SUPPORTING INFORMATION

Five coordination Cu(II) cluster-based MOF and its application in synthesis of pharmaceuticals via Sp³ C-H/N-H oxidative couplings


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PART 1: SYNTHESIS AND CHARACTERIZATION OF VNU-18

Section S1. Synthesis and Characterization of VNU-18: Chemicals

$N,N$-dimethylformamide (DMF), methanol, and dichloromethane (DCM) were all purchased from Sigma Aldrich Chemical Company. 3,5-pyridinedicarboxylic acid, and coppernitratetrihydrate were purchased from Acros Organics. All chemicals were used without further purification.
Section S2. Synthesis, Activation, and Characterization of VNU-18, including Thermal Gravimetric Analysis and Gas Adsorption

Synthesis and Activation of Cu$_5$O$_2$(PDC)$_5$Cu(OH)$_2$, VNU-18

Cu(NO$_3$)$_2$·3H$_2$O (210 mg, 0.869 mmol) and H$_2$PDC (135 mg, 0.808 mmol) were dissolved in 60 mL DMF. The solution was subsequently heated at 100 ºC for 24 h in an isothermal oven to yield light blue single crystals. After cooling to room temperature, the light blue solid product was separated from the mother liquor by filtration.

VNU-18 as-synthesized samples were activated (removal of guest molecules from the pores) as follows: The obtained solid was washed three times in 1 day with 240 mL of DMF. After this period, the DMF was replaced with methanol and the product was washed three times over two days with fresh methanol each time. Then the solvent was replaced with dichloromethane and washed three times over one day with fresh dichloromethane each time. Solvent-exchanged VNU-18 was dried under vacuum at 85 ºC for 24 h to yield 192 mg of activated material (92% based on H$_2$-PDC linker). PXRD patterns were measured on activated samples and demonstrated excellent agreement with the simulated pattern generated from single crystal structure.

ICP of activated sample: Calcd. for C$_{35}$H$_{17}$N$_5$O$_{26}$Cu$_6$ = [Cu$_5$O$_2$(OH)$_2$(C$_7$H$_3$NO$_4$)$_5$]: Cu, 29.22%. Found: Cu, 29.6 %.

Thermal Gravimetric Analysis

In general, a ~12 mg sample of activated VNU-18 was run on a TA Instrumental Q-500 Series Thermal Gravimetric Analyzer. The sample was held in a platinum pan under a continuous air flow. During the process, the sample was heated at a constant rate of 5 ºC.
min⁻¹. The initial weight loss is attributed to adsorbed water molecules when transferring sample to the platinum pan. It is noted that the presumably CuO residue (35.2%) in the activated sample is in satisfactory agreement with the calculated theoretical value (37.0%).

Fig. S1. Thermal gravimetric analysis trace of activated (guest-free) VNU-18.
Gas Adsorption

Low-pressure N\textsubscript{2} adsorption measurements were carried out on a Micromeritics 3Flex Surface Characterization Analyzer. A liquid N\textsubscript{2} bath was used for measurements at 77 K. Helium was used as estimation of dead space. Ultrahigh-purity-grade N\textsubscript{2} and He (99.999\% purity) were used throughout adsorption experiments.

![Figure S2](image-url)  

**Figure S2.** N\textsubscript{2} isotherm of VNU-18 at 77 K. Filled and open symbols represent adsorption and desorption, respectively. The connected lines are inserted as guides for the eyes.
Section S3. X-ray Crystallography

Powder X-ray diffraction data collection

Powder X-ray diffraction (PXRD) data was collected on a Bruker D8-Advance \( \theta-\theta \) diffractometer, equipped with a LynxEye detector, in reflectance Bragg-Brentano geometry employing Ni filtered (0.2 mm) Cu K\( \alpha \) lines focused radiation (1.54059 Å, 1.54439 Å) at 1600 W (40 kV, 40 mA) power. A dried and activated powder sample of VNU-18 was mounted on a zero background holder and then leveled using a razor blade. The procedure for obtaining the best counting intensity used a measurement step scan of 0.02° 2\( \theta \) from 3 – 30° with exposure time of 0.3 second per step. The measurement was performed at room temperature and atmospheric pressure.

