

# Exploring Methods for Capping Metal Organic Framework 5



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## Abstract

Metal organic framework 5 (MOF-5) is a crystalline, porous crystal made up of zinc centers and organic linkers. Previous research conducted by the Grimm, Burdette and MacDonald labs determined the mechanism and structure, as well as capping MOF-5 to trap crystal violet, a large sterically bulky dye, within the pores. Previous caps have included triphenyl and diphenyl acetic acid. This paper demonstrates that silanization and derivative capping with non-nucleophilic bases could be an effective method of capping, whereas derivative capping with silanes and acyl chlorides is less effective. Furthermore, the determination whether the application of heat is necessary for effective and consistent capping is explored within this paper.

## Acknowledgements

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## 1. Introduction

Metal organic frameworks took the chemistry world by storm when they were discovered in 1990. Metal organic frameworks, or better known as MOFs, are porous, crystalline compounds that are formed from inorganic molecules and organic ligands. Certain MOFs can hold up to 90% of their volume within their porous structure, making them ideal vehicles for drug delivery, or even trapping gases. Furthermore, most MOFs can be synthesized in a one-pot reaction, making their synthesis both time and financially desirable. (Gongke et al.)

Current and future uses of MOFs are primarily drug delivery and compound trapping. In a published study, one high school held a demonstration showing various MOFs ability to adsorb toxic chemicals out of solution. (Yoshie et al.) Another study demonstrated the use of MOFs in cancer drug delivery due to their small size and adsorbent abilities. Within the world of drug delivery, vehicles that are easy to synthesize, biodegradable, and non-toxic are highly sought after. (Ibrahim et al.)

MOF-5 is a large, porous, zinc-based crystal that is able to trap and contain large bulky guests. The zinc molecules coordinate with benzene-1,4-dicarboxylic acid (BDC) linkers to form the porous structure shown below.

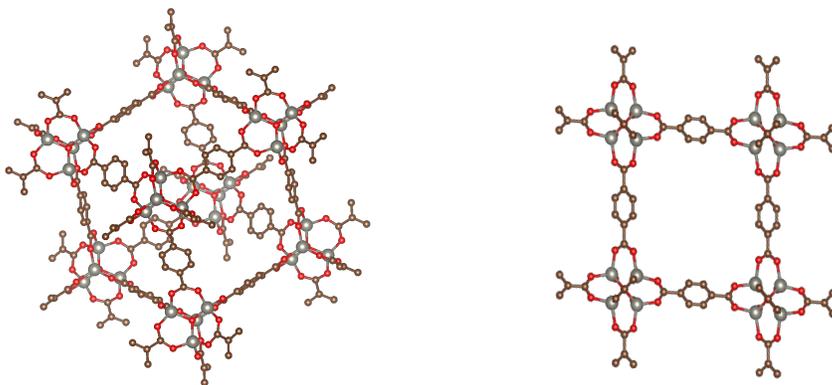


Figure 1.1. Above are two different angles of the structure of metal organic framework 5. The structure and surface chemistry of MOF-5 is the subject of this research. The large silver atoms are zinc, the red atoms are oxygen coordination groups, and the brown atoms are the organic carbon linkers.

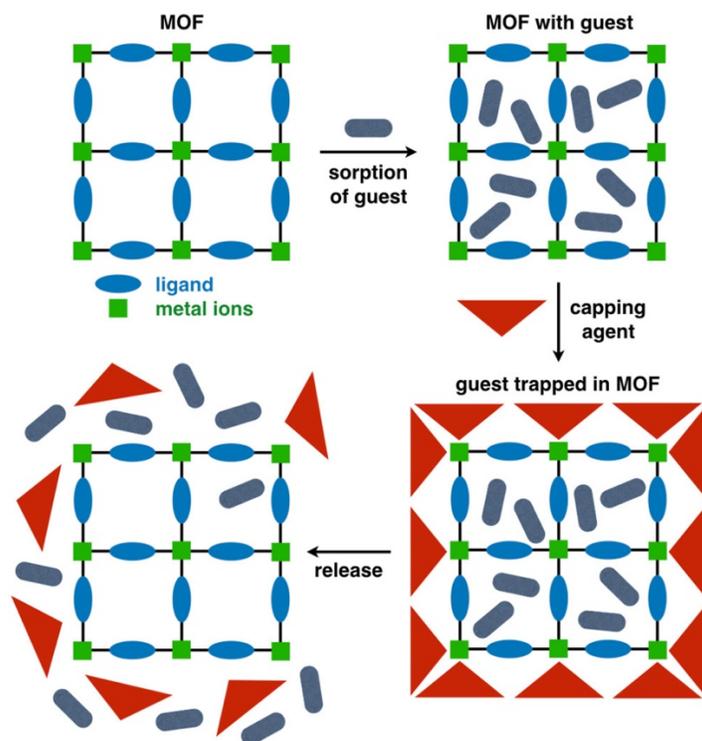


Figure 1.2. The figure above demonstrates the general idea of capping MOF-5. The guest would be introduced to and enter the crystal, then the MOF-5 containing the guest would be put in the presence of a cap, effectively trapping the guest within the pores of the crystal. (Homan et al.)

## 2. Background

### Silanes

Silane based caps were the primary reagent used to cap MOF-5. Silanes are frequently used as coupling agents and are known to have organic and inorganic reactivity. This allows for silane-based compounds to react with the inorganic surface of MOF-5 while being able to act as the interphase region between MOF-5 and an organic cap. The general structure of a silane consists of four substituents attached to a silicon atom. The substituent groups are usually three alkoxy groups, and an organic group.

