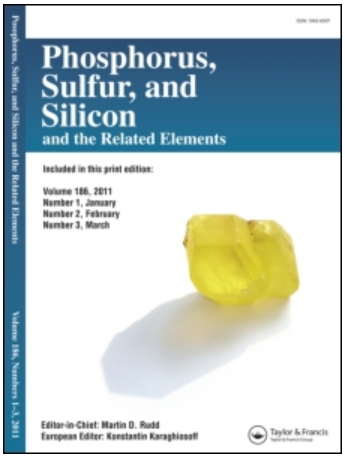

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CHLOROSULFONATION OF 9-ARYLOCTAHYDROXANTHEN-1,8-DIONES

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CHLOROSULFONATION OF 9-ARYLOCTAHYDROXANTHEN-1,8-DIONES

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*The 9-aryloctahydroxanthen-1,8-diones (**3, 4–24**) were prepared by reaction of cyclohexan-1,3-dione (**1**) with selected arylaldehydes. The xanthendiones (**4–9**, **11**, **12**, **18**, **21**, **22**) were successfully reacted with chlorosulfonic acid, and the crude sulfonyl chlorides were converted into 15 sulfonamides (**26–40**) for screening as potential pesticides. Attempted chlorosulfonation of the xanthendiones (**13–17**) was unsuccessful. α-Methylcinnamaldehyde was reacted with cyclohexandione (**1**) to yield the corresponding xanthendione derivative (**23**). On the other hand, with* o*-methoxycinnamaldehyde an impure product formed and the p-methoxy isomer afforded the corresponding 2-arylpyran (**25**). The NMR spectral data of the compounds are briefly discussed.*

Keywords: 9-Aryloctahydroxanthen-1,8-diones; chlorosulfonation; sulfonamides

Chlorosulfonic acid is extensively employed as a reagent for the sulfonation and chlorosulfonation of aromatic and heteroaromatic compounds.[1] The work described in this article forms part of our previous studies on the chlorosulfonation of heterocyclic compounds as a route to the synthesis of arylsulfonyl derivatives of potential biological activity.[2–5]

The chlorosulfonation of dimedione (5,5-dimethylcylohexan-1,3-dione) derivatives of aromatic aldehydes was studied previously.[6] The work has now been extended to the chlorosulfonation of the analogous derivatives of cyclohexan-1,3-dione and aromatic aldehydes. Cyclohexan-1,3-dione (**1**), like dimedione, is known to be a reagent for the identification of aldehydes in organic qualitative analysis.[7–10] The dione (**1**;2 moles) reacts with aldehydes (1 mole) by a Michael-type condensation to yield the 2:1 adducts (**2**); the latter are readily dehydrated by acids, yielding the corresponding octahydroxanthen-1,8-dione (**3**, Scheme 1).

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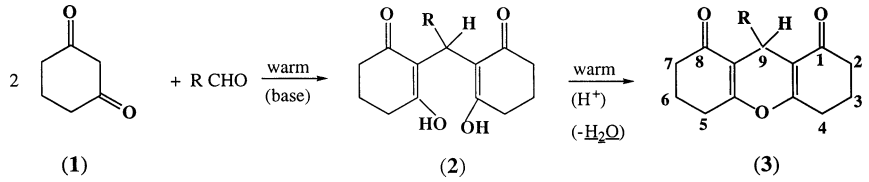


SCHEME 1

The xanthendiones (**3**) are solids; however, they do not generally form such well-defined crystals as the analogous dimedione derivatives, and consequently they are not so widely used in organic qualitative analysis.

Reaction of cyclohexan-1,3-dione with aromatic aldehydes provides a series of 9-aryloctahydroxanthendiones (**3**, R=Ar), which should be susceptible to chlorosulfonation by treatment with excess chlorosulfonic acid because they are essentially alkyl-substituted benzene derivatives which are known[1] to react with chlorosulfonic acid under comparatively mild conditions. In addition, previous studies have demonstrated that keto groups are unaffected by treatment with chlorosulfonic acid: thus hydantoins[11] and chalcones[12] have been successfully converted into the corresponding chlorosulfonyl derivatives.

