

Christopher M. Kane

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OBJECTIVE

Seeking a laboratory research position in Materials Science/Synthetic Chemistry and characterization.

HIGHLIGHTS

- Extensive experience in synthetic (organic/inorganic) and physical organic chemistry. Specifically, skill in performing a variety of reaction types (cross-couplings, photochemical, catalytic) in purification techniques (Recrystallization, chromatography, distillation), and knowledge in handling and using gases (inert, toxic, and/or flammable). Mastery in standard characterization techniques (NMR, IR/ATR, UV-Vis, GC-MS, EA, Raman).
- High proficiency in single crystal (SCXRD) and powder (PXRD) X-ray diffraction techniques. Expertise: in crystal growth, data collection, twinning, structure refinement, and CIF file preparation (>800 crystal structures collected, ~75 in CSD). Experience in general x-ray diffractometer operation (synchrotron – ALS, Diamond Light Source) and maintenance (APEX DUO, D8). PXRD experience: transmission (capillary) or reflection (Bragg-Bentano, zero background discs).
- Experience in the characterization and study of materials. Specifically, thermogravimetric analysis (TGA/tandem TGA-MS), differential scanning calorimetry (DSC), and an Autosorb sorption analyzer.
- Proficiency with MSOffice (Word, Powerpoint, Excel), chemical drawing (Chemdraw), imaging (Canvas, POV-RAY) programs, X-ray crystallography software (SHELX, X-Seed, Jade, Platon, enCIFer, Mercury, pubCIF) and databases (Cambridge Structural Database, FIZ/NIST Inorganic Crystal Structure Database).
- Experience in teaching and advising younger scientists in the laboratory (NSF-REU, ACS Project SEED program, MChem students), specifically in instrument operation, materials analysis, and safe lab practices. Tutored and taught lab sections for general chemistry and organic chemistry (2 semesters each).
- Proficiency in developing flow chemistry syntheses towards organic intermediates and target compounds.

EDUCATION

- Ph.D. in Chemistry (Synthetic Organic and Materials), Georgetown University, August 2015 (GPA 3.5) Advisor: Professor K. Travis Holman, Thesis title: “Crystalline Organic Cavitanas as Microcavity Materials.”
- M.S., Organic Chemistry, University of North Carolina at Charlotte, May 2009 (GPA 4.0) Advisor: Professor Markus Etzkorn, Thesis title: “Glycoluril-Derived Fluorinated Molecular Tweezers for Host-Guest Association Studies.”
- B.S., Organic Chemistry, James Madison University, May 2007, Advisor: Professor Scott Lewis, Research Project: “The Synthesis of 1-Hexylcyclobutene for Synthetic Studies With Difluorocarbene.”

RESEARCH AND WORK EXPERIENCE

May 2019-Present, Uppsala University (Postdoctoral Research Associate)

- Developed flow chemistry syntheses for the production of pyrrole from biomass for implementation into paper batteries.

January 2016-April 2019, University of Liverpool (Postdoctoral Research Associate)

- Engineered and developed organic tectons with strong, directional extended bonding for crystallographic studies and fruitful materials properties (in conjunction with computational collaborators on Crystal Structure Prediction).

August 2009-August 2015, Georgetown University

- Synthesized organic cavitand materials and characterized them using standard spectroscopic techniques and analyzed their materials properties for use in gas storage and separations applications.

June 2007-June 2009, University of North Carolina – Charlotte

- Synthesized molecular tweezer derivatives for application in supramolecular chemistry; furnished fluorinated naphthalenes to expand the scope of Bergman cyclization reactivity.

September 2005-May 2007, James Madison University

- Synthesized cyclobutene derivatives towards *m*-difluorinated benzenes as potential pathways for the synthesis and development of novel pharmaceutical compounds.

