Synthesis of heterocyclic derivatives from Schiff bases

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Summary

A new derivatives of heterocyclic were compounds have been prepared through the reaction of Schiff bases with glycolic acid , alanine and cysteine in toluene and the reaction was refluxed for some time. In addition to the study include identification for all derivatives by Mp, TLC and FTIR spectroscopy.

تحضير مركبات حلقية غير متجانسة من قواعد شف

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الخلاصة

يتضمن البحث تحضير مركبات حلقية غير متجانسة من قواعد شف عن طريق معادلتها مع حامض الكلايكوليك والالنين والسيستاين في التلوين بوجود عامل مساعد والتصعيد لفترة زمنية معينة. تم تشخيص المركبات المحضرة بواسطة نقاط الانصهار وكرموتوغرافيا الطبقة الرقيقة واطياف الاشعة تحت الحمراء .

Keywords: synthesis, Schiff bases , glycolic acid, cystiene and alanine

Introduction

Hugo Schiff described the condensation between an aldehyde and an amine leading to a Schiff bases in 1864 [(1)]. Schiff bases one class of important compounds in medicinal and pharmaceutical field. They show biological activities including antibacterial[(2-5)] antifungal[(6-7)] anticancer[(8-10)] and antiherbieidal [(11)] activities further more, Schiff bases are utilized as starting material in the synthesis of industrial[(12-14)] and biological compounds such as β-Lactames[(15)]. Schiff bases are able to stabilize many different metals in various oxidant state[(1)]. Some of Schiff bases have liquid crystalline properties[(16).] Schiff bases are used as intermediate in the synthesis heterocyclic compounds.

A heterocyclic compounds is one that contains airing made up of more that one kind of atom [(17)]. Heterocyclic compounds intermediates are beings used in synthesis as protecting groups, readily generated and when their job is done, readily removed. In the biological word, as he have seen heterocyclic compounds are every where carbohydrates are heterocyclic [(18-19)]. Five memberaed hetero aromatic rings are one of heterocyclic compounds and well recognized as central in organic synthesis and in the synthesis of biological active compounds [(20)].

Experimental

All the chemicals used were supplied by Merck, Fluka and BDH chemicals. FTIR spectra were recorded on SHIMADZU – FTIR 8400 Fourier transform infrared spectrophotometer using KBr discs. Melting point were determined in open capillaries on Thomas Hoover apparatus and were uncorrected. Thin layer chromatography (TLC) was carried out using Fertigfollen precoate sheets type Polygram SilG, and the plates were developed with iodine vapor

2-1 Synthesis of Schiff bases

A mixture of amine (0.01mole) with aromatic aldehyde (0.01mole) in 15 ml of absolute ethanol were refluxed for appropriate time then cooled to the room temperature the produced precipitates were filtrated and washed with ethanol The end of reaction is detected by TLC by using appropriate mixtures of solvent physical properties of the prepared compounds are shown in table 2-1

Comp No.	Yield%	Solvents of TLC	M.P^{0}c	Color	Time
1	97	benzene-Ethanol	112-114	Yellow	20 minutes
2	98	benzene-Ethanol	160-162	Deep yellow	30 minutes

Table 2-1 show the physical properties of the prepared Schiff bases

2-2 preparation of oxazolidin-4-one derivatives

A mixture of (0.01) mole compounds (1,2) and (0.01) mole of glycolic acid and few drops of trimethylamine in 25ml of toluene have been refluxed for appropriate time the end of reaction indicated by TLC by using a mixture of chloroform and ethanol then cooled to the room temperature the produced precipitates were filtrated and washed with toluene the physical properties of the prepared compounds are shown in table 2-2

Table 2-2 show the physical properties of oxazolidin-4-one compounds

Comp No.	Yield%	Solvents of TLC	M.P^{0}c	Color	Time
3	40	chloroform-Ethanol	157-159	Orange	20 h
4	44	chloroform-Ethanol	203-205	yellow	17 h

2-3 Preparation of imidazolidin-4-one

A mixture of (0.01) mole compounds (1,2) and (0.01) mole of alanine and few drops of trimethylamine in 25ml of toluene have been refluxed for appropriate time the end of reaction indicated by TLC by using a mixture of toluene and ethanol then cooled to the room temperature the produced precipitates were filtrated and washed with toluene the physical properties of the prepared compounds are shown in table 2-3

Table 2-2 show the physical properties of imidazolidin-4-one compounds

Comp No.	Yield%	Solvents of TLC	M.P^{0}c	Color	Time
5	43	benzene - ethanol	133-135	White	18 h
6	37	benzene - ethanol	188-190	white	22 h

2-4 preparation of 1,3-thiazinan-4-one

A mixture of (0.01) mole compounds (1,2) and (0.01) mole of cytiene and few drops of trimethylamine in 25ml of toluene have been refluxed for appropriate time the end of reaction indicated by TLC by using a mixture of toluene and ethanol then cooled to the room temperature the produced precipitates were filtrated and washed with toluene the physical properties of the prepared compounds are shown in table 2-4