### X-ray Spectroscopy

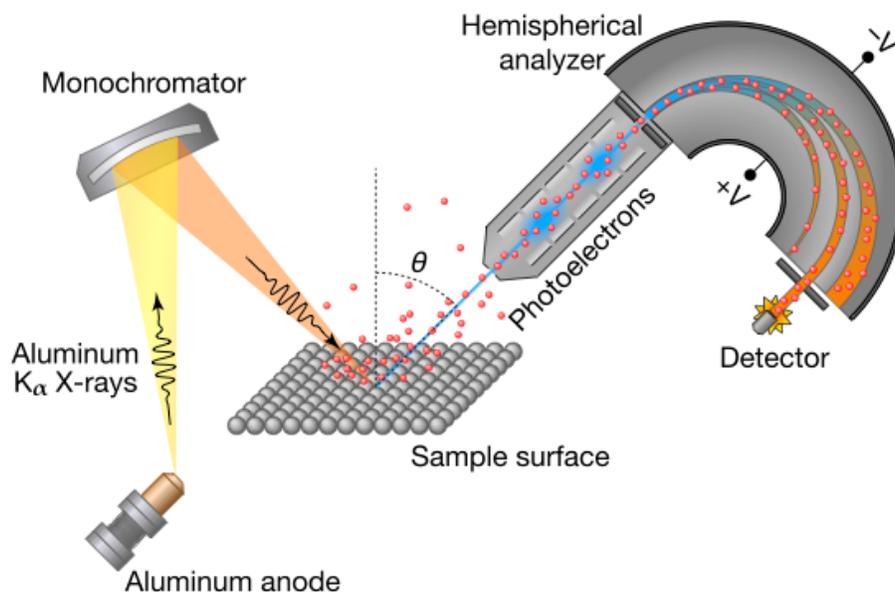


Figure 2.1. Sourced from GrimmGroup.net. The image shows how X-ray spectroscopy spectra are gained, starting from x-rays being shot towards a sample, and electrons being shot out of the sample towards a detector.

X-Ray Spectroscopy (XPS) is a surface-sensitive quantitative spectroscopic technique that allows us to determine atoms on a sample's surface, as well as what atoms are bonded to. Photons are shot on the surface of a sample by an aluminum anode and are absorbed by the sample. When a sample has absorbed a certain number of photons, electrons are ejected out of the sample and are analyzed by a detector. The energies at which electrons are ejected is determined and a spectra is created. Peaks on the spectra are associated with different binding energies (eV). Electrons that are further away from the nucleus require less energy to be ejected, so they have lower binding energies. XPS also allows for us to determine chemical shifts within a sample. For example, carbons bonded to carbons would have a different peak in comparison to carbons bonded to oxygen. (LibreTexts)

## Derivative Capping/Click Chemistry

Derivative capping is the creation of a new cap by replacing a molecule or compound on the original cap with something new. Click chemistry was introduced by Dr. Barry Sharpless in 1999. Click chemistry refers to the idea of reactions that are one pot and simple to perform without the use of protecting agents. (Hein)

A goal of this project was to utilize the concept of one-pot, derivative click chemistry when capping MOF-5. After synthesis, MOF-5 would be derivatively capped with 3-aminopropyltriethoxysilane and 4-(trifluoromethyl)benzoyl chloride.

## Previous Research

This project is a part of an ongoing research process to better understand the chemical structure and surface chemistry of MOF-5. Previous research was conducted by the MacDonald lab, Burdette group and Grimmgroup of WPI.

Previous research determined the protocol in order to grow MOF-5 crystals, and identified that N,N-diethylformamide (DEF) synthesizes MOF-5, while N,N-dimethylformamide (DMF) does not. This protocol stands out from several other published protocols, which require the use of DMF over DEF. Previous research also determined that acetic acid-based compounds can react and attach to the surface of MOF-5. This conclusion was determined by filling MOF-5 pores with a sterically bulky dye, Crystal Violet (CV) while observing dye loss over time. Acetic acid-based compounds with larger R-groups had slower rates of dye release, while smaller compounds had faster rates. Uncapped MOF-5 had significant rates of dye loss. (MacDonald)

This research concluded that carboxylates could react and attach to the surface of MOF-5, and this research has provided a solid foundation for this project to start.

## 3. Experimental Section

### Materials and Chemicals

Chemicals used for synthesis and capping of MOF-5 included zinc nitrate hexahydrate, terephthalic acid, N,N-dimethylformamide [DMF], N,N-diethylformamide [DEF], Toluene, Ethanol, 4-(trifluoromethyl)benzoyl chloride, 97% [benzoyl chloride], 4-(trifluoromethyl)benzoic acid, 98% [benzoic acid], (3,3,3-trifluoropropyl)trimethoxysilane, 97% [methoxysilane], (3,3,3-trifluoropropyl)trichlorosilane [trifluoro trichlorosilane], 3-aminopropyltriethoxysilane [aminosilane], 1,8-diazabicyclo[5.4.0]undec-7-ene [DBU].

### Instrumentation

A Phi5600 XPS system with a third-party data acquisition system (RBD Instruments, Bend Oregon) acquired all photoelectron spectra. Analysis chamber base pressures were  $<1 \times 10^{-9}$  Torr. A hemispherical energy analyzer that was positioned at  $90^\circ$  with respect to the incoming monochromated Al K $\alpha$  X-ray flux and  $45^\circ$  with respect to standard sample positioning collected the photoelectrons. Survey spectra utilized a 117 eV pass energy, a 0.5 eV step size, and a 50-ms-per-step dwell time. High-resolution XP spectra employed a 23.5 eV pass energy, 0.025 eV step size, and a 50 ms dwell time per step. Inconsistent 285 eV position of features ascribed to adventitious carbon; acquisitions necessitated a charge neutralization.

