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FORMATION OF 5-OXO-5,6,7,8-TETRAHYDROCHROMENYLIUM AND -THIOCHROMENYLIUM SALTS FROM TRIKETONES OF THE 2-(3-OXOPROPYL)CYCLOHEXANE-1,3-DIONE SERIES

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We have worked out the optimal conditions for synthesis of 2,4-diaryl-5-oxo-5,6,7,8-tetrahydrochromenylium and -thiochromenylium salts based on triketones of the 2-(1,3-diaryl-3-oxopropyl)cyclohexane-1,3-dione series. For the first time, along with 5-oxo-substituted salts, we have obtained 5-thioxo-5,6,7,8-tetrahydrothiochromenylium salts. A necessary condition for the formation of the latter on treatment with acids and S-nucleophiles is the presence of electron-donor groups on the aryl substituents of the indicated triketones.

Keywords: 2-(1,3-diaryl-3-oxopropyl)cyclohexane-1,3-diones, 5,6,7,8-tetrahydrochromenylium salts.

Triketones of the 2-(3-oxopropyl)cyclohexane-1,3-dione series containing phenyl substituents are not converted to 5-oxotetrahydrothiochromenylium salts when treated with hydrogen sulfide in the presence of protic and aprotic acids. The process is limited by formation of 5-oxo-5,6,7,8-tetrahydro-4H-thiochromenes, which we explained by the insufficient hydride mobility of the hydrogen atom in the 4 position of this heterocycle [1,2]. In this connection, we were interested in determining the effect of electron-donor groups in aryl substituents of the indicated triketones on their behavior under the same conditions.

With this objective, we studied the ratio of 2-(3-oxopropyl)cyclohexane-1,3-diones **1a-f** to hydrogen sulfide in the presence of protic acids (perchloric, trifluoroacetic), and also hydrogen chloride and boron trifluoride etherate. We found that only triketones **1a-e**, containing methoxy groups in the phenyl substituents, reacting with hydrogen sulfide in glacial acetic acid medium in the presence of perchloric acid or boron trifluoride etherate, can be converted to the corresponding salts: 5-oxo-5,6,7,8-tetrahydrothiochromenylium perchlorates (**3a,c-e**) or tetrafluoroborates (**5c-3**). In the case of triketones **1b,d**, we see not only heterocyclization with formation of a thiochromenylium salt but also substitution of the oxygen of the carbonyl group by a sulfur atom in the cation of the latter, as a result of which 5-thioxo-5,6,7,8-tetrahydrothiochromenylium perchlorates **4b,d** or tetrafluoroborates **6b,d** are formed. However, the yields of salts **3-6** under the described conditions are 8%-31%.

Considering that acetic anhydride together with acids results in an active hydride ion acceptor, the acylium cation, we studied the reaction of triketones **1a-c,e** with hydrogen sulfide in a mixture of acetic anhydride and glacial acetic acid (1:4). As a result, we established the feasibility of obtaining the corresponding salts **3a,c,e, 4b**,

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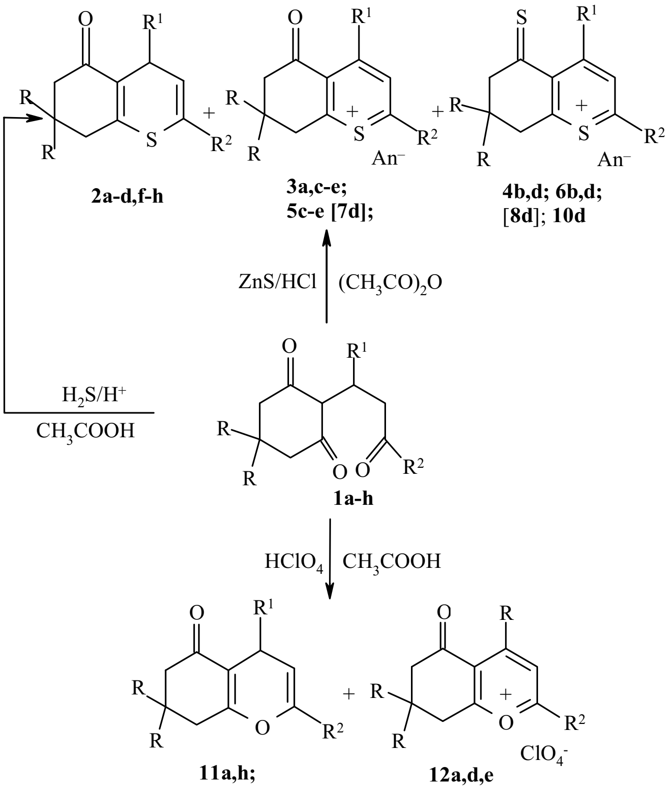
5c,e, and **6b** in preparative yields (33%-88%) (Table 1). When triketones **1a-d** were reacted with hydrogen sulfide in the presence of boron trifluoride etherate or perchloric acid, along with the corresponding tetrafluoroborates **5c-d**, **6b,d** and perchlorates **3a,c,d**, **4b,d,** we isolated 5-oxo-5,6,7,8-tetrahydro-4H-thiochromenes **2a-d** in 22%-36% yields from the ether filtrates after separation of the salts by column chromatography on aluminum oxide (hexane–ether as the eluent).