Table 2-4 the physical properties of 1,3-thiazinan-4-one compounds

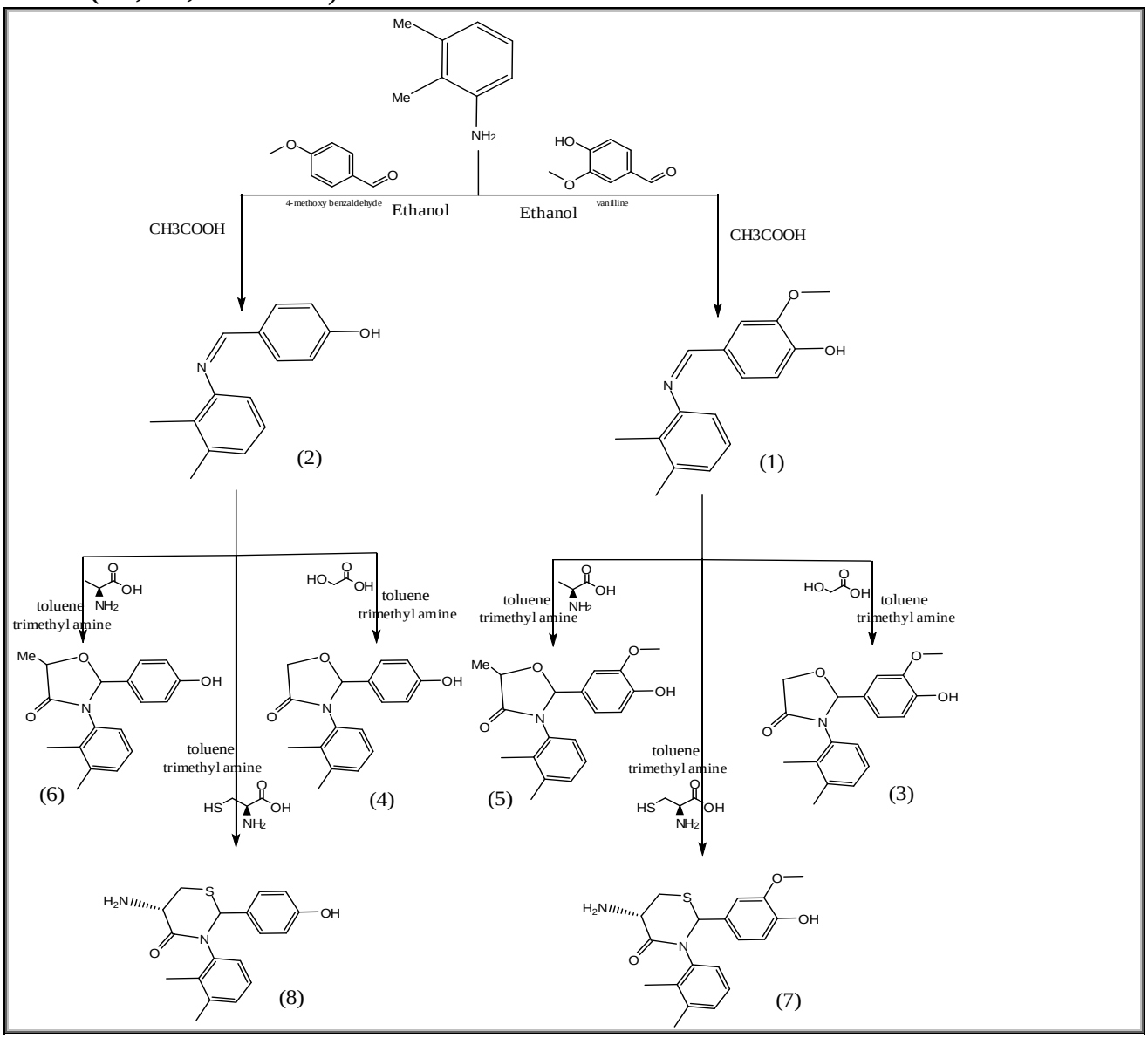
Comp No.	Yield%	Solvents of TLC	M.P^{0}c	Color	Time
7	65	toluene - ethanol	210-212	White	18 h
8	71	toluene - ethanol	188-190	white	22 h

Results and discussion:

Many new heterocyclic derivatives prepared from Schiff bases. The first derivative is Oxazolidine prepared by reaction Schiff base with glycolic acid in toluene and refluxed for appropriate time.n

The second derivative is imidazolidine prepared by reaction Schiff base with amino acid alanine in toluene and refluxed for appropriate time. The third derivative is 1, 3-Thiazinane prepared by reaction Schiff base with cystiene in toluene and refluxed for appropriate time.

The analytical data with some physical properties of the new derivatives are summarized in Tables (2-1, 2-2, 2-3and 2-4).



