Synthesis and Characterization of New Heterocyclic Thioxanthone Derivatives

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Abstract:

This work comprises the synthesis of new thioxanthone derivatives containing C-substituted thioxanthone. To obtain these derivatives, the o-mercapto benzoic acid was chosen as the starting material, o-mercapto benzoic acid with phenoxy acetic acid / thio phenoxy acetic acid gave 2-(oxy acetic acid) thioxanthone (**1**) and 2-(thio acetic acid) thioxanthone (**2**) respectively. Treatment of (**1**,**2**) with thionyl chloride gave 2-(oxy acetyl chloride) thioxanthone (**3**) and 2-(thio acetyl chloride) thioxanthone (**4**) respectively. Compounds (**3**,**4**) were treated with hydrazine hydrate (99%) to form the hydrazide (**5**,**6**) which is the desired Chiron, the hydrazide (**5**,**6**) was used to react with phenyl isothiocyanate / phenyl isocyanate to give thiosemicarbazide (**7**,**8**) / semicarbazide (**9**,**10**) derivatives respectively, which were used in the preparation of eight types of heterocyclic derivatives.

Keywords: Thioxanthone, 1,2,4-Triazole, 1,3,4-Oxadiazole, 1,3,4-Thiadiazole.

Introduction:

Thioxanthone and derivatives are an important class of molecules and are a common heterocyclic scaffold in biologically active and medicinally significant compounds [1,2]. The thioxanthone and derivatives are the core structure of a wide variety of naturally occurring and synthetic compounds that exhibit extraordinary anti-tumor [3-5], anti-parasitic [6,7], anti-cancer activity [8,9], anti-hypertensive, anti-oxidative, anti-thrombotic [10,11] and are potential anti-cancer drugs and some thioxanthones containing plant extract are directly used in traditional medicine [4,5]. The thioxanthone [12,13] possess a number of interesting pharmacological activities [14,15]. Certain members of these classes of compounds exhibit significant anti-tumor and cytotoxic effects [16,17]. On the other hand, substituted thioxanthone are often used in industry field as surface coating, printing inks, microelectronics, photoresists, adhesives [18] and used in free radical polymerization as a photosensitizer [19-21] or photoinitiator [22], because of their absorption characteristics at near-UV range [22-25]. Silva and coworkers [26] in European food safety authority (EFSA) have found that isopropyl thioxanthone [ITX] was used as food packaging in milk products, fruit juices, fruit nectars and drinks [27].

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Materials and Methods:

General

All reactions were monitored by thin-layer chromatography (TLC) using 0.25 mm pre-coated silica-gel F254 plates, spots were detected with iodine vapour. The IR spectra were recorded on (SHIMDZU) FT-IR 8400 spectrophotometer; solid samples were run in KBr discs, Liquid samples were run as smears. UV spectra were recorded with UV-Visible spectrophotometer (CARY) UV-100 Conc. Melting points were determined on a Gallenkamp meting point apparatus with sample contained in open capillary tube in an electrically heated metal block apparatus and were un corrected. ^{1}H-NMR spectra were recorded on ultra shield 300 MHz NMR spectrophotometer in acetone-d_6 solvent and with tetra methylsilane (TMS) as an internal standard.

General procedure for preparation of 2-(oxy acetic acid) thioxanthone (1) and 2-(thio acetic acid) thioxanthone (2)

O-mercapto benzoic acid (1.6 g, 0.01 mol) was slowly added to sulfuric acid (98 %) (15 ml) and the mixture was stirred for (5 min) to ensure thorough mixing. Phenoxy acetic acid (6.7 g, 0.044 mol) was added slowly to the stirred mixture over a period of (30 min). After the addition, the reaction mixture was stirred at room temperature for (2 hrs). The product was checked by (T.L.C) using (methanol: carbon tetra chloride) (2:1), then heated at (80)°C for (2 hrs), after which it was left to stand at room temperature overnight. The resulting mixture was poured carefully with stirring into a 10-fold excess of boiling water then boiled for further (5 min). The solution was cooled, filtered and the residue was recrystallized from (dioxane / water) (1:1) to give a yellow crystal. Yield: 2.38 g, 80 %, m.p. 200-202 (dec)°C.

Compound (**2**) was prepared by the same method described for the preparation of compound (**1**) except using of thiophenoxy acetic acid (7.4 g, 0.044 mol) instead of phenoxy acetic acid. The solid product was recrystallized from (dioxane / water) (1:1) afford a green crystal. Yield: 2.5 g, 80 %, m.p.195-197 (dec)°C. Similarly the following compounds were prepared in a similar manner.

Preparation of 2-(oxy acetyl chloride) thioxanthone (3) and 2-(thio acetyl chloride) thioxanthone (4)

A mixture of compound (**1**) or **(2)** (0.001 mol) and excess of thionyl chloride (1.45 ml, 0.02 mol) and one drop of DMF were refluxed for (4 hrs). After evaporation of thionyl chloride under reduced pressure, the residue was recrystallized from carbon tetra chloride to give **(3)** a yellow-reddish crystal. Yield: 0.2 g, 66 %, m.p. 240-242 (dec)°C.

Compound (**4**). Green reddish crystal (from methanol). Yield: 0.18 g, 57 %, m.p. 237-239 (dec)°C.