a,b,c,f,g R = H; **d,e,h** R = CH_3 ; **a** $R^1 = C_6H_5$, $R^2 = C_6H_4OCH_3$-4;
b,d $R^1 = C_6H_4OCH_3$-4, $R^2 = C_6H_5$; **c,e** $R^1 = R^2 = C_6H_4OCH_3$-4; **f** $R^1 = C_6H_5$, $R^2 = CH_3$;
g,h $R^1 = R^2 = C_6H_5$; **3,4** $An^- = ClO_4^-$; **5,6** $An^- = BF_4^-$; **7,8** $An^- = Cl^-$;
9,10 $An^- = Cl^- \cdot ½ ZnCl_2$

The results obtained provide a basis for assuming that reactions of triketones **1a-e** with hydrogen sulfide proceed through a stage of formation of 5-oxotetrahydro-4H-thiochromenes, which are converted to the corresponding tetrahydrothiochromenylium salts. This is also supported by the conversion of 5-oxo-tetrahydro-4H-thiochromene **2a** when treated with 70% perchloric acid in Ac_2O–AcOH (1:4) medium to 5-oxotetrahydro-thiochromenylium perchlorate **3a** in 62% yield. The reaction of triketone **1d** with hydrogen sulfide in the presence of hydrogen chloride in glacial acetic acid or a mixture of acetic acid and acetic anhydride also proceeds with formation of 5-oxotetrahydro-4H-thiochromene **2d** and 5-oxo- (**7d**) and 5-thioxotetrahydrothiochromenylium (**8d**) chlorides. But we could not separate the latter by treatment of the reaction mixture with excess absolute ether or hexane, and accordingly they were identified by conversion to the corresponding tetrafluoroborates **5d** and **6d**.

When triketone **1f**, containing phenyl and methyl substituents, is treated with hydrogen sulfide in the presence of perchloric acid in glacial acetic acid or trifluoroacetic acid medium, only the corresponding 5-oxo-5,6,7,8-tetrahydro-4H-thiochromene **2f** is formed in 29% and 34% yield; in this case, no salt formation occurs.

A reaction that is certainly of interest is the reaction of triketones **1d,g,h** with *in situ* generated hydrogen sulfide, in which zinc sulfide in acid medium is used as the source of hydrogen sulfide. The reaction of 1,5-diketones with zinc sulfide in acid medium is a rather simple synthesis route for six-membered cyclic sulfides [3], but the behavior of triketones of type **1** has not been previously studied under the conditions given above. We

