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# Synthesis and photophysical properties of 2-azolyl-6-piperidinylpurines

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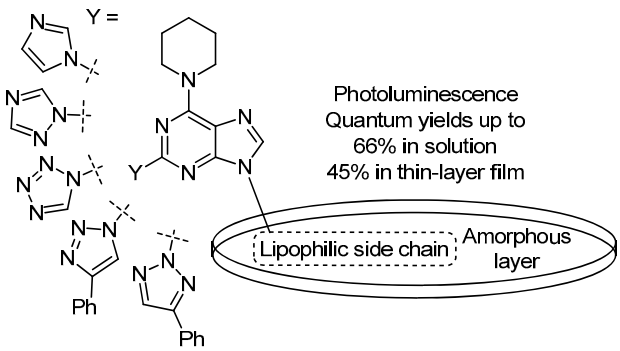



A synthesis of novel fluorescent 2-azolyl-6-piperidinylpurine derivatives was designed. Azolyl substituent at purine C-2 atom was introduced *via* nucleophilic aromatic substitution or in the case of tetrazolyl and 1,2,3-triazolyl substituents *via* a ring formation on a pre-installed amine or azide moiety, respectively. The obtained purine intermediates were functionalized at N-9 position using Mitsunobu reaction conditions to achieve amorphous compounds, which form thin-layer films of good quality. The synthesized push-pull systems exhibited fluorescence with emission in range of 360–400 nm and quantum yields up to 66% in $CH_2Cl_2$ solution and up to 45% in the thin-layer film.

**Keywords**: azoles, purines, fluorescence, nucleophilic aromatic substitution, ring formation.

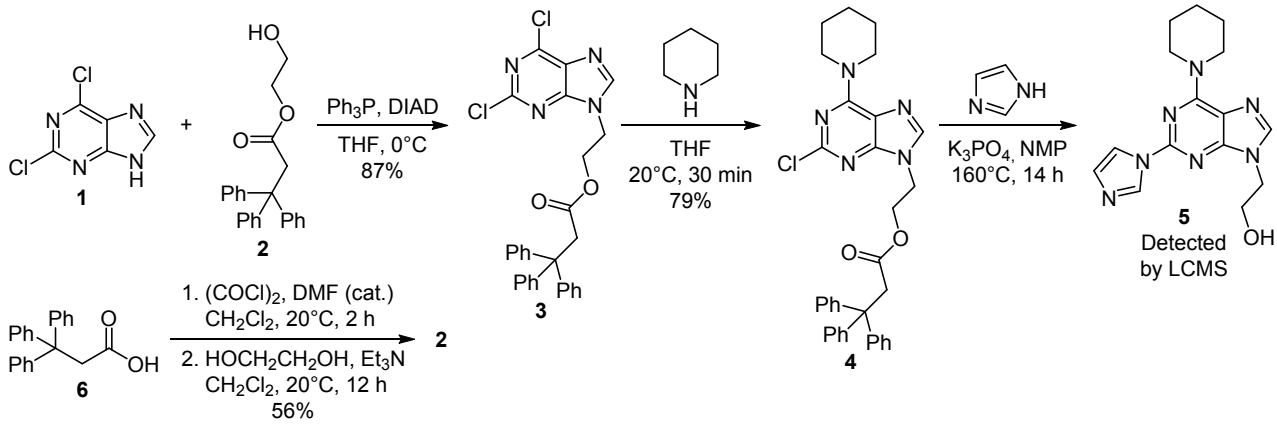
For decades, purine derivatives have been extensively studied due to their wide spectrum of biological activities.[1–4] Purine cycle is known as the most common nitrogen heterocycle in nature.[5,6] Fluorescence properties exhibited by certain purine derivatives are often used in cell imaging.[7–11] On the contrary, materials science applications of fluorescent purine derivatives are poorly studied. Lately Castellano's group has developed organic light emitting diodes (OLEDs) with purine emitters[12,13] and Yang's group obtained the first thermally activated delayed fluorescence (TADF) emitter using purine ring as the electron-accepting group.[14]

Applications of organic compounds in materials science are often limited by complicated and expensive processes, for example, vacuum deposition, which is used for creating thin substance layers. The alternative is a solution processing, which requires compounds that form stable amorphous phase and possess good solubility. These properties can be achieved by introducing a trityl moiety in the molecule.[15,16]

Expanding the previous work of our group,[11] we developed a synthesis of 2-azolyl-6-piperidinylpurine derivatives as push-pull systems with different substituents at the N-9 atom. While there is a wide range of information on introduction of various azoles at purine C-6 position,[17–21] transformations at purine C-2 position are less common, as C-6 position is more reactive and preliminary functionalization of C-6 position deactivates substitution at C-2 position.[6] The known examples represent pyrazole ring construction on hydrazine,[23] imidazole ring introduction *via* an oxidated intermediate of 8-oxoadenine and 8-oxohypoxanthine,[23] and substituted benzimidazole introduction using Buchwald–Hartwig cross coupling[24,25] or a $S_NAr$ reaction.[26]

The initial synthetic approach to the target compounds included $N^9$-functionalization using Mitsunobu reaction between 2,6-dichloropurine (**1**) and 2-hydroxyethyl 3,3,3-tri-

**Scheme 1**. Attempted synthesis of 2-[2-(1*H*-imidazol-1-yl)-6-(piperidin-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoates



