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SYNTHESIS OF IMINES VIA CARBONYL-AMINE CONDENSATION IN THE TRICYCLIC PYRROLO[2,3-d]PYRIMIDINE SYSTEM

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Abstract. Pyrrolo[2,3-*d*]pyrimidinones were made by reacting tricyclic pyrrolo[2,3-*d*]pyrimidinones with 4-halogenated phenylamines. The structures of all the new compounds were figured out using ^{1}H-NMR, ^{13}C-NMR analyses.

Keywords: pyrrolo[2,3-*d*]pyrimidine-imines; 2-methoxypyridine; *N*-halosuccinimide; 4-*halo*-substituted phenylanilines; pyrrolo[2,3-*d*]pyrimidine-imines.

Trisiklik pirrolo[2,3-*d*]pirimidin tizimida karbonil-amin kondensatsiyasi orqali iminlar sintezi

Annotatsiya. Trisiklik pirrolo[2,3-*d*]pirimidinonlarni 4-galogenalmashgan fenilaminlar bilan reaksiyasi natijasida pirrolo[2,3-*d*]pirimidin-iminlar sintez qilingan. Barcha yangi birikmalarning tuzilishi ^{1}H-YaMR, ^{13}C-YaMR tahlillari bilan aniqlandi.

Kalit so‘zlar: pirrolo[2,3-*d*]pirimidinlar; N-galosuksinimid; 4-galogenalmashgan fenilanilinlar; pirrolo[2,3-*d*]pirimidin-iminlar.

Синтез иминов путем карбонил-аминной конденсации в трициклической пирроло[2,3-*d*]пиримидиновой системе

Аннотация. Пирроло[2,3-*d*]пиримидиноны были синтезированы путем взаимодействия трициклических пирроло[2,3-*d*]пиримидинонов с 4-галогенированными фениламинами. Структуры всех новых соединений были определены с помощью анализов ^{1}H-ЯМР, ^{13}C-ЯМР.

Ключевые слова: пирроло[2,3-*d*]пиримидины; N-галосукцинимид; 4-галогензамещенные фениланилины; пирроло[2,3-*d*]пиримидин-имины.

Introduction. Pyrimidine-derived chemical frameworks incorporating various five-membered heterocycles have gained significant attention in cancer drug discovery due to their favorable pharmacological profiles and ability to modulate key biological targets. Such heterocyclic systems are frequently represented in clinical candidates, including MCT1 blockers AZD3965 and AR-C155858 [1-4], the WEE1 kinase modulator AZD1775 [5, 6], ALK inhibitors AZD3463 and AZD5363 [7, 8], and the DNA-PK suppressor AZD7648 [9, 10], as well as other AZD analogues under oncological evaluation [11, 12].

Among privileged scaffolds, the pyrrolo[2,3-d]pyrimidine (7-deazapurine) ring system stands out due to its occurrence in a wide range of biologically essential structures, such as natural nucleotides in DNA and RNA, phytochemicals, and synthetic analogs with pharmacological relevance[13]. Structurally, it consists of a fused six-membered pyrimidine ring and a five-membered pyrrole moiety, forming a compact, electron-rich heterocyclic core.

Recently, our group introduced a streamlined route to tricyclic pyrrolo[2,3-d]pyrimidinones, providing access to these motifs in satisfactory yields[14]. We have long pursued the design and

reactivity of condensed pyrimidine-based systems, including their interaction with biological targets and their anticancer potential in human tumor cell lines[15, 16].

The tricyclic scaffold bears multiple chemically addressable positions, such as the nitrogen atoms within both rings, a carbonyl group embedded in the pyrimidine unit, and a methylene linker. These features enable diverse electrophilic substitution reactions, which may contribute to depurination processes or serve synthetic purposes in constructing complex bioactive compounds[17].

In this study, we focused on exploring imine formation via carbonyl–amine condensation as a route to structurally modify the tricyclic pyrrolo[2,3-d]pyrimidine skeleton.

