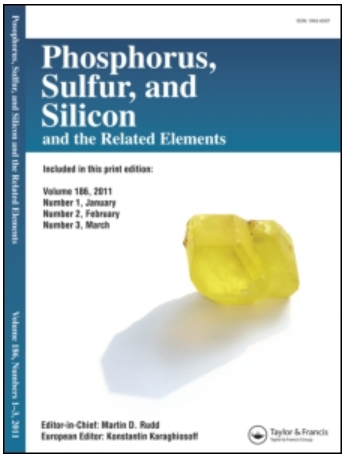


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# Synthesis and Studies of Some Nitrogen and Sulfur Compounds

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# SYNTHESIS AND STUDIES OF SOME NITROGEN AND SULFUR COMPOUNDS

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*Pyrazolo-, pyrimidino-, isoxazolo-, thiozolo-, and β-lactam incorporating thienopyridazine has been synthesised by cyclocondensation addition reaction and cycloaddition reaction of hydrazine hydrate phenyl hydrazine, hydroxylamine hydrochloride, urea, thiourea, mercapto acetic acid and monochloroacetyl chloride.*

*Keywords:* Nitrogen; pyrazolo; sulfur

## INTRODUCTION

A five and six membered heterocyclic systems containing nitrogen-, oxygen-, and sulfur atoms of fused and spiro pyrazolo-, pyrimidino-, isoxazolo-, thiazolo-, and $\beta$-lactam incorporating thienopyridazine were found to possess interesting biological properties[1–5] and as synthetic drugs.[6–10] The chemistry of these heterocyclic compounds has received much attention in recent years. This principally due to the unique physical and chemical properties of such compounds, which enable their wide application as plant growth regulators.[11] Recently, we have reported the synthesis and structure activity relationship of variety heterocyclic and aromatic derivatives of theinopyridazines.[12,13] In our screening substances showing interesting biological activity and unusual chemical structures have been detected. The molecules described in this work are composed of a five-, and six-membered heterocyclic systems containing nitrogen-, oxygen-, and sulfur atoms. However various styryl compounds and Schiff base compounds incorporating the theinopyridazine residue we have been synthesized and studies.

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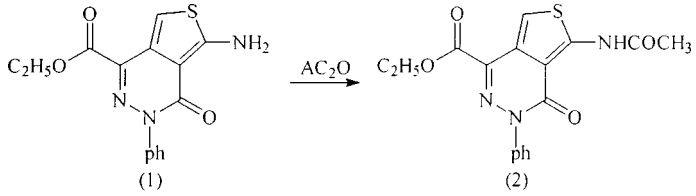
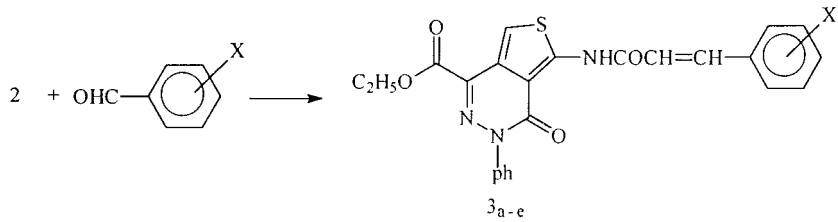
Therefore, several fused and spiro pyrazolo-, pyrimidino-, isoxazolo-, thiazolo-, and $\beta$-lactam derivatives are synthesized and tested for biological activity.

## RESULTS AND DISCUSSION

Acetylation of aminotheinopyridazine derivative (**1**) with one mole equivalent of acetic anhydride yielded the corresponding acetylaminotheinopyridazine derivative (**2**).[14]