Preparation of 2-(oxy acetyl hydrazide) thioxanthone (5) and 2-(thio acetyl hydrazide) thioxanthone (6)

To a solution of compound **(3)** or **(4)** (0.01 mol) in ethanol (30 ml), hydrazine hydrate (99%) (1ml, 0.02 mol) was added then the resulting mixture was refluxed on water-bath for (3 hrs). The formed precipitate was filtered and

recrystallized from toluene to give the hydrazide derivative **(5)** a yellow-reddish crystal. Yield: 2.13 g, 71%, m.p. 276-278 °C.

Compound (**6**). Dark red crystals (from benzene). Yield: 1.9 g, 60 %, m.p.116-118°C.

Preparation of N-phenyl-2-[(oxyacetyl) thiosemicarbazide] thioxanthone (7) and N-phenyl-2-[(thio acetyl) thiosemicarbazide] thioxanthone (8)

To a solution of compound (**5**) or (**6**) (0.3 g, 0.001 mol) in absolute ethanol (25 ml) phenyl isothiocyanate (0.6 ml, 0.005 mol) was added with continuous stirring and the mixture was refluxed for (4-5 hrs), then reaction mixture was cooled and the resulting solid (**7**) was recrystallized from hexane to give dark yellow crystal. Yield: 0.24 g, 53%, m.p.180-182 °C.

Compound (**8**). Black reddish crystal (from benzene). Yield: 0.22 g, 43 %, m.p.219-221°C.

Preparation of N-phenyl-2-[(oxy acetyl) semicarbazide] thioxanthone (9) and N-phenyl-2-[(thio acetyl) semicarbazide] thioxanthone (10)

Compounds (**9**) and (**10**) were prepared by the same method described for the preparation of thiosemicarbazide (**7**,**8**) except using phenyl isocyanate (0.5 ml, 0.005 mol) instead of phenyl isothiocyanate. The solid product was recrystallized from methanol afford light crystal. Yield: 0.3 g, 73 %, m.p. 190-192°C. Compound (**10**). Black crystal (from ethanol). Yield: 0.26 g, 61 %, m.p. 244-246 (dec)°C.

Preparation of N_4-phenyl-3-[(thioxanthone-2-yl) oxy methyl, thio methyl-1,2,4-triazole-5-thiol (11,12)]

Compound (**7**) or (**8**) (0.001 mol) was refluxed with (10 %) aqueous sodium hydroxide solution (30 ml) for (4 hrs). The reaction mixture was cooled, filtered and neutralized by gradual addition with stirring of (10 %) acetic acid solution. The resulting solid (**11**) was filtered and recrystallized from acetone to give brown crystal. Yield: 0.18 g, 40 %, m.p. 284-286 °C.

Compound (**12**). Pale red crystal (from ethanol).Yield: 0.16 g, 35 %, m.p. 113-115 °C.

Preparation of 5-phenyl amine-2-[(thioxanthone-2-yl) oxy methyl, thio methyl-1,3,4-thiadiazole (13,14)]

Compound (**7**) or (**8**) (0.001 mol) was dissolved in syrup of phosphoric acid (10 ml), heated at (120)°C for (50 min), kept overnight and then poured into an ice-cold water. The resulting solid (**13**) was filtered and recrystallized from ethanol to give dark yellow crystal. Yield: 0.19 g, 44 %, m.p. 223-225 °C.

Compound (**14**). Dark yellow crystal (from ethanol). Yield: 0.3 g, 68 %, m.p. 211-213 (dec)°C.

Preparation of 5-phenyl amine-2-[(thioxanthone-2-yl) oxy methyl, thio methyl-1,3,4-oxadiazole (15,16)]

Compounds (**15**) and (**16**) were prepared by the same method described for the preparation of compounds (**13,14**) except using (**9,10**) instead of (**7,8**).

Compound (**15**). Black crystal (from benzene). Yield: 0.15 g, 37 %, m.p. 136-138 °C. Compound (**16**). Brown crystal (from dioxane). Yield: 0.2 g, 50 %, m.p. 243-245 °C.

Preparation of N_4-phenyl-3-[(thioxathone-2-yl) oxy methyl, thio methyl-1,2,4-triazole-5-ol (17,18)]