Experimental section. All reagents and solvents were purchased from Sigma and used without further purification. Thin-layer chromatography (TLC) was performed on glass plates coated with silica gel (Qingdao Haiyang Chemical Co., G60F-254) and visualized by UV light (254 nm). The products were purified by column chromatography over silica gel (Qingdao Haiyang Chemical Co., 200–300 mesh). Melting points were determined on a Buchi B-540 apparatus and were uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded with a Varian 400 MHz NMR spectrometer in $CDCl_3$ and DMSO-d_6 using TMS as an internal standard.

General synthetic procedure for the preparation of tricyclic pyrrolo[2,3-*d*]pyrimidines 6a-e. Compound (**4**) (5.0 mmol) and 2-methoxypyridine (5.5 mmol) were added to anhydrous DCM, placed at 0 °C, protected with nitrogen, and stirred for ten min. Then Tf_2O (10 mmol) was slowly added dropwise, and stirring was continued at 0 °C for 1 h. Then the aromatic amines (**5a-e**) (10 mmol) were slowly added, stirred at 0° C for 1 h, and then transferred to room temperature to continue the reaction, and the reaction was detected by TLC until the reaction was completed. Saturated sodium bicarbonate was then added, and the organic phase was separated, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to give the crude product, which was purified by silica gel chromatography to give compounds **6a-e**.

Compound (6a). Yield 99%, m.p. 281-283. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 3H), 7.26 (d, J = 1.7 Hz, 1H), 7.16 – 7.11 (m, 2H), 7.06 – 7.00 (m, 1H), 6.95 – 6.90 (m, 2H), 5.10 (s, 1H), 4.16 (t, J = 6.2 Hz, 2H), 3.61 (s, 3H), 2.95 (t, J = 6.7 Hz, 2H), 2.03 (p, J = 6.1 Hz, 2H), 1.93 (p, J = 6.4 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.96, 151.70, 148.58, 145.48, 133.31, 132.66, 130.64, 129.63, 128.99, 128.65, 122.16, 122.10, 104.14, 101.84, 43.88, 32.51, 29.83, 22.85, 19.41.

Compound (6b). Yield 48%, m.p. 240-242. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.30 (m, 2H), 7.18 – 7.13 (m, 2H), 7.02 – 6.95 (m, 2H), 6.90 – 6.83 (m, 2H), 5.16 (s, 1H), 4.14 (t, J = 6.2 Hz, 2H), 2.95 (t, J = 6.7 Hz, 2H), 2.03 (p, J = 6.5, 6.1 Hz, 2H), 1.93 (p, J = 6.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.03, 157.65, 153.94, 149.15, 145.53, 133.51, 132.91, 130.48, 129.69, 128.73, 123.14, 123.07, 115.68, 115.46, 103.85, 101.74, 43.98, 32.50, 29.86, 22.83, 19.36.

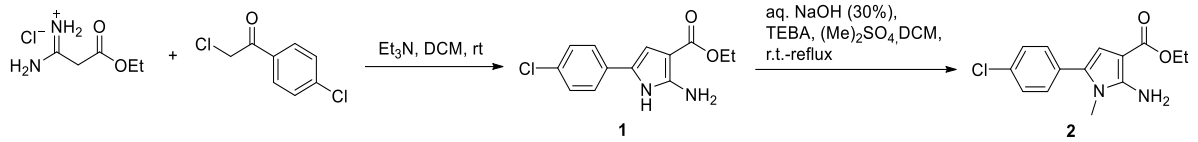
Compound (6c). Yield 68%, m.p. 273-275. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H), 7.27 – 7.21 (m, 2H), 7.20 – 7.14 (m, 2H), 6.89 – 6.83 (m, 2H), 5.26 (s, 1H), 4.13 (t, J = 6.2 Hz, 2H), 3.61 (s, 3H), 2.95 (t, J = 6.7 Hz, 2H), 2.03 (p, J = 6.2 Hz, 2H), 1.93 (p, J = 6.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.94, 150.35, 148.65, 145.53, 133.57, 133.00, 130.46, 129.77, 128.97, 128.75, 126.96, 123.41, 103.86, 101.67, 43.93, 32.49, 29.85, 22.82, 19.37.