AWARDS & DISTINCTIONS

- **2013 Ludo Frevel Crystallography Scholarship** from the International Centre For Diffraction Data (\$2,500). Topic – “Low Packing Fraction Crystalline Cavitands Exhibiting Molecule Sized Cavities.” The committee selected 13 of 69 applications for the Scholarship Program. <http://www.icdd.com/resources/awards/frevelwinner.htm#2013>
- **International Research Fellowship Award (2)** from the University of California – Santa Barbara (UCSB) [Fall 2012 (\$5,000) and Fall 2010 (\$2,000)]. These fellowships support research visits to non-US labs for graduate students and postdocs from US institutions. Fostered a collaboration with Dr. Len Barbour at the University of Stellenbosch, South Africa. <http://www.icmr.ucsb.edu/programs/exchange.html>
- **2012 Bruker AXS/MIT Symposium Poster Prize Award**, Boston, MA (2012 Motto – Metal-Organic Frameworks). This is an annual event for chemists and crystallographers alike. A qualified group of judges awards one poster with the Bruker/MIT Poster Prize (\$500). http://web.mit.edu/pmueller/www/BMIT2012/content/D7C_2870_cs4_large.html
- **2008 Thomas D. Walsh Graduate Research Fellowship**, UNC-Charlotte. The Chemistry Department at UNCC presents this most prestigious fellowship to recognize outstanding graduate research. (RA/tuition support - \$15,500).
- **2008 UNCC Graduate Research Fair**, 1st Prize (Oral Presentation). Topic - “Glycoluril-based Molecular Tweezers for Host-Guest Association Studies”. The Graduate Research Fair recognizes excellent research in all graduate disciplines. (\$300)
- **2006 Research Experience for Undergraduates (REU)** awardee, James Madison University. The REU program supports active research participation by undergraduate students funded by the National Science Foundation (w/ Dr. Scott B. Lewis).

PATENTS

- Holman, K. T.; Kane, C. M. Cavitand Compositions and Methods of Use Thereof, US Patent Appl. 61/799,037, March 15, 2013.

PUBLICATIONS

9. Brekalo, I.; Deliz, D.; Kane, C. M.; Friščuóm T.; Holman, K. T. “Exploring the Scope of Macrocyclic ‘Shoe-last’ Templates in the Mechanochemical Synthesis of RHO Topology Zeolitic Imidazolate Frameworks.” *Molecules* **2020**, *Just Accepted*.
8. Brekalo, I.; Kane, C. M.; Ley, A. N.; Ramirez, J. R.; Friščić, T.; Holman, K. T. “Use of a ‘Shoe-last’ Solid-State Template in the Mechanochemical Synthesis of High-Porosity RHO-Zinc Imidazolate.” *J. Am. Chem. Soc.* **2018**, *140*, 10104-10108.
<https://pubs.acs.org/doi/pdfplus/10.1021/jacs.8b05471>
7. Pulido, A.; Chen, L.; Kaczorowski, T.; Holden, D.; Little, M. A.; Chong, S. Y.; Slater, B. J.; McMahan, D. P.; Bonillo, B.; Stackhouse, C. J.; Stephenson, A.; Kane, C. M.; Clowes, R.; Hasell, T.; Cooper, A. I.; Day, G. M. “Functional materials discovery using energy-structure-function maps.” *Nature* **2017**, *543*, 657-664.
<http://www.nature.com/nature/journal/vaop/ncurrent/full/nature21419.html>
6. Ramirez, J. R.; Yang, H.; Kane, C. M.; Ley, A. N.; Holman, K. T. “Reproducible Synthesis and High Porosity of mer-Zn(Im)₂ (ZIF-10): Exploitation of an Apparent Double-Eight Ring Template.” *J. Am. Chem. Soc.* **2016**, *138*, 12017-12020.
<http://pubs.acs.org/doi/abs/10.1021/jacs.6b06375>
5. Kane, C. M.; Banisafar, A.; Dougherty, T. P.; Barbour, L. J.; Holman, K. T. “Enclathration and Confinement of Small Gases by the Intrinsically 0D Porous Molecular Solid, Me₂H₂SiMe₂.” *J. Am. Chem. Soc.* **2016**, *138*, 4377-4392.
<http://pubs.acs.org/doi/abs/10.1021/jacs.5b11395>
4. Kane, C. M., Ugono, O.; Barbour, L. J.; Holman, K. T. “Many Simple Molecular Cavitands are Intrinsically Porous (Zero-Dimensional Pore) Materials” *Chem. Mater.* **2015**, *27*, 7337-7354.
<http://pubs.acs.org/doi/abs/10.1021/acs.chemmater.5b02972>
3. Joseph, A. I.; Lapidus, S. H.; Kane, C. M.; Holman, K. T. “Extreme Confinement of Xenon by Cryptophane-111 in the Solid State.” *Angew. Chem. Int. Ed.* **2014**, *54*, 1471.
<http://onlinelibrary.wiley.com/doi/10.1002/anie.201409415/abstract>
2. Kane, C. M.; Meyers, T. B.; Yu, X.; Gerken, M.; Etzkorn, M. “Bergman Cyclization of Fluorinated Benzo-Fused Eneidyne to Naphthalene Derivatives: Syntheses and Structures.” *Eur. J. Org. Chem.* **2011**, 2969-2980.
<http://onlinelibrary.wiley.com/doi/10.1002/ejoc.201001747/abstract>
1. Kane, C. M.; Drake, S. D.; Holman, K. T. “2-[4-(2,6-Dimethoxyphenyl)butyl]-1,3-dimethoxybenzene” *Acta Cryst.* **2010**, *E66*, o1921.

