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Introduction

The discovery of new energy resources, in particular natural gas in the form of shale gas, will facilitate the development of natural gas as a viable alternative energy source in the near future. Abundant resources and the capability to produce shale gas commercially on a large scale make natural gas particularly appealing as a new fuel. To accelerate the switching of fuel from coal/petroleum to natural gas, it is necessary to establish an infrastructure and to develop suitable materials for the storage

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A porous metal-organic framework with an elongated anthracene derivative exhibiting a high working capacity for the storage of methane[†]

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We have developed a new porous metal–organic framework (MOF) (UTSA-80) with an elongated anthracene derivative as a linker. The activated UTSA-80a has a pore volume of 1.03 cm³ g⁻¹ and a gravimetric Brunauer–Emmett–Teller surface area of *ca.* 2280 m² g⁻¹, higher than those of PCN-14. The volumetric methane storage capacity of UTSA-80a at 35 bar and 298 K is 192 cm³ (STP) cm⁻³, which makes it one of the few porous MOFs with a storage capacity >190 cm³ (STP) cm⁻³ at 35 bar. The volumetric uptake of methane by UTSA-80a reaches 233 cm³ (STP) cm⁻³ at room temperature and 65 bar; this is 88.6% of the new volumetric target of the US Department of Energy if the packing density loss is ignored. This capacity is comparable with that of PCN-14. However, as a result of the lower methane uptake of UTSA-80a at 5 bar, it has a much higher methane storage working capacity (deliverable amount of methane between 65 and 5 bar) of 174 cm³ (STP) cm⁻³ achieved by HKUST-1, suggesting that it is a promising material for methane storage in transport applications. Such an exceptionally high working capacity can probably be attributed to the elongated anthracene derivative used as a linker within UTSA-80a, which adjusts the pore sizes/cages and interactions with the methane molecules to optimize the methane working capacity.

and transportation of natural gas. Currently, compressed natural gas, stored as a supercritical fluid at room temperature and 200–300 bar in steel cylinders, is used in vehicles powered by natural gas; however, the large tank size required restricts the application of compressed natural gas to larger vehicles such as trucks. Adsorbed natural gas is better suited for everyday use in cars, in terms of both cost and safety.

Porous metal-organic frameworks (MOFs), a new type of porous crystal material,¹ have shown great potential for the storage of gases such as hydrogen,^{2,3} methane (the main compound in natural gas)4-7 and carbon dioxide8,9 due to their high surface area and porosity,¹⁰ tunable pore size and shape^{1h,11} and functional sites which can be readily immobilized to optimize their storage capacity.12 The utilization of MOFs for methane storage in particular has received increasing attention in recent years.4-6 BASF has also demonstrated model vehicles fueled by natural gas using BASF MOF materials.13 Therefore it is important to develop new adsorbents with high methane storage capacities for natural gas fuel systems used in vehicles. Recently, in order to promote research on methane storage, the US Department of Energy (DOE) has begun a new methane storage program (MOVE) with the ambitious target of a volumetric storage capacity of 350 cm³ (STP) cm⁻³ (adsorbent) and a gravimetric storage capacity of 0.5 g(CH₄) g⁻¹ (adsorbent) at room temperature.¹⁴ If we ignore the loss of adsorbent material

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due to packing (*i.e.* we assume that the gas tank is filled with a large single crystal of adsorbent),¹⁵ the new volumetric target also demands 263 cm³ (STP) cm⁻³ (adsorbent).