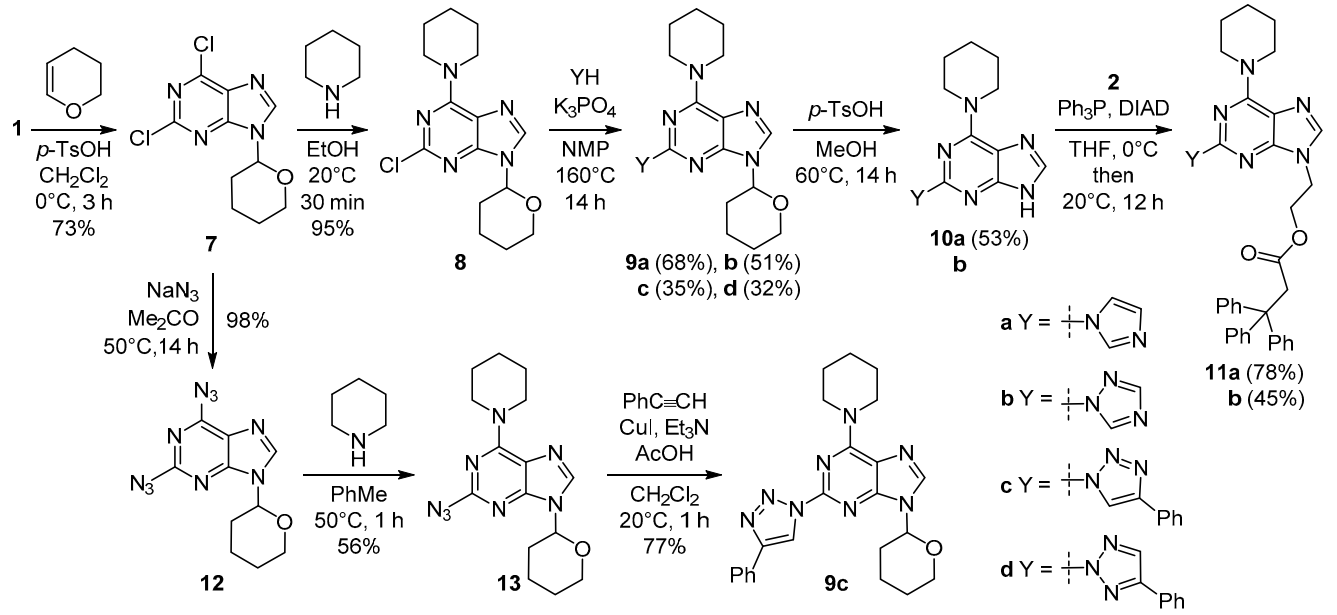

phenylpropanoate (**2**)[27] (Scheme 1). The selected side chain is foreseen to ensure the amorphous properties of otherwise crystalline heterocycles. Compound **2** containing trityl group was obtained by esterification of commercially available or readily prepared[28] acid **6**. Next, a $S_NAr$ reaction of adduct **3** at C-6 position with piperidine produced intermediate **4** under mild conditions (20°C, 30 min). Then the next $S_NAr$ reaction was tried for the introduction of imidazole at C-2 position.[24] At 160°C in the presence of $K_3PO_4$ as a base, we observed the $S_NAr$ process at the C-2 atom. However, these conditions were too harsh, and the ester moiety was cleaved at the N-9 side chain producing compound **5** regardless of its relatively high stability which arises due to sterical hindrance.

To avoid the ester cleavage of substituent at N-9 position we decided to perform firstly the $S_NAr$ reaction with piperidine at C-6 and then with imidazole at C-2 position of $N^9$-unsubstituted purine. Again, the second $S_NAr$ process did not provide the desired product, most likely due to the presence of a negative charge on the purine ring arising from its deprotonation.

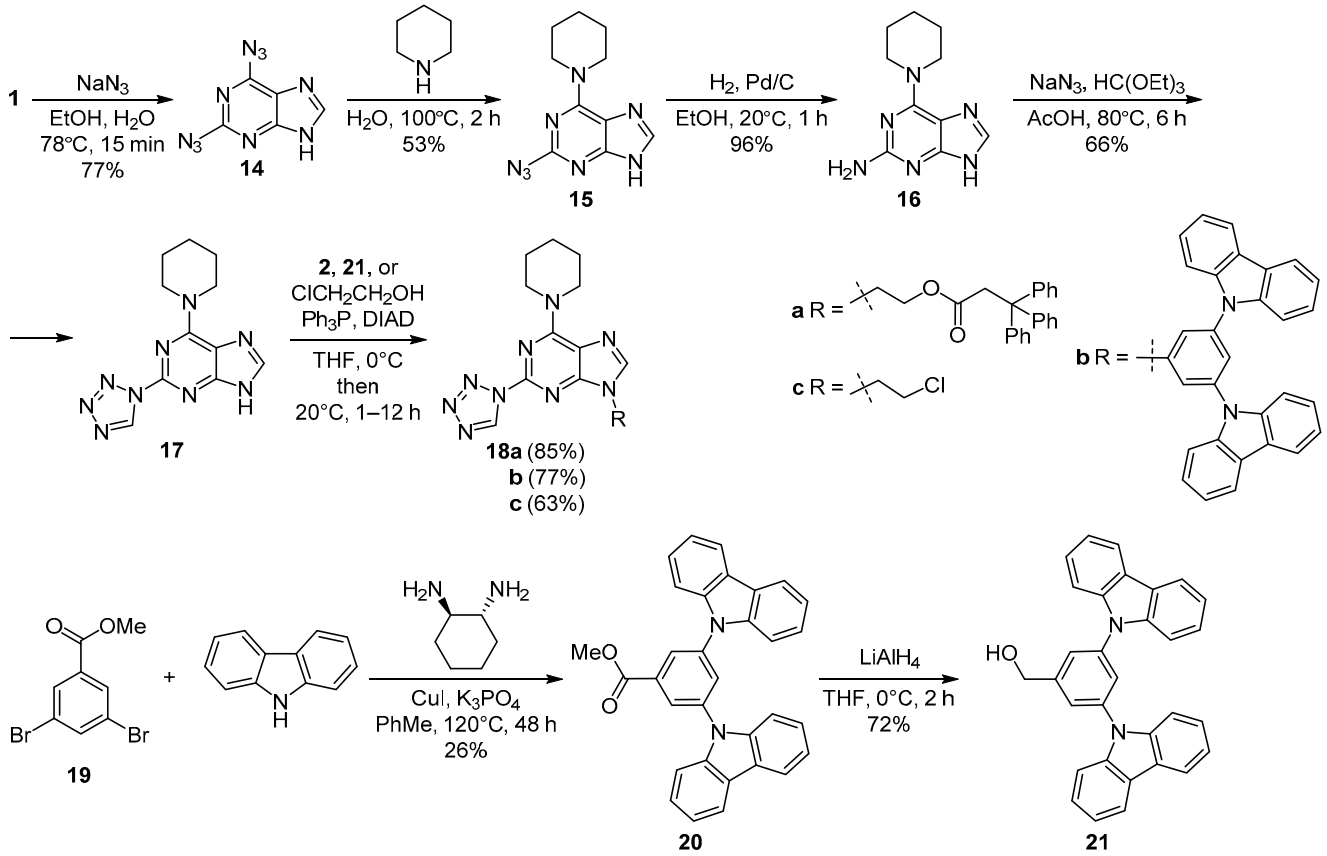
Then a thermally stable and base-resistant tetrahydropyranyl (THP) protecting group was introduced at N-9 position, and product **7**[29] was obtained in 73% yield starting from 2,6-dichloropurine (**1**) (Scheme 2). Compound **7** reacted smoothly with piperidine at C-6 position (20°C, 30 min) providing the key intermediate **8**[30,31] in 95% yield. The latter underwent $S_NAr$ reactions at C-2 position with azole nucleophiles such as imidazole, 1,2,4-triazole, and 4-phenyl-1,2,3-triazole.