In Preparation

- Kane, C. M.; Ley, A. N.; Barbour, L. J.; Holman, K. T. “Rare Gas Permselectivity as Monitored by *in situ* Diffraction Analysis of a Dynamically 0D Porous Molecular Crystal.”
- Kane, C. M.; Banisafar, A.; Dougherty, T. P.; Holman, K. T. “A 0D Porous Cavitand Exhibits High CH₃Cl:CH₃OCH₃ Enclathration Selectivity.”
- Kane, C. M.; Tammela, P.; Lindh, J. “Flow Chemistry Synthesis of Pyrrole from Biomass-Derived Furfural”.

PRESENTATIONS

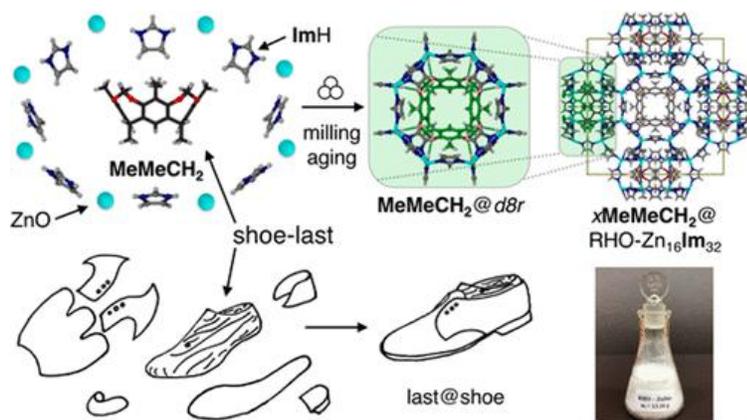
- C. M. Kane, Pulido, A.; Little, M. A.; McMahon, D. P.; Day, G.; Cooper, A. I. “*Using Energy-Structure-Function Maps to Predict Low-Density Solids.*” (Oral Presentation), MOF2018 – Auckland, New Zealand – December 2018
- C. M. Kane, K. Travis Holman “*Crystalline Organic Cavitands As Microcavity Materials.*” (Poster), 2014 ACS Midwest Regional Meeting, University of Missouri - Columbia – November 2014
- C. M. Kane, K. Travis Holman “*Crystalline Organic Cavitands Towards Microcavity Materials.*” (Selected Student Oral Presentation), Northeast Corridor Zeolite Association, Philadelphia, PA – December 2012
- C. M. Kane, A. Banisafar, T. P. Dougherty, K. Travis Holman “*Guest-Free Cavitands: Low Packing-Fraction Materials.*” (1st Place Poster), 2012 Bruker AXS/MIT Symposium Meeting, Boston, MA – February 2012
- C. M. Kane, A. Banisafar, T. P. Dougherty, L. J. Barbour, K. T. Holman: “*Guest-Free Cavitands: Low Packing-Fraction Materials.*” (Poster), 243rd ACS National Meeting, San Diego, CA – March 2012
- C. M. Kane, K. T. Holman: “*Guest-Free Cavitands: Low Packing-Fraction Materials.*” (Oral Presentation), 2011 ACA Meeting, New Orleans, LA – June 2011 (Travel Award)
- C. M. Kane, M. Etzkorn: “*Glycoluril-based Molecular Tweezers for Host-Guest Association Studies*” (Oral Presentation), 2008 Graduate Research Fair, UNCC (1st place)
- C. M. Kane, D. T. Nguyen, M. Etzkorn: “*Glycoluril-based Fluorinated Molecular Tweezers – Toward Extended Pincer Units*” (Poster), 235th ACS National Meeting, New Orleans, LA – April 2008
- C. M. Kane, S. B. Lewis: “*The synthesis of 1-hexylcyclobutene for synthetic studies with difluorocarbene*” (Oral Presentation), 21st National Conference For Undergraduate Research, San Rafael, CA - April 2007
- C. M. Kane, S. B. Lewis: “*The synthesis of 1-hexylcyclobutene for synthetic studies with difluorocarbene*” (Poster), 233rd ACS National Meeting, Chicago, IL – March 2007

