Exp. 1 (Text #3): The Molecular Sieve Zeolite X

Lab Work 1/24
Report Due 2/6

What is a zeolite?

- A microporous solid, containing pores or channels in its structure that can accommodate guest molecules

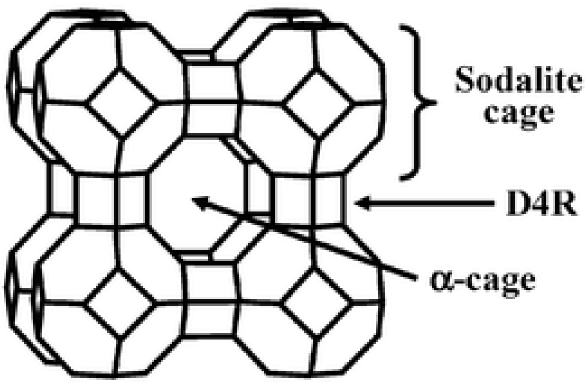
- An aluminosilicate

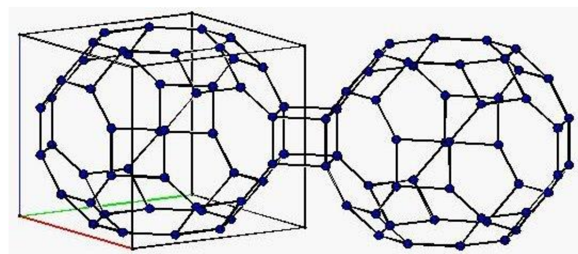
  - Framework stoichiometry: $(Si, Al)_nO_{2n}$
  - Si or Al atoms are tetrahedrally coordinated to bridging O's ("vertex-sharing" tetrahedra)
  - Cations (e.g., Na^+, K^+) *required for charge balance*

    Si^{4+} vs. $(Al^{3+} + Na^+)$

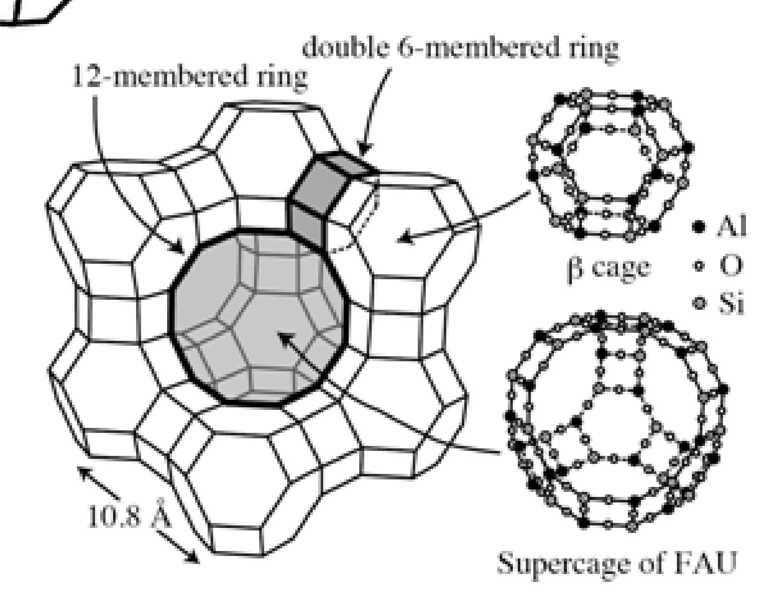
Zeolite Structure

Several aluminosilicate structures are based on a **truncated octahedron** with stoichiometry $\mathbf{M_{24}O_{36}}$ (where M = Si, Al), also called the **sodalite** or β **cage**:





Zeolite A
(showing connectivity of "octahedra")