TABLE 1. Characteristics of Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, %				mp, °C	IR spectrum, ν, cm^{-1}				Yield, %
		C	H	Cl	S		$\nu_{C=O}$	$\nu_{C=C}$	ν_{cation}	ν_{anion}	
1	2	3	4	5	6	7	8	9	10	11	12
2a	$C_{22}H_{20}O_2S$	75.9 75.8	5.7 5.8	—	9.2 9.2	107-108	1665	1620 1640	—	—	36
2b	$C_{22}H_{20}O_2S$	75.8 75.8	5.8 5.8	—	9.2 9.2	93-94	1660	1610 1640	—	—	28
2c	$C_{23}H_{22}O_3S$	73.2 73.0	5.9 5.8	—	8.2 8.4	103-105	1660	1610 1635	—	—	22
2d	$C_{24}H_{24}O_2S$	76.5 76.6	6.7 6.4	—	8.5 8.5	79-80	1675	1610 1635	—	—	27
2f	$C_{16}H_{16}OS$	75.3 75.0	6.6 6.3	—	12.1 12.5	83-85	1675	1615 1645	—	—	29*/34*^{3}
3a	$C_{22}H_{19}ClO_6S$	59.4 59.1	4.3 4.3	8.3 7.9	7.6 7.2	148-150	1710	—	1380, 1460, 1560	1100	17*/63*^{2}
3d	$C_{24}H_{23}ClO_6S$	60.8 60.7	4.9 4.9	7.2 7.5	7.1 6.8	88-90	1710	—	1380, 1470, 1560	1100	20*
3c	$C_{23}H_{21}ClO_7S$	57.8 57.9	4.9 4.4	7.5 7.4	7.1 6.7	116-119	1720	—	1380, 1460, 1550	1100	8*/48*^{2}
3e	$C_{25}H_{25}ClO_7S$	59.0 59.6	4.9 5.0	7.4 7.0	6.8 6.4	120-123	1710	—	1380, 1470, 1550	1100	14*/88*^{2}
4b	$C_{22}H_{19}ClO_5S_2$	56.8 57.0	4.3 4.1	7.6 7.7	13.9 13.8	138-140	—	—	1390, 1480, 1550	1100	29*/68*^{2}
4d	$C_{24}H_{23}ClO_5S_2$	58.7 58.7	4.8 4.7	7.0 7.2	13.4 13.1	112-114	—	—	1375, 1460, 1550	1100	15*

TABLE 1 (continued)

1	2	3	4	5	6	7	8	9	10	11	12
5c	$C_{23}H_{21}BF_4O_3S$	59.8 59.5	4.6 4.6	—	7.2 6.9	130-133	1710	—	1380, 1470, 1550	1070	31*/57*^{2}
5d	$C_{24}H_{23}BF_4O_2S$	62.2 62.3	5.2 5.0	—	7.1 6.9	125-128	1710	—	1380, 1480, 1550	1070	29*
5e	$C_{25}H_{25}BF_4O_3S$	60.6 61.0	5.1 5.1	—	6.8 6.5	98-100	1710	—	1380, 1470, 1550	1070	21*/62*^{2}
6b	$C_{22}H_{19}BF_4OS_2$	58.8 58.7	4.5 4.3	—	14.6 14.2	136-138	—	—	1380, 1470, 1550	1070	14*/33*^{2}
6d	$C_{24}H_{23}BF_4OS_2$	60.2 60.3	4.5 4.9	—	13.3 13.4	182-186			1380, 1470, 1550	1070	19*
9d	$C_{24}H_{23}Cl_2OS{\cdot}1/2Zn$	60.1 60.2	4.9 4,8	14.5 14,8	6.8 6.7	167-171	1710	—	1380, 1470, 1555	—	9
10d	$C_{24}H_{23}Cl_2OS_2{\cdot}1/2Zn$	58.0 58.2	4.6 4.7	14.1 14.3	12.6 13.0	230-234	—	—	1380, 1470, 1555	—	16
12a	$C_{22}H_{19}ClO_7$	60.9 61.3	4.4 4.5	8.7 8.2	—	203-205	1710	—	1380, 1470, 1570	1100	9*/37*^{2}
12d	$C_{24}H_{23}ClO_7$	63.0 62.8	5.3 5.1	7.7 7.7	—	143-46	1710	—	1380, 1475, 1570	1100	8*/50*^{2}
12e	$C_{25}H_{25}ClO_8$	61.1 61.4	5.3 5.2	7.8 7.3	—	177-180	1710	—	1380, 1475, 1560	1100	11*/35*^{2}

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* Yield of salt and compound **2f** in glacial acetic acid medium.

*^{2} Yield of salt in glacial acetic acid and acetic anhydride (4:1) medium.

*^{3} Yield of 5-oxo-5,6,7,8-tetrahydro-4H-thiochromene **2f** in trifluoroacetic acid medium.

found that phenyl-substituted triketones **1g,h** when treated with *in situ* generated hydrogen sulfide are converted only to the corresponding 5-oxotetrahydro-4H-thiochromenes **2g,h**, while the methoxyphenyl-substituted triketone **1d** under the indicated conditions forms a mixture of 5-oxotetrahydro-4H-thiochromene **2d** and tetrahydrothiochromenylium chlorozincates **9d** and **10d**, which were isolated in 9% and 16% yields respectively (Table 1).

