Rafidain Journal of Science
https://rsci.mosuljournals.com
Vol. 31, No.3, pp.9-18, 2022

Synthesis of some New Derivatives of Triazole using Ortho-Carboxybenzaldehyde as a Synthone

Maysaa K. Al-Mala Khder
Department of Chemistry/ College of Education for Girls/ University of Mosul
Moayed S. Al-Jawady
Department of Chemistry/ College of Science/ University of Mosul

p-ISSN: 1608-9391
e -ISSN: 2664-2786

Article information

Received: 30/1/2022
Accepted: 13/4/2022

DOI: **10.33899/rjs.2022.175388**

corresponding author:
Maysaa K. Al-Mala Khder
maysaa.emad@uomosul.edu.iq

Moayed S. Al-Jawady
moyed.salim@yahoo.com

ABSTRACT

In this work, the 1,2,4-triazole ring was prepared by introducing the starting material ortho-carboxybenzaldehyde (1) in the usual esterification reaction, in ethanol as a solvent and in the presence of concentrated sulfuric acid to give ethyl-2-formylbenzoate (2), then introducing the resulting ester in a condensation reaction with thiosemberazide in the presence of ethanol as a solvent to afforded 2-(2-formylbenzoyl)hydrazine-1-carbothioamide (3), which on cyclisation using a solution of sodium hydroxide to obtain the 1,2,4-triazole compound (4), which on treatment with different primary aromatic amines to obtain Schiff bases (5a-e) the prepared Schiff bases were introduced into a reaction with chloroacetyl chloride in the presence of triethylamine as an auxiliary base for the reaction to prepare the tetracyclic azitidinone compounds (beta-lactams) (6a-e) along with the triazole ring in the same compound. Finally, these prepared compounds were characterized by physical and spectroscopic measurements such as melting point, thin layer chromatography (m.p., TLC) infrared spectroscopic measurements FT-IR, and nuclear magnetic resonance spectra of some compounds ^{1}H-NMR.

Keywords: ortho-carboxybenzaldehyde, triazole, azetidinone.

INTRODUCTION

The chemistry of heterocyclic compounds is a separate field of organic chemistry with a long history and future prospects. During this time, heterocyclic chemistry progresses the large number of compounds that have biological efficacy and therapeutic significance and synthetic methods to the synthesis of drugs. (Joule and Mills, 2010; D'Souza *et al.*, 2021)

Therefore, among the heterocyclic compounds triazoles represents a class of compounds key component of noteworthy course due to their applications in areas as different as pharmacology, (Kaur *et al.*, 2017) industry, (Lazar *et al.*, 2014) agriculture, (Toda *et al*., 2021) It is known that the vast majority of the drugs acquire derivatives of heterocyclic compounds as active ingredients. Mainly triazole the interest in this course of compounds is detailed by a wide range of biological activities; an immense number of them are already utilized in practical medicine as drugs, for example, voriconazole, triazolam, fluconazole, itraconazole, etc. (Wang *et al.*, 2014; Zhang *et al.*, 2014; Khan *et al.*, 2015; Galstyan *et al.*, 2019). In addition to its use as an anticancer, (Pokhodylo *et al.*, 2013) anti-HIV, (Feng *et al.*, 2021) antibacterial, (Santhanalakshmi *et al.*, 2020) antioxidant, (Saad *et al.*, 2018) antimalarial, (Batra *et al.*, 2018) anti-inflammatory, (Kotla and Chunduri, 2013) antifungal, (Harkala *et al.*, 2014) antitubercular, (Al-Jawady *et al.*, 2018) pesticides and herbicides, (Shneine and Alaraji, 2016) plant growth regulators, (Mohammad and Hassan, 2006; Mohammad *et al.*, 2007)

It is well known that the Schiff bases are characterized when a carbonyl compound is condensed with a primary amine (Ghosh *et al.*, 2019) which have a lot of applications in several fields such as inorganic chemistry, biological chemistry, and organic chemistry. Altogether, they symbolize extremely frequently utilized and useful scaffolds in medicinal chemistry. (Saleh *et al.*, 2019) which react with different reagents to give heterocyclic compounds.

