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Synthesis of o-tolylphenoxyastatin through a nucleophilic substitution reaction from o-toluoyl chloride and examination of it's biological activity

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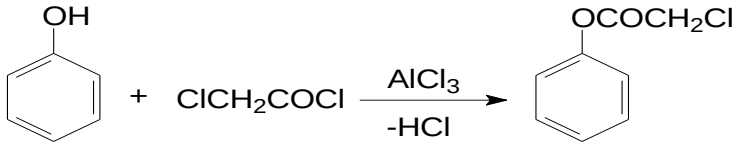
Abstract

o-Kresol was first carried out in the presence of catalytic iron salts and the composition of the resulting product was determined. The reaction mechanism suggested. Research has been conducted to synthesize the individual substance and a method for the synthesis of o-tolylchloracetate in benzene solution has been developed. Reactions with phenol under different conditions were carried out in order to synthesize a new organic substance. As a result of the research, a new organic substance o-tolylphenoxyacetate was synthesized and its structure was proved by physicochemical methods. This substance has been proven to have biological activity.

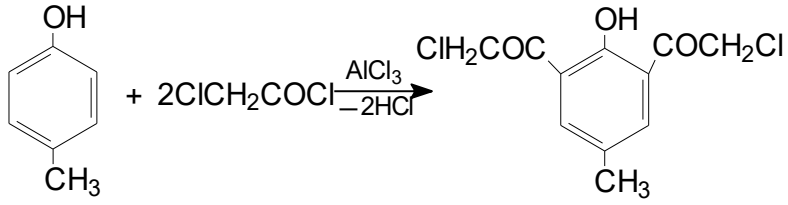
Keywords: o-kresol, phenol, acidification, catalyst, temperature, chloraacetyl, chloride, isomer, gas-liquid chromatography, nucleophilic, solvent, synthesis, biologic, vacuum, bactericidal, fungicide, extraction

Introduction

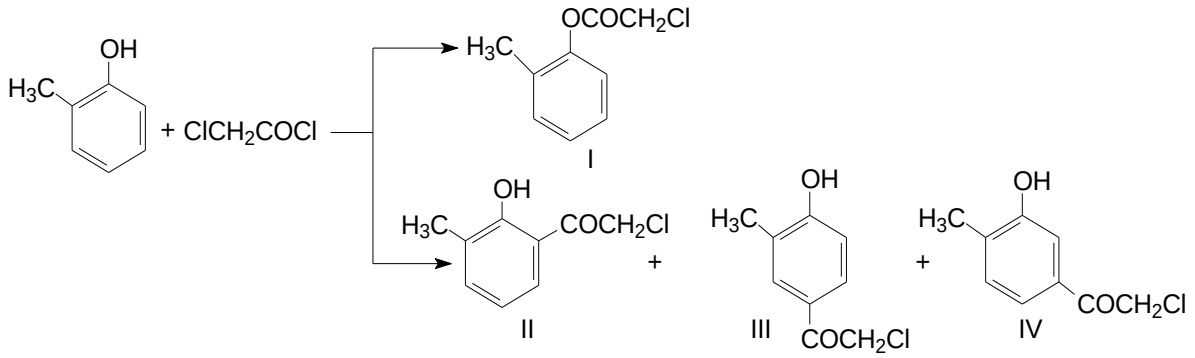
German scientists use phenol chloracetylation reaction with the participation of $AlCl_3$, at 0^0 CS_2 solutions and went to O-acylation response they determined the formation of phenylchloracetate [1].



Chloracetylation of British scientists p-kresol with the participation of $AlCl_3$ In the reaction monoacyl product formed 2-hydroxy 5-methylphenacylchloride be discovered when the amount of catalyst is increased 26% diacylproduct 2,6-dixloratsetyl-4-methylphenol is formed [2].



Chloracetylation of o-kresol, according to literature reactions, are not studied at all. therefore, these reactions are rare theoretical and practical aspects of the study with the participation of catalysts in quantities it is necessary; the result of the chloracetylation reaction of o-kresol is as follows combination can be formed [3].