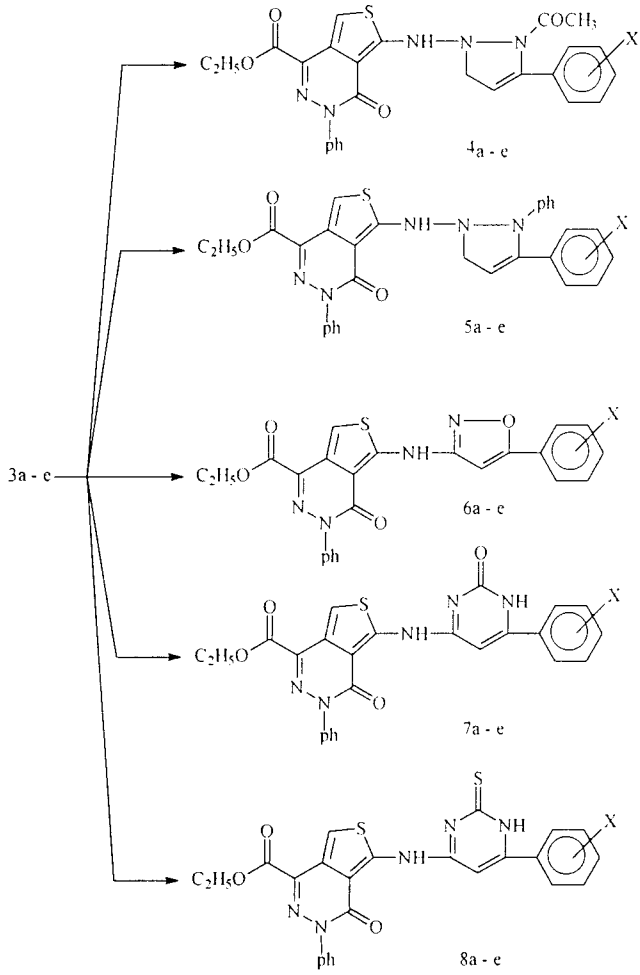



Synthesis of chalcone compound derivatives (**3a–e**) were achieved through the interaction of the theinopyridazine (**2**) with aromatic aldehydes such as p. hydroxybenzaldehyde $\rho$-nitrobenzaldehyde, benzaldehyde, $\rho$-N-dimethylbenzaldehyde and $\rho$-chlorobenzaldehyde using piperidine as catalyst in the presence of ethanol as solvent afforded chalcone compound dervative **3a–e**. The structure of **3a–e** were confirmed by Microanalysis data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II).




Where: a, X = ρ. OH; b, X = ρ. NO2; c, X = H; d, X = ρ. N(CH3)2; e, X = ρ. Cl

The chalcone compound derivatives **3a–f** when interacted with one mole of hydrazine hydrate in presence of acetic acid, and one mole of phenyl hydrazine under piperidine catalyzed, gave the required N-acetyl-, and N-phenyl-, pyrazolo derivatives (**4a–e** and **5a–e**) respectively, (Scheme 1). The structure of (**4a–e** and **5a–e**) were confirmed by Microanalysis data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II).




**SCHEME 1**

Synthesis of isoxazolo compound derivatives (**6a–e**) were achieved through the interaction of chalcone compound derivatives (**3a–e**) with one mole of hydroxylamine hydrochloride in ethanol as organic solvent under effect of sodium hydroxide as catalyst formed isoxazolo compound derivatives **6a–e**, Scheme (1). The structure of (**6a–e**) were confirmed by Microanalysis data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II). Also, synthesis of pyrimidines compound derivatives (**7a–e** and **8a–e**), were achieved through the interacted of chalcone compound derivatives (**3a–e**) with one mole of urea in ethanol as solvent