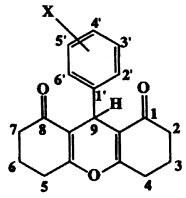
The aromatic aldehydes (1 mole) reacted with cyclohexan-1,3-dione (2 moles), in boiling aqueous ethanol-piperidine as catalyst to give the 1,2-adducts (**2**, R=Ar), which were dehydrated by heating with ethanol-concentrated hydrochloric acid under standard conditions,[13,14] yielding the 9-aryloctahydroxanthendiones (**3**, R=Ar). By this procedure, the xanthendiones (**3**) were obtained from benzaldehyde (**4**) and the following substituted benzaldehydes: 2′-methyl (**5**); 4′-methyl (**6**); 2′-, 3′-, and 3′-methoxy (**7**–**9**); 4′-fluoro (**10**); 3′-, 4′-dichloro (**11**); 2′-, 4′-, and 4′-nitro (**12**–**14**); 2′- and 4′-trifluoromethyl (**15**, **16**); and 4′-dimethylamino (**17**).

Other analogous derivatives were prepared from thiophen-2- and 3-carboxaldehyde (**18**, **19**), pyridine-4-carboxaldehyde (**20**), biphenyl-4-carboxaldehyde (**21**), and cinnamaldehyde (**22**) and α-methylcinnamaldehyde (**23**) (Chart 1).

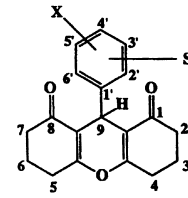
Reaction of cyclohexan-1,3-dione (**1**) with an equimolar quantity of p-methoxycinnamaldehyde afforded the pyran derivative (**25**) (Chart 1). This product was formed irrespective of the presence or absence of base (piperidine), which supports the pyran structure. On the other hand, attempted reaction of the dione (**1**) with *o*-methoxycinnamaldehyde gave an impure product (an oil).

The ^{1}H NMR spectra of the 9-aryloctahydroxanthendiones (**3**) showed that the tertiary allylic proton (9-H) resonated as relatively low-field singlet (δ 5.0 approx.) because this proton is deshielded by the combined effects of the adjacent double bond and the aromatic ring.

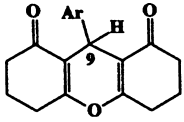
CHART I 9-Aryloctahydroxanthen-1,8-diones and their Sulfonyl Derivatives. Numbers Under the Y Heading Indicate the Position of the SO_2 Y Group.



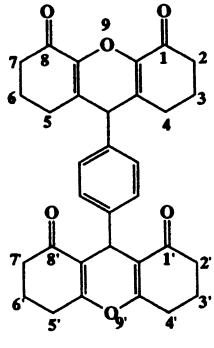
(i) excess $ClSO_3H$
(ii) Y H



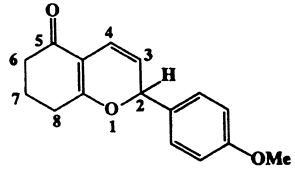
	X		X	Y
(**4**)	H	(**26**)	H	4′-NMe_2
(**5**)	2′-Me	(**27**)	H	4′-NHEt
(**6**)	4′-Me	(**28**)	H	4′-pyrrolidino
(**7**)	2′-OMe	(**29**)	2′-Me	5′-NMe_2
(**8**)	3′-OMe	(**30**)	4′-Me	5′-NMe_2
(**9**)	4′-OMe	(**31**)	2′-OMe	5′-NHEt
(**10**)	4′F	(**32**)	2′-OMe	5′-piperidino
(**11**)	3′,4′Cl_2	(**33**)	3′-OMe	4′-NMe_2
(**12**)	2′-NO_2	(**34**)	3′-OMe	4′-piperidino
(**13**)	3′-NO_2	(**35**)	4′-OMe	5′-pyrrolidino
(**14**)	4′-NO_2	(**36**)	3′,4′Cl_2	5′-NMe_2
(**15**)	2′-CF_3	(**37**)	2′-NO_2	4′-NMe_2
(**16**)	4′-CF_3	(**38**)	4′-dimethylsulfonamide of (**18**)	
(**17**)	4′-NMe_2	(**39**)	4′-dimethylsulfonamide of (**21**)	
		(**40**)	4′-dimethylsulfonamide of (**22**)	



