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The Preparation of some New Mannich and Shiff bases derived from 2-Mercaptobenzimidazole

Mohammed R. Ahamed [*] ***Shetha F. Narren*** [**]
Amaal S. Sadiq [**]

*Department of Chemistry, College of Science, University of Baghdad, Iraq
**Department of Chemistry, College of Science for Women, University of Baghdad, Iraq

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Abstract:

The present work involved two steps: the first step include Mannich reaction was carried out on 2- mercaptobenzimidazole using formaldehyde and different secondary amine or amide to gives the compounds(2-16). The secnd step include preparation of (Ethylbenzimidazoly-2-mercaptoacetate)(17) from the reaction of 2- mercaptobenzimidazole with ethylchloroacetate than prepared hydrazide derivative[18]from reaction of compound(17) with hydrazinehydrate. Followed Preparation of shiff bases(19-24) and there reaction with mercaptoacetic acid to give a new compounds containing thiazolidinderivetives(25-30).Structure confirmation of all prepared compound were proved using FTIR and element analysis (C.H.N.S) and mesurmentedmelting point.

Key words: 2Mercaptobenzimidazole, Mannich reaction, Shiff bases.

Introduction:

2-Mercaptobenzimidazole derived from benzimidazole with thiol group in the 2-position. It possesses other chemical names such as, .o-phenylenthiourea, benzirnidazol-2-thion with formula of $C_7H_6N_2S$ [1,2]. Some characteristic of 2-mercaptobenzimidazole are containing of thioamide group (-N-C=S), therefore it is considered one of thioamide compounds for its ability to react under special conditions to give derivatives having substituent at either nitrogen or sulfur atoms[3'4], 2-mercaptobenzimidazole possess the form dimer, because it has (C=S) group, this preferable product is the dimer[5], it is known to exist in two tautomerism forms , the thiol and thione[6'7]. Various derivatives of 2-mercapto benzimidazole have been synthesized by several investigators and have been reported to exhibit a wide range of biological activities such as antimicrobial[8] antihistamine[9] and neutropic[10] activities. Although a great deal of the scientific literature concerning 2-mercaptobenzimidazole is in the area of medicinal chemistry 2-mercaptobenzimidazole is also used in non-biological application, it serve as plant growth regulators[11] and used as corrosion inhibitor for mild steel in sulfuric acid solution[12], stainless steel in aqueous solutions of NaCI[13], mild steel and zinc in phosphoric acid[14,15] Also, it is widely used as an accelerator in rubber processing[16], and anti oxidant for rubber and plastics[17].

Mercaptobenzimidazole and its derivatives display insecticidal properties [18], it is also a well-known analytical reagent. for mercury, and have been used for the determination Fe(Il), Cu(II), and Cd(II) metal ions in sewage water and industrial waste waters samples[19-20].

Methods:

All chemicals with high purity are used as the manufactures spilled them. The FT-IR spectra in the range (4000-200)cm^{-1}were recorded as KBr disc on a Shimadzu FT-IR 8300 spectrophotometer, elemental analysis (C.H.N.S) was carried out in Ministry of Oil. Melting points were recorded on Stuart scientific CO-LTD melting point SMP 1 and were uncorrected. Mercaptobenzimidazole was prepared in autoclave , it's local mode from stainless steel with capacity (300 ml) and diameter (12.5 cm).