Compound (6d). Yield 99%, m.p. 262-264. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.31 (m, 4H), 7.20 – 7.15 (m, 2H), 6.84 – 6.79 (m, 2H), 5.27 (s, 1H), 4.13 (t, J = 6.2 Hz, 2H), 3.61 (s, 3H), 2.95 (t, J = 6.7 Hz, 2H), 2.02 (p, J = 6.2 Hz, 2H), 1.93 (p, J = 6.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.94, 150.86, 148.52, 145.53, 133.58, 133.03, 131.89, 130.45, 129.79, 128.75, 123.91, 114.50, 103.85, 101.65, 43.92, 32.48, 29.82, 22.82, 19.37.

Compound (6e). Yield 61%, m.p. 202-204. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, J = 8.3 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.18 – 7.13 (m, 2H), 7.05 (d, J = 8.3 Hz, 2H), 5.20 (s, 1H), 4.19 (t, J = 6.2 Hz, 2H), 2.99 (t, J = 6.7 Hz, 2H), 2.05 (p, J = 6.1 Hz, 2H), 1.95 (p, J = 6.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.90, 148.51, 145.94, 133.86, 133.83, 130.14, 129.69, 128.81, 126.21, 124.47, 124.15, 123.36, 122.51, 122.46, 103.54, 101.64, 44.41, 32.47, 29.90, 22.76, 19.27.

Results and discussion. The target 5-substituted 2-amino-1*H*-methyl-3-carboxylates **2** were obtained from 1-amino-3-ethoxy-3-oxopropane-1-iminium chloride in two steps (Scheme 1). The reaction of 1-amino-3-ethoxy-3-oxopropane-1-iminium chloride with the corresponding halogenated ketones in the presence of triethylamine (Et_3N) in DCM at room temperature yielded 2-amino-1*H*-pyrrole in up to 70% yield. Methylation of the intermediate products of 2-amino-1*H*-pyrrole was carried

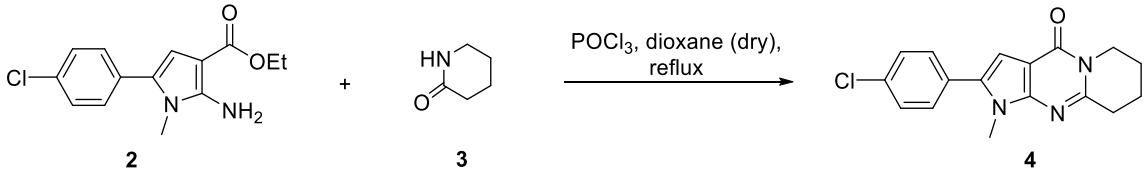
out with dimethyl sulfate in DCM using TEBA (benzyltriethylammonium chloride) as a phase transfer catalyst.



Scheme 1. Synthesis of target 2-aminopyrrole **2.**

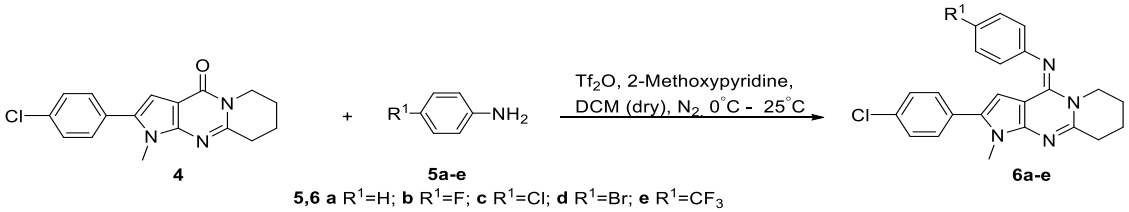
Next, the direct one-reactor formation of pyrrolo[2,3-*d*]pyrimidinone **4** derivatives was investigated. Cyclization of substituted 2-aminopyrroles can be achieved under acidic conditions using $POCl_3$ and proceeds similarly to their furo analogues (Scheme 2). After careful adjustment of the reaction conditions, pyrrolo[2,3-d]pyrimidinones were obtained in only two cases: in the first, without a solvent at 140°C for 2 hours (product yield up to 100%), in the second case, using the nonpolar solvent dioxane as the reaction solvent, where the desired pyrrolo[2,3-*d*]pyrimidinones were formed with a yield of up to 92% after 1 hour. However, when dioxane was used as the reaction solvent, a simpler procedure for obtaining and purifying the final products was observed (Scheme 2).