(**18**)	2′thienyl
(**19**)	3′thienyl
(**20**)	4′pyridyl
(**21**)	4-Ph.C_6H_4
(**22**)	PhCH═CH
(**23**)	PhCH═CMe



(24)



(25)

The remaining aliphatic protons generally appeared as a broad multriplet (δ 3.0–1.2); furthermore, relatively low field resonances (δ 2.6, 2.4) could often be clearly identified corresponding to the protons of the CH_2CO and $CH_2C{=}C$ moieties, namely the 2- and 4-H protons, respectively.

Several 9-aryloctahydroxanthen-1,8-diones (**4**–**9**, **18**, **21**, and **22**) reacted with excess chlorosulfonic acid (10–12 moles) at room temperature to give the crude sulfonyl chlorides. In the majority of the compounds, the pure sulfonyl chlorides were not isolated (c.f. Cremlyn and Saunders[6]). The sulfonyl chlorides were, however, purified and characterized as the alkyl sulfonamide derivatives (Chart 1).

EXPERIMENTAL

Melting points were determined with a Gallenkamp electric apparatus and are uncorrected. IR spectra were recorded as nujol mulls using a Perkin Elmer 237 spectrophotometer. ^{1}H NMR spectra were measured with a Bruker AC 250 spectrometer using tetramethylsilane (TMS) as internal standard and $CDCl_3$ as solvent. Resonances removed by D_2O treatment are indicated by an asterisk. ^{13}C NMR spectra were recorded using a Jeol EX 270 instrument. EI mass spectra were obtained with a V.G. Micromass 16F spectrometer operating at 70 ev.

Preparation of 9-Aryl-1,2,3,4,5,6,7,8-octahydroxanthen-1,8-diones (3)

Cyclohexane-1,3-dione (5.6 g, 0.05 mole) was dissolved in warm 10% aqueous ethanol (50 ml), and then a solution of the appropriate arylaldehyde (0.025 mole) in ethanol (10 ml) was added followed by piperidine (3 drops). The solution was heated in a steam bath for 10 min. After cooling in ice water, the crystalline 1,2-adduct (**2**) separated out, filtered off, and dried. The product was dissolved in ethanol (30 ml) containing concentrated hydrochloric acid (3 drops), and the solution was refluxed on the steam bath for 30 min. When the solution was cooled (ice water), the xanthendione (**3**) crystallized out and was collected and dried (vacuum desiccator). By this procedure, the xanthendiones (**4**–**24**) were prepared. The following xanthendiones were novel:

2′-Methyl (5)

m.p. 220–222°C. ^{1}H NMR: δ 7.1–6.8 (m, 4H, Ar H), 4.82 (s, 1H, 9-H), 2.9 (s, 3H, Ar Me), 2.6–2.0 (M, 12H, alip H).

2′-Methoxy (7)

m.p. 221°C. ^{1}H NMR: δ 7.4–6.78 (m, 4H, Ar H), 4.85 (s, 1H, 9-H), 3.8 (s, 3H, OMe), 2.7 (m, 4H, 2-H), 2.5 (m, 4H, 4-H), 1.8 (m, 4H, 3-H).

4′-Fluoro (10)

m.p. 235°C $C_{19}H_{17}O_3F$ requires C, 73.1; H, 5.4. Found: C, 73.0; H, 5.6.

3′,4′,-Dichloro (11)

m.p. 243–245°C. $C_{19}H_{16}O_3Cl_2$ requires C, 62.8; H, 4.4. Found: C, 63.1; H, 4.5.

2′-Trifluoromethyl (15)

m.p. 235–236°C. $C_{20}H_{17}O_3F_3$ requires C, 66.3; H, 4.7. Found: C, 66.1; H, 5.0.

