UCSB
Department of Chemistry and Biochemistry

# Mobility Studies and 3-D Structural Characterization of POSS Compounds

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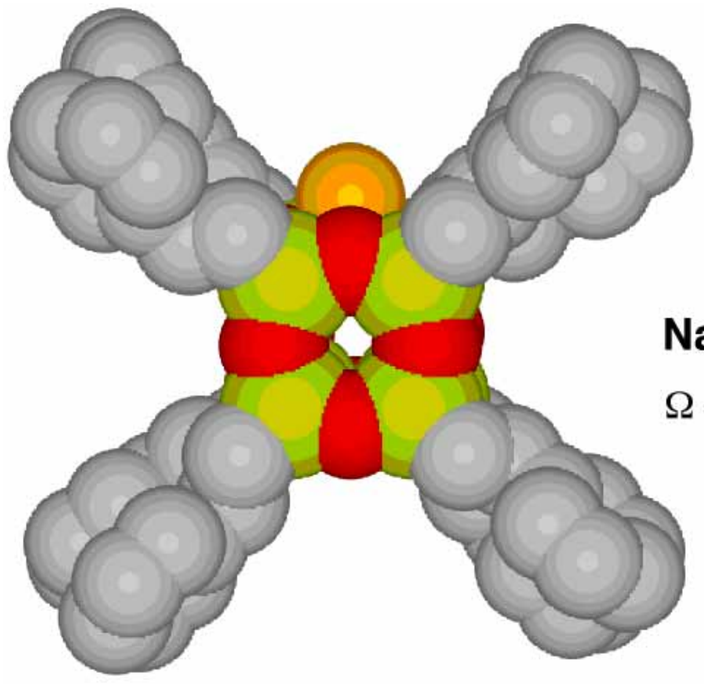
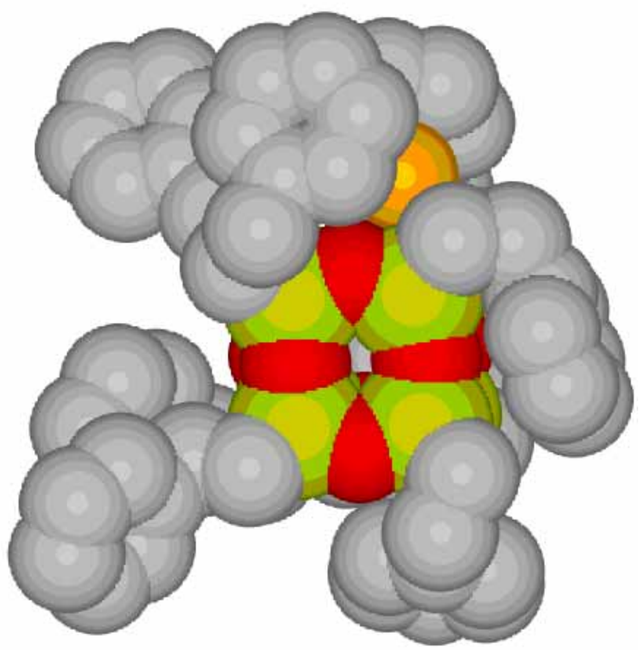


