

**Science
Division**



Doc No: SCI-BLP-027-0101

Issue: 1

Date: 21 March 2003

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Diamond Beamline Proposal 027

A Versatile X-ray Spectrometer for Diamond

A proposal prepared for the SAC May 2003

Acknowledgements

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Beamline Proposal 027

A Versatile X-ray Spectrometer for Diamond

1. Summary

It is proposed to build a versatile modular X-ray spectrometer on Diamond that will set the definitive world standards for stability and beam quality at high flux. Coupled with state of the art detector systems and secondary spectrometers, this beamline will make experiments feasible in the large areas of fundamental science and technology that are currently out of the reach of synchrotron radiation.

The construction of this beamline will involve significant but realistic developments in beamline and detector technology that will bring benefits to SR technology worldwide.

The outline design of the beamline is optimised for operation between 4 and 35 keV based upon a multipole wiggler source. An optical system has been designed in which each component is optimised for a single function, in order to maximise spectral purity, stability and flux. A cryogenically cooled twin axis monochromator suitable for fast scanning is proposed to produce a constant height exit beam without the use of any translational motion. An energy dispersive spectrometer is incorporated into the design to allow studies of fast chemical and photochemical kinetics. The beamline will be equipped with state of the art detectors for XAS and X-ray diffraction and a high resolution secondary X-ray spectrometer

Trace levels of elements play a vital role in geology, environmental science, biology, chemistry and materials science. This beamline aims for an order of magnitude improvement in signal to noise over any currently available XAS beamline and will permit study of samples in their natural functioning state.

In the study of nucleation and crystallisation of ceramics, minerals, clays catalysts and biomineralisation this beamline will permit collection of a complete combined XAS/XRD structure datasets in seconds. This facility will be ideal for spectroscopy experiments under extreme conditions of pressure and temperature. The philosophy of the beamline is to achieve maximum information content by collecting the maximum amount of data available from each sample.

Secondary X-ray Spectroscopy (SXS) will be developed to unravel structural information from different valence or spin states of the same element existing in a material. Charge and spin fluctuations play a fundamental role in condensed matter physics, transition metal compounds and in biological tissue. SXS permits X-ray magnetic circular dichroism spectra, to be measured on bulk materials even under high pressure conditions. SXS can remove a large component of the lifetime broadening, and thereby go beyond the core hole lifetime limit. X-ray Raman scattering can also be measured to produce structural information on light elements such as B in borate like glass.

This proposal forms part of a coherent strategy for X-ray spectroscopy in the UK, based around three beamlines: a microfocus beamline (BL-13), a bending magnet beamline (BL-C) and a high flux/stability beamline (BL-27). The facilities on each beamline are complementary, and each beamline possesses unique characteristics that will meet the needs of distinct areas of research activity. Within a European context, the beamline will complement lower-energy beamlines ID26, for ultra dilute studies, and ID24, for dispersive measurements, at the ESRF. It will attract many new users from HEIs and industry.

This beamline will ensure the secure development of XAS at Diamond into new and promising and challenging, areas, and will give the UK a unique tool for future scientific discovery.

2. Scientific Case

2.1 Introduction

X-ray spectroscopy (XAS) is one of the most versatile tools that has arisen from the development of synchrotron radiation. This technique provides insight at an atomic level for work spanning magnetism and collective behaviour of electron, catalysis nucleation, crystal growth, materials science, environmental and earth science. Applications of XAS encompass fundamental science and large scale industrial processes. Although exceedingly diverse, the science described below has one characteristic in common – it lies at or beyond the boundary of what is possible using existing spectrometers, or those planned already for Diamond.

2.2 Scientific Impact

2.2.1 Condensed Matter Physics

The ability to identify the valence state and local interactions around particular atoms in a solid is paramount to understanding the behaviour of many of the most important materials for the 21st Century.

Electronic and magnetic materials

On BL-27 the Secondary X-ray Spectroscopy facility will open up large areas of new science. Non-resonant Raman scattering may be used to probe magnetic dichroism in 3d and 4f magnetic materials where the selection-rules for probing magnetic states otherwise require soft x-ray transitions. Also of interest would be resonant Raman scattering, and studies of collective electronic excitations

XAS can make a major contribution to the field of doped magnetic systems, in which competing degrees of freedom often lead to several electronically-ordered ground states with similar energies. Layered transition-metal oxides, such as cuprates exhibiting high-temperature superconductivity, colossal magnetoresistance manganites, and charge-ordered nickelates, all involve such a balance of microscopic interactions. SXS has the unique ability to determine the local structure as a function of the valence state.

For a number of years there have been apparently conflicting results emerging from high-energy core X-ray spectroscopy and low energy techniques such as neutron scattering on the valence state of lanthanide compounds e.g. oxides of cerium and praseodymium. The high-energy techniques see a mixed valence, whereas the low energy techniques find a single stable valence. This problem strikes at the heart of our understanding of electron transfer, valence and bonding.

A knowledge of the hybridisation effects upon band structure could improve understanding of correlated electron behaviour in systems that can accommodate more than one ordered state, e.g magnetism and superconductivity in the heavy fermion family of compounds CeTIn_5 (T=Co, Rh, Ir) around their quantum critical point.

Cuprous halides are technologically important, laser components and have potential as novel quantum dots. There are currently two competing models of the observed physical properties: Fermi-coupling and off-centre atomic displacements of copper atoms. It should be possible to verify or disprove the off-centre model for explanation of the structure of CuCl at low temperatures and high pressure by employing combined XAS/XRD/ND methods

High Pressure Studies

Pressure is an important variable not only in earth sciences but also as parameter to investigate subtle behaviour of correlated electron systems. None of the beamlines thus far approved for Diamond are optimised for high pressure time resolved XAS. By combining cumulants (from XAS) with long range structural constraints (XRD) and dynamic information on phonons (Raman spectroscopy), in a single combined experiment it is possible to determine the interatomic potentials under high pressure. These potentials are of fundamental importance in the field, being the point at which theory and experiment meet.

High pressure melts and amorphisation

XAS/XRD/Raman is an ideal tool for study the structure and dynamics of melts (Ga, In, Se, Fe, Fe-S etc.) under pressure. These investigations will have significant impact on the understanding of the evolution of the Earth's inner core and, consequently, its magnetic field and will stimulate further development of theoretical models of the behaviour of melts under extreme P/T conditions.

Low temperature melting of crystalline materials under pressure – amorphisation – is a complex phenomenon whose characteristics are poorly understood. There is no theoretical foundation for this order-disorder transition, and it presents an experimental challenge since rapid spectroscopy and diffraction is required under extreme conditions. The amorphisation of zeolites has been found to have a dynamic response whereby the crystalline phase survives to higher pressures or temperatures the greater the rate at which the stress is applied. The time resolution currently for following the collapse of microporous structures is several minutes. XAS-3 would improve this to ms and permit determination of the frequency of the soft phonon modes or floppy modes that appear to drive this unusual non-reversible transformation to the amorphous state. The ultimate goal is to follow the formation of diaplectic glasses, amorphising minerals under T and P on ms timescales activated by shock waves.