**TABLE I** Characterization of Compounds **3–8**

| Comp. no. | Solvent of crystalization | m.p. °C | Yield % | Color | Formula (m. wt) | Analytical data found/required % C | H | N | MS (m/z) |
|---|---|---|---|---|---|---|---|---|---|
| **3a** | Ethanol | 170 | 40 | Green | $C_{24}H_{19}N_3O_5S$ | 62.42 | 4.12 | 9.11 | 460 |
| | | | | | (461) | 62.40 | 4.12 | 9.10 | |
| **3b** | Ethanol | 220 | 11 | Move brown | $C_{24}H_{18}N_4O_6S$ | 58.77 | 3.67 | 11.42 | 492 |
| | | | | | (490) | 58.77 | 3.66 | 11.41 | |
| **3c** | Ethanol | 160 | 45 | Green | $C_{24}H_{19}N_3O_5S$ | 64.92 | 4.08 | 9.46 | 445 |
| | | | | | (445) | 64.93 | 4.08 | 9.44 | |
| **3d** | Ethanol | 170 | 25 | Greenish blue | $C_{26}H_{24}N_4O_4S$ | 63.99 | 4.95 | 11.47 | 488 |
| | | | | | (488) | 63.97 | 4.91 | 11.45 | |
| **3e** | Ethanol | 165 | 59 | Pale green | $C_{24}H_{18}N_3O_4SCl$ | 60.12 | 3.75 | 8.76 | 479 |
| | | | | | (479) | 60.10 | 3.77 | 8.77 | |
| **4a** | Methanol | 230 | 60 | Yellow | $C_{26}H_{21}N_5O_5S$ | 60.58 | 4.01 | 13.59 | 515 |
| | | | | | (515) | 60.60 | 11.00 | 13.57 | |
| **4b** | Ethanol | 190 | 55 | Green | $C_{26}H_{20}N_6O_6S$ | 57.35 | 3.67 | 15.44 | 544 |
| | | | | | (544) | 57.35 | 3.68 | 15.41 | |
| **4c** | Methanol | 183 | 50 | Green | $C_{26}H_{21}N_5O_4S$ | 62.52 | 4.20 | 14.02 | 499 |
| | | | | | (499) | 62.50 | 4.00 | 14.00 | |
| **4d** | Ethanol | 160 | 45 | Brown | $C_{28}H_{26}N_6O_4S$ | 61.99 | 4.79 | 15.49 | 542 |
| | | | | | (542) | 62.00 | 4.76 | 15.51 | |
| **4e** | Ethanol | 180 | 40 | Green | $C_{26}H_{19}N_5O_4SCl$ | 58.53 | 3.75 | 13.13 | 532 |
| | | | | | (532) | 58.54 | 3.73 | 13.10 | |
| **5a** | Ethanol | 150 | 45 | Brown | $C_{30}H_{23}N_5O_4S$ | 65.5 | 4.18 | 12.75 | 551 |
| | | | | | (549) | 65.49 | 4.20 | 12.76 | |
| **5b** | Ethanol | 180 | 35 | Brown | $C_{30}H_{22}N_6O_5S$ | 62.28 | 3.80 | 14.53 | 578 |
| | | | | | (578) | 62.30 | 3.80 | 14.55 | |
| **5c** | Ethanol | 135 | 40 | Orange | $C_{30}H_{23}N_5O_3S$ | 67.66 | 4.31 | 13.13 | 533 |
| | | | | | (533) | 67.65 | 4.30 | 13.14 | |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **5d** | Ethanol | 130 | 55 | Green | $C_{32}H_{28}N_6O_3S$ | 66.66 | 4.86 | 14.58 | 576 |
| | | | | | (576) | 66.64 | 4.66 | 14.58 | |
| **5e** | Ethanol | 160 | 45 | Green | $C_{30}H_{22}N_5O_3SCl$ | 63.44 | 3.88 | 12.34 | 567 |
| | | | | | (567) | 63.44 | 3.88 | 12.35 | |
| **6a** | Ethanol | 230 | 65 | Green | $C_{24}H_{18}N_4O_5S$ | 60.75 | 3.79 | 11.81 | 474 |
| | | | | | (474) | 60.76 | 3.80 | 11.80 | |
| **6b** | Methanol | 170 | 60 | Yellow | $C_{24}H_{17}N_5O_6S$ | 57.25 | 3.37 | 13.91 | 503 |
| | | | | | (503) | 57.27 | 3.37 | 13.40 | |
| **6c** | Methanol | 158 | 35 | Green | $C_{24}H_{18}N_4O_4S$ | 63.15 | 3.94 | 12.28 | 458 |
| | | | | | (458) | 63.15 | 3.94 | 12.30 | |
| **6d** | Ethanol | 142 | 50 | Brown | $C_{26}H_{23}N_5O_4S$ | 62.27 | 4.59 | 13.97 | 501 |
| | | | | | (501) | 62.30 | 4.58 | 13.94 | |
| **6e** | Ethanol | $>300$ | 25 | Brown | $C_{24}H_{17}N_4O_4SCl$ | 58.53 | 3.46 | 11.38 | 492 |
| | | | | | (492) | 58.51 | 3.45 | 11.40 | |
| **7a** | Ethanol | 210 | 30 | Green | $C_{25}H_{18}N_5O_5S$ | 61.98 | 3.71 | 14.46 | 484 |
| | | | | | (484) | 62.00 | 3.70 | 14.45 | |
| **7b** | Methanol | 225 | 55 | Green | $C_{25}H_{18}N_6O_6S$ | 56.71 | 3.21 | 15.87 | 530 |
| | | | | | (530) | 56.70 | 3.20 | 15.89 | |
| **7c** | Ethanol | 130 | 45 | Brown | $C_{25}H_{19}N_5O_5S$ | 61.80 | 3.91 | 14.43 | 485 |
| | | | | | (485) | 61.9 | 3.90 | 14.43 | |
| **7d** | Ethanol | 145 | 70 | Yellow | $C_{27}H_{24}N_6O_4S$ | 61.36 | 4.54 | 15.91 | 528 |
| | | | | | (528) | 61.35 | 4.55 | 15.89 | |
| **7e** | Ethanol | 142 | 50 | Pale green | $C_{25}H_{18}N_5O_4SCl$ | 57.8 | 3.46 | 13.48 | 519 |
| | | | | | (519) | 57.6 | 3.46 | 13.50 | |
| **8a** | Methanol | 210 | 55 | Black | $C_{25}H_{18}N_5O_4S_2$ | 60 | 3.60 | 14.00 | 500 |
| | | | | | (500) | 60.03 | 3.60 | 14.35 | |
| **8b** | Ethanol | 200 | 50 | Brown | $C_{25}H_{18}N_5O_5S_2$ | 54.94 | 3.29 | 15.38 | 546 |
| | | | | | (546) | 54.92 | 3.31 | 15.40 | |
| **8c** | Methanol | 220 | 45 | Green | $C_{25}H_{19}N_5O_3S_2$ | 59.88 | 3.79 | 13.97 | 501 |
| | | | | | (501) | 59.91 | 3.74 | 13.95 | |