Although relatively harsh reaction conditions have been applied ($K_3PO_4$, *N*-methylpyrrolidone (NMP), 160°C) the $S_NAr$ substitution process in compound **8** proceeded with good yields (51–68%). $S_NAr$ reaction with 1,2,4-triazole selectively gave 1*H*-1,2,4-triazol-1-ylpurine **9b**, and no 1,2,4-triazol-4-yl derivative was observed. Reaction with 4-phenyl-1,2,3-triazole yielded a 1:1 mixture of 2-(1*H*-1,2,3-triazol-1-yl)purine and 2-(2*H*-1,2,3-triazol-2-yl)purine derivatives **9c**,**d** with isolated yields 35 and 32%, respectively (Scheme 2). The identity of compound **9c** was confirmed by an alternative synthetic pathway, in which the 1*H*-1,2,3-triazol-1-yl substituent was installed by Cu(I)-catalyzed

**Scheme 2**. Synthesis of 2-[2-azolyl-6-(piperidin-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoates **11a**,**b**



**Scheme 3**. Synthesis of 9-alkylated 6-(piperidin-1-yl)-2-(1*H*-tetrazol-1-yl)-9*H*-purine derivatives **18a–c**




azide-alkyne cycloaddition reaction of azide derivative **13**. The latter was obtained by a $S_NAr$ reaction of 2,6-diazidopurine **12**. Toluene as relatively nonpolar solvent and slightly elevated temperature ensured the C-6 reactivity due to the prevalence of 6-azido form of the substrate, which can enter azide-tetrazole tautomeric equilibrium.[11,32–35]

THP deprotection of compounds **9a**,**b** yielded azolylpurines **10a**,**b**, which were further functionalized at N-9 position *via* Mitsunobu reaction with diisopropyl azodicarboxylate (DIAD) to give compounds **11a**,**b** (Scheme 2). Deprotected compound **10b** exhibited extremely poor solubility in DMF, DMSO, THF, and various alcohols, thus preventing compound characterization. It was, therefore, telescoped into the Mitsunobu reaction giving compound **11b**.

Attempts to introduce a tetrazole ring at C-2 position of purine **6** by the $S_NAr$ process of 2-chloropurine derivative **8** did not provide the desired product. Since tetrazole is rather acidic (p$K_a$ 4.9), its deprotonated form is a weak nucleophile. Aiming to obtain the desired product we switched to a ring construction on 2-aminopurine derivative **16** (Scheme 3).[36] The latter was synthesized starting from 2,6-dichloropurine (**1**). Firstly, 2,6-diazidopurine (**14**)[37] was obtained. Subsequent $S_NAr$ reaction with piperidine yielded adduct **15**.[38] The remaining 2-azido group was catalytically reduced to amino group. The obtained 2-aminopurine **16**[39] was used in ring formation with $NaN_3$ and $HC(OEt)_3$ and gave the expected 2-tetrazolylpurine derivative **17** in 66% yield. Similarly to 9*H*-purine derivatives **10a**,**b**, also intermediate **17** underwent selective alkylation at N-9 atom under Mitsunobu conditions yielding compounds **18a–c** in 63–85% yields (Scheme 3). Alcohols such as 2-hydroxyethyl 3,3,3-triphenylpropanoate (**2**), [3,5-di(9*H*-carbazol-9-yl)phenyl]methanol (**21**), and 2-choroethanol were used. The latter provides compound **18c**, the chloroethyl substituent of which can serve as a "sticky end" for further modifications of the side chain. Reagent **21** for the introduction of the dicarbazolylphenyl moiety was synthesized from compound **19** using copper-catalyzed N-arylation to give compound **20**,[40] followed by ester reduction with $LiAlH_4$ to compound **21**.

Photophysical properties of compounds **11a**,**b** and **18a**,**b** were investigated in $5\cdot10^{-5}$ M $CH_2Cl_2$ solution and in the thin films (Figs. 1, 2 and Table 1). For compounds **11a**,**b** and **18a**, the lowest energy absorption bands correspond to intramolecular charge transfer transition (ICT) of purine chromophores indicated by shoulders in the 300–310 nm range. For compound **18b**, the ICT band overlaps with carbazole absorption as indicated by characteristic maxima $\lambda_{abs}$ at 323 and 338 nm. The emission maxima $\lambda_{em}$ for compounds **11a**,**b** and **18a** were in the 356–401 nm range, which corresponds to near UV and purple light. The increase of number of nitrogen atoms in the azole ring resulted in a higher electron deficiency, which caused a bathochromic shift in emission maxima.[41] The 3,5-dicarbazolylphenyl moiety in compound **18b** was intended to improve hole transfer capabilities for potential use in OLEDs.[42] In the case of purine derivative **18b** we observed

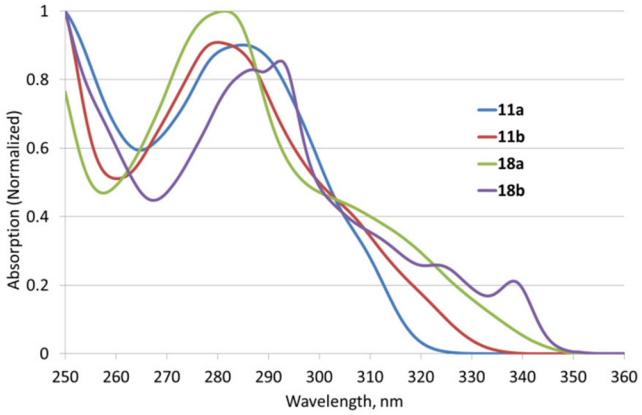



**Figure 1**. Absorption spectra of 2-azolyl-6-piperidinylpurines **11a**,**b** and **18a**,**b** in $5 \cdot 10^{-5}$ M $CH_2Cl_2$ solution.

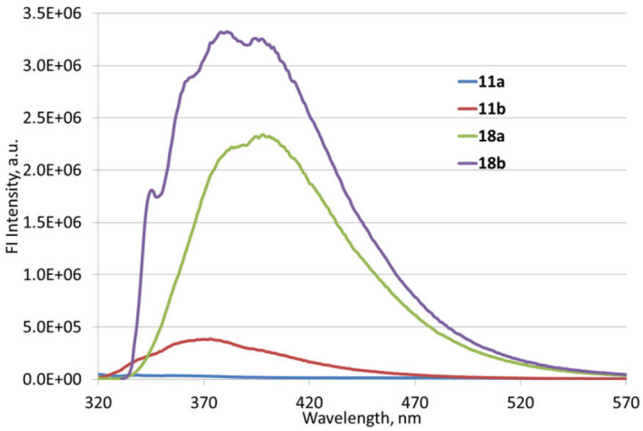



**Figure 2**. Emission spectra of 2-azolyl-6-piperidinylpurines **11a**,**b** and **18a**,**b** in $5 \cdot 10^{-5}$ M $CH_2Cl_2$ solution.

an emission spectrum, which is a combination of emission from 6-piperdinyl-2-tetrazolylpurine and 3,5-dicarbazolylphenyl moieties. We explain this by similar lowest energy singlet levels for carbazole and purine chromophores. Onset of absorption for both **18a**,**b** which contain the same purine moiety is at 350 nm. Characteristic maxima of carbazole emission are also distinguishable in PL band (362, 378, 397 nm: entry 7, Table 1).

