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J. S. C. McKee
University of Manitoba

G. R. Smith
University of Manitoba

A. A. Mirzai
University of Manitoba

M. S. Mathur
University of Manitoba

N. M. Halden
University of Manitoba

See next page for additional authors

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HIGH ENERGY PROTON INDUCED X-RAY EMISSION AND ITS APPLICATIONS

J.S.C. McKee*¹, G.R. Smith¹, A.A. Mirzai¹, M.S. Mathur¹, N.M. Halden², C. Pinsky³ and R. Bose³

¹Accelerator Centre, Department of Physics, University of Manitoba, Winnipeg, Manitoba, Canada R3T 2N2

²Department of Geological Sciences, University of Manitoba, Winnipeg, Manitoba, Canada R3T 2N2

³Department of Pharmacology and Therapeutics, University of Manitoba, Winnipeg, Manitoba, Canada R3E 0W3

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Abstract

High energy protons of energies between 30 and 60 MeV are particularly valuable in particle induced X-ray analysis of elements in the rare earth region and above. Transmission samples of thickness up to 100 μm can be used, and damage to both biological and mineral samples is minimal. Detection limits are estimated, and the use of Fourier transformation techniques in the analysis of data is discussed in some detail. The quality of the raw data from mineralogical and biological experiments is such as to permit immediate identification of all elements above Germanium present at the part per million level or above in a sample. Particle induced K X-ray spectra from Au ore, sperrylite, rutheniridosmine and autonite are shown, indicating that all elements from As to U are readily identified when K X-rays are generated by protons of appropriately high energy.

KEY WORDS: X-rays, high energy protons, particle-induced X-rays

*Address for correspondence:

J. S. C. McKee,
Accelerator Center, Department of Physics,
University of Manitoba,
Winnipeg, Manitoba, Canada R3T 2N2
Telephone No. (204) 474-9874

Introduction

For over a decade Particle Induced X-Ray Emission (PIXE) analysis has been carried out at the University of Manitoba using high energy protons between 30 and 50 MeV as the inducing particles. K X-rays are then detected by means of hyper pure Ge (HPGE) and Si(Li) detectors, and detection limits are typically 1 ppm or below for most elements above silicon in the Periodic Table.

The rationale for high energy particle induced X-ray emission or high energy proton PIXE (HEPP) as it is currently referred to is that it yields data of high resolution in a region of study where ambiguity is a feature of low-energy PIXE experiments, which utilize protons of 3 MeV energy as the inducing particle.

The fact that K X-rays are the signature for each element studied by HEPP ensures that for the rare earth region and above unambiguous determination is possible even when complex mixtures of elemental concentrations are present.

Prior to 1989, virtually all high energy proton induced K X-ray spectroscopy was studied at the University of Manitoba Accelerator Centre (McKee et al., 1976; Ramsay et al., 1979; Durocher et al., 1988; McKee et al., 1990). However, Peisach and Pineda (1990) reported confirmation of much of the earlier Manitoba work and produced a detection limit analysis of elemental standards in the rare earth region that indicated dramatically the power of HEPP at their chosen energy of 60 MeV (Peisach and Pineda, 1990).

High energy proton PIXE uses protons to remove inner shell electrons. At the energies quoted for both the Canadian and South African experiments the yield of K X-rays is high for all medium Z elements and good for both high and low Z materials. Indeed, data from K X-ray studies of moderately light elements exhibit high signal to background quality and enable elemental concentrations to be determined at least as precisely as by traditional low energy PIXE methods.

The preparation of targets in HEPP is often straightforward. Because of the low dE/dx of 40 to 60 MeV protons, little energy is dropped in (for example) a 40 μm ore sample. As a result, no visible damage is

observed on the bombarded surface of our solid targets. Not only is radiation damage from low current beams almost non-existent, self-absorption of the K X-rays generated is low for medium and high Z elements and the raw data from such studies require little correction prior to the determination of precise concentrations. For analysis of biological specimens, the high energy protons, in conjunction with a thin transmission target (often the thickness of a stopping target at 1-3 MeV), ensure that analysis is both of high resolution and simultaneously non-destructive. Standard HEPP experiments use millibeams of 2 mm diameter, and microbeams are being developed which can probe materials at the micron level as and when such a facility is required (McKee and Smith, 1989).