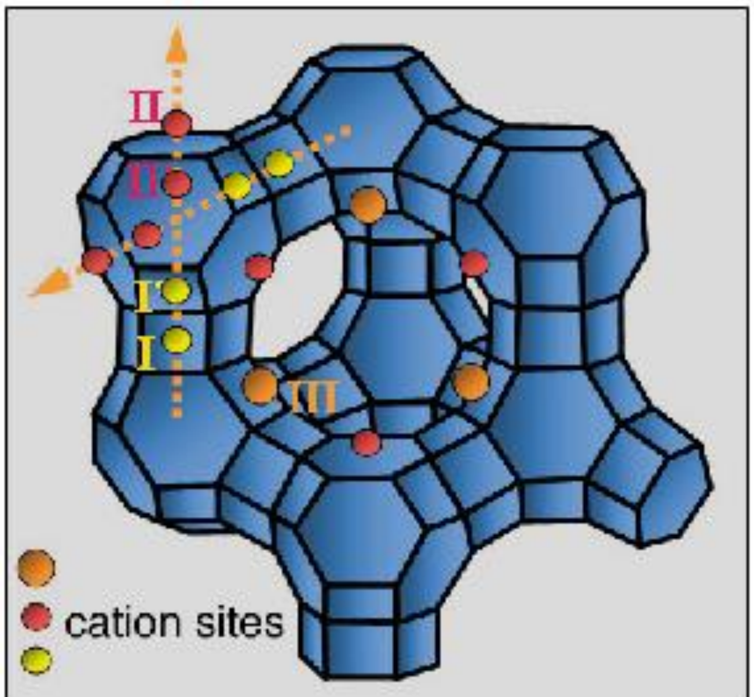


Zeolite X

Zeolite Structure, continued

Cations occupy numerous sites within the framework, and help to determine the size of the pores (α- or supercage).

- Also influenced by Si/Al ratio



We will use Na^+ to balance charge, so the **hydrated sodium ion** helps determine pore size.

How would pore size change if K^+ were used, instead?

K^+ is larger; pore size would be smaller.

Applications of Zeolites

- Molecular sieves (separation by size):
  - Desiccants/Adsorbents

- Ion exchange
  - Water softening

- Catalysis
  - Introduction of transition-metal ions affords numerous sites for catalytic reactions

NaX Synthesis and Ion-Exchange:

Synthesis of NaX:

$$(24\text{-}n)\ SiO_2 + n\ NaAlO_2 \rightarrow Na_nSi_{24-n}Al_nO_{48}$$

n = 9.6 – 12 for X-type zeolites; For us, ***n* = 10.7** (pore size = 7.4 Å)

Completed by mixing prepared solutions of sodium aluminate and sodium silicate (Solutions 1 and 2 in text)

Characterization by IR spectroscopy (1/25): See Balkus, K. J. et al. *J. Chem. Educ.* **1991**, *68*, 875-877 for published spectra.

Ion-Exchange Reaction (1/25):

$$Na_nSi_{24-n}Al_nO_{48} + x\ CoCl_2 \rightarrow Co_xNa_{n-2x}Si_{24-n}Al_nO_{48} + 2x\ NaCl$$

What is the specific ion-exchange process that occurs here?

Uptake of 1 Co^{2+} results in release of 2 Na^+ ions

NaX Synthesis: Procedural Notes and Tips

- You will work in pairs on this experiment.
- We will perform the experiment at **50% scale**.
- Next week, we will complete Part A to the stopping point mentioned in the text (filtering NaX crystals and leaving them in your drawer to dry).
- Our aim is to allow 2 hours for reaction in the oven, ideally starting around 3:00. We cannot begin heating until everyone is ready.
    - Make water bath immediately and start heating (watch temp as directed)
    - Work on Solutions 1 and 2 simultaneously
    - Note that the specified masses are not very precise (e.g., 1.2 g). **Don't** waste time trying for 1.200 g; just record the exact mass you obtain.
- After cooling, you will suction-filter your product using a Buchner funnel and filter paper. Wash the crystals with ~3 portions of water and continue suction as long as possible.
- Be careful when removing crystals from filter paper.

Overview of Activities for Next Week

In Lab Next Week:

1. Determine total mass of dry NaX product

2. Acquire IR spectrum of solid NaX product

3. Perform cobalt-exchange reaction:

$$Na_{10.7}Si_{13.3}Al_{10.7}O_{48} + x\ CoCl_2 \rightarrow Co_xNa_{10.7-x}Si_{13.3}Al_{10.7}O_{48}$$

1. Percent Yield of NaX Product

You prepared two solutions – sources of alumina and silicate – and mixed them together to form NaX:

1. $Al(OC_3H_7)_3 \rightarrow NaAlO_2$

2. Silica gel $\rightarrow$ Silicate ("SiO_2")

Overall reaction:

$$13.3\ SiO_2 + 10.7\ NaAlO_2 \rightarrow Na_{10.7}Si_{13.3}Al_{10.7}O_{48}$$

How will you calculate the theoretical yield of NaX?

Find limiting reactants from preparation of Solutions 1 & 2 and for the overall reaction