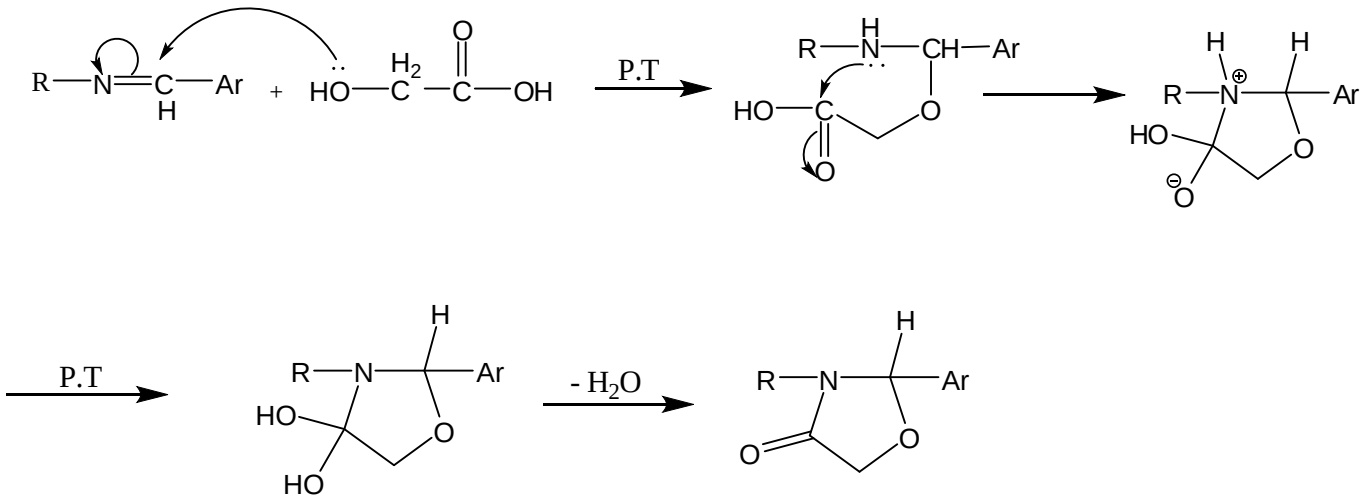
Scheme 1 show the prepared compounds

FTIR spectra:

FTIR spectroscopic studies shows for Schiff bases appear weak peak at (2950-2813) cm^{-1} which are belong to methyl groups. Another strong peaks (1654 and 1510) cm^{-1} belongs to imine group, figs (1 and 2). For second derivatives appears middle peak at (1700-1720) cm^{-1} for carbonyl group, and weak peak at (3300-3380) cm^{-1} for hydroxyl group, figs (3and4). Third derivative appears strong peak at 1750cm^{-1} for carbonyl group and weak peak at (3400-3450) cm^{-1} for amine group of immidazolidine, figs (4and6). Four derivatives appear strong peak at (1650-1750) cm^{-1} which is belong to carbonyl group and weak peak (3200-3350) cm^{-1} for amine group, figs (7and8).

The proposed mechanism of these reactions

1-



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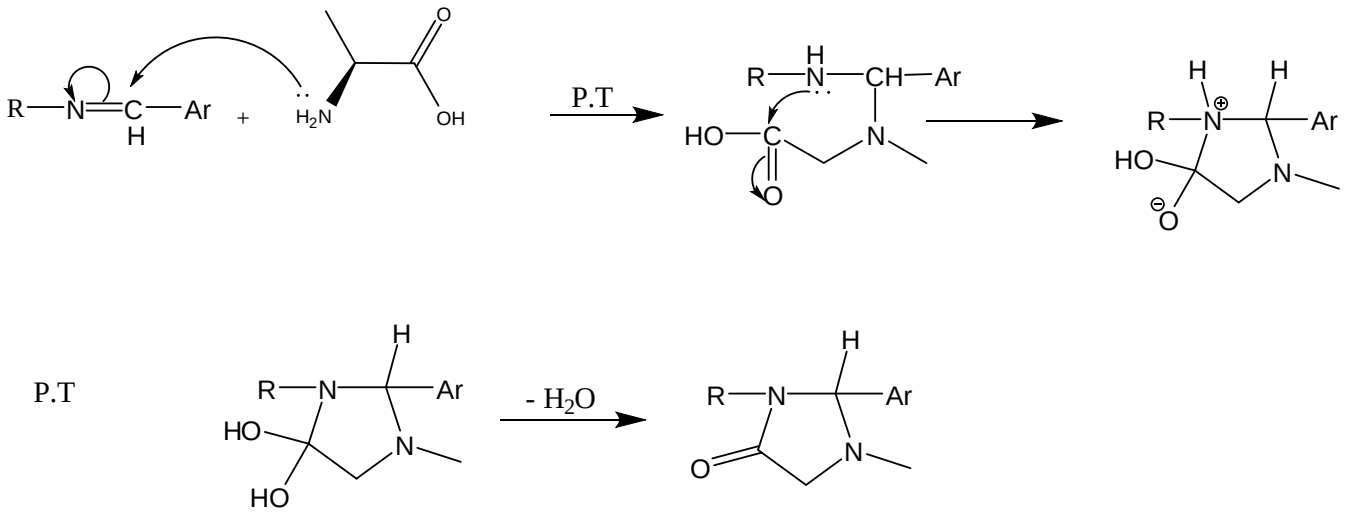