REFERENCES

- Dr. Jonas Lindh, Uppsala University, +46 18 471 3073, Jonas.lindh@angstrom.uu.se
- Dr. K. Travis Holman, Georgetown University, +1 202 687 4027, kth7@georgetown.edu
- Dr. Len Barbour, University of Stellenbosch, +27 21 808 3355, ljb@sun.ac.za
- Dr. Markus Etzkorn, UNC-Charlotte, +1 704 687 1468, metzkorn@uncc.edu
- Dr. Marc Little, University of Liverpool, +44 151 795 7139, M.A.Little@liverpool.ac.uk

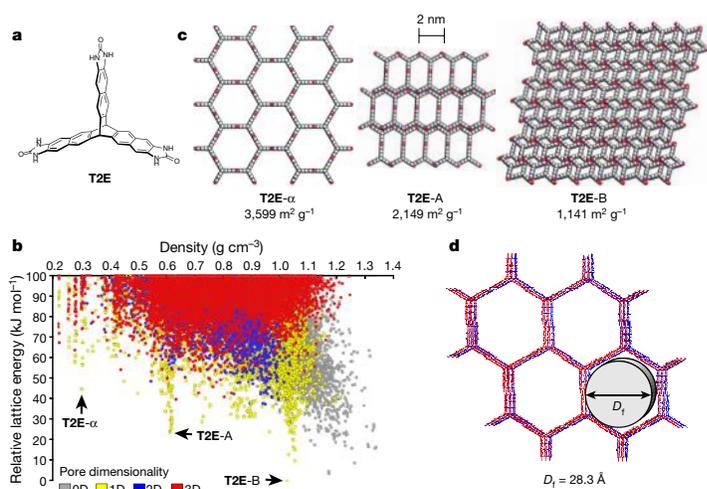
PUBLICATION HIGHLIGHTS

Brekalo, I.; Kane, C. M.; Ley, A. N.; Ramirez, J. R.; Friščić, T.; Holman, K. T. “Use of a ‘Shoe-last’ Solid-State Template in the Mechanochemical Synthesis of High-Porosity RHO-Zinc Imidazolate.” *J. Am. Chem. Soc.* **2018**, *140*, 10104-10108.



Abstract: We report the first use of a nonionic solid (NIS) as a template in mechanochemical synthesis of a metal–organic framework. Through eight intermolecular C–H···O hydrogen bonds, the macrocyclic MeMeCH₂ template predictably functions as a “shoe-last” for the assembly of double-eight rings in the liquid-assisted reaction of ZnO and imidazole (ImH). The resulting new form of ZnIm₂ (namely xMeMeCH₂@RHO-Zn₁₆Im₃₂) is available in multigram amounts, highly porous, and thermally stable.

Pulido, A.; Chen, L.; Kaczorowski, T.; Holden, D.; Little, M. A.; Chong, S. Y.; Slater, B. J.; McMahon, D. P.; Bonillo, B.; Stackhouse, C. J.; Stephenson, A.; Kane, C. M.; Clowes, R.; Hasell, T.; Cooper, A. I.; Day, G. M. “Functional materials discovery using energy-structure-function maps.” *Nature* **2017**, *543*, 657-664.