Although the new DOE MOVE program has promoted research targeting adsorbents for methane storage, it is still challenging to achieve these DOE targets. Several groups have recently reported that HKUST-1 shows an exceptionally high volumetric methane storage capacity of 267 cm^3 (STP) cm^{-3} and a working capacity of 190 cm^3 (STP) cm^{-3} at room temperature and 65 bar,¹⁶ which is the first MOF material with a volumetric methane storage capacity that meets the new DOE target if the packing density loss is ignored. Our own recent work highlighted a novel NbO MOF, UTSA-76a, with dynamic pyrimidine groups, which showed a record methane storage working capacity of \sim 200 cm³ (STP) cm⁻³ at 298 K and 65 bar.¹⁷ These results encouraged us to develop further MOFs to achieve the DOE target. It is well known that PCN-14, reported by Ma et al.,18 shows an exceptional methane storage capacity of 230 cm^3 (STP) cm^{-3} at 290 K and 35 bar. This high methane storage capacity is mainly attributed to the open metal sites and small cages within PCN-14 due to the polycyclic anthracene derivative linker.4ª Although the methane uptake of PCN-14 reported by two other groups4b,16 was found to be slightly lower at 195–202 cm³ (STP) cm⁻³ at 35 bar and 230–239 cm³ (STP) cm⁻³ at 65 bar, respectively (both studies were carried out at 298 K), their results further confirmed its high methane storage capacity. In addition, to optimize the volumetric methane storage capacity, an ideal MOF should have balanced porosities and framework densities and suitable pore sizes/cages for the recognition of methane molecules.16-21

To optimize the pore sizes/cages to enhance the methane storage capacity, we developed a new ligand with an elongated anthracene derivative by adding a triple-bond spacer (Scheme 1) to construct its copper MOF $[Cu_2L(H_2O)_2]$ (UTSA-80). As expected, the activated UTSA-80a has a larger Brunauer-Emmett-Teller (BET) surface area and pore volume than PCN-14 because of the elongated anthracene linker. More importantly, UTSA-80a shows a high volumetric methane storage capacity of 233 cm^3 (STP) cm^{-3} , which is comparable with that of PCN-14 [230-239 cm³ (STP) cm⁻³]. However, to our surprise, UTSA-80a has a much higher methane storage working capacity [174 cm³ (STP) cm⁻³] than PCN-14 [157-160 cm³ (STP) cm⁻³],^{4b,16} comparable with HKUST-1 [190 cm³ (STP) cm⁻³]. The working capacity (also called the deliverable capacity), which determines the driving range of natural gas vehicles, is more important than the total storage capacity values. Our results suggest that

Scheme 1 Schematic structure of the organic ligands serving as linkers in (a) PCN-14 and (b) UTSA-80.

the new MOF UTSA-80a reported here is a very promising material for methane storage applications.

Experimental

Materials

All reagents and solvents were commercially available and used without further purification. Dimethyl 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) isophthalate²² and 1,3-diethylcarboxy-late-4-ethynylbenzene were prepared according to previously published procedures.^{10a}

General procedures

¹H-NMR spectra were recorded on a Varian Mercury 500 MHz spectrometer using tetramethylsilane as an internal standard. The coupling constants are reported in Hz. FTIR spectra were performed on a Bruker Vector 22 spectrometer at room temperature. The elemental analyses were performed with a Perkin-Elmer 240 CHN analyzer from Galbraith Laboratories (Knoxville). Thermogravimetric analysis (TGA) was carried out using a Shimadzu TGA-50 analyzer under a nitrogen atmosphere, with a heating rate of 5 °C min⁻¹. Powder X-ray diffraction (PXRD) patterns were measured by a Rigaku Ultima IV diffractometer operated at 40 kV and 44 mA with a scan rate of 1.0 deg min⁻¹. A Micromeritics ASAP 2020 surface area analyzer was used to measure gas adsorption isotherms. To remove all the guest solvents in the framework, the fresh sample of UTSA-80 was guest-exchanged with dry acetone at least 10 times, filtered and degassed at room temperature for 1 day and then at 393 K for another 5 h until the outgas rate was 5 μ mHg min⁻¹ before the measurements were made. The activated sample of UTSA-80a was maintained at 77 K with liquid nitrogen. High-pressure CH_4 sorption isotherms were measured using a Sieverts-type apparatus. A detailed description of the experimental setup, calibration and the isotherm has been published previously.16,23

Synthesis of H₄L

As shown in Scheme 2, the asymmetric elongated anthracene linker H₄L was synthesized by a two-step reaction procedure.



Scheme 2 Synthesis procedure for the organic linker H_4L .

