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ORIGINAL PAPER

# Pseudo-three-component synthesis of substituted 1,2,4-triazolo[1,5-*a*]pyridines

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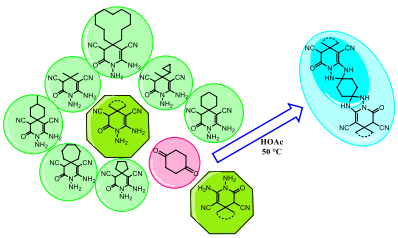
## Abstract

Heterocyclic 1,2,4-triazoles and their derivatives, such as triazolopyridines, have been used as leading components for the synthesis of numerous heterocyclic compounds; and because of their various biological activities, they have a distinct location in pharmaceutical and medicinal chemistry. In this article, we demonstrate a new way of synthesizing 1,2,4-triazolo[1,5-*a*]-pyridine derivatives, as a one-pot pseudo-three-component reaction, via the reaction of pyridine-2-(1*H*)-one derivatives and 1,4-cyclohexadione in the adjacency of acetic acid both as solvent and an inexpensive and green catalyst at 50 °C. The desired products were synthesized in good yields, and the chemical structure of the synthesized compounds was recognized using $^{1}$H and $^{13}$C NMR spectra, FT-IR, melting point, mass spectroscopy, and elementary analysis.

### Graphic abstract




**Keywords** Green synthesis · 1,4-Cyclohexadione · 1,2,4-Triazolo[1,5-*a*]pyridine · Pseudo-three-component reaction · Pyridine-2-(1*H*)-one derivatives

## Introduction

Multi-component reactions (MCRs) have appeared as highly impressive instrumentation in modern synthetic organic chemistry due to properties such as atom economy, direct and straight reaction design, and the opportunity to manufacture target organic molecules by presenting various elements. Typically, purification of products resulting from MCRs is also reasonably modest since all the occupied reactants are used and are synthesized into the target compound [1–10]. MCRs leading to interesting heterocyclic compounds are individually crucial for the preparation of diverse chemical libraries of drug-like molecules [11].

Heterocyclic chemistry is a highly competing and amply rewarding field, and by far, heterocycles form the largest class in organic chemistry. The majority of biologically active agrochemicals, pharmaceuticals, modifiers, and additives used in industrial usages are heterocyclic by nature [12]. Synthetic organic chemists have made remarkable progress in detecting and developing a vast range of heterocyclic compounds human benefits. Among the heterocyclic compounds, pyridines and triazoles are among the key heterocycles exhibiting significant pharmacological activity as they are fundamental constituents of all cells and

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living matter [13, 14]. Triazole is a five-membered heterocyclic ring, which contains three nitrogen atoms 1, 2, and 4 locations. Several methods have previously been related to triazolo[1,5-*a*]pyridines production from pyridines [15–18], especially 1,6-diaminopyridines [19–23]. As heterocyclic components, triazolopyridines have been applied in the construction of pharmaceutical ingredients with diverse biological effects [24–27], including antiproliferative, anti-inflammatory, and antithrombotic agents. Triazolopyridines act as inhibitors at growth hormone secretagogs [28] or mitogen-activated protein (MAP) kinases [29, 30]. They are also used to treat antithrombotic agents [31] and gastrointestinal disorders [32]. Therefore, various suitable methods are of remarkable interest for their synthesis. 1,2,4-Triazole-based compounds exhibit vastly potential usages in supramolecular, medicinal, chemical, agricultural, as well as material sciences [33, 34]. In medicinal chemistry, the unique structure of triazole ring made its derivatives easily fasten with a diversity of enzymes and receivers such as antioxidants, anticoagulants, anticancer, as well as antifungal, antiviral, antibacterial, and anti-inflammatory agents in biological activities [35–37].