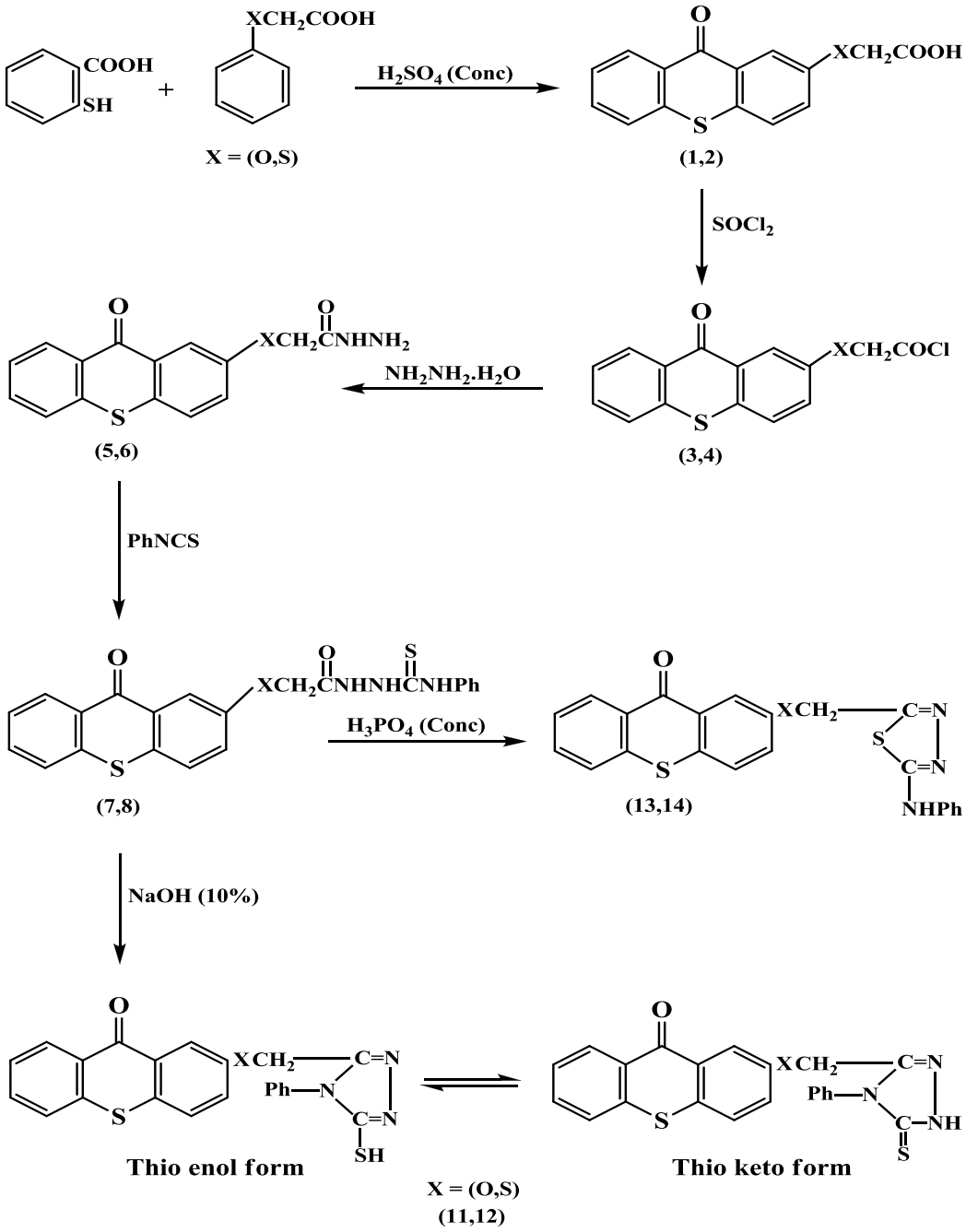
Compounds (**17**) and (**18**) were prepared by the same method described for the preparation of compounds (**11,12**) except using (**9,10**) instead of (**7,8**).

Compound (**17**). Deep-yellow crystal (from dioxane).Yield: 0.16 g, 40 %, m.p. 191-193°C.

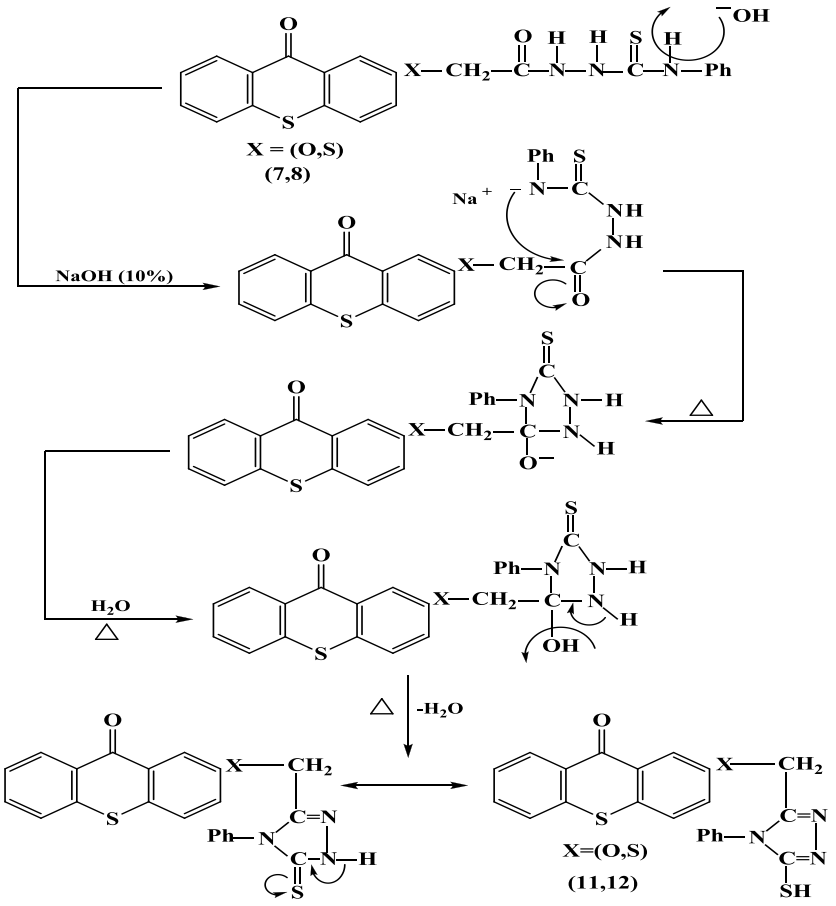
Compound (**18**). Black crystal (from ethanol).Yield: 0.17 g, 40 %, m.p.104-106 (dec) °C. Characteristic absorption bands of FT-IR and U.V spectra of oxy and thio derivatives are listed in Table (1) and (2) respectively. Table (3) represent the ^{1}H-NMR spectra for some compounds (5,12,14,17).