The HEPP technique requires a cyclotron as a particle source since most electrostatic accelerators are limited in accelerated particle energy to less than 20 MeV by electrical breakdown of insulators. At present, there are two laboratories that are pursuing HEPP as an analytical technique, Manitoba and the National Accelerator Laboratory in Faure, South Africa (Peisach and Pineda, 1990). Because most cyclotron laboratories are experiencing a reduction in pure nuclear physics research, projects like HEPP have become more attractive. At the same time, cyclotrons dedicated to medical applications are proliferating in major population centres. HEPP provides these medical facilities with an analytical technique that is both inexpensive and easy to implement subsidiary to their isotope production or therapeutic programmes.

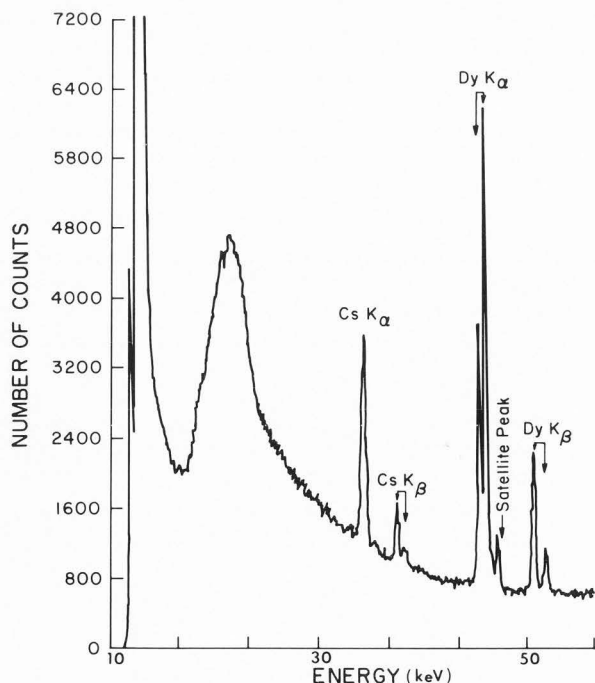


Fig. 1 Typical X-ray spectrum of mouse brain sample. Dy has been added to the homogenized samples for concentration analysis.

Minimum Detectable Limit using HEPP

In determining the minimum detectable limit (MDL) for elemental concentrations using HEPP, it is important to obtain some relationship between proton bombardment time, beam current, target thickness and K X-ray detection efficiency for the sample under study. In PIXE analysis at 40 MeV, concentrations at the 1 ppm level can be measured for all elements from As to U. This level is, in practise, approximately three orders of magnitude below that of scanning electron microscopy (SEM) and two orders of magnitude better than low energy PIXE results for elements above Germanium. This result can be deduced from analysis of samples incorporating known concentrations of a marker element. In the case of studies of Cs uptake in mouse brain (McKee et al., 1981; McKee et al., 1985), Dy was used as a calibration standard and was present in brain homogenate at tens of ppm levels (see Figure 1). On some occasions, however, it is necessary to look for elements at a lower level. An experiment which searched for single photon emission following the double K-shell ionisation of Rubidium using the HEPP detection facility was successful in measuring concentrations to a limit of 100 ppb (Al-Ghazi, 1982). This technique can be tailored to the needs of a particular analysis. However, electronically removing pile up and limiting count rates to values consistent with minimal noise levels, increases overall running time significantly. Whereas 1 ppm may be achieved routinely in a 20 minute run for a transmission target with 1 nA current, 100 ppb may require days and considerable effort. The K X-ray data, however, in both cases will be clearly resolved from competing processes.

The determination of elemental concentrations from PIXE analysis requires doping the sample with a standard where that is possible, or comparison with a standard where this is not. In a typical experiment at Manitoba using a liquid homogenate, a drop of approximately 20 mg mass is placed on a low background mylar slide and then exposed to a 2 mm proton beam. When a marker is included as a component of the drop in known concentration, then comparative concentrations of adjacent elements can be obtained directly. In the absence of a dopant, however, estimation of elemental concentration must be performed with care. For example, a 2 mm beam falling on a 5 mm diameter drop may give excellent spectra. However, under vacuum, the drop may dry under exposure and more of the material under investigation be drawn to the centre of the drop. The final result is that the elemental concentration, as deduced, increases with time because of the contraction of the drop to what may eventually be the size (diameter) of the 2 mm beam.