Figure S3. PXRD patterns of VNU-18 demonstrated the good agreement between simulated, as-synthesized and activated samples.
Single crystal X-ray diffraction data collection

A single crystal of VNU-18 was isolated from the mother liquor of the reaction by a nylon loop and mounted. The X-ray diffraction data for these materials were both collected on a Bruker D8 Venture diffractometer outfitted with a PHOTON-100 CMOS detector using monochromatic microfocus CuKa radiation (λ=1.54178 Å) that was operated at 50 kW and 1.0 mA. The single crystal data was collected at room temperature. Unit cell determination was performed in the Bruker SMART APEX II software suite. The data sets were reduced and a multi-scan spherical absorption correction was implemented in the SCALE interface. The structures were solved with direct methods and refined by the full-matrix least-squares method in the SHELXL-97 program package. Once the framework atoms were located in the difference Fourier maps, the SQUEEZE routine in PLATON was performed to remove scattering from disordered guest molecules residing in the pores. Detailed descriptions of structural refinement can be found in Table S1.

Table S1. Structural refinement of VNU-18

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<tr>
<td></td>
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<tr>
<td></td>
<td>$c = 15.2457(8) \text{Å}$</td>
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<td>$\gamma$</td>
<td>$90°$</td>
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Largest diff. peak and hole

| 2.003 and -0.451 e·Å⁻³ |

**Figure S4.** As-symmetry unit of VNU-18 drawn by ORTEP with thermal ellipsoids style at 50% probability.

CIF-file has been deposited with Cambridge Crystallographic Data Center (CCDC) and can be obtained free of charge. CCDC number: 1515280.
Section S4. Topological analysis

Topology for VNU-18 was analyzed by TOPOS 4.0 software. The structure was firstly simplified to generate the point of extension. The single pentanuclear Cu cluster connected to two PDC units, which was simplified as square planar building unit. Three-dimensional topology of VNU-18 was found as $4, 4, 4 \text{T}10$ constructed based two 4-connected points of linking square.

Figure S5. Topological presentation for VNU-18. Type of Cu-building block (a), which is represented by square connection in (b). The combination of two square connections yields the $4, 4, 4, \text{T}10$ topology (c).
PART 2: CATALYSIS

Section S5. Catalysis: Materials and Instrumentation

All reagents and starting materials were obtained commercially from Sigma-Aldrich and Acros, and were used as received without any further purification unless otherwise noted. Gas chromatographic (GC) analyses were performed using a Shimadzu GC 2010-Plus equipped with a flame ionization detector (FID) and an SPB-5 column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μm). The temperature program for GC analysis heated samples from 100 °C for 1.0 minutes; heated from 100 to 280 °C at 40 °C/min; held at 280 °C for 3 min. Inlet and detector temperature were set constant at 280 °C. Diphenyl ether was used as internal standard to calculate the GC yield of reaction. The ¹H and ¹³C NMR were recorded on a Brucker AV 500 MHz with tetramethylsilane as standard. ICP was conducted using PerkinElmer 350D.

Section S6. Catalysis: Optimization

Synthesis of Cu₃(BTC)₂

The procedure to prepare Cu₃(BTC)₂ was similar to that previously reported.⁵ In a typical preparation, a solid mixture of Cu(NO₃)₂·3H₂O (0.438 g, 1.81 mmol) and 1,3,5-benzenetricarboxylic acid (H₃BTC) (0.236 g, 1.12 mmol) was dissolved in a mixture of DMF (3 mL), ethanol (4 mL) and water (2 mL) in a 20 mL vial. The vial was heated at 85 °C in an isothermal oven for 24 h, yielding light blue crystals. After cooling the vial to room temperature, the solid product was obtained by decanting with mother liquor and washed with DMF (3 x 8 mL). Solvent exchange was then carried out with ethanol (3 x 8 mL) at room temperature. The product was then dried under vacuum at 170 °C for 6 h,
yielding 0.285 g of Cu$_3$(BTC)$_2$ in the form of deep purple crystals (84% based on 1,3,5-benzenetricarboxylic acid).