**$Na^+Sty_8T_8$**

$\Omega$ = 338 Å$^2$



**$Na^+PhenEt_8T_8$**

$\Omega$ = 266 Å$^2$

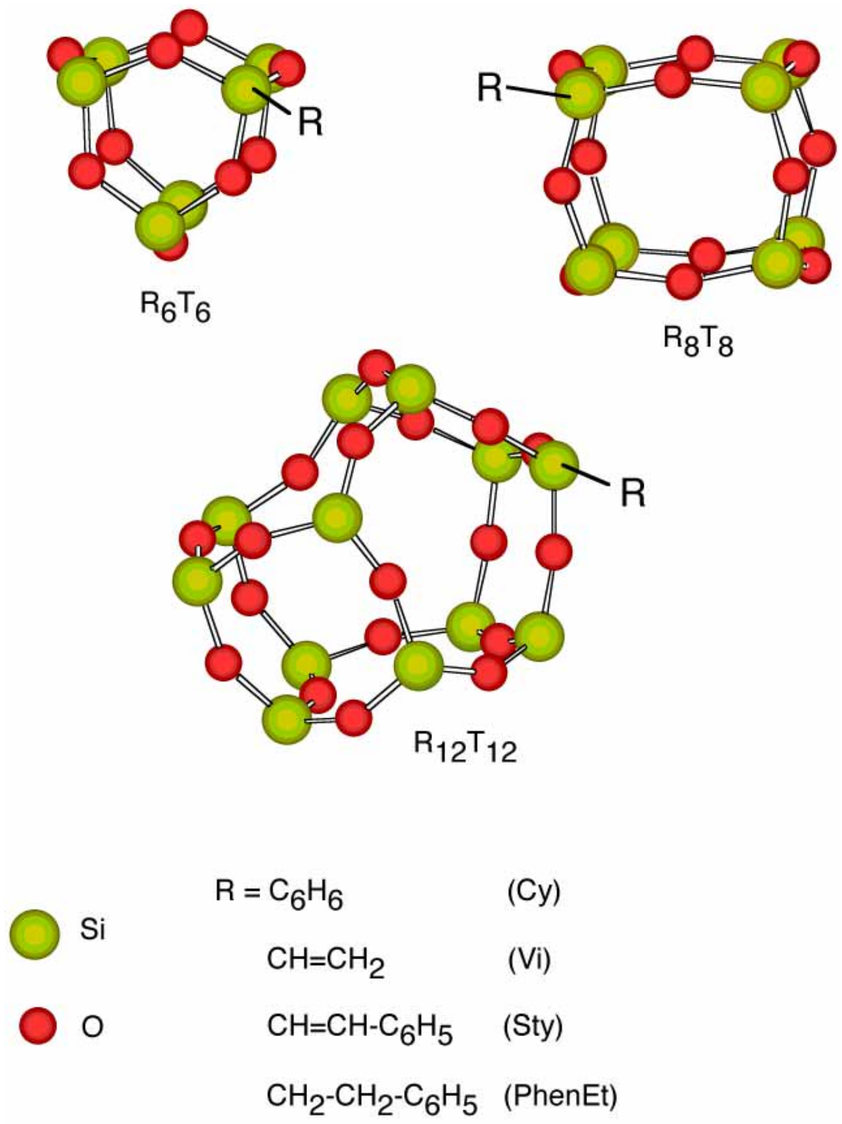
# INTRODUCTION

Polyhedral Oligomeric Silsesquioxanes (POSS)

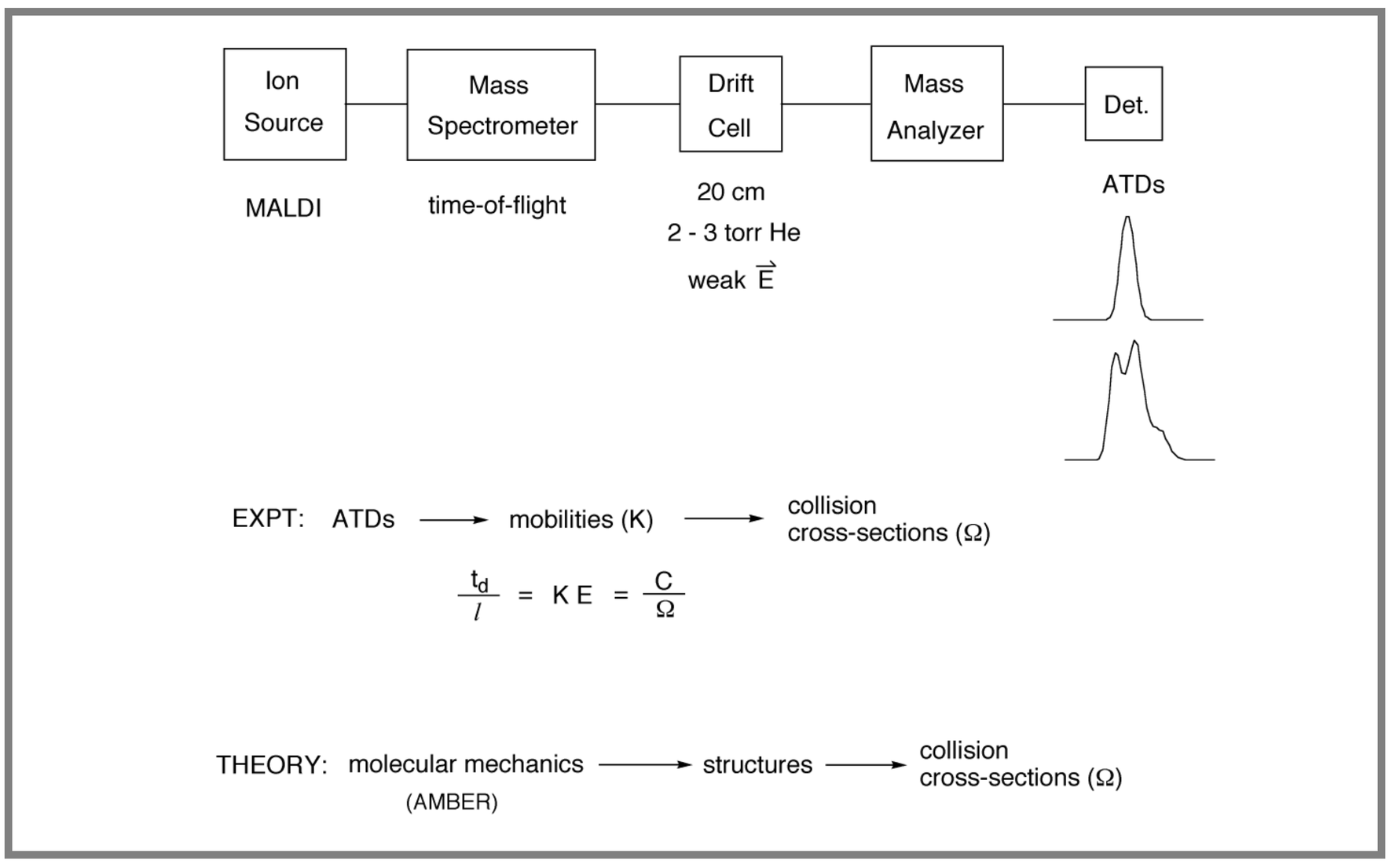
- have a thermally and chemically robust hybrid silicon-oxygen framework of the form $(RSiO_{3/2})_n$
- use organic substituents such as esters, epoxies, methacrylates, siloxanes and nitriles to add functionality
- are incorporated into traditional polymer systems to increase thermal and chemical stability of polymers, and to control physical and mechanical properties of polymers
  - addition of POSS to polymers offers:
    - extend temperature ranges
    - reduce flammability
    - lower thermal conductivity
    - reduced viscosity
    - lower density
- improve high performance polymers and transform commodity polymers into high performance polymers

