophile, substitution, extraction, solvent, synthesis, analysis, chloroacetylation, technological scheme*

Introduction

Chemists are conducting scientific research to find a simple way to obtain new organic substances through simple and basic organic synthesis methods and to create a technology for their production.

Currently, one of the urgent problems facing specialists is the synthesis and production of nutritional yeasts, amino acids, vitamins, enzyme preparations, antibiotics, preparations for protecting plants from pests

and diseases, etc., for the microbiology industry through organic synthesis.

Through acylation reactions of aromatic hydrocarbons, preparations against microorganisms that cause corrosion in the oil and gas industry have been synthesized, and a production technology has been created and put into practice (Ochilov M., Mamatkulov N. N., Abdushukurov A. K., 2025).

It has been found that compounds obtained by the chloroacetylation reaction of aminophenols exhibit high biological activity against gram-positive bacteria (Bacillus subtilis and Bacillus thuringiensis), gram-negative bacteria (Escherichia coli and Pseudomonas aeruginosa), and fungi Fusarium oxysporum and Botrytis fabae (Abdushukurov A. K., Yusufov M. S., 2020).

Substances synthesized by chloroacetylation reactions of phenol and its derivatives have high biological activity and are widely used in pharmaceuticals as antibacterial, analgesic, and bactericidal drugs against pathogenic microorganisms (Abdushukurov A. K., Mamatkulov N. N., Ibragimov T. E., 2023). In medicine, these include immunostimulants, drugs with antidiabetic and anticancer activity (Mamatkulov N. N., Ochilov M., Tursunova D. R. 2023; Azomova G.Z., Yuldasheva M.R., Tadjimuhamedov K.S. 2020), as well as substances with high biological activity such as adrenaline, noradrenaline, and isradipine, which have been used as hormonal drugs in pharmaceuticals (Vartanyan R.S., 2005; Sibrikov S.G., 2022). Substances synthesized by acylation reactions of phenols are used in practice as stabilizers for polymers, pesticides, and dyes (Mamatkulov N.N., Yakubov L E., 2021; Mamatkulov N.N., Yakubov L. E. Madusmanova N.K., Khoshimkhanova M.A., 2021).

Materials and methods

Synthesis of phenyl-3-methylphenoxypropionate

Experiment No. 1. In a two-necked flask equipped with a reflux condenser and a stirrer, 30 ml of absolute benzene and 5.4 g (0.05 g-mol) of 3-methylphenol were dissolved. 1.15 g (0.05 g-mol) of sodium metal, free from oxide film, was added little by little to it. After the formation of sodium 3-methylphenolate slowed down, the reaction mixture was heated in a flask heater for 4 hours. Then, 9.2 g (0.05 g-mol) of phenyl-2-chloropropionate was slowly added, and the reaction mixture was boiled for 5 hours. The reaction mixture was washed with alkaline water, extracted three times with benzene, and dried with $CaCl_2$. After the benzene was pumped out in a water pump, the product was collected in a vacuum at 215-225 °C/10 mm. sim. overhead. The yield of phenyl-3-methylphenoxypropionate was 8.2 g (64%).

Experiment No. 2. 5.4 g (0.05 g-mol) of 3-methylphenol, 6.9 g (0.05 g-mol) of potassium carbonate, and 9.2 g (0.05 g-mol) of phenyl-2-chloropropionate taken for the reaction were placed in a two-necked flask equipped with a reflux condenser and a stirrer and boiled for 9 hours. A white precipitate of potassium chloride was formed during boiling. After the reaction was complete, the precipitate was separated, and the acetone was evaporated under normal conditions. The reaction mixture was washed with 5% alkaline water and extracted with benzene. After the benzene was evaporated, the reaction product was evaporated in vacuo. The yield of phenyl-3-methylphenoxypropionate was 8.4 g (66%).