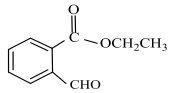
Finally, we are interested in heterocyclic compounds as we found that four-membered ring azitidinones derivatives are a key component group of heterocyclic compounds that have a wide range of biological activities it acts as anticancer, (Salunkhe and Piste, 2014) antimicrobial, (Bagherwal *et al.*, 2011) antibacterial, antifungal, (Patel *et al.*, 2011) and anti-inflammatory, (Rajasekaran *et al.*, 2010) These compounds were prepared through the reaction of Schiff bases with chloroacetylchloride.

EXPERINENTAL

All reagents and compounds are from fluka and BDH. Melting points were measured using an open capillary tube on Stuart SMP30 Melting point apparatus and they were uncorrected. The purity of the compounds was confirmed by TLC using silica gel and visualized in iodine. FT-IR spectra, fourier transform infrared SHIMADZU (8400) in the region between 400-40 cm^{-1} by using potassium bromide (KBr disc). ^{1}H-NMR spectra were recorded by Bruker ultra-shield 400 MHz origin: University of Ghazi Othman Pasha in Turkey and are reported in ppm (δ) DMSO-d_6 was used as a solvent with TMS as an internal standard.

General procedure for synthesis of ester compound.

Synthesis of ethyl-2-formylbenzoate (2): (Farooq and Ngaini, 2018; Jin, 2008; Kshirsagar *et al.*, 2009)

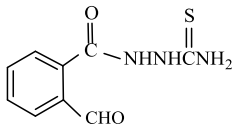


In round bottom flask, a mixture of (1.50 g, 0.01 mole) ortho-carboxy benzaldehyde (1) and (25ml) of absolute ethanol was added dropwise (1ml) of concentrated sulfuric acid with stirring and cooling, then refluxed for (6) hours, (monitored by TLC) the solvent system is (4:1) benzene: MeOH. After completion the reaction mixture was cooled and poured into crushed ice, then treated

with sodium bicarbonate with stirring to obtain the solid product, that was collected washed with water and recrystallized from ethanol as a white powder the product is (75%) (m.p. 62-63 ºC).

General procedure for synthesis of thiosemicarbazide derivative.

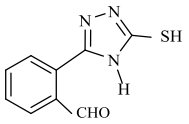
Synthesis of 2-(2-formylbenzoyl)hydrazine-1-carbothioamide (3): (Farag *et al.*, 2010)



To a mixture of ester compound (2) (1.78 g, 0.01 mole) in (25ml) of absolute ethanol was added (1.36 g, 0.015 mole) of thiosemicarbazide, then refluxed for (15) hours. (Monitored by TLC) the solvent system is (4:1) benzene: MeOH, after completion, filtered the hot mixture and the mixture was evaporated to its half volume, then let it overnight. Solid separated, which was filtered off washed with cold ethanol and recrystallized from ethanol to give the product as a white, (84%), with (m.p. 199-200 ºC).

General procedure for synthesis of triazole compound.

Synthesis of 2-(5-sulfanyl-4H-1,2,4-triazol-3-yl) benzaldehyde (4): (Cretu *et al*., 2010)



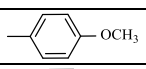
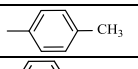
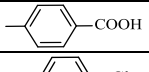
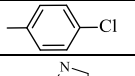
A mixture of thiosemicarbazide derivatives (3) (2.23 g, 0.01 mole) and 2N sodium hydroxide solution (8%, 25 mL) was heated under reflux for 4 h., the reaction mixture was cooled and poured into crushed ice, then neutralized with 10% dilute solution of HCl. The crude product was filtered off, washed with water and recrystallized from ethanol to afford the product as a white, (m.p. 247-249 °C), with yield (95%).

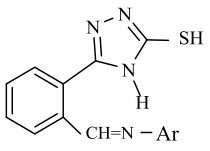
General procedure for the synthesis of Schiff bases.

Synthesis of 5-(2-{[(4- substituted phenyl)imino]methyl}phenyl)-4H-1,2,4-triazole-3-thiol (5a-e): (Khattab *et al*., 2007; Al-Ne'aimi *et al*., 2012)

A mixture of compound (4) (1.50 g, 0.01 mole) and substituted amines (0.01mole) in (20 ml) methanol. Two drops of acetic acid were added to the mixture, the mixture was refluxed for (6) hours (monitored by TLC) the solvent system is (4:1) benzene: MeOH, after completion, filtered the hot mixture and the mixture was evaporated to its half volume, then let it overnight. Solid separated, which was filtered off washed two times with (10 ml) of cold water and recrystallized from absolute ethanol to give the compounds (5a-e). Physical data of compounds (5a-e) are listed in (Table 1), Scheme (1).

