An elegant one-pot synthesis of isoxazolo[2,3-α]pyrimidines

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An elegant one-pot synthesis of isoxazolo[2,3-α]pyrimidines has been achieved by reaction of 3-amino-5-methylisoxazole with 4-arylidene-2-phenyl-5-oxazolones. Treatment of 3-amino-5-methylisoxazole with chalcones also resulted in the formation of isoxazolo[2,3-α]pyrimidines in a single step. The structure of the products have been elucidated by spectral and analytical data.

Keywords: One-pot synthesis, 3-amino-5-methylisoxazole, 4-arylidene-2-phenyl-5-oxazolone, chalcone, isoxazolo[2,3-α]pyrimidines.

Heterocycles are widely utilized compounds in both pharmaceutical and agricultural fields. Consequently, the development of methodologies useful for the assembly of molecules containing heterocyclic templates continues to attract the attention of both the academic and industrial communities. Among aromatic heterocycles, the isoxazole unit constitute an easily accessible nucleus that is present in a number of natural and pharmacological compounds, and displays a wide range of organic reactivities and could be used as an effective means of preparing new molecular scaffolds. Isoxazoles have been repeatedly shown as useful synthons in organic synthesis.

The pyrimidine nucleus is also present in a wide range of bioactive natural products. In addition, the pharmacological and biological activities of pyrimidine derivatives are well documented in the literature. Pyrimidines and their ring-fused derivatives have a broad spectrum of biological activity, best known as the heterocyclic core of the nucleic acid bases. These systems are often incorporated into drugs designed for anticancer, antiviral, antihypertensive and analgesic agents. Encouraged by these findings and as a sequel to our interest in the synthesis of a fused heterocyclic system carrying isoxazole as one of the moiety, we found that 3-amino-5-methylisoxazole 1 is a versatile, readily accessible building block for the synthesis of isoxazolopyrimidines of expected biological potency. We report in this paper, a one-pot synthesis of isoxazolo[2,3-α] pyrimidines, by reaction of 3-amino-5-methylisoxazole with 4-arylidene-2-phenyl-5-oxazolones or with chalcones.

The reaction of 3-amino-5-methylisoxazole 1 with 4-arylidene-2-phenyl-5-oxazolones in dioxane under refluxing condition for 6 hr led to the formation of N1-(2-methyl-7-oxo-5-aryl-6,7-dihydro-5H-isoxazolo[2,3-α]pyrimidin-6yl)benzamides 3 on the basis of elemental analyses and spectral data instead of the expected isoxazolyl imidazolones 4 (Scheme I, Table I).

Usually, the amino group of isoxazole makes a nucleophilic attack on carbonyl group of 4-arylidene-2-phenyl-5-oxazolone 2, the ring opening of oxazolone nucleus and recyclization to the imidazolone ring takes place by dehydration, that leads to the formation of isoxazolyl imidazolones 4. Instead of this expected reaction, we have observed that the amino group of isoxazole is attacking the carbon of benzylidene group, which is probably electron deficient due to electron withdrawing influence of carbonyl group. It will be a sort of Michael addition to the highly polarized benzylidene ethylenic linkage. Later, imino group of isoxazole by the influence of secondary amino group attacks the carbonyl group, which results in the ring opening of oxazolone and subsequently cyclizes to isoxazolo pyrimidine 3. All the spectral and analytical data clearly evidenced the formation of product 3 but not 4. Hence, we could achieve the synthesis of isoxazolo[2,3-α]pyrimidines 3 by a one-pot synthesis from readily available materials. Such observation was not reported earlier with 4-arylidene-2-phenyl-5-oxazolone (Scheme II).

The IR spectrum of 3 displayed strong absorption bands at 3250, 1685, 1640 cm⁻¹ assignable to amide NH, pyrimidine ring carbonyl and amide carbonyl respectively. 1H NMR spectrum of 3 displayed two doublets at δ 4.0 and 4.5 assignable for benzylic-H adjacent to C=N and tertiary-H adjacent to carbonyl group respectively. The amide proton appeared as a broad singlet at δ 8.3 disappeared on shaking with D2O. The mass spectrum of 3 showed molecular ion peak at m/z 347 confirming the formation of isoxa-
![Chemical structures](image)

**Scheme I**

**Table I — Physical and analytical data of compounds 3a-i and 7a-i**

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<th>Compd</th>
<th>Ar</th>
<th>Ar'</th>
<th>Yield (%)</th>
<th>m.p. (°C)</th>
<th>Mol. Formula</th>
<th>Found (%)</th>
<th>Calcd (%)</th>
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<td>C_{16}H_{15}N_2OCl</td>
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</table>

NOTES

- Ar = Aryl
- Ar' = Alkyl
- m.p. = Melting point
- Found (Calcd) = Percentual analysis
zolo[2,3-a]pyrimidine 3 but not 4. If 4 is the product, the molecular ion peak should appear at m/z 329 involving loss of H₂O from 1 and 2 to give 4. The base peak present at m/z 105 is assignable to benzoyl cation.

