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Synthesis of some new bis(pyrazol-5-ols), Pyridones and benzo-[f]-thiochromene-2-carbonitrile derivatives bearing N-(4-chlorophenyl)-2-(4-formylphenoxy) acetamide moiety

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Abstract

Novel 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol) derivative **2** was synthesized via interaction of *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1**) with diverse available reagent (two mole from 3-methyl-1H-pyrazol-5(4*H*)-one). Moreover, One-pot pseudo three-component reaction of hydrazine hydrate, ethyl acetoacetate and aldehydes in ethanole using pipridine at 70°C afforded the corresponding aminopyrazole derivative **3**. on the other hand, cyanoacetamide scaffolds **4a,b** was reacted with aromatic aldehyde particularly *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1**), to afford arylidenes **5a,b** that undergoes cyclization by heating in ethanol containing drops of piperidine as catalyst, and malononitrile afforded the corresponding pyridinone derivatives **6a,b**. All freshly synthesized scaffolds were elucidated by considering the data of both elemental and spectral analyses.

Keywords: *N*-(4-chlorophenyl)-2-(4-formylphenoxy) acetamide, bis(pyrazol-5-ols), pyridones and benzo-[f]-thiochromene-2-carbonitrile.

Introduction

Pyrazoles are five-membered aza-heterocyclic compounds with two adjacent nitrogen atoms. Pyrazoles are known to ex- hibit a broad spectrum of pharmacological characteristics (P. Chauhan et al., 2017; M.A. Abdallah et al., 2017; S.M. Gomha et al., 2018; A.R. Sayed et al., 2019; S. Gomha et al., 2015; A.O. Abdelhamid et al., 2019; I.M. Abbas et al.,2017; S.M. Gomha et al., 2014). Currently, pyrazole motif-containing drugs such as Cele- coxib (non-steroidal drugs are used for the treatment of arthritis and acute pain, while Fipronil and others are explored as insecticides and for other applications (M. Adib et al., 2005; S. Liu et al., 2018). The pyrazole derivatives have display various biological and pharmacological properties, which include anticancer (S. Ozkınalı et al., 2018), antibacterial (S.B. Otvos

et al., 2019), antimicrobial (Y. Zou et al., 2012), antitumor (M. Srivastava et al., 2013), antipyretic (P.A. Moraes et al., 2019), analgesic (M. Srivastava et al., 2014), anti-inflammatory (F. Shirini et al., 2015), anti-diabetic (M. Driowya et al., 2018), anti-hyperglycemic (F. Nemati et al., 2015), antidepressive (A.R.F. Carreira et al., 2019) and anti-angiogenic (P. Thangarasu et al., 2019) activities. Bis-pyrazolones have exhibited significant antioxidant agents to slow down the process of oxidation by protecting from the reactive oxygen species (ROS) (K. Niknam et al., 2010). In view of the previous importance of Pyrazole scaffolds, the authors have synthesized and screened a novel bis(3-methyl-1*H*-pyrazol-5-ols) derivatives.

Experimental

Materials and Methods

Melting points were determined on Gallenkamp electric device and were uncorrected. The infrared spectra were recorded with KBr on Thermo Scientific Nicolet iS10 FTIR spectrometer. The ^{1}H NMR spectra were recorded in DMSO-d_6 on Bruker's spectrometer at 400 MHz. The mass spectra were determined on Quadrupole GC-MS (DSQII) mass spectrometer at 70 eV. Elemental analyses of carbon, hydrogen, and nitrogen were estimated on Perkin Elmer 2400 analyzer.

General procedure for the synthesis of pyrazole compound 2.

A suspension of *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide scaffold **1** (1.44 g, 5 mmol) and 3-methyl-1H-pyrazol-5(4H)-one (0.98 g, 10 mmol) was heated under reflux for 3 h in EtOH (25 ml) containing five drops of piperidine as catalyst. The resulting products (on cooling) were separated by filtration and then recrystallized from the suitable solvent to obtain 4,4′-(arylmethylene)bis(3-methyl-1*H*-pyrazol-5-ols) derivative **2**.