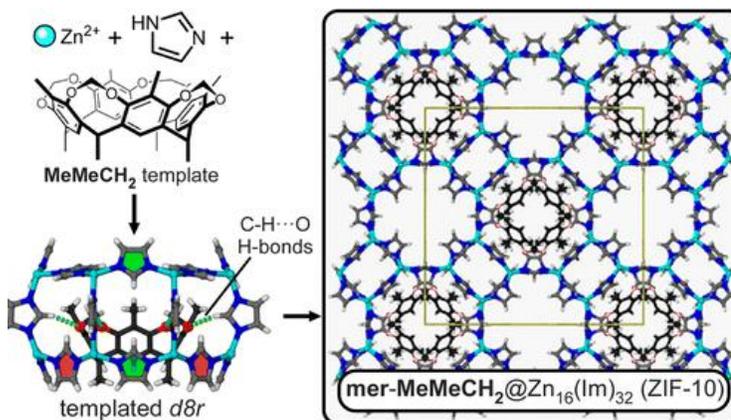


Abstract: Molecular crystals cannot be designed in the same manner as macroscopic objects, because they do not assemble according to simple, intuitive rules. Their structures result from the balance of many weak interactions, rather than from the strong and predictable bonding patterns found in metal–organic frameworks and covalent organic frameworks. Hence, design strategies that assume a topology or other structural blueprint will often fail. Here we combine computational crystal structure prediction and property prediction to build energy–structure–function maps that describe the possible structures and properties that are available to a candidate molecule. Using these maps, we identify a highly porous solid, which has the

lowest density reported for a molecular crystal so far. Both the structure of the crystal and its physical properties, such as methane storage capacity and guest-molecule selectivity, are predicted using the molecular structure as the only input. More generally, energy–structure–function maps could be used to guide the experimental discovery of materials with any target function that can be calculated from predicted crystal structures, such as electronic structure or mechanical properties.

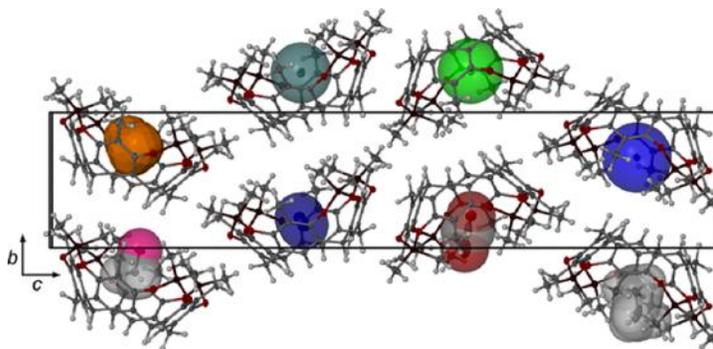
Ramirez, J. R.; Yang, H.; Kane, C. M.; Ley, A. N.; Holman, K. T. “Reproducible Synthesis and High Porosity of mer-Zn(Im)₂ (ZIF-10): Exploitation of an Apparent Double-Eight Ring Template.” *J. Am. Chem. Soc.* **2016**, *138*, 12017-12020.

Abstract: Reproducible synthesis of the elusive merlinoite (**mer**) topology of zinc imidazolate (**mer**-Zn(Im)₂, or ZIF-10) has been achieved by employing a simple macrocyclic solute—**MeMeCH₂**—as a kinetic template. The corresponding phase-pure material, **mer**-**MeMeCH₂**@Zn₁₆(Im)₃₂, is confirmed to be porous and exhibits one of the highest experimental surface areas (1893 m²/g, BET) yet reported for any ZIF. The X-ray single crystal structure of **mer**-**MeMeCH₂**@Zn₁₆(Im)₃₂·xsolvent reveals the role of the macrocycle as an 8-fold hydrogen bond acceptor in templating the requisite double-eight rings (*d8r*) of the **mer** framework.