9,10-Dibromoanthracene reacts with 1 equiv. 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) isophthalate by Suzuki crosscoupling to give compound 1, following by reaction with 1 eauiv. 1,3-diethylcarboxylate-4-ethynylbenzene to give compound 2. Compound 2 (980 mg, 1.6 mmol) was suspended in 50 mL THF and then a 2 M KOH aqueous solution (75 mL) was added. The mixture was stirred under reflux overnight until it became clear. The THF was then removed under reduced pressure and dilute HCl was added to the remaining aqueous solution to acidify to pH 2. The precipitate was collected by filtration, washed several times with water and then dried to give a pale yellow powder. Yield: 810 mg (95%). ¹H-NMR (500 MHz, d_6 -DMSO, ppm): $\delta = 13.45$ (s, 4H), 8.65 (d, J = 8.8 Hz, 2H), 8.58 (s, 1H), 8.44 (s, 2H), 8.41 (s, 1H), 8.03 (s, 2H), 7.66 (t, J = 6.6 Hz, 2H), 7.42 (t, J = 7.8 Hz, 4H).

Synthesis of UTSA-80

A mixture of the organic linker H₄L (8.0 mg, 0.015 mmol) and $Cu(NO_3)_2 \cdot 2.5H_2O$ (20.0 mg, 0.086 mmol) was dissolved in 3.5 mL of mixed solvent (DMF/H₂O, 3 mL/0.5 mL) in a screw-capped vial (20 mL), to which three drops of HBF₄ were added. The vial was capped and heated in an oven at 60 °C for 24 h. Green block crystals were obtained by filtration and washed with DMF several times to give UTSA-80 at a yield of 65%. UTSA-80 has a best formula of $[Cu_2L(H_2O)_2] \cdot 4DMF \cdot 3H_2O$, which was obtained based on single-crystal X-ray structural determination, elemental analysis and TGA. Anal. calculated for $C_{44}H_{52}N_4O_{17}Cu_2$: C, 51.01; H, 5.06; N, 5.41%. Anal. found: C, 49.98; H, 5.14; N, 5.48%. TGA data for loss of 4DMF and 5H₂O: calculated, 37.00%; found, 37.75%. IR (neat, cm⁻¹): 1629, 1564, 1427, 1366, 1248, 1099, 766, 717, 656.

Single-crystal X-ray crystallography

Crystal data were collected on an Agilent Supernova CCD diffractometer equipped with graphite monochromatic enhanced CuK α radiation ($\lambda = 1.54184$ Å) at 100 K. The data sets were corrected by empirical absorption correction using spherical harmonics, implemented in the SCALE3 ABSPACK scaling algorithm. The structure was solved by direct methods using SIR92 and refined by full matrix least-squares methods with the SHELX-2014 program package.24 The central anthracene ring and triple bond of the organic ligand are disordered and the solvent molecules in the compound are highly disordered. The SQUEEZE subroutine of the PLATON software suite was used to remove the scattering from the highly disordered guest molecules.25 The resulting new files were used to further refine the structures. The H atoms on the C atoms were generated geometrically. The crystal data are summarized in Table S1.†

Results and discussion

The formula of UTSA-80 was determined as $[Cu_2L(H_2O)_2]$ · 4DMF·3H₂O by single-crystal XRD analysis, TGA and elemental analysis. The phase purity of the bulk material was also confirmed by PXRD (Fig. S4[†]). Single-crystal XRD analysis

revealed that UTSA-80 crystallizes in the $R\bar{3}m$ space group, exactly the same structure as PCN-14.18 The framework nodes in UTSA-80 consist of paddlewheel dinuclear Cu₂(COO)₄ secondary building units (SBUs), bridged by the carboxylates of L^{4-} to form a 3D NbO-type structure. There are two types of cage in the resulting framework. One cage (Fig. 1; green) about 12.1 Å in diameter is composed of 12 ligands connecting six paddlewheel SBUs, which is much larger than that of PCN-14 (10.0 Å). Another large irregular elongated cage of about 14.4 \times 28.8 Å consists of six ligands connecting 12 paddlewheel SBUs, which is remarkably elongated by these six ligands. This large irregular cage is considered to consist of one squashed cuboctahedral cage at the center (blue) and two extraordinarily small cages of \sim 3 Å on the top and bottom (pink); these two types of cage are separated by two series of three anthracenyl rings in close contact. These elongated pores/cage sizes and the extraordinarily small cages may play an important part in optimizing the methane storage capacity.