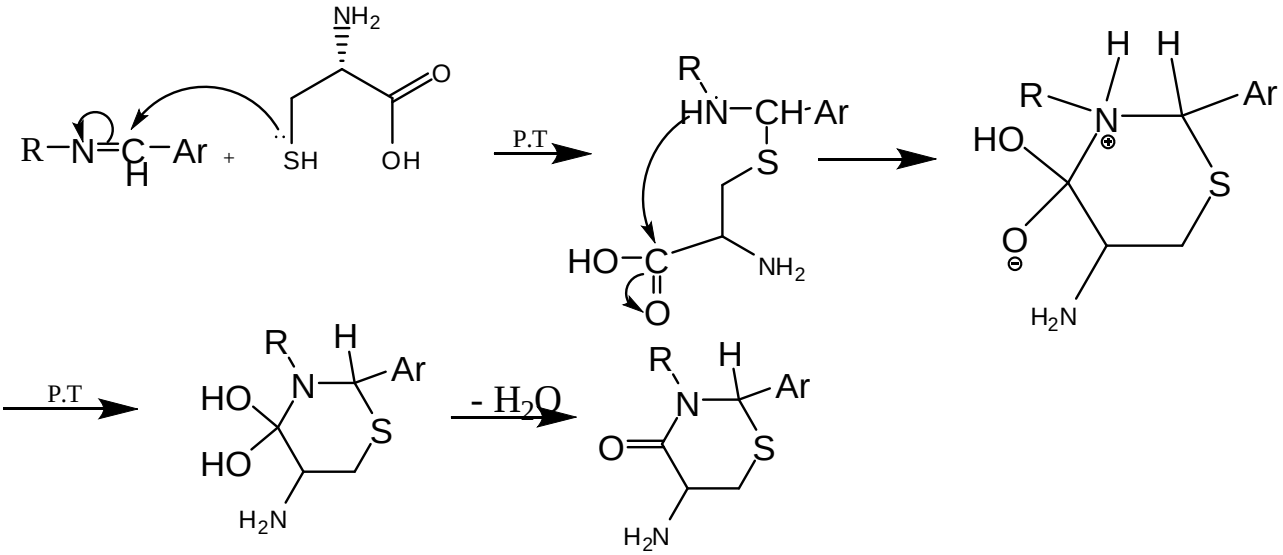

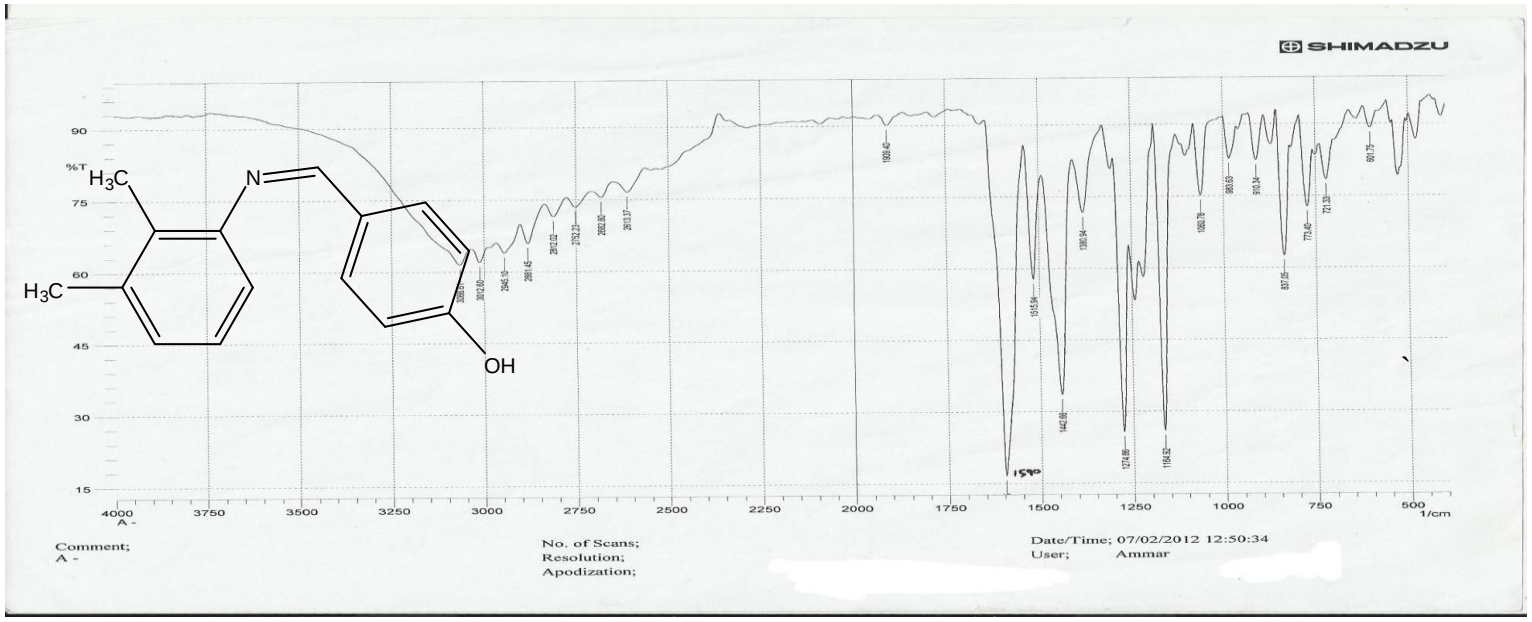

Fig.(3-1) FTIR spectrum of compound 1 (Z)-4-((2,3-dimethylphenylimino)methyl)phenol