Post-acquisition data fitting employed W-Tougaard-style baselines and GL(70) peak shapes, a Shirley-style baseline and GL(70) peak shape, a W-Tougaard baseline and GL(30) peak shape, and linear baselines with GL(30) peak shapes for C 1s, O 1s, N 1s, Zn 2p 3/2, Si 2p, F 1s, and Cl 2p regions. Fits that employ multiple peaks within a spectral region utilized identical fwhm values for each peak to minimize mathematically optimized but possibly chemically unrealistic fits.

### Synthesis of MOF-5

MOF-5 was synthesized by massing out 0.90 mg zinc nitrate, 0.160 mg terephthalic acid, and 25mL of N,N-diethylformamide. The reagents were placed in a round bottom vial, capped, and sonicated for 3-5 minutes, until all solids had dissolved, and the solution did not appear cloudy. The vial was then placed in a furnace to cook. The furnace warmed up to 120°C over 5 hours, stayed at 120°C for 24 hours, and cooled to 0°C over 7 hours, for a total cooking time of 36 hours.

### Capping Procedures

#### Silane Capping Preparation

After cooking, the vial of freshly synthesized MOF-5 was opened, and the leftover DEF solvent was decanted. If the MOF-5 was anticipated to be capped with 3-aminopropyltriethoxysilane, toluene was poured over the MOF-5 to displace any DEF that would be leftover in the pores from synthesis. If the MOF-5 was anticipated to be capped with either

(3,3,3-trifluoropropyl)trimethoxysilane or (3,3,3-trifluoropropyl)trichlorosilane, DMF was poured over the MOF-5 to displace any remaining DEF within the pores of the MOF-5.

#### Silane Capping

75mg of MOF-5 was scooped from the side of the vial and placed in a clean tapered vial. 2mL of the appropriate diluted silane stock solution was poured over the MOF-5. The vial was then capped and left overnight.

#### Benzoyl Chloride Capping

75mg of newly synthesized MOF-5 was scraped from the sides and bottom of the vial and placed in a clean vial. 5mL DEF and 5 $\mu$ L acid chloride was poured over the MOF-5, and the vial was left to sit between 5-8 hours.

#### Benzoic Acid Capping

75mg of newly synthesized MOF-5 was scraped from the sides and bottom of the vial and placed in a clean vial. 5mL DEF was poured into the vial and 0.033 mol carboxylic acid was massed and placed in the vial. The vial was left to sit overnight.

#### Benzoyl Chloride and Amino silane Capping

There were two experiments to determine the feasibility of capping MOF-5 with the acid chloride. The first was to determine that the acid chloride would be able to attach to the amino surface of the amino silane capped MOF-5. The second was to determine that the acid chloride did not interact with the uncapped surface of MOF-5.

For the first experiment, newly synthesized MOF-5 was capped with amino silane following the previously mentioned procedure. Following the amino silane capping, the amino silane diluted stock was decanted from the vial and replaced with 5mL of DEF and 5 $\mu$ L of the acid chloride.

Two methods of capping were proposed. The first method was to cook the MOF-5 now in DEF and acid chloride under normal synthesis conditions. The second method was to leave the vial alone, and let it sit overnight.

#### DBU and Benzoic Acid Capping

75mg of newly synthesized MOF-5 was scraped off from the sides and bottom of the vial and placed in a clean vial. 5mL of DEF was poured over the MOF-5. 55 $\mu$ L of DBU and 0.033 mol of carboxylic acid was massed out and put in the vial.

Two methods of capping were proposed. The first method was to cook the MOF-5 now in DEF with the DBU and carboxylic acid under normal synthesis conditions. The second method was to leave the vial alone, and let it sit overnight.

## XPS Sample Preparation

An XPS puck was obtained and wiped down with isopropanol. A small piece of carbon tape was cut and placed on the puck. With a spatula, a small scoopful of MOF-5 was taken from the vial, and gently placed on the carbon tape. The puck was transferred to drying chamber, which was attached to the Schlenk line. The drying chamber was sealed, and the puck was left to dry overnight or over the weekend.