4′-Trifluoromethyl (16)

m.p. 240–242°C. $C_{20}H_{17}O_3F_3$ requires C, 66.3; H, 4.7. Found: C, 66.0; H, 4.8.

2′-Thienyl (18)

m.p. 218–220°C. ^{1}H NMR: δ 7.3–6.8 (m, 3H, Ar H), 5.2 (s, 1H, 9-H), 2.65–1.8 (m, 12H, Alip H). MS: 300 (M^+).

3′-Thienyl (19)

m.p. 208–210°C. ^{1}H NMR: δ 7.5–6.8 (m, 3H, Ar H), 4.8 (s, 1H, 9-H), 2.6–2.0 (m, 12H, Alip H). MS: 300 (M^+).

4′-Bipyridyl (21)

m.p. 196–198°C. ^{1}H NMR: δ 7.6–6.8 (m, 9H, Ar H), 4.82 (s, 1H, 9-H), 2.6 (m, 4H, 2-H), 2.35 (m, 4H, 4-H), 2.0 (m, 4H, 3-H). MS: 370 (M^+).

Cinnamaldehyde (22)

m.p. 184–186°C. $C_{21}H_{20}O_3$ requires C, 78.7; H, 6.2. Found: C, 78.4; H, 6.4. ^{1}H NMR: δ 7.35–7.1 (m, 5H, Ar H), 6.2 (d, 2H, CH=CH), 4.4 (s, 1H, 9-H), 2.7–2.0 (m, 12H, alip H). ^{13}C NMR: δ 196.7 (C=O), 164.8, 137.2, 131.1 (3 signals, alkenic C), 129.8–115.3 (6 signals, aryl C), 36.9–20.4 (4 signals, alkyl C). MS: 320 (M^+).

α-Methylcinnamaldehyde (23)

m.p. 153–155°C. $C_{22}H_{22}O_3$ requires C, 79.0; H, 6.0. Found: C, 79.3; H, 5.8. ^{1}H NMR: δ 7.4–7.1 (m, 5H, Ar H), 5.8 (s, H, CH=C(Me), 4.5 (s, 1H, 9-H), 2.8 (s, 3H, CH=C (Me), 2.6–1.8 (m, 12H, Alip H).

Terephthaldehyde (24)

m.p. 338–340°C. $C_{32}H_{30}O_6$ requires C, 75.2; H, 5.9. Found: C, 74.9; H, 6.1. ^{1}H NMR: δ 8.2–7.4 (m, 4H, Ar H, AA′ BB′ pattern), 4.9 (s, 2H, 9,9′-H), 2.6 (m, 4H, 2,2′-H), 2.4 (m, 4H, 4,4′-H), 2.0 (m, 4H, 3,3′-H).

Reaction of Cyclohexan-1,3-Dione (1) with p-methoxycinnamaldehyde

A solution of cyclohexan-1,3-dione (4.4 g, 0.02 mole) in ethanol (100 ml) was warmed with p-methoxycinnamaldehyde (3.3 g, 0.02 mole) for 30 min, and the yellow solution was left at room temperature for 1 week. Cooling in ice-water mixture afforded the 2-(p-methoxy) pyran (**25**) as yellow crystals, yield of 4.2 g (80%), m.p. 197–198°C. $C_{16}H_{15}O_3$ requires C, 75.3; H, 5.9. Found: C, 75.0; H, 6.1. ^{1}H NMR: δ 7.0–6.8 (m, 4H, Ar H, AA′ BB′ pattern), 5.0 (d, 1H, 2-H), 3.7 (s, 3H, OMe), 3.4 (d, 2H, alkenic H), 2.6–1.2 (m, 6H, alkyl H). MS: 255 (M^+).

When the reaction was carried out in the presence of base (piperidine), the identical product (**25**) was isolated.