2.2.2 Chemistry and Catalysis

XAS has become an established tool in catalysis, but despite the progress that has been made, there are many systems for which it not possible to make measurements under authentic operating conditions, either because the concentrations are too low or because real catalysts have to contend with rapidly changing gas flows and/or composition. Temporal analysis of products (TAP) and FTIR are two of the most powerful diagnostic tools in catalysis, in which reactants and the catalyst are analysed on the micro- to milli- second timescale. For example, TAP studies have shown that transient composition fluctuation have a significant effects in regenerating spent catalysts. On BL-27 it will be feasible to elucidate the structural basis of this type of phenomenon and to identify transient reaction centres by use of EDE, time-sliced scanning XAS or REFLEXAS. These studies will complement spatially studies of catalyst beds proposed for BL-13. Typical systems include, oxide supported metal and surface organometallic catalysts applied to 3-way exhaust gas catalysts, alkane hydrogenolysis catalysts for polymer recycling.

Secondary X-ray spectrometry (SXS) could have a revolutionary impact in catalysis, since it is feasible to circumvent the Achilles heel of XAS, of polydispersity and multiple sites. Valence selected XAS would make it possible to distinguish between large particle and clusters embedded in the support, or between active atoms localised at the metal- support interface and other spectator material.

For homogeneous transition metal catalysts such as the ultra-dilute catalysts for C-C coupling reactions, alcohol carbonylation, and catalysis in supercritical fluids as "green" processes, the

working concentrations lie at or beyond the limit of current spectrometers. Study of these catalytic systems under realistic conditions by XAS, IR, and NMR is an achievable goal.

The reaction mechanisms of transition metal compounds are studied in solution by stopped-flow systems, flow cells in microchannel reactors and photochemical activation to generate transients utilising IR, UV-visible NMR. XAS has been applied down to 10^{-3} sec, the challenge in the future is reduce the time step to the $10^{-10} - 10^{-6}$ sec region. This would open up the vast field of redox reactions, including intermediates in electron transfer reactions (including those initiated at electrodes); the primary steps in homogeneous catalysis from unsaturated transients, and transition metal photochemistry. Many preparations of nanoparticles in solution start with a reduction reaction and the early steps are both important and difficult to study. The combination of a higher flux beamline enabling low concentrations of dissolved metal complex coupled with improved time resolution would allow the early stages of particle growth to be investigated. In ultra dilute solution, complex-complex interactions would decrease and nanocluster formation slowed down allowing the measurements to be made. In the concentration limit that the nanoclusters did not form, it would be possible to examine the discrete species in solution under reaction conditions without being swamped by metal-metal distances from nanoclusters. This could have important implications for our understanding of the Heck reaction in ionic liquids where it is not clear whether the nanoclusters formed are the active species or a spectator and that the active complex is too small to be detected.

An alternative route for the study of highly reactive and unstable species is stabilised in cryogenic solids such as argon at *ca.* 10 K. Such information, when combined with other experimental data and high level calculations provides great insight into geometric and electronic structures and bonding. This is an established field, where the future challenges include use of SXS to not only elucidate symmetries but also distinguish between different species in the matrix. Matrix isolation is a standard tool in photophysics, but it is not currently possible to perform time resolved studies because of flux limitations. Pump-probe time-resolved studies will be possible on BL-27 and offer exciting prospects in structural photodynamics. 'Atomic XAFS' is a controversial topic that is still the subject of much debate. Matrix isolation experiments offer the possibility of unambiguous identification of these features with either 'atomic XAFS' or double excitation in transition metals. Such data is not available for the transition metals. The ultimate experiment in this area to seek to obtain XAS data directly from the vapour phase atoms as they exit the sputtering source, but this is not only a very photon hungry but also a technically challenging experiment.

2.2.3 Materials

Nanocrystalline materials are recognised as one of the most tangible outcomes of the present interest in nanotechnology. Combined X-ray techniques are fundamental to the determination of not only the exact crystalline/amorphous content, but also size, shape and size distribution of nanostructure, and the role of dopants.

Ion conducting oxides, batteries

Development of next generation of battery materials will increasingly adopt a holistic approach. Battery systems contain complex materials, often disordered and usually containing small quantities of dopants to improve the electrical and or mechanical properties. In situ combined techniques are a recognised tool in this field. BL27 will permit studies of real time charge/discharge cycles of all parts of the battery including the electrochemical interface. XAS studies of electrochemical interfaces have fallen out of favour in recent years since experiments have taken too long and the results have not been as good as those obtained via diffraction or SPM. On BL27, data collection time of a few seconds should be possible permitting the local structure of disordered surfaces to be determined in real time.

Fuel cells

Carbon supported metal nano-particle catalysts with sub-monolayers of modifiers, lie at the heart of low temperature PEM fuel cells. The challenge in future will be to characterise electrocatalyst degradation and poisoning of these materials and their reactions under near-working conditions. At present the metal loading and flux permit only limited time resolution. With increased flux it will be possible to study the structure of adsorbates on metal nano-particle catalysts. SEXAFS studies of adsorbed species and surface layer structures on planar catalysts will provide complementary information. EDE could be used for model systems to study potential dependent changes with msec (or better) time resolution. Time-resolved XRD could be employed to study structural changes in the carbon support where the high flux is essential for study of low Z materials and may allow the polydispersity of electrocatalysts to be more fully characterised.

Biomaterials

Biomaterials will be the subject of some of the most intense development in materials science in the next decade. Novel biocompatible materials are required for dentition, bone regeneration / replacement and other prosthetic materials. These will be complex materials, derived from amorphous or sol-gel precursors in most cases. The majority of materials in current use have been developed by trial and error. The challenge now is to determine the defining characteristics of successful materials in order to permit rational design of materials with improved mechanical and biological characteristics. XAS in combination with SAXS / WAXS will be an important technique for successful developments in this area. Ca XAS studies of the silicon – neuron interface interfaces would be possible. Studies on the release of metals from implanted materials (such as Zr from the current generation of artificial joints for limbs, metal-on-metal and alternative material combinations for hip replacements) will become possible. Studies of these are required, both individually and in conjunction with other materials such as semiconductors for the development of intelligent biomaterials and implants.

Glasses and Amorphous solids

At present, nucleation and growth processes in the vicinity of the glass transition can only be followed rather slowly by combined XAS/XRD. Nucleation cannot be readily differentiated from growth using either structural probe. The much higher flux levels on BL-27 coupled with the improvements in detector speed and resolution will bring these experiments into the arena of practical measurement, by reducing time frames for XRD and XAS to minutes or even seconds. This will enable dilution levels of impurities for XAS as low as a few ppm. Recent use of laser-heated levitator furnaces is permitting studies of the structure and dynamics in fragile inorganic melts and super-cooled liquids. Because of dust-free contactless conditions, liquids like alumina that would otherwise promptly crystallize on cooling can now be vitrified to temperatures 1000 deg or more below the melting point. When crystallization eventually happens the configurational enthalpy stored in the glass is so rapidly released that the temperature rises abruptly with the emission of light – recalescence. For typical drop sizes of a few mm crystallization occurs in 1-10ms. Recording this on these timescales with energy dispersive EXAFS and also with XRD is a huge challenge but one which is a feasible target for BL-27.

Sol gel materials, particular those containing metals for optical coatings, ultra-low expansion materials, filters, dielectrics. System containing Ti, have immense potential as a catalyst (beyond the existing TS1 zeolites, which have a limited pore size). The key issue is the *detailed* nature of the metal site, and how this is affected by synthesis, processing and environment. The combination of EXAFS/XANES with XRD, neutron diffraction, NMR, etc. has been extraordinarily successful in the past. SXS combined with a controlled sample environment has the potential to give quantitative understanding of the *mechanisms* of the synthesis/processing stages. This requires use of *time*

resolved, in situ facilities for studying the gelling-to-xerogel/aerogel stage, calcination/phase separation/crystallisation and finally, the *in situ* study of catalytic reactions.