The results of a study of reactions of triketones **1a-h** with hydrogen sulfide suggest that the process of salt formation under the studied conditions is possible only in the case when the starting compounds contain phenyl substituents with electron-donor methoxy groups in the 1 and (or) 3 position of the side chain. In this case, two or three carbonyl groups of the starting triketones can participate in the reaction with the sulfur reagent. Probably formation of the thiochromenylium cation occurs initially, the onium center of which increases the activity of the carbonyl group in the 5 position in nucleophilic reactions with hydrogen sulfide.

In the absence of hydrogen sulfide, triketones **1a,d,e** also react with perchloric acid with formation of salts: 5-oxotetrahydrochromenylium perchlorates **12a,d,e** and the corresponding 5-oxotetrahydro-4H-chromenes **11a,d**. In the absence of acetic anhydride, formation of salts **12a,d,e** occurs very slowly and in fairly low yields (8%-11%); carrying out the same reaction in a 1:4 acetic anhydride–acetic acid mixture increases the yield of perchlorates **12a,d,e** up to 35%-50%. Evidently in this case, the acylium perchlorate also promotes formation of the 5-oxotetrahydrochromenylium cation.

The composition and structure of the synthesized compounds are supported by elemental analysis data and IR spectroscopy. Compounds **2g,h, 11a,d** were identified from the melting point of a mixed test sample with samples obtained earlier in [1]. The IR spectra of compounds **2a-d,f** agree well with literature data [1] for sulfides **2g,h**. We should assign the absorption bands in the 1660-1675 cm^{-1} region to vibrations of the conjugated carbonyl group; the stretching vibration bands for the double bonds of the thiopyran ring have weaker intensity, and are found in the 1610-1620 cm^{-1} and 1635-1645 cm^{-1} region (Table 1).

In the IR spectra of salts **3a-e, 4b,d, 5c-e, 6b,d, 9d, 10d** we can distinguish intense absorption bands in the region 1375-1390, 1460-1480, 1550-1560 cm^{-1}, which are assigned to vibrations of the tetrahydrochromenylium cation; and the band at 1460-1480 cm^{-1} is split. Intense absorption bands at 1470-1485, 1590-1600 cm^{-1} correspond to the tetrahydrochromenylium cation in compounds **12a,d,e**. The broad intense band in the 1100 cm^{-1} region in the IR spectra of the salts **3a-e, 4b,d, 12a,d,e** corresponds to absorption by the ClO_4^- anion; the strong absorption at 1070 cm^{-1} in the spectra of the tetrafluoroborates **5c-e, 6b,d** is due to the presence of the BF_4^- ion.

The presence of an absorption band at 1710-1720 cm^{-1} in the spectra of compounds **3a-e, 5c-e, 9d** suggests retention of the carbonyl group. The shift of the 1660-1675 cm^{-1} $\nu_{(C=O)}$ absorption band in the IR spectra of oxotetrahydro-4H-thiochromenes **2a-d** toward lower frequency compared with $\nu_{(C=O)}$ 1710-1720 cm^{-1} in the salts **3a-e, 5c-e, 9d** can be explained by the presence in the former of conjugation of the π-bond with the free electron pairs of the sulfur atom.

In the IR spectra of compounds **4b,d, 6b,d, 10d** we see no absorption in the carbonyl group absorption region (1650-1750 cm^{-1}).

EXPERIMENTAL

The IR spectra were taken on a UR-20 spectrometer in nujol mulls and perchloro-1,3-butadiene. The course of the reaction was monitored by TLC on Silufol UV-254 plates in the system 2:1:1 hexane–ether–chloroform, visualized by iodine vapors.

Reaction of Triketones 1a-e with Hydrogen Sulfide and Perchloric Acid (or Boron Trifluoride Etherate) in Glacial Acetic Acid. A suspension of the starting triketone **1** (0.01 mol) in glacial acetic acid (50 ml) was saturated for 2 h with hydrogen sulfide at 20°C, and then 70% perchloric acid (0.04 mol) or boron trifluoride etherate (0.04 mol) was added. Saturation with hydrogen sulfide was continued for five more hours and it was allowed to stand for 15-20 h until conversion of the starting triketone was complete (monitored by TLC). Then the reaction mixture was poured into dry ether (500 ml), the precipitate of the salt was separated and washed with dry ether and then dried in air. After reprecipitation from chloroform with ether, we obtained the corresponding 5-oxo-

5,6,7,8-tetrahydrothiochromenylium salt (**3a,c-e, 4, 5, 6**). The ether filtrate, after separation of the salt, was washed with water and dried with Na_2SO_4, and the ether was driven off. The corresponding 5-oxo-5,6,7,8-tetrahydro-4H-thiochromene **2** was isolated from the remaining oil by column chromatography on aluminum oxide (eluent hexane).