The behaviour of drops under vacuum can, however, be studied independently of the experiment, using a Nuclear Magnetic Resonance (NMR) technique (Guillot et al., 1989). This involves observation of the drying process through one dimensional profiles and three dimensional NMR imaging in a manner similar to that of

High Energy Proton Induced X-Ray Emission

Guillot in a similar connection. For solid samples, direct comparison with a known standard of transmission thickness will provide useful data at the ppm level directly.

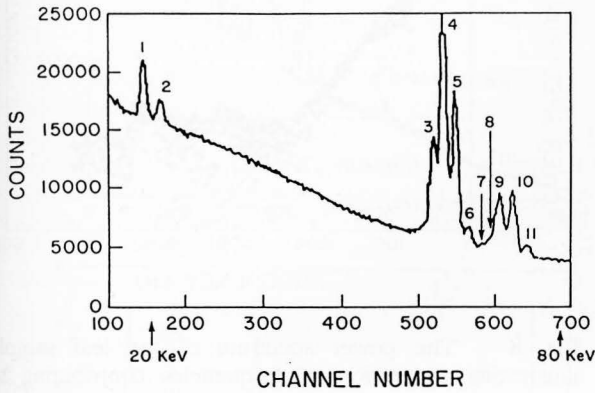


Fig. 2 Typical X-ray spectrum of a rock sample at an incident proton energy of 40 MeV (HPGe detector). Elements identified were: 1-RuK α , 2-RuK β , 3-ReK α , 4-OsK α , 5-IrK α , 6-PtK α , 7-AuK α , 8-ReK β , 9-OsK β , 10-IrK β , 11-PtK β .

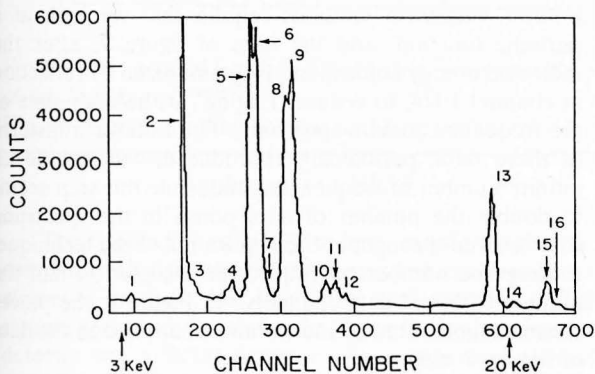


Fig. 3 Spectrum of the same rock sample as in Fig. 2 (Si(Li) detector). Elements identified were: 1-CaK α , 2-MnK α , 3-MnK β , 4-CuK α , 5-ZnK α , 6-GaK α , 7-ZnK β , 8-GaK β , 9-AsK α , 10-AsK β , 11-BrK α , 12-BrK β , 13-RuK α , 14-RhK α , 15-RuK β , 16-RuK β . Peaks 13 through 16 correspond to peaks 1 and 2 of Fig. 2.

Analysis of Data

Figures 2, 3, 4, 5, 6 show data from a variety of experiments which vary in spectroscopic quality as the complexity of the sample, its thickness, composition and transparency to the proton beam differ. Analysis of all samples depends upon the separation of K X-ray peaks from background, despite the fact that peaks are almost always evident in the raw data prior to reduction.

Most PIXE data analysis follows the rather traditional path of fitting some kind of function to the slowly varying

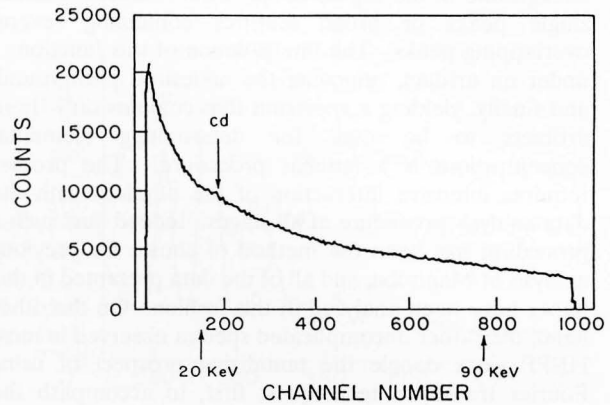


Fig. 4 Typical X-ray spectrum of a tree bark sample at an incident proton energy of 40 MeV (HPGe detector).