The stability of glass in contact with iron-containing metals is strongly affected by the fractional population of Fe^{2+} and Fe^{3+} in the glass in coordination different sites. This information could be extracted by use of SXS, and would be of industrial importance. But perhaps the most important advance for complex glasses would be the ability to determine the local structure for dopants whose edges overlap – a practical outcome might be a better understanding of immobilisation of high level radioactive waste.

Zeolites and Microporous Materials

There is a continued challenge and interest from both industry and academia in the area of multicomponent dense framework materials such bismuth and iron molybdates, and perovskite related oxides. The nature of active sites on the surface are thought to be which significantly different from the bulk. A high-flux XAS/XRD beam line is required to permit study of first row transition metal ions promoters in presence of a highly absorbing matrix.

A significant question to be addressed in the preparation of framework catalyst, is the structure of specific metal ions when they are in the precursor state and how do they get modified during preparative processes. The goal of SR studies of solvothermal synthesis is to determine the structure of metal ions at every stage of the crystallisation processes from the gel state to the final product, in order to design specific materials.

Inclusion compounds are important but often neglected materials, that are difficult to study because of a lack of order. Combined XRD/XAS is a useful tool for study of these materials. Medium and long range order is gained via diffraction and the local conformation of the guest by XAS since this varies for each system. By following kinetics of order/disorder transitions in real time it may be possible to understand the ordering mechanisms in these materials.

2.2.4 Environmental and Earth Sciences

Fundamental advances in our understanding of natural environmental and anthropogenic systems have been achieved over the past decade using XAS, in particular by deriving element specific structural information on complex materials. XAS has had special impact in the area of environmental contamination, which often comprises non-crystalline (amorphous) species, sorbates and solution species. A legacy of Britain's industrial past is large areas of contaminated land and waterways that present both a health hazard and an inhibition to the productive use of the land. Contamination of the environment continues, an inevitable consequence of industrial and domestic processes, with the containment and environmental impact of wastes, a particular problem. Here XAS is able to provide critical and previously unobtainable information on the speciation of toxic metals and the role of sorption on mineral surfaces in the cycling of the metals; these factors control toxicity, mobility and bio-availability and their understanding allows remediation and containment strategies to be devised. XAS is also particularly relevant to the increasing demand for high calibre information relating to the science underpinning environmental regulation and risk assessment. To make further advances requires analysis of systems at ***environmentally relevant concentrations***, and BL 27 will provide this capability as its incident photon flux will be up to 1000 times higher compared with BL C; thus the minimum level of concentration required is reduced by more than two orders of magnitude, to the sub ppm level in many cases. Of particular interest are bio-geochemical and inorganic:organic interface systems where toxicity must be studied at particularly low concentrations. In all these studies the identification of the species and their behaviour is essential. XAS has provided, and will continue to provide, information on 'heavy' metals such as Cd, As, Se,

Sb, Cu, Zn, Hg, Cr, U, Pb, Tc and Pu, all products of industrial processes. Furthermore, the *energy dispersive EXAFS* facility will enable key information on *the development and properties of amorphous phases* in the first millisecond after precipitation. Such phases often predominate in many surface environments and are a major suspended constituent of discharges from waste disposal sites.

XAS has also been very successful in the study of systems that are crucial to the understanding of fundamental Earth processes, particularly by the study both the bulk and surface chemistry of rock-forming and economically important minerals; such information is relevant to both Earth and environmental studies. The importance of the biogenic contribution to surface processes is increasingly being realised - for instance, many phases form biogenically and BL 27 will enable the *in situ* characterisation of metal sorption and mineral phases within biofilms, enabling the effects of aerobic and anaerobic conditions to be studied. These studies will also determine the influence of contaminants and plant nutrients on precipitation processes.

Examples of future investigations are:

- ***Arsenic is one of the most prevalent causes of water contamination*** in developing and developed countries and consequently is stimulating much research into the biogeochemistry of this exotoxic element. The geochemical behaviour of arsenic is governed predominantly by adsorption onto iron oxyhydroxides. XAS is ideal for determining the oxidation state and local chemical environment of arsenic. Recent studies indicate that micro-organisms have a significant impact on the mobilisation of arsenic. Fluids and biogenic material tends to have very low concentrations of arsenic, hence at present cannot be studied by XAS in the UK. BL 27 will enable this and similar studies on other toxic species and they will provide vital data in the elucidation of arsenic cycling and the development of strategies to provide safe drinking water.

- ***Selenium is recognised as a potentially serious environmental contaminant.*** Microbiological processes are an integral part of bio-geochemical cycles, but may also be harnessed for the treatment of contaminated solid and liquid wastes. Little is known about the nature of microbially-derived selenium-containing minerals, however sulphate-reducing bacteria are known to reduce selenium oxyanions to selenides or elemental selenium. XAS is an ideal tool to study such speciation changes. BL 27 is needed to study of systems at environmentally realistic concentrations. Currently only systems with artificially elevated concentrations can be studied.

- ***Remediation strategies*** that can immobilise contaminants *in situ* such as phosphate treatment, offer the potential for large savings in clean-up costs, however, to trace the fate of all the contaminants in a soil sample is not simple. Contaminated soil often contains a variety of contaminants at very different concentrations, while a large background signal is often present in soil. Thus to obtain XAS data on all the elements in such samples requires not just the capability to reach ppm detection levels but also to exclude background signals from the detector. BL 27 will enable such studies to be undertaken enabling the accurate assessment, and hence improvement, of such remediation technologies.

- ***Chromium use has left a legacy of waste residues*** deposited in urban environments. These are often in close proximity to water tables. The role of Fe/Mn oxyhydroxides on the Cr(III)/(VI) partition is of particular interest in identifying remediation strategies, as the valence state is key to chromium bio-availability and toxicity. XAS is excellent for determining the valence state of metals such as Cr, which is often present in low concentration (ppm level), BL 27 will be ideal for such studies.

- In order to construct records of ***ocean conditions related to climate change***, it is necessary to rely on geochemical proxies. The Sr/Ca ratio in coral has been used as a measure of local surface sea

temperature (SST). However the potential to improve the temporal resolution exists. There are many elements in coral, notably Ba, Cu, Mn, Fe, Sn, U and Zn, at concentrations between 1 and 100 ppm. Their use as palaeoenvironmental indicators is inhibited by a lack of understanding of their mode of incorporation into the coral skeleton. BL 27 will enable their local environment to be determined and enable determining whether they encode SST or document episodic changes in seawater concentration. Similar studies on trace elements in other biogenic carbonates will give insights into the elemental ordering and structure in natural carbonates. This information will also provide data on variations in climatic conditions in a ‘geological’ timeframe.

- **Uranium contamination** has a dramatic impact on all aspects of the natural environment. There is increasing pressure to understand, and predict, the mobility and geochemical cycling of uranium within the geosphere. Previous XAS experiments investigating uranium speciation have focussed on highly acidic solutions to keep the uranium concentration at detectable levels ($\gg 10$ ppm). In order to understand U mobility better, experimental conditions must replicate those that prevail in the surface environments where uranium concentrations are at ppm or sub ppm levels. BL 27 will enable XAS studies on these solutions and other systems such as Hg and Cd in sulfidic systems and the geochemically relevant understanding of the role of thiosulphate complexes in the transport of gold. Furthermore, combined analysis with other spectroscopic techniques (such as IR and Raman) will produce coincidental complementary information, and continuing development of sample cells will allow analysis at a range of temperatures and solution chemistries; the latter are particularly important in the study of metal transport in crustal fluids ($t = 100-500^{\circ}\text{C}$).