Results and Discussion:

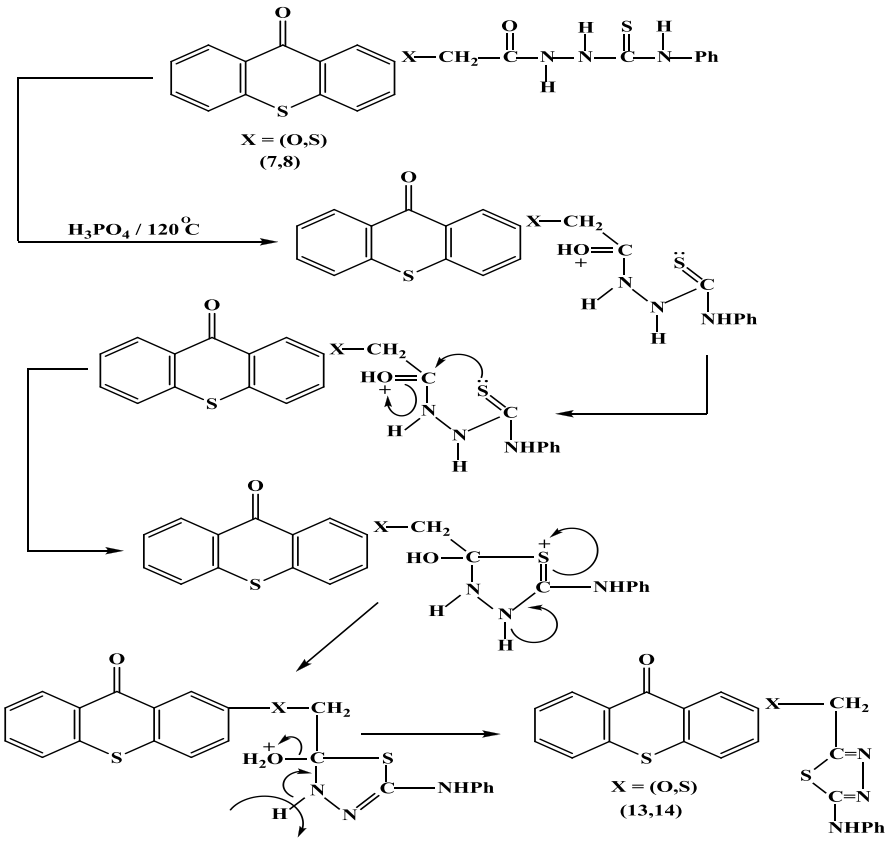
2-(Oxy acetic acid) thioxanthone (**1**) and 2-(thio acetic acid) thioxanthone (**2**) [28] were obtained from condensation of o-mercapto benzoic acid with phenoxy acetic acid and thio phenoxy acetic acid respectively in the presence of sulfuric acid (98%) with high yields and as a single isomer [23]. When the acids (**1**,**2**) were treated with excess of thionyl chloride to give oxy and thio acetyl chloride (**3**,**4**). The compounds (**3**,**4**) were reacted with hydrazine hydrate (99%) to form the hydrazide (**5**,**6**). The reaction of (**5**,**6**) with phenyl isothiocyanate and phenyl isocyanate to give thiosemicarbazide (**7**,**8**) and semicarbazide (**9**,**10**) derivatives respectively which were used in preparation of eight types of heterocyclic derivatives. Treatment of (**7**,**8**) with NaOH (10 %) under refluxing condition affected intramolecular cyclization through the loss of H_2O giving the desired 1,2,4-triazole-5-thiol derivative (**11**,**12**). When (**7**,**8**) were treated with phosphoric acid at (120)°C, it was affected by intramolecular cyclization through the loss of H_2O and giving the expected 1,2,4-thiadiazole derivative (**13**,**14**) (**Scheme 1**).


XCH2COOH
COOH
SH
+
H2SO4 (Conc)
X = (O,S)
(1,2)
SOCl2
XCH2COCl
(3,4)
NH2NH2.H2O
XCH2CNHNH2
(5,6)
PhNCS
XCH2CNHNHCNHPh
(7,8)
H3PO4 (Conc)
(13,14)
NHPh
NaOH (10%)
Thio enol form
Thio keto form
X = (O,S)
(11,12)