![Fig S6. X-ray powder diffractogram of the Cu$_3$(BTC)$_2$](image)

**Synthesis of Cu(BDC)**

The Cu(BDC) was prepared according to literature procedure.$^6$ In a typical preparation, a mixture of 1,4-benzenedicarboxylic acid (H$_2$BDC) (0.332 g, 2 mmol), and Cu(NO$_3$)$_2$.3H$_2$O (0.4235 g, 1.75 mmol) was dissolved in DMF (DMF = N,N'-dimethylformamide; 45 mL), and the resulting solution was distributed to six 20 mL vials. The vial was then heated at 130 $^\circ$C in an isothermal oven for 48 h. After cooling the vial to room temperature, the solid product was removed by decanting with mother liquor and washed in DMF (3 x 10 mL) for 3 days. Solvent exchange was then carried out with dichloromethane (DCM) (3 x 10 mL) at room temperature for 3 days. The material was then evacuated under vacuum at 160 $^\circ$C for 6 h, yielding 0.308 g of Cu(BDC) in the form of blue crystals (77% based on copper nitrate).
The Cu$_2$(BDC)$_2$(BPY) was prepared according to literature procedure.$^7$ A solid mixture of Cu(NO$_3$)$_2$.3H$_2$O (0.105 g, 0.4 mmol), 1,4-benzenedicarboxylic acid (H$_2$BDC) (0.068 g, 0.4 mmol), and 4,4’-bipyridine (4,4’-bpy) (0.062 g, 0.4 mmol) was dissolved in DMF (30 mL). The resulting solution was then distributed in four 20 mL vials. The vials were heated at 120 °C in an isothermal oven for 24 h, yielding light blue crystals. After cooling the vials to room temperature, the solid product was obtained by decanting with mother liquor and washed with DMF (3 x 10 mL). Solvent exchange was then carried out with methanol (3 x 10 mL) at room temperature. The product was then dried under vacuum at 120 °C for 6 h, yielding 0.184 g of Cu$_2$(BDC)$_2$(BPY) (75% based on 1,4-benzenedicarboxylic acid).
Fig S8. X-ray powder diffractogram of the Cu$_2$(BDC)$_2$(BPY)

**Synthesis of Cu$_2$(PBDC)$_2$(BPY)**

In a typical preparation,$^{[8]}$ a solid mixture of H$_2$BPDC (H$_2$BPDC = 4,4’-biphenyldicarboxylic acid; 0.630 g, 2.4 mmol), bpy (bpy = 4,4’-bipyridine; 0.198 g, 1.2 mmol), and Cu(NO$_3$)$_2$.3H$_2$O (0.630 g, 2.4 mmol) was dissolved in a mixture of DMF (DMF = N,N’-dimethylformamide; 180 mL), pyridine (1.8 mL), and methanol (18 mL). The resulting solution was stirred at 70 °C for 5 min, and then distributed to 20 mL vials. The vials were then heated at 120 °C in an isothermal oven for 24 h. After cooling the vials to room temperature, the solid product was removed by decanting with mother liquor and washed in DMF (3 x 20 mL) for 3 days. Solvent exchange was carried out with methanol (3 x 20 mL) at room temperature for 3 days. The material was then evacuated under vacuum at 140 °C for 6 h, yielding 0.605 g of Cu$_2$(BPDC)$_2$(BPY) in the form of blue crystals, correspond to 66 % based on copper.
Synthesis of Cu₂(BDC)₂(DABCO)

Cu₂(BDC)₂(DABCO) was prepared according to literature procedure,[9] a mixture of H₂BDC (H₂BDC = 1,4-benzenedicarboxylic acid; 0.506 g, 3.1 mmol), DABCO (DABCO = 1,4-diazabicyclo[2.2.2]octane; 0.188 g, 1.67 mmol), and Cu(NO₃)₂·3H₂O (0.8 g, 3.3 mmol) was dissolved in DMF (DMF = N,N’-dimethylformamide, 80 mL). The mixture was stirred for 2 h, and the resulting solution was then distributed to eight 10 mL vials. The vial was heated at 120 °C in an isothermal oven for 48 h, forming blue crystals. After cooling the vial to room temperature, the solid product was removed by decanting with mother liquor and washed with DMF (3 x 10 mL). Solvent exchange was carried out with methanol (3 x 10 mL) at room temperature. The product was then dried at 140 °C for 6 h under vacuum, yielding 0.57 g of the metal–organic framework Cu₂(BDC)₂(DABCO) as light blue crystals (66% yield).
**Fig. S10.** PXRD of as-synthesized Cu₂(BDC)₂(DABCO) and simulated patterns.