Experiment No. 4. DMF was used as the solvent, and sodium 3-methylphenolate was synthesized by the reaction of 5.4 g (0.05 mol) of 3-methylphenol with 1.15 g (0.05 mol) of sodium metal. When 9.2 g (0.05 g-mol) of phenyl-2-chloropropionate was added to it, the reaction lasted for a short time of 1.5 hours. The sodium chloride salt was filtered off from the reaction mixture, and DMF was separated by distillation under normal conditions. Then the product was washed with 5% alkaline water, extracted with benzene, and dried with $CaCl_2$. First, the benzene was distilled off, then the product. The yield of phenyl-3-methylphenoxypropionate was 9.8 g (77%).

Experiment No. 5. The amount of reagents in the reaction was taken to be 0.05 mol. Sodium 3-methylphenolate was prepared in a DMSO solution. The reaction of phenyl-2-chloropropionate with 3-methylphenol lasted for 1.5 hours. After the reaction was complete, the sodium chloride was first filtered, and the DMSO was evaporated under normal conditions. The reaction product was washed with 5% alkaline water, extracted with benzene, and dried with $CaCl_2$. First, the

benzene was evaporated, then the product. The yield of phenyl-3-methylphenoxypropionate was 10.6 g (83%).

Phenyl-3-methylphenoxypropionate is a colorless liquid, and in the hexane: ethyl acetate: chloroform system in a volume ratio of 2:1:1, a single spot was observed on silifol UV-254, Rf=0.61. Its refractive index nD^{20} =

IR spectrum of phenyl-3-methylphenoxypropionate: $\nu_{C=0}$=766; $\nu_{C=C}$=592; ν_{C-O-C}=093, 1160, 1234; δ_{CH}=688 (mono alm. ben.); δ_{CH}=750 (1,3 alm. ben.); $\delta^{s}_{CH_3}$=1454; $\delta^{as}_{CH_3}$=1488; $\nu_{=CH}$=3052; $\delta^{as}_{CH_3}$=2935,2996.

1H NMR spectrum of phenyl-3-methylphenoxypropionate: δ 1.50 (3H, d, *J* = 6.9 Hz), 2.32 (3H, s), 4.82 (1H, q, *J* = 6.9 Hz), 6.79 (1H, ddd, *J* = 2.8, 2.0, 0.5 Hz), 6.85-6.99 (2H, 6.91 (ddd, *J* = = 8.0, 2.7, 2.0 Hz), 6.93 (dt, *J* = 8.2, 2.7 Hz)), 7.13-7.35 (4H, 7.20 (ddd, *J* = 8.2, 8.0, 0.5 Hz), 7.28 (tt, *J* = 7.6, 1.3 Hz), 7.29 (dtd, *J* = = 8.1, 1.3, 0.5 Hz)), 7.47 (2H, dddd, *J* = 8.1, 7.6, 1.5, 0.5 Hz).

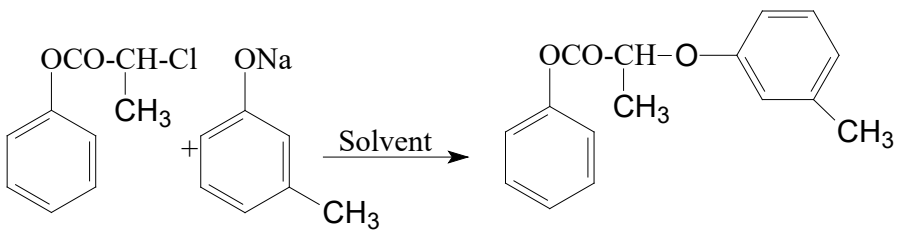
13C NMR spectrum of phenyl-3-methylphenoxypropionate: δ 17.9 (1C, s), 21.3 (1C, s), 74.3 (1C, s), 112.4 (1C, s), 116.7 (1C, s), 121.3 (2C, s), 123.5 (1C, s), 126.2 (1C, s), 129.1 (1C, s), 129.5 (2C, s), 139.5 (1C, s), 150.6 (1C, s), 158.5 (1C, s), 168.4 (1C, s).

