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Reversible carbon monoxide binding at copper(I) P–S–X (X = N, O) coordination polymers

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ABSTRACT

Reaction of $Ph_2PCH_2CH_2S(2\text{-}C_6H_4NH_2)$ (**1**) or $Ph_2PCH_2CH_2S(2\text{-}C_6H_4OMe)$ (**2**) with CuCl and $AgSbF_6$ in the presence of THF affords $\{[Cu(\mathbf{1})THF](SbF_6)\}_n$ (**3**) and $\{[Cu(\mathbf{2})THF](SbF_6)\}_n$ (**4**). In the solid state, THF-inclusive **3** exists as a one dimensional zigzag coordination polymer in which each P–S–N ligand is connected to two copper(I)-centres. Both **3** and **4** are able to bind CO reversibly in solution or can be desolvated at 110 °C *in vacuo* to give $\{[Cu(\mathbf{1})](SbF_6)\}_n$ (**7**) and $\{[Cu(\mathbf{2})](SbF_6)\}_n$ (**8**). These solvent-free compounds have been found to bind carbon monoxide reversibly over multiple cycles in solution and in the solid state, under mild conditions.

1. Introduction

Although metal compounds that can reversibly bind carbon monoxide have long been known, several new and interesting examples have been published in recent years [1–5]. The ongoing interest in CO-coordinating agents is driven not only by the ubiquity of CO as a ligand in organometallic chemistry but also by the possibility of their varied and wide-ranging application as CO sources/sensors, molecular switches and in biologically related systems for example. Copper(I) species in particular have a rich history in the reversible binding of CO and are used in this context in the industrially important COSORB process [6–10].

More recently, our group has reported the use of a Tp*Cu complex (Tp* = tris(3,5-dimethylpyrazolyl)borate) in the pre-concentration of ^{11}CO for subsequent release to palladium catalysed coupling reactions and use in the area of positron emission tomography [11]. We have also described reversible CO binding at several first-row transition metal complexes of a new P–S–N ligand [12]. In extension of this work, we have chosen to look at copper(I) complexes of potentially tridentate ligands containing hard and soft donor functionalities.

Previously reported examples of copper(I) CO binding complexes have been based on a variety of ligand types including those containing mixed donor ligands such as Schiff bases [13] or tripodal bis(imidazole)thioethers [14]. In general, when ligands inclusive of thioether substituents are coordinated to Cu^I, the resulting complexes often adopt multimeric structures, may contain unsaturated or weakly coordinated metal centres and frequently exhibit reactivity toward small molecules [15–19]. The formation of unsaturated, yet structurally stable, Cu^I species can allow for reversible CO binding in the solid state and there are examples of solid supported copper complexes for which this is true, however, unsupported metal complexes that bind CO in solution often do not bind CO as crystalline solids due to lattice constraints [20,21]. We report here a rare example of a copper(I) coordination polymer containing a P–S–N ligand together with a P–S–O ligand analogue and describe their behaviour toward CO in solution and in the solid state.

2. Results and discussion

$Ph_2PCH_2CH_2S(2\text{-}C_6H_4NH_2)$ (**1**) and $Ph_2PCH_2CH_2S(2\text{-}C_6H_4OMe)$ (**2**) (Fig. 1) were synthesised by deprotontion of the relevant C2-substituted arylthiol precursors followed by reaction with (2-chloroethyl)diphenyl-phosphine [12,22,23]. A mixture of one equivalent of **1** or **2** and CuCl was reacted with $AgSbF_6$ in the presence of THF to give species of the formulation $\{[Cu(\mathbf{1})THF](SbF_6)\}_n$ (**3**) and $\{[Cu(\mathbf{2})THF](SbF_6)\}_n$ (**4**), respectively (Fig. 2).

The ^{31}P NMR spectra of these complexes revealed broad singlets at chemical shifts located downfield with respect to the free ligands (**3**: −6.7 ppm, **4**: −4.1 ppm) and the presence of a single molecule of THF per Cu(ligand) unit was confirmed by analysis of the relevant

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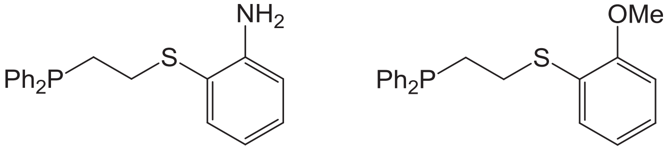


1 **2**

Fig. 1. The P–S–N and P–S–O ligands **1** and **2**.