Material and Methods:

Experimental

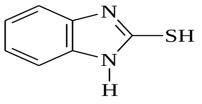
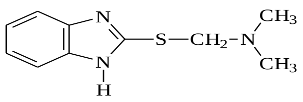
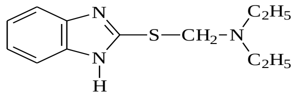
Synthesis of 2-Mercaptobenzjmidazole [1]:

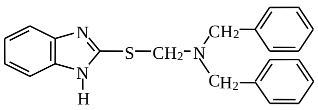
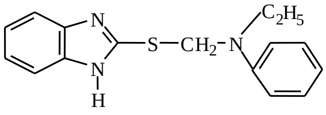
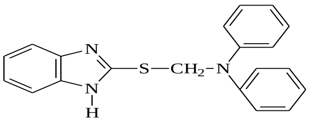
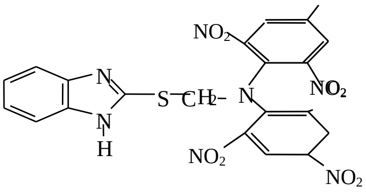
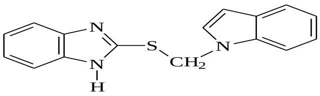
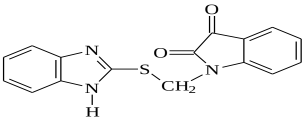
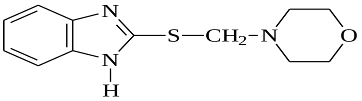
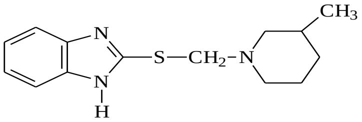
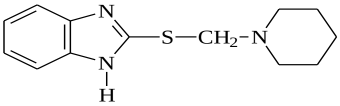
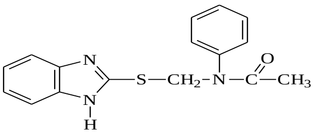
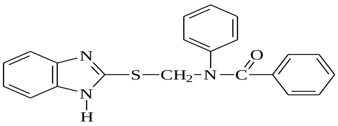
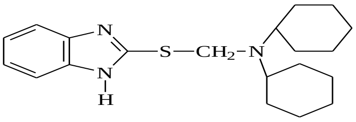
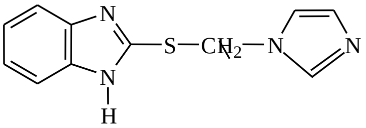
o-Phenylenediamine (5.0 g , 0.083 mol) was dissolved in absolute ethanol (75 ml) and added (35 ml) of carbon disulfide then the mixture was transferred in to autoclave with closing it very well to get high temperature and pressure. The set-up washeated in sand both at (150 C) for (15 hours). Then the mixture was put in beaker and added (9 ml) of (10%) sodium hydroxide to get rid of o-phenylenediamine and some concentrated hydrochloric acid until the mixture become acidic, to precipitate 2- mercaptobenzimidazole, filtered and dried then recrystallized from ethanol and water. Table (1) Listed the physical properties of compound [I].

Preparation of Mannich Bases [2-16][21]

Equimolar quantities (0.01 mol, 1.5 g) of 2-mercaptobenzimidazole and the respective compounds having secondary amine were dissolved in methanol (30 ml) in a beaker under perfect ice-cold condition and stirred constantly. To this solution, formaldehyde (0.01 mol) was added slowly and heated to reflux for (3 hours) the content was kept overnight in the freezer. The corresponding crystals of mannich base of 2-mercaptobenzimidazole, obtained was recrystallized from ethanol.Physical properties are listed in table (1).

Table (1) shows the nomenclature, physical properties of Mannichbases .

Compd. No.	Nomenclature	Structure formula	Yield %	Color	M. P. ºC
1	2-Mercapto benzaimidazole		75	White	282-284
2	2-(Dimethylamine-N-methyl)mercpto-1H-benzimidazole		50	White	143-145
3	2-(Diethylamine-N-methyl)mercpto-1H-benzimidazole		54	White	151-153