Results and discussion

Extensive efforts are being made worldwide to synthesize effective drugs for use in agriculture and medicine based on phenol and its derivatives, which, in turn, has led to significant progress in the creation of competitive drugs based on natural raw materials and synthetic organic chemical products, which is leading to intensive development of agriculture on a scientific basis. Scientific research has been carried out to synthesize similar competitive compounds.

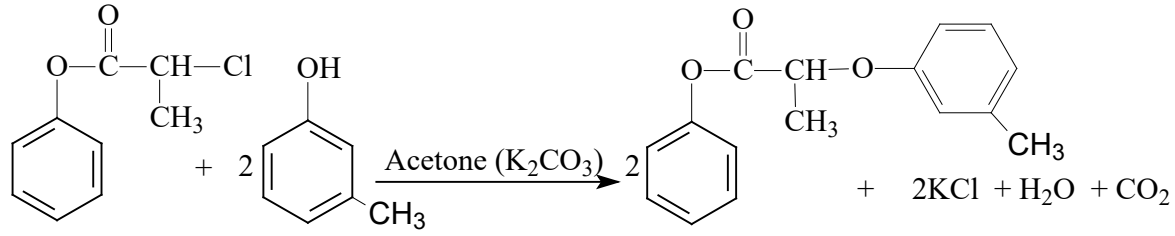
Since there is no information in the literature on the nucleophilic substitution reactions of phenyl-2-phenoxypropionate with 3-methylphenol, this reaction was carried out for the first time. Benzene, acetone, dioxane, DMF DMSO were used as solvents. The reaction for the formation of phenyl-3-methylphenoxypropionate proceeded according to the following scheme:

$$3\text{-}CH_3\text{-}C_6H_4OH + 2Na \xrightarrow{\text{Solvent}} 3\text{-}CH_3\text{-}C_6H_4OH + 2Na$$



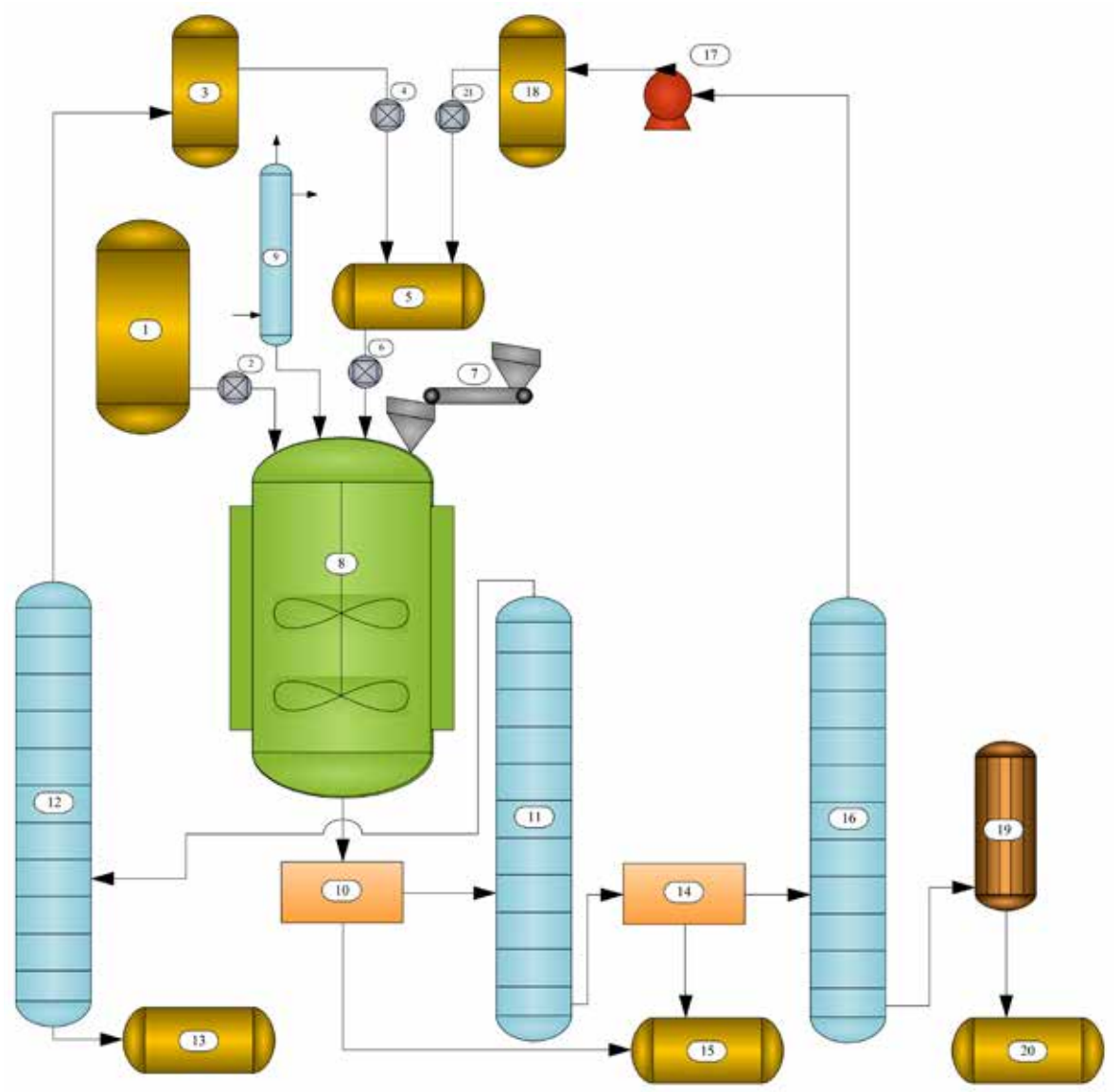
Solvent=Benzene, 1,4-Benzodioxane, DMFA,DMSO