Scheme 1

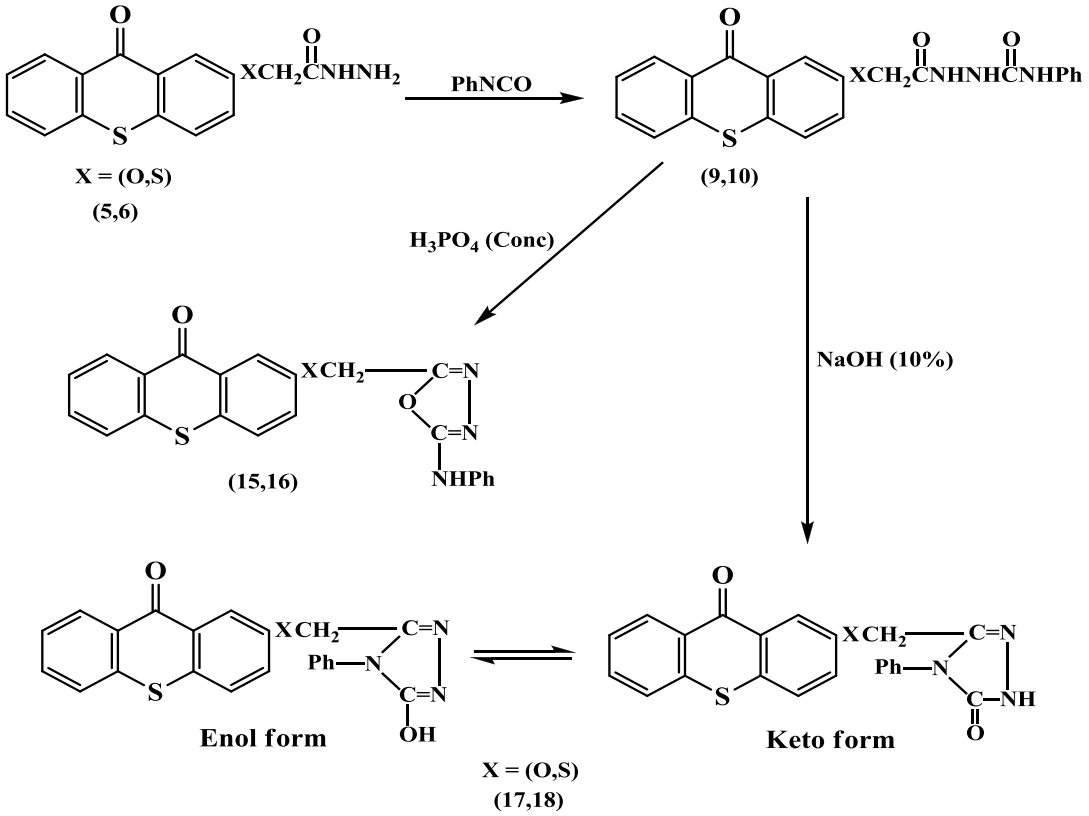


Reaction mechanism for the formation of compounds (11,12)



Reaction mechanism for the formation of compounds (13,14)

1,2,4-oxadiazole derivative (**15**,**16)** was prepared by the same method described for the preparation of compounds (**13,14**) except using (**9,10**) instead of (**7,8**). The intramolecular cyclization of (**9,10**) via reflux with NaOH (10 %) for (4 hrs) to form 1,2,4-triazole-5-ol derivative **(17,18) (Scheme 2)**.



Scheme 2

The structures of all derivatives the thioxanthone were proven on the basis of melting points (m.p.), thin layer chromatography (T.L.C) and spectral data.

Table (1): Characteristic absorption bands of FT-IR and U.V spectra of oxy-derivatives

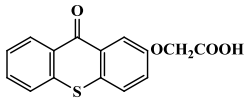
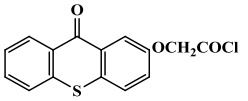
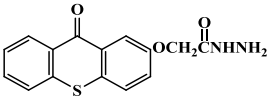
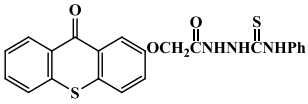
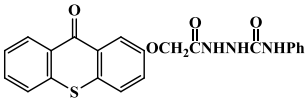
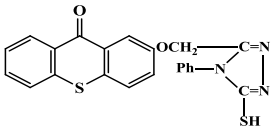
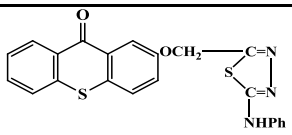
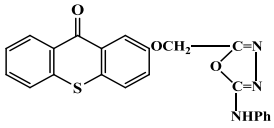
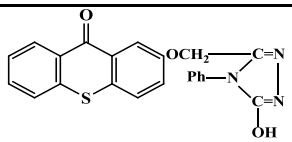
Comp. No.	Compound structure	FTIR spectral data cm^{-1}								U.V. (λ_{max}) nm
		υ(N-H)	υ(C-H) Aromatic	υ(C-H) Aliphatic	υ(C=C) Aromatic	υ(C=O) Ketone	υ(C=N) Imine	υ(C-O-C) Ether	Others	
1		-	3047	2856 2984	1585 1564 1471	1635 1719	-	1227 1054	υ(O-H) acid 3360 υ(C-O) acid 1281	391
3		-	3036	2928	1589 1441	1637 1783	-	1253 1055	υ(C-L) 663	386
5		3401 3343	3051	2933	1590 1438	1644 1685	-	1263 1080	-	387
7		3348 3304	3042 3080	2943	1545 1507 1437	1638 1681	-	1238 1078	υ(C=S) 1281	397
9		3345 3320	3056	2954	1585 1443	1644 1694	-	1227 1085	-	386
11		3361	3007 3039	2929	1574 1521 1454	1650	1617	1231 1042	υ(S-H)Thio enol form 2565 υ(C=S)Thio keto form 1256	385
13		3370	3036	2936	1561 1508 1444	1654	1631	1229 1041	-	392
15		3359	3056	2862	1590 1443	1645	1620	1245 1071	-	386
17		3299	3006 3034	2863	1562 1457	1649 1704	1617	1240 1069	υ(O-H) Enol form 3442	398

Table (2): Characteristic absorption bands of FT-IR and U.V spectra of thio derivatives