^{1}H NMR spectra. Solid samples of **3** and **4** could be dried *in vacuo* at room temperature and stored under N_2 for weeks without loss of THF. The molecular structure of **3** is shown in Fig. 3 and was determined by single crystal X-ray diffraction of crystals grown from 1:1 THF/hexane at room temperature.

In contrast to the facial coordination modes seen in the recently reported iron, cobalt and nickel complexes of **1** [12], here in the copper species the tridentate P–S–N ligand acts in a bridging manner, the nitrogen and phosphorus donor atoms coordinating to one copper centre whilst the sulphur donor coordinates to an adjacent copper atom, forming a cationic chain polymer that extends along the crystallographic *c* axis direction (Fig. 3). The orientation of the ligands along the chain puts the N(1) hydrogen atoms all on one "side" of the chain (the back as viewed in Fig. 3), and the SbF_6 anions serve to bridge adjacent, glide-related, N(1) centres via N–H···F hydrogen bonds. Unfortunately, the anions were found to be disordered (see supporting information) and so the geometries (which have been calculated using the major occupancy orientation) have to be considered as indicative only; the [N···F], [H···F] distances (Å) and the [N–H···F] angle (°) are 3.011(6), 2.13, 165 and 3.120(8), 2.41, 136. There are no N–H···F links between chains. The geometry at the copper centre is severely distorted tetrahedral with angles in the range 96.31(9) to 122.35(3)°, and the eight-membered chelate ring has a twist-boat-chair conformation [24].

Although crystallographic evidence was not obtained for **4**, it is likely that this compound has a solid-state structure analogous to **3**. This is inferred by the strong similarities observed in their NMR spectra and the closely matching CO binding properties of both compounds, although no further supporting evidence was obtained.

By bubbling CO through DCM solutions of **3** or **4** the new copper(I) carbonyl species {[Cu(**1**)CO](SbF$_6$)}$_n$ (**5**) and {[Cu(**2**)CO](SbF$_6$)}$_n$ (**6**) were formed. The generation of compounds containing terminal Cu–CO bonds was confirmed by infrared spectroscopy (**5**: 2091 cm^{-1}, **6**: 2114 cm^{-1}) and ^{1}H NMR spectroscopy in CD_2Cl_2 revealed the presence of free THF indicating that it is replaced in its metal binding sites by CO. Release of CO was achieved by bubbling N_2 through DCM solutions of the respective carbonyl complexes to give {[Cu(**1**)](SbF$_6$)}$_n$ (**7**) and {[Cu(**2**)](SbF$_6$)}$_n$ (**8**). Subsequent reaction with CO resulted in complete reconversion to **5** and **6** and the reversible gas binding process between these species was found to be reproducible over at least 5 cycles. Alternatively, addition of an equal volume of THF to DCM solutions of **5** or **6** followed by application of an N_2 stream led to the reformation of the parent complexes **3** and **4** (Scheme 1).

$$\mathbf{1} + \mathrm{CuCl} + \mathrm{AgSbF_6} \xrightarrow{\mathrm{THF}} \{[\mathrm{Cu}(\mathbf{1})\mathrm{THF}](\mathrm{SbF_6})\}_n + \mathrm{AgCl}$$

3

$$\mathbf{2} + \mathrm{CuCl} + \mathrm{AgSbF_6} \xrightarrow{\mathrm{THF}} \{[\mathrm{Cu}(\mathbf{2})\mathrm{THF}](\mathrm{SbF_6})\}_n + \mathrm{AgCl}$$

4

Fig. 2. Preparation of **3** and **4**.

In a separate experiment, it was possible to directly desolvate **3** or **4** by heating solid samples at 110 °C *in vacuo*. Complete removal of THF to give **7** and **8**, respectively, was achieved in 2 h under these conditions. Other than the absence of THF, the ^{1}H NMR spectra showed little change in the chemical shifts and patterns of diagnostic resonances compared with data recorded for **3** and **4** and matched those obtained in the solution studies reported above, indicating that this process occurs without any significant decomposition. The singlet peaks found in the ^{31}P NMR spectra (**7**: −5.3 ppm, **8**: −3.4 ppm) were also subtly shifted from chemical shift values obtained for the THF-inclusive complexes and this possibly suggests that no major structural rearrangements take place upon desolvation. Elemental analysis conducted on **7** and **8** confirm their THF-free assignment.