When triketone **1b** reacts with hydrogen sulfide in the presence of perchloric acid or boron trifluoride etherate under the described conditions, we obtain only 5-thioxo-5,6,7,8-tetrahydrothiochromenylium perchlorate **4b** or tetrafluoroborate **6b**. Triketone **1d** under the same conditions forms mixtures of 5-thioxo-5,6,7,8-tetrahydrothiochromenylium perchlorate **3d** or tetrafluoroborate **5d** with 5-thioxo-5,6,7,8-tetrahydrothiochromenylium perchlorate **4d** or tetrafluoroborate **6d**. The perchlorates **3d** and **4d** or the tetrafluoroborates **5d** and **6d** were separated by fractional reprecipitation from chloroform with dry ether.

The characteristics of the synthesized compounds are presented in Table 1.

The reaction of triketones **1a,c-e** with hydrogen sulfide and perchloric acid (or boron trifluoride etherate) in a mixture of glacial acetic acid and acetic anhydride, 4:1, was carried out by the procedure given above. The yields of the corresponding salts **3a,c-e, 4b, 5c,e, and 6b** are given in Table 1.

Reaction of 2-[1-(4-Methoxyphenyl)-3-oxo-3-phenylpropyl]-5,5-dimethylcyclohexane-1,3-dione (1d) with Hydrogen Sulfide and Hydrogen Chloride in Glacial Acetic Acid. A suspension of triketone **1d** (10 g, 0.026 mol) in glacial acetic acid (100 ml) was saturated for 2 h with hydrogen sulfide at 20°C, then for 7 h with a mixture of hydrogen sulfide and hydrogen chloride. Then the reaction mixture was held at room temperature until conversion of the starting triketone was complete, then poured into absolute ether (800 ml). The precipitate formed when attempting to isolate it in air is converted to a dark oil, which was separated, washed with hexane, and treated in hexane with a three-fold molar excess of boron trifluoride etherate. The precipitate obtained of a mixture of tetrafluoroborates **5d** and **6d** was separated, washed with dry ether, and dried. This was separated by fractional reprecipitation from chloroform with hexane. The yield of salts **5d** and **6d** was respectively 16% and 1.2%. The ether phase, after removal of the oil, was washed with water and dried with Na_2SO_4, then the ether was driven off. We isolated 4.6 g (46%) of sulfide **2d** from the oily residue by column chromatography on aluminum oxide (eluent hexane).

The reaction of triketone **1d** with hydrogen sulfide and hydrogen chloride was conducted similarly in glacial acetic acid and acetic anhydride (4:1). The yields of tetrafluoroborates **5d, 6d** and sulfide **2d** were respectively 23%, 29%, and 21%.

Reaction of 2-[1-(4-Methoxyphenyl)-3-oxo-3-phenylpropyl]-5,5-dimethylcyclohexane-1,3-dione (1d) with Hydrogen Sulfide Generated *in situ*. Acetic anhydride (120 ml) was added to a mixture of ether (60 ml) and concentrated hydrochloric acid (*d* = 1.19) (30 ml) with cooling. Triketone **1d** (10 g, 26 mmol) and zinc sulfide (4.5 g, 46 mmol) was added to the solution obtained. The mixture was held for 48 h until conversion of the starting triketone was complete. The precipitate was separated, washed with chloroform and then ether, and dried. Obtained 2.1 g (16%) of 4-(4-methoxyphenyl)-7,7-dimethyl-2-phenyl-5-thioxo-5,6,7,8-tetrahydrothiochromenylium chlorozincate **10d**. The filtrate was poured into dry ether (600 ml); the precipitate formed was separated, washed with dry ether, dried, and reprecipitated from chloroform with ether. Obtained: 1.2 g (9%) of 4-(4-methoxyphenyl)-7,7-dimethyl-5-oxo-2-phenyl-5,6,7,8-tetrahydrothiochromenylium chlorozincate **9d**. The ether filtrate, after separation of salt **9d**, was washed with water and dried with Na_2SO_4, and then the ether was evaporated. 3.3 g (33%) of 4-(4-methoxyphenyl)-7,7-dimethyl-5-oxo-2-phenyl-5,6,7,8-tetrahydro-4-thiochromene **2d** was isolated from the oily residue by column chromatography on aluminum oxide (eluent hexane).