## Results and discussion

The enormous biological and pharmacological importance of triazolopyridine derivatives encouraged us to design a modern and effective protocol for their synthesis. In this scope, being commercially available, economically cost-effective, and environmentally friendly, acetic acid could be used as an essential, efficient, and green catalyst in the synthesis of novel 1,2,4-triazolo[1,5-*a*] pyridine derivatives. In our experiment for the integration of 1,2,4-triazolo[1,5-*a*]pyridines, we performed the reaction between 1,6-diaminopyridines **1** (2 mmol) and 1,4-cyclohexadione (**2**, 1 mmol) in the adjacency of heated acetic acid at 50 °C (Scheme 1). The reaction was carried out in different conditions and the best result was obtained for acetic acid as both solvent and an inexpensive and green catalyst at 50 °C (Table 1, entry 7). The separated product 7,7,7″,7″-tetramethyl-5,5″-dioxo-1,1″,5,5″,6,6″,7,7″-octahydro-3*H*,3″*H*-dispiro[[1,2,4] triazolo[1,5-*a*]pyridine-2,1′-cyclohexane-4′,2″-[1,2,4] triazolo[1,5-*a*]pyridine]-6,6″,8,8″-tetracarbonitrile (**3a**) was fully described based on FT-IR, $^{1}$H, $^{13}$C NMR, and MS spectroscopy and elemental analysis. In the $^{1}$H NMR spectra, the four protons at NH of triazole rings display two singlets at 6.36, 6.39 and two singlets at 8.70, 8.73 ppm; moreover, two protons at CH of pyridine rings display two singlets at 4.59, 4.61 ppm, and the presence of this number of peaks in these areas indicates the reaction of two components of 1,6-diaminopyridine with 1,4-cyclohexadione (see experimental section). To study the possibility of this work, diversity of pyridine-2(1*H*)-one derivatives synthesis by a reported manner [38] reacted with 1,4-cyclohexadione under reaction conditions and led to final products in acceptable yields (Table 2).

The proposed mechanism for the acid-catalyzed reaction between 1,4-cyclohexadione and pyridine-2-(1*H*)-one derivatives is depicted in Scheme 2. Initially, the active group of amine that bonded to the *N* atom of the pyridine ring attacks the carbonyl group and immediately other amine group attacks the same carbon after aqueous distillation by acetic acid as catalyst, leading to the production of 1,2,4-triazole biologically active heterocyclic helper headings, and then the same happens to the other carbonyl group in 1,4-cyclohexadione.

**Scheme 1**

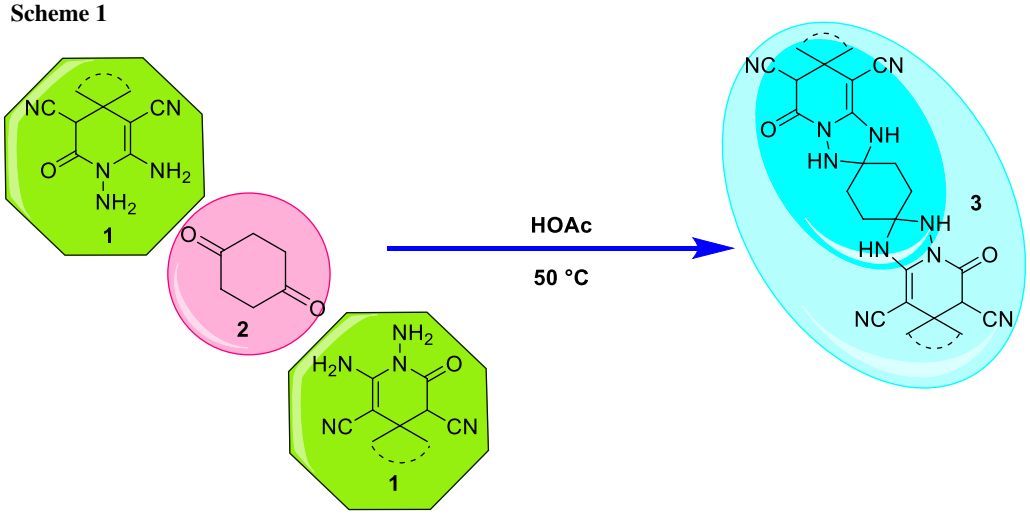
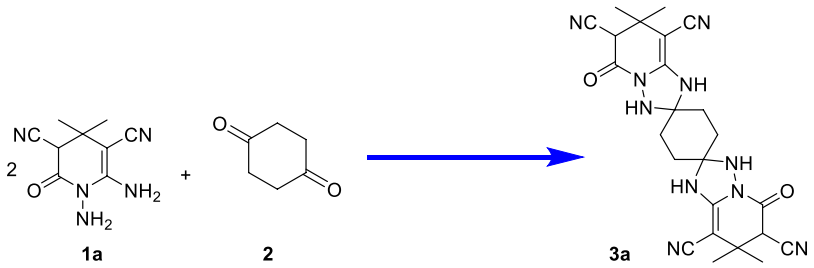