- The **development of metastable amorphous phases** are a crucial stage in the cycling of metals at the Earth’s surface, often forming in geochemical ‘sinks’ where toxic metals accumulate. Particularly important are Fe and Mn oxyhydroxide phases and ‘heavy metal’ sulfides in reducing conditions – these are also a major suspended constituent of discharges resulting from waste sites. Using the EDEXAFS facility on BL 27, the structural development and properties of Fe, Mn, Co and Ni sulfides and oxides, toxic phases containing Cr, As, Sb, Se and a range of transition metals will be determined. Investigations will also include development of novel flow through cells that will cause precipitation *in situ* in the beam, allowing structures of neo-formed solids to be determined, and development of special cells for redox sensitive systems (such as those containing Fe and Mn).

These exemplar studies are only partially indicative of the enormous range of science relevant to the environmental and Earth sciences that would be achieved on the versatile BL 27. It will ensure that the UK’s research communities in these areas remain as world-leaders in their fields.

2.2.5 Biological Science

Biological XAS is a broad field that overlaps chemistry and environmental sciences, hence the systems of interest span small therapeutic molecules, isolated proteins and membrane complexes, to microbial transformations, biomineralisations and bioremediation. In biological sciences, BL-27 will complement the microfocus and Core XAS beamlines.

Most biological systems are disordered, even when the solid state is involved. This places limits on the utility of diffraction and scattering techniques. Although there has been an explosive growth in protein crystallography over recent years, the bond length accuracy achieved at metal binding sites in proteins (typically 0.1 \AA) is still less than desired. The recent trend of refining a PX-determined metal site in a protein against XAS data, to obtain a more accurate picture of the metal site is likely to continue. Clearly XAS will continue to make a significant contribution to this area, especially when XAS used in tandem with PX.

With currently available spectrometers, XAS data for metalloproteins have to be measured at artificially high, (0.1-10 mM) concentrations. In many cases, this is not thought to produce artefacts. However, many proteins have limited solubility at these concentrations, or form oligomers: The copper metallochaperone Atx1 (from *Synechocystis* PCC 6803) forms homodimers at concentrations >0.3 mM, displacing one of the monomer Cu ligands with a ligand from the neighbouring molecule. The oligomerisation problem is largely absent at μM concentrations. The flux from BL-27 will allow high quality data to be obtained, in favourable cases, *at biologically relevant concentrations*.

Vanadium is an essential component of several metalloproteins, and recently titanium binding to a bacterial iron binding protein has been confirmed. It is currently extremely difficult to obtain high quality XAS data on these elements at the concentrations available, due to a combination of the low penetrating power of 4-5 keV x-rays and low fluorescence yield. Such studies will be tractable on BL-27.

The capabilities of BL-27 will open up a whole new range of possibilities in time-resolved biological XAS, both in EDE and time-sliced scanning XAS, including studies of anti-HIV macrocycles, photo-activated Pt anti-cancer complexes, Rh(II) anti-cancer complexes, radiopharmaceuticals (both diagnostic and medicinal), biomimetic reactions, and ultimately time-resolved biological XAS. The flux from BL-27 will reduce the minimum concentrations (and/or the minimum time) required for EDE. For systems that remain too dilute for EDE, the stability of the beamline optical scheme (0.01 eV) will make time-sliced XAS in scanning configuration achievable.

SXS could have a significant impact in biological XAS. For instance, it will allow the understanding of the reaction/redox cycle in the di-nuclear, 2Fe enzyme, mammalian purple acid phosphatase, and ultimately may allow a complete understanding of the redox cycle of the 4Mn oxygen evolving complex of Photosystem II.

At the interface with environmental sciences, microbial bioremediation has the potential to have a significant impact, both environmentally and economically. Microorganisms including fungi and their products can be highly efficient bioaccumulators of soluble and particulate (nano-crystalline) forms of metals, especially from dilute external concentrations. In addition to micro-organisms, the nematode *Lumbricus Rubellus*, is a known accumulator of arsenic. Characterisation of accumulated or precipitated minerals is a high priority in elucidating precise pathways of toxic heavy metal removal, leading to further opportunities for biotechnology process development. Whilst analytical chemistry and conventional electron-microscope techniques are useful in determining and locating metals precipitated by fungal mycelium and bacterial biofilms, they lack the ability to characterise element speciation. Beamline 27 will revolutionise this important research area. The high flux will deliver the ability to work with sub-ppm concentrations for most elements and the multi-pole wiggler will be vital for delivering the high energies needed for studying the K-edge spectra for environmentally-important elements such as Cd. At slightly higher concentration, time-resolved studies of the adsorption of metals and precipitation of nano-crystalline oxide phases on microbial surfaces (such as *B. subtilis*, *Shewanella*, *Calothrix*) will be possible. There is a natural synergy between this work and studies proposed for the microfocus XAS beamline to localise these bio-accumulated metals.

2.3 Alternative Sources

ESRF ID26:

X-ray Absorption of Ultra-dilute Samples (XAUS). This will be the main competitor to XAS-3, especially with its brighter beam from the undulator source. The beamline designed for XAS and EXAFS studies requiring high-flux for dilutions below 10mM, down to about 4ppm. Focal spot size quoted is $200\mu\text{m} \times 80\mu\text{m}$, although latterly focussing down to micron size has become available. Energy range: 3.2–30keV. Flux $> 10^{12}$ p/s. Inelastic scattering is also available.

APS PNC-CAT:

On BL 20-ID line, much of the XAFS is micro-XAFS in conjunction with K-B mirrors. They have a standard multi-element Ge and ion chamber detectors, but have been working with some log-spiral laue analyzers with some success. These provide better energy resolution and similar solid angle as the 13-element detector and can help if a sample has many overlapping fluorescence lines.

APS GSECARS:

This is a dedicated facility for environmental and earth sciences research. It operates over the energy range 4 to 80keV. On BL 13-ID-C, 90% of the dilute XAFS is microprobe work, with typically a $\sim 2 \times 3$ micron beam, focussed with K-B mirrors. For fluorescence a Canberra 16 element detector, but is used also a wavelength-dispersive-spectrometer has been used on occasion. They also have a grazing incidence capability using the SSD.

APS BioCAT:

The BioCAT beamline BL 18-ID is designed for biological problems and will be very competitive with XAS-3. It has been optimized for rapid energy scanning so that scans over 1 keV ranges can be completed in 13 s or less. The range of energies accessible with either of two monochromators is from 3.5 to 35 keV. XAS detectors include novel BioCAT-developed analyzers to reject background. It has bent Laue analyzers suitable for studies near the molybdenum (K-edge 20 KeV), cadmium (K-edge 26.7 KeV) and zinc edges (K-edge 9.66 KeV). For low and moderate energies the Multilayer Analyzer Array Detector (MAAD) provides background rejection with no count rate limits for dilute and complex systems.

APS MRCAT:

The MRCAT beamline BL 10-ID is setup for XAFS from 4.8-90 keV with a Si(111) and a Si(333) monochromator. It is able to take complete EXAFS scans of 1000eV in under 10s. The MR-CAT instrumentation includes a single-element solid state detector and a 13-element solid state detector for dilute XAFS and Fluorescence measurements. A project has begun to fabricate a bent Laue fluorescence analyzer. This detector will be capable of easily separating close-lying fluorescence lines and will not be as count rate limited as a solid state detector. Site-selective XAS at the Fe K-edge has been done here using a Ge (620) crystal spectrometer.