Unlike tetrazole-substituted derivatives **18a**,**b**, compounds **11a**,**b** showed better quantum yield in the thin-layer film than in the $CH_2Cl_2$ solution. This is unusual, since typically quantum yields in the thin-layer films are lowered due to an intermolecular quenching, which is reduced in solution. Compounds **11b** and **18a** showed the best quantum yields in the thin-layer films, 0.45 and 0.42 respectively.

To conclude, we have developed several approaches for the introduction of various azolyl moieties at C-2 position of purine ring system containing an electron-donating piperidinyl substituent at its C-6 position. $S_NAr$ reactions were used for the introduction of imidazolyl, 1*H*-1,2,4-triazol-1-yl, and 2*H*-1,2,3-triazol-2-yl substituents at the purine C-2 position. On the other hand, selective construction of 1*H*-1,2,3-triazol-1-yl group on 2-azidopurine derivative was carried out by Cu(I)-catalyzed azide-alkyne cycloaddition reaction, and a tetrazolyl substituent was introduced by three-component assembling on 2-aminopurine derivative. The developed synthetic pathways allowed a late stage $N^9$-functionalization of the obtained heterocyclic assemblies to achieve amorphous states of the desired push-pull purines for potential materials science applications in the future. An introduction of 2-chloroethyl side chain at N-9 position will serve as a functionalization site for possible chemical modifications in future. The obtained conjugates exhibited emission in the near UV region in both solution (up to 66% quantum yield) and in the thin-layer films (up to 45% quantum yield).

## Experimental

UV-Vis absorption spectra were recorded with a PerkinElmer Lambda 35 spectrometer. Films for optical measurements were prepared using spin-coating technique with a Laurell WS-400B-6NPP/LITE spin-coater on glass slides, using 30 mg/ml THF solutions. After the coating, all films were dried in oven at 100°C for 2 h. Emission spectra and quantum yields for solutions and thin films were recorded using a QuantaMaster 40 steady state spectrofluorometer (Photon Technology International, Inc.) equipped with a 6 inch integrating sphere by LabSphere, using the software package provided by the manufacturer. $^1$H and $^{13}$C NMR spectra were recorded on a Bruker 300 (300 and 75 MHz, respectively) spectrometer. Internal standard – residual non-deuterated solvent peak for $^1$H nuclei (7.26 ppm in $CDCl_3$, 2.50 ppm in DMSO-$d_6$) and deuterated solvent peak for $^{13}$C nuclei (77.2 ppm in $CDCl_3$, 39.5 ppm in DMSO-$d_6$). Nontrivial peak assignments were confirmed with $^1$H–$^{13}$C HSQC and/or $^1$H–$^{13}$C HMBC spectra. High-resolu-

**Table 1**. Photophysical properties of 2-azolyl-6-piperidinylpurines **11a**,**b** and **18a**,**b**

| Entry | Compound | State | $\lambda_{abs}$, nm | $\lambda_{em}$, nm | Emission quantum yield |
|---|---|---|---|---|---|
| 1 | **11a** | $CH_2Cl_2$ solution | 284, 305 (shoulder) | 356 | 0.01 |
| 2 | **11a** | Thin-layer film | 286, 300 (shoulder) | 366 | 0.12 |
| 3 | **11b** | $CH_2Cl_2$ solution | 280, 300 (shoulder) | 373 | 0.07 |
| 4 | **11b** | Thin-layer film | 283, 310 (shoulder) | 373 | 0.45 |
| 5 | **18a** | $CH_2Cl_2$ solution | 281, 310 (shoulder) | 398 | 0.66 |
| 6 | **18a** | Thin-layer film | 285, 310 (shoulder) | 401 | 0.42 |
| 7 | **18b** | $CH_2Cl_2$ solution | 292, 323, 338 | 362, 378, 397 | 0.51 |
| 8 | **18b** | Thin-layer film | 295, 326, 340 | 401, 422 | 0.20 |

tion mass spectra (ESI) were recorded on a Waters Q-TOF Micromass spectrometer. Melting points were determined on a Fisher Digital Melting Point Analyzer Model 355. HPLC analysis was performed using an Agilent Technologies 1200 Series system equipped with XBridge C18 column, 4.6 × 150 mm, particle size 3.5 μm, with flow rate of 1 ml/min, using 0.1% aqueous TFA (A) and MeCN (B) for mobile phase; wavelength of detection was 260 nm; eluent: gradient 30–95% B in A for 5 min, 95% B for 5 min, 95–30% B for 2 min. All reactions were followed by TLC on E. Merck Kieselgel 60 $F_{254}$ plates with detection by UV light. Silica gel (60 Å, 40–63 μm, ROCC) was used for flash chromatography.

Commercially available reagents were used as received. Starting materials **1**, **6**, and **19** are commercially available. Compounds **2**,[27] **3**,[27] **7**,[29] **8**,[30,31] **14**,[37] **15**,[38] and **16**[39] are known from literature.

**2-[2-Chloro-6-(piperidin-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoate (4)**. Piperidine (0.57 ml, ρ 0.86 g/cm$^3$, 5.79 mmol, 3.0 equiv) was added to a solution of compound **3** (1.0 g, 1.93 mmol, 1.0 equiv) in THF (30 ml), and the reaction mixture was stirred at 20°C for 30 min. Then the reaction mixture was evaporated, dissolved in $CH_2Cl_2$ (30 ml), and washed with aqueous $KH_2PO_4$ solution (3 × 20 ml), saturated $NaHCO_3$ solution (20 ml), and brine (10 ml). The organic phase was dried over anhydrous $Na_2SO_4$, filtered, and evaporated. Yield 865 mg (79%), colorless solid. $R_f$ 0.32 ($CH_2Cl_2$–MeCN, 20:1). HPLC: $t_R$ 8.22 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 1.63–1.81 (6H, m, $3CH_2$); 3.72 (2H, s, $CH_2$); 4.03–4.14 (4H, m, $2CH_2$); 4.10–4.34 (4H, m, $2CH_2$); 7.12–7.31 (15H, m, H Ph); 7.40 (1H, s, H-8 ). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.8; 26.3; 42.5; 46.4; 46.7; 56.1; 62.3; 118.8; 126.5; 128.0; 129.3; 138.5; 146.5; 152.5; 154.2; 154.3; 170.5. Found, *m*/*z*: 566.2328 $[M+H]^+$. $C_{33}H_{33}ClN_5O_2$. Calculated, *m*/*z*: 566.2317.