Chlorosulfonation of the 9-Aryloctahydroxanthen-1,8-diones (4–9, 11, 12, 18, 21–22)

The 9-aryloctahydroxanthen-1,8-dione (**3**, 0.01 mole) was gradually added, with swirling, to chlorosulfonic acid (14 g, 0.12 mole) at 0°C. The solution was left at room temperature until all effervence had ceased (2–7 days) and was poured onto crushed ice (100 g) with stirring. After the ice had melted, the product was collected, washed with ice water, and dried (vacuum desiccator) to yield the sulfonyl chloride. In many cases, the crude sulfonyl chloride was used and characterized as the sulfonamide derivative.

Preparation of the Sulfonamides

The 9-(chlorosulfonylaryl)xanthendione (0.01 mole) was gradually added to a solution of the appropriate amine (0.03 mole) in ethanol (30 ml) at 0°C. The mixture was left at room temperature (2–3 days), added to ice water containing dilute hydrochloric acid, and the precipitate filtered off, washed with water, and dried. The solid product was recrystallized from ethanol to give the sulfonamide derivative.

Compound 26

m.p. 205–206°C. $C_{21}H_{23}NO_5S$ requires C, 62.0; H, 5.65; N, 3.4. Found: C, 62.3; H, 5.7; N, 3.5. ^{1}H NMR: δ 7.8–7.4 (m, 4H, Ar H, AA′ BB′ pattern),

4.85 (s, 1H, 9-H), 2.75 (s, 6H, NMe_2), 2.6–1.8 (m, 12H, alip H). MS: 407 (M^+).

Compound 27

m.p. 225–227°C. $C_{21}H_{23}NO_5S$ requires C, 62.0; H, 5.65; N, 3.4. Found: C, 61.6; H, 5.6; N, 3.5. ^{1}H NMR: δ 7.8–7.2 (m, 4H, Ar H, AA′ BB′ pattern), 5.5* (s, 1H, NH), 4.8 (s, 1H, 9-H), 3.0–1.2 (m, 17H, alip H). MS: 407 (M^+).

Compound 28

m.p. 189–191°C. $C_{23}H_{25}NO_5S$ requires C, 64.6; H, 5.85; N, 3.3. Found: C, 64.3; H, 5.8; N, 3.4. ^{1}H NMR: δ 7.85–7.1 (m, 4H, Ar H, AA′ BB′ pattern), 4.82 (s, 1H, 9-H), 3.3 (m, 4H, NCH_2), 2.62–1.2 (m, 16H, alip H). ^{13}C NMR: δ 196.5 (C=O), 164.7–146 (4 signals, alkenic C), 136–125.4 (6 signals, aryl C), 47.8–20.2 (7 signals, alip C).

Compound 29

m.p. 198–200°C. $C_{22}H_{25}NO_5S$ requires C, 63.6; H, 6.0; N, 3.4. Found: C, 63.3; H, 5.8; N, 3.5. ^{1}H NMR: δ 7.7–7.0 (m, 3H, Ar H), 4.9 (s, 1H, 9-H), 2.8 (s, 6H, NMe_2), 2.3 (s, 3H, Ar-Me), 2.6–1.8 (m, 12H, alip H). MS: 415 (M^+).

Compound 30

m.p. 205–206°C. $C_{22}H_{25}NO_5S$ requires C, 63.6; H, 6.0; N, 3.4. Found: C, 64.0; H, 9.9; N, 3.3. ^{1}H NMR: δ 7.8–7.1 (m, 3H, Ar H), 4.8 (s, 1H, 9-H), 2.8 (s, 6H, NMe_2), 2.6–1.9 (m, 12H, alip H), 2.3 (s, 3H, Ar-Me). MS: 415 (M^+).

Compound 31

m.p. 230°C. $C_{22}H_{26}NO_6S$ requires C, 61.1; H, 6.0; N, 3.2. Found: C, 60.9; H, 6.2; N, 3.3. ^{1}H NMR: δ 7.8–6.9 (m, 3H, Ar H), 5.0 (s, 1H, 9-H), 4.8* (s, 1H, SO_2NHEt), 3.8 (s, 3H, OMe), 2.9–1.5 (m, 14H, alip H), 1.2 (t, 3H, CH_2Me). ^{13}C NMR: δ 196.6 (C=O), 164.7, 160.9 (2 signals, alkenic C), 133.2–111.1 (6 signals, aryl C), 56.1–14.9 (7 signals, alkyl C).