*Catalytic procedure:*

To a 8 mL vial were added VNU-18 (5.31 mg, 0.025 mmol, 0.5 mol%), KBr (18.0 mg, 0.15 mmol, 30 mol%), diphenyl ether (50 µL, as an internal standard), propiophenone 98% (68 mg, 0.5 mmol, 1 equiv.). The vial was stirred for 5 minutes. Then, morpholine (65.5 mg, 0.75 mmol, 1.5 equiv., in 2 mL of DMF) was drop-wise added during 10 minutes under air at room temperature. The vial was then sealed with rubber septum and stirred in indicated time. The resulting mixture was extracted with ethyl acetate (20 mL) and pure water (20 mL). The organic phase was dried over anhydrous Na₂SO₄ and concentrated under vacuum. After chromatography, pure products were obtained.
Figure S11. Effect of KBr amount on reaction yields.

Figure S12. Effect of reagent molar ratios on reaction yields.
Section S7. Catalysis: Leaching Test and Recycling Studies

For the leaching test, a catalytic reaction was stopped after 2 h, analyzed by GC, and filtered to remove the solid catalyst. The reaction solution was then stirred for a further 10 h after adding KBr. Reaction progress, if any, was monitored by GC as previously described.
Figure S14. Leaching test with catalyst removal after 2 hours

In recycling studies, the catalyst was separated from the reaction mixture by simple filtration, washed with copious amounts of DMF and methanol, dried 150 °C under vacuum in 8 h, and reused under identical conditions with previous runs.

Fig. S15. Recycling studies
Mechanistic studies:

**Step 1:** To a 8 mL vial were added VNU-18 (5.31 mg, 0.025 mmol, 0.5 mol%), KBr (18.0 mg, 0.15 mmol, 30 mol%), diphenyl ether (50 μL, as an internal standard), propiophenone 98 % (68 mg, 0.5 mmol, 1 equiv.). The vial was then sealed with rubber septum and stirred in 12 hour at room temperature. The resulting mixture was extracted with ethyl acetate (20 mL) and pure water (20 mL). The organic phase was dried over anhydrous Na₂SO₄ and concentrated under vacuum. After chromatography, α-bromo propiophenone was obtained in 81 % yield.

**Step 2:** To a 8 mL vial were added α-bromo propiophenone (108 mg, 0.5 mmol). The vial was stirred for 5 minutes. Then, morpholine (65.5 mg, 0.75 mmol, 1.5 equiv., in 2 mL of DMF) was drop-wise added during 10 minutes under air at room temperature. The vial was then sealed with rubber septum and stirred in 8 hours at room temperature. The

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**Fig. S16. PXRD of VNU-18 after 6\textsuperscript{th} run**
resulting mixture was extracted with ethyl acetate (20 mL) and pure water (20 mL). The organic phase was dried over anhydrous Na₂SO₄ and concentrated under vacuum. After chromatography, 2-morpholino-1-phenylpropan-1-one was obtained in 72 % yield.

2-Morpholino-1-phenylpropan-1-one

This compound is known. Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 87.6 mg (81 % of yield).

^1H NMR (500 Hz, CDCl₃) δ 8.09-8.08 (d, 2H), 7.58-7.54 (m, 1H), 7.45-7.27 (m, 2H), 4.10-4.06 (q, 1H), 3.72-3.65 (m, 4H), 2.66-2.60 (m, 2H), 2.59-2.55 (m, 2H), 1.31 (d, 3H)

^13C NMR (125 Hz, CDCl₃) δ 200.19, 136.19, 133.00, 128.75, 128.40, 77.27, 77.02, 76.76, 67.11, 64.70, 50.02, 11.62.

1-phenyl-2-(piperidin-1-yl)propan-1-one

This compound is known. Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 82.5 mg (76% of yield).

^1H NMR (500 Hz, CDCl₃) δ 8.12-8.10 (d, 2H), 7.55-7.51 (m, 1H), 7.45-7.42 (m, 2H), 4.10-4.06 (q, 1 H), 2.59-2.55 (m, 2H), 2.55-2.48 (m, 2H), 1.56-1.53 (m, 4H) 1.52-1.49 (m, 2H), 1.4 (m, 3H)

2-(4-methylpiperazin-1-yl)-1-phenylpropan-1-one

This compound is known. Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 91.5 mg (80 % of yield).

^1H NMR (500 Hz, CDCl₃) δ 8.00 (m, 2H), 7.61-7.59 (m, 1H), 7.49-7.44 (m, 2H), 4.29-4.25 (q, 1H), 3.11-3.0 (m, 4H), 2.96-2.88 (m, 2H), 2.75-2.53 (s, 3H), 1.35-1.24 (m, 3H)
Methyl 2-morpholino-2-phenylacetate (4)

This compound is known.\textsuperscript{[10]} Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 56.4 mg (48% of yield).