Spring-8 BL10XU:

In BL10XU, the High Brilliance XAFS Station, a high-intensity and quasi-monochromatized SR beam is obtained by an in-vacuum undulator (Spring-8 standard). The undulator beam is monochromatized by a Si (111) double crystal, resulting in intense X-ray ($>10^{13}$ photons/s) with a energy resolution ($\Delta E/E \sim 10^{-4}$). There are two 19 or 100 multi-element solid-state detectors (SSD) mounted in a high performance goniometer on a standard optical table with a stage height control. Site selective XAS at the Cu edge has been done using a Ge (111) crystal spectrometer

SPEAR-3 4.1, 4.2, 7.2, 7.3, 9.3

When SPEAR3 returns to service in 2004 there are a number of beamlines with 16 or 20 pole 2/2.1T wigglers that will be very competitive with XAS-3. They generally operate in a similar energy regime 2.4-35 keV and 2 beamlines have a 30-element Ge detector optimised for dilute biological XAS.

2.4 Beneficiaries

The list of beneficiaries is as diverse as the new areas of science described above. The list includes earth and environmental scientists, chemists and materials scientists. Those involved in tackling the fundamental challenges of condensed matter physics will have a new tool. This beamline will also nurture a new community of biological scientists. The programme described above is very relevant to the core missions of EPSRC and NERC in particular..

3 Beamline Requirements

The principal requirements are maximal flux and beam purity, maximal stability and minimal noise. The flux requirement can only be satisfied by a MPW, since only this source will give the broad and stable energy range. It is mandatory for QEXAFS and EDE.

A MPW has been requested by the community for a number of reasons:

- Whilst in general undulators are the ideal source for diffraction experiments, the requirements of spectroscopy to change energy imposes additional requirements such as undulator gap scanning. This whilst being actively developed at 3rd generation synchrotrons, is likely to limit the ultimate data quality, and most certainly precludes time-resolved experiments except in special circumstances (e.g. ID24 at the ESRF).
- With a 3 GeV machine in order to cover a continuous energy range from it is unlikely that an undulator with a period of less than 20mm can be used. To reach even 20keV relies on the 11th or 13th harmonic, which could easily be degraded by magnet errors.

The advantages of a MPW are a continuous spectrum through the whole energy range. The disadvantages include the high heat load on the optics, and the large source size which in particular for off-axis applications will severely limit the size of x-ray focus.

The philosophy adopted on this beamline is to ensure that each optical component performs only one operation in a modular fashion and that no component deflects the beam out of the horizontal plane. The system proposed should be easy to set-up and maintain. There are a minimum of movements allowing components to be removed or inserted as necessary and most importantly providing unprecedented beam quality. The monochromator proposed is of the twin axis variety which has mirror systems to match the acceptance from the source. The design will accept 0.14 millirad vertically and 1 millirad horizontally.

The front end of the optical system collimates the beam into the twin axis monochromator, the front crystal receiving the bulk of the heat load. The second axis will provide tail stripping, to enable thick specimens to be examined with minimal distortion. In this configuration only rotation is employed on the monochromator axes, thus permitting rapid scanning. Harmonic rejection is not attempted by the monochromator, thus eliminating complex feedback systems. Instead, harmonic rejection will be achieved by a pair of flat mirrors under the control of the experimenter. Re-alignment of the equipment when transferring to a different edge will not be necessary, merely the adjustment of the harmonic rejection mirrors.

The post monochromator mirrors will again be normally fixed and focus the beam to an image which can subsequently be used either for fluorescence detection, straight-forward absorption, diffraction, input to catalytic reactor, or high pressure cell.

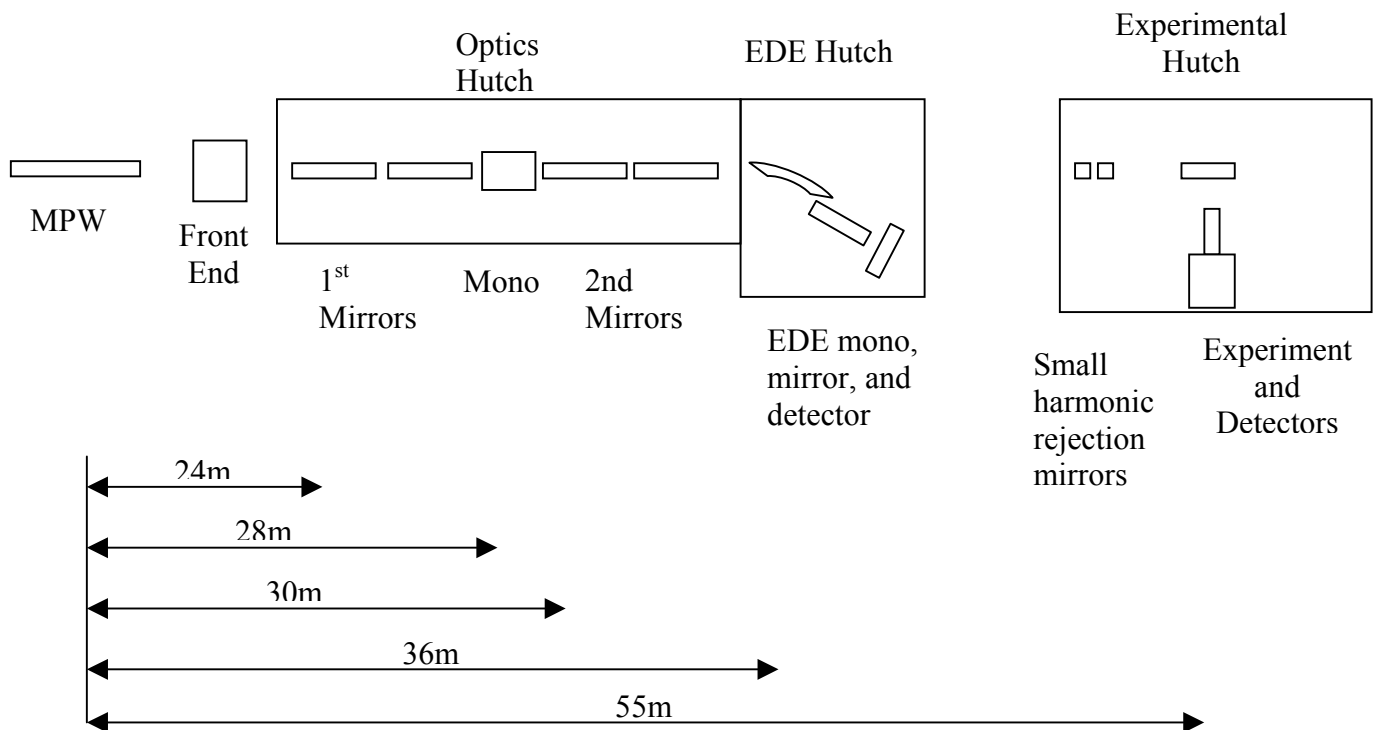
3.1 Sample Environments, Modules and Offline Facilities

Sample environments will be modular and dependant on the application. We expect that almost all states of matter will be investigated, involving high and low temperatures and pressures. Located on kinematic mounts with 10 micron precision, modules will be optimised and prealigned offline, in order to maximise beamline throughput. A wide variety of sample environments will be required and should be developed in collaboration with user groups. These include cells capable of operating at high and low temperatures and pressures (HT, LT, HP, LP) equipped for simultaneous XRD measurements and Raman, optical, electrical resistivity and magnetic properties, utilising as much as possible the 4π solid angle for x-ray and optical beam delivery and for data collection. Process monitoring will be available for reacting systems. The data acquisition system should allow seamless integration of user-defined sample environments and ancillary data collection into the experiment. This will ease automation of tasks such as setting up, control of multiple techniques and remote access for monitoring. Offline facilities will permit initial alignment and sample characterisation to be undertaken. These facilities would be shared with BL-13 and BL-C. It is however extremely important that BL-27 has its own laboratory facilities, as it is likely to be performing many more preparative experiments, in far more hostile conditions than the microfocus beamline and in addition space will be required for setting up, testing and development of the above non-ambient cells.