Understanding physical conformations of the substituted POSS is essential in tailoring both application and synthetic processes.

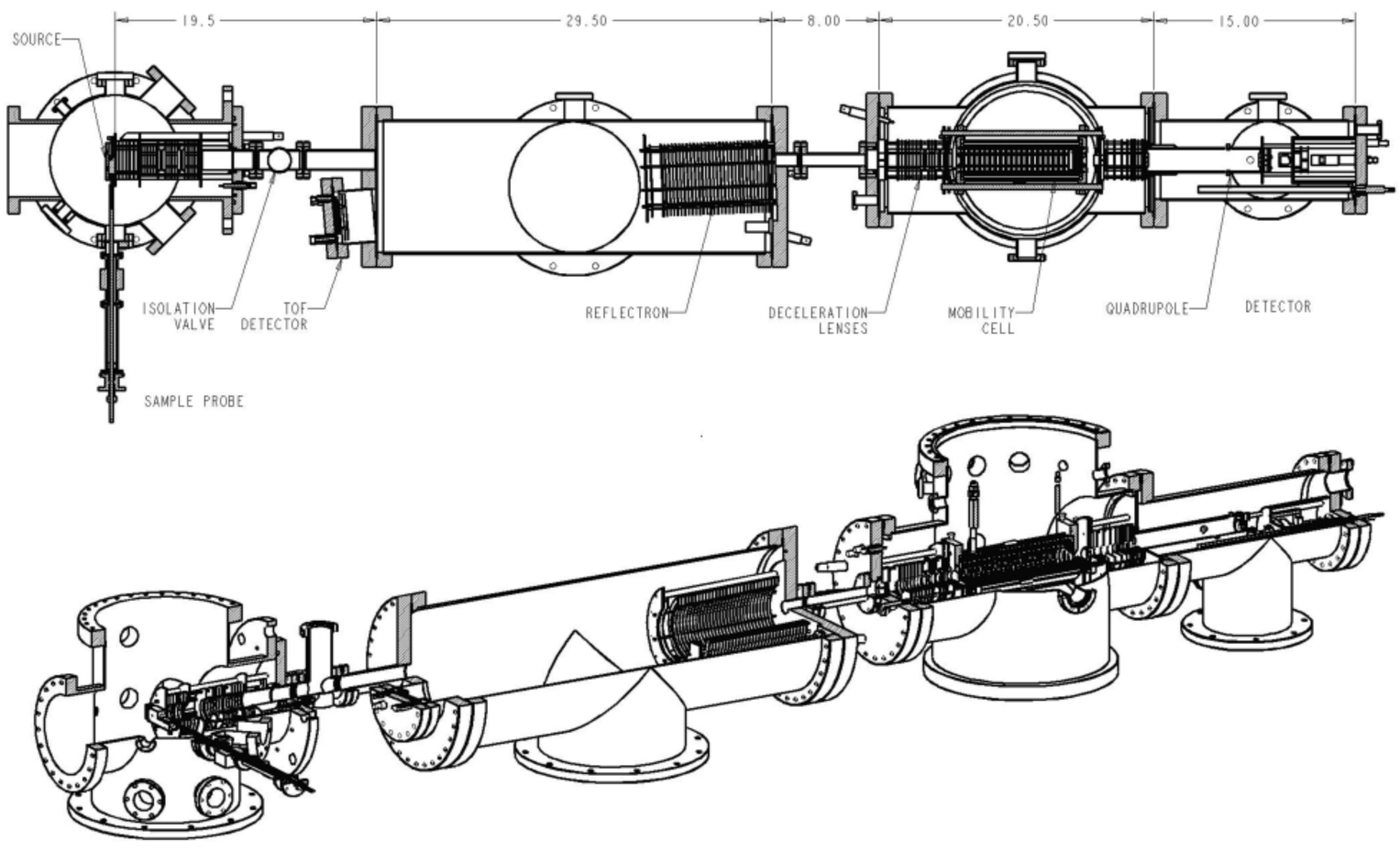
## Sample POSS Cages



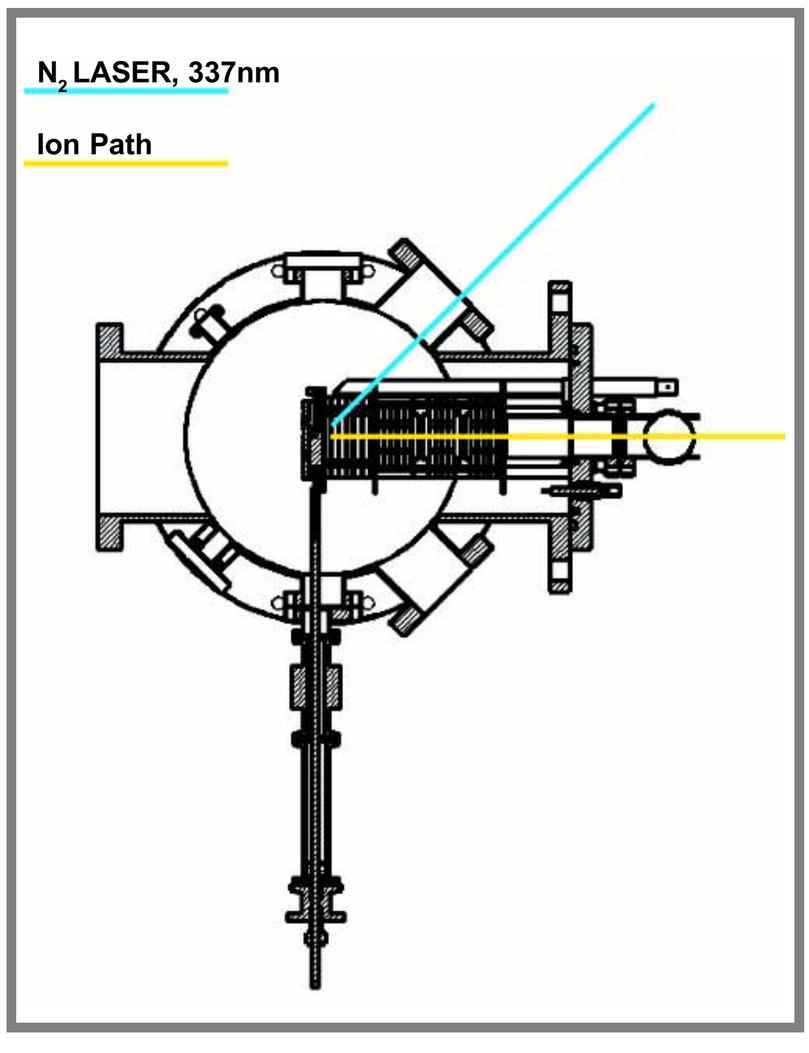

## Procedure Overview



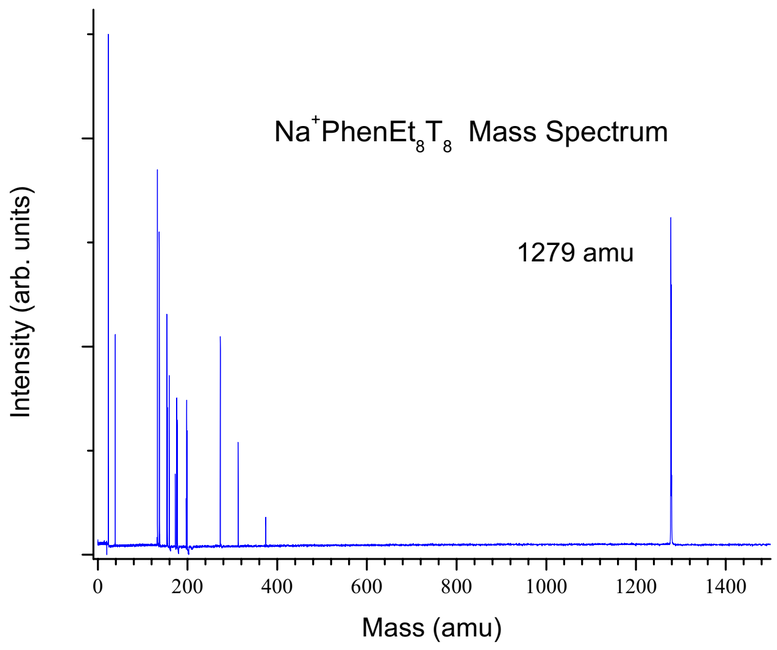