Table 1: Physical properties of compounds (5a-e).

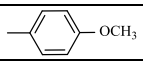
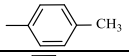
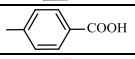
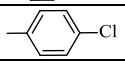
Comp. No.	Ar	M.P (°C)	Yield %	Colour
5a		230-232	64	Gray
5b		189-191	70	Brown
5c		209-211	60	White
5d		220-222	58	White
5e		178-180	53	Brown

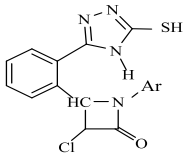


General procedure for the synthesis of azetidin-2-one compounds.
synthesis of 3-chloro-1-(4- substituted phenyl)-4-[2-(5-sulfanyl-4H-1,2,4-triazol-3-yl) phenyl]azetidin-2-one (6a-e): (Baviskar *et al.*, 2009; Al-Jawady and **Younis, 2017).**

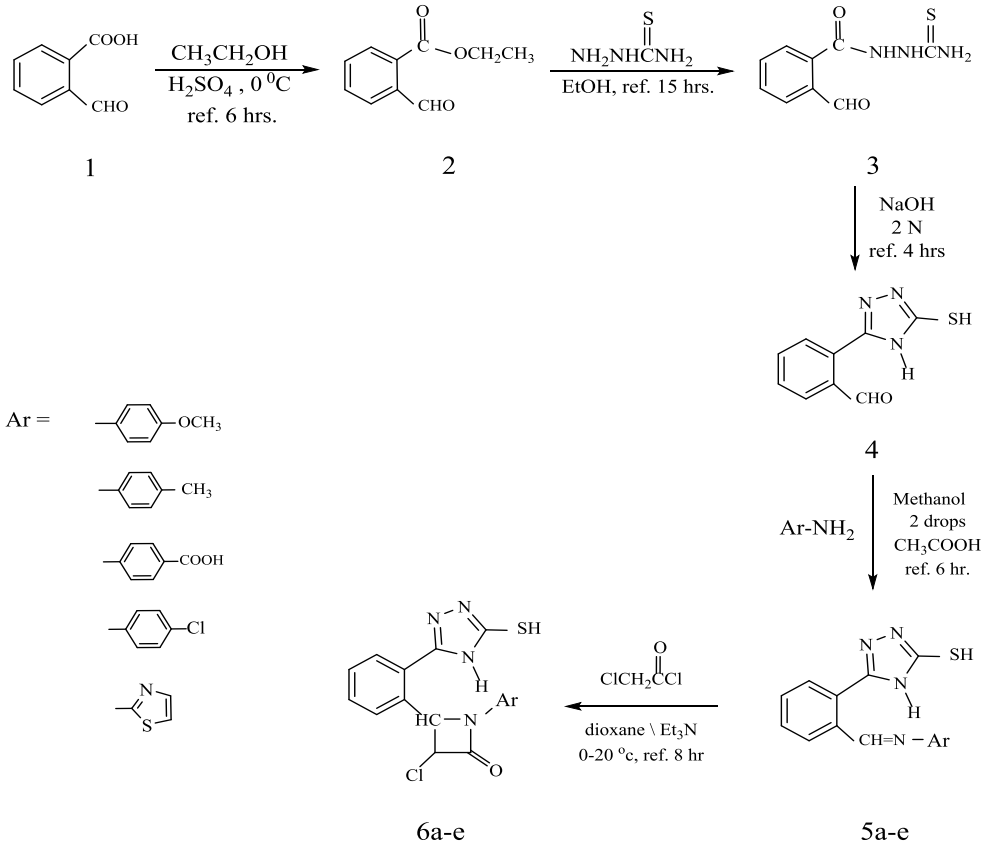
To a stirred solution of compounds (5a-e) (0.001mole) in 20 ml of dry 1,4-dioxane, triethylamine (0.05 ml, 0.001 mole) and chloroacetylchloride (0.1 ml ,0.001 mole) were added slowly dropwise with stirring at 0-20 °C. The reaction mixture was kept at room temperature for 30 minutes. Then reflux for (8) hours (monitored by TLC) the solvent system is (4:1) benzene: MeOH**,** the residue obtained after removal of the solvent under vacuum poured into 50 ml ice-cold water with stirring, solid separated filtered off recrystallized from ethanol to give compounds (6a-e). Physical data are listed in (Table 2), Scheme (1).