**Table 1** Optimization of the reaction condition for the synthesis of highly substituted 1,2,4-triazole




| Entry | Catalyst | Solvent | *T*/°C | Time/min | Isolated yields/% |
|---|---|---|---|---|---|
| 1 | – | EtOH | R.T. | 120 | – |
| 2 | PTSA (10 mol%) | EtOH | R.T. | 120 | Trace |
| 3 | HOAc (20 mol%) | EtOH | R.T. | 120 | 20 |
| 4 | – | HOAc | R.T. | 120 | 65 |
| 5 | HOAc (10 mol%) | EtOH | 50 | 60 | 43 |
| 6 | HOAc (20 mol%) | EtOH | 50 | 60 | 56 |
| **7** | **–** | **HOAc** | **50** | **60** | **90** |

Reaction conditions: pyridine-2-(1*H*)-one derivative **1a** (2.0 mmol), 1,4-cyclohexadione (**2**, 1.0 mmol)

## Conclusion

In conclusion, in a novel and efficient approach, we characterized and synthesized 1,2,4-triazolo[1,5-*a*]pyridine derivatives as new spiro biologically active compounds with the reaction of pyridine-2(1*H*)-one derivatives with 1,4-cyclohexadione compounds and by applying acetic acid both as solvent and a green catalyst. This method offers several benefits including green conditions, short reaction times, mild reaction conditions, excellent yields, and a simple workup procedure, but no need for column chromatography. Via its Brϕnsted acid nature, acetic acid advances reactions by being involved in nucleophilic addition as well as dehydration steps.

## Experimental

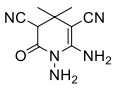
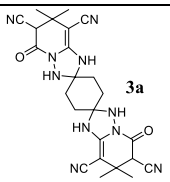
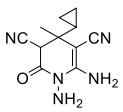
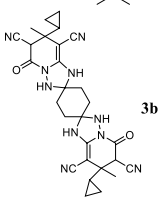
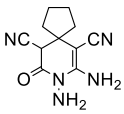
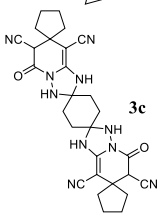
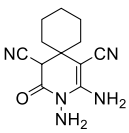
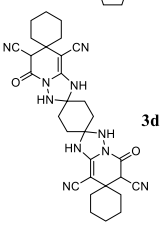
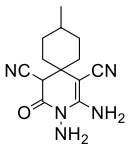
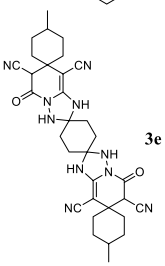
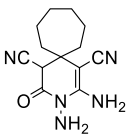
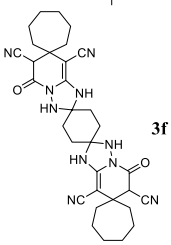
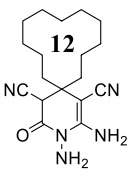
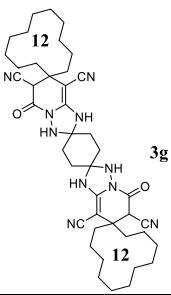
Melting points of all compounds were measured with an Electrothermal 9100 apparatus. All of the reagents were purchased from Fluka, Merck, and Aldrich companies, and were used without further purification. In addition, the $^{1}$H NMR and $^{13}$C NMR spectra were recorded on a Bruker Avance DPX-300 instrument using DMSO-$d_6$ as an internal standard at 300 and 75 MHz, respectively. FT-IR spectra of all compounds were measured with a JASCO FT-IR-460 plus spectrometer. Elemental analyses for C, H, and N, and mass spectra were recorded using a Heraeus CHN–O-Rapid analyzer and on an Agilent Technology (HP) 5973 mass spectrometer operating at an ionization potential of 70 eV, respectively.