## 4. Results

### XPS Characterization

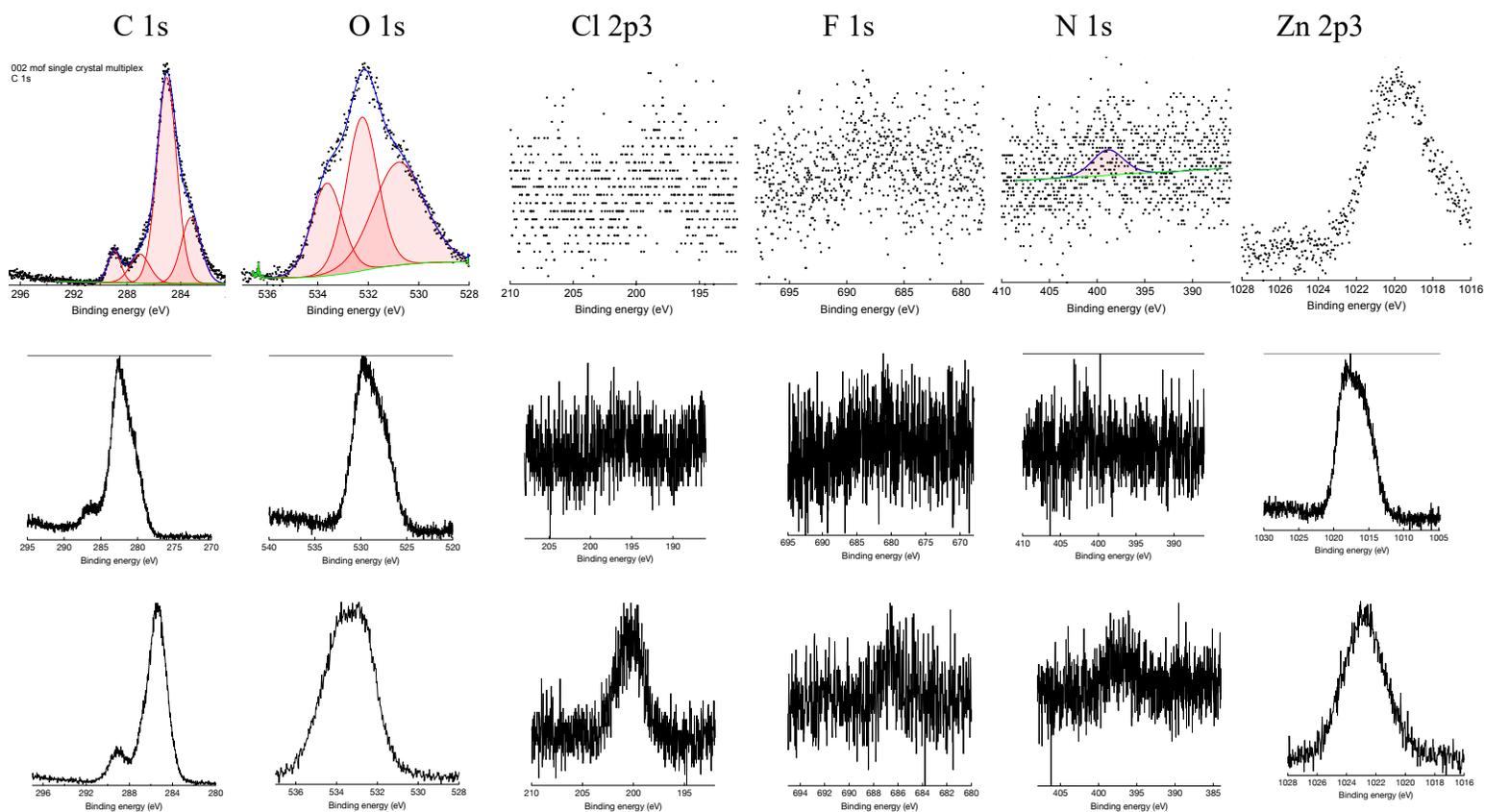


Figure 4.1. Above are three different XPS scans of uncapped MOF-5. After synthesis, these crystals were placed on a puck and dried over the Schlenk line between 1-3 days, then scanned. The peaks at columns C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was successfully synthesized, because MOF-5 is composed of those molecules. The peak within the chlorine column is likely contamination, from the puck or lab utensils not being cleaned between samples. The peaks within the nitrogen column are likely leftover solvent within the pores of MOF-5.

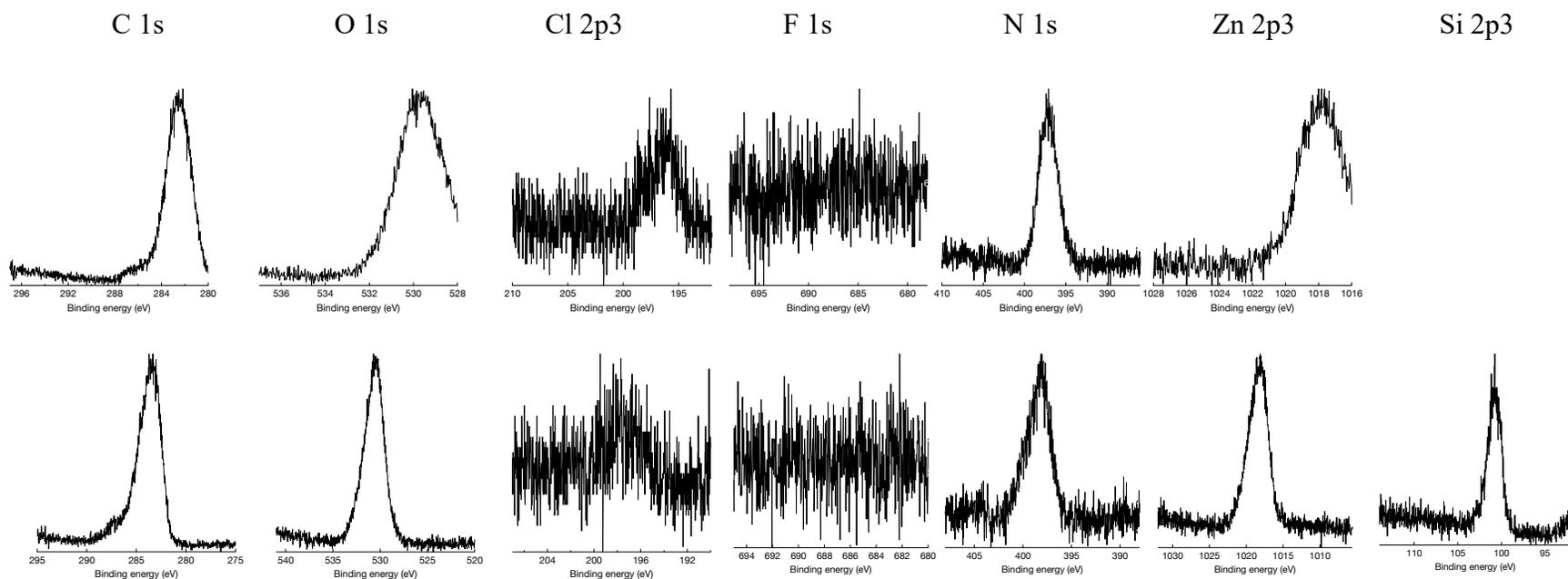


Figure 4.2. These scans show MOF-5 capped with 3-aminopropyltriethoxysilane. The distinct peaks at C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was not only synthesized but did not degrade while being capped. The peaks within the nitrogen and silicone columns are indicative of the aminosilane reacting with and attaching to the surface of the MOF-5 crystals. The peaks within the chlorine column are likely contamination, from either the puck or lab utensils not being cleaned between samples.