## Matrix Assisted LASER Desorption/Ionization and Time of Flight Mass Spectrometer

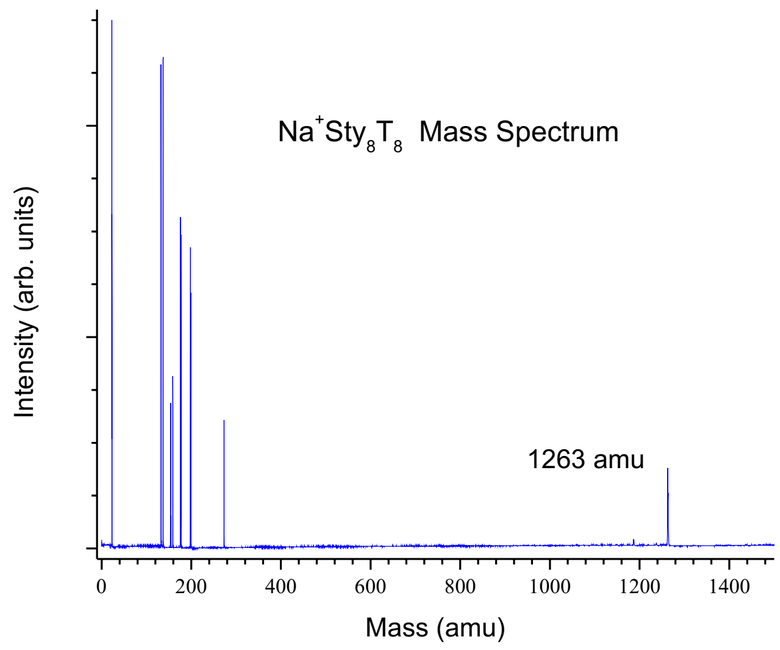



## MALDI Source

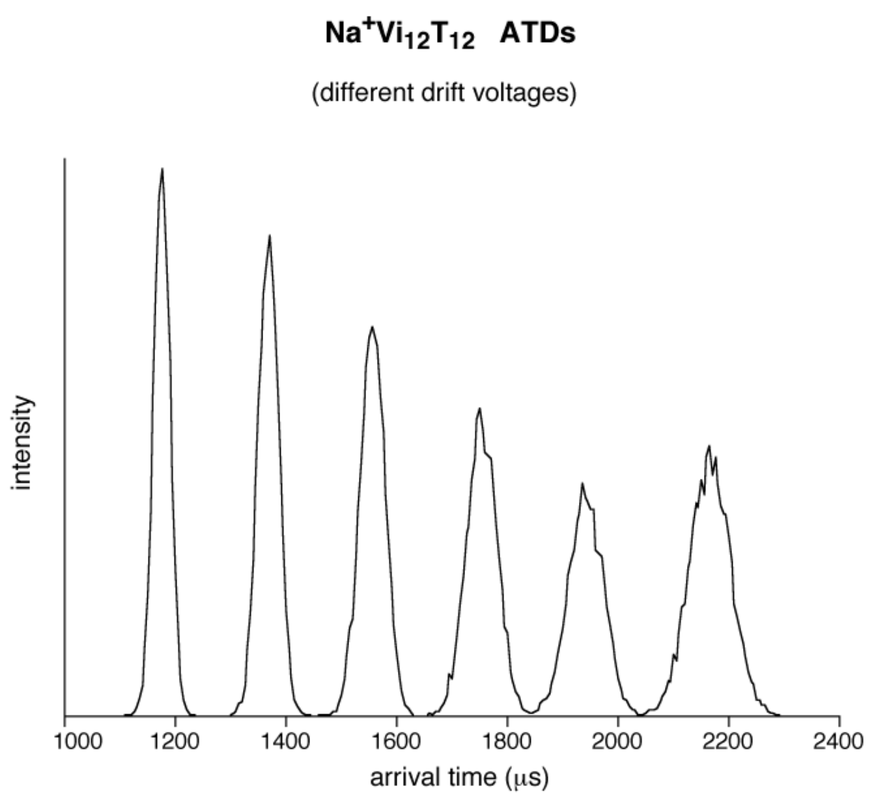


# Time of Flight (TOF) Mass Spectra

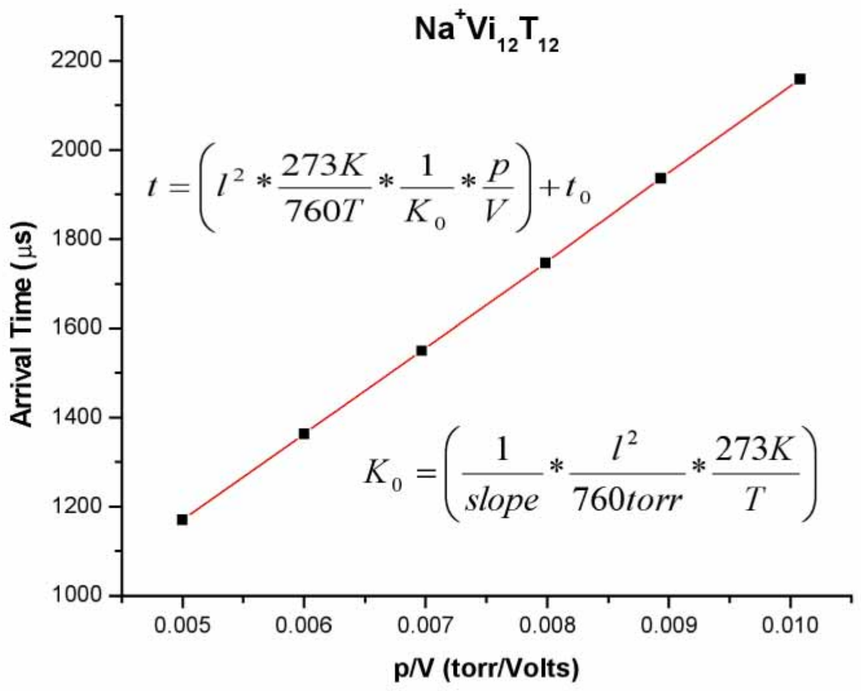