Table 2: Physical properties of compounds (6a-e).

Comp. No.	Ar	M.P (°C)	Yield %	Colour
6a		154-156	57	Gray
6b		148-150	54	Brown
6c		165-167	62	White
6d		132-134	63	Brown
6e		162-164	43	Brown



RESULTS AND DISCUSSION



Scheme (1)

Ortho-carboxybenzaldehyde was used as a synthone for this work used for synthesis of ester (2) by refluxing in ethanol in presence of conc. sulfuric acid, the ester (2) was characterized by physical and spectral data. The FT-IR spectrum revealed stretching absorption bands at 1782 cm^{-1} for carbonyl of ester (C=O) and 1703 cm^{-1} for carbonyl of aldehyde, and stretching absorption bands at 1141, 1288 cm^{-1} for sym. and a sym. of C-O bond, thus we noticed that the carboxyl group was disappeared. (Sherbeh *et al.*, 1985)

The ^{1}H-NMR spectrum for compound (2) in (DMSO-d6) in ppm showed significant peaks as the following a triplet peak at (1.25) ppm for protons of (CH_3) group, and quaternary peak at (3.89) ppm for protons of (CH_2) group, and a signal peak at (10.4) ppm for aldehydic proton and there is a multiple peak at (7.68-7.91) ppm for aromatic protons. (Table 5), Fig. (1).

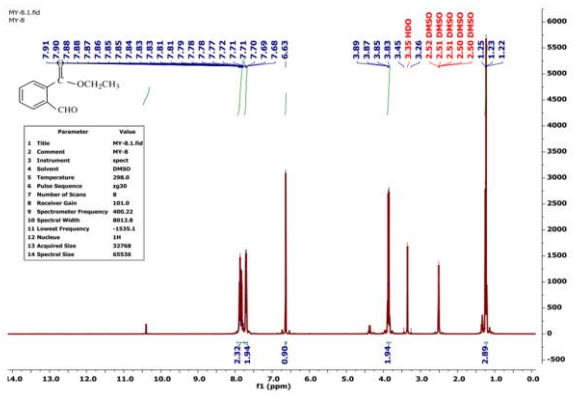


Fig. 1: ^{1}H-NMR spectrum of compound (2) is shown

The ester (2) was treated with thiosemicarbazide gave compound (3), the thiosemicarbazide derivative proved structure through the FT-IR spectrum revealed stretching absorption bands at 1683 cm^{-1} for carbonyl of amide (C=O) and 1743 cm^{-1} for carbonyl of aldehyde, and stretching absorption bands at 3431,3311,3277, and 3180 cm^{-1} for N-H and NH_2 groups and bending absorption bands at 1230, 819 cm^{-1} for C=S and C-S bond respectively.

The thiosemicarbazide derivative (3) was transformed to the triazole (4) by heating with 2N sodium hydroxide solution. The IR spectrum of the latter product confirmed function bands at 3431 and 2841 cm^{-1} corresponding to a N-H and SH functional groups for triazole ring, respectively. Similarly, to a band at 1680 cm^{-1} for carbonyl of aldehyde, and 821 cm^{-1} for C-S bond. (Kotla and Chunduri, 2013). Its ^{1}H-NMR spectrum displayed signals peak at (11.2) and (13.31) ppm corresponding to NH and SH protons, respectively, similarly to a multiple band at (7.47-8.27) ppm for aromatic protons. (Table 5), Fig. (2).