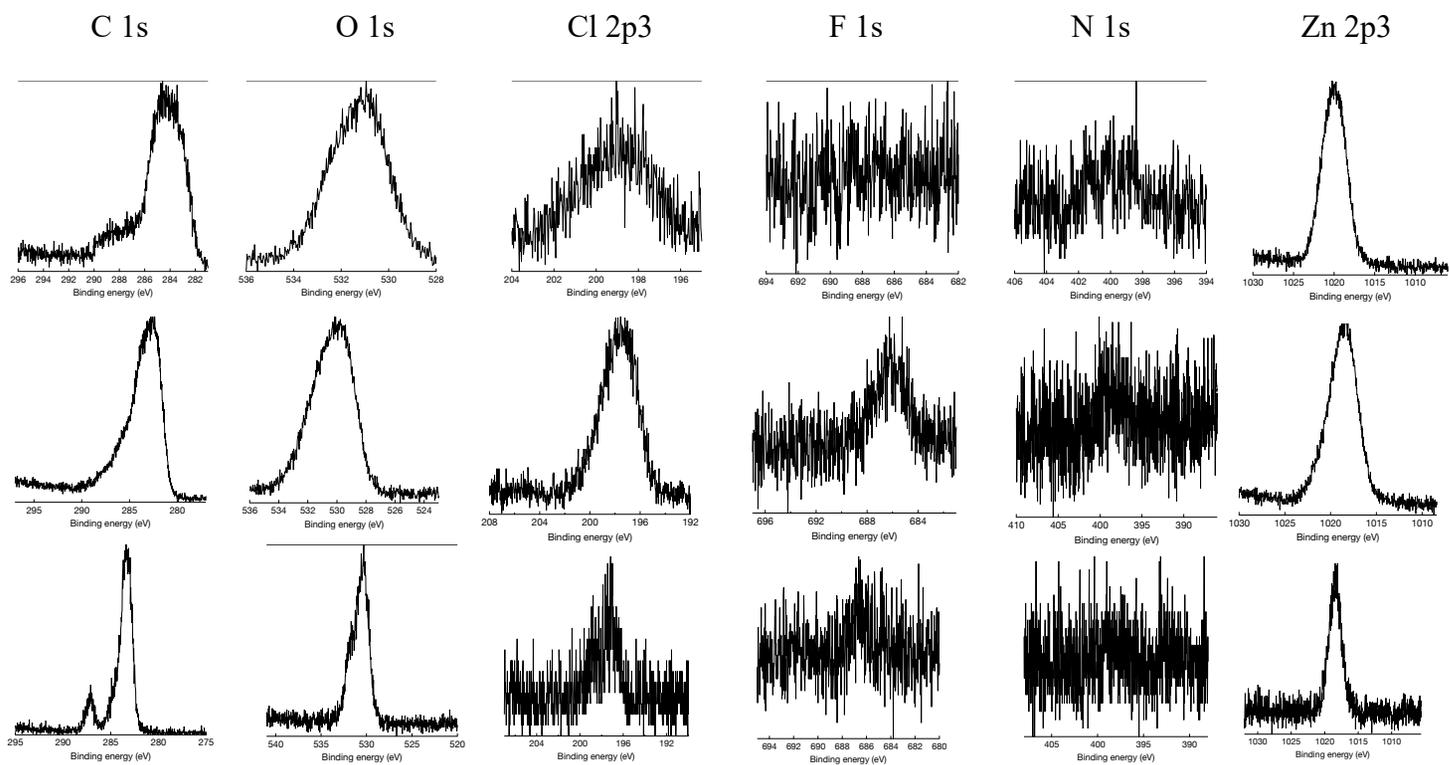


Figure 4.3. The scans above are MOF-5 capped with 4-(trifluoromethyl)benzoyl chloride. The distinct peaks at C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was not only synthesized but did not degrade while being capped. There are distinct peaks within both the chlorine and fluorine columns, indicating that the benzoyl chloride did not react and attach to the surface of MOF-5.