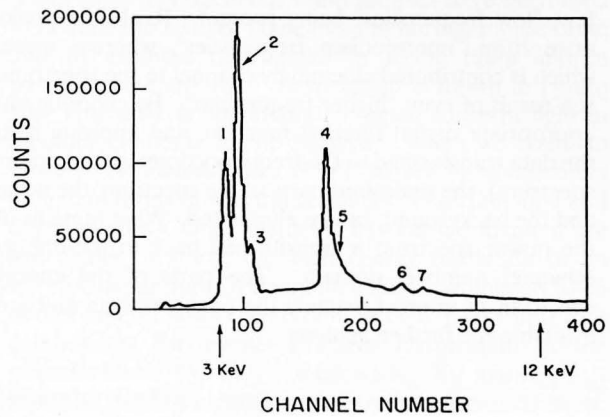


Fig. 5 Spectrum of the same tree sample as in Fig. 4 (Si(Li) detector). Elements identified were: 1-KK α K β , 2-CaK α , 4-MnK α , 5-MnK β , 6-CuK α , 7-ZnK α .

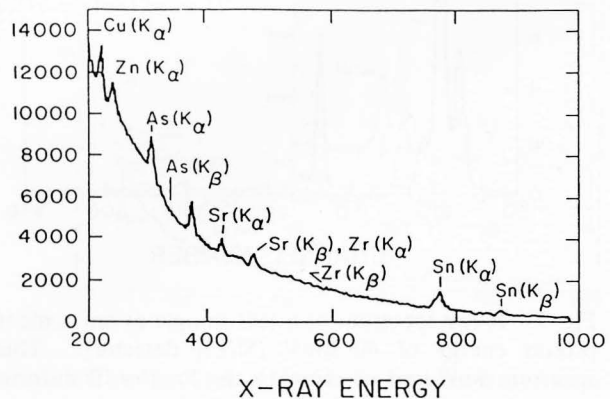


Fig. 6 Spectrum of 500 μ m grains of float glass irradiated by a 1 nA 40 MeV proton beam. Note the Sn signature. The composition and concentrations of the elements help identify the source of the glass.

background in the region of spectrum artifacts, whether single peaks or broad features containing several overlapping peaks. The interpolation of this function under an artifact, removing the undesired background, and finally, yielding a spectrum that contains only those artifacts to be used for determining elemental concentrations is a familiar procedure. The process requires intensive interaction of the physicist with the data analysis procedure at all stages. Indeed, just such a procedure has been the method of choice for previous analysis at Manitoba, and all of the data presented in this paper have been analyzed in this fashion. On the other hand, the rather uncomplicated spectra observed in most HEPP work dangle the tantalizing prospect of using Fourier transform techniques: first, to accomplish the background stripping process and second, to include noise reduction, or data smoothing, as a part of the data reduction procedure. This decreases the amount of interactive involvement by the physicist during the process. Conceptually, the slowly varying background spectrum, contributed by all channels, is the result of a few, "low frequencies" being present. Real data peaks arise from "intermediate frequencies", whereas noise, which is contributed channel by channel to the spectrum, is a result of even "higher frequencies". By choosing an appropriate digital filtering function, and applying it to the data transformed to the frequency domain (the power spectrum), the undesired parts of the spectrum, the noise and the background, can be eliminated. What remains of the power spectrum is transformed back to the energy (channel number) domain. The parts of the energy spectrum of interest, namely the peaks, remain and are available for further analysis.

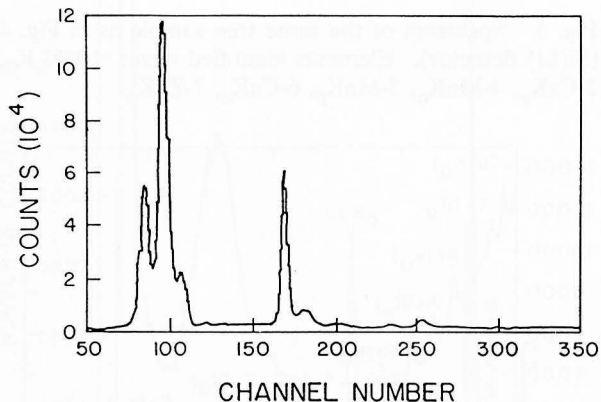


Fig. 7 X-ray spectrum of a leaf sample at an incident proton energy of 40 MeV (Si(Li) detector). This spectrum was used as input to the Fourier Transform spectrum stripping programme.