4	**2-(Dibenzylamine-N-methyl)mercpto-1H-benzimidazole**		**72**	**Light Yellow**	**76-78**
5	**2-(Ethylphenylamine-N-methyl)mercpto-1H-benzimidazole**		**68**	**Dark Brown**	**181-183**
6	**2-(Diphenylamine-N-methyl)mercpto-1H-benzimidazole**		**61**	**Light Gray**	**128-130**
7	**2-(Hexnitrodiphenylamine-N-methyl)mercpto-1H-benzimidazole**		**60**	**Dark Yellow**	**162-164**
8	**2-(Indo-N-methyl) mercpto-1H-benzimidazole**		**54**	**Pink**	**100-102**
9	**2-(Indol-2,3-dione-N-methyl)mercapto-1H-benzimidazole**		**72**	**Orange**	**112-114**
10	**2-(Morpholin-N-methyl)mercapto-1H-benzimidazole**		**83**	**White**	**199-200**
11	**2-(3-Methyl piperidine-N-methyl) mercapto-1H-benzimidazole**		**50**	**Yellow**	**110-112**
12	**2-(Piperidine-N-methyl) mercapto-1H-benzimidazole**		**55**	**White**	**97-99**
13	**2-(N-phenylacetamide-N-methyl) mercapto-1H-benzimidazole**		**48**	**White**	**120-121**
14	**2-(N-phenylbenzamide-N-methyl) mercapto-1H-benzimidazole**		**78**	**Gray**	**140-142**
15	**2-(Dicyclohexyl amine -N-methyl) mercapto-1H-benzimidazole**		**63**	**Light Brown**	**158-160**
16	**2-(Imidazole-N-methyl) mercapto-1H-benzimidazole**		**45**	**White**	**146-148**

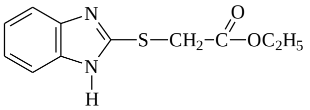
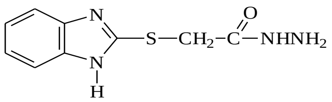
Preparation of Compound [17] Ethylbenzimidazolyl-2-Mercaptoacetate[22]

In a reaction flask provided with a refluxing cooler, (50 ml)of absolute ethyl alcohol was introduced and (1.15g , 0.05mol) metallic sodium was carefully added to the obtained solution of sodium ethoxide (7.5g, 0.05 mol)2-mercaptobenzimidazole was added as small portions under stirring and gentle heating on water bath for the solution homogenization, (6.75g , 0.1mol) ethylchloroacetate were added as several doses to the hot alcoholic solution of the 2-mercaptbenzimidazole sodium derivative. The mixture was refluxed on a water bath for about (1 hour). The sodium chloride was then removed by filtration under vacuum and the obtained yellow solution cooled to the room temperature and poured as thin thread, under stirring, into cold water. A cream – colored precipitate separated was filtered off under vacuum and dried. The crude product was purified by recrystallization from boiling ethyl alcohol.Table (2) listed he nomenclature , physical properties of Compound [17].

Preparation of Compound [18] Benzimidazolyl-2-Mercapto-aceticacid Hydrazide[22]

About (2.36g,0.01mol) ethyl Benzimidazolyl-2-mercaptoacetate[17] was suspended into (10 ml) absolute ethyl alcohol in a reaction vessel. Then (3 ml, 0.06 mol) hydrazine hydrate(80%) was added and gently heated on a water both. The viscous solution resulting within (15-20 minutes) was intermittently stirred. It was then allowed to stay at room temperature for (6 hours) for the reaction completion. A white precipitate thus separated which was collected by filtration under vacuum and dried. Recrystallization from boiling ethanol.Table (2) listed thenomenclature, physical properties of compound [18].

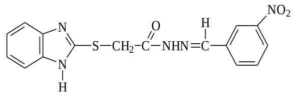
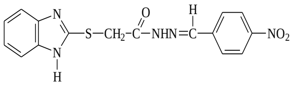
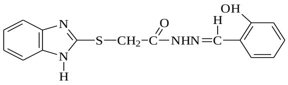
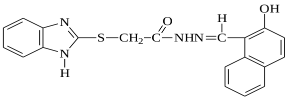
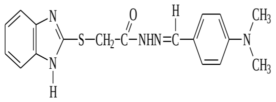
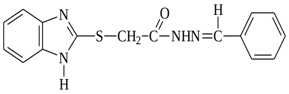
Table (2): The nomenclature , physical properties of Compound [17].