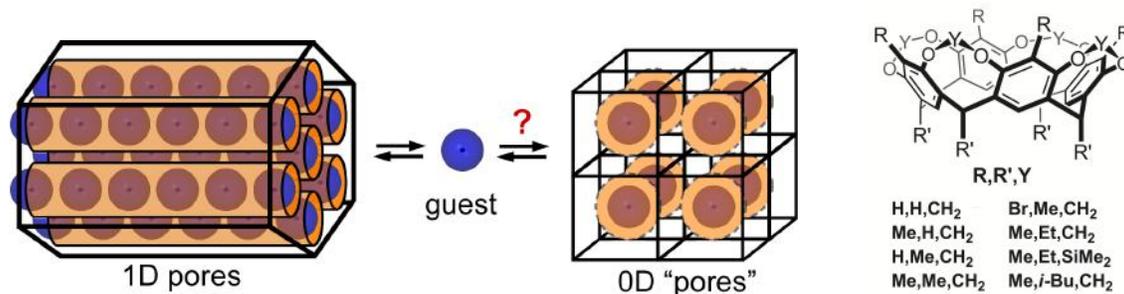
Kane, C. M.; Banisafar, A.; Dougherty, T. P.; Barbour, L. J.; Holman, K. T. “Enclathration and Confinement of Small Gases by the Intrinsically 0D Porous Molecular Solid, Me₃H₂SiMe₂.” *J. Am. Chem. Soc.* **2016**, *138*, 4377-4392.

Abstract: The stable, guest-free crystal form of the simple molecular cavitant, **Me₃H₂SiMe₂**, is shown to be intrinsically porous, possessing discrete, zero-dimensional (0D) pores/microcavities of about 28 Å³. The incompressible 0D pores of **Me₃H₂SiMe₂** have been exploited for the enclathration and room temperature (and higher) confinement of a wide range of small gases. Over 20 isostructural *x*(gas/guest)@**Me₃H₂SiMe₂** (*x* ≤ 1) clathrates (guest = H₂O, N₂, Ar, CH₄, Kr, Xe, C₂H₄, C₂H₆, CH₃F, CO₂, H₂S, CH₃Cl, CH₃OCH₃, CH₂Br, CH₂SH, CH₃CH₂Cl, CH₂Cl₂, CH₃I, CH₃OH, BrCH₂Cl, CH₃CH₂OH, CH₃CN, CH₃NO₂, I₂), and a propyne clathrate (CH₃CCH@**Me₃H₂SiMe₂**·2CHCl₃), have been prepared and characterized, and their single crystal structures determined.



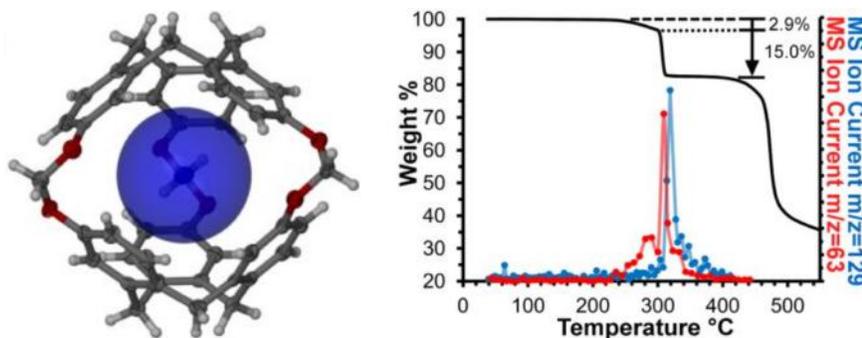
Gas enclathration is found to be highly selective for gases that can be accommodated by the predefined, though slightly flexible 0D pore. The structure determinations provide valuable insight, at subangstrom resolution, into the factors that govern inclusion selectivity, gas accommodation, and the kinetic stability of the clathrates, which has been probed by thermal gravimetric analysis. The activation (emptying) of several clathrates (guest = H₂O, N₂, CO₂, Kr, CH₃F) is shown to occur in a single-crystal-to-single-crystal (SC → SC) fashion, often requiring elevated temperatures. Akin to open pore materials, water vapor and CO₂ gas are shown to be taken up by single crystals of empty **Me₃H₂SiMe₂** at room temperature, but sorption rates are slow, occurring over weeks to months. Thus, **Me₃H₂SiMe₂** exhibits very low, but measurable, gas permeability, despite there being no obvious dynamic mechanism to facilitate gas uptake. The unusually slow exchange kinetics has allowed the rates of gas (water vapor and CO₂) sorption to be quantified by single crystal X-ray diffraction. The data are well fit to a simple three-dimensional diffusion model.