### General procedure for the synthesis of compound 3

Initially, pyridine-2-(1*H*)-one derivatives **1** were prepared by a reported process [38]. Then, a mixture of pyridine-2-(1*H*)-one derivatives **1** (2.0 mmol) and 1,4-cyclohexadione (**2**, 1.0 mmol) in 2.0 cm$^3$ acetic acid was located in a 5.0 cm$^3$ round-bottomed flask mounted over a magnetic stirrer. The contents in an oil bath maintained at 50 °C for an adequate time were stirred magnetically as shown in Table 2. The reaction time was monitored by TLC; after the reaction was complete, the reaction mixture was allowed to cool at room temperature. Then, the solid was obtained smooth, and the reliable product was separated. The separated product was washed twice with water ($2 \times 10$ cm$^3$) to afford pure products. The spectral and analytical data of all the compounds are given below.

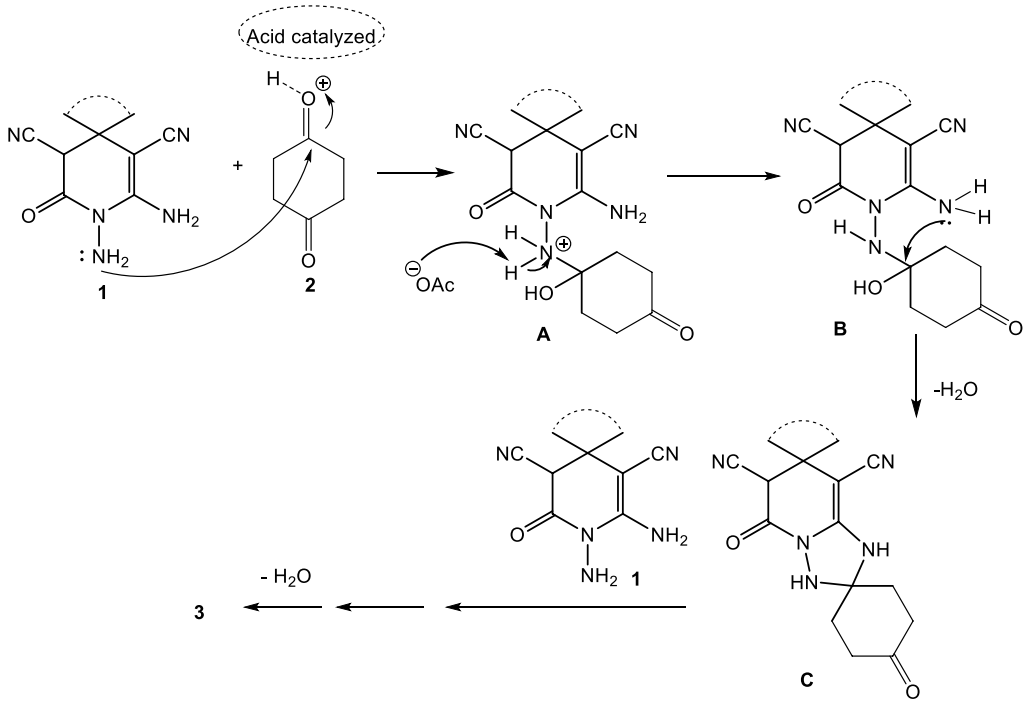
**7,7,7″,7″-Tetramethyl-5,5″-dioxo-1,1″,5,5″,6,6″,7,7″-octahydro-3*H*,3″*H*-dispiro[[1,2,4]triazolo[1,5-*a*]pyridine-2,1′-cyclohexane-4′,2″-[1,2,4]triazolo[1,5-*a*]pyridine]-6,6″,8,8″-tetra-carbonitrile (3a, $C_{24}H_{26}N_{10}O_2$)** Green-blue solid; yield 90%; m.p.: 287–289 °C; $^{1}$H NMR (DMSO-$d_6$, 300 MHz): $\delta = 8.73$ (s, NH, 1H), 8.70 (s, NH, 1H), 6.39 (s, NH, 1H), 6.36 (s, NH, 1H), 4.61 (s, CH, 1H), 4.59 (s, CH, 1H), 1.93–1.66 (m, 4CH$_2$, 8H), 1.31 (s, 2CH$_3$, 6H), 1.17 (s, 2CH$_3$, 6H) ppm; $^{13}$C NMR (DMSO-$d_6$, 75 MHz): $\delta = 21.53$, 24.70, 26.78, 30.80, 31.20, 31.41, 35.61, 48.23, 59.98, 60.05, 77.57, 115.83, 118.78, 118.80, 151.10, 151.42, 157.94, 158.10 ppm; FT-IR (KBr): $\bar{\nu} = 3368$, 3229, 2928, 2864, 2184,