Compd. No.	Nomenclature	Structure formula	Yield %	Color	M. P. ºC
17	**Ethylbenzimidazole -2-mercaptoacetate**		**77**	**White**	**80-82**
18	**Benzimidazolyl -2-mercaptoaceticacidhydrazide**		**90**	**White**	**138-140**

Preparation of Schiff Bases [19-24](2-Aryliden–Hydrazinoacet-yl) – Merc-aptobenzimidazole[23]

A mixture of compound [18] (0.5g , 0.003mol),substuted aromatic respective aldehyde (0.4g , 0.003 mol) in absolute ethanol (15 ml) and drops of glacial acetic acid was refluxed for (8 hours). The mixture was cooled and collected by filtration and recrystallized by DMF. Table (3) the nomenclature,listed physical properties of shiff bases.

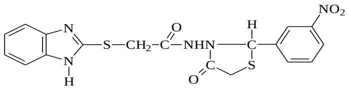
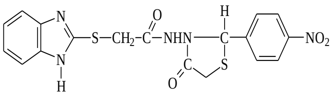
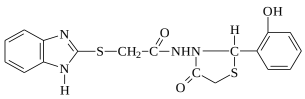
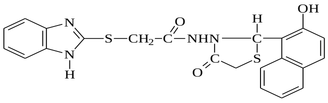
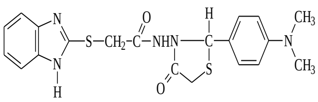
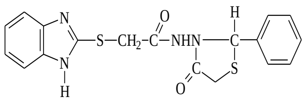
Table (3): The nomenclature , physical properties of Schiff bases [19-24]

Compd.No.	Nomenclature	Compound Structure	Yield%	Color	M. P.°C
19	[2(3-Nitrobenzylidine-hydrazinoacetyl)-mercaptobenzimidazole]		68	Light Yellow	160-162
20	[2(4-Nitrobenzylidine-hydrazinoacetyl)-mercaptobenzimidazole]		70	Light Yellow	174-176
21	[2(2-hydroxy benzylidine-hydrazinoacetyl)-mercaptobenzimidazole]		62	Beige	192-194
22	[2(2-Hydroxy-1-naphthyl-hydrazinoacetyl)-mercaptobenzimidazole]		60	Yellow	204-206
23	[2(4-Dimethlamine-benzylidine-hydrazinoacetyl)-mercaptobenzimdazole]		55	Dark Yellow	185-187
24	[2(benzylidine-hydrazinoacetyl)-mercaptobenzimidazole]		64	White	141-143

Preparation of Compounds [25-30][(2-Aryl-4-Oxo-1,3-Thiazolidin) –Hydrazinoacetyl] Mercaptobenzimidazole [24]

Mercaptoaceticacid (0.1g, 0.001mol) dissolved in dry benzene (7.5 ml) was added slowly to (0.4g , 0.001mol) of compound [34-39] respective dissolved in dry benzene. The addition continued about 10 seconds with stirring then the mixture was refluxed for (10 hours) excess solvent was evaporated and residues were treated with sodium bicarbonate, solid precipitate was recrystallized from ethanol. Table (4) listed nomenclature, physical properties of prepared compounds.

Table (4) shows the nomenclature, physical properties of prepared compounds.