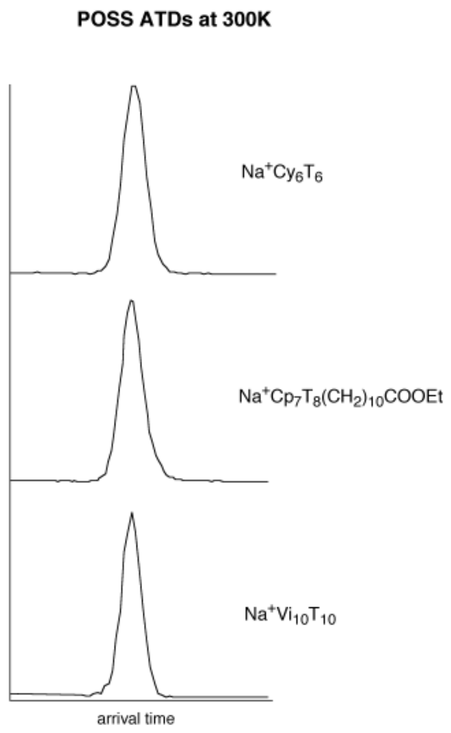

## Effect of Drift Voltage on ATD's




## Obtaining Mobility ($K_0$) from ATD Data




## More Sample ATD's




## Experiment vs. Theory

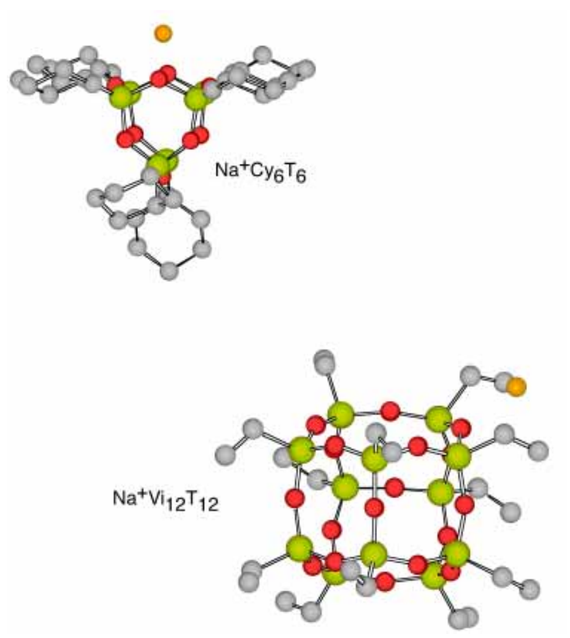
POSS cross-sections (Å$^2$)