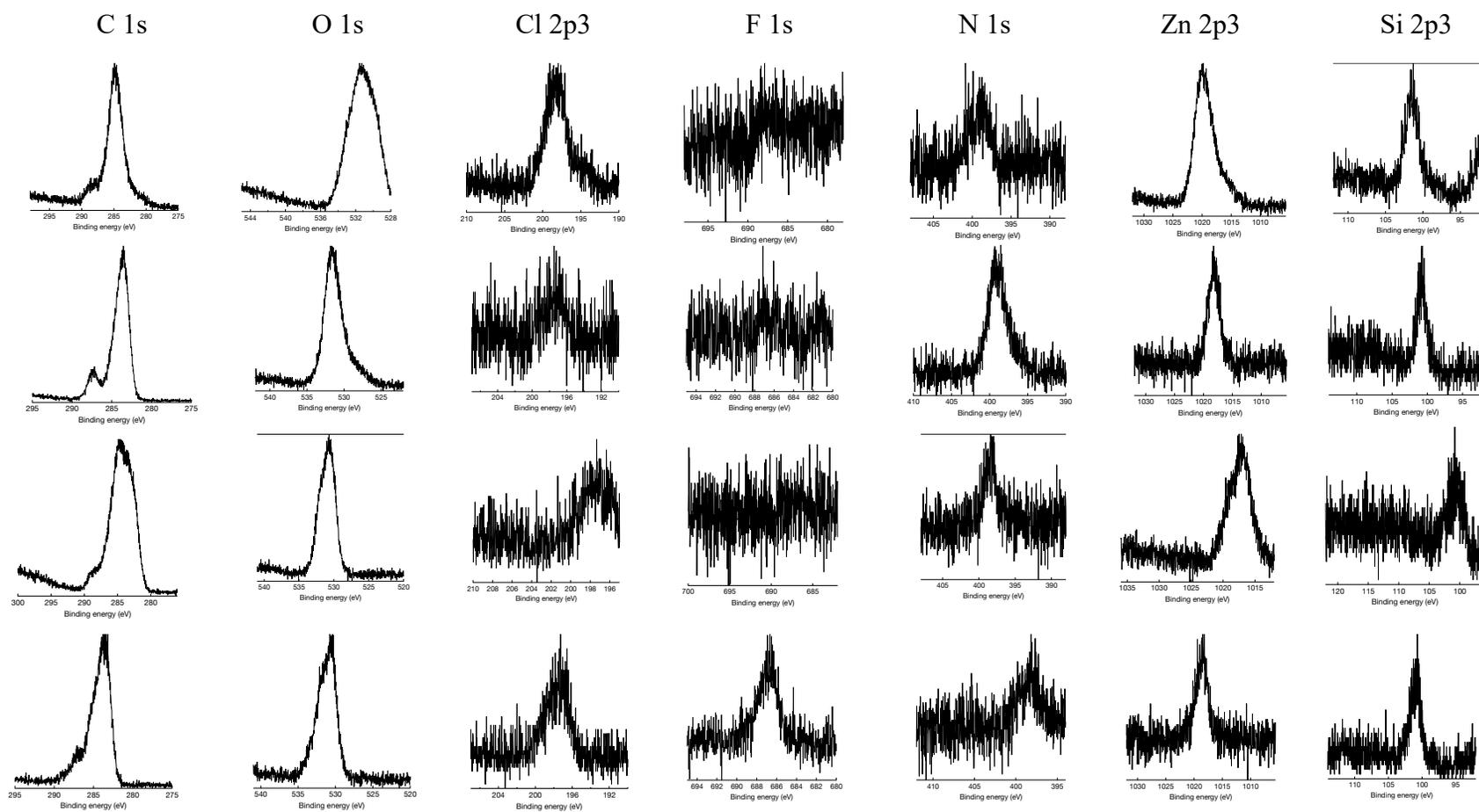


Figure 4.4. The scans above are MOF-5 capped with 3-aminopropyltriethoxysilane and 4-(trifluoromethyl)benzoyl chloride. The distinct peaks at C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was not only synthesized but did not degrade while being capped. The peaks within the nitrogen and silicone columns indicate that the aminosilane reacted and attached to the surface of the MOF-5 crystals. The presence of both chlorine and fluorine indicates that the benzoyl chloride did not interact with the aminosilane cap on the surface of the MOF-5 crystals, leaving the aminosilane as the only cap.

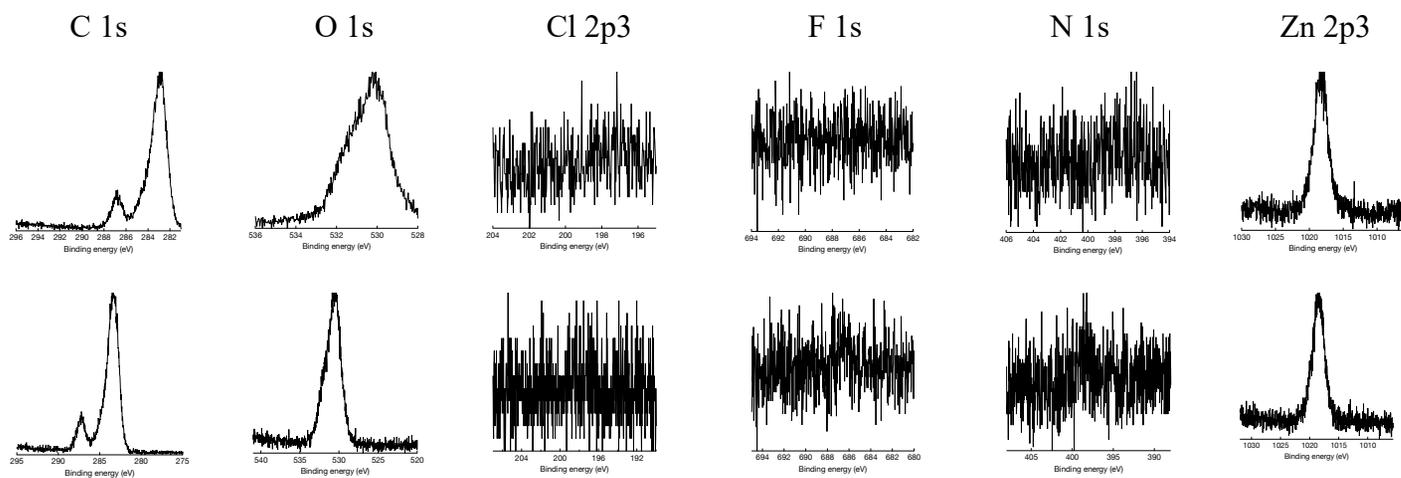


Figure 4.6. The scans above are MOF-5 capped with 4-(trifluoromethyl)benzoic acid. The distinct peaks at C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was not only synthesized but did not degrade while being capped. There are no other distinct peaks, indicating that the benzoic acid did not react and attach to the surface of MOF-5.