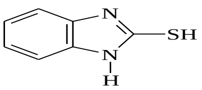
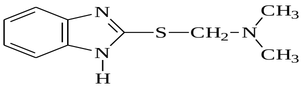
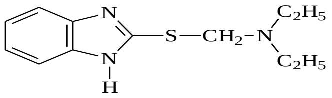
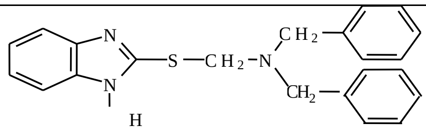
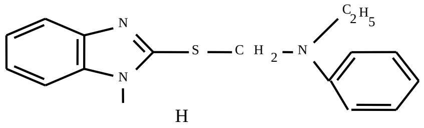
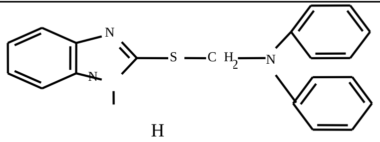
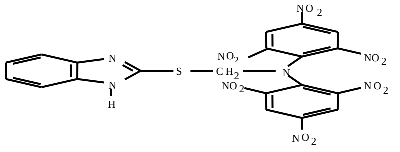
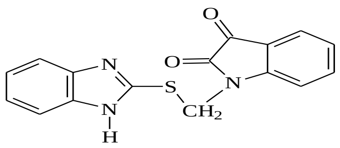
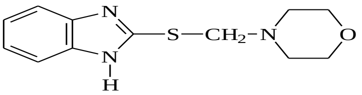
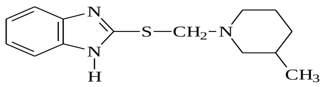
Compd. No.	Nomenclature	Compound Structure	Yield %	Color	M. P. °C
25	[2(3-Nitrobezyl -4-oxo-1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		62	Yellow	201-203
26	[2(4-Nitrobezyl-4-oxo-1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		60	Yellow	198-200
27	[2(2-hydroxybezyl-4-oxo-1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		55	Light Brown	215-217
28	[2(2-hydroxy-1- naphyl-4-oxo-1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		64	Dark Yellow	265-267
29	[2(4-N-dimethyl phenyl-4-oxo1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		61	Yellow	220-232
30	[2-(-phenyl)-4-oxo-1,3-thiazolidin)-hydrazino-acetyl-mercaptobenzimidazole]		65	Beige	196-198

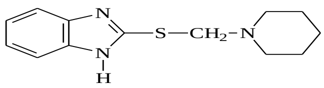
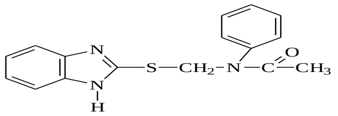
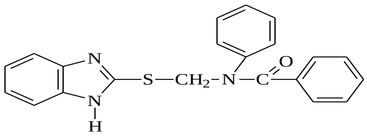
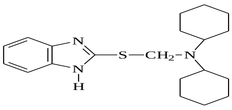
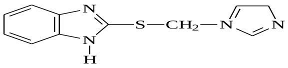
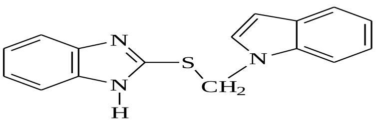
Result and Discussion:

The present work involved two steps.

First step:mannich bases of 2-mercaptobenzimidazole derivatives were synthesized using mannich reaction by the reaction between compounds having secondary amine and formaldehyde .The FT-IR spectrum show the appearance stretching υ (C-H) aromatic at (3096-3025) cm^{-1} (C-H) aliphatic at (2996-2925) cm^{-1}, and (C-N) band at (1392-1323) cm^{-1}, other bands were also absorbed in FT-IR spectra of these compounds which are listed in table (5) and C.H.N.S analysis for some of them in table(9).

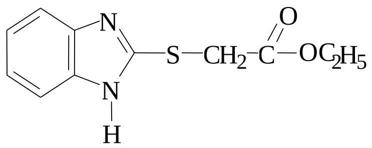
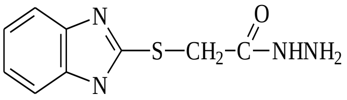
Table(5) FT-IR spectra data of compounds [1-16]