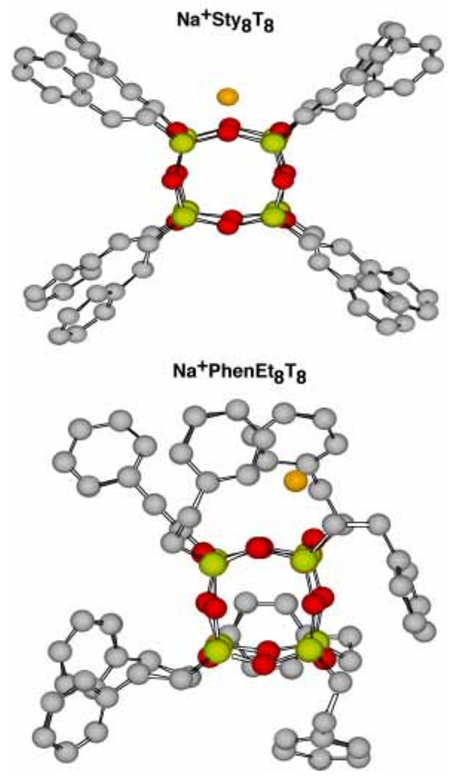
| | x-ray* | MALDI -TOF | theory |
|---|---|---|---|
| | | $Na^+$ | $Na^+$ |
| $Cy_6T_6$ | 224 | 227 | 220 |
| $Cp_7T_8(CH_2)_{10}COOEt$ | | 273 | |
| $PhenEt_8T_8$ | | 267 | 266 |
| $Vi_{10}T_{10}$ | | 193 | 193 |
| $Vi_{12}T_{12}$ | 212 | 218 | 215 |