We at Manitoba have been exploring this possibility for the past year. A report on our progress, illustrated by data taken on tree leaves bombarded by 40 MeV protons,

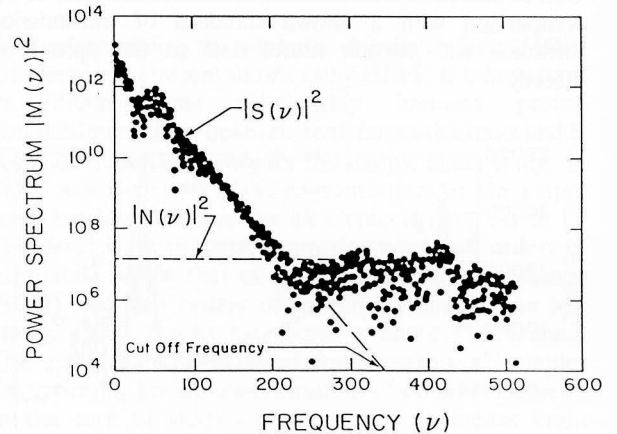


Fig. 8 The power spectrum of the leaf sample illustrating separation of frequencies contributing to statistical fluctuation in the data.

is shown in figure 7. One problem that arises in applying Fourier Transform techniques to these experimental data is that the peaks of an X-ray spectrum have regularly varying widths. Following a technique developed by Kennett et al. (1978), the horizontal scale of the spectrum is adjusted to produce peaks with constant widths. Fourier transform methods require that we look at a periodic function, and the data of figure 7, after the indicated energy adjustment, is symmetrized by reflection at channel 1024, to reduce "ringing" at the endpoints of the frequency domain spectrum. The Fourier transform of these data, periodically reproduced, will include an infinite number of frequencies, and while this step seems to double the number of data points in the spectrum, care is taken throughout the application of the technique, to keep the number of frequencies accepted to half the number of initial data channels. In Figure 8, the power spectrum generated by the technique, applied to the data of Figure 7 is shown.

Notice that the spectrum divides nicely into contributions from the signal (peaks plus background) and noise (statistical fluctuations). The background spectrum is contributed by the information contained in the first few channels of the power spectrum. The cutoff frequency, characteristic of the signal, is identified on Figure 8. An optimal filter, is developed and applied to the data. An indication of the degree of success, and of our progress is seen in Figures 9a and 9b. Figure 9a shows how successfully the background spectrum is reproduced by the technique. Note that an "oscillation" extends from about channel 400 to channel 700. Finally, in Figure 9b, the background stripped noise suppressed version of the spectrum of Figure 7 is shown. Success in applying the Fourier Transform technique to the problem of HEPP data analysis will now be defined as regularizing the small problem indicated in Figure 9a. This work is proceeding.