**Table 2** Reaction of pyridine-2-(1*H*)-one derivative and 1,4-cyclohexadione in the present of acetic acid

| Entry | 1a-1g | Product | Time /min | Isolated yield /% | M.p. /°C | Color |
|---|---|---|---|---|---|---|
| **1** |  |  **3a** | 60 | 90 | 287–289 | Green-blue |
| **2** |  |  **3b** | 120 | 85 | 285–288 | Green-blue |
| **3** |  |  **3c** | 90 | 80 | 267–268 | Green-blue |
| **4** |  |  **3d** | 60 | 95 | 286–288 | Green-blue |
| **5** |  |  **3e** | 120 | 82 | 289–290 | Green-blue |
| **6** |  |  **3f** | 60 | 87 | 266–267 | Green-blue |
| **7** |  |  **3g** | 60 | 93 | 250–252 | Green |

Reaction conditions: pyridine-2-(1*H*)-one derivative (2.0 mmol), 1,4-cyclohexadione (1.0 mmol), 2.0 $cm^3$ acetic acid, 50 °C

**Scheme 2**




1711, 1651, 1395, 1099 $cm^{-1}$; MS (EI, 70 eV): *m/z* (%) = 485 ($M^+$, 31).

**7,7″-Dicyclopropyl-7,7″-dimethyl-5,5″-dioxo-1,1″,5,5″,6,6″,7,7″-octahydro-3*H*,3″*H*-dispiro[[1,2,4]triazolo[1,5-*a*]pyridine-2,1′-cyclohexane-4′,2″-[1,2,4]triazolo[1,5-*a*]pyridine]-6,6″,8,8″-tetracarbonitrile (3b, $C_{28}H_{30}N_{10}O_2$)** Green-blue solid; yield 85%; m.p.: 285–288 °C; $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta$ = 8.61 (s, 1H, NH), 8.56 (s, 1H, NH), 6.35 (s, 1H, NH), 6.32 (s, 1H, NH), 4.61 (s, 1H, CH), 4.59 (s, 1H, CH), 1.78–1.46 (m, 8H, 4$CH_2$), 1.21 (s, 6H, 2 $CH_3$), 0.81–0.79 (m, 2H, 2CH), 0.28–0.25 (m, 4H, 2$CH_2$), 0.00 to − 0.06 (m, 4H, 2$CH_2$) ppm; $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta$ = − 1.51 ($CH_2$ cyclopropyl), − 0.50 ($CH_2$ cyclopropyl), 15.31 (CH cyclopropyl), 15.36 ($CH_3$), 23.44, 23.61, 28.37, 28.46, 29.31, 29.38, 36.41, 36.43, 46.57, 51.44, 51.47, 75.49, 75.52, 113.79, 117.60, 117.62, 150.08, 150.15, 156.17, 156.19 ppm; IR (KBr): $\bar{\nu}$ = 3362, 3238, 2935, 2862, 2191, 1716, 1651, 1411 $cm^{-1}$; MS (EI, 70 eV): *m/z* (%) = 537 ([M-1]$^+$, 100).