Compd. No.	Structure	υ(C-H) cm^{-1} Aromatic	υ(C-H) cm^{-1} Aliphatic	υ(C=N) cm^{-1} Aromatic	υ(C-N) cm^{-1}	υ(N-H) cm^{-1}	Other Band cm^{-1}
1		3040	-	1512	1624	3159	υ(S-H) 2569
2		3042	2966	1617	1340	3426	-
3		3040	2980	1620	1332	3350	-
4		3028	2943	1612	1392	3315	-
5		3072	2932	1630	1334	3425	-
6		3070	2925	1605	1354	3354	-
7		3093	2930	1612	1368	3432	υ(C-No_2) 1450
8		3025	2962	1616	1338	3448	υ(C=O) 1720
9		3052	2935	1616	1354	3433	υ(C-O) ether 1041
10		3056	2945	1622	1342	3422	-

11		**3062**	**2932**	**1618**	**1344**	**3423**	**-**
12		**3096**	**2996**	**1620**	**1352**	**3450**	**υ(C=O) amide 1665**
13		**3051**	**2990**	**1600**	**1323**	**3344**	**υ(C=O) amide 1654**
14		**3062**	**2927**	**1612**	**1338**	**3433**	**-**
15		**3052**	**2990**	**1616**	**1357**	**3342**	**-**
16		**3062**	**2942**		**1337**	**3339**	**-**

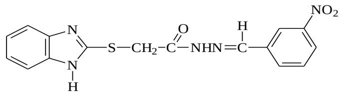
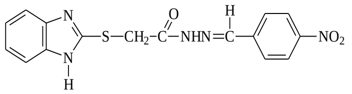
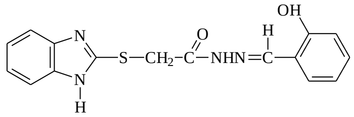
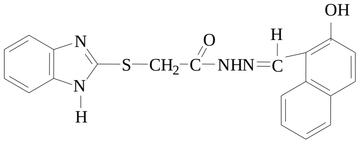
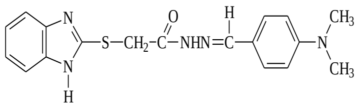
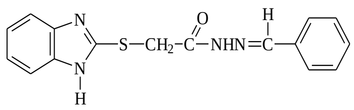
Second step: Reaction of ethyl alcohol with metallic sodium to obtain of sodium ethoxide, after adding 2-mercaptobenzimidazole under stirring and gentle heating to give 2-mercaptobenzimidazole sodium derivative followed ethylchloroacetate was added as several doses and refluxed on water bath for about (1) hour to give ethylbenzimidazolyl-2-mercaptoacetate[17]. Thanthe reaction of hydrazine hydrate with ester is one of the most common reactions to synthesize the acid hydrazide[18], the product compounds have been characterized by FT-IR spectrum besides the (C.H.N.S) analysis in table(9).The FT-IR spectra showed the bands of the stretching vibrations due to(ester –C=O, amine –$NH_2$and amide –C=O) groups in the (1739 cm^{-1}), (3344, 3282cm^{-1}) and (1654 cm^{-1}) respectively.The diagnostic bands were assigned and the band positions are given in table(6).

Table (6): FT-IR spectral data of compounds [17-18]

Compd. No.	Structure	υ (C-H) cm^{-1} Aromatic	υ (C-H) cm^{-1} Aliphatic	υ (C=C) cm^{-1} Aromatic	υ (C=O) cm^{-1}	υ(C=N) cm^{-1}	Other Band cm^{1}
17		**3020**	**2970**	**1589**	**1739**	**1620**	**υ (N-H) 3456 υ(C-O) 1180**
18		**3043**	**2962**	**1535**	**1654**	**1593**	**υ(NH_2) 3344 3282**