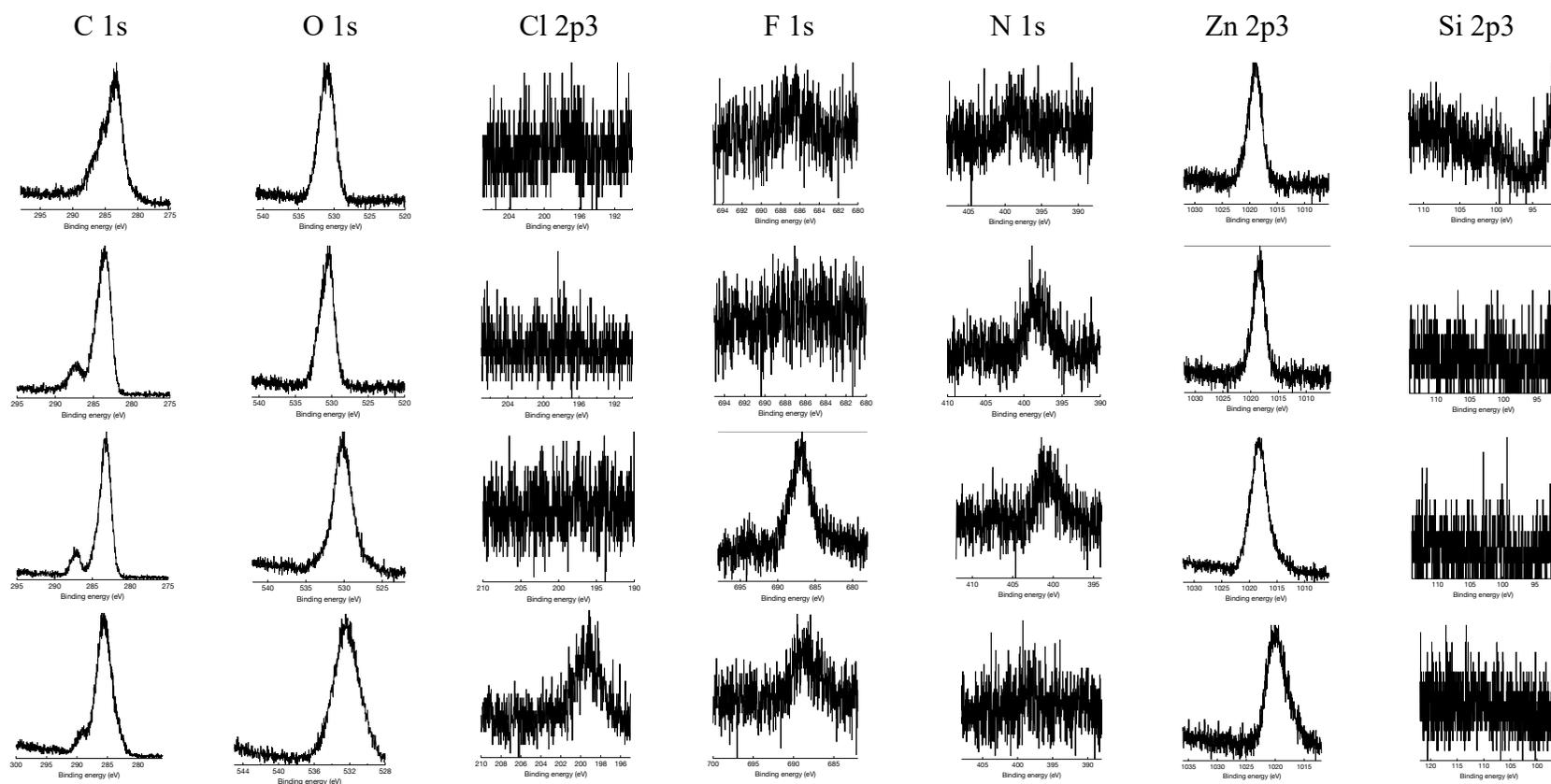


Figure 4.6. The scans above are MOF-5 capped with 4-(trifluoromethyl)benzoic acid with the presence of 1,8-diazabicyclo [5.4.0]undec-7-ene (DBU), a non-nucleophilic base. The distinct peaks at C 1s, O 1s, and Zn 2p3 indicate that MOF-5 was not only synthesized but did not degrade while being capped. There are peaks within the nitrogen and fluorine columns, that suggests that the benzoic acid did react and attach to the surface of MOF-5 in the presence of DBU.

## 5. Discussion

### Synthesis and Characterization of MOF-5

This project is continuing the ongoing research to understand the structure and surface chemistry of MOF-5. As mentioned earlier, previous research conducted by the MacDonald lab and Grimmgroup determined the protocol to synthesize MOF-5 effectively and successfully. With the set protocol, this project was able to synthesize MOF-5 for the purpose of capping and determining which capping agents were successful and which were not.

Because MOF-5 was not a frequently studied crystal, protocols online varied in amounts of reagent and solvents, and synthesis temperatures were different. Therefore, the protocol used for this project was published by a research lab at WPI. MOF-5 is composed of carbon, oxygen, and zinc. XPS spectra showed tall peaks within those regions, which is a good indication that MOF-5 was synthesized. Furthermore, resulting spectra from various capping experiments shows the same regioselective peaks in the same locations, indicating that the protocol used for MOF-5 synthesis was correct and synthesis was consistent.

### X-Ray Photoelectron Spectroscopy

The success of synthesizing MOF-5 was further verified by XPS spectra of other caps. The first cap to determine this was the 3-aminopropyltriethoxysilane cap. To verify that this cap attached to the surface of MOF-5 crystals, XPS spectra needed to show five peaks in five different regions: carbon, oxygen, zinc, nitrogen, and silicone. As mentioned earlier, carbon, oxygen and zinc make up the structure of MOF-5, while silicone, nitrogen, chlorine, and fluorine peaks suggest that MOF-5 has been capped.