Kane, C. M.; Ugono, O.; Barbour, L. J.; Holman, K. T. "Many Simple Molecular Cavitands Are Intrinsically Porous (Zero-Dimensional Pore) Materials." *Chem. Mater.* **2015**, *27*, 7337-7354.



Abstract: Guest-free crystal structures of eight related small molecule cavitands (Scheme 1; simplified nomenclature: **R,R',Y**) are reported. The compounds are studied as candidate discrete molecule microcavity materials (DMMMs) under the hypothesis that, due to their rigid bowl-like molecular structures, many cavitands ought to be incapable of efficient crystal packing in pure form. Emphasis is placed on the engineering of structures that exhibit uniform microcavities that are large enough to potentially accommodate small molecules of interest (*e.g.*, gases or volatile organic compounds) without necessitating a change in host crystal packing. The guest-free structures of several cavitands—namely, **H,H,CH₂**, **H,Me,CH₂**, α -**Me,H,CH₂**, **Me,Me,CH₂**, β -**Br,Me,CH₂**, **Me,Et,CH₂**, **Me,Et,SiMe₂**, and **Me,*i*-Bu,CH₂**—appear to be as-close-packed-as-possible, yet exhibit relatively large microcavities (or, zero-dimensional pores) in the range of 27-115 Å³. Except for **Me,Et,CH₂**, the microcavities generally involve the intrinsic cavitand molecular cavity, yet the ultimate size and shape of cavities are also strongly influenced by crystal packing. It is suggested that these DMMMs may be particularly suited to the formation of stable gas clathrates and may find application in small molecule separations and/or gas storage. It is demonstrated that some cavitand solvates, namely **CH₂Cl₂@H,Me,CH₂**, x **H₂O@Me,Et,SiMe₂**, and **CH₂Cl₂@Me,*i*-Bu,CH₂** (84:16 *rcccr:rcct*) maintain host packings that are more or less identical to their empty, intrinsically porous host phases. Crystals of x (H₂O)@**Me,Et,SiMe₂** are dehydrated in a single-crystal-to-single-crystal fashion and are therefore permeable to water. The structures and thermal properties of several other new cavitand solvates are also reported.

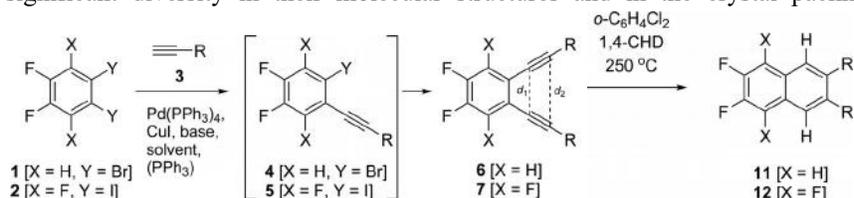
Joseph, A. I.; Lapidus, S. H.; Kane, C. M.; Holman, K. T. "Extreme Confinement of Xenon by Cryptophane-111 in the Solid State." *Angew. Chem. Int. Ed.* **2015**, *54*, 1471-1475.



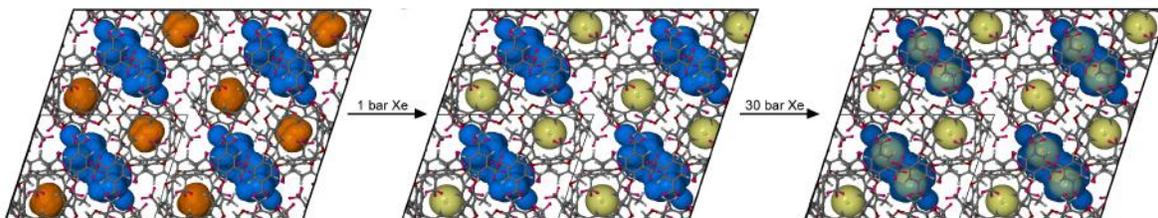
Abstract: Solids that sorb, capture and/or store the heavier noble gases are of interest because of their potential for transformative rare gas separation/production, storage, or recovery technologies. Herein, we report the isolation, crystal structures, and thermal stabilities of a series of xenon and krypton clathrates of (\pm)-cryptophane-111 (**III**). One trigonal crystal form, **Xe@111 \cdot y(solvent)** (1,2-dichloroethane), is exceptionally stable, retaining xenon at temperatures of up to about 300°C (above). The high kinetic stability is attributable not only to the high xenon affinity and cage-like nature of the host, but also to the crystal packing of the clathrate, wherein each window of the molecular container is blocked by the bridges of adjacent containers, effectively imprisoning the noble gas in the solid state. The results highlight the potential of discrete molecule materials exhibiting intrinsic microcavities or zero-dimensional pores.