* from Tim Haddad at ERC Inc., Air Force Research Laboratory

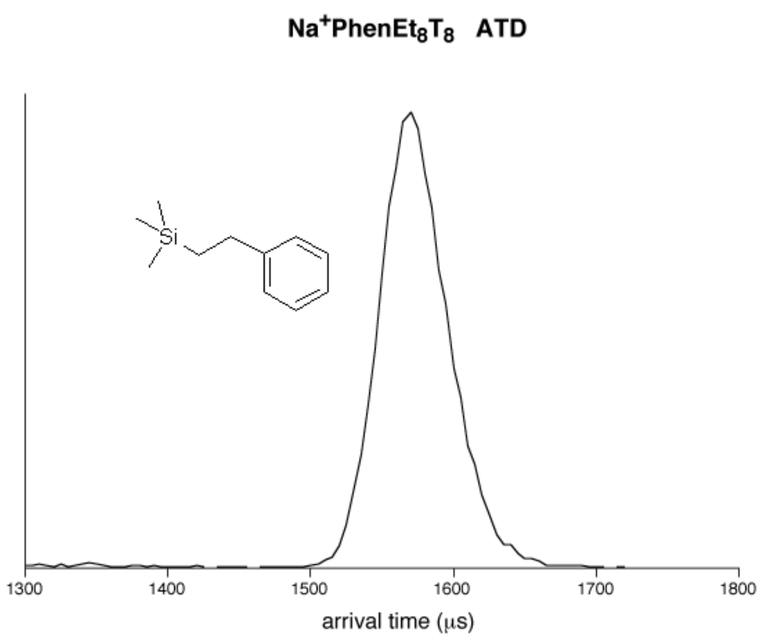
# Minimum Energy Structures calculated by AMBER

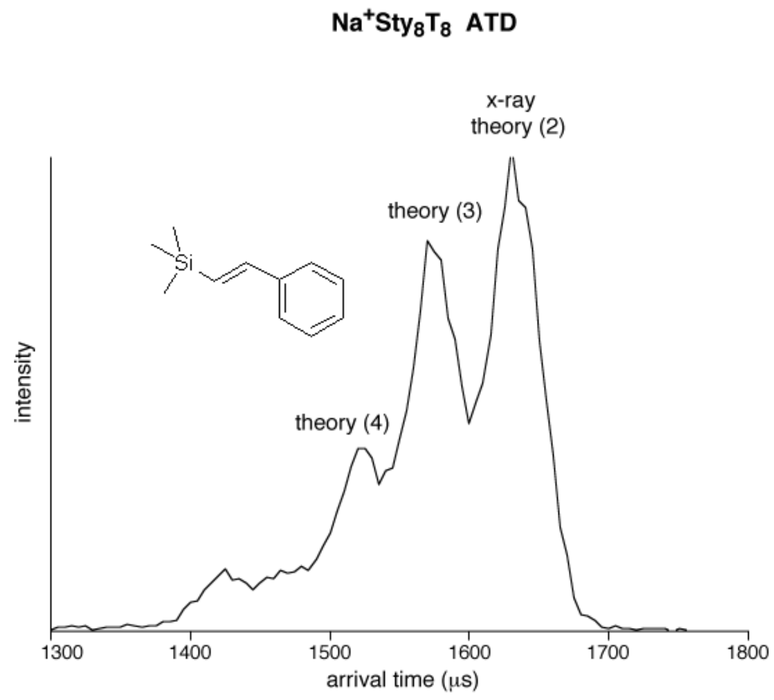
### AMBER Molecular Mechanics software Package, Peter Kollman, UCSF