To assess the ability of these compounds to bind CO in the solid state, a stream of CO (1 atm) was passed over finely powdered samples for 10 min. This resulted in the complete conversion of **7** and **8** to the carbonyl species **5** and **6**, respectively, which were identified by elemental analysis of the solids and NMR and IR spectroscopy in CD_2Cl_2. Solid samples of **5** and **6** were stable in a stream of N_2 (1 atm), however, CO could be fully removed under vacuum conditions (1×10^{-3} mbar) at room temperature. Reversible binding of carbon monoxide was recorded for both compounds in the solid state over at least 5 cycles. Bubbling N_2 through THF solutions of **5** and **6** obtained via this route furnished the parent complexes **3** and **4**. A summary of the relationship between compounds **3**–**8** is shown in Scheme 1.

3. Conclusion

Several copper(I) species containing P–S–X (*X* = N, O) heterodonor ligands **1** and **2** have been synthesised and characterised. Interestingly, the molecular structure of THF-inclusive **3** has revealed it to be a one dimensional zigzag coordination polymer in the solid state. In solution, compounds **3** and **4** can be converted, in a facile and reversible manner, to the carbonyl species **5** and **6** which can in turn undergo decarbonylation to give **7** and **8**. Alternatively, the latter solvent-free compounds are obtained by desolvation of **3** or **4** using a combination of heat and vacuum conditions. Notably, compounds **7** and **8** were found to bind CO gas reversibly under mild conditions in solution and in the solid state over multiple cycles.

The versatile carbon monoxide trap and release properties of these compounds may be useful in a variety of applications although initially we intend to assess their suitability for use as ^{11}CO-trapping agents in systems developed for the radiolabelling of drug molecules [11,25,26].

4. Experimental

4.1. General

All air and moisture-sensitive compounds were manipulated under an inert atmosphere of nitrogen using conventional Schlenk or glove-box techniques. General solvents were passed through drying columns and freeze-thaw degassed before use. Deuterated solvents were purchased from the Cambridge Isotope Laboratories. All other chemicals were purchased from commercial sources and used as received. Compound **1** was synthesised according to a literature procedure [12]. NMR spectra were recorded on a Bruker Av-400 or DRX-400 MHz NMR spectrometer. ^{1}H and ^{13}C spectra were referenced to residual proton resonances in the deuterated solvent, while ^{31}P{^{1}H} spectra were referenced to an external H_3PO_4 standard. NMR spectroscopic resonances are reported in ppm at 298 K unless otherwise stated. Infrared absorption spectra were

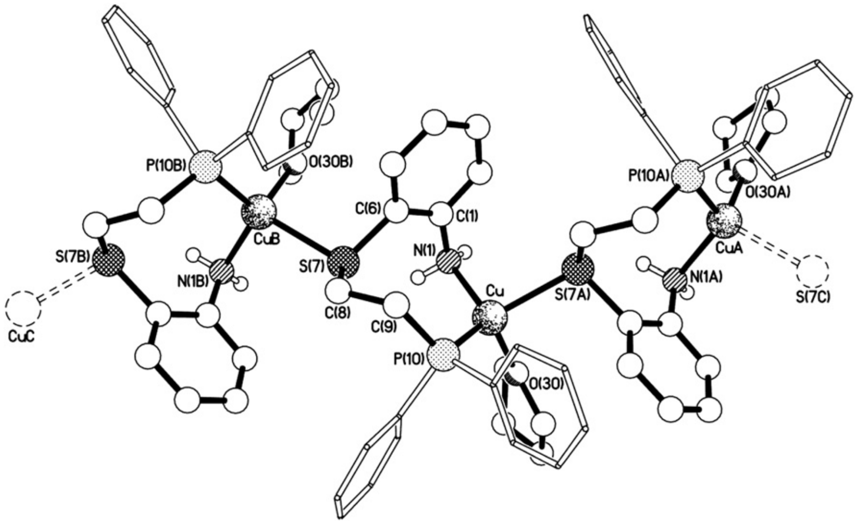


Fig. 3. Part of one of the extended cationic chains present in the crystals of **3**. Selected bond lengths (Å) and angles (°); Cu–N(1) 2.152(3), Cu–P(10) 2.2180(9), Cu–O(30) 2.145(3), Cu–S(7A) 2.4172(9), N(1)–Cu–P(10) 119.14(8), N(1)–Cu–O(30) 98.73(11), N(1)–Cu–S(7A) 99.18(8), P(10)–Cu–O(30) 116.31(8), P(10)–Cu–S(7A) 122.35(3), O(30)–Cu–S(7A) 96.31(9).

collected with a Perkin Elmer RX FT-IR spectrometer using a nitrogen purged cell containing KBr discs. Elemental analyses were performed by Stephen Boyer, Science Centre, London Metropolitan University, London.