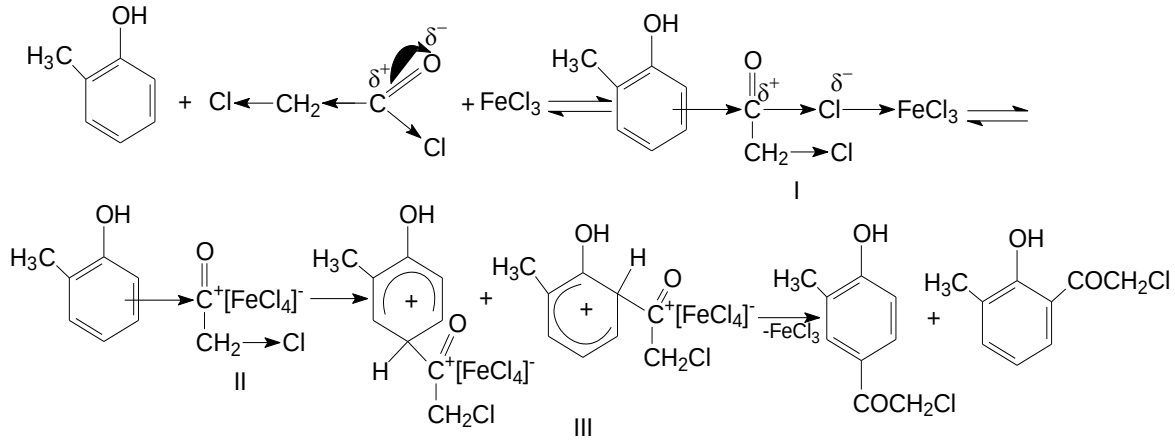


Theoretically O–acylation reaction in the first direction o-tolylchloroacetate (I) is formed.

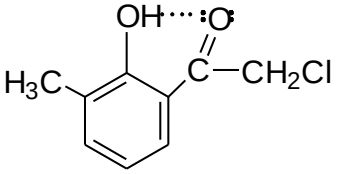
In the second direction, gidroxycetons go C-acylation reaction (2-hydroxyfrom-3-methylphenacylchlorides (II), 4-hydroxyfrom-3-methylphenacylchloride (III) and 3-hydroxy-4-methylphenacyl chloride (IV)) s can be formed.

We took o-kresol under the same conditions as the chloracetylation reaction we went. o-Kresol catalytic catalysts $FeCl_3$, $FeCl_3*6H_2O$, $ZnCl_2$ was conducted with the participation of $Fe_2(SO_4)_3$ and TAA. Chloracetylation product to determine the composition of its thin layer chromatography (CUQX) and gas-liquid was analyzed using chromatography (GSX). Reaction the resulting o-tolylchloroacetate (I),(2-hydroxy-3-methylphenacylchloride (II) and 4- hydroxyl-3-methylphenacylchloride (III) mixture of was formed.

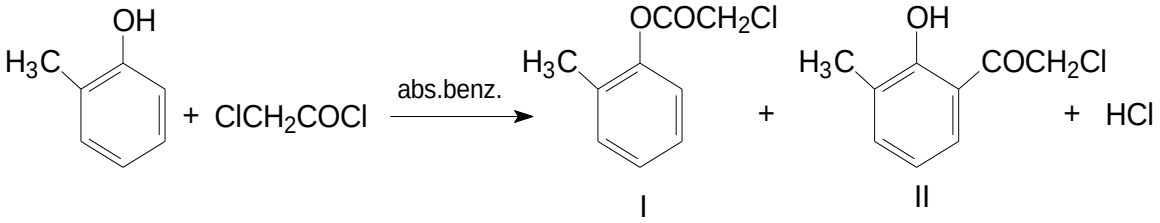
C-Acidification reaction with the participation of catalytic catalysts goes reaction mechanism $FeCl_3$ example 2-hydroxy-3-methylphenacylchloride and 4-hydroxy-3-methylphenacylchloride formation mechanism suggested as follows we did in the response of o-kresol with chloracetylchloride initially the double bond of the ring on account of p-complex (I-II) is formed. Then para and ortho concerning the hydroxyl group in cases, c-complex (III)is formed. During the reaction, the catalyst decomposes, and as a product, a mixture of gidroxycetos is formed.