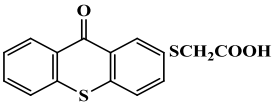
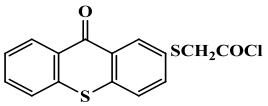
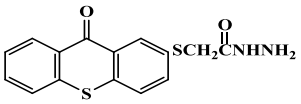
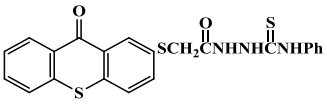
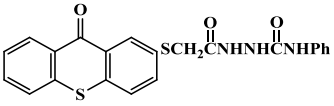
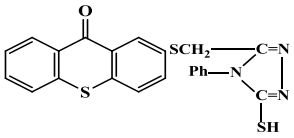
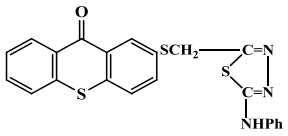
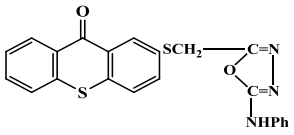
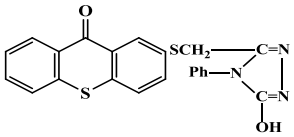
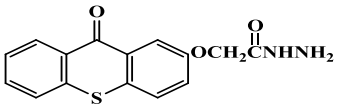
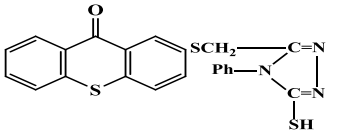
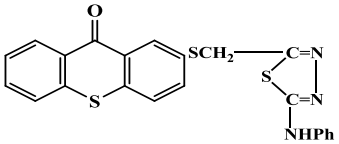
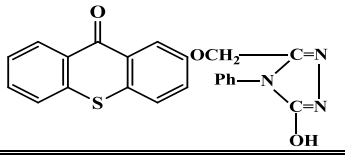
Comp. No.	Compound structure	FTIR spectral data cm^{-1}							U.V. (λ_{max}) nm
		υ(N-H)	υ(C-H) Aromatic	υ(C-H) Aliphatic	υ(C=C) Aromatic	υ(C=O) Ketone	υ(C=N) Imine	Others	
2		-	3062	2891	1589 1498	1637 1701	-	υ(O-H) acid 3268 υ(C-O) acid 1297	393
4		-	3067	2849	1588 1447	1640 1784	-	υ(C-L) 691	394
6		3363 3304	3051	2864	1592 1442	1640 1678	-	-	398
8		3347 3289	3008 3060	2858	1584 1444	1642 1677	-	υ(C=S) 1268	392
10		3329 3263	3026 3059	2849	1561 1443	1651 1683 1711	-	-	394
12		3311	3041	2857	1571 1447	1642	1616	υ(S-H) Thio enol form 2559 υ(C=S) Thio keto form 1222	387
14		3257	3032	2868	1566 1520 1447	1649	1618	υ(C-O-C) Ether 1268 (asym) 1092 (sym)	398
16		3281	3041	2853	1564 1466	1648	1615	-	397
18		3271	3073	2861	1570 1464	1641 1689	1612	υ(O-H) Enol form 3415	397

Table (3): ^{1}H-NMR spectra for some compounds

Comp. No.	Compound structure	δH aromatic ppm	δH other bands ppm
5		(7.1-8.3) (m,7H)	3.3 (s,2H) CH_2 group protons 4.9 (s, 2H) NH_2 group protons 9.3 (s,1H) (NH) group proton
12		(7.1-8.5) (m,12H)	2.6 (s,2H) CH_2 group protons 6.3 (s,1H) (NH) group proton 10.2 (s,1H) (SH) group proton
14		(7.1-8.4) (m,12H)	2.5 (s,2H) CH_2 group protons 6.1 (s,1H) (NH) group proton
17		(7.2-8.5) (m,12H)	3.2 (s,2H) CH_2 group protons 6.7 (s,1H) (NH) group proton 11.1-12.9 (br,1H) (OH) proton

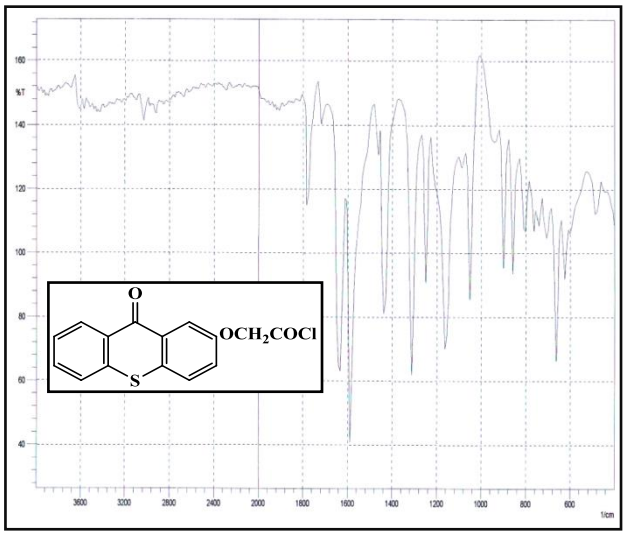


Fig.(1): FT-IR spectrum for compound (3)

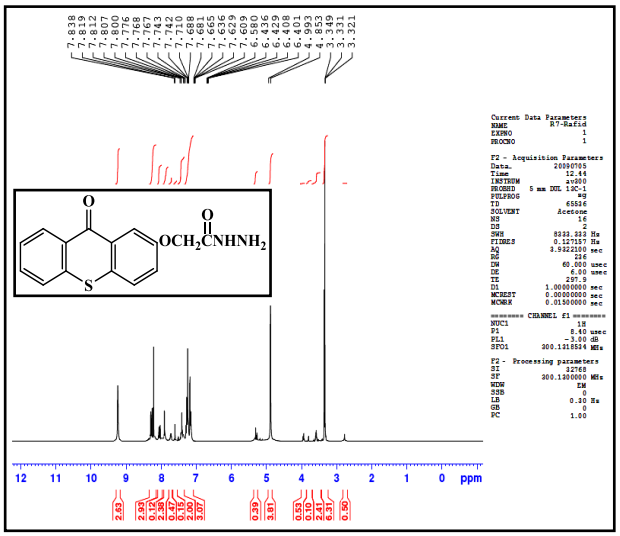


Fig.(2): ^{1}H-NMR spectrum for compound (5)

