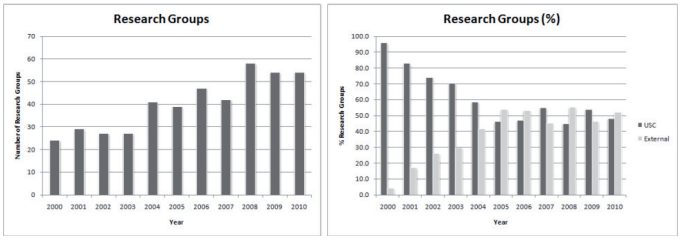
as a general crystallographic service to the scientific community either at USC or at any other national or international research organization or industry.

The X-Ray Unit at the USC offers nowadays the following techniques:

- Single crystal diffraction for all size of molecules.
- General X-Ray diffraction: powders, micro-diffraction, high resolution, grazing incidence.
- Small Angle Scattering (SAXS).
- Reflectometry.
- Energy dispersive X-Ray fluorescence.

Gathering all X-Ray related techniques in one place has many advantages: easier management in a homogeneous environment, better planning and future development, more synergy and complementarity between different techniques due to close collaboration among specialized personnel, low running costs and finally even the possibility to offer specific solutions to scientific questions asked by the researchers instead of individual techniques alone.

The X-Ray Unit facilitates the application of crystallographic techniques to inexperienced users and to an increasing number of research groups working in other fields that were not traditionally linked to crystallography. For example, the evolution with time of the number of research groups demanding single crystal diffraction experiments is displayed in the graphs below.



The main tasks to achieve are: keep routine techniques available, implement the most demanded ones, advice users to help them to solve specific scientific problems, provide training to enable them to perform their own experiments and data analysis, disseminate the results obtained, spread the capabilities of the techniques and finally implement and develop new methodological approaches to solve non-routine problems (disorder, twins, unstable crystals, macromolecules, etc.).

Keywords: service, X-ray, crystallography

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Improved DFT calculation of raman spectra of silicates and similar compounds

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With advances in modern technology, Raman spectrometers have become smaller, cheaper, and easier to use. As a result, Raman spectroscopy is nowadays a standard method in the characterization of materials. Beyond simple fingerprinting, vibrational analysis is extremely useful for obtaining information about structural features of molecules and solids. The theoretical tools used for vibrational analysis have also advanced very much in recent years. Among the most powerful is certainly the *ab initio* calculation of vibrational spectra from the structure alone, using quantum-mechanical methods. The benefit of this approach is not so much in the calculated frequencies themselves, which after all are easily gained from the experimental spectrum. But the calculations yield additional information about the mode symmetries and the movements of the atoms involved in the particular vibrations. These can be used to understand changes in frequencies or other features of the experimental spectrum, like peak intensity or broadening, that result from changes in pressure, temperature, or chemical composition. Furthermore, deviations from the ideal crystal structure and its expected spectrum can be identified, and the presence of impurity phases can be spotted.

In order to join the information from the experimental spectrum with the information from the calculation, it is necessary to assign the observed bands to specific calculated modes. The foremost criterion used in this context is the agreement of the observed and calculated frequencies. To allow for an unequivocal assignment, the uncertainty of both values needs to be lower than the distance between neighboring modes. For moderately complex silicates, this is ≈ 20 cm^{-1} on average, and considerably less in some regions of the spectrum. Experimentally, this level of accuracy does not present a problem. Modern Raman spectrometers reach spectral resolutions of 2 cm^{-1} and accuracies of 0.5 cm^{-1}.

On the theoretical side, very accurate vibrational spectra of silicates are obtained from DFT calculations if the appropriate Hamiltonian is used. For silicates and compounds with similar electronic character, theoretical considerations [1,2,3] suggest that the Hartree-Fock component of ACM1 hybrid functionals should be 1/6 instead of 1/4 for this class of materials. When applied to the PBE / PBE0 [2] functional this removes the scaling error of the calculated vibrational frequencies. Calculations using this PBE(n=6) functional in combination with optimized Gaussian basis sets result in very small remaining deviations between observed and calculated Raman shifts, with standard uncertainties of ≈ 3.5 cm^{-1}, maximum deviations of ≈ 10 cm^{-1}, and no significant systematic trends. These deviations are lower than the experimental variations when comparing results from different samples or authors. This has been confirmed for a wide range of silicate structures, for which high-quality Raman spectra have been published: Forsterite α-Mg_2SiO_4 (nesosilicate), γ-$Y_2Si_2O_7$ (sorosilicate), $K_2Ca_3Si_3O_{10}$ (oligosilicate), $K_2Ca_4Si_8O_{21}$ (phyllosilicate), and α-quartz SiO_2 (tectosilictae).

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Keywords: raman, calculation, silicate

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Design and characterization of high performance CMOS area detectors for X-ray crystallography

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CCD-based area detectors are well established as a principal technology for X-ray crystallography, both in the home lab and at synchrotron beam lines. However, recently CMOS detector technology has begun to replace CCD detectors in some applications. CMOS has a number of potential advantages including faster readout, shutter free operation and better detective quantum efficiency. We report on the design and operating characteristics of a new CMOS detector and compare the performance to CCD-based detectors.