High Energy Proton Induced X-Ray Emission

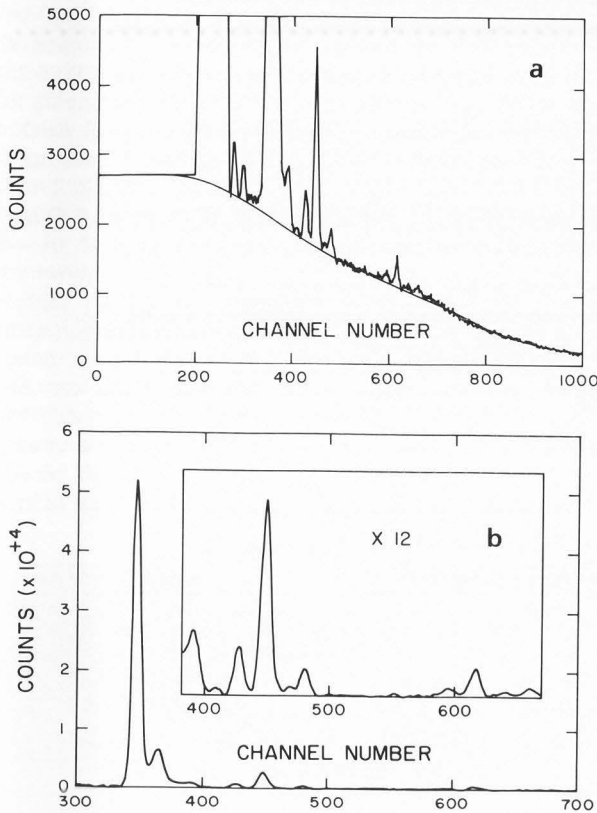


Fig. 9a Background function fitted to the X-ray spectrum of the leaf sample.

Fig. 9b Corresponding background-free peaks.

Applications of HEPP

The application of HEPP to leaf analysis, despite the comparatively light mass of the micro- and macro-nutrient elements involved, has been discussed recently, and a sample spectrum was shown in Figure 7. In this case the detector was a Si(Li) detector with energy resolution of 150-170 eV full width half maximum (FWHM) at 6 keV. Line widths from calibration vary from 100 eV at 1 keV to 250 eV at 20 keV, the top of the useful energy range for most purposes. This analysis can detect metallic elements in leaf and may be used as a mineralogical tool (Mirzai et al., 1990).

Another figure, 6, shows a spectrum from really thick 500 μm grains of float glass. Again, this is raw data showing directly the visibility of K X-ray peaks from lighter elements. Spectra like this can be used in forensic science applications and often identify the source of the glass splinters concerned. Current research relating to aging - in particular Parkinson's Disease - involves the analysis of mouse brain from an experimental genetically pure strain of mice after manganese chloride has been administered in measurable quantities by interperitoneal injections. It is known that miners of manganese ore have a more than normal incidence of Parkinson's Disease and uptake in the brain in relation to the environment is an appropriate observation to make.

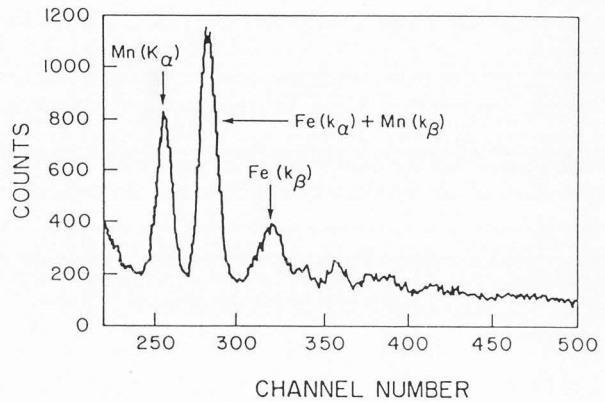


Fig. 10 Spectrum of homogenized mouse brain after interperitoneal injections of MnCl.

Data from homogeneous brain samples have been used to deduce the level of manganese in normal mouse brain and to examine the uptake in whole brain and in particular the striatum. A spectrum is shown in Figure 10. This work is continuing but again concerns light to medium elements in the analysis. When we come to samples of mineral ore, HEPP leaves its competitors behind in terms of spectral resolution, detection limit and depth studies. Once elements beyond Germanium are the object of study, the high energy resolution of an intrinsic hyper pure Germanium detector, nominally 0.4% at 122 keV enables the resolution of K peaks into four components for elements above Dysprosium in the periodic table. Not only can known K_{α}/K_{β} and $K_{\alpha 1}/K_{\alpha 2}$ ratios be used to identify and quantify concentrations of

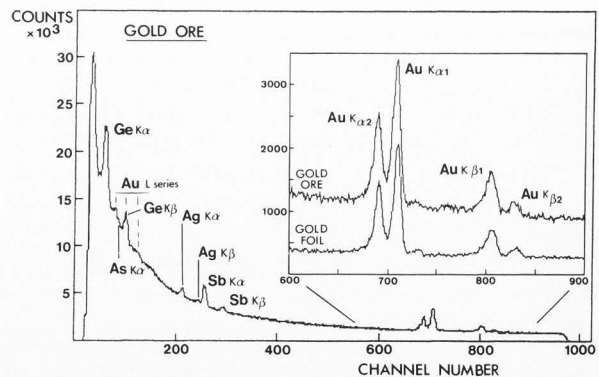


Fig. 11 Raw spectrum of the Cochenour-Williams Au ore sample showing K x-ray lines. The inset spectrum shows an expansion of the region containing the four Au K x-ray lines, and a reference spectrum from Au foil for comparison. The position of the As K_{α} peak was checked in a similar fashion against a reference spectrum collected from As_2O_3 .