**5′,5‴-Dioxo-5′,5‴,6′,6‴-tetrahydro-1′*H*,1‴*H*,3′*H*,3‴*H*-tetraspiro[cyclopentane-1,7′-[1,2,4]triazolo[1,5-*a*]pyridine-2′,1″-cyclohexane-4″,2‴-[1,2,4]triazolo[1,5-*a*]pyridine-7‴,1′′′′-cyclopentane]-6′,6‴,8′,8‴-tetracarbonitrile (3c, $C_{28}H_{30}N_{10}O_2$)** Green-blue solid; yield 80%; m.p.: 267–269 °C; $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta$ = 8.76 (s, 1H, NH), 8.75 (s, 1H, NH), 6.36 (s, 2H, 2NH), 4.66 (s, 1H, CH), 4.64 (s, 1H, CH), 2.10–1.62 (m, 24H, 12$CH_2$) ppm; $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta$ = 24.44, 24.60, 30.92, 31.13, 31.25, 36.54, 36.60, 37.17, 45.79, 46.30, 46.34, 60.02, 77.77, 116.33, 119.41, 119.44, 151.52, 158.69, 158.73 ppm; IR (KBr): $\bar{\nu}$ = 451, 3329, 2928, 2800, 2251, 2180, 1702, 1645, 1415, 1104 $cm^{-1}$; MS (EI, 70 eV): *m/z* = 538.

**5′,5‴-Dioxo-5′,5‴,6′,6‴-tetrahydro-1′*H*,1‴*H*,3′*H*,3‴*H*-tetraspiro[cyclohexane-1,7′-[1,2,4]triazolo[1,5-*a*]pyridine-2′,1″-cyclohexane-4″,2‴-[1,2,4]triazolo[1,5-*a*]pyridine-7‴,1′′′′-cyclohexane]-6′,6‴,8′,8‴-tetracarbonitrile (3d, $C_{30}H_{34}N_{10}O_2$)** Green-blue solid; yield 95%; m.p.: 286–288 °C; $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta$ = 8.79 (s, 1H, NH), 8.78 (s, 1H, NH), 6.36 (s, 1H, NH), 6.35 (s, 1H, NH), 4.54 (s, 1H, CH), 4.52 (s, 1H, CH), 1.93–1.31 (m, 28H, 14$CH_2$) ppm; $^{13}$C NMR (75 MHz, DMSO-$d_6$): $\delta$ = 21.52, 21.68, 25.26, 31.00, 31.10, 33.37, 34.18, 38.24, 38.26, 46.45, 46.86, 58.18, 58.31, 77.63, 116.06, 120.15, 120.25, 152.22, 152.28, 158.06, 158.18 ppm; IR (KBr): $\bar{\nu}$ = 3368, 3208, 2931, 2858, 2175, 1703, 1639, 1413, 1106 $cm^{-1}$; MS (EI, 70 eV): *m/z* = 565.

**4,4′′′′-Dimethyl-5′,5‴-dioxo-5′,5‴,6′,6‴-tetrahydro-1′*H*,1‴*H*,3′*H*,3‴*H*-tetraspiro[cyclohexane-1,7′-[1,2,4]triazolo[1,5-*a*]pyridine-2′,1″-cyclohexane-4″,2‴-[1,2,4]triazolo[1,5-*a*]pyridine-7‴,1′′′′-cyclohexane]-6′,6‴,8′,8‴-tetracarbonitrile (3e, $C_{32}H_{38}N_{10}O_2$)** Green-blue solid; yield 82%; m.p.: 289–290 °C; $^1$H NMR (300 MHz, DMSO-$d_6$): $\delta$ = 8.82 (s, 1H, NH), 8.80 (s, 1H, NH), 6.35 (s, 2H, 2NH), 4.58 (s, 1H, CH), 4.56 (s, 1H, CH), 1.83–1.66 (m, 26H, 2CH, 12$CH_2$), 0.96 (d, *J* = 3 Hz, 6H,