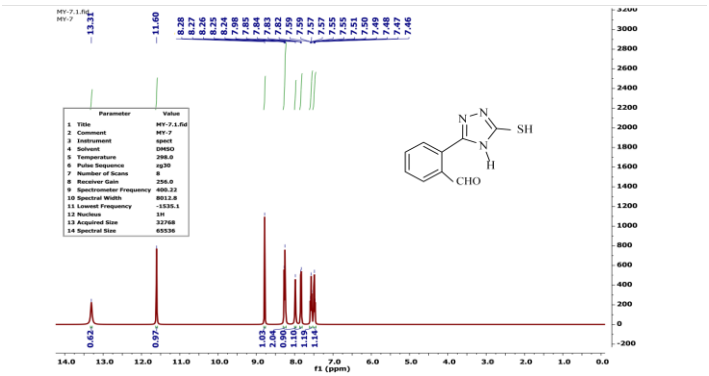
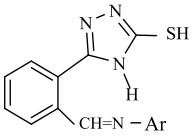
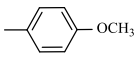
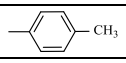
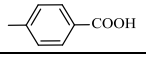
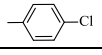


Fig. 2: ^{1}H-NMR spectrum of compound (4) is shown

Schiff bases compounds (5a-e) were prepared by reaction of compound (4) with different aromatic amines. The structure of these Schiff bases was established on the bases of their spectral data. The FT-IR spectra of Schiff base (5a-e) compounds confirmed an absorption seemed at (1597-1641) cm^{-1} for the new bond C=N, and there is a bending absorption seemed on the range of (634-713) cm^{-1} for the C-S group. We observed that the aldehydic carbonyl became disappeared, and the stretching absorption of N-H bond appeared at (3306-3431) cm^{-1}. (Table 3).

Table 3: IR spectral data for compounds (5a-e)



Comp. No.	Ar	IR (KBr) ν cm^{-1}			
		C=N	C-S	N-H	Others
5a		1625	669	3377	1294 asym. 1143 sym. for C-O-C
5b		1602	669	3431	
5c		1610	713	3306	
5d		1597	667	3429	759 for C-Cl
5e		1641	634	3309	

T

he ^{1}H-NMR spectra, we take the spectrum of compound (5b) as a sample, there is a band at (2.42) ppm for protons of methyl group, and the proton of CH=N group, we noticed it at (8.78) ppm, and there is a multiple peak at (7.47-8.27) ppm for aromatic protons, and there is another peak at (11.6) ppm for the NH group of traizole ring. The S-H band, it appears at (13.31) ppm which disappeared when we added deuterated water. (Table 5), Fig. (3).

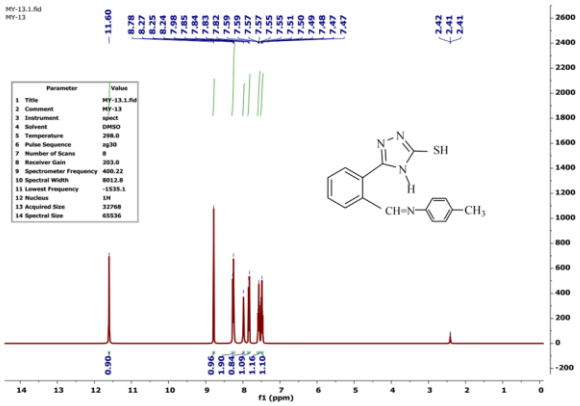
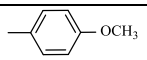
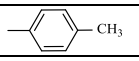
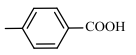
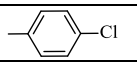
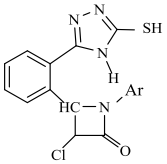


Fig. 3: ^{1}H-NMR spectrum of compound (5b) is shown

The azetidine compounds (6a-e) that have a four membered ring had been elucidated at the bases in their spectral data. The FT-IR spectra confirmed an absorption at (742-769) cm^{-1} for C-Cl bond and there may be absorption at (1377-1383) cm^{-1} for C-N bond and there is an absorption on the range of (1666-1708) cm^{-1} for a lactam carbonyl and the C-S bond appeared at (682-690) cm^{-1} and N-H group of traizole seemed at (3315- 3429) cm^{-1}. (Table 4).