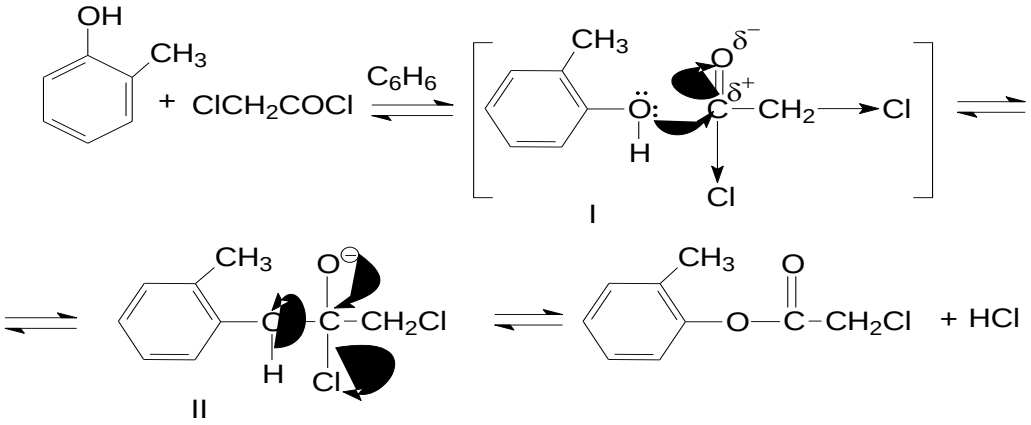
In reactions with the participation of catalysts, 2-hydroxy-3-there will be a lot of methylphenacyl chloride because of this; the response is high-temperature rise and yield of hydrogen bond in a C-acylation product as a result of the high-temperature stability of this isomer too we made the conclusion that the cause is and described it as follows synthesis of o-tolylchloracetate and exchange of nucleophile on its basis to carry out their reactions, research was carried out.



As a result of rectal when chloracetylating o-kresol in a solution of absolute benzene, two different substances; o-tolylchloracetate (I) 95% and 2-hydroxy-3-methylphenacylchlorides (II) 5% are formed;

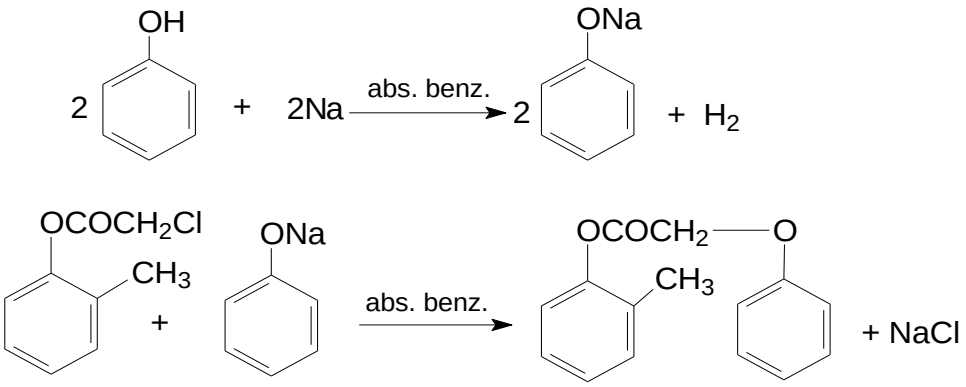


With the help of colonic chromatography, o-tolylchloroacetate separated from this mixture—the study of nucleic acid exchange reactions to allocate the required amount of o-tolylchloroacetate. o-Kresol O-acylation reaction mechanism has been proposed [4]. It can be described as follows