To establish the permanent porosity, acetone-exchanged UTSA-80 was activated at room temperature for 24 h and then heated at 100 °C under high vacuum to yield the activated UTSA-80a. The porosity was characterized by nitrogen sorption at 77 K. As shown in Fig. 2, the N₂ isotherm showed a reversible type I sorption behavior, characteristic of microporous materials, with an N₂ uptake of 664.4 cm³ g⁻¹. The BET surface area of UTSA-80a is 2280 m² g⁻¹ and the pore volume calculated from the maximum amount of N₂ adsorbed is 1.03 cm³ g⁻¹; these are much higher than those of PCN-14 (Table 1).

The high methane storage capacity of PCN-14 prompted us to examine the capacity of UTSA-80a. Temperature-dependent total methane sorption isotherms for UTSA-80a are shown in Fig. 3. At 125 K and 2.2 bar, the absolute methane adsorption of UTSA-80a reaches 379 cm³ (STP) cm⁻³, which is 64.1% of that of liquid methane (0.423 g cm⁻³) at 113 K. The pore volume of UTSA-80a derived from the saturated methane uptake at 125 K is 1.030 cm³ g⁻¹, in good agreement with the nitrogen pore volume. At room temperature and 35 bar, UTSA-80a shows a high volumetric methane storage capacity of 192 cm³ (STP)



Fig. 1 X-ray crystal structures of PCN-14 and UTSA-80 indicating three types of cages, respectively.



Fig. 2 N₂ sorption isotherms of UTSA-80a at 77 K. Closed symbols, adsorption; open symbols, desorption.

cm⁻³, surpassing the DOE's previous target of 180 cm³ (STP) cm⁻³ when the packing density loss is ignored. This capacity is comparable with that of PCN-14 [195 cm³ (STP) cm⁻³] and UTSA-20 [195 cm³ (STP) cm⁻³], but is higher than NU-135 [187 cm³ (STP) cm⁻³], NU-125 [182 cm³ (STP) cm⁻³] and NU-111 [138 cm³ (STP) cm⁻³]. In fact, UTSA-80a is one of the very few porous MOFs with a methane storage capacity >190 cm³ (STP) cm⁻³ at 35 bar, suggesting that it is a promising adsorbent for practical methane storage.

When the pressure increases to 65 bar, the volumetric methane storage capacity of UTSA-80a reaches 233 cm³ (STP) cm⁻³, which is 88.6% of the new DOE volumetric target if the packing density loss is ignored. This value is comparable with PCN-14 [230–239 cm³ (STP) cm⁻³] and other promising MOFs for volumetric methane storage (Table 1). However, UTSA-80a has a much higher methane storage density of 0.240 g(CH₄) g⁻¹ than the 0.197–0.204 g(CH₄) g⁻¹ of PCN-14. This is because UTSA-80a has a larger pore volume and a lower framework density than PCN-14 due to its longer organic linker. In addition, the methane storage density of UTSA-80a is higher than



Fig. 3 High-pressure CH_4 total adsorption isotherms measured over a broad range of temperatures. Closed symbols, adsorption; open symbols, desorption.

most of the other promising MOFs, *e.g.* HKUST-1 [0.216 g(CH₄) g^{-1}], UTSA-20 [0.181 g(CH₄) g^{-1}], Ni-MOF-74 (ref. 16) [0.148 g(CH₄) g^{-1}] and NU-135 [0.219 g(CH₄) g^{-1}].