*(Continued on next page)*

**TABLE I** Characterization of Compounds **3–8** *(Continued)*

| Comp. no. | Solvent of crystalization | m.p. °C | Yield % | Color | Formula (m. wt) | Analytical data found/required % C | H | N | MS (m/z) |
|---|---|---|---|---|---|---|---|---|---|
| **8d** | Ethanol | 150 | 79 | Green | $C_{27}H_{24}N_6O_3S_2$ | 59.55 | 4.41 | 15.44 | 544 |
| | | | | | (544) | 59.54 | 4.40 | 15.41 | |
| **8e** | Ethanol | 150 | 60 | Green | $C_{25}H_{18}N_5O_2Cl$ | 56.07 | 3.36 | 13.08 | 535 |
| | | | | | (535) | 56.09 | 3.36 | 13.10 | |
| **9a** | Methanol | 150 | 85 | Deep violet | $C_{27}H_{20}N_5O_5S$ | 63.15 | 4.09 | 10.91 | 512 |
| | | | | | (512) | 63.17 | 4.08 | 10.90 | |
| **9b** | Methanol | 170 | 80 | Deep violet | $C_{27}H_{20}N_5O_5S$ | 63.15 | 4.09 | 10.91 | 512 |
| | | | | | (512) | 63.15 | 4.10 | 10.92 | |
| **9c** | Ethanol | 175 | 65 | Green | $C_{27}H_{23}N_6O_5S$ | 59.66 | 4.23 | 15.46 | 543 |
| | | | | | (543) | 59.65 | 4.24 | 15.47 | |
| **10a** | Ethanol | 168 | 50 | Move | $C_{29}H_{22}N_4O_6S_2$ | 59.38 | 3.75 | 9.55 | 586 |
| | | | | | (586) | 59.40 | 3.72 | 9.57 | |
| **10b** | Ethanol | 158 | 60 | Black | $C_{29}H_{22}N_4O_6S_2$ | 59.38 | 3.75 | 9.55 | 586 |
| | | | | | (586) | 59.37 | 3.76 | 9.58 | |
| **10c** | Ethanol | 170 | 65 | Green | $C_{29}H_{23}N_6O_6S_2$ | 56.58 | 3.73 | 13.65 | 615 |
| | | | | | (615) | 56.60 | 3.74 | 13.67 | |
| **11a** | Ethanol | 150 | 70 | Move | $C_{29}H_{21}N_4O_6SCl$ | 59.18 | 3.57 | 9.52 | 588 |
| | | | | | (588) | 59.17 | 3.56 | 9.50 | |
| **11b** | Methanol | 150 | 50 | Violet | $C_{29}H_{21}N_4O_6SCl$ | 59.18 | 3.57 | 9.52 | 588 |
| | | | | | (588) | 59.21 | 3.57 | 9.55 | |
| **11c** | Ethanol | 160 | 45 | Brown | $C_{29}H_{21}N_6O_6SCl$ | 56.40 | 3.40 | 13.61 | 617 |
| | | | | | (617) | 56.39 | 3.41 | 13.63 | |