O-Chloracetylchloride reacts with kresol and chloracetyl chloride toward the electron density electric oxygen in the molecule, and oxygen will have a partial negative charge. Electromagnetic chlorine and due to the action of oxygen atoms, the carbon fraction is partially positive. It has an account and doubles the hydroxyl group in the phenol molecule it interacts with electrons and forms. During the formation of a valence bond between oxygen and carbon II complex formed and separated from it the yield of the reaction with hydrogen chloride, it turns out.

m-Kresol, phenol, and p-kresol chloratsetyl products nucleophile synthesis of biologically active substances exchange reactions conducted [5-7]. Research to obtain a sense with this activity carried out, synthesis of new importance based on o-tolylchloroacetate and its nucleophilic substitution with phenol to study biological properties reactions were conducted and a new organic substance o-tolylphenoxyacetate it was synthesized.



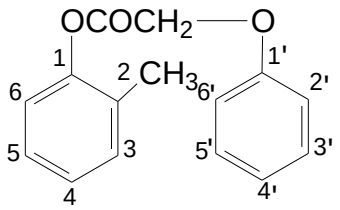
Conditions of Obtaining GSX, IR,-, PMR-spectra

Analysis of o-kresol in chloracetylation products chromium-41 equipment was done. These column lengths are 2.5 m, diameter 3 mm, cellulite -545 with 17% polyethene glycol filled thermostat temperature 170 ℃ evaporator temperature 250 ℃, hydrogen speed 20 Ml/min. In 50% chloroform solution received.

IR-spectrum of the broadcast synthesized based on o-tolylchloroacetate Phillips Pye Unicum SP3-080 marking equipment of the firm Pye pail with potassium bromine Halida was taken.

PMR spectrum 1)INM-4N-100MGS 2)Tesla 567 100 MGS 3)varint XL-100 from TMS and GMDs internal standards in MGS (solvent CCI4,CD3CI)equipment written using.

Table 1. IR-PMR spectra of synthesized o-tolylphenoxyacetate

o-Tolylphenoxyacetate structural formula	
IR spectrum(v,CM)	$\nu_{C=O}$=1782, $\nu_{C=C}$=1608, 1589, ν_{C-O-C}=1086, 1159, 1253, δ_{CH}= 689, 722, δ_{CH}= 755, $\nu_{=CH}$=3063
PMR spectrum (BM)	δ_{Ar-CH_3} = 2,85 (c) δ_{COCH_2} =4,90 (c), δ_{Ar-CH} = 6,76-7,50 (m)

o-Tolylphenoxyacetate biological activity. In Uzbekistan, one of the technical crops in the Republic is cotton. In recent years root rot and gummosis in Goose varieties due to climate change diseases have been widespread. This leads to a decrease in productivity, A substance synthesized by the gummous disease inhibitor micro organism –mustard to the bacterium malvacearum, and black root microorganism that causes disease. Basicola black root rots the condition against was tested.

Table 2

№	Name of article	Stopping the growth of microorganisms zone % on account	
		X. malvacearum E (bactericidal)	Th. B(fungicidal)
1.	o-Tolilfenoxiate	44,0	50,0
2.	Comparison “fentiuram”	41,2	45

The test result was compared to the comparator synthesized because tolylphenoxy acetate is active in bactericidal and fungicidal properties.

Experimental Results

Experiment №1

a) 10,8 g (0,1 mol) o-kresol 11,3g (0,1g mol) chloracetylchloride, 0,025g $FeCl_3$ mixture (beef-1,5•10^{-4}g) heated for 130 hours at 140 ℃ 2 hour. Hydrogen o-kresol, which did not react after the release of chloride gas, stopped separating. The main product is 170-180 ℃/28mm in a vacuum; the sim top drive was also received. Product flour 14,9 g (81%).