2-(4-(bis(5-hydroxy-3-methyl-1H-pyrazol-4-yl)methyl)phenoxy)-N-(4-chlorophenyl)acetamide (2).

White powder (yield 75%). mp 245–247 °C. IR ($\bar{\nu}$ /cm^{-1}): 3405, 3397 (NH, OH), 1685 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 2.02 (6H, s, 2CH_3), (2NH and 2OH exchanged with water of DMSO-d6), 4.95 (3H, s, CH_2 and CH), 6.79 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.10 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.34 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 10.20 (1H, s, NH). Anal. Calcd for $C_{23}H_{22}ClN_5O_4$ (467.14): C, 59.04; H, 4.74; N, 14.97. Found: C, 59.04; H, 4.74; N,14.70.

General procedure for the synthesis of aminopyrazole compound 3.

A solution of hydrazine hydrate (2 mmol), ethyl acetoacetate (2 mmol), and pipridine (0.2 mmol) in 20 mL of EtOH 70% was stirred at 70 °C. After 15 min, *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide scaffold **1** (1 mmol) was added and the mixture stirred at 70 °C for 3h. After completion of the reaction, as indicated by TLC, the precipitated solid was filtered and washed with mixture water/ethanol (1:1) and products obtained as pure.

2-(4-((3-amino-5-hydroxy-1H-pyrazol-4-yl)(5-hydroxy-3-methyl-1H-pyrazol-4-yl)methyl)phenoxy)-N-(4-chlorophenyl)acetamide (3).

White powder (yield 65%). mp 275–278 °C. IR ($\bar{\nu}$ /cm^{-1}): 3448, 3386 (NH, OH), 1674 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 2.12 (6H, s, 2CH3), 2.02 (3H, s, CH_3), (2NH, NH_2 and 2OH exchanged with water of DMSO-d6), 4.79 (3H, s, CH_2 and CH), 7.10 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.37 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.82 (d, 2H, *J* = 9.00 Hz, H-Ar), 10.28 (1H, s, NH). Anal. Calcd for $C_{22}H_{21}ClN_6O_4$ (468.13): C, 56.35; H, 4.51; N, 17.92. Found: C, 56.35; H, 4.51; N, 17.92.

Synthesis of N-(4-acetamidophenyl)-3-aryl-2-cyano-acrylamides 5a,b.

A suspension of cyanoacetamide scaffolds 4a,b (5 mmol) and *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1)** (1.44 g, 5 mmol) was heated under reflux for 3 h in EtOH (25 ml) containing five drops of piperidine as catalyst. The resulting products (on cooling) were separated by filtration and then recrystallized from the suitable solvent to obtain 5a,b.

3-(4-(2-(4-chlorophenylamino)-2-oxoethoxy)phenyl)-2-cyano-N-(1,3,4-

thiadiazol-2-yl)acrylamide (5a).

Yellow powder; yield (85%); m.p. 205-207°C (EtOH). IR ($\overline{\nu}$ /cm^{-1}): 3447, 3389 (NH), 2213 (C≡N), 1675, 1636 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 4.86 (s, 2H, CH_2), 7.20 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.37 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 8.04 (d, 2H, *J* = 9.50 Hz, H-Ar), 8.41 (s, 1H, CH), 9.10 (s, 1H, CH), 10.32 (1H, s, NH). Anal. Calcd. for $C_{20}H_{14}ClN_5O_3S$ (439.05): C, 54.61; H, 3.21; N, 15.92%. Found: C, 54.61; H, 3.21; N, 15.92%.

3-(4-(2-(4-chlorophenylamino)-2-oxoethoxy)phenyl)-2-cyano-N-(thiazol-2-yl)acrylamide (5b).