Compound 32

m.p. 240–242°C. $C_{25}H_{29}NO_6S$ requires C, 63.7; H, 6.1; N, 3.0. Found: C, 63.5; H, 6.0; N, 3.2. ^{1}H NMR: δ 7.62–7.0 (m, 3H, Ar H), 5.0 (s, 1H, 9-H), 3.9 (s, 3H, OMe), 2.9–1.2 (m, 22H, alip H).

Compound 33

m.p. 135°C. $C_{22}H_{26}NO_6S$ requires C, 61.1; H, 6.0; N, 3.2. Found: C, 60.8; H, 5.9; N, 3.0. MS: 432 (M^+).

Compound 34

m.p. 140–142°C. $C_{25}H_{29}NO_6S$ requires C, 63.7; H, 6.1; N, 3.0. Found: C, 63.5; H, 5.9; N, 2.8. ^{1}H NMR: δ 8.1–6.2 (m, 3H, Ar H), 4.8 (s, 1H, 9-H), 3.9 (m, 3H, OMe), 3.1–1.4 (m, 22H, alip H). ^{13}C NMR: δ 196.6 (C=O), 166.5, 164.4 (2 signals, alkenic C), 135–118 (6 signals, aryl C), 56.5–20.1 (7 signals, alkyl C).

Compound 35

m.p. 208–210°C. $C_{24}H_{27}NO_6S$ requires C, 64.4; H, 3.0; N, 3.1. Found: C, 64.2; H, 2.8; N, 3.4. ^{1}H NMR: δ 7.8–6.8 (m, 3H, Ar H), 4.85 (s, 1H, 9-H), 3.8 (s, 3H, OMe), 3.3 (m, 6H, CH_2NCH_2), 2.6–1.7 (m, 16H, alip H).

Compound 36

m.p. 228°C. $C_{21}H_{21}NCl_2O_5S$ requires C, 53.6; H, 4.5; N, 3.0. Found: C, 53.4; H, 4.7; N, 3.1. MS: 473, 469 (M^+).

Compound 37

m.p. 206–208°C. $C_{21}H_{22}N_2O_7S$ requires C, 56.5; H, 4.9; N, 6.3. Found: C, 56.3; H, 5.0; N, 6.1. MS: 446 (M^+).

Compound 38

m.p. 212°C. $C_{19}H_{21}NO_3S_2$ requires C, 60.8; H, 5.6; N, 3.7. Found: C, 60.6; H, 3.5; N, 4.0. MS: 375 (M^+).

Compound 39

m.p. 191–193°C. $C_{27}H_{27}NO_5S$ requires C, 67.8; H, 5.6; N, 2.9. Found: C, 67.6; H, 5.5; N, 3.1. ^{1}H NMR: δ 7.8–6.8 (m, 8H, Ar H), 4.82 (s, 1H, 9-H), 2.85 (s, 6H, NMe_2), 2.5–1.8 (m, 12H, alip H). MS: 477 (M^+).

Compound 40

m.p. 188–190°C. $C_{23}H_{15}NO_5S$ requires C, 66.2; H, 3.6; N, 3.35. Found: C, 66.0; H, 3.9; N, 3.3. ^{1}H NMR: δ 7.3–7.1 (m, 4H, Ar H, AA′ BB′ pattern), 6.2 (s, 2H, alkenic H), 4.8 (s, 1H, 9-H), 2.9 (s, 6H, NMe_2), 2.6–2.0 (m, 12H, alip H). ^{13}C NMR: δ 196.7 (C=O), 164.8, 137.2, 131 (3 signals, alkenic C), 129.8–115.3 (6 signals, aryl C), 36.3–20.4 (4 signals, alkyl C). MS: 417 (M^+).

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