\textit{^1}H NMR (500 Hz, CDCl\textsubscript{3}) δ 7.45-7.43 (m, 2H), 7.36-7.26 (m, 3H), 4.00 (s, 1H), 3.74-3.73 (m, 4H), 3.72-3.68 (s, 3H), 2.46-2.44 (m, 4H)

Methyl 2-(4-hydroxyphenyl)-2-morpholinoacetate

This compound is known.\textsuperscript{[10]} Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 91.8 mg (73% of yield).

\textit{^1}H NMR (500 Hz, CDCl\textsubscript{3}) δ 7.29-7.26 (m, 2H), 6.81-6.80 (m, 2H), 3.90 (s, 1H), 3.75-3.73 (d, 4H), 3.72-3.68 (s, 3H), 2.45 (m, 4H)

3-Morpholinopentan-2-one

This compound is known.\textsuperscript{[10]} Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 66 mg (77% of yield).

\textit{^1}H NMR (500 Hz, CDCl\textsubscript{3}) δ 3.73-3.67 (m, 4H), 2.88-2.85 (m, 1H), 2.59-2.56 (m, 2H), 2.47-2.42 (m, 2H), 2.18-2.17 (s, 3H), 1.69-1.61 (m, 2H), 0.86 (t, 3H).

2-(2-Methyl-1-piperidinyl)-1-phenyl-1-propanone

This compound is known.\textsuperscript{[11]} Column chromatography using hexane:ethyl acetate = 7:1. Product was achieved with 66 mg (45% of yield).

\textit{^1}H NMR (500 Hz, CDCl\textsubscript{3}) δ 7.26-7.65 (m, 5H), 4.51 (q, J = 3.4, 1H), 2.95 – 3.05 (m, 1H), 2.48 – 2.54 (m, 2H), 1.35 – 1.65 (m, 6 H), 1.24 (d, J = 2.1, 3H), 1.12 (d, J = 2.1, 3H).

2-(3-Methyl-1-piperidinyl)-1-phenyl-1-propanone
This compound is known.[12] Column chromatography using hexane:ethyl acetate = 7:1.
Product was achieved with 85.4 mg (74 % of yield).

\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 8.10–8.15 (m, 2H), 7.48–7.52 (m, 1H), 7.40–7.46 (m, 2H), 4.05–4.11 (m, 1H), 2.80–2.83 (m, 1H), 2.63–2.67 (m, 1H), 1.72–2.35 (m, 2H), 1.44–1.64 (m, 4H), 1.23–1.26 (d, \(J = 6.9\) Hz, 3H), 0.71–0.85 (m, 4H).

2-(3,4-Dihydro-2-isoquinolinyl)-1-phenyl-1-propanone

This compound is known.[13] Column chromatography using hexane:ethyl acetate = 7:1.
Product was achieved with 85.4 mg (74 % of yield).

\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 8.11–8.18 (m, 2H), 7.50–7.56 (m, 1H), 7.40–7.48 (m, 2H), 7.01–7.15 (m, 4H), 4.33 (q, \(J = 6.8\) Hz, 1H), 3.85–3.95 (m, 1H), 3.75–3.82 (m, 1H), 2.79–2.85 (m, 4H), 1.36 (d, \(J = 7.1\) Hz, 3H).

\(\alpha\)-[Methyl(phenylmethyl)amino]-methylbenzenacetate

This compound is known.[14] Column chromatography using hexane:ethyl acetate = 7:1.
Product was achieved with 87.4 mg (65 % of yield).

\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 7.44 – 7.50 (m, 2H), 7.30 – 7.48 (m, 7H), 7.22-7.28 (m, 1H), 4.33 (s, 1H), 3.74 (s, 3H), 3.66 (d, \(J = 11.8\) Hz, 1H), 3.54 (d, \(J = 11.8\) Hz, 1H), 2.25 (s, 3H).

\(\alpha\)-(Methylphenylamino)-methylbenzenacetate

This compound is known.[15] Column chromatography using hexane:ethyl acetate = 5:1.
Product was achieved with 59.9 mg (47 % of yield).
\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 7.25 – 7.42 (m, 7H). 6.84 – 6.90 (m, 3H), 5.70 (s, 1H), 3.80 (s, 3H), 2.81 (s, 3H).