Table 4: IR spectral data for compounds (6a-e)

Comp. No.	Ar	IR (KBr) ν cm^{-1}					
		C=O lactam	C-Cl	C-N	C-S	N-H	Others
6a		1678	742	1377	688	3363	
6b		1666	750	1381	682	3429	
6c		1697	769	1379	690	3315	1776,3344 for C=O, OH acid
6d		1678	750	1383	686	3402	
6e		1708	754	1377	686	3348	



For ^{1}H-NMR we take compound 6b as a sample for compounds (10-14). There is an absorption at (2.42) ppm for CH_3 group, (3.57) ppm for proton of CH-Cl group and at (4.29) ppm for CH-N group, and for aromatic protons, they regarded at (7.49- 8.11) ppm. The proton of N-H bond, it seems at (11.6) ppm. The S-H band, it appears at (13.31) ppm which disappeared when we added deuterated water. (Table 5), Fig. (4).

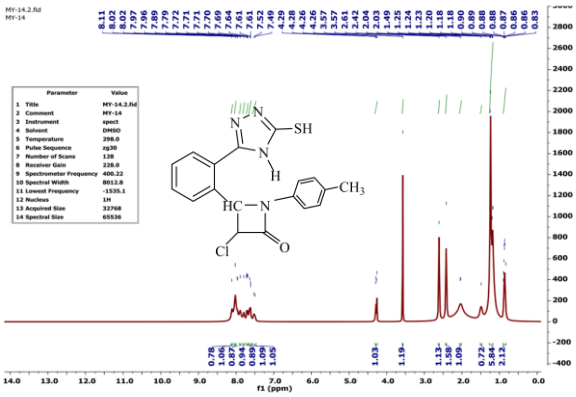


Fig. 4: ^{1}H-NMR spectrum of compound (6b) is shown

Table 5: ^{1}H-NMR spectral data for compounds

Comp. No.	^{1}H-NMR δ (ppm) DMSO-$_6$
2	1.25, t,3H, (CH_3), 3.89, q, 2H, (CH_2), 10.4, s,1H, (CHO), 7.68-7.91, m,4H, (ArH)
4	7.46-8.27, m,4H, (ArH), 11.6, s, 1H, (N-H), 13.31, s,1H, (S-H)
5b	2.42, s,3H, (CH_3), 8.78, s,1H, (CH=N), 7.47-8.27, m, 4H, (ArH), 11.6, s,1H, (N-H), 13.31, s,1H, (S-H)
6b	2.42, s,3H, (CH_3), 3.57, s,1H, (CH-Cl), 4.29, s,1H, (CH-N), 7.49-8.11, m, 4H, (ArH), 11.6, s,1H, (N-H), 13.31, s,1H, (S-H)

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تحضير بعض مشتقات الترايازول الجديدة باستعمال اورثو- كاربوكسي بنزالديهايد كمادة أولية

الملخص

في هذا العمل تم تحضير حلقة 4,2,1- ترايازول من خلال ادخال المادة الأولية اورثو- كاربوكسي بنزالديهايد في تفاعل الاسترة والتي تعرف باسترة فيشر في الايثانول كمذيب وبوجود حامض الكبريتيك المركز ومن ثم ادخال الاستر الناتج (2) في تفاعل تكاثف مع الثايوسيمسكاربازيد بوجود الايثانول كمذيب لينتج عن هذا التفاعل مركب الثايوسيميكاربازيد (3) الذي سيتم إدخاله في تفاعل حولقة باستخدام محلول هيدروكسيد الصوديوم للحصول على حلقة 4,2,1- ترايازول (4) وفيما بعد يتفاعل مركب 4,2,1- ترايازول (4) الناتج من المادة الأولية (1) مع امينات اروماتية اولية مختلفة باستعمال الميثانول كمذيب واضافة حامض الخليك للحصول على قواعد شيف المقابلة (5a-e) ومن ثم ادخال قواعد شيف المحضرة في تفاعل مع كلوريد كلورو اسيتيل بوجود ثلاثي اثيل امين كقاعدة مساعدة للتفاعل لتحضير مركبات الازيتيدينون رباعية الحلقة (بيتا- لاكتام) (6a-e) الى جانب حلقة الترايازول في المركب نفسه. وفي النهاية شخصت هذه المركبات المحضرة من خلال قياس درجة الانصهار وتتبع التفاعل بكروماتوكرافيا الطبقة الرقيقة (*TLC, m.p.*) والقياسات الطيفية لطيف الاشعة تحت الحمراء FT-IR وطيف الرنين النووي المغناطيسي ^{1}HNMR لبعض المركبات.

الكلمات الدالة: اورثو- كاربوكسي بنزالديهايد، ترايازول، ازيتيدينون.