**2-(1*H*-Imidazol-1-yl)-6-(piperidin-1-yl)-9-(tetrahydro-2*H*-pyran-2-yl)-9*H*-purine (9a)**. Anhydrous $K_3PO_4$ (1.38 g, 6.52 mmol, 3.0 equiv) was added to a solution of compound **8** (700 mg, 2.17 mmol, 1.0 equiv), imidazole (222 mg, 3.26 mmol, 1.5 equiv) in dry NMP (3.5 ml) under Ar, and the reaction mixture was stirred at 160°C for 14 h. Then the reaction mixture was poured into $H_2O$ (30 ml) and extracted with PhMe (3 × 20 ml). The organic phase was washed with brine (3 × 20 ml), $H_2O$ (2 × 20 ml), and again with brine (10 ml), dried over anhydrous $Na_2SO_4$, filtered, and evaporated. Silica gel column chromatography (eluent $CH_2Cl_2$ in MeOH, gradient 0–5%) of the residue provided product **9a**. Yield 523 mg (68%), colorless solid. $R_f$ 0.41 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 4.75 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.57–2.15 (12H, m, $6CH_2$); 3.76 (1H, td, $^2J$ = $^3J$ = 11.2, $^3J$ = 2.8) and 4.15 (1H, d, $^2J$ = 11.2, $CH_2O$); 4.10–4.36 (4H, m, $2CH_2$); 5.66 (1H, dd, $^3J$ = 9.8, $^3J$ = 2.4, NCHO); 7.09 (1H, s, H imidazole); 7.85 (1H, s, H imidazole); 7.87 (1H, s, H-8); 8.54 (1H, s, H imidazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 23.0; 24.8; 25.0; 26.2; 31.8; 46.6; 68.9; 81.8; 117.0; 118.2; 129.7; 136.0; 136.3; 149.7; 151.1; 153.8. Found, *m*/*z*: 354.2033 $[M+H]^+$. $C_{18}H_{24}N_7O$. Calculated, *m*/*z*: 354.2037.

**6-(Piperidin-1-yl)-9-(tetrahydro-2*H*-pyran-2-yl)-2-(1*H*-1,2,4-triazol-1-yl)-9*H*-purine (9b)** was prepared analogously to compound **9a** from compound **8** (700 mg, 2.17 mmol, 1.0 equiv), 1,2,4-triazole (225 mg, 3.26 mmol, 1.5 equiv), dry NMP (3.5 ml), anhydrous $K_3PO_4$ (1.38 g, 6.52 mmol, 3.0 equiv). Yield 388 mg (51%), colorless solid. $R_f$ 0.39 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 5.43 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.57–2.16 (12H, m, $6CH_2$); 3.78 (1H, td, $^2J$ = $^3J$ = 11.3, $^3J$ = 2.7) and 4.12 (1H, d, $^2J$ = 11.3, $CH_2O$); 4.11–4.38 (4H, m, $2CH_2$); 5.82 (1H, dd, $^3J$ = 10.2, $^3J$ = 1.9, NCHO); 7.94 (1H, s, H-8); 8.09 (1H, s, H triazole); 9.12 (1H, s, H triazole). $^{13}$C NMR spectrum ($CDCl_3$, δ, ppm: 22.8; 24.7; 25.0; 26.2; 32.4; 46.5; 68.8; 81.3; 118.8; 136.6; 143.8; 149.4; 150.9; 153.1; 153.8. Found, *m*/*z*: 355.1998 $[M+H]^+$. $C_{17}H_{23}N_8O$. Calculated, *m*/*z*: 355.1989.

**2-(4-Phenyl-1*H*-1,2,3-triazol-1-yl)-6-(piperidin-1-yl)-9-(tetrahydro-2*H*-pyran-2-yl)-9*H*-purine (9c) and 2-(4-phenyl-2*H*-1,2,3-triazol-2-yl)-6-(piperidin-1-yl)-9-(tetrahydro-2*H*-pyran-2-yl)-9*H*-purine (9d)**, 1:1 mixture, were prepared analogously to compound **9a** from compound **8** (296 mg, 0.92 mmol, 1.0 equiv), 4-phenyl-1*H*-1,2,3-triazole (200 mg, 1.38 mmol, 1.5 equiv), dry NMP (2 ml), anhydrous $K_3PO_4$ (585 mg, 2.76 mmol, 3.0 equiv). Products **9c**,**d** were separated by silica gel column chromatography (eluent PhMe in MeCN, gradient 0–10%).

**Compound 9c**. Yield 137 mg (35%), colorless solid. $R_f$ 0.28 ($CH_2Cl_2$–MeCN, 10:1). HPLC: $t_R$ 6.87 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.57–2.23 (12H, m, $6CH_2$); 3.82 (1H, td, $^2J$ = $^3J$ = 11.2, $^3J$ = 2.7) and 4.17 (1H, d, $^2J$ = 11.2, $CH_2O$); 4.15–4.55 (4H, m, $2CH_2$); 5.85 (1H, dd, $^3J$ = 10.2, $^3J$ = 2.2, NCHO); 7.35 (1H, t, $^3J$ = 7.5, H Ar); 7.45 (2H, t, $^3J$ = 7.6, H Ar); 7.93–8.01 (3H, m, H Ar, H-8); 8.69 (1H, s, H triazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 23.0; 24.9; 25.2; 26.3; 32.4; 46.8; 68.9; 81.9; 118.9; 119.3; 126.3; 128.3; 128.9; 130.9; 136.9; 147.5; 149.6; 151.2; 154.1. Found, *m*/*z*: 431.2332 $[M+H]^+$. $C_{23}H_{27}N_8O$. Calculated, *m*/*z*: 431.2302.

**Compound 9d**. Yield 125 mg (32%), colorless solid. $R_f$ 0.20 (PhMe–MeCN, 10:1). HPLC: $t_R$ 6.78 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.55–2.11 (11H, m) and 2.15 (1H, d, $^2J$ = 11.2, $6CH_2$); 3.83 (1H, td, $^2J$ = $^3J$ = 11.4, $^3J$ = 2.8) and 4.15 (1H, d, $^2J$ = 11.2, $CH_2O$); 4.23–4.48 (4H, m, $2CH_2$); 5.93 (1H, dd, $^3J$ = 10.1, $^3J$ = 2.3, NCHO); 7.39 (1H, tt, $^3J$ = 7.5, $^4J$ = 1.5, H Ar); 7.47 (2H, t, $^3J$ = 7.6, H Ar); 7.93–8.01 (3H, m, H Ar, H-8 ); 8.15 (1H, s, H triazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 23.0; 24.9; 25.2; 26.4; 32.6; 46.8; 68.9; 81.5; 119.1; 126.8; 129.0; 129.1; 130.3; 133.9; 136.8; 149.8; 151.1; 151.4; 154.3. Found, *m*/*z*: 431.2275 $[M+H]^+$. $C_{23}H_{27}N_8O$. Calculated, *m*/*z*: 431.2302.