The working capacity for methane storage is more important than the total volumetric and gravimetric methane uptake; this determines the driving range of vehicles powered by natural gas. Although the volumetric methane uptake of UTSA-80a at room temperature and 65 bar shows no significant increase compared with PCN-14, it has a much higher methane storage working capacity of 174 cm³ (STP) cm⁻³ than PCN-14 [157-160 cm³ (STP) cm⁻³] (Fig. 4). This higher working capacity of UTSA-80a is mainly due to its much lower methane uptake than PCN-14 at 5 bar, attributed to the larger pores and slightly weaker methane binding in UTSA-80a. In fact, this value is slightly lower than HKUST-1 [190 cm³ (STP) cm⁻³], but higher than that of UTSA-20 and NU-135. UTSA-80a also has a methane working capacity density of 0.178 g(CH₄) g^{-1} (adsorbent), which is much higher than that of 0.154 g(CH₄) g^{-1} (adsorbent) in HKUST-1 and 0.136–0.138 g(CH₄) g^{-1} (adsorbent) in PCN-14. Given the fact that the working capacity is more important than the total

Table 1	Comparison of some micro	porous MOFs for high-pressure	e methane storage at room te	mperature and 65 (35) bar

	a d	$V_{\rm p}^{\ b} \ ({\rm cm}^3 {\rm g}^{-1})$	$D_{\rm c}^{\ c}$ (g cm ⁻³)	Total uptake ^d at 65 bar (35 bar)		Working capacity ^e at 65 bar (35 bar)			
MOFs	S_{BET}^{a} $(\text{m}^2 \text{ g}^{-1})$			$(g g^{-1})$	$(cm^{3} cm^{-3})$	Density	$(g g^{-1})$	$(\mathrm{cm}^3 \mathrm{cm}^{-3})$	(kJ mol ^{-1})
UTSA-80a	2280	1.03	0.694	0.240 (0.198)	233 (192)	0.167 (0.138)	0.178 (0.136)	174 (133)	15.95
UTSA-76 (ref. 17)	2820	1.09	0.699	0.263 (0.216)	257 (211)	0.184 (0.151)	0.201 (0.154)	197 (151)	15.44
HKUST-1 (ref. 16)	1850	0.78	0.883	0.216 (0.184)	267 (227)	0.191 (0.162)	0.154 (0.122)	190 (150)	17.0
NOTT-101(ref. 4h)	2805	1.08	0.684	0.247 (0.202)	237 (194)	0.169 (0.138)	0.189 (0.144)	181 (138)	15.49
NU-125 (ref. 15)	3120	1.29	0.578	0.287 (0.225)	232 (182)	0.166 (0.130)	0.227 (0.165)	183 (133)	15.1
NU-135 (ref. 7a)	2530	1.02	0.751	0.219 (0.178)	230 (187)	0.164 (0.134)	0.178 (0.133)	170 (127)	16.6
PCN-14 (ref. 16)	2000	0.85	0.829	0.197 (0.167)	230 (195)	0.164 (0.139)	0.136 (0.106)	157 (122)	18.7
PCN-14 (ref. 4b)	1984	0.83	0.829	0.204 (0.172)	239 (202)	0.170 (0.143)	0.138 (0.108)	160 (125)	17.6
UTSA-20 (ref. 16)	1620	0.66	0.909	0.181 (0.145)	230 (184)	0.164 (0.131)	0.134 (0.098)	170 (124)	18.2
NU-111 (ref. 15)	4930	2.09	0.409	0.360 (0.241)	206 (138)	0.147 (0.098)	0.313 (0.194)	179 (111)	14.2

^{*a*} BET surface areas calculated from N₂ isotherms at 77 K. ^{*b*} Pore volumes calculated from the maximum amounts of N₂ adsorbed. ^{*c*} Framework densities without guest molecules and terminal waters. ^{*d*} At 298 K and 65 (35) bar. ^{*e*} Defined as the difference of methane uptake between 65 (35) bar and 5 bar.



Fig. 4 Schematic diagram of the determination of the deliverable amount of UTSA-80a, defined as the difference in uptake between 65 and 5 bar.

storage capacity values, our results suggest that UTSA-80a is superior to PCN-14 for practical methane storage applications.