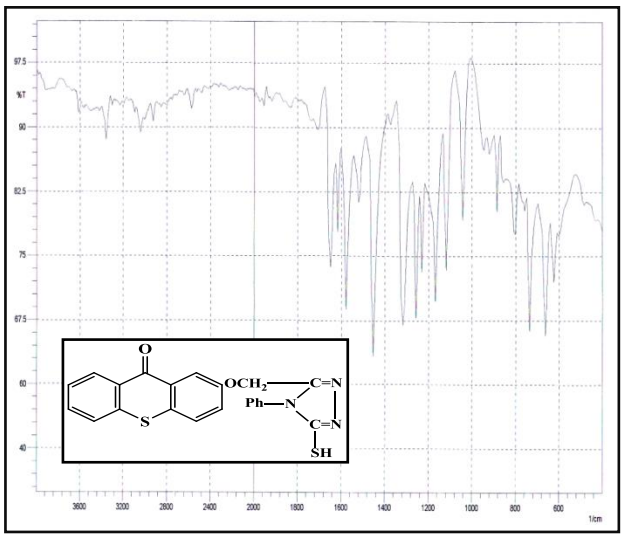


Fig.(3): FT-IR spectrum for compound (11)

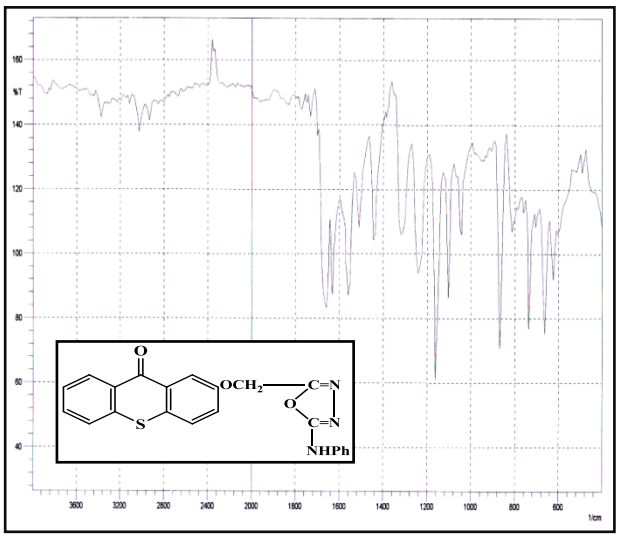


Fig.(4): FT-IR spectrum for compound (15)

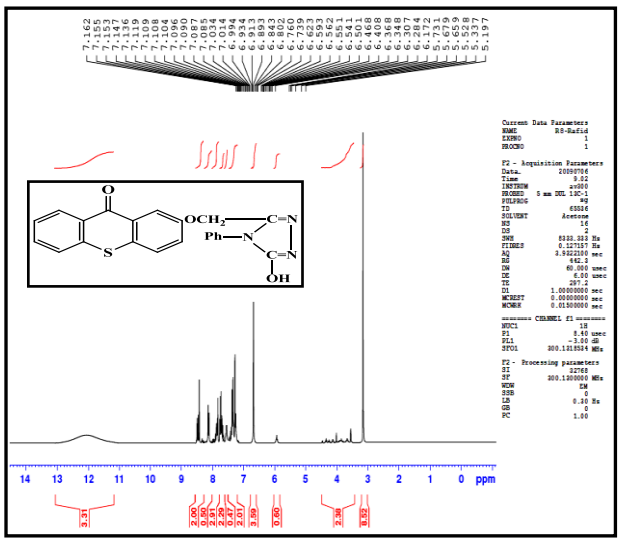


Fig.(5): ^{1}H-NMR spectrum for compound (17)

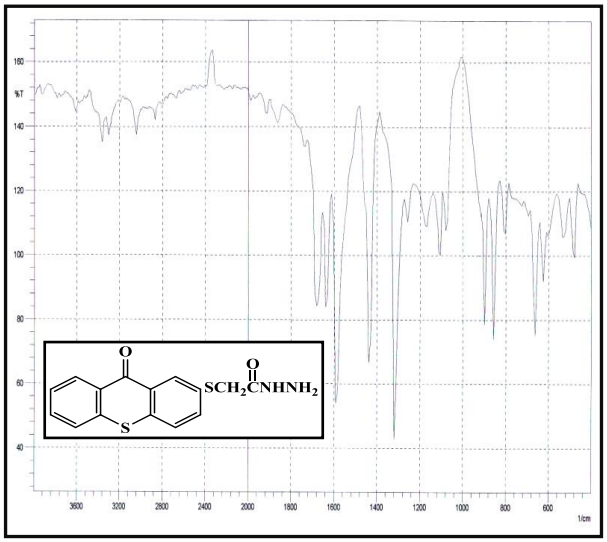


Fig.(6): FT-IR spectrum for compound (6)