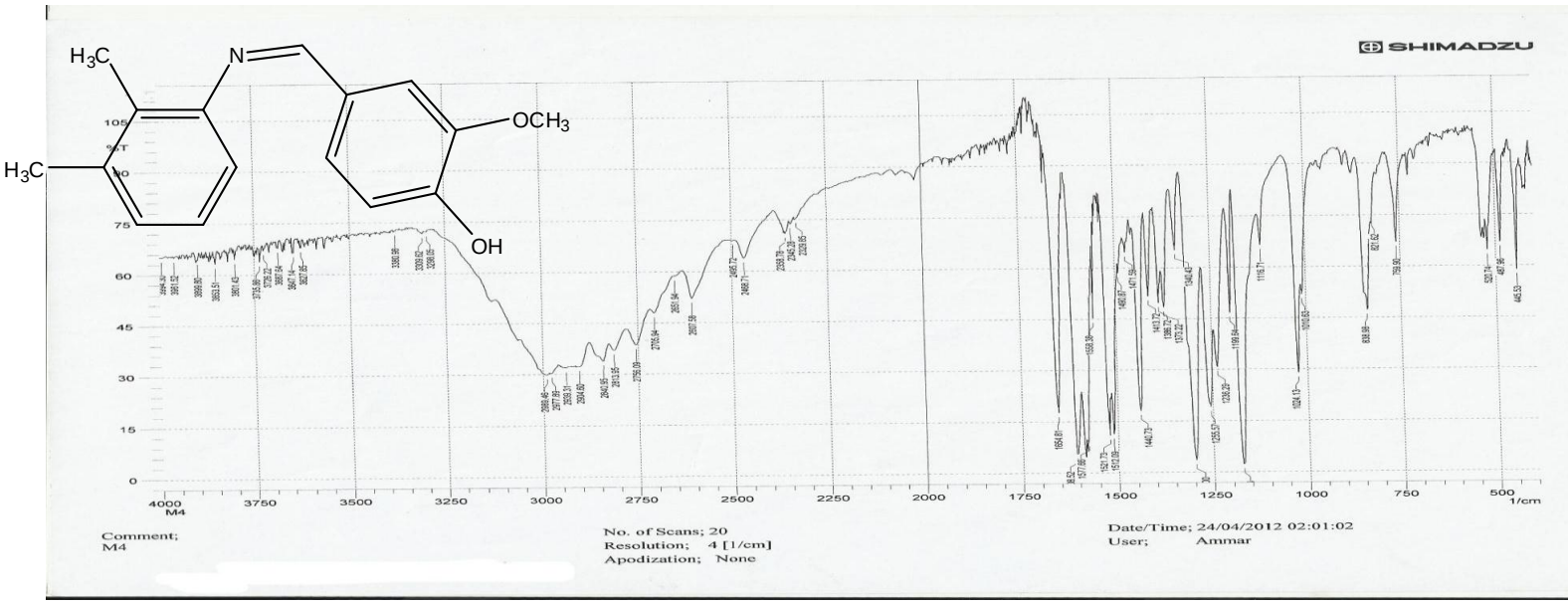

Fig.(3-2) FTIR spectrum of compound 2 (Z)-4-((2,3-dimethylphenylimino)methyl)-2-methoxyphenol

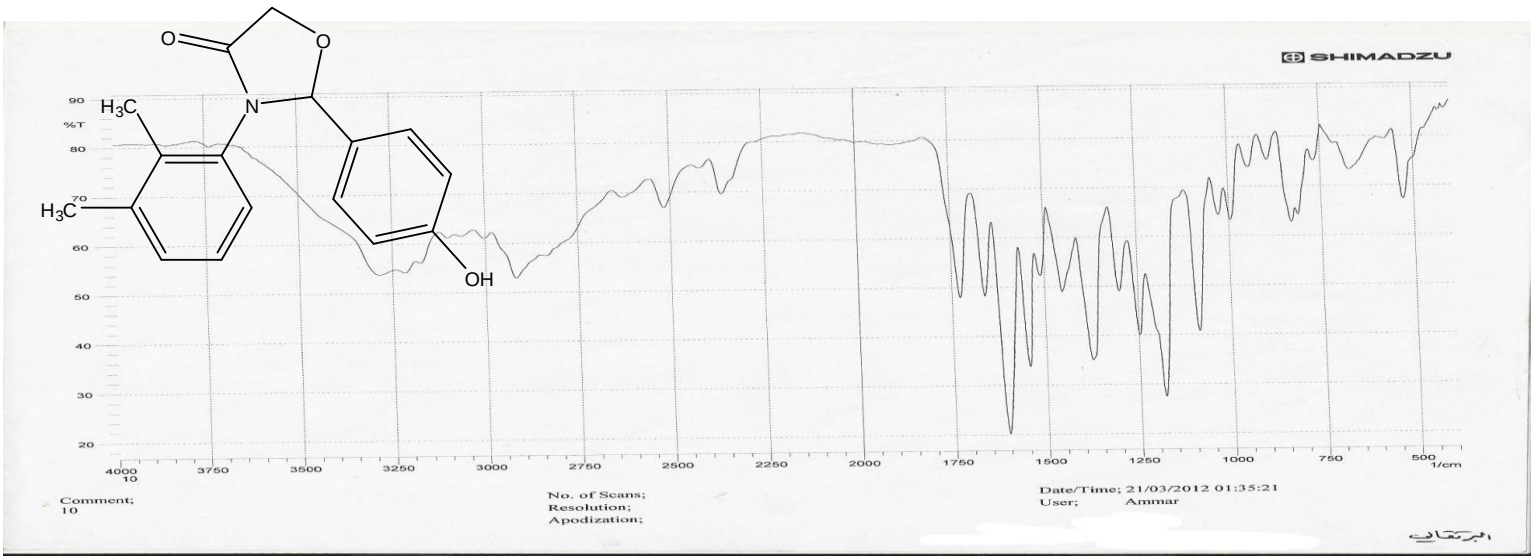

Fig .(3-3) FTIR spectrum of of compound 3 3-(2,3-dimethylphenyl)-2-(4-hydroxyphenyl)oxazolidin-4-one