Gas-liquid chromatography shows that the reaction yield is 51% o-tolylchloroacetate 37%, 2-hydroxy-3-methylphenacylchloride and 12%-4-hydroxy-3-methylphenacylchloride it turned out that it consists of.

b) 10,8 г (0,1 mol) o-kresol 5,65 g (0,05 g-mol) chloracetylchloride 0,012 g $FeCl_3$ mixture (7.4•10^{-5}g mol) heated for 14 hours at 140-150 ℃. Reaction after completion in the vacuum, first o-kresol and then the product 170-180 ℃ /28 mm the sim top were also expelled. Product flour 7g (77%)

Gas-liquid chromatography shows that the reaction yield is 70% o-tolylchloroacetate, 18% 2-hydroxy-3-methylphenacylchloride and 12 % 4-hydroxy-3-methylphenacylchloride turned out that it consists of.

Experiment №2

a) Return installed in the pipe adapted to hydrogen chloride output 5,4 g (0,05g-mol) o-kresol 5,65 g per round tube, equipped with a refrigerator a mixture of G (0,05g-mol) chloracetylchloride and 50 ml of absolute acetone boiled for 20 hours. After the end of the reaction, acetone is expelled; the main product is 175-180 s/30 mm in a vacuum; the sim to drive is obtained 5 g (54%) the product was separated.

When the composition of the products is checked with GSX, the ketone fraction is 81% o-tolylchloroacetate 7% 2-hydroxy-3-methylphenacylchloride 12%-4-hydroxy-3-methylphenacylchloride showed that it consists of.

b) 5,4 g (0,05 g –mol) o-kresol per equipped tube similar to the above 5,65g (0,05 g-mole) chloracetylchloride and 50 ml abs. benzene is injected 30 hours during boiling until the output of hydrogen chloride is finished. The mixture was twice washed in alkaline water and twice in ordinary water. Benzene extraction done dried at $CaCl_2$. Benzene was driven by a water pump, a substance in a vacuum. The composition of this substance is from 95% o-tolylchloroacetate, according to GSX, and 5%-2-hydroxy-3-methylphenacylchloride consists of.

Study of reaction product using thin layer chromatography the incidence of o-kresol chloracetylation reaction product was studied and to isolate it a system was found. System CCl_4; $CHCl_3$ =1;1 from the volume

ratio formed the reaction yield was chromatogram, and there were two stains on it the presence was determined. Above yellowish stain R_f=0.68, lower murky stain R_f=0.26 equal to o-tolylchloroacetate colonic chromatography method with R_f=0.68 (CCl_4; $CHCl_3$=1;1) separated using silica gel.

Experiment №3

a) Two-mouthed tubes with a return refrigerator and mixer set 3,57g (0,038g mol) of phenol was injected and dissolved in 50 ml of absolute benzene. His sodium metal slightly purified from 0,9 g (0,038 g-mol) oxide membrane on top it's swinging. As soon as the reaction slows down, the mixture is heated in a water bath. In this hungry, a bruise appeared, then drip 7 g (0.038g-mol) o-tolylchloroacetate was put in and the Belshteyn sample reaction mixture washed in alkaline water, benzene is driven in a water pump, the product is in a vacuum he was driven. o-tolylphenoxyacetate flour 5,52g (60%) .

b) 25 ml of DMFA 3,57 g (0.038 g-mol) phenol was injected into the tube, and on top of it 0.9 g (0.038 g-mol) sodium metal purified from oxide stains it is swinging. During the reaction, the solution entered a reddish colour. Reaction 20 minutes continued. The reaction mixture is cooled to 7 g (0.038 g-mol) o-tolylchloroasetate slightly infused, and the reaction mixture is boiled for 7 hours. At the end of the reaction, the product was filtered and separated from the DMFA. Then the reaction mixture was washed in 10% alkaline water and extracted in benzene with $CaCl_2$. It was dried. o-tolylphenoxy acetate expelled in the vacuum after benzene expelled received-substance flour 6,62g (72%).

Calculated% C 74,36; H 5,80 $C_{15}H_{14}O_3$

FOUND % C 73,50 H 5,40.

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