**TABLE II** Selected IR, $^1$H NMR Spectra Data for the New Compounds Listed in Table I

| Compound no. | IR ($cm^{-1}$) | $^1$H NMR $\delta$ (ppm) |
|---|---|---|
| **3a** | 3274 (OH), 3133 (NH), 1702 (C=O ester), 1654 (C=O amide), 1540 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 2.7 (s, 1H), 4.4 (q, 2H, J = 7.2 HZ), 6.20 (d, 1H, J = 15.90 $H_2$), 6.40 (d, 1H, J = 15.90 $H_2$), 7.46–7.57 (m, 9H), 7.92 (s, 1H), 11.06 (s, 1H). |
| **3b** | 3150 (NH), 1750 (C=O ester), 1670 (C=O amide), 1555 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.20 (d, 1H, J = 15.9), 6.40 (d, 1H, J = 15.9 Hz), 7.46–7.57 (m, 9H), 7.92 (s, 1H), 11.06 (s, 1H). |
| **3c** | 3140 (NH), 1725 (C=O ester), 1680 (C=O amide), 1550 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.20 (d, 1H, J = 15.90 Hz), 6.40 (d, 1H, J = 15.90 Hz), 7.46–7.57 (m, 10H), 7.92 (s, 1H), 11.06 (s, 1H). |
| **3d** | 3155 (NH), 1710 (C=O ester), 1650 (C=O amide), 1525 (C=N) | 1.2 (m, 6H), 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.20 (d, 1H, J = 15.90 Hz), 6.40 (d, 1H, J = 15.90 Hz), 7.46–7.57 (m, 9H), 7.92 (s, 1H). 11.06 (s, 1H). |
| **3e** | 3150 (NH), 1699 (C=O ester), 1660 (C=O amide), 1530 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.20 (d, 1H, J = 15.90 Hz), 6.40 (d, 1H, J = 15.90 Hz), 7.46–7.57 (m, 9H), 7.92 (s, 1H), 11.06 (s, 1H),. |
| **4a** | 3260 (OH), 3120 (NH), 1700 (C=O ester), 1690 (C=O ketone), 1540 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 3.5 (s, 3H), 6.60 (s, 1H), 7.50–7.60 (m, 9H), 7.94 (s, 1H), 11.00 (s, 1H), 11.05 (s, 1H). |
| **4b** | 3113 (NH), 1709 (C=O ester), 1699 (C=O ketone), 1561 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 3.5 (s, 3H), 4.4 (q, 2H, J = 7.2 Hz), 6.60 (s, 1H), 7.50–7.60 (m, 9H), 7.94 (s, 1H), 11.05 (s, 1H). |
| **4c** | 3145 (NH), 1705 (C=O ester), 1700 (C=O ketone), 1570 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 3.5 (s, 3H), 4.4 (q, 2H, J = 7.2 Hz), 6.60 (s, 1H), 7.46–7.57 (m, 10H), 7.92 (s, 1H), 11.06 (s, 1H). |
| **4d** | 3140 (NH), 3400 (NH), 1720 (C=O ester), 1690 (C=O ketone), 1540 (C=N) | 1.2 (m, 6H), 1.4 (t, 3H, J = 7.2 Hz), 3.5 (s, 3H), 4.4 (q, 2H, J = 7.2 Hz), 6.55 (s, 1H), 7.43–7.58 (m, 9H), 7.90 (s, 1H), 11.00 (s, 1H). |
| **4e** | 3130 (NH), 1739 (C=O ester), 1680 (C=O keton) 1555 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 3.5 (s, 3H), 4.4 (q, 2H, J = 7.2 Hz), 6.60 (s, 1H), 7.46–7.57 (m, 9H), 7.92 (s, 1H), 11.00 (s, 1H). |

*(Continued on next page)*

**TABLE II** Selected IR, $^1$H NMR Spectra Data for the New Compounds Listed in Table I *(Continued)*

| Compound no. | IR ($cm^{-1}$) | $^1$H NMR $\delta$ (ppm) |
|---|---|---|
| **5a** | 3300 (OH), 3110 (NH), 1750 (C=O ester), 1690 (C=O ketone), 1550 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 2.7 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.58 (m, 14H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **5b** | 3100 (NH), 1765 (C=O ester), 1710 (C=O ketone), 1530 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.58 (m, 14H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **5c** | 3115 (NH), 1750 (C=O ester), 1715 (C=O ketone), 1515 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H) 7.45–7.58 (m, 15H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **5d** | 3225 (NH), 1746 (C=O ester), 1720 (C=O ketone), 1520 (C=N) | 1.2 (m, 6H), 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.58 (m, 14H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **5e** | 3220 (NH), 1740 (C=O ester), 1720 (C=O ketone), 1525 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.58 (m, 14H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **6a** | 3295 (OH), 3125 (NH), 1760 (C=O ester), 1710 (C=O ketone), 1555 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.58 (m, 9H), 7.95 (s, 1H), 8 (s, 1H), 11.08 (s, 1H). |
| **6b** | 3122 (NH), 1770 (C=O ester), 1712 (C=O ketone), 1525 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H) 7.45–7.58 (m, 9H), 7.95 (s, 1H), 11.08 (s, 1H). |
| **6c** | 3120 (NH), 1755 (C=O ester), 1720 (C=O ketone), 1520 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.63 (s, 1H) 7.42–7.55 (m, 910H), 7.93 (s, 1H), 11.05 (s, 1H). |
| **6d** | 3240 (NH), 1750 (C=O ester), 1715 (C=O ketone), 1526 (C=N) | 1.2 (m, 6H), 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.60 (s, 1H), 7.40–50 (m, 9H), 7.90 (s, 1H), 11.00 (s, 1H). |
| **6e** | 3220 (NH), 1735 (C=O ester), 1710 (C=O keton) 1530 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.65 (s, 1H), 7.45–7.55 (m, 9H), 7.95 (s, 1H), 11.06 (s, 1H). |
| **7a** | 3280 (OH), 3010 (NH), 1700 (C=O ester), 1635 (C=O ketone), 1560 (C=N) | 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H), 7.50–8.01 (m, 9H), 7.96 (s, 1H), 11.08 (s, 1H), 11.50 (s, 1H). |
| **7b** | 3105 (NH), 1715 (C=O ester), 1689 (C=O ketone), 1540 (C=N) | 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 7.50–8.01 (m, 9H), 6.70 (s, 1H) 7.96 (s, 1H), 11.08 (s, 1H) 11.50 (s, 1H). |
| **7c** | 3155 (NH), 1740 (C=O ester), 1695 (C=O ketone), 1555 (C=N) | 1.4 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.2–7.7 (m, 10H), 7.6 (s, 1H), 11.05 (s, 1H) 11.40 (s, 1H). |