In order to establish the evidence for the above reaction, we performed another reaction between 3-amino-5-methylisoxazole 1 and chalcones 6 (having α,β-unsaturated carbonyl system). The reaction of 3-amino-5-methylisoxazole 1 with chalcone 6 in dioxane under refluxing conditions for 4 hr resulted in the formation of N1-(2-methyl-5,7-diaryl)-5H-isoxazolo[2,3-a]pyrimidines 7 (Scheme III, Table I). Once again, we could able to achieve the synthesis of isoxazolo[2,3-a]pyrimidines by a one-pot synthesis. The structure of the products 7 have been elucidated by microanalytical and spectral data. The amino group of isoxazole, influenced by secondary amino group attacks the carbonyl group resulting in cyclization, which spontaneously loses water molecule to give isoxazolo[2,3-a]pyrimidine 7 (Scheme IV).

The IR spectrum of 7 displayed absorption bands at 1640 and 1625 cm⁻¹ due to C=N and C=C stretching vibrations. ¹H NMR spectrum of 7 exhibited two doublets at δ 4.0 and 5.5 due to benzylic-H and ethylenic-H of pyrimidine ring confirming cyclization. The mass spectrum of 7 confirms the proposed structure by exhibiting the molecular ion peak at m/z 288.

**Conclusion**

In conclusion, an elegant one-pot synthesis of isoxazolo[2,3-a]pyrimidines by interaction of 3-amino-5-methylisoxazole with 4-arylidene-2-phenyl-5-oxazolones or with chalcones using inexpensive and commercially available materials. This synthesis benefits from a simple method of purification, which does not require chromatography. This ease of purification compliments the one-pot synthesis,
making the technology practical, easy to perform and facile. Moreover, fused pyrimidine ring derivatives are potent pharmacological agents, this study may activate the researchers concerned in this field to explore the pharmacological activity of the compounds.

**Experimental Section**

All the melting points were determined on a Cintex melting point apparatus and are uncorrected. Analytical TLC was performed on Merck precoated 60 F<sub>254</sub> silica gel plates. Visualization was done by exposing to Iodine vapour. IR spectra (KBr pellet) were recorded on a Perkin-Elmer BX series FT-IR spectrophotometer. <sup>1</sup>H NMR spectra were recorded on a Varian Gemini 200 MHz spectrometer. Chemical shift values are given in ppm (δ) with tetramethylsilane as internal standard. Mass spectral measurements were carried out by EI method on a Jeol JMC-300 spectrometer at 70 eV. Elemental analyses were performed on a Carlo Erba 106 Perkin-Elmer model 240 analyser.

**General procedure for the synthesis of N<sub>1</sub>-(2-methyl-7-oxo-5-aryl-6,7-dihydro-5H-isoxazolo[2,3-a]pyrimidine-6-yl)benzamides, 3a-i**

A mixture of 4-arylidene-2-phenyl oxazolo-5-one (0.01 mole) and 3-amino-5-methylisoxazole (0.01 mole) were refluxed in dioxane (15 mL) for 6 hr. The reaction mixture was concentrated, cooled and then poured into water (50 mL). The white precipitate obtained was filtered, washed with water and recrystallized from ethanol to afford the isoxazolo[2,3-a] pyrimidine as white crystals.

**Compound 3a**: White crystalline solid; IR (KBr): 3250 (NH), 1685 (C=O), 1640 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ 2.2 (s, 3H, CH<sub>3</sub>), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 6.7 (s, 1H, isoxazole-H), 7.2-7.7 (m, 9H, Ar-H), 8.2 (bs, 1H, NHCO, D<sub>2</sub>O exchangeable); MS (EI): m/z 347 [M]+, 305, 278, 277, 267, 242, 223, 105, 77, 69, 57.

**Compound 3b**: White crystalline solid; IR (KBr): 3260 (NH), 1680 (C=O), 1645 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ 2.3 (s, 3H, CH<sub>3</sub>), 3.9 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.3 (d, 1H, -N-CH-C=O, J = 7Hz), 6.6 (s, 1H, isoxazole-H), 7.2-7.8 (m, 9H, Ar-H), 8.2 (bs, 1H, NHCO, D<sub>2</sub>O exchangeable); MS (EI): m/z 382 [M]+, 381, 363, 336, 285, 283, 276,169, 150, 106, 105, 91, 89, 77, 51, 43, 384[M+2]+.