3.2 Detectors

A range of detectors will be available which will include incident/transmitted flux monitors which may be either of the photodiode or ion chamber type. Area detectors will be used for diffraction experiments, and parallel fluorescence detectors for fluorescence analysis. The situation with regard to these detectors is extremely fluid at present so firm commitment to specific detectors is difficult. The secondary x-ray spectroscopy falls into two parts. The first is a wavelength dispersive detector with modest energy resolution, but capable of scanning over a wide energy spectrum. The second spectrometer is a high resolution spectrometer consisting of a scanning vertical arm 1m in length, secondary bent monochromator crystals and an area detector.

4 Beamline Specification



A schematic layout of the proposed beamline is shown below:

Source:	5m 1.6T 100-pole permanent field multi-pole wiggler
Photon Energy:	4-35 keV with energy resolution better than $\frac{1}{4}$ core-hole lifetime limit
Energy stability:	0.01 eV
Bandpass:	$\sim 10^{-4} - 10^{-5}$ using interchangeable crystals Si(111) and (311) or (511)
1 st Mirrors	1.5m Pt coated collimating vertically set at 2.25 mrad + plane mirror
Mono	Direct LN ₂ cooled, 4-bounce QEXAFS
2 nd Mirrors	1.5m Pt coated toroid + plane mirror
Beam size:	~ 1.5 mm (h) x 125 μ m (v) FWHM (Fan of 1mrad (h) x 0.14 mrad (v))
Flux:	7.5×10^{13} p/s Si(111) at 10keV (ESRF ID26 10^{13} , SRS MPW6.2 10^{12})
For EDE:	
Photon Energy:	6-35 keV
Beam size:	~ 70 μ m (h) x 25 μ m (v) FWHM
Flux:	7.5×10^{13} p/s Si(111) at 10keV (ESRF ID24 2×10^{12} , SRS 9.3 5×10^{11})

4.1 MPW Specification

Figure 2.2-4 from the Green Book is reproduced below in Figure 1. This shows the flux as a function of photon energy with a beam current of 300mA. The 1.6 and 2.5 T devices are 5m and 2m long permanent field magnets, but the 3.5T device is a superconducting 1m long device. The reason that the higher field devices have less flux at lower energy is that in order to restrict the total power output to about 20kW the length of the device, and hence number of poles has been restricted. The preferred device will be specified in the design stage. In terms of the energy spectrum a 5m 1.6 T permanent field magnet device is sufficient, however in order to reduce the effective source size a 1m superconducting device (2 - 2.5 T) may offer advantages by reducing the effective horizontal source size.

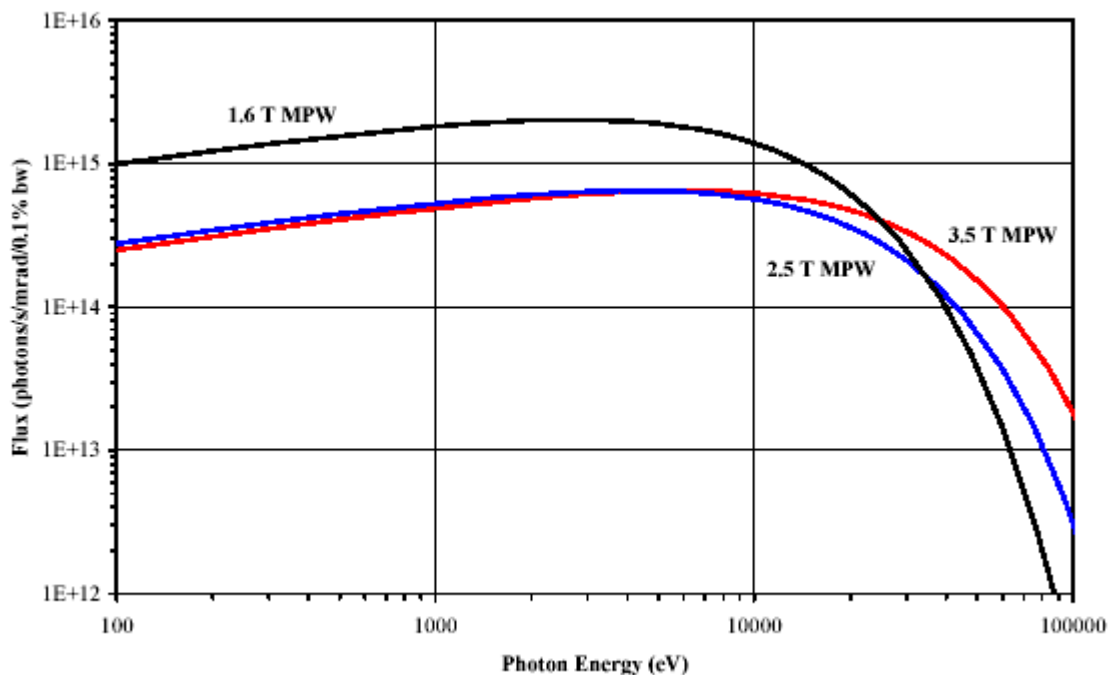


Figure 1: Spectral flux for 3 possible MPWs. (Reproduced from figure 2.2-4 in the Green Book)

4.2 Optics Specification:

In order to utilise efficiently the vertical aperture of radiation offered by an MPW without degrading energy resolution, the first optic is required to be a mirror. This will produce a collimated beam vertically and reduce the heat load on the primary monochromator. This will impose a maximum energy cut-off; proposed to be 35keV so that all elements are covered. However consideration will be given to make the mirror removable to extend the energy range. A first mirror of length 1.5m at 24m from the source would be able to collect 0.14 mrad which is 50% of the vertical flux from the MPW assuming a Pt coated mirror at 2.25 mrad which will reflect X-rays up to 36 keV. The power absorbed by the mirror is about 450 W/mrad. Recent high heat load mirrors using Ga eutectic cooling channels are predicted to be able to cope with up to 2 kW of power. Certainly a few hundred watts is not a problem. A second mirror, downwardly deflecting is then required to bring the beam

back into the horizontal plane in order to facilitate ease of use and flexible operation of the beamline. This is particularly important as the proposed twin axis monochromator acts as a collimator, so without this mirror the beam will be going uphill between the two axes. To adjust the mirror angle, then becomes very difficult. The mirror will be a cylindrical mirror to collimate the beam in the horizontal plane. In order to calculate vertical beam sizes, slope errors of $0.5\mu\text{rad}$ have been assumed. Whilst this is better than can be achieved now ($1\text{-}2\mu\text{rad}$) it is expected that ion beam milling or equivalent will have developed to enable polishing errors to be removed.