Within the spectra of MOF-5 being capped with silanes, silicone peaks were a good suggestion of silanes reacting and attaching to the surface of MOF-5. Two of the three silanes contained a trifluoromethyl tag, meaning that should the silane cap to the surface, it would be indicated by the presence of fluorine. However, one cap did not possess fluorine, instead there was an amine tag. The presence of nitrogen would suggest that the aminosilane capped on the surface of MOF-5. However, the solvent in which MOF-5 is synthesized contains nitrogen, so it could also be suggested that nitrogen peaks on the XPS spectra are indicative of the fact the MOF-5 crystals are not dry and there is leftover solvent within the pores. Luckily, there are very distinct nitrogen peaks between MOF-5 capped with aminosilane and uncapped MOF-5.

The remaining caps are the benzoic acid that was capped in the presence of DBU, a non-nucleophilic base, and benzoyl chloride, which was used for derivative capping. For the benzoyl chloride cap, the presence of both chlorine and fluorine suggest the cap did not interact and attach to the surface of MOF-5. The tall chlorine peaks and lack of fluorine peaks very strongly suggest that benzoyl chloride cannot derivatively cap with aminosilane under synthesis conditions. However, benzoic acid appeared to cap on the surface of MOF-5 in the presence of DBU. Comparisons between MOF-5 capped with just benzoic acid and MOF-5 capped with benzoic acid and DBU have significantly different sized fluorine peaks, and MOF-5 capped in the presence of DBU also showed small peaks of nitrogen, which is a component of DBU.

## Issues and Obstacles

Throughout this project, there were several notable obstacles that had to be overcome. The first obstacle encountered was the synthesis of MOF-5. In the early stages there were many inconsistencies with the crystals size and color, creating confusion over whether MOF-5 was synthesized. These inconsistencies stemmed from technical issues with one of the two ovens used to synthesize MOF-5. The display temperature of the Burdette oven was about 40°C lower than the internal temperature. Because of this, the temperature at which MOF-5 was synthesized was predicted to be anywhere between 115°C and 125°C from temperature fluctuations between the oven and the thermocouple used to determine temperature.

Another notable difference in the synthesis of MOF-5 was the DEF used. Previous research conducted by the MacDonald research group determined that DEF should be used over DMF as the solvent for synthesis. Unfortunately, DEF is more expensive than DMF, requiring us to save and distill DEF to recycle and reuse it for continued MOF-5 synthesis. When using DEF from the bottle, the MOF-5 crystals were noticeably larger, whereas crystals that were synthesized later from distilled DEF were much smaller in size. Although there was no question whether MOF-5 was synthesized, scanning the surface of the crystals became more difficult due to the smaller surface area.



Figure 5.1. The above four samples are MOF-5 synthesized using fresh and distilled DEF. The sample of MOF-5 furthest to the left was synthesized using fresh DEF, while the remaining three were synthesized with distilled DEF.

To synthesize MOF-5, 30mL round-bottom vials were used with crimp caps and contained 25mL of reagent. Often, the caps would expand due to pressure buildup within the vial, and on occasion would break. The introduction of air into the vial degrades any MOF-5 that was synthesized. To mitigate this, the recipe used was halved, creating a total of 13mL of reagent instead of the original 25mL. Although this stopped the crimp caps from expanding, the

amount of MOF-5 synthesized was also halved. Additionally, at the time a different oven was used; Pluto, which allowed MOF-5 to grow at the correct temperature. Unfortunately, Pluto was the only working oven so multiple batches of MOF-5 could not be made simultaneously.

As previously mentioned, MOF-5 readily degrades while in the presence of air and water, so moving quickly and efficiently while working with MOF-5 is essential. Because of this, it should be noted that several of the peaks associated with zinc on the XPS scans are smaller than they should be. This could be because the MOF-5 took several days to dry, took several days to be pumped down to be scanned, or was out on a bench while being capped. Suggestions to mitigate this would be working with MOF-5 under nitrogen in a glovebox, and to make sure that solvents used to cap MOF-5 are completely dry.

Other contributions to the lowering of zinc peaks could be the caps used. Four out of the five caps used contained fluorine as a tag. Due to fluorine's reactive nature, it is possible that a prolonged presence of fluorine could have degraded MOF-5, causing a lower zinc peak. To mitigate this, limiting MOF-5 in the presence of caps containing fluorine to no more than 48 hours should prevent the degradation of MOF-5.

## 6. Conclusions and Next Steps

This project studied the structure and surface chemistry of MOF-5, identifying new caps and new capping methods to limit the pore sizes on the surface of MOF-5. Although not all the caps were able to react and attach to the surface, the results of this study gave us a better understanding of the surface of the crystal. The success of one-pot silane capping gives confidence in future one-pot capping experiments with different reagents.

Due to the time constraints of the project, repeating the experiments done would allow us to better understand the surface chemistry of MOF-5. Because MOF-5 readily degrades in the presence of air and water, moving quickly and efficiently when capping, drying and scanning MOF-5 is essential to successful experiments.

Recommended experiments include capping with the silanes because the diluted solutions started to polymerize and could not be used after an initial capping experiment. In continuation of this research, using a wider range of reagents for capping should be considered. In this research, only five distinct caps were used, and only three were studied in-depth.

Another idea that should be considered would be to use other metal organic frameworks to cap MOF-5, such as UiO-66. The crystal structure and pores of UiO-66 are smaller than those of MOF-5, so it would act as a pseudo cap or a type of filter to allow certain sizes of guests to be released.

Finally, new caps that should be considered should be able to cap diagonally at the intersections of the MOF-5 structure. Theoretically these caps would be long to span the surface of MOF-5, instead of a single cap at each intersection. The goal for this method of capping would be to have on-demand guest release. A suggestion for this type of cap would have the coordination sites of the cap be linked by a compound that could be degradable in the presence of UV light.

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