**Compound 3c**: White crystalline solid; IR (KBr): 3250 (NH), 1670 (C=O), 1650 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ 2.2 (s, 3H, CH<sub>3</sub>), 2.4 (s, 3H, CH<sub>3</sub>), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.5 (d, 1H, -N-CH-C=O, J = 7Hz), 6.7 (s,1H, isoxazole-H), 7.0-7.6 (m, 9H, Ar-H), 8.8 (bs, 1H, NHCO, D<sub>2</sub>O exchangeable); MS (EI): m/z 361 [M]+, 319, 292, 241,222, 178, 105, 91, 77, 51, 43.

**Compound 3d**: White crystalline solid; IR (KBr): 3250 (NH), 1670 (C=O), 1660 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ 2.2 (s, 3H, CH<sub>3</sub>), 2.4 (s, 3H, CH<sub>3</sub>), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.5 (d, 1H, -N-CH-C=O, J = 7Hz), 6.7 (s,1H, isoxazole-H), 7.0-7.6 (m, 9H, Ar-H), 8.8 (bs, 1H, NHCO, D<sub>2</sub>O exchangeable); MS (EI): m/z 361 [M]+, 319, 292, 241,222, 178, 105, 91, 77, 51, 43.
(200 MHz, CDCl₃): δ 2.3 (s, 3H, CH₃), 3.8 (s, 3H, OCH₃), 4.1 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.6 (d, 1H, -N-CH-C=O, J = 7 Hz) 6.6 (s, 1H, isoxazole-H), 7.0-7.7 (m, 9H, Ar-H), 8.8 (bs, 1H, NHCO, D₂O exchangeable); MS (EI): m/z 377 [M⁺], 335, 317, 266, 265, 241, 222, 169, 105, 77, 69, 51.

**Compound 3e:** White crystalline solid; IR (KBr): 3280 (NH), 1680 (C=O), 1660 (C=O) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 3.7 (s, 3H, OCH₃), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.4 (d, 1H, -N-CH-C=O, J = 7 Hz), 6.4 (s, 1H, isoxazole-H), 7.2-7.9 (m, 9H, Ar-H), 8.2 (bs, 1H, NHCO, D₂O exchangeable); MS (EI): m/z 377 [M⁺], 317, 265, 241, 223, 169, 150, 106, 91, 89, 77, 43.

**Compound 3f:** White crystalline solid; IR (KBr): 3270 (NH), 1680 (C=O), 1665 (C=O) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.3 (s, 3H, CH₃), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.6 (d, 1H, -N-CH-C=O, J = 7 Hz), 6.7 (s, 1H, isoxazole-H), 7.0-8.0 (m, 8H, Ar-H), 8.5 (bs, 1H, NHCO, D₂O exchangeable); MS (EI): m/z 415 [M⁺], 373, 322, 312, 205, 186, 142, 130, 105, 77, 51, 43.

**Compound 3g:** White crystalline solid; IR (KBr): 3260 (NH), 1685 (C=O), 1660 (C=O) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.3 (s, 3H, CH₃), 4.1 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.5 (d, 1H, -N-CH-C=O, J = 7 Hz), 6.4 (s, 1H, isoxazole-H), 7.1-7.8 (m, 9H, Ar-H), 8.6 (bs, 1H, NHCO, D₂O exchangeable); MS (EI): m/z 392 [M⁺], 351, 333, 306, 255, 236, 131, 130, 106, 105, 89, 77, 57.

**Compound 3h:** White crystalline solid; IR (KBr): 3255 (NH), 1680 (C=O), 1665 (C=O) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 4.0 (d, 1H, Ph-CH-N=, J = 7 Hz), 4.4 (d, 1H, -N-CH-C=O, J = 7 Hz), 5.0 (s, 2H, OCH₂O), 6.6 (s,1H, isoxazole-H), 7.0-7.7 (m, 8H, Ar-H), 8.5 (bs, 1H, NHCO, D₂O exchangeable); MS (EI): m/z 391 [M⁺], 340, 314, 296, 271, 257, 139, 120, 77, 69, 51, 43.

**General procedure for the synthesis of N₇(2-methyl-5,7-diaryl-5H-isoxazolo [2,3-a]pyrimidines) 7a-i**

A mixture of chalcone (0.01 mole) and 3-amino-5-methyl isoxazole (0.01 mole) were refluxed in dioxane (15 mL) for 4 hr. The reaction-mixture was concentrated, cooled and then poured into water (50 mL). The pale yellow precipitate obtained was filtered, washed with water and recrystallized from ethanol to afford the isoxazolo[2,3-a] pyrimidine as pale yellow crystals.