**2-(N-Benzylamino)-1-phenyl-1-propanone**

This compound is known.\(^{[16]}\) Column chromatography using hexane:ethyl acetate = 5:1. Product was achieved with 59.9 mg (47 % of yield).

\(^1\)H NMR (500 MHz, C\(_6\)D\(_6\)) \(\delta\) 7.72 – 7.80 (m, 2H), 7.40 – 6.89 (m, 8H), 4.08 (q, \(J = 7.2\) Hz, 1H), 3.58 – 3.64 (m, 2H), 1.14 (d, \(J = 7.2\) Hz, 3H).

**2-(Diethylamino)-1-phenyl-1-propanone**

This compound is known.\(^{[17]}\) Column chromatography using hexane:ethyl acetate = 9:1. Product was achieved with 76.8 mg (75 % of yield).

\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 8.06 – 8.12 (m, 2H), 7.50 – 7.54 (m, 1H), 7.39 – 7.45 (m, 2H), 4.40 (q, \(J = 7.1\) Hz, 1H), 2.52 – 2.68 (m, 4H), 1.24 (d, \(J = 7.1\) Hz, 3H), 1.03 (t, \(J = 7.4\) Hz, 6H).

**Methyl-2-(2-chlorophenyl)-2-(6,7-dihydrothieno[3,2-c]pyridine-5(4H)-acetate**

This compound is known.\(^{[18]}\) Column chromatography using hexane:ethyl acetate = 6:1. Product was achieved with 97.8 mg (61 % of yield).

\(^1\)H NMR (500 Hz, CDCl\(_3\)) \(\delta\) 7.70 (dd, \(J = 8.0, 1.0\) Hz, 1H), 7.41 (dd, \(J = 8.0, 1.6\) Hz, 1H), 7.25 (m, 2H), 7.00 (d, \(J = 4.0\) Hz, 1H), 6.61 (d, \(J = 4.0\) Hz, 1H), 4.85 (s, 1H), 3.70 (d, \(J = 12.0\) Hz, 1H), 3.64 (s, 3H), 3.57 (d, \(J = 12.0\) Hz, 1H), 2.82 (s, 4H);

**1-Phenyl-2-(pyrrolidin-1-yl)pentan-1-one (α-pyrrolidinovalerophenone)**
This compound is known.\textsuperscript{[19]} Column chromatography using hexane:ethyl acetate = 4:1. Product was achieved with 94.6 mg (82\% of yield).

$^1$H NMR (500 Hz, CDCl$_3$) $\delta$ 8.15 – 8.05 (m, 2H), 7.60 – 7.55 (m, 1H), 7.47 – 7.41 (m, 2H), 3.94 (q, $J$ = 4.8 Hz, 1H), 2.75 – 2.71 (m, 4H), 1.95 – 1.82 (m, 2H), 1.79 – 1.65 (m, 4H), 1.36 – 1.19 (m, 2H), 0.87 (t, $J$ = 7.8 Hz, 3H).

\textbf{2-(1-Pyrroloidinyl)-3-pentanone}

This compound is known.\textsuperscript{[20]} Column chromatography using hexane:ethyl acetate = 7:1. Product was achieved with 40.1 mg (51\% of yield).

$^1$H NMR (500 Hz, CDCl$_3$) $\delta$ 2.97 (q, 1H, $J$ = 7.0 Hz), 2.68 - 2.40 (m, 6H), 1.91-1.72 (m, 4H), 1.23 (d, 3H, $J$ = 7.0 Hz), 1.03 (t, 3H, $J$ = 7.1 Hz).

\textbf{1-(Diethylamino)-1-phenyl-2-propanone}

This compound is known.\textsuperscript{[21]} Column chromatography using hexane:ethyl acetate = 5:1. Product was achieved with 63.5 mg (62\% of yield).

$^1$H NMR (500 Hz, CDCl$_3$) $\delta$ 8.05 – 7.91 (m, 2H), 7.81 – 7.35 (m, 3H), 3.36 (q, $J$ = 7.1 Hz, 2H), 3.28 (q, $J$ = 7.2 Hz, 2H), 2.08 (s, 3H), 1.16 (t, $J$ = 7.1 Hz), 1.10 (t, $J$ = 7.1 Hz, 3H).
Section SX. References


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