4.2. Procedures

*4.2.1. Preparation of $Ph_2PCH_2CH_2S(2\text{-}C_6H_4OMe)$ (**2**)*

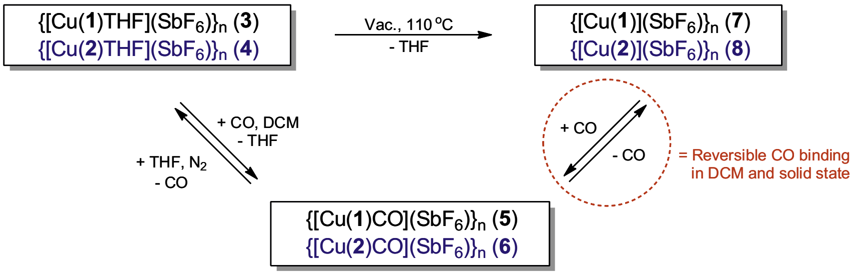
$KPPh_2$ (20.3 ml, 10 mmol) was added dropwise to a solution of 1,2-dichloroethane (4.8 ml, 61 mmol) in THF (80 ml) at −78 °C. The resulting yellow solution was allowed to reach room temperature and was then stirred for 12 h. Removal of the solvent *in vacuo* led to the isolation of an off-white glassy solid. Addition of Cs_2CO_3 (3.3 g, 10 mmol), MeCN (80 ml) and 2-methoxybenzenethiol (1.43 g, 10 mmol) was followed by heating of the mixture to reflux for 2 days. The resulting suspension was filtered through celite and the solvent was removed *in vacuo* to give **2** as a white solid. Yield = 2.95 g, 83%. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −17.1 (s). ^1H NMR (CD_2Cl_2): 7.36 (m, 10H), 7.18 (m, 1H), 7.11 (m, 1H), 6.86 (m, 2H), 3.80 (s, 3H), 2.92 (m, 2H), 2.33 (m, 2H). Anal. Calcd. for $C_{21}H_{21}OPS$: C, 71.57; H, 6.01. Found: C, 71.41; H, 5.79.

*4.2.2. Preparation of $\{[Cu(\mathbf{1})THF](SbF_6)\}_n$ (**3**)*

1 (0.2 g, 0.59 mmol) and CuCl (0.058 g, 0.59 mmol) were combined and dissolved in DCM (20 ml). $AgSbF_6$ (0.2 g, 0.58 mmol) was added in one portion and the resulting suspension was stirred for 30 min. Filtration and subsequent removal of the solvent *in vacuo* gave a green solid. Hexane (1 ml) was added followed by THF (5 ml) and the resulting solution was heated briefly to reflux and then allowed to cool to room temperature. A green solution was filtered from the precipitated white product which was washed with Et_2O (2 × 5 ml) and dried *in vacuo*. Yield = 0.28 g, 68%. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −6.7 (s). ^1H NMR (CD_2Cl_2): 7.93–9.93 (m, 14H), 5.08 (s, 2H), 3.84 (s, 4H), 3.26 (s, 2H), 2.37 (s, 2H), 1.98 (4H). $^{13}C\{^1H\}$ NMR (CD_2Cl_2): 133.67 (s), 133.11 (d, 15 Hz), 132.13 (s), 131.48 (s), 130.21 (d, 10 Hz), 129.57 (s), 128.39 (s), 126.17 (s). Anal. Calcd. for $C_{24}H_{28}CuF_6NOPSSb$: C, 40.65; H, 3.98; N, 1.98. Found: C, 40.55; H, 3.81; N, 1.87. Crystal data for **3**: $M = 708.79$, monoclinic, $P2_1/c$ (no. 14), $a = 10.36016(6)$, $b = 23.69555(15)$, $c = 11.23626(7)$ Å, $\beta = 99.8468(6)°$, $V = 2717.75(3)$ Å^3, $Z = 4$, $D_c = 1.732$ g cm^{-3}, μ(Cu-Kα) = 10.665 mm^{-1}, $T = 173$ K, colourless needles, Oxford Diffraction Xcalibur PX Ultra diffractometer; 5306 independent measured reflections ($R_{int} = 0.0265$), F^2 refinement, $R_1(obs) = 0.0326$, $wR_2(all) = 0.0840$, 4860 independent observed absorption-corrected reflections [$|F_o| > 4\sigma(|F_o|)$, $2\theta_{max} = 145°$], 390 parameters. CCDC 885922.