**Compound 7a:** Pale yellow solid; IR (KBr): 1640 (C=O), 1625 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 4.0 (d, 1H, -CH₂-, J = 4 Hz), 5.5 (d, 1H, =CH-, J = 4 Hz), 6.6 (s, 1H, isoxazole-H), 7.0-7.5 (m, 10H, Ar-H); MS (EI): m/z 288 [M⁺], 246, 181, 143, 107, 102, 55, 43.

**Compound 7b:** Pale yellow solid; IR (KBr): 1630 (C=O), 1620 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.3 (s, 3H, CH₃), 4.0 (d, 1H, -CH₂-, J = 4 Hz), 5.8 (d, 1H, =CH-, J = 4 Hz), 6.5 (s, 1H, isoxazole-H), 7.1-7.6 (m, 9H, Ar-H); MS (EI): m/z 322 [M⁺], 280, 215, 136, 104, 55, 43. 324 [M+2].

**Compound 7c:** Pale yellow solid; IR (KBr): 1635 (C=O), 1610 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.3 (s, 3H, CH₃), 2.5 (s, 3H,CH₃), 4.2 (d, 1H, -CH₂-, J = 4Hz), 5.8 (d, 1H, =CH-, J = 4Hz), 6.8 (s, 1H, isoxazole-H), 7.1-7.7 (m, 9H, Ar-H); MS (EI): m/z 302 [M⁺], 287, 242, 195, 116, 107, 91, 77, 51, 43.

**Compound 7d:** Pale yellow solid; IR (KBr): 1640 (C=O), 1610 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.4 (s, 3H, CH₃), 3.8 (s, 3H, OCH₃), 4.1 (d, 1H, -CH₂-, J = 4Hz), 5.6 (d, 1H, =CH-, J = 4Hz), 6.6 (s, 1H, isoxazole-H), 7.1-7.8 (m, 9H, Ar-H); MS (EI): m/z 318 [M⁺], 275, 211, 196, 171, 103, 55, 43.

**Compound 7e:** Pale yellow solid; IR (KBr): 1650 (C=O), 1600 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 4.1 (d, 1H, -CH₂-, J = 4 Hz), 5.4 (d, 1H, =CH-, J = 4 Hz), 6.4 (s, 1H, isoxazole-H), 7.0-7.7 (m, 9H, Ar-H), 8.5 (bs, 1H, OH, D₂O exchangeable); MS (EI): m/z 304 [M⁺], 262, 235, 197, 155, 118, 104, 93, 55, 43.

**Compound 7f:** Pale yellow solid; IR (KBr): 1640 (C=O), 1620 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 2.4 (s, 3H, CH₃), 4.0 (d, 1H, -CH₂-, J = 4 Hz), 5.3 (d, 1H, =CH-, J = 4 Hz), 6.6 (s, 1H, isoxazole-H), 6.9-7.7 (m, 9H, Ar-H); MS (EI): m/z 302 [M⁺], 287, 256, 220, 118, 116, 102, 91, 77, 55, 43.

**Compound 7g:** Pale yellow solid; IR (KBr): 1640 (C=O), 1600 (C=C) cm⁻¹; ¹H NMR (200 MHz,
CDCl₃): δ 2.4 (s, 3H, CH₃), 3.8 (s, 3H, OCH₃), 4.2 (d, 1H, -CH-), 5.6 (d, 1H, =CH, J = 4 Hz), 6.5 (s, 1H, isoxazole-H), 7.2-7.9 (m, 9H, Ar-H); MS (EI): m/z 318 [M]+, 275, 195, 134, 102, 77, 43.

Compound 7h: Pale yellow solid; IR (KBr): 1645 (C=N), 1610 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 4.1 (d, 1H, -CH-, J = 4 Hz), 5.4 (d, 1H, =CH, J = 4 Hz), 6.6 (s, 1H, isoxazole-H), 7.0-7.7 (m, 9H, Ar-H); MS (EI): m/z 322 [M]+, 280, 252, 215, 137, 103, 101, 55, 43, 324 [M+2]+.

Compound 7i: Pale yellow solid; IR (KBr): 1640 (C=N), 1610 (C=C) cm⁻¹; ¹H NMR (200 MHz, CDCl₃): δ 2.2 (s, 3H, CH₃), 4.0 (d, 1H, -CH-, J = 4 Hz), 5.3 (d, 1H, =CH, J = 4 Hz), 6.5 (s, 1H, isoxazole-H), 7.0-7.7 (m, 9H, Ar-H); MS (EI): m/z 322 [M]+, 280, 252, 220, 135, 111, 103, 101, 55, 43, 324 [M+2]+.

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References