Aftersynthesis (2-aryliden-hydrazinoacetyl) -mercaptobenzimid-azolefrom the reaction between compound [18] and appropriate aldehydes in absolute ethanol and glacial acetic acid the structure of the compounds [19-24] have been characterized and confirmed by FT-IR spectrum besides the C.H.N.S analysis for some of them in table (9).The FT-IR spectrum shows the disappearance absorption band at (3344, 3282) cm^{-1} due to the asymmetric and symmetric stretching vibration of the (NH_2) group and appearance the (C=N) band of the imine in the range (1651-1608) cm^{-1} and (C=O) band of the amide at (1678-1654) cm^{-1}, also bands appear at (3086-3040) cm^{-1}, (2989-2924) cm^{-1} and (1589-1489) cm^{-1} due to υ(C-H) aromatic, υ(C-H) aliphatic and υ(C=C) aromatic respectively, these bands and other are shown in table (7).End step refluxequimolor amounts from the imine compounds[19-24] with mercaptoaceticacide in dry benzene.The structure of the synthesized compounds[25-30] have been characterized and confirmed by FT-IR spectrum besides the (C.H.N.S) analysis for some of them table (9). The FT-IR spectrum shows the appearance of carbonyl group band of the oxo-thiazolidine ring at (1692-1656) cm^{-1} and (C=O) amide at (1635-1593)cm^{-1}, other bands are shown in table (8).

Table(7): FT-IR spectral data of compounds [19-24]

Compd. No.	Structure	υ(C-H) cm^{-1} Aromatic	υ(C-H) cm^{-1} Aliphatic	υ(C=C) cm^{-1} Aromatic	υ(C=O) cm^{-1}	υ(C=N) cm^{-1}	Other Band cm^{-1}
19		**3070**	**2974**	**1527**	**1678**	**1616**	**υ(NO_2) 1350**
20		**3070**	**2924**	**1523**	**1674**	**1608**	**υ(NO_2) 1356**
21		**3043**	**2989**	**1489**	**1670**	**1608**	**υ(O-H) 3425 Interference with V(N-H)**
22		**3086**	**2932**	**1589**	**1678**	**1651**	**υ(O-H) 3603** **υ(N-H) 3286**
23		**3055**	**2986**	**1520**	**1654**	**1616**	**υ(N-Me) 842**
24		**3040**	**2982**	**1586**	**1672**	**1612**	**-**

Table(8): FT-IR spectral data of compounds [25-30]

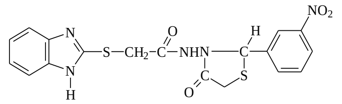
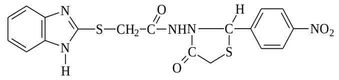
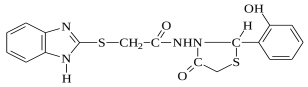
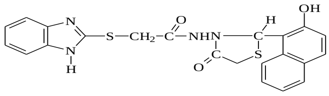
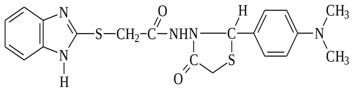
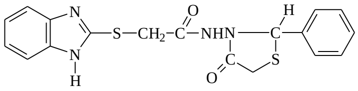
Compd. No.	Structure	υ(C-H) cm^{-1} Aromatic	υ(C-H) cm^{-1} Aliphatic	υ(C=C) cm^{-1} Aromatic	υ(C=O) cm^{-1}	υ(C=N) cm^{-1}	Other Band cm^{-1}
25		3047	2970	1527	1666	1600	υ(NO_2) 1365
26		3032	2972	1532	1678	1618	υ(NO_2) 1338
27		3076	2968	1586	1659	1620	υ(OH) 3520
28		3031	2962	1585	1675	1624	υ(OH) 3612
29		3051	2958	1500	1692	1635	υ(N-Me) 867
30		3095	2945	1522	1656	1593	-

