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CONDENSATION REACTIONS OF HETEROCYCLIC SULFUR COMPOUNDS

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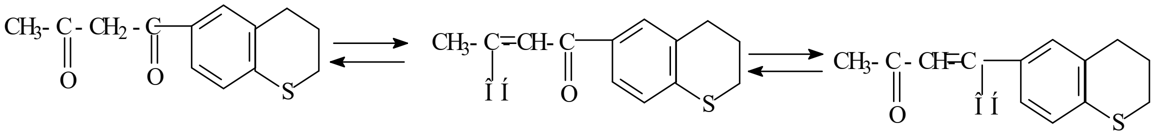
Abstract: Condensation reactions of sulfur bicyclic organic compounds with circaethyl ether were studied. as a result of the reaction, it was found that compounds of the acetylthiochroman series react with circaethyl ether in the same way as aliphatic and aromatic ketones, and as a result of the reaction, the corresponding thiochromanoylacetones are formed with good yield.

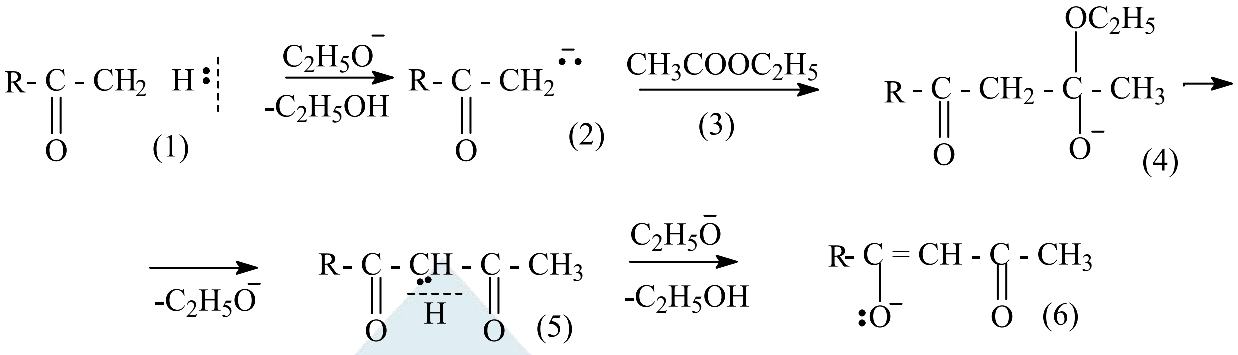
Keywords: thiochroman, acylthiochroman, circaethyl ether, diketone, sodium thiochromanoylacetone, filtrate, desiccator, electron donor, electron acceptor, carbonyl group, ketone, absolute ether, charge, reagent.

It is known that the condensation reactions of complex esters with ketones lead to the formation of β-diketones. Most ketones can readily undergo enol tautomerization in the presence of bases or acids if they have hydrogen atoms on the α-carbon atom relative to the keto group. For diketones and ketones, the amount of enols in equilibrium is stabilized by the additional unsaturation groups in their enol form. The amount of enols formed from ketones strongly depends on the structure of the ketone and the nature of the solvent used.

It was found that the appearance of β-diketones in the enol form is higher in solution than in the keto form. A similar pattern is observed in β-diketones of the 1-thiochroman series.

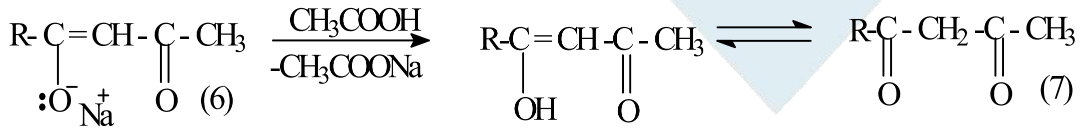


The resulting compounds are crystalline substances, soluble in organic solvents, but insoluble in water. The mechanism of the condensation reaction of ketones of the acetylthiochroman type with circaethyl ether can be expressed in the following order:



where: R= thiochromene molecule.

The catalyst (ethylate anion) removes a proton from ketone (1), which is a methylene component. The resulting highly reactive carbanion (2) attacks the partially positively charged carbon atom of ethyl ether (3) of acetic acid, which is a carbonyl component. As a result, anion (4) is formed, and thiochromanoylacetone (5) is obtained due to the separation of the ethylate ion. This compound exists as the enolate form (6) in alkaline media. The reaction mixture is acidified to isolate thiochromanoylacetones:



The structure of the synthesized compounds was determined using IR- and PMR-spectra, and the composition was determined using the method of elemental combustion analysis. Monitoring the progress of the reaction and determining the purity of the obtained substances was carried out using the thin-layer chromatographic method on the "Silufol" plate.

In the PMR-spectrum of 2-Methyl-1-thiaindanoylacetone (I), the protons of the methyl group in the sixth position are singlet at 1.27 m.u. show their signals in , the signals of methylene group protons are 2.13 m.u. in the form of a complex multiplet. and 2.32 m.u. give their signals, while the signals of protons in the aromatic ring are 8.28 m.u. and 8.32 m.u. appears. The characteristic aspect of the spectrum of this substance lies in the presence of a strong internal molecular hydrogen bond in compounds with this structure. As the acidity of β-diketones in the form of enol increases, the shift of the signals of the hydroxyl group shows a shift towards the dark region. A similar appearance is shown by the enol-form molecule of 1-thiochroman β-diketones in the complex multiplet form of the proton in the hydroxyl group at 2.08 m.u. manifestation and the signals of the proton in the SN group of α-carbon atom are 7.12 m.u. can be seen in their manifestations.

In the IR-spectrum of the synthesized thiochromane series β-diketones, broad absorption lines of the ON group in the form of enol can be seen in the region of 3570-3420 cm^{-1}. Also, the absorption lines belonging to the broad carbonyl group can be observed in the region of 1600 cm^{-1}, and the absorption lines in the region of 1310-1360 cm^{-1} indicate that they belong to SN3SO. It shows the characteristic absorption lines belonging to the methylene group at 1445 cm^{-1}, and the absorption lines at 1100, 1065 and 1010 cm^{-1} belong to the S=S double bond in the enol form. Absorption lines of characteristic vibrations of SN bonds in the benzene ring are observed in the region of 840 cm^{-1}, while absorption lines corresponding to valence vibrations of hydrogen atoms in the benzene ring can be observed in the region of 930 and 950 cm^{-1}. From the obtained results, it can be said that in these compounds, the lifetime of the structure in the enol form is much better than in the keto form.

The following table presents the physicochemical data of β-diketones of the thiochromane series.

Thiochromane series β-diketones
physical and chemical data

Table 1

s.n.	percentage %	Liquefaction temperature °C.	Found,%		Gross formula	Calculated,%	
			C	H		C	H
I	64	41-42	71,85 71,80	6,59 6,54	$C_{13}H_{14}O_2S$	71,61	6,46
II	56	38-39	67,91 67,87	6,58 6,55	$C_{14}H_{16}O_2S$	67,76	6,45

As can be seen from the above data, it was found that in the reaction of 6-acetyl-1-thiochroman and 6-methyl-7-acetylthiochroman molecules with circaethyl ether, the reactivity of 6-acetyl-1-thiochroman molecule is slightly higher than that of 6-methyl-7-acetylthiochroman molecule. The main reason for this can be attributed to two factors. The first factor is the steric effect of the methyl group in the 6-position. The second factor can be explained by the influence of electron-donor and electron-acceptor groups in the aromatic ring. Thus, it was found that semi-aromatic sulfur-containing bicyclic compounds, like aliphatic and aromatic ketones, are well suited for complex ether condensation reactions.

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