Kane, C. M.; Meyers, T. B.; Yu, X.; Gerken, M.; Etzkorn, M. "Bergman Cyclization of Fluorinated Benzo-Fused Enediynes to Naphthalene Derivatives: Syntheses and Structures." *Eur. J. Org. Chem.* **2011**, 2969-2980.

Abstract: Fluorinated naphthalene derivatives were prepared by Bergman cyclization of fluorinated benzo-fused enediynes. This route provides access to the aromatic target compounds in a two-step procedure from commercially available precursors, via a Sonogashira cross-coupling and a subsequent, thermally initiated Bergman cyclization. Crystal structures of six fluorinated benzo-enediynes and three fluorinated naphthalene derivatives display significant diversity in their molecular structures and in the crystal packing arrangements. The rather subtle structural change from di- to tetrafluorobenzo-enediynes, as well as the variation in the terminal acetylene subunit led to different non-covalent interactions in the solid state that ultimately govern their crystal structures.



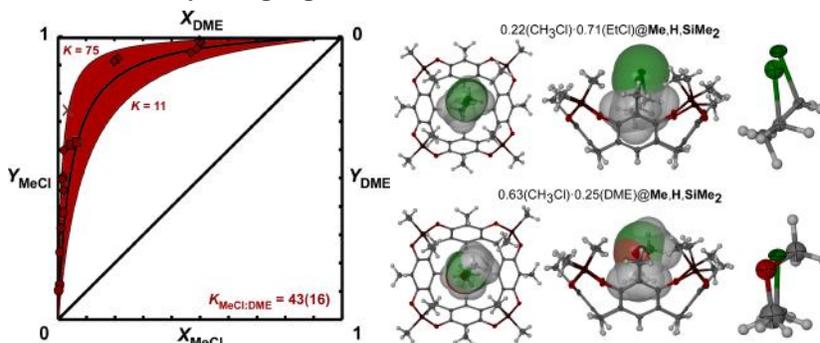
Kane, C. M.; Ley, A. N.; Barbour, L. J.; Holman, K. T. "Rare Gas Permselectivity as Monitored by *in situ* X-ray Diffraction Analysis of a Dynamically 0D Porous Molecular Crystal," *in preparation*.



Synopsis: Porous materials have proven to be fruitful in gas storage and separations applications due to their "accessibility by external fluids". It has recently been noted however, that microcavity materials (*e.g.* cavitands), which do not possess molecular scale channels throughout their packing, may provide greater enthalpic binding capabilities for volatile gaseous species due to greater intermolecular contacts between gas and host. Though there is no pore leading to the cavities that are present in such materials, complexation of guests may still be achieved on a reasonable timescale by using dynamic functional groups (-CF₃ or *t*-bu). Most interestingly, we have developed a cavitand material (**Me₃,5-CF₃Ph,CH₂ *rctt***) that possesses large, interpenetrating cavities and have investigated its ability to sorb various gases [N₂, O₂, CO₂, Ar, Kr, Xe] by bulk phase sorption experiments and *in situ* single crystal x-ray diffraction.

Kane, C. M.; Banisafar, A.; Dougherty, T. P.; Holman, K. T. "A 0D Porous Cavitand Exhibits High CH₃Cl:CH₃OCH₃ Enclathration Selectivity," *in preparation*.

Synopsis: Materials that have isolated cavities (*e.g.* cavitands) as opposed to open, accessible pores have been shown to boast unusual properties. We have begun to assess the potential for use of the cavitand **Me₃H,SiMe₂** in the industrial separation of dimethyl ether and chloromethane. These gases inevitably appear as a mixture by the action of hydrochloric acid on methanol and current methods of



separation call for the wasteful destruction of CH₃OCH₃ by acids, even though DME is a useful commodity. Efforts to quantify the selectivity of chloromethane (over DME) by **Me₃H,SiMe₂** are discussed.