Pale Yellow crystal; yield (88%); m.p. 190-192°C (EtOH). IR ($\overline{\nu}$ /cm^{-1}): 3445, 3377 (NH), 2222 (C≡N), 1685, 1645 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 4.86 (s, 2H, CH_2), 7.20 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.37 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 8.04 (d, 2H, *J* = 9.50 Hz, H-Ar), 8.41 (s, 2H, CH), 9.10 (s, 1H, CH), 10.32 (1H, s, NH). Anal. Calcd. for $C_{21}H_{15}ClN_4O_3S$ (438.06): C, 57.47; H, 3.44; N, 12.77%. Found: C, 57.47; H, 3.44; N, 12.77%.

Synthesis of 6-amino-4-aryl-3,5-dicyano-2-oxopyridine derivatives 6a,b.

Method A: To a mixture of arylidene derivatives **5a,b** (5 mmol) and malononitrile (0.33 g, 5 mmol) in 20 mL of ethanol, five drops of piperidine was added and then heated under reflux for 3 h. On cooling to room temperature, the precipitated solid was filtered and recrystallized from EtOH/DMF mixture (3:1) to obtain the pyridine scaffolds **6a,b**.

2-(4-(6-amino-3,5-dicyano-2-oxo-1-(1,3,4-thiadiazol-2-yl)-1,2-dihydropyridin-4-yl)phenoxy)-N-(4-chlorophenyl)acetamide (6a).

Pale yellow powder; yield (70%); m.p. > 300°C. IR ($\overline{\nu}$ /cm^{-1}): 3451, 3373, 3191 (NH_2 and NH), 2211 (C≡N), 1684 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 4.86 (s, 2H, CH_2), 6.03 (s, 2H, NH_2), 7.20 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.37 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 8.04 (d, 2H, *J* = 9.50 Hz, H-Ar), 8.41 (s, 2H, CH), 10.32 (1H, s, NH).

Anal. Calcd. for $C_{23}H_{14}ClN_7O_3S$ (503.06): C, 54.82; H, 2.80; N, 19.46%. Found: C, 54.82; H, 2.80; N, 19.46%.

2-(4-(6-amino-3,5-dicyano-2-oxo-1-(thiazol-2-yl)-1,2-dihydropyridin-4-yl)phenoxy)-N-(4-chlorophenyl)acetamide (6b).

Yellow crystal; yield (75%); m.p. >300°C. IR ($\overline{\nu}$ /cm^{-1}): 3446, 3305, 3196 (NH_2 and NH), 2214 (C≡N), 1681, 1658 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 4.86 (s, 2H, CH_2), 6.05 (s, 2H, NH_2), 7.20 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.37 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.66 (d, 2H, *J* = 8.50 Hz, H-Ar), 8.04 (d, 2H, *J* = 9.50 Hz, H-Ar), 8.41 (s, 2H, CH), 10.32 (1H, s, NH). Anal. Calcd. for $C_{24}H_{15}ClN_6O_3S$ (502.06): C, 57.32; H, 3.01; N, 16.71%. Found: C, 57.32; H, 3.01; N, 16.71%.

Synthesis of N-(4-acetamidophenyl)-3-aryl-2-cyano-acrylamides 8.

A suspension of naphthalene-2-thiol scaffold (5 mmol), *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1)** (1.44 g, 5 mmol) and malononitrile (0.33 g, 5 mmol) was heated under reflux for 3 h in EtOH (25 ml) containing five drops of piperidine as catalyst. The resulting products (on cooling) were separated by filtration and then recrystallized from the suitable solvent to obtain **8**.

N-(4-chlorophenyl)-2-(4-(2-cyano-3-imino-3H-benzo[f]thiochromen-1-yl)phenoxy)acetamide (8).