**Alternative method for the synthesis of compound 9c**. Phenylacetylene (251 μl, ρ 0.93 g/cm$^3$, 2.29 mmol, 1.5 equiv) was added to a solution of compound **13** (500 mg, 1.52 mmol, 1.0 equiv), CuI (58 mg, 0.30 mmol, 0.20 equiv), AcOH (95 μl, ρ 1.05 g/cm$^3$, 1.67 mmol, 1.1 equiv), and $Et_3N$ (230 μl, ρ 0.73 g/cm$^3$, 1.67 mmol, 1.1 equiv) in $CH_2Cl_2$ (15 ml). The reaction mixture was stirred isolated from daylight for 1 h at 20°C. Then the reaction mixture was poured into $H_2O$

(30 ml) and extracted with $CH_2Cl_2$ (3 × 20 ml). The combined organic phase was washed with aqueous $NaHSO_3$ (15 ml) and saturated NaCl (15 ml), dried over anhydrous $Na_2SO_4$, filtered, and evaporated. Silica gel column chromatography (eluent $CH_2Cl_2$ in MeCN, gradient 0–10%) of the residue provided product **9c** as a colorless solid. Yield 506 mg (77%).

**2-(1*H*-Imidazol-1-yl)-6-(piperidin-1-yl)-9*H*-purine (10a)**. *p*-TsOH·$H_2O$ (429 mg, 2.49 mmol, 2.2 equiv) was added to a solution of compound **9a** (400 mg, 1.13 mmol, 1.0 equiv) in MeOH (12 ml), and the reaction mixture was stirred for 14 h at 60°C. Then the reaction mixture was evaporated, the residue was suspended in 0.5 M solution of $K_2CO_3$ in $H_2O$–MeOH, 10:1 (20 ml) and filtered off. Recrystallization of the precipitate from EtOH provided product **10a**. Yield 161 mg (53%), colorless solid, mp 272–274°C (decomp., EtOH). $R_f$ 0.23 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 3.71 min. $^1$H NMR spectrum (DMSO-$d_6$, 60°C), δ, ppm: 1.55–1.80 (6H, m, $3CH_2$); 4.12–4.39 (4H, m, $2CH_2$); 7.05 (1H, s, H imidazole); 7.85 (1H, s, H imidazole); 8.04 (1H, s, H-8); 8.46 (1H, s, H imidazole); 13.00 (1H, br. s, NH). $^{13}$C NMR spectrum (DMSO-$d_6$, 60°C), δ, ppm: 24.2; 25.7; 45.8; 116.8; 117.2; 129.4; 135.5; 138.1; 148.8; 152.0; 153.0. Found, *m*/*z*: 270.1450 $[M+H]^+$. $C_{13}H_{16}N_7$. Calculated, *m*/*z*: 270.1462.

**2-[2-(1*H*-Imidazol-1-yl)-6-(piperidin-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoate (11a)**. DIAD (0.09 ml, ρ 1.03 g/cm$^3$, 0.45 mmol, 1.2 equiv) was added over 15 min to a solution of compound **9a** (100 mg, 0.37 mmol, 1.0 equiv), $Ph_3P$ (117 mg, 0.45 mmol, 1.2 equiv), and 2-hydroxyethyl 3,3,3-triphenylpropanoate (**2**) (154 mg, 0.45 mmol, 1.2 equiv) in dry THF (5 ml) at 0°C, followed by stirring for 12 h at 20°C. Then the reaction mixture was evaporated, the residue was suspended in MeOH (15 ml) and $H_2O$ (1 ml) and cooled at –10°C for 30 min. The formed precipitate was filtered off and washed with cold MeOH (2 × 5 ml) to give compound **11a**. Yield 173 mg (78%), colorless solid. $R_f$ 0.39 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 6.64 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 1.64–1.88 (6H, m, $3CH_2$); 3.71 (2H, s, $CH_2$); 4.04–4.18 (4H, m, $2CH_2$); 4.14–4.42 (4H, m, $2CH_2$); 7.06–7.32 (16H, m, H Ph, H imidazole); 7.43 (1H, s, H-8); 7.84 (1H, s, H imidazole); 8.53 (1H, s, H imidazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.8; 26.3; 42.4; 46.2; 46.5; 55.9; 62.1; 117.1; 118.2; 126.5; 128.0; 129.2; 129.9; 136.4; 138.4; 146.3; 149.8; 151.6; 153.9; 170.6. Found, *m*/*z*: 598.2930 $[M+H]^+$. $C_{36}H_{36}N_7O_2$. Calculated, *m*/*z*: 598.2925.

**2-[6-(Piperidin-1-yl)-2-(1*H*-1,2,4-triazol-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoate (11b)**. *p*-TsOH·$H_2O$ (283 mg, 1.49 mmol, 2.2 equiv) was added to a solution of compound **9b** (240 mg, 0.68 mmol, 1.0 equiv) in MeOH (10 ml), and the reaction mixture was stirred for 14 h at 60°C. Then the reaction mixture was evaporated and suspended in 0.5 M $K_2CO_3$ solution in $H_2O$–MeOH, 10:1 (20 ml), and filtered. The crude 9*H*-deprotected compound **10b** (100 mg, 0.37 mmol, 1.0 equiv) was used for the synthesis of compound **11b**, using 2-hydroxyethyl 3,3,3-triphenylpropanoate (**2**) (154 mg, 0.45 mmol, 1.2 equiv), $Ph_3P$ (117 mg, 0.45 mmol, 1.2 equiv), DIAD (0.09 ml, ρ 1.03 g/cm$^3$,, 0.45 mmol, 1.2 equiv), and THF (5 ml) according to the procedure for the synthesis of compound **11a**. Yield 185 mg (45%), colorless solid. $R_f$ 0.46 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 7.33 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 1.66–1.90 (6H, m, $3CH_2$); 3.75 (2H, s, $CH_2$); 4.10–4.24 (4H, m, $2CH_2$); 4.08–4.54 (4H, m, $2CH_2$); 7.08–7.35 (15H, m, H Ph); 7.50 (1H, s, H-8); 8.10 (1H, s, H triazole); 9.13 (1H, s, H triazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.7; 26.2; 42.4; 46.1; 46.4; 55.9; 62.4; 118.8; 126.4; 128.0; 129.1; 139.0; 143.8; 146.3; 149.4; 151.5; 153.1; 153.8; 170.5. Found, *m*/*z*: 599.2879 $[M+H]^+$. $C_{35}H_{35}N_8O_2$. Calculated, *m*/*z*: 599.2878.