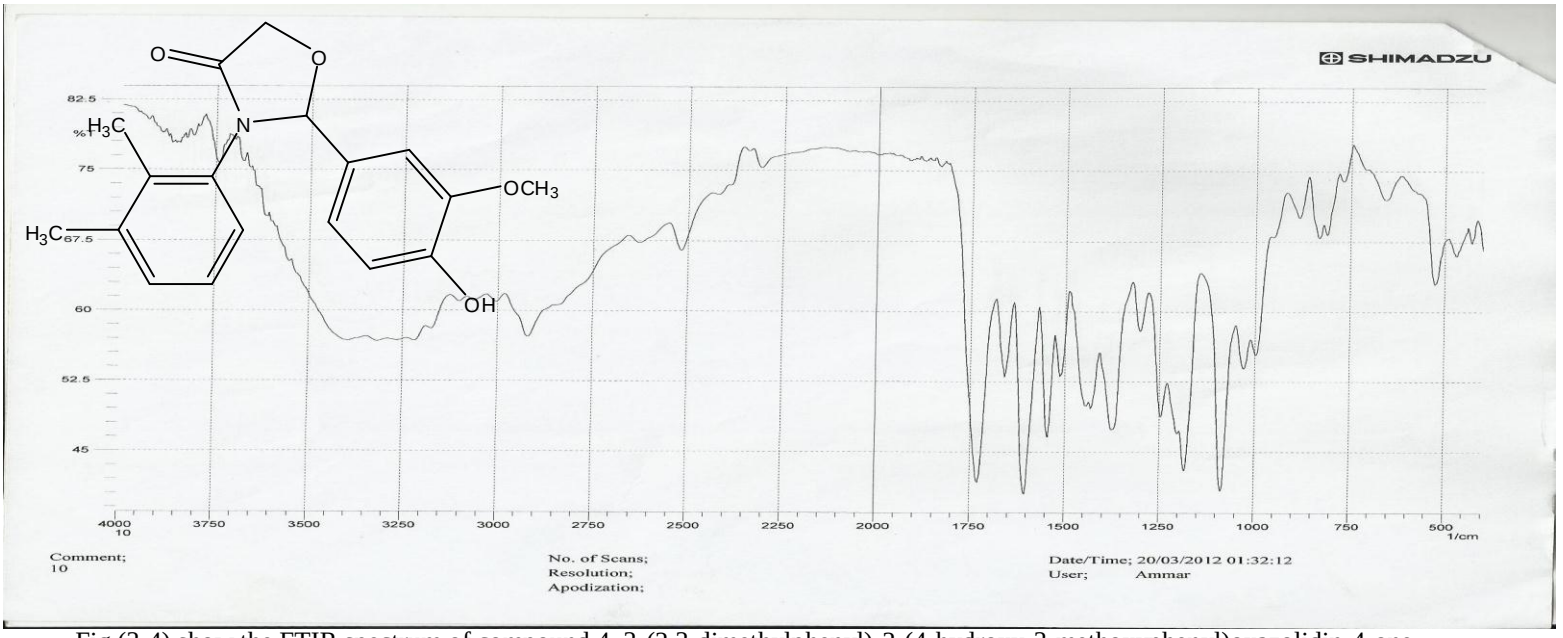

Fig (3-4) show the FTIR spectrum of compound 4 3-(2,3-dimethylphenyl)-2-(4-hydroxy-3-methoxyphenyl)oxazolidin-4-one

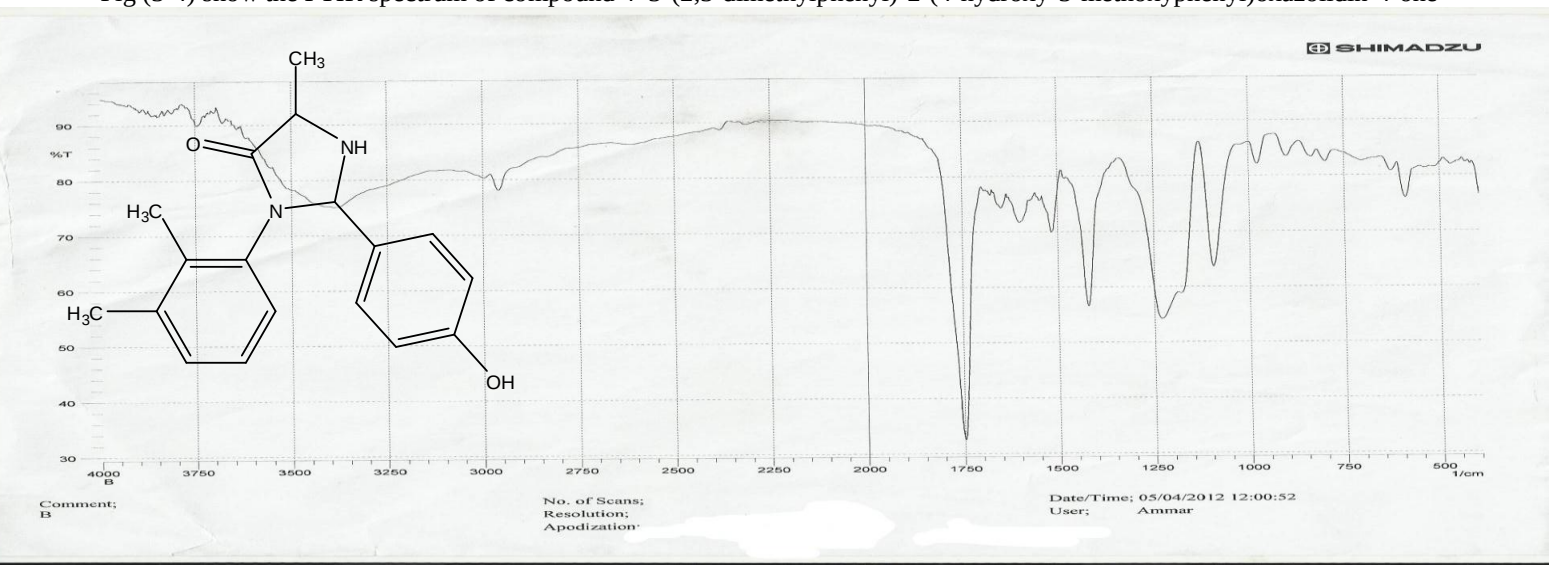

Fig (3-5) show the FTIR spectrum of compound 5 (2,3-dimethylphenyl)-2-(4-hydroxyphenyl)-5-methylimidazolidin-4-one