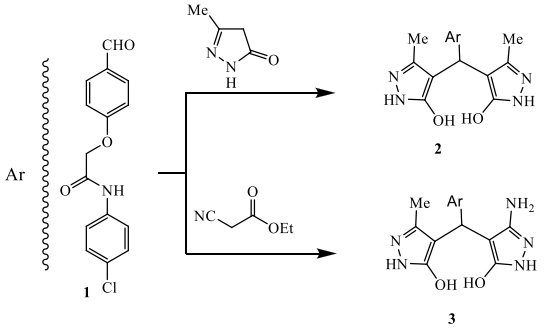
Yellow crystal; yield (55%); m.p. >300°C. IR ($\overline{\nu}$ /cm^{-1}): 3470, 3320, 3214 (NH), 2217 (C≡N), 1702 (C=O). ^{1}H NMR (DMSO-d_6): δ/ppm 4.82 (s, 2H, OCH_2), 7.18 (d, 2H, *J* = 9.00 Hz, H-Ar), 7.38 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.55 (d, 2H, *J* = 8.50 Hz, H-Ar), 7.62 (t, 3H, *J* = 7.00 Hz, H-Ar), 7.69 (d, 2H, *J* = 8.50 Hz, H-Ar), 8.00 (t, 3H, *J* = 8.50 Hz, H-Ar), 8.24 (s, 1H, NH), 10.31 (1H, s, NH). Anal. Calcd. for $C_{28}H_{18}ClN_3O_2S$ (495.08): C, 67.81; H, 3.66; N, 8.47%. Found: C, 67.81; H, 3.66; N, 8.47%.

Result and Discussion

Treatment of *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide with of two mole

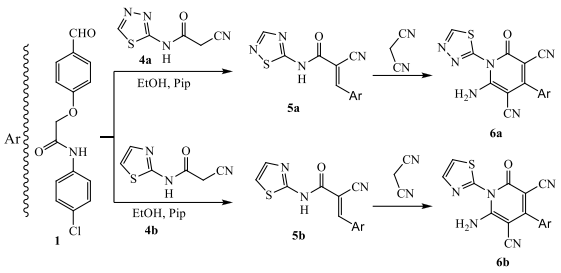
from 3-methyl-1*H*-pyrazol-5(4*H*)-one, in EtOH in the presence of an equimolar amount of pipridine afforded the corresponding 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) derivative **2**.

One-pot pseudo three-component reaction of hydrazine hydrate, ethyl acetoacetate and aldehydes in ethanole using pipridine at 70°C afforded the corresponding aminopyrazole derivative **3**, (Scheme 1). The absorption bands (3448, 3386 and 1674 cm^{-1}) in the IR of scaffold **3** clearly indicated the presence of OH, NH, nitrile and cyclic carbonyl functions. ^{1}H NMR of the scaffold demonstrated The singlet signal for methyl protons at 2.02 ppm, singlet for the protons of methylene function (4.79 ppm), (NH_2 and 2OH exchanged with water of DMSO-d6), multiplet for eight aromatic protons (7.10-7.82 ppm), singlet signal for the proton of one NH function (10.28 ppm).



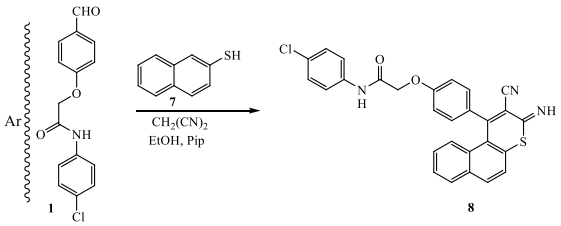
Scheme 7. Synthesis of 4,4′-(arylmethylene)bis(3-methyl-1H-pyrazol-5-ols) derivatives. **2**, and **3**.

In this manner, cyanoacetamide scaffolds was reacted with aromatic aldehyde particularly *N*-(4-chlorophenyl)-2-(4-formylphenoxy) acetamide (**1**), to afford the conformity unsaturated nitrile scaffolds **5a**,**b** (Scheme 2). The IR of **5a**, as an example of the synthesized scaffolds, clearly demonstrated absorptions at 3447, 3389, 2213, 1675 and 1636 cm^{-1} to indicate the presence of NH, nitrile and carbonyl functional groups, respectively. ^{1}H NMR of the same scaffold demonstrated singlet for the protons of methylene function (4.03 ppm), multiplet for nine aromatic protons (7.20-8.04 ppm), singlet for olefinic proton (8.41 ppm) and two singlet signals for the protons of two NH functions (9.10 and 10.32 ppm). By heating in ethanol containing drops of piperidine as catalyst, pyridinone derivatives **6a**,**b** were obtained by the reaction of malononitrile with the synthesized arylidenes **5a**,**b**. The resulted compounds **6a**,**b** were in perfect assent with the proposed structure according to elemental analyses and spectroscopic data.