ipso-Imination of ketone, carbonyl, and sulfoxide functional groups with amines (1°-3°) is rapid access to the synthesis of imine-tethered compounds [18-20]. Tricyclic pyrrolo[2,3-*d*]pyrimidinone 4 contain a C=O group and together with the neighboring-nitrogen heteroatom of the pyrimidine ring, it becomes an amide-type functional fragment. Therefore, it was interesting to study the *ipso*-imination reaction of these tricyclic pyrimidinone ring systems *via* carbonyl-amine condensation. The three-step total synthesis of compound **4** was carried out according to the following scheme.



Scheme 2. Synthetic route of tricyclic pyrrolo[2,3-*d*]pyrimidine **4**.

During a choice of amide activation agent for carbonyl-amine condensation, here triflic anhydride (Tf_2O) [21] was selected. Tf_2O is successfully used as an electrophilic activation agent for the cyclic amides [22], chemo-selective synthesis process of imines [23], and even used in a nucleophilic substitution reaction towards various generated phosphates [24]. Except for an aniline, 4-*halo*-substituted phenyl anilines (**5a-e**) were selected for the condensation, while a base additive was used 2-methoxypyridine. The carbonyl-amine condensation was performed using dichloromethane (DCM) as a reaction solvent at low temperature: starting from 0°C to r.t. 25°C. The results revealed that transformation easily occurred under the above condition, and desired pyrrolo[2,3-*d*]pyrimidine-imines **6a-e** were formed up to 99% (Scheme 3). Therefore, Tf_2O/2-methoxypyridine-mediated carbonyl-amine condensation was found as a convenient and mild pathway on pyrrolo[2,3-*d*]pyrimidine-imines **6a-e** formation, and no other condition for *ipso*-imination was investigated in these systems.



Scheme 3. Synthetic pathway of pyrrolo[2,3-*d*]pyrimidine-imines **6a-e**.

All synthesized pyrrolo[2,3-*d*]pyrimidine-imines **6a-e** were elucidated by ^{1}H-NMR and ^{13}C-NMR as described in the experimental section (see Supplementary Information), as well as X-ray analysis for (*E*)-*N*,2-*Bis*(4-chlorophenyl)-1-methyl-6,7,8,9-tetrahydropyrido[1,2-*a*]pyrrolo[2,3-*d*]pyrimidin-4(1*H*)-imine (**6c**) (Fig. 1).

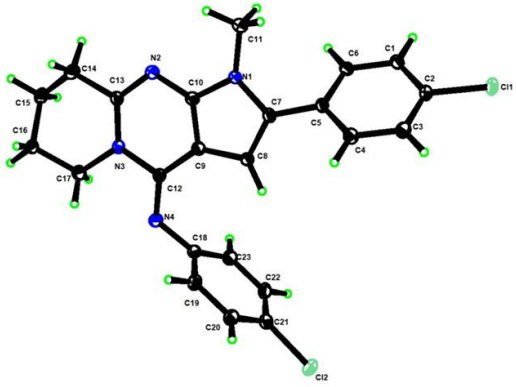


Fig. 1. Crystal structure of compound **6c** (CCDC: 2169282).

Conclusion. The process begins with a carbonyl compound and an amine, which form an imine. Tf_2O acts as a strong acid catalyst that increases the electrophilicity of the carbonyl. 2-Methoxypyridine serves as a nucleophilic base. The reaction involves condensation, in which Tf_2O accelerates imine formation by removing water. Optimized conditions ensure high yields and minimal side reactions. After the reaction, the imines are characterized using nuclear magnetic resonance (NMR), infrared (IR), and mass spectrometry. This method provides a versatile approach to synthesizing imines from the tricyclic pyrrolo[2,3-d]pyrimidine framework, allowing for further functionalization in medicinal chemistry and materials science. Together, Tf_2O and 2-methoxypyridine facilitate the synthesis of complex nitrogen heterocycles, which are valuable in research and development.

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