**2,6-Diazido-9-(tetrahydro-2*H*-pyran-2-yl)-9*H*-purine (12)**. $NaN_3$ (2.38 g, 36.63 mmol, 4.0 equiv) was added to a solution of compound **7**[27] (2.50 g, 9.16 mmol, 1.0 equiv) in $Me_2CO$ (60 ml), and the reaction mixture was stirred in dark for 14 h at 50°C. Then the reaction mixture was evaporated, the residue was suspended in $H_2O$ (50 ml), filtered off, and washed with $H_2O$ (3 × 20 ml) to give compound **12**. Yield 2.58 g (98%), colorless solid. $R_f$ 0.26 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 5.74 min. $^1$H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.58–2.16 (6H, m, $3CH_2$); 3.75 (1H, td, $^2J$ = $^3J$ = 11.3, $^3J$ = 2.6) and 4.16 (1H, d, $^2J$ = 11.3, $CH_2O$); 5.68 (1H, dd, $^3J$ = 10.1, $^3J$ = 2.2, NCHO); 8.10 (1H, s, H-8 ). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 22.7; 24.9; 31.9; 68.9; 82.1; 121.4; 141.5; 153.2; 153.7; 156.0. Found, *m*/*z*: 287.1121 $[M+H]^+$. $C_{10}H_{11}N_{10}O$. Calculated, *m*/*z*: 287.1112.

**2-Azido-6-(piperidin-1-yl)-9-(tetrahydro-2*H*-pyran-2-yl)-9*H*-purine (13)**. Piperidine (2.59 ml, ρ 0.86 g/cm$^3$, 26.2 mmol, 3.0 equiv) was added to a solution of compound **12** (2.5 g, 8.74 mmol, 1.0 equiv) in PhMe (30 ml). The reaction mixture was stirred in dark for 1 h at 50°C. Then the reaction mixture was evaporated, and the residue was purified by silica gel column chromatography (eluent $CH_2Cl_2$ in MeCN, gradient 0–10%). Yield 1.61 g (56%), pale-yellow solid. $R_f$ 0.45 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 7.20 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.55–2.11 (12H, m, $6CH_2$); 3.73 (1H, td, $^2J$ = $^3J$ = 11.4, $^3J$ = 2.4) and 4.12 (1H, d, $^2J$ = 11.4, $CH_2O$); 4.09–4.35 (4H, m, $2CH_2$); 5.64 (1H, dd, $^3J$ = 10.0, $^3J$ = 2.0, NCHO); 7.82 (1H, s, H-8). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 23.0; 24.8; 25.0; 26.2; 32.0; 46.5; 68.9; 81.4; 117.3; 135.5; 151.7; 153.8; 156.2. Found, *m*/*z*: 329.1835 $[M+H]^+$. $C_{15}H_{21}N_8O$. Calculated, *m*/*z*: 329.1833.

**6-(Piperidin-1-yl)-2-(1*H*-tetrazol-1-yl)-9*H*-purine (17)**. $HC(OEt)_3$ (240 mg, ρ 0.89 g/cm$^3$, 11.75 mmol, 1.6 equiv) was added to a solution of compound **16** (1.60 g, 7.34 mmol, 1.0 equiv) and $NaN_3$ (716 mg, 11.02 mmol, 1.5 equiv) in AcOH (40 ml), and the reaction mixture was stirred for 6 h at 80°C. Then the reaction mixture was evaporated and suspended in saturated aqueous $NaHCO_3$ solution (40 ml). The precipitate was filtered off, washed with saturated aqueous $NaHCO_3$ solution (2 × 10 ml), $H_2O$ (2 × 10 ml), and dried to provide product **17**. Yield 1.31 g (66%), colorless solid. $R_f$ 0.39 ($CH_2Cl_2$–MeOH, 20:1). HPLC: $t_R$ 4.48 min. $^1$H NMR spectrum (DMSO-$d_6$, 60°C), δ, ppm: 1.54–1.80 (6H, m, $3CH_2$); 4.19–4.39 (4H, m, $2CH_2$); 8.17 (1H, s, H-8 ); 9.97 (1H, s, H tetrazole). $^{13}$C NMR spectrum (DMSO-$d_6$, 60°C), δ, ppm: 24.1: 25.7; 45.8 (br); 118.3;

139.3; 142.9; 146.8; 151.7; 152.9. Found, *m*/*z*: 272.1003 $[M+H]^+$. $C_{11}H_{14}N_9$. Calculated, *m*/*z*: 272.1367.

**2-[6-(Piperidin-1-yl)-2-(1*H*-tetrazol-1-yl)-9*H*-purin-9-yl]ethyl 3,3,3-triphenylpropanoate (18a)** was prepared analogously to compound **11a** from compound **17** (300 mg, 1.12 mmol, 1.0 equiv), 2-hydroxyethyl 3,3,3-triphenylpropanoate (**2**) (465 mg, 1.34 mmol, 1.2 equiv), $Ph_3P$ (352 mg, 1.34 mmol, 1.2 equiv), and DIAD (0.27 ml, ρ 1.03 g/cm$^3$, 1.34 mmol, 1.2 equiv) in THF (10 ml) for 12 h at 20°C. Yield 570 mg (85%), colorless solid. $R_f$ 0.38 ($CH_2Cl_2$–MeCN, 10:1). HPLC: $t_R$ 7.48 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 1.67–1.89 (6H, m, 3$CH_2$); 3.72 (2H, s, $CH_2$); 4.10–4.22 (4H, m, 2$CH_2$); 4.02–4.62 (4H, m, 2$CH_2$); 7.12–7.30 (15H, m, H Ph); 7.52 (1H, s, H-8 ); 9.38 (1H, s, H tetrazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.6; 26.2; 42.5; 46.0; 46.4; 55.8; 62.0; 119.3; 126.4; 127.9; 129.0; 139.4; 141.7; 146.2; 147.4; 151.1; 153.7; 170.4. Found, *m*/*z*: 600.2823 $[M+H]^+$. $C_{34}H_{34}N_9O_2$. Calculated, *m*/*z*: 600.2830.