The finding that UTSA-80a has a much higher methane storage density and working capacity than PCN-14 is very encouraging. It indicates that the elongation of the organic linker in MOFs may remarkably enhance their methane storage performance. To obtain a better insight into the origin of the enhanced methane working capacity by the elongation of the linker in UTSA-80a, we examined the isosteric heats of adsorption (Q_{st}) from the temperature-dependent isotherms shown in Fig. S8,[†] calculated using the viral method. The initial Q_{st} value for CH₄ adsorption in UTSA-80a is \sim 15.95 kJ mol⁻¹ which is slightly lower than that of PCN-14 (17.6–18.7 kJ mol⁻¹). Moreover, the Q_{st} for CH₄ adsorption gradually decreases with the CH_4 loading to 6 mmol g⁻¹. These two features indicate that there is weaker methane binding in UTSA-80a compared with PCN-14 at low methane loadings, leading to the lower methane uptake of UTSA-80a at 5 bar and the higher working capacity. This lower CH₄ uptake below 5 bar is also consistent with the fact that UTSA-80a has larger pores and a higher pore volume than PCN-14 due to the elongated linker. When the CH₄ loading increased further, the $Q_{\rm st}$ began to increase. We speculate that this increase is due to attractive CH₄-CH₄ interactions, which dominate the uptake of methane at higher pressures.

Conclusions

In summary, we developed a new porous MOF UTSA-80a with a high methane storage capacity using an elongated anthracene derivative as a linker. UTSA-80a has a greatly enhanced surface area and pore volume compared with PCN-14. It also has a comparable volumetric methane storage capacity to PCN-14 at 298 K and 65 bar, and is one of the most promising MOFs for methane storage. Most importantly, compared to PCN-14, UTSA-80a has a much higher methane storage working capacity of 174 cm³ (STP) cm⁻³ as well as a larger storage density of 0.240 g g⁻¹, suggesting that it is a promising material for methane storage in transport applications. We also found that the elongated pores/cages within UTSA-80a result in a much lower

methane uptake below 5 bar compared with PCN-14, while the uptake at 65 bar is comparable, thus leading to a much higher working capacity. These results suggest a promising new route to optimizing the methane working capacity of MOF, using elongating linkers with suitable pore sizes/cages to improve the methane storage working capacity.

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- 15 If one sample has a large enough single crystal which can directly fill the storage tank, it does not have the packing loss issue, then a 263 cm³ (STP) cm⁻³ storage capacity can meet the target. If we have a powder sample, and

assuming the material subjects to 25% packing loss of density, then the storage capacity of the material itself (each crystal) needs to be 350 cm³ (STP) cm⁻³. Current emphasis is on discovering promising MOFs with higher methane storage and working capacities. Future close collaboration with industrial partners on examining some practical issues including packing densities will be required before some MOFs will be eventually utilized for their dairy usages of methane storage.

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Supporting Information

A Porous Metal-Organic Framework with an Elongated Anthracene Derivative Exhibiting High Methane Working

Capacity

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Synthesis of dimethyl 5-(10-bromoanthracen-9-yl)isophthalate (1).

9,10-Dibromoanthracene (3.3 g, 10 mmol), 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) isophthalate (1.6 g, 5 mmol), K₃PO₄ (2.55g, 12 mmol) and tetrakis(triphenylphosphine) palladium(0) (0.3 g, 0.26 mmol) were dissolved in dry 1,4-dioxane (60 mL) under N₂ atmosphere. The mixture was stirred at 80 °C for two days. After removal of the solvent under reduced pressure, the residue was extracted with dichloromethane, washed with brine, dried over anhydrous MgSO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel using CH₂Cl₂/hexane (1/2, v/v) as eluent to give 1.35 g of the product. Yield: 60 %. ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.91 (t, *J* = 1.6 Hz, 1H), 8.65 (d, *J* = 8.9 Hz, 2H), 8.30 (t, *J* = 1.5 Hz, 2H), 7.62 (dd, *J*₁ = 8.9 Hz, *J*₂= 3.2 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 7.41 (dd, *J*₁ = 9.3 Hz, *J*₂ = 3.3 Hz, 2H), 3.97 (s, 6H).