$2CH_3$) ppm; $^{13}C$ NMR (75 MHz, DMSO-$d_6$): $\delta = 22.51, 30.51, 30.80, 31.09, 31.19, 31.57, 32.94, 34.09, 37.74, 49.10, 49.13, 56.98, 57.03, 77.70, 115.92, 120.84, 120.86, 152.70, 158.77, 158.79$ ppm; IR (KBr): $\bar{\nu} = 3361, 3202, 2925, 2856, 2176, 1702, 1641, 1415, 1109$ cm$^{-1}$; MS (EI, 70 eV): $m/z = 593$.

**5′,5‴-Dioxo-5′,5‴,6′,6‴-tetrahydro-1′*H*,1‴*H*,3′*H*,3‴*H*-tetraspiro[cycloheptane-1,7′-[1,2,4]triazolo[1,5-*a*]pyridine-2′,1″-cyclohexane-4″,2‴-[1,2,4]triazolo[1,5-*a*]pyridine-7‴,1⁗-cycloheptane]-6′,6‴,8′,8‴-tetracarbonitrile (3f, $C_{32}H_{38}N_{10}O_2$)** Green–blue solid; yield 87%; m.p.: 266–267 °C; $^1H$ NMR (300 MHz, DMSO-$d_6$): $\delta = 8.72$ (s, 2H, 2NH), 6.37 (s, 2H, 2NH), 4.49 (s, 1H, CH), 4.47 (s, 1H, CH), 1.93–1.45 (m, 32H, $16CH_2$) ppm; $^{13}C$ NMR (75 MHz, DMSO-$d_6$): $\delta = 21.51, 22.68, 23.10, 30.10, 30.38, 30.99, 35.87, 38.66, 41.39, 48.20, 60.59, 77.60, 116.45, 120.09, 120.13, 151.66, 158.12, 158.17$ ppm; IR (KBr): $\bar{\nu} = 3451, 3329, 2928, 2800, 2251, 2180, 1702, 1645, 1415, 1104$ cm$^{-1}$; MS (EI, 70 eV): $m/z = 593$.

**5′,5‴-Dioxo-5′,5‴,6′,6‴-tetrahydro-1′*H*,1‴*H*,3′*H*,3‴*H*-tetraspiro[cyclododecane-1,7′-[1,2,4]triazolo[1,5-*a*]pyridine-2′,1″-cyclohexane-4″,2‴-[1,2,4]triazolo[1,5-*a*]pyridine-7‴,1⁗-cyclododecane]-6′,6‴,8′,8‴-tetracarbonitrile (3g, $C_{42}H_{58}N_{10}O_2$)** Green solid; yield 93%; m.p.: 250–252 °C; $^1H$ NMR (300 MHz, DMSO-$d_6$): $\delta = 8.99$ (s, 1H, NH), 8.74 (s, 1H, NH), 6.62 (s, H, NH), 6.41(s, 1H, NH), 4.20 (s, 1H, CH), 4.14 (s, 1H, CH), 1.93–1.36 (m, 52H, $26CH_2$) ppm; $^{13}C$ NMR (75 MHz, DMSO-$d_6$): $\delta = 19.94, 21.51, 21.94, 22.11, 22.20, 22.46, 26.00, 26.28, 26.40, 30.73, 30.79, 31.40, 31.56, 32.01, 44.48, 44.49, 57.80, 58.37, 77.45, 116.04, 119.64, 119.69, 150.92, 151.20, 57.12, 157.41$ ppm; IR (KBr): $\bar{\nu} = 3362, 3238, 2935, 2862, 2191, 1716, 1651, 1411$ cm$^{-1}$; MS (EI, 70 eV): $m/z = 734$.

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