Scheme 8. Synthesis of pyridones derivatives **5**, and **6**

A suspension of naphthalene-2-thiol scaffold (**7**), *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1**) and malononitrile was heated under reflux for 3 h in EtOH in the presence of an equimolar amount of pipridine afforded the corresponding 3-imino-3*H*-benzo[*f*]thiochromen-1-yl)phenoxy)acetamide derivative **8**, (Scheme 3). The chemical structure of **8** was established based on its ^{1}H NMR which revealed singlet signal at δ 4.82 ppm assigned for OCH_2 group in addition to multiplet signal at δ 7.18-8.00 ppm due to aromatic protons, and two singlet signals for the protons of two NH functions (8.84 and 10.31 ppm).



Scheme 3. Synthesis of 3-imino-1-aryl-3H-benzo[f]thiochromene-2-carbonitrile 8.

Conclusion

Novel 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol) derivative **2** was synthesized via interaction of *N*-(4-chlorophenyl)-2-(4-formylphenoxy)acetamide (**1**) with diverse available reagent (two mole from 3-methyl-1H-pyrazol-5(4*H*)-one). Moreover, treatment of hydrazine hydrate, ethyl acetoacetate and aldehydes in ethanole using pipridine afforded the corresponding aminopyrazole derivative **3**. on the other hand, cyanoacetamide scaffold was reacted with

aromatic aldehyde, to afford arylidenes **5a,b** that undergoes cyclization by heating in ethanol containing drops of piperidine as catalyst, and malononitrile afforded the corresponding pyridinone derivatives **6a,b**. Structures of the new compounds using IR, and ^{1}H NMR spectroscopic techniques were characterized.

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الملخص العربي

عنوان البحث: تشييد بعض مشتقات البيرازول، البيريدين و الكرومين الجديدة

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[٢] قسم الكيمياء-كلية العلوم- جامعة المنصورة
[٣] قسم الكيمياء الهندسية - المعهد العالي للهندسة والتكنولوجيا – دمياط الجديدة

تهدف الدراسة الحالية الى تحضير مشتقات جديدة من البيرازول، البيريدين و الكرومين. عند معالجة المركب الالدهيد (١) مع ٣-ميثيل بيرازولون ، في وجود ايثانول وأضافة قطرات من البيبريدين أعطى مشتق ثنائي ميثيل بيرازول ٢)). وعند غليان المركب الالدهيد (٢) مع هيدرات الهيدرازين ، وإيثيل أسيتو أسيتات في وجود ايثانول وأضافة قطرات من البيبريدين أعطى مشتق الأمينوبيرزاول ٣)). بالإضافة الى ذلك، عند تسخين مشتق السيانواسيتاميد مع الالدهيد الاروماتي ؛ أدي ذلك الي تكوين مشتقات البنزاليدين ٥ .((a,bوعند معالجة مشتقات البنزاليدين ٥ ((a,bمع المالونونيتريل في وجود ايثانول وأضافة قطرات من البيبريدين أعطى مشتقات الأمينوبيريدين ٦ .((a,bعند غليان المركب الالدهيد مع نفثالين ثايول والمالونونيتريل في وجود ايثانول وأضافة قطرات من البيبريدين أعطى مشتق ثايوكرومين (٨). تم اثبات التركيب الكيميائي للمركبات المشيدة الجديدة بواسطة التحليل العناصري ودراسة التحاليل الطيفية الحديثة المختلفة مثل طيف الاشعة تحت الحمراء، وطيف الرنين النووي المغناطيسي لنواة ذرة الهيدروجين وكذلك طيف الكتلة.