Synthesis of diethyl 5-((10-(3,5-bis(methoxycarbonyl)phenyl)anthracen-9-yl)ethynyl) isophthalate (2)

Compound 1 (1.3 g, 2.9 mmol) and 1,3-diethylcarboxylate-4-ethynylbenzene (0.73 g, 3.0 mmol) were dissolved in THF/diisopropylamine (v/v = 1/1, 120 mL) at room temperature under N₂ atmosphere. To the solution were added Pd(PPh₃)₄ (60 mg) and CuI (5 mg). The reaction mixture was stirred for 24 h at room temperature. The reaction was monitored by TLC. Upon completion, the solution was first filtered and the filtrate was then concentrated under reduced pressure. The product was purified by silica gel column chromatography using dichloromethane-hexane (v/v = 1:1) as eluent. Yield: 55% (980 mg). ¹H NMR (500 MHz, CDCl₃, ppm): δ 8.92 (t, *J* = 1.6 Hz, 1H), 8.77 (d, *J* = 8.5 Hz, 2H), 8.73 (t, *J* = 2.0 Hz, 1H), 8.63 (d, *J* = 1.7 Hz, 2H), 8.33 (d, *J* = 1.5 Hz, 2H), 7.67 (dd, *J*₁ = 8.7 Hz, *J*₂ = 3.2 Hz, 2H), 7.56 (d, *J* = 8.8 Hz, 2H), 7.45 (dd, *J*₁ = 8.8 Hz, *J*₂ = 3.2 Hz, 2H), 4.51 (q, *J* = 7.1 Hz, 4H), 3.98 (s, 6H), 1.50 (t, *J* = 7.1 Hz, 6H).



Figure S1. ¹H (CDCl₃, 500MHz) spectra of compound 1.



Figure S2. ¹H (CDCl₃, 500MHz) spectra of compound 2.



Figure S3. ¹H (DMSO-d₆, 500MHz) spectra of the ligand H₄L.



Figure S4. PXRD patterns of as-synthesized UTSA-80 (red) and activated UTSA-80a (blue) along with the simulated XRD pattern from the single-crystal X-ray structure (black).



Figure S5. TGA curves of as-synthesized UTSA-80.



Figure S6. X-ray single crystal structure of UTSA-80: (a) the 4-connected $Cu_2(O_2CR)_4$ paddlewheel unit; (b) one tetracarboxylate ligand connects with four $Cu_2(O_2CR)_4$ clusters. Turquoise, red, and gray spheres represent Cu, O, and C atoms, respectively.



Figure S7. Excess volumetric high-pressure methane sorption isotherms of UTSA-80a at different temperatures. Filled and open symbols represent adsorption and desorption data, respectively.



Figure S8. Isosteric heats of adsorption (Q_{st}) for CH₄ as a function of gas loading in mmol(gas) g⁻¹(UTSA-80a), calculated using the virial method.

Table S1. Crystallographic data and structure refinement results for UTSA-80 (from single-crystal X-ray diffraction analysis on the as-synthesized sample).

	UTSA-80			
Formula	$C_{48} \ H_{24} \ Cu_3 \ O_{15}$			
Formula weight	1031.29			
Temperature/K	100.00(19)			
Crystal system	Trigonal			
Space group	R-3m			
<i>a</i> , <i>b</i> (Å)	18.4951(7)			
<i>c</i> (Å)	46.337(3)			
α (°)	90			
β (°)	90			
γ (°)	120			
$V(Å^3)$	13726.8(13)			
Ζ	6			
$D_{\text{calcd}} (\text{g cm}^{-3})$	0.749			
$\mu (\mathrm{mm}^{-1})$	1.106			
<i>F</i> (000)	3114			
Crystal size/mm ³	$0.2\times0.16\times0.10$			
GOF	1.202			
R_{int}	0.0257			
$R_{I}, wR_{2}[I \ge 2\sigma(I)]$	0.1073, 0.2991			
R_1 , wR_2 [all data]	0.1290, 0.3276			
Largest diff. peak and hole (e Å ⁻³)	0.946 and -0.766			

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