Table (9): The C.H.N.S analysis of some prepared compounds

17	$C_{22}H_{21}N_3S$	Calc. 73.50	5.89	11.69	8.92
		Found. 73.22	5.66	11.32	8.59
18	$C_{16}H_{17}N_3S$	Calc. 67.81	6.05	14.83	11.31
		Found. 67.71	6.02	14.66	11.01
20	$C_{20}H_{11}N_9SO_{12}$	Calc. 39.94	1.84	20.69	5.33
		Found. 39.81	1.77	20.54	5.01
21	$C_{16}H_{13}N_3S$	Calc. 68.79	4.69	15.04	11.48
		Found. 68.56	4.59	15.03	11.32
22	$C_{16}H_{11}N_3SO_2$	Calc. 62.12	3.58	13.58	10.36
		Found. 62.01	3.44	13.42	10.22
23	$C_{12}H_{15}N_3SO$	Calc. 57.81	6.06	16.85	12.86
		Found. 57.71	6.02	16.75	12.79
25	$C_{13}H_{17}N_3S$	Calc. 63.12	6.93	16.99	12.96
		Found. 63.01	6.79	16.81	12.84
27	$C_{21}H_{17}N_3SO$	Calc. 70.17	4.77	11.60	8.92
		Found. 70.07	4.56	11.55	8.81
28	$C_{20}H_{29}N_3S$	Calc. 69.93	8.51	12.23	9.33
		Found. 69.81	8.41	12.11	9.23
30	$C_{11}H_{12}N_2SO_2$	Calc. 55.91	5.12	11.86	13.57
		Found. 55.79	5.01	11.77	13.40
31	$C_9H_{10}N_4SO$	Calc. 48.63	4.53	25.21	14.43
		Found. 48.55	4.22	25.01	14.23
32	$C_{16}H_{13}N_5SO_3$	Calc. 54.08	3.69	19.71	9.02
		Found. 54.01	3.49	19.55	8.99
34	$C_{16}H_{14}N_4SO_2$	Calc. 58.88	4.32	17.17	9.82
		Found. 58.76	4.22	17.09	9.66
37	$C_{16}H_{14}N_4SO$	Calc. 61.92	4.55	18.05	10.33
		Found. 61.77	4.23	17.99	10.24
38	$C_{18}H_{15}N_5S_2O_4$	Calc. 50.34	3.52	16.31	14.93
		Found. 50.11	3.41	16.22	14.81
39	$C_{18}H_{15}N_5S_2O_4$	Calc. 50.34	3.52	16.31	14.93
		Found. 50.11	3.30	16.11	14.55
41	$C_{22}H_{18}N_4S_2O_3$	Calc. 58.65	4.03	12.44	14.23
		Found. 58.42	3.99	12.22	14.10
43	$C_{18}H_{16}N_4S_2O_2$	Calc. 56.23	4.19	14.57	16.68
		Found. 56.11	4.01	14.31	16.21

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تحضير مشتقات مانخ و قواعد شف جديدة للمركب 2-مركبتوبنزاميدازول

محمد رفعت احمد* **شذى فاضل نارين**** **أمال سمير صادق****

*قسم الكيمياء/ كلية العلوم/جامعة بغداد
**قسم الكيمياء/كلية العلوم للبنات/جامعة بغداد

الخلاصة:

يتضمن البحث خطوتين :
الخطوة الاولى تحضير قواعد مانخ لمركب 2-مركبتوبينزاميدازول باستخدام امينات ثانوية وبوجود الفورمالديهاد لتحضيرالمركبات [2-16].الخطوة الثانية تحضير انواع من قواعد شف من خلال تكثيف 2-مركبتوبينزاميدازول مع بعض الالديهايدات الاروماتية وبوجود حامض الخليك كعامل مساعد ومن ثم يتم استخدام قواعد شف للحصول على مركبات تحتوي على حلقة الثايوزولدين باستخدام مركبتو استك اسد الذي يساعد في الخلق الحلقي. تم تشخيص المركبات المحضرة بمطياف ال FT-IR و التحليل الدقيق لعناصر (C.H.N.S.) بجانب قياس درجة الانصهار.

الكلمات المفتاحية: 2-مركبتوبنزاميدازول، تفاعل مانخ، قواعد شف.