**TABLE II** Selected IR, $^1$H NMR Spectra Data for the New Compounds Listed in Table I *(Continued)*

| Compound no. | IR ($cm^{-1}$) | $^1$H NMR $\delta$ (ppm) |
|---|---|---|
| **7d** | 3205 (NH), 1760 (C=O ester), 1699 (C=O ketone), 1570 (C=N) | 1.3 (m, 6H), 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.6–8.20 (m, 9H), 8.4 (s, 1H), 11.06 (s, 1H) 11.45 (s, 1H). |
| **7e** | 3189 (NH), 1750 (C=O ester), 1705 (C=O ketone), 1585 (C=N) | 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.6–8.2 (m, 9H), 8.4 (s, 1H), 11.06 (s, 1H), 11.45 (s, 1H). |
| **8a** | 3298 (OH), 3190 (NH), 1718 (C=O ester), 1645 (C=O ketone), 1592 (C=N) | 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.6–8.2 (m, 9H), 8.4 (s, 1H), 11.06 (s, 1H), 11.45 (s, 1H), 12 (s, 1H). |
| **8b** | 3199 (NH), 1765 (C=O ester), 1690 (C=O ketone), 1550 (C=N) | 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.6–8.2 (m, 9H), 8.4 (s, 1H), 11.06 (s, 1H), 11.45 (s, 1H). |
| **8c** | 3210 (NH), 1755 (C=O ester), 1705 (C=O ketone), 1560 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 4.4 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H) 7.4–8.0 (m, 10H), 8.4 (s, 1H), 11.45 (s, 1H) 11.05 (s, 1H). |
| **8d** | 3195 (NH), 1750 (C=O ester), 1710 (C=O ketone), 1549 (C=N) | 1.3 (m, 6H), 1.6 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H), 7.6–8.2 (m, 9H), 8.4 (s, 1H),11.06 (s, 1H), 11.45 (s, 1H). |
| **8e** | 3205 (NH), 1765 (C=O ester), 1715 (C=O keton) 1559 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 4.6 (q, 2H, J = 7.2 Hz), 6.70 (s, 1H), 7.6–8.2 (m, 9H), 8.4 (s, 1H), 11.06 (s, 1H), 11.45 (s, 1H). |
| **9a** | 3300 (OH), 3200 (NH), 1700 (C=O ester), 1650 (C=O amide), 1540 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 3.5 (s, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.57 (m, 11H), 7.95 (s, 1H), 11.06 (s, 1H). |
| **9b** | 3300 (OH), 3200 (NH), 1700 (C=O ester), 1650 (C=O amide), 1540 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 3.5 (s, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.57 (m, 11H), 7.95 (s, 1H), 11.06 (s, 1H). |
| **9c** | 3300 (OH), 3200 (NH), 1700 (C=O ester), 1650 (C=O amide), 1540 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 1.5 (s, 3H), 3.5 (s, 1H) 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 10H), 7.9 (s, 1H). 11.06 (s, 1H). |
| **10a** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1540 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 2 (s, 2H), 3.2 (b, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 11H), 7.9 (s, 1H), 11.06 (s, 1H). |

*(Continued on next page)*

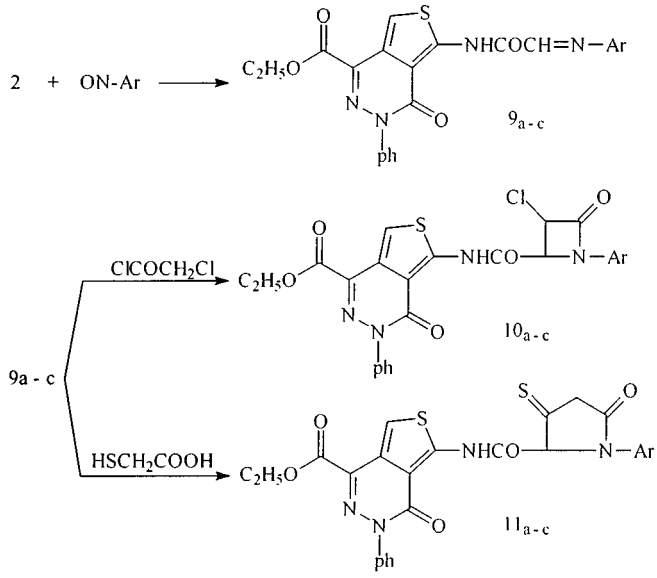
**TABLE II** Selected IR, $^1$H NMR Spectra Data for the New Compounds Listed in Table I *(Continued)*