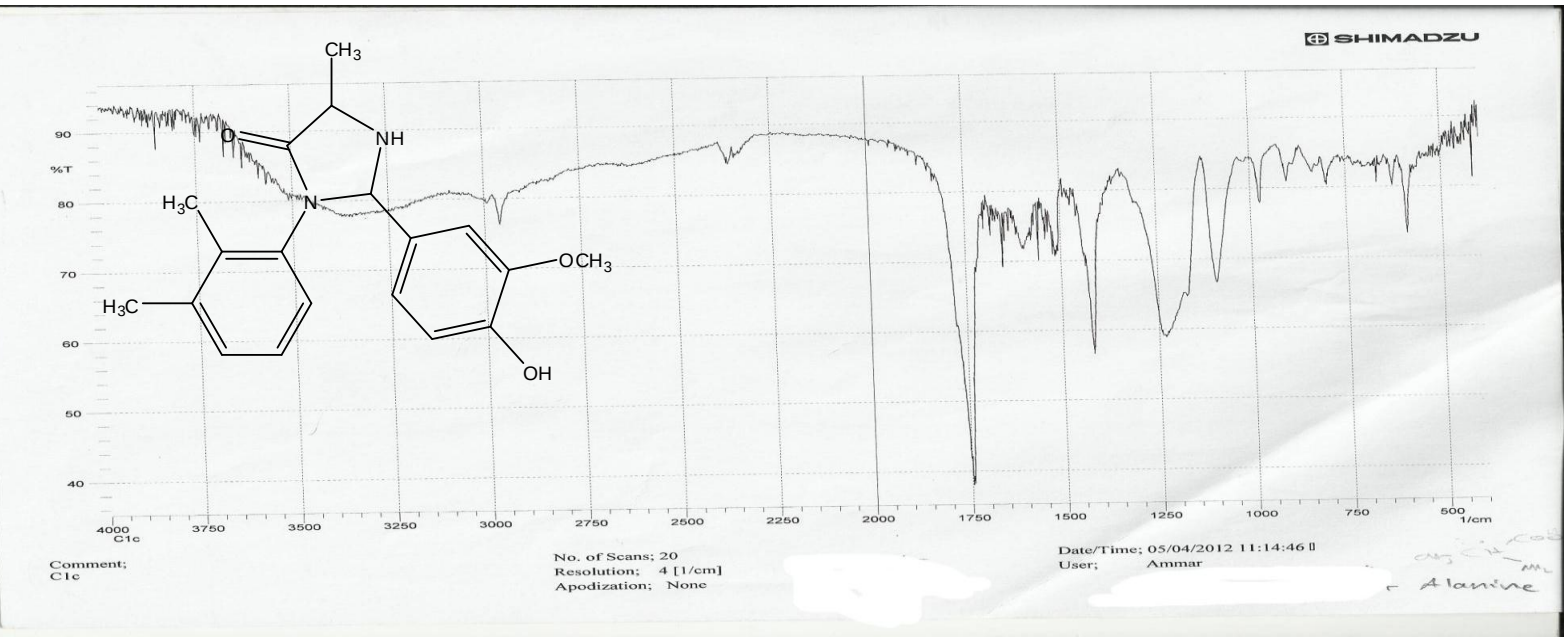

Fig (3-6) show the FTIR spectrum of compound 6 3-(2,3-dimethylphenyl)-2-(4-hydroxy-3-methoxyphenyl)-5-methylimidazolidin-4-one