**9,9'-(5-{[6-(Piperidin-1-yl)-2-(1*H*-tetrazol-1-yl)-9*H*-purin-9-yl]methyl}-1,3-phenylene)bis(9*H*-carbazole) (18b)** was prepared analogously to compound **11a** from compound **17** (118 mg, 0.44 mmol, 1.0 equiv), [3,5-di(9*H*-carbazol-9-yl)-phenyl]methanol (**21**) (212 mg, 0.48 mmol, 1.1 equiv), $Ph_3P$ (138 mg, 0.53 mmol, 1.2 equiv), and DIAD (0.11 ml, ρ 1.03 g/cm$^3$, 0.53 mmol, 1.2 equiv) in THF (5 ml) for 1 h at 20°C. Yield 233 mg (77%), colorless solid. $R_f$ 0.75 ($CH_2Cl_2$–MeCN, 10:1). HPLC: $t_R$ 9.57 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.62–1.93 (6H, m, 3$CH_2$); 3.91–4.67 (4H, m, 2$CH_2$); 5.60 (2H, s, $CH_2$); 7.28 (4H, t, $^3J$ = 7.5, H Ar); 7.37 (4H, t, $^3J$ = 7.5, H Ar); 7.45 (4H, d, $^3J$ = 7.5, H Ar); 7.64 (2H, s, H Ar); 7.79 (1H, s, H Ar); 7.93 (1H, s, H-8 ); 8.10 (4H, d, $^3J$ = 7.5, H Ar); 9.36 (1H, s, H tetrazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.7; 26.3; 46.8; 47.2; 109.7; 119.8; 120.7; 120.9; 124.0; 124.6; 125.1; 126.4; 138.9; 139.6; 140.5; 140.6; 141.8; 148.1; 151.7; 154.2. Found, *m*/*z*: 692.2542 $[M+H]^+$. $C_{42}H_{34}N_{11}$. Calculated, *m*/*z*: 692.2993.

**9-(2-Chloroethyl)-6-(piperidin-1-yl)-2-(1*H*-tetrazol-1-yl)-9*H*-purine (18c)** was prepared analogously to compound **11a** from compound **17** (400 mg, 1.48 mmol, 1.0 equiv), 2-chloroethanol (0.11 ml, ρ 1.20 g/cm$^3$, 1.63 mmol, 1.1 equiv), $Ph_3P$ (466 mg, 1.78 mmol, 1.2 equiv), and DIAD (0.35 ml, ρ 1.03 g/cm$^3$, 1.78 mmol, 1.2 equiv) in THF (8 ml) for 3 h at 20°C. The product was purified by silica gel column chromatography (eluent $CH_2Cl_2$ in MeCN, gradient 0–6%). Yield 312 mg (63%), colorless solid. $R_f$ 0.15 ($CH_2Cl_2$–MeCN, 20:1). HPLC: $t_R$ 5.14 min. $^1$H NMR spectrum ($CDCl_3$, 50°C), δ, ppm (*J*, Hz): 1.67–1.88 (6H, s, 3$CH_2$); 3.96 (2H, t, $^3J$ = 5.7, $CH_2$); 4.10–4.50 (4H, m, 2$CH_2$); 4.57 (2H, t, $^3J$ = 5.7, $CH_2$); 7.88 (1H, s, H-8); 9.41 (1H, s, H tetrazole). $^{13}$C NMR spectrum ($CDCl_3$, 50°C), δ, ppm: 24.7; 26.3; 42.5; 46.0; 47.0; 119.8; 139.9; 141.9; 147.8; 151.4; 154.1. Found, *m*/*z*: 334.1268 $[M+H]^+$. $C_{13}H_{17}ClN_9$. Calculated, *m*/*z*: 334.1290.

**Methyl 3,5-di(9*H*-carbazol-9-yl)benzoate (20)**.[43] (1*R*,2*R*)-Cyclohexane-1,2-diamine (0.96 ml, ρ 0.95 g/cm$^3$, 7.96 mmol, 0.6 equiv) was added to a solution of methyl 3,5-dibromobenzoate (**19**) (3.90 g, 13.27 mmol, 1.0 equiv), CuI (756 mg, 3.98 mmol, 0.3 equiv), 9*H*-carbazole (5.54 g, 33.18 mmol, 2.5 equiv), and anhydrous $K_3PO_4$ in dry PhMe (100 ml). The reaction mixture was stirred at 120°C for 48 h. Then the reaction mixture was centrifuged, the supernatant was extracted with $H_2O$ (3 × 40 ml) and brine (10 ml). The organic phase was evaporated, and silica gel column chromatography (eluent hexane in MTBE, gradient 0–20%), followed by precipitation from MeOH (50 ml) and washing with cold MeOH (3 × 20 ml), provided product **20**. Yield 1.61 g (26%), colorless solid. $R_f$ 0.79 ($CH_2Cl_2$). HPLC: $t_R$ 10.26 min. $^1$H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 4.00 (3H, s, $CH_3$); 7.34 (4H, t, $^3J$ = 7.5, H Ar); 7.46 (4H, t, $^3J$ = 7.5, H Ar); 7.54 (4H, d, $^3J$ = 7.5, H Ar); 8.04 (1H, t, $^4J$ = 1.9, H Ar); 8.17 (4H, d, $^3J$ = 7.5, H Ar); 8.39 (2H, d, $^4J$ = 1.9, H Ar). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 52.9; 109.7; 120.7; 120.8; 123.9; 126.5; 126.8; 129.4; 134.0; 139.9; 140.5; 168.5. Found, *m*/*z*: 467.1772 $[M+H]^+$. $C_{32}H_{23}N_2O_2$. Calculated, *m*/*z*: 467.1754.

**[3,5-Di(9*H*-carbazol-9-yl)phenyl]methanol (21)**.[44] A solution of $LiAlH_4$ (406 mg, 10.20 mmol, 5.0 equiv) in dry THF (20 ml) was stirred at 0°C for 30 min, then a solution of compound **20** (1.00 g, 2.14 mmol, 1.0 equiv) in dry THF (10 ml) was added dropwise, and the reaction mixture was stirred at 0°C for 2 h. Then the reaction mixture was evaporated, suspended in $CH_2Cl_2$ (20 ml), and centrifuged. Silica gel column chromatography ($CH_2Cl_2$) provided product **21**. Yield 676 mg (72%), colorless solid. $R_f$ 0.36 ($CH_2Cl_2$). HPLC: $t_R$ 8.41 min. $^1$H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.99 (1H, br. s, OH), 4.95 (2H, s, $CH_2$); 7.33 (4H, t, $^3J$ = 7.5, H Ar); 7.46 (4H, t, $^3J$ = 7.5, H Ar); 7.56 (4H, d, $^3J$ = 7.5, H Ar); 7.73 (2H, s, H Ar); 7.76 (1H, s, H Ar); 8.17 (4H, d, $^3J$ = 7.5, H Ar). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 64.6; 109.8; 120.5; 120.6; 123.8; 123.9; 124.3; 126.3; 139.7; 140.7; 144.9. Found, *m*/*z*: 439.1835 $[M+H]^+$. $C_{31}H_{23}N_2O$. Calculated, *m*/*z*: 439.1805.

Supplementary information file containing experimental procedures for compounds **2**, **8**, **14**–**16** and $^1$H and $^{13}$C NMR spectra of compounds **4**, **9a**–**d**, **10a**, **11a**,**b**, **12**, **13**, **17**, **18a**–**c**, **20**, and **21**, is available at the journal website http://hgs.osi.lv.

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