| Compound no. | IR ($cm^{-1}$) | $^1$H NMR $\delta$ (ppm) |
|---|---|---|
| **10b** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1640 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 2 (s, 2H), 3.2 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 11H), 7.9 (s, 1H), 11.06 (s, 1H). |
| **10c** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1640 (C=N) | 1.5 (s, 3H), 1.3 (t, 3H, J = 7.2 Hz), 2 (s, 2H), 3.2 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 10H), 7.9 (s, 1H), 11.06 (s, 1H). |
| **11a** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1640 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 3.2 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 11H), 7.9 (s, 1H), 11.06 (s, 1H). |
| **11b** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1640 (C=N) | 1.3 (t, 3H, J = 7.2 Hz), 3.2 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 11H), 7.9 (s, 1H), 11.06 (s, 1H). |
| **11c** | 3250 (OH), 3150 (NH), 1700 (C=O ester), 1650 (C=O amide), 1640 (C=N) | 1.5 (s, 3H), 1.3 (t, 3H, J = 7.2 Hz), 3.2 (br, 1H), 4.4 (q, 2H, J = 7.2 Hz), 5.8 (s, 1H), 7.46–7.56 (m, 10H), 7.9 (s, 1H), 11.06 (s, 1H),. |



under the effect of concentrated hydrochloride acid as catalyst and one mole of thiourea in ethanol as organic solvent under the effect of sodium hydroxide as catalyst produced **7a–e** and **8a–e** respectively, (Scheme 1). The structure of (**7a–e** and 8**a–e**) were confirmed by Microanalytical data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II). When chalcone compound derivatives (**3a–f**) were condensed with nitroso compounds such as $\alpha$-nitroso-$\beta$-naphthol, $\beta$-nitroso-$\alpha$-naphthol, 3-methy1-4-oxime-1-pheny1-5-pyrazolone in the presence piperidine as catalyst to give corresponding Schiff base derivatives (**9a–c**), (Scheme 2). The structure of (**9a–c**) were confirmed by Microanalytical data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II). Cycloaddition reaction of thioglycolic acid to previously prepared Schiff bases compound derivatives **9a–c** proceeded successfully. Thus, thioglycolic acid was added to **9a–c** in boiling benzene using a water separator to give thiazolidinone derivatives **10a–c** (Scheme 2). The structure of **10a–c** were confirmed by Microanalytical data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II).

The reaction of chloroacetyl chloride in dioxane in the presence of triethylamine as catalyst afforded $\beta$-lactam derivatives **11a–c** (Scheme 2).




Where : a, Ar = α-nitroso-β-naphthol; b, Ar = β-nitroso-α-naphthol; c, Ar = 3-methyl-4-ox ime-1-phenyl-5-pyrazolone.

**SCHEME 2**

The structures of (**11a–c**) were confirmed by Microanalytical data, Mass spectra, Infrared spectra and $^1$H NMR spectra (c.f. Tables I and II).

## EXPEREMENTAL

All melting points are uncorrected. The IR spectra were recorded on a Perkin Elmer/1650 ET-IR spectrum, $^1$H NMR spectra on an EM-390 90MHz NMR spectrometer, mass spectra on an MS 5988 and Analytical data were determined with a CE 440 Elemental Analyzer-Automatic Injector at Cairo University.

### Synthesis of Chalcone Compound Derivatives (3a–e)

#### *General Procedure (1)*

To a solution of 2 (0.001 mol, 0.5 g) and aromatic aldehydes (0.001 mol, 0.17 g, 0.21 g, 0.14 ml, 0.20 g, and 0.19 g resp.) in absolute alcohol, two drops of piperidine were added, the reaction mixture was refluxed for 12–15 h, concentrated, cooled, filtered

and the chalcones, so obtained were purified and crystallized from ethanol. Physical and chemical properties are summarized in Tables I and II.

## Synthesis of N-acetyl Pyrazolo Derivatives (4a–e)

### *General Procedure (2)*

To a solution of chalcones **3a–e** (0.002 mol, 0.922 g, 0.980 g, 0.890 g, 0.976 g, and 0.959 g resp.) in ethanol as a solvent, hydrazine hydrate (50%, 0.1 ml) was added in presence of few drops of acetic acid as a catalyst. The reaction mixture was refluxed for 12–15 h, and then the reaction mixture was concentrated, cooled. On trituration with 20 ml of petroleum ether a resins material was separated, decanted from excess of petroleum ether. This material was triturated with water, then the precipitate has separated, filtered, washed several times by water and crystallized from ethanol. Physical and chemical properties were summarized in Tables I and II.