The heat load on the monochromator is immense at about 1800 W/mrad . (This would be for a full 5 m straight 100-pole 1.6 T device). Stanford have developed a directly cooled LN_2 double crystal monochromator. It has already been demonstrated to work superbly at 600 W and is designed to take at least 1 kW on SPEAR-3. Clearly further work will be required in order to assess what power loading is feasible, but in the timescale of this Diamond beamline SPEAR-3 will be working very soon. A twin axis monochromator is required in order to facilitate a 4-bounce system to reduce monochromator tails and to produce a constant height exit beam without the use of any translational motion. Careful design will be required in order to ensure that the 2 axes move exactly in sequence – 1 arcsecond reproducibility has recently been demonstrated on the radiometry beamline D-07-1B at BESSYII.

In order to focus the monochromatic beam vertically 2 further mirrors will be required to produce a focussed fixed exit beam coaxial with the output from the MPW. The first of these will be a toroid to focus the beam horizontally and vertically at the sample position. The second will be a plane mirror used to bring the beam back to the horizontal plane, but also is used for vertically focusing for EDE. A variety of plane paired smaller mirrors will then be employed in the experimental and EDE hutch for harmonic rejection.

The optics for the energy dispersive component of this beamline is probably the most challenging aspect of monochromator design for the whole spectrometer. By placing the EDE hutch immediately behind the optics hutch advantage of the still broad horizontal beam can be used to disperse the beam across the horizontal monochromator. This provides a virtual source point for the spectrometer which will focus the dispersed beam within 1 m of the sample as is standard. However the power loading is as yet an unsolved problem. Various possibilities exist, such as the Stanford asymmetric I-beam mono designed to take 1 kW , and the APS triangular crystal which has a thinned section for Laue diffraction. However one cannot be too prescriptive at this point and it may be that a depth-graded carbon-carbon multilayer (currently being prototyped) may allow pre filtering of the intense white beam.

4.3 Detectors:

As noted above there is no single solution to the challenge presented to detector design on high flux beamlines on 3rd generation sources.

Ultradilute Spectroscopy

A range of detectors will be required to obtain optimum data for the many types of sample that would be studied on the station. Compact multielement solid state detectors based upon either Ge or GaAs with digital signal processing will be valuable for low scattering matrices. Pixellated detectors could be produced, but there may be substantial issues of reliability and servicing to be considered.

For ultradilute samples with a strongly scattering matrix it is likely that multilayer Bragg filters currently under development at the APS will emerge as the only viable route to collect good data. A design study is needed to model the performance of the detectors with prototypical samples for ultradilute XAS.

Secondary X ray Spectroscopy

There is a clear need for two spectrometers. The first would be for a wide range *X-ray fluorescence* spectrometer with 5-10eV resolution – possibly purchased from a commercial supplier but with integrated software and capable of operating in the vertical plane. The second Raman Inelastic X-ray Scattering spectrometer (*RIXS*), operating in vertical plane would have a resolution set to the lifetime of the final core hole in the one step process. This is approximately one quarter of the primary core-hole lifetime, and for transition metals is about 0.2eV. This spectrometer will require a range of spherically bent silicon and germanium crystals with a 150mm diameter to cover the required energy range. These two spectrometers will require a number of detectors including both 1D photon counting and integrating detectors. It is important that these detectors give linear behaviour at high count rates. In addition a quarter wave phase plate will be needed for some applications.

X-ray diffraction

There is a need for high resolution 1D and 2D detectors, for which a number of possible solutions exist. For 1D, gas microstrip detectors would offer an immediate solution, but over the lifetime of the design phase of this project is very likely that silicon microstrip (e.g. DIFEX) will emerge from the prototype stage to become viable practical detectors. The situation for a 2D detector is less clear at and requires more investigation.

Energy Dispersive XAS

The next generation of silicon microstrip detector, XSTRIP2, should offer a 1024 channel PSD with $<1\mu\text{s}$ minimum integration time. Development of a suitable I_0 detector should be given a high priority if this technique is to be capable of fulfilling its potential in time resolved XAS.

5. Costs

Costings: BL 27 in thousands of pounds.

MPW ID	
Front end	
Pre-Mono optics	
First mirror	
Second mirror	
4 bounce Monochromator	
2nd set of two mirrors	
Harmonic Rejection mirrors	
EDEXAFS mono	
Optics Hutch	
Vacuum legs	
Experimental hutch	
Io, It, Im detectors	
Fluorescence detectors (solid state)	
Fluorescence detectors multi-layers energy selecting	
1 d silicon detector	
EDEXAFS detector	
Secondary X-ray spectrometer	
RIXS detector	
Sample stages	
User area	
Controls for BL and experiment	
Ancillary labs	
Total	

XAS-3 a Versatile X-ray Spectrometer for diamond

Expressions of interest are invited from research groups in using a versatile X-ray Spectrometer on the **diamond** light source.

diamond <http://www.diamond.ac.uk/> will be built at the Rutherford Appleton Laboratory and is due to be available to users in September 2006. With 24 cells, and at 3.0 GeV, it will be unique among the medium energy synchrotron radiation sources and will present great opportunities for fundamental and applied research in both the physical and the life sciences. It will provide very bright radiation from undulators up to 20keV and high flux from multipole wigglers and wavelength shifters to energies greater than 100keV. Bending magnet sources will provide intense radiation over a wide spectral region from 40 keV to the IR.

After consultation with the Science Advisory Committee we are proceeding with the preparation of detailed proposals for beamlines for consideration for the third year of operation, that is, from September 2008. It is proposed that one of these should be a versatile X-ray spectrometer. The beamline will be optimised for applications that need the maximum flux, beam stability and beam purity. It will be ideal for experiments that demand sample dilution, speed, combined techniques and secondary spectroscopy.

The outline specification of the beamline is set out below.

Source: multipole wiggler

Optics:A Cryogenically cooled 4-bounce monochromator with cooled primary mirror
Energy 4-35 keV
Resolution less than 1/4 of core hole lifetime limit
Beamsize at sample 5 – 100 μ m
Harmonic Content $10^{-5} - 10^{-6}$ at all energies
Flux 10^{14} ph/s at 10 keV

Optics:B Cryogenically cooled polychromator for dispersive XAS
Energy range: 6-35 keV,
Resolution $\Delta E/E 1 \times 10^{-3}$;
Photon flux $3 \times 10^{13} \text{ s}^{-1}$ with Si(111) at 10keV
Beam size 50 μ m (v) \times 30 μ m(h) FWHM

Detectors and Secondary Optics:

Spectroscopy: multielement compact solid state detectors, multilayer Bragg filters, μ -strip detector for EDE. Secondary monochromator for high resolution secondary spectroscopy.

X-ray diffraction: curved μ -strip detector, 2D pixellated area detector

Sample environment and offline facilities .

Modular sample stages for rapid interchange of complex environments.
Online in-situ sample characterisation via optical, electrical and magnetic probes.

Expressions of interest, not longer than two sides of A4 should be sent as a word document to diamond@rl.ac.uk by **February 20th 2003**. They should be marked: **BEAMLINE XAS-3** and include:

your name and affiliation;
the overall objectives of your proposed research;
the likely long-term impact on science and technology
and your comments on the outline beamline specifications.

The information will be used in the preparing the case for the beamline and defining its primary aims.

