❖ **How do you judge an X-ray structure's quality?**

➢ **Look at statistics table**

In **crystallography**, the **R-factor** (sometimes called residual **factor** or reliability **factor**or the **R-value** or R_{Work}) is a measure of the agreement between the **crystallographic**model and the experimental **X-ray diffraction** data. In other words, it is a measure of how well the refined structure predicts the observed data.

1) **Resolution**
   - **> 3.2 A:** approximate backbone and overall fold, poor side chains
   - **3.0 A:** a few errors in backbone, approximate side chains, atom positions to **0.5-1 A**
   - **2.5 A:** good backbone, some side chains errors, atom positions to **0.3-0.5 A**
   - **2.0 A:** good side chains, hydrogen bond distances, positions to **0.15-0.3 A**
   - **> 1.5:** precise atom locations, individual atoms as separate balls

2) **R-factor =** $\frac{\sum |F_o - F_c|}{\sum F}$ **, agreement between observed and calculated F_s (spot**

work	free	
>30		BAD
25-30	>30	marginal
20-25	25-30	acceptable
<20	<25	high quality

- when model matches the obs data, R-factor = 0
- anything about 50 % is weird => model doesn't match the data
- 50 % is random, but 49 % is NOT
- anything above 30% is bad, 25% marginal, 22% acceptable, anything below 20% is good
- small R-factor = high resolution

*** Discussion about linear fits, parabola, and models/prediction

Free R factor

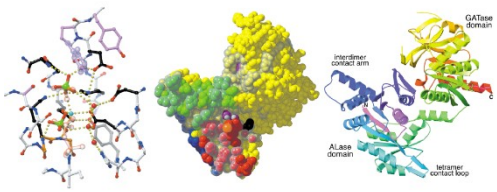
A **residual function** calculated during structure refinement in the same way as the conventional *R* factor, but applied to a small subset of reflections that are not used in the refinement of the structural model.

R free will always be greater than **R normal** because the model is not fitted to the reflections that contribute to R free, but the two statistics should be similar because a correct model should predict *all* the data with uniform accuracy

Point is that you can **get a good R factor**, but have a **wrong model** because
1) Poor Resolution
2) Bad Data
3) Overfit the Data (too many parameters and not enough info)

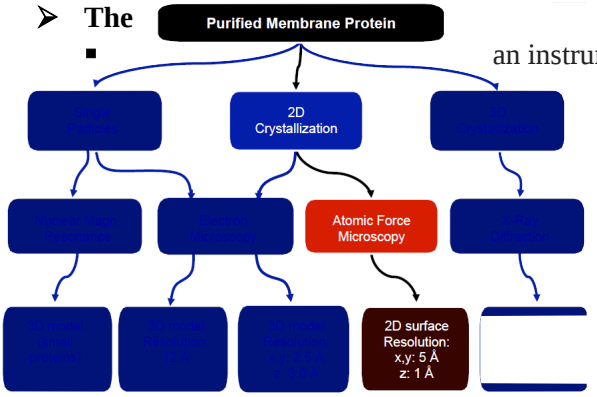
3) **Acceptable geometry deviations**
   - Angles < **2.5** °
   - Bonds < **0.2 A**

Different Structural Representation



Ball-and-stick Space-filling (CPK) Ribbon

➢ **The**



Scanning Tunneling Microscope

- an instrument for imaging surfaces at the atomic level, atom by atom
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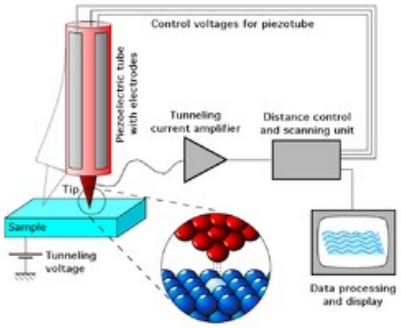
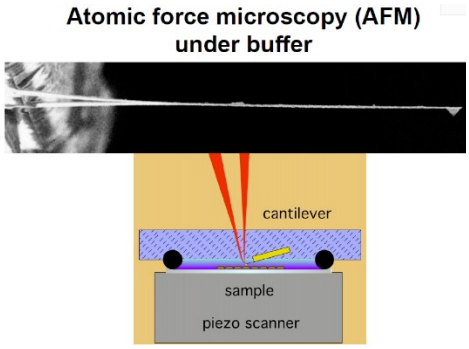
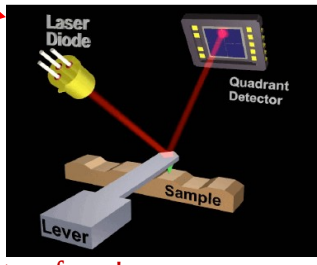


➢ **The Atomic Force Microscope**



- resolution on the order of fractions of a nanometer
- information is gathered by "feeling" or "touching" the surface with a mechanical probe
- can use it with bio samples because it actually touch the surface (STM cannot because it is based on conductivity)
- can have **different modes**

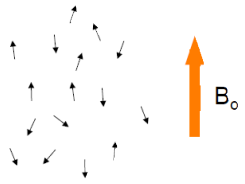


- **Constant Height Mode**
- **Constant Force Mode**
- **Friction Mode**
- **Tapping Mode**
- **Functionalized Cantilevers**

*** Assume a flat surface when you work with this! Crystals have flat surfaces!

❖ **Nuclear Magnetic Resonance (NMR) Spectroscopy**

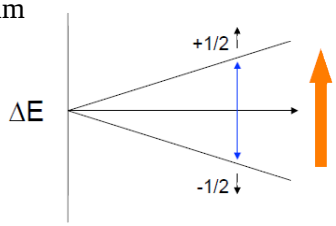
a spectroscopic technique to observe local magnetic fields around atomic nuclei. The sample is placed in a magnetic field and the NMR signal is produced by excitation of the nuclei sample with radio waves into nuclear magnetic resonance, which is detected with sensitive radio receivers. The intramolecular magnetic field around an atom in a molecule changes the resonance frequency, thus giving access to details of the electronic structure of a molecule and its individual functional groups.



➢ Certain isotopes have an intrinsic atomic momentum

➢ With no magnetic field, these are **randomly oriented**

➢ With magnetic field, they tend to **align with or against the field** according to their nuclear spin quantum number($\mp\frac{1}{2}$)



- Now there are two populations of different spins, with different energies depending on their alignment