## Synthesis of N-phenyl Pyrazolo Derivatives (5a–e)

### *General Procedure (3)*

To a solution of chalcones **3a–e** (0.001 mol, 0.461 g, 0.490 g, 0.445 g, 0.488 g, and 0.480 g resp.) in ethanol as a solvent, phenyl hydrazine (0.001 mol, 0.11 ml) was added in presence of few drops of piperdine as a catalyst, the reaction mixture was refluxed for 15–20 h and then it was concentrated to one-third of its volume and allowed to cool, then the reaction mixture was poured to a mixture of ice/HCl, a brown precipitate was separated, filtered, washed for several times of water, dried and crystallized from ethanol. Physical and chemical properties of **5a–e** were summarized in Tables I and II.

## Synthesis of Isoxazolo Derivatives (6a–e)

### *General Procedure (4)*

(0.001 mol, 0.461 g, 0.490 g, 0.445 g, 0.488 g, and 0.480 g resp.) of chalcones **3a–c** and (0.001 mol, 0.069 g) of hydroxylamine hydrochloride were refluxed in presence of sodium hydroxide as a catalyst and ethanol as a solvent for 15–20 h, the reaction mixture was filtered off from unreacted material, the filtrate was concentrated, added to ice-HCl mixture, the precipitate was separated, filtered, washed by water, dried and crystallized from ethanol, physical and chemical properties of **6a–e** were summarized in Tables I and II.

## Synthesis of Pyrimidino Derivatives (7a–e)

### *General Procedure (5)*

An alcoholic solution of chalcones **3a–e** (0.002 mol, 0.922 g, 0.980 g, 0.890 g, 0.976 g, and 0.959 g resp.) was refluxed with urea (0.002 mol, 0.12 g) and 2 ml of HCl concentrated on water bath for 12–15 h. The reaction mixture was filtered on hot, allowed to cool, neutralized with 5N NaOH, then a precipitate was separated, filtered, washed by water for several times, dried and crystallized from ethanol. Physical and chemical properties of **7a–e** were summarized in Tables I and II.

## Synthesis of Thio Pyrimido Derivatives (8a–e)

### *General Procedure (6)*

Chalcones **3a–e** (0.002 mol, 0.922 g, 0.980 g, 0.890 g, 0.976 g, and 0.959 g resp.) were refluxed with thiourea (0.002 mol, 0.152 g) in presence of sodium hydroxide as a catalyst, and ethanol as a solvent for 15–20 h, the reaction mixture was concentrated poured on ice-HCl mixture, the precipitate was separated, filtered, washed by water for several times, dried and crystallized from ethanol, physical and chemical properties of **8a–e** were summarized in Tables I and II.



## Synthesis of Schiff Base Compound Derivatives (9a–c)

### *General Procedure (7)*

To a solution of 2 (0.001 mol, 0.5 g) and nitroso compounds such as $\alpha$-nitroso-$\beta$-naph-thol, $\beta$-nitroso-$\alpha$-naphthol and 3-methyl-1-phenyl-4-oxime-5-pyrazolone (0.001 mol, 0.173 g, 0.173, and 0.203 g) respectively in absolute ethanol, two drops of piperidine were added. The reaction mixture was refluxed for 8–10 h, filtered on hot, concentrated and cooled, the Schiff base so obtained were purified and crystallized from ethanol. Physical and chemical properties of **9a–c** were summarized in Tables I and II.

## Synthesis of Thiazolidino Derivatives (10a–c)

### *General Procedure (8)*

An equimolar mixture of **9a–c** (0.001 mol, 0.512 g, 0.512 g, and 0.452 g resp.) and mercaptoacetic acid (0.001 mol, 0.092 g) in dry benzene (50 ml) were refluxed for 15 h. The reaction mixture was then evaporated to one-third under reduced pressure and allowed to stand at room temperature. In most cases the thiazolidinone was separated with addition of ice-water on the triturated residue, and the product was washed

with water, and crystallized from ethanol physical and chemical properties of **10a–c** were summarized in Tables I and II.

## Synthesis of $\beta$-lactam Derivatives (11a–c)

### *General Procedure (9)*

To a well stirred solution of **9a–c** (0.001 mol, 0.512 g, 0.512 g, and 542 g resp.) and triethylamine (0.002 mol, 0.202 g) in dry dioxane (50 ml) was added chloroacetyl chloride (0.002 mol, 0.226 g) dropwise at room temperature. The mixture was stirred for an additional 9 h, and left at room temperature for 3 dyes. The precipitate of triethylamine hydrochloride was filtered off and washed throughly with dioxane. The filterate was evaporated to one-third of its original volume, cooled and ice-water was added. The residue was washed with water and crystallized from ethanol, physical and chemical properties of **11a–c** were summarized in Tables I and II.



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