Colin Norris
Science Director for **diamond**

Beamline Proposal 027: A Versatile X-ray Spectrometer for Diamond

Beamline Proposal 027 – Expressions of Support

Names	Affiliations	Areas of Interest / Techniques
N. Allison	Geography and Geosciences, University of St Andrews	Geochemistry, Paleoclimatology.
E.H. Bailey	Life and environmental Science, University of Nottingham	Environmental and geochemistry, Speciation, Heavy metal contamination
S. Bailey	Department of Chemistry, De Montfort University	Semiconductor physics, High Pressure, Combined techniques, Biomaterials
L. G. Benning	School of Earth Sciences, University of Leeds	Microbiology, biomineralisation, metal uptake, biofilms, XAS
F. Berry	Chemistry Department, The Open University	Materials chemistry, heterogeneous catalysis, Pigments, Interplanetary geochemistry, Cracking catalysts
A.T. Boothroyd	Department of Physics, Oxford University	Condensed matter physics, Magnetism, Valence/charge fluctuations, SXS
R. Burch	School of Chemistry, Queen's University of Belfast	Heterogeneous catalysis, Time resolved XAS, Combined XAS/TAP,
C. R. A Catlow	Davy Faraday Research Laboratory, The Royal Institution	Materials chemistry, combined techniques, catalysis, theoretical modelling, microporous solids
A. Chadwick	School of Physical Sciences, University of Kent	Materials chemistry, Nanomaterials, Combined techniques, Solid state ionics.
J. M Charnock	Department of Chemistry, University of Manchester	Geochemistry, Earth and environmental science, speciation and mineralisation

Names	Affiliations	Areas of Interest / Techniques
S. P. Collins	Diamond Light Source	Magnetism, Resonant Raman Scattering, Electronic Excitations, SXS,
S. Doherty	School of Chemistry, Queen's University of Belfast	Nanoclusters, Ionic liquids, Homogenous catalysis.
J. E. Evans	Department of Chemistry, Southampton University	Time-resolved XAS, Heterogeneous catalysis, homogeneous catalysis.
A. A Finch	Geography and Geosciences, University of St Andrews	Geochemistry, Paleoclimatology.
G. M. Gadd	Biological Sciences, University of Dundee	Microbiology, Bioremediation, Biogeochemistry
G. N. Greaves	Department of Physics, University of Wales	Glasses, Devitrification, Amorphisation, Time resolved XAS, Laser levitation techniques, Recalescence, Combined techniques.
C. Hardacre	School of Chemistry, Queen's University of Belfast	Heterogeneous catalysis, time resolved XAS, combined XAS/TAP, nanoclusters, Ionic liquids, Water remediation via colloids, Heavy metal sorption.
K. D. M. Harris	School of Chemical Sciences, , University of Birmingham	Chemical Crystallography, powder diffraction, structure solution methods, inclusion compounds
I. Harvey	CLRC, Daresbury Laboratory	Bio-inorganic chemistry, Ultra-dilute spectroscopy, Time resolved XAS, Combined techniques, Biomimetic reactions, Transport proteins.
C.M.B. Henderson	Department of Earth Sciences, University of Manchester	Earth and environmental science, Geochemistry, XAS, Biomineralisation
M.E. Hodson	Department of Soil Science, University of Reading	Soil Science, Speciation, Heavy metal contamination, Remediation.
D. Holland	Department of Physics, University of Warwick	Glasses, NMR, Neutron scattering, Mossbauer, SXS
A. S. Hursthouse	School of Engineering & Science, University of Paisley	Environmental Science, Geochemistry, Toxic metals in the environment, Ore Processing residues

Names	Affiliations	Areas of Interest / Techniques
C.J. Langdon	Department of Soil Science, University of Reading	Soil science, Heavy metal and arsenic uptake in bio-organisms, XAS, XANES
M. R. Lees	Department of Physics, University of Warwick	Superconductivity and magnetism, Magnetoresistance, Heavy fermions, heat capacity, Neutron scattering, SXS
A. Lennie	CLRC Daresbury Laboratory	Earth and Environmental science, Mineral chemistry, Metal sulfides and carbonates, Complexation.
R. Linford	Department of Chemistry, De Montfort University	Solids state electrochemistry, Battery materials, Interfacial chemistry
F R Livens	Department of Chemistry, University of Manchester	Geochemistry, Earth and environmental science, speciation and mineralisation
J. Lloyd	Earth Sciences, University of Manchester	Geochemistry, Earth and environmental science, Speciation and Arsenic contamination.
A.J. Morgan	Biosciences, Cardiff University	Soil science, Heavy metal and arsenic uptake in bio-organisms, XAS, XANES
Mosselmans	CLRC Daresbury	Environmental and Earth Sciences, Geochemistry, XAS technique development.
P.J. Murphy	Earth Science and Geography, Kingston University	Environmental and geochemistry, Speciation, Heavy metal contamination
R. J. Newport	Material Physics, University of Kent	Materials chemistry and physics, Sol-gel synthetic methods, optical coatings, Amorphous materials and glasses, Biomaterials, Time resolved XAS/XRD
R Patrick	Department of Earth Sciences, University of Manchester	Geochemistry, Earth and environmental science, speciation and mineralisation
D. McK. Paul	Department of Physics, University of Warwick	Magnetism, superconductivity, High pressure, SXS
D A Polya	Earth Sciences, University of Manchester	
R. Rickaby	Earth Sciences Department, University of Oxford,	Earth and Environmental Science, Mineralisation, Time resolved XAS, Speciation.

Names	Affiliations	Areas of Interest / Techniques
J. Robinson	Department of Physics, University of Warwick	Condensed matter physics, SXS, Electrochemistry
A. E. Russell	Department of Chemistry, Southampton University	Electrochemistry, Fuel cells, Surface XAS, Atomic XAS
P. J. Sadler	School of Chemistry, University of Edinburgh	Chemical biology, Transport proteins, Chelation and metal uptake, Therapeutic materials,
G. Sankar	Davy Faraday Research Laboratory, The Royal Institution	Materials chemistry, Combined techniques, Catalysis, Microporous solids
A. V. Sapelkin	Department of Chemistry, De Montfort University	High Pressure, Melts, Condensed matter physics, Combined techniques, Biomaterials. Software development.
G.C. Saunders	School of Chemistry, Queen's University of Belfast	Sorption, Water remediation, Flocculation and nucleation, Time Resolved XAS.
P.F. Schofield	Department of Mineralogy, Natural History Museum	Environmental and Earth Science, Mineralogy, Heavy metal contamination
K. R. Seddon	School of Chemistry, Queen's University of Belfast	Ionic liquids, Homogenous catalysis, Heavy metal sorption
S. Shaw	Earth Sciences Department, , University of Oxford	Microbiology, biomineralisation, metal uptake, biofilms, XAS
M. E. Smith	Department of Physics, The University of Warwick	Solid state NMR, biomaterials, SXS, Amorphous materials, Nanocrystalline materials
S.R. Stürzenbaum	Biosciences, Cardiff University	Soil science, Heavy metal and arsenic uptake in bio-organisms, XAS, XANES
D. J. Vaughan	Earth Sciences, University of Manchester	Geochemistry, Earth and environmental science, speciation and mineralisation
M.J. Went	Physical Sciences, University of Kent	Radiopharmaceutical drugs
R. Wogelius	Earth Sciences, University of Manchester	Earth Science, Geochemistry, Surface chemistry, Speciation.

Names	Affiliations	Areas of Interest / Techniques
N. A. Young	Department of Chemistry, The University of Hull	Combined XAS / Matrix isolation studies,