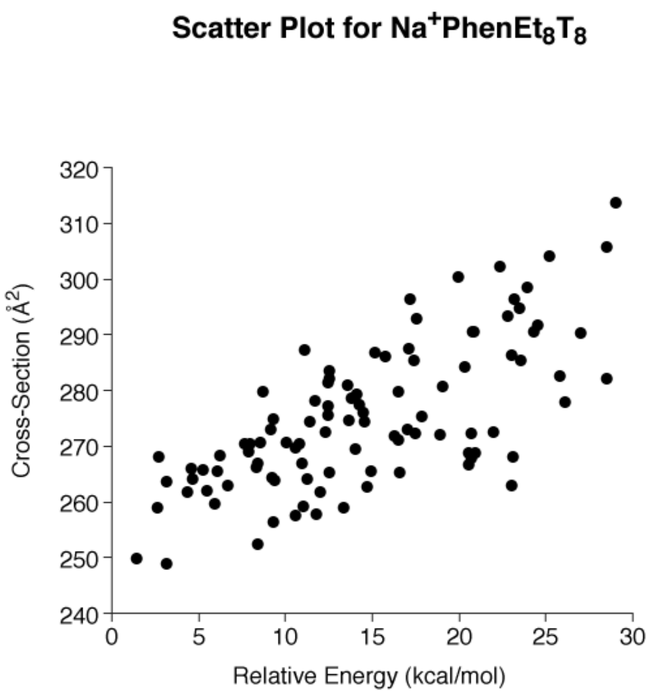



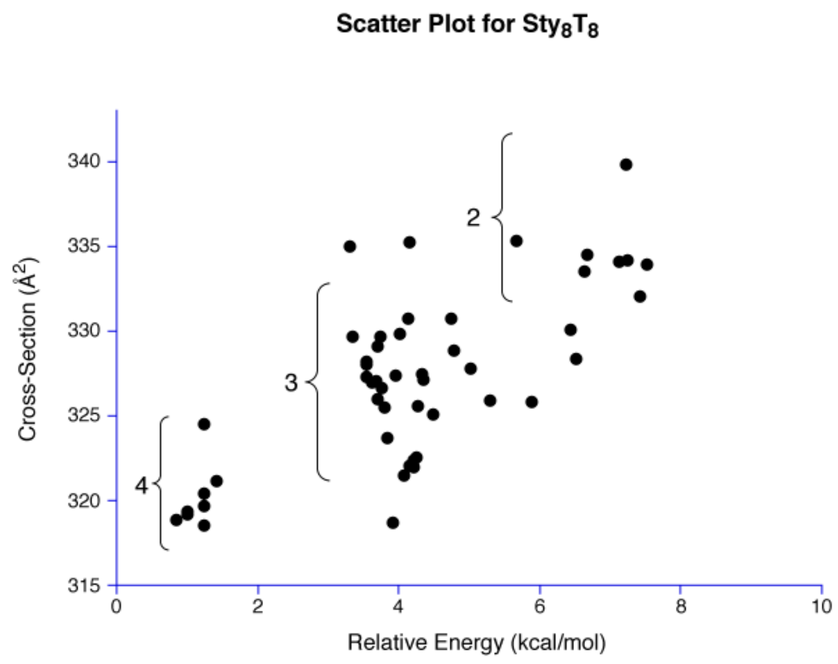
# Arrival Time Distributions (ATD's)



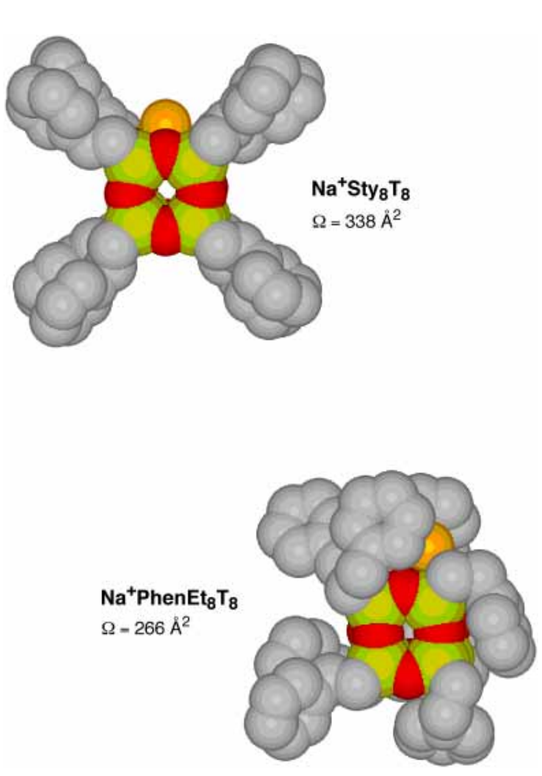



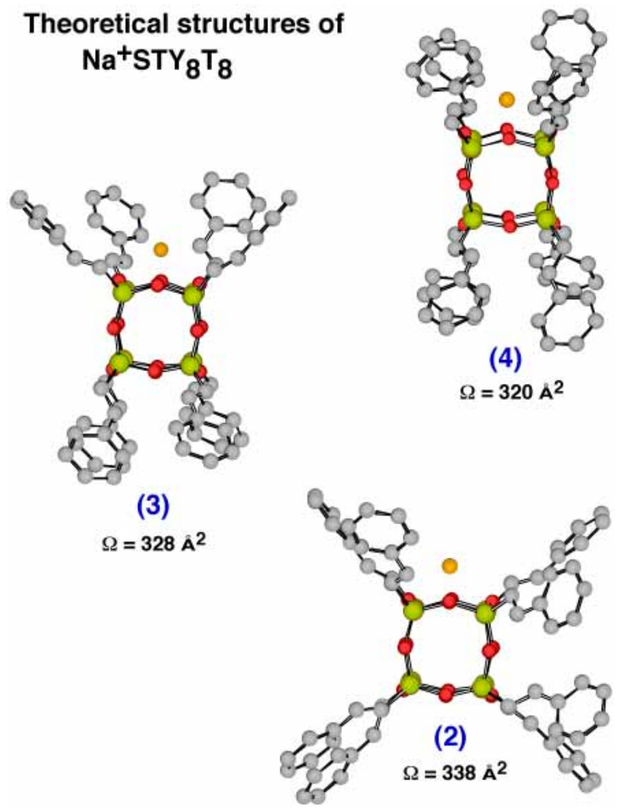

# Scatter Plots (Cross Section vs. Energy)




# Minimum Energy Structures (AMBER)




# SUMMARY

- Experimental results for collision cross sections show good agreement with both X-ray crystal structures and theoretical models.
- This technique reveals and identifies structural detail. In the case of the styrene-capped system $Sty_8T_8$, 3 main peaks were observed experimentally and theoretical calculations produced structures with matching cross sections.
- Unexpectedly large cross section and structural changes observed with small chemical change. $PhenEt_8T_8$ was found to have a cross section 30% smaller than that of $Sty_8T_8$.
- We are confident in our results for POSS systems, as well as in our previous work on polymer systems. We look forward now to structural study of POSS-modified polymer systems.