When nucleophilic substitution reactions of phenyl-2-phenoxypropionate were carried out with 3-methylphenol in acetone solution in the presence of K2CO3, the reaction proceeded according to the following scheme.



In the nucleophilic substitution reactions of phenyl-2-phenoxypropionate with 3-methylphenol, solvents affected the yield of the resulting product. In benzene solution, the reaction yield was 64%, while in DMSO solution, it was found that phenyl-3-methylphenoxypropionate was formed with a yield of 83%.

Figure 1. *Technological scheme for the synthesis of phenyl-3-methylphenoxypropionate in acetone solution*



The technology of nucleophilic substitution reaction of phenyl-2-phenoxypropionate with 3-methylphenol in acetone solvent to form phenyl-3-methylphenoxypropionate was developed.

The synthesis of phenyl-4-methoxyphenoxypropionate was carried out according to the following technological scheme. Phenyl-2-chloropropionate is fed from the tank (pos 1) through a rheometer (pos 2) into a reactor (pos 8) equipped with a mixer. From the tank for acetone (pos 3) through a rheometer (pos 4), 4-methoxyphenol and acetone are fed from the mixing unit (pos 5) through a rheometer (pos 6) into the reactor (pos 8). Crushed K_2CO_3 is fed from the ribbon saturator (pos 7) through a funnel into the reactor (pos 8). During the reaction, volatile substances formed are cooled in a cooler (pos 9) installed at the top of the reactor (pos 8). The reaction mixture enters the filter (pos. 10) and from there passes to the distillation column (pos. 11).

From the top of the distillation column (pos. 11), the acetone and water mixture is sent to the next distillation column (pos. 12). From the distillation column (pos. 12), acetone is returned to the process, and the remaining water is collected in the water tank (pos. 13). The reaction mixture purified from acetone and water is transferred from the distillation column (pos. 11) to the

filter (pos. 14) and collected in the KCl tank (pos. 15). The reaction mixture is sent from the filter (pos. 14) to the distillation column (pos. 16). From the top of the column, 4-methoxyphenol is sent to the tank (pos. 18) via a pump (pos. 17) and 4-methoxyphenol is returned to the process. The remaining phenyl-4-methoxyphenoxypropionate from the distillation column (item 16) was purified by vacuum evaporation (item 19) and collected in a phenyl-4-methoxyphenoxypropionate (item 20) container.

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