*4.2.3. Preparation of $\{[Cu(\mathbf{2})THF](SbF_6)\}_n$ (**4**)*

2 (0.2 g, 0.57 mmol) and CuCl (0.056 g, 0.57 mmol) were combined and dissolved in DCM (20 ml). $AgSbF_6$ (0.2 g, 0.58 mmol)



Scheme 1. Reversible binding of carbon monoxide in solution and in the solid state.

was added in one portion and the resulting suspension was stirred for 30 min. Filtration and subsequent removal of the solvent *in vacuo* gave a green solid. THF (5 ml) was added and the resulting solution was heated briefly to reflux. Removal of the solvent gave **4** as a white solid which was washed with Et_2O (2 × 5 ml) and dried *in vacuo*. Yield = 0.25 g, 61%. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −4.1 (s, broad). ^1H NMR (CD_2Cl_2): 7.75–7.24 (m, 12H), 7.09 (d, J_{H-H} = 8 Hz, 1H), 6.97 (d, J_{H-H} = 8 Hz, 1H), 4.08 (s, 4H), 3.67 (s, 3H), 3.19 (s, 2H), 2.50 (s, 2H), 2.09 (s, 4H). Anal. Calcd. for $C_{25}H_{29}CuF_6O_2PSSb$: C, 41.47; H, 4.04. Found: C, 41.49; H, 3.96.

4.2.4. Preparation of $\{[Cu(\mathbf{1})CO](SbF_6)\}_n$ (**5**)

A stream of CO (1 atm) was either bubbled through a DCM (or CD_2Cl_2) solution of **3** or passed over finely powdered samples of **7** for 10 min to give **5**. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −2.1 (s). ^1H NMR (CD_2Cl_2): 7.70 (d, J_{H-H} = 7 Hz, 1H), 7.61–7.13 (m, 13H), 4.92 (s, 2H), 3.17 (s, 2H), 2.71–1.90 (m, 2H), 3.72 (s, 4H), 1.88 (s, 4H). IR = 2091 cm^{-1} (CO). Anal. Calcd. for $C_{21}H_{20}CuF_6NOPSSb$: C, 37.93; H, 3.03; N, 2.11. Found: C, 37.89; H, 2.91; N, 1.87.

4.2.5. Preparation of $\{[Cu(\mathbf{2})CO](SbF_6)\}_n$ (**6**)

A stream of CO (1 atm) was either bubbled through a DCM (or CD_2Cl_2) solution of **4** or passed over finely powdered samples of **8** for 10 min to give **6**. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −1.1 (s, broad). ^1H NMR (CD_2Cl_2): 8.00–6.80 (m, 14H), 3.81 (s, 3H), 3.73 (s, 4H), 3.15 (s, 2H), 2.43 (s, 2H), 1.86 (s, 4H). IR = 2114 cm^{-1} (CO). Anal. Calcd. for $C_{22}H_{21}CuF_6O_2PSSb$: C, 38.86; H, 3.12. Found: C, 38.97; H, 3.10.

4.2.6. Preparation of $\{[Cu(\mathbf{1})](SbF_6)\}_n$ (**7**)

Solid **3** was finely powdered and then heated for 2 h at 110 °C *in vacuo* to give **7**. Alternatively, **7** was obtained by bubbling a stream of N_2 through a DCM (or CD_2Cl_2) solution of **5** for 10 min. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −5.3 (s). ^1H NMR (CD_2Cl_2): 7.95–6.84 (m, 14H), 5.03 (s, 2H), 3.24 (s, 2H), 2.46 (s, 2H). Anal. Calcd. For $C_{20}H_{20}CuF_6NPSSb$: C, 37.71; H, 3.17; N, 2.20. Found: C, 37.82; H, 3.25; N, 2.29.

4.2.7. Preparation of $\{[Cu(\mathbf{2})](SbF_6)\}_n$ (**8**)

Solid **4** was finely powdered and then heated for 2 h at 110 °C *in vacuo* to give **8**. Alternatively, **8** was obtained by bubbling a stream of N_2 through a DCM (or CD_2Cl_2) solution of **6** for 10 min. $^{31}P\{^1H\}$ NMR (CD_2Cl_2): −3.4 (s, broad). ^1H NMR (CD_2Cl_2): 7.70–7.23 (m, 12H), 7.08 (t, J_{H-H} = 8 Hz, 1H), 6.96 (d, J_{H-H} = 8 Hz, 1H), 3.61 (s), 3.28 (s), 2.65 (s). Anal. Calcd. for $C_{21}H_{21}CuF_6OPSSb$: C, 38.69; H, 3.25. Found: C, 38.69; H, 3.34.

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Appendix A. Supporting material

Supplementary data related to this article can be found online at doi:10.1016/j.jorganchem.2012.05.032.

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