The reaction of triketones **1g,h** with *in situ* generated hydrogen sulfide was conducted according to the procedure given for triketone **1**. From triketone **1g** (10 g, 0.03 mol) we obtained 6.2 g (65%) of 5-oxo-2,4-diphenyl-5,6,7,8-tetrahydro-4H-thiochromene **2g**. From triketone **1h** (10 g, 0.029 mol) we obtained 5.5 g (55%) of 7,7-dimethyl-5-oxo-2,4-diphenyl-5,6,7,8-tetrahydro-4H-thiochromene **2h**.

2-Methyl-5-oxo-4-phenyl-5,6,7,8-tetrahydro-4H-thiochromene (2f). A. A solution of triketone **1f** (5 g, 0.019 mol) in anhydrous trifluoroacetic acid (30 ml) was saturated for 6 h with hydrogen sulfide at 20°C and allowed to stand for 15-20 h until conversion of the starting triketone was complete. Then the reaction mixture was poured into ether (200 ml). The ether solution was washed with water, dried with Na_2SO_4, and then the ether was driven off. The precipitate of thiochromene **2f** was separated, washed with alcohol, dried, and recrystallized from alcohol. Obtained 1.9 g (34%) oxotetrahydrothiochromene **2f**.

B. A suspension of triketone **1f** (5 g, 0.019 mol) in glacial acetic acid (50 ml) was saturated for 1 h with hydrogen sulfide at 20°C and then perchloric acid (*d* = 1.67) (5.7 g, 0.057 mol) was added dropwise. Then the reaction mixture was saturated with hydrogen sulfide for another five hours and allowed to stand for 15-20 h until conversion of the starting triketone was complete. After treatment of the reaction mixture by the method described in A, we isolated 1.6 g (29%) of oxotetrahydrothiochromene **2f**.

2-(4-Methoxyphenyl)-5-oxo-4-phenyl-5,6,7,8-tetrahydrothiochromenylium Perchlorate (3a). Perchloric acid (*d* = 1.67) (0.34 g, 3 mmol) was added to a suspension of 2-(4-methoxyphenyl)-5-oxo-4-phenyl-5,6,7,8-tetrahydro-4H-thiochromene **2a** (0.4 g, 1 mmol) in a mixture of glacial acetic acid (4 ml) and acetic anhydride (1 ml). This was allowed to stand for 10-15 h until conversion of the starting oxotetrahydrothiochromene was complete. Then the reaction mixture was poured into dry ether (200 ml). The precipitate of perchlorate **3a** was separated and then washed several times with dry ether, dried, and reprecipitated from chloroform with dry ether. Obtained 0.31 g (62%) of perchlorate **3a**.

2-(4-Methoxyphenyl)-5-oxo-4-phenyl-5,6,7,8-tetrahydrochromenylium Perchlorate (12a). 70% Perchloric acid (2.6 ml, 42 mmol) was added to a suspension of 2-[3-(4-methoxyphenyl)-3-oxopropyl-1-phenyl]cyclohexane-1,3-dione **1a** (5 g, 14 mmol) in of a 4:1 mixture of glacial acetic acid and acetic anhydride (50 ml). The mixture was stirred until conversion of the starting triketone **1a** was complete (monitored by TLC), then it was poured into dry ether (300 ml). The precipitate of the perchlorate **12a** formed was separated, washed with dry ether, dried, and reprecipitated from chloroform by dry ether. Yield 2.27 g (37%). The ether filtrate was washed with water, dried with Na_2SO_4, and then the ether was evaporated. 2-(4-Methoxyphenyl)-5-oxo-4-phenyl-5,6,7,8-tetrahydro-4H-chromene **11a** (1.56 g, 33%) was isolated from the oily residue by column chromatography on aluminum oxide (eluent hexane).

Perchlorates **12d,e** and tetrahydrochromene **11d** were obtained similarly.

Reaction of Triketones 1a,d,e with Perchloric Acid in Glacial Acetic Acid was accomplished by the procedure given above for reaction of triketone **1a** with perchloric acid in a mixture of glacial acetic acid and acetic anhydride. The yields of perchlorates **12a,d,e** were 9%, 8%, and 11% respectively.

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