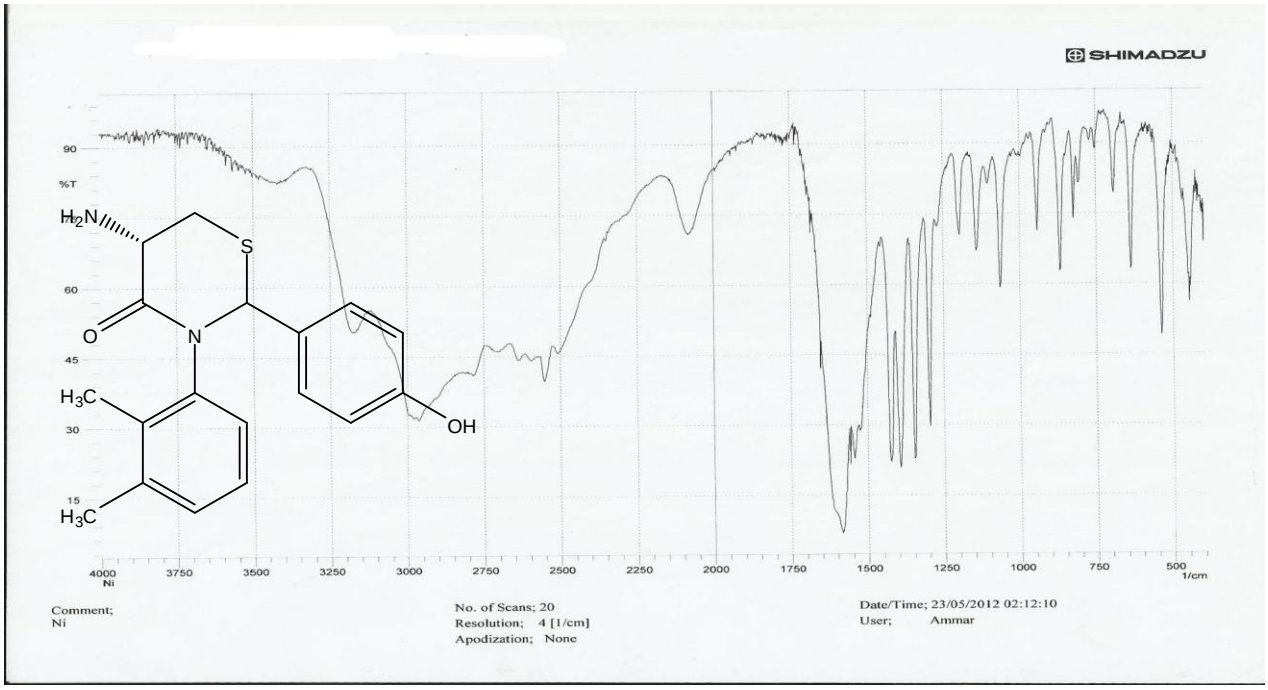

Fig (3-7) show the FTIR spectrum of compound 7 (5S)-5-amino-3-(2,3-dimethylphenyl)-2-(4-hydroxyphenyl)-1,3-thiazinan-4-one

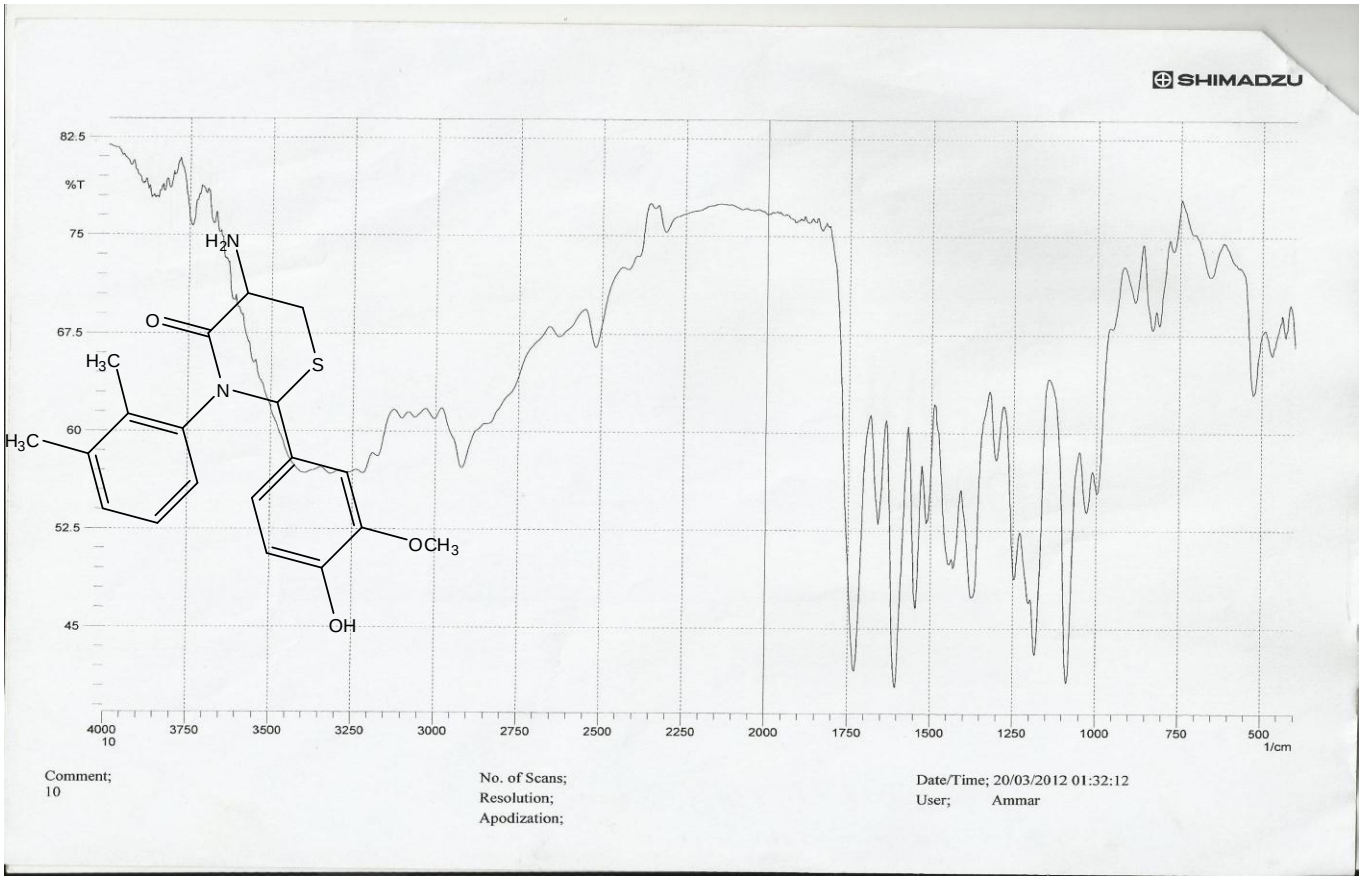

Fig (3-8) show the FTIR spectrum of compound 8 5-amino-3-(2,3-dimethylphenyl)-2-(4-hydroxy-3-methoxyphenyl)-1,3-thiazinan-4-one

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