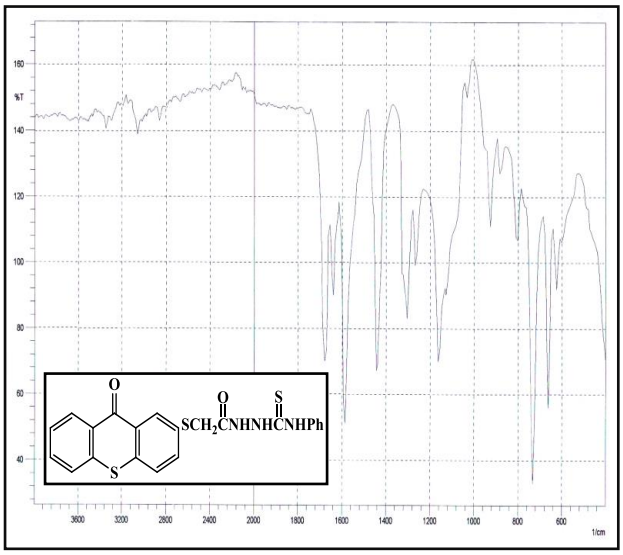


Fig.(7): FT-IR spectrum for compound (8)

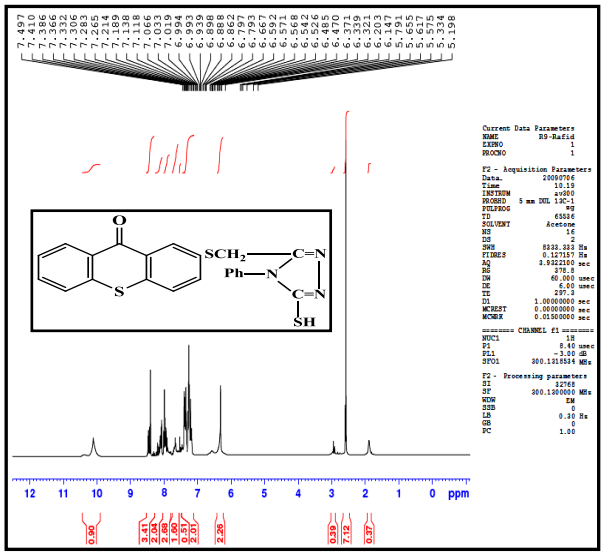


Fig.(8): ^{1}H-NMR spectrum for compound (12)

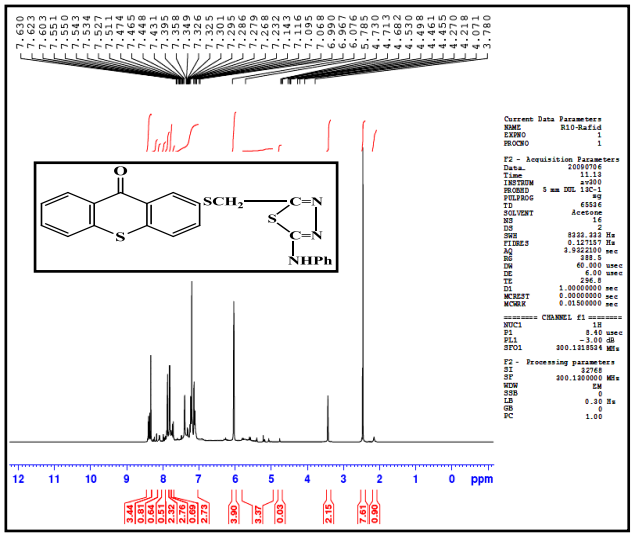


Fig.(9): ^{1}H-NMR spectrum for compound (14)

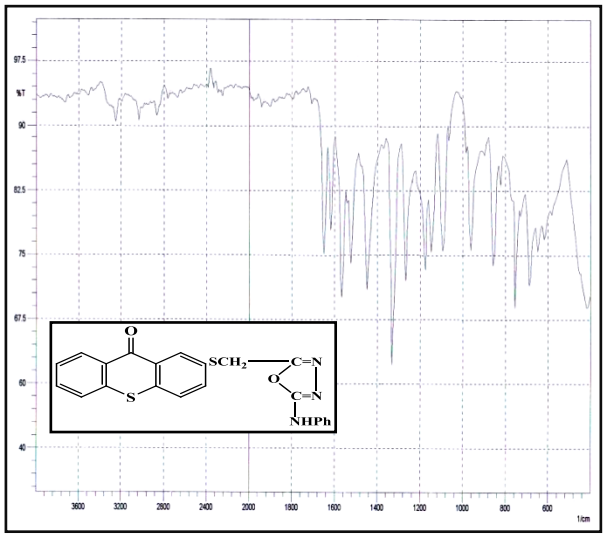


Fig.(10): FT-IR spectrum for compound (16)

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تحضير وتشخيص مشتقات حلقية جديدة للثايوكسانثون

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الخلاصة: يتضمن البحث تحضير مشتقات جديدة للثايوكسانثون المعوضة على ذرة الكاربون. وقد اعتبرت المادة الاولية وهي اورثو مركبتوبنزويك اسيد لتحضير هذه المشتقات. عند معاملة اورثو مركبتو بنزويك اسيد مع فينوكسي استيك اسيد/ ثايو فينوكسي استيك اسيد ليعطي 2-(اوكسي اسيتك اسيد) ثايوكسانثون (**1**) و 2-(ثايو اسيتك اسيد) ثايوكسانثون (**2**) على التوالي، والتي بمعاملتها مع ثايونيل كلورايد لتعطي 2- (اوكسي اسيتيل كلورايد) (**3**) و 2-(ثايو اسيتيل كلورايد) (**4**) على التوالي، المركبات (**4,3**) عند معاملتها مع الهايدرازين المائي تحول الى مشتق الهايدرازيد (**6,5**) وهذا بدوره يتفاعل مع فنيل ايزوثايوسيانيت / فنيل ايزوسيانيت نحصل على المشتق ثايوسيميكاربازايد (**8,7**) وسيميكاربازايد (**10,9**) التي تستخدم في تحضير ثمانية أنواع من المشتقات الحلقية.