# Review of BM30B FAME

## November 2009

## Introduction

BM30B is a French CRG instrument for X-ray absorption spectroscopies. It became operational in 2002 and was previously reviewed in 2005. The scientific programme is centred on Environmental Science and Geochemistry projects, with a special emphasis on speciation of elements in complex natural samples and geological fluids. Over the past 5 years, several new capabilities have been added which complement the programme in x-ray absorption spectroscopy, including development of a crystal analyser for eliminating fluorescence backgrounds, microbeam capabilities, a high pressure and temperature cell for XAFS studies, and x-ray Raman and emission spectroscopies.

The staff have developed their own high quality experimental programme with an emphasis on geological fluids and have provided superb user support. The user community is comprised principally of environmental scientists and geochemists, with a significant proportion of materials scientists. These users are mainly drawn from France but groups from countries as far away as Australia have made regular use of this beamline.

Continuation of this excellent beamline and associated scientific programme is strongly recommended. Several proposals to ensure future success through addition of a new staff member and technical upgrades ranging from a new detector to the possible relocation on a new source are presented.

## Research by Users

Research publications are predominantly in Environmental Sciences (46% of 102 publications from 2005-9) and Geochemistry (19%). The remainder are primarily

Materials Science (25%), Biology (5%), Catalysis (3%), and Instrument/Methods Development (3%). The panel considered the geochemical research in the area of hydrothermal fluids to be world-leading (e.g., gold speciation in S-bearing fluids, Cu chloride speciation in brines at high pressure and temperature). This work is leading to new understanding of hydrothermal transport and deposition of metals. The Environmental Sciences research is considered to be world-class (e.g., toxicity of nanoparticles, speciation of Zn in plants, adsorption of As on iron oxide nanoparticles, speciation of heavy metals in soils). This work may lead to applications for mitigating potential toxic effects of nanoparticles and the removal of ground water contaminants. The materials science research is generally good, but this is not the major strength of the beamline.

We recommend a continuing emphasis on environmental science and geochemical research. There does not seem to be a strong need to expand the activities to further communities given the high oversubscription rate and quality of applications for the available beamtime (3 for CRG, 7 for ESRF, overall average 4.3). Users are drawn mainly from France but also from Italy and other countries within and outside Europe (e.g., Australia).

**Research by Staff**

The main interest of the beamline staff is in geothermal fluids and their research in this area has contributed to a world-leading position. The development of specialized high pressure and temperature cells and their implementation on the beamline have been an important part of this activity. The staff have also developed productive collaborations with many of the user groups.

**Technical Status of the Beamline**

The previous panel report recommended that KB focusing optics and a crystal analyser spectrometer should be added to the instrument, and these have been successfully

implemented during the past five years. The new analyser is allowing high quality XANES spectra to be collected from complex and dilute samples with high fluorescence backgrounds. Further improvements are expected when the five crystals are all implemented. Additional analyser crystals would be desirable but are not practical due to space limitations in the hutch.

The present state of the beamline is as good as it can be given the location on a shared Bending Magnet. The minimum spot size of 15 x 15 micron in the present configuration is useful for high spatial resolution experiments. The beam stability is good. The present detector is aging and several detector elements have failed, and there is a clear need for a new detector to increase count rates significantly and hence the quality of data. In the current configuration and depending on sample type, the sensitivity is limited to >100 ppm even for heavy elements. X-ray Raman and emission experiments are now possible in addition to absorption spectra, extending the capabilities of the beamline.

Various sample environments are provided by a liquid helium cryostat, a high pressure and temperature autoclave, a gas mixing cell, an electrochemical cell, and a furnace. Other specialized environments such as DAC's and Paris-Edinburgh cells have also been used but are not generally available.

We support the proposal to add an optical Raman system that will provide complementary information for many experiments. The possibility of collecting x-ray diffraction data in combination with absorption spectra is attractive, but this may be difficult to implement in the present available space.

The facility is very user friendly due to the engagement of the staff and training activities such as the annual FAME+ school.

**Future Technical Developments**

The present space available for the instrumentation and its development is very limited and is an obstacle to future improvements. In addition, the control cabin is very small and uncomfortable for users. The beamline is limited by the small angular fan (2 out of 6 mrad) available from the shared bending magnet, and by the low brilliance of this source. The result is a lack of sensitivity to very dilute samples and a limit to the microfocusing that can be achieved. A new source will be needed to improve this situation. We recommend that relocation to a dedicated undulator source is considered very seriously. The world-class science performed on this beamline justifies this upgrade. A less desirable option could be a dedicated bending magnet. This relocation would provide more space for future upgrades of instrumentation and better facilities for users.

**Future Scientific Directions**

The extension of the scientific programme to higher pressure and temperature studies of synthetic fluids is recommended, linked with studies of natural fluids in inclusions. A new cell capable of pressures up to 10 kbar will be required to enable this research, which could include studies of supercritical water/$CO_2$ fluids of relevance to geochemical processes and carbon sequestration.

An important goal for environmental science studies is to extend the sensitivity of XAFS spectroscopy to lower element concentrations typical of many contaminated sites. This can be achieved by the provision of a new, more efficient detector, further development of the crystal analyser, and a brighter source. Geomicrobiology and environmental nanotechnology are other areas that will benefit from these improvements.

Both of the above areas have potential technological applications that could result in societal benefit, further expanding the user base. With continuing improvements, a strong user base, and dedicated and innovative staff, BM30B should remain a competitive and productive beamline for the coming decade.

## Staff

The panel was impressed by the enthusiasm, dedication, and scientific and technical expertise of the beamline staff. There are currently two 50% beamline scientists, a full time operations manager, and a postdoctoral scientist providing additional support only until 2/2010. Technical support is provided by two CNRS technical engineers. This present support is adequate for user operations (with an effective participation of somewhat more than 50% by the two beamline scientists) but this does not allow for the progression of in-house technical and scientific developments. This situation will become more severe when the postdoctoral support ends in 2010, as this position is unlikely to be refilled.

We recommend that an additional permanent engineer position is created to provide much-needed user support and take charge of some of the technical developments of the beamline and of novel sample environments.

## Summary of Recommendations

• Continuation of the outstanding geochemistry and environmental science programmes is recommended, with exploration of potential applications that could result in societal benefit.

• To strengthen these programmes as well as user support, we recommend that an additional permanent engineer position is created.

• We recommend that relocation to a dedicated undulator source is considered very seriously. The world-class science performed on this beamline justifies this upgrade. A less desirable option could be a dedicated bending magnet.

• There is a clear need for a new detector to increase count rates significantly and hence data quality.

• The construction of a new cell capable of pressures up to 10 kbar is required for future hydrothermal fluids research.

• We support the proposal to add an optical Raman system which will provide complementary information for many experiments. The possibility of x-ray diffraction facilities should also be considered.

**Review Committee**

John Paul Attfield

Gordon E. Brown Jr.

Simone De Panfilis

Jean Dubessy

Carmen Sanchez Valle