neighbouring elements in a sample, but on the rare occasions when γ -rays from two or multiple stage nuclear processes are present, these, being single, are easily removed. Indeed, the analysis of spectra obtained for various proton beam energies 20 MeV and above can indicate the specific process generating the additional and unwanted visitor: e.g. the (p,xn) reaction concerned. For a discussion of the philosophy behind HEPP and the selection of an appropriate energy for the initiating proton beam see McKee et al., (1990).

Figure 11 shows a recent spectrum from gold ore bombardment with a 40 MeV proton beam compared to a similar spectrum from gold foil (Halden et al., to be

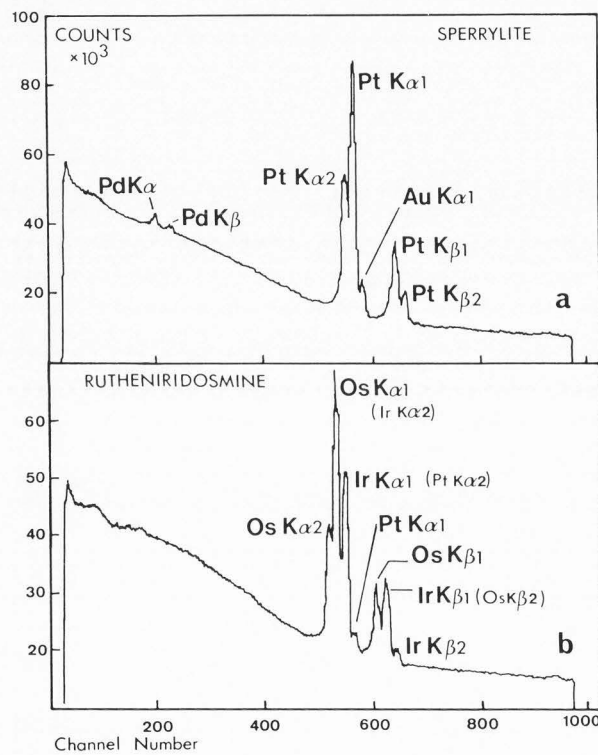


Fig. 12a Spectrum collected from sperrylite showing the four K X-ray lines for platinum. The Au $K_{\alpha 1}$ X-ray line is produced from Au created by a Pt(p,1n)Au reaction. The low-energy part of the spectrum is suppressed with a 32 mm paraffin filter. Still visible in the low energy part of the spectrum are K X-rays for Pd and escape peaks of Ge caused by proton back-scattering in the detector.

Fig. 12b Spectrum collected from rutheniridosmine, showing K_{α} X-rays for Os, Ir and Pt, and K_{β} X-rays for Os and Ir. The overlapping X-rays are shown in parenthesis after the label corresponding to the principal peak at these positions. The low-energy region is suppressed using the paraffin filter. Just visible are poorly developed peaks that correspond to K X-rays for Ru and Ge, the latter created by scattering in the detector.

published). The ease with which elements can be identified from the raw spectra is clear, and the region of L X-rays from Au that interferes so dramatically with arsenic in low energy proton PIXE measurements is indicated for interest. The K lines from gold in the HEPP experiment cannot interfere with the L lines from any element, and beam energy could in fact be tailored to peak yield for Au specifically, if required. Other spectra obtained recently are shown in Figure 12. A transmission diamond-cut sample of sperrylite is shown in figure 12a.) and rutheniridosmine in 12b.). Short experimental runs of ten to twenty minutes duration with 1 nA of proton beam on target produced these data. Again, as mentioned earlier, detection limits can be tailored to need. The background can be reduced by thinning the sample in each case. The data however are compressed, coming directly from a VAX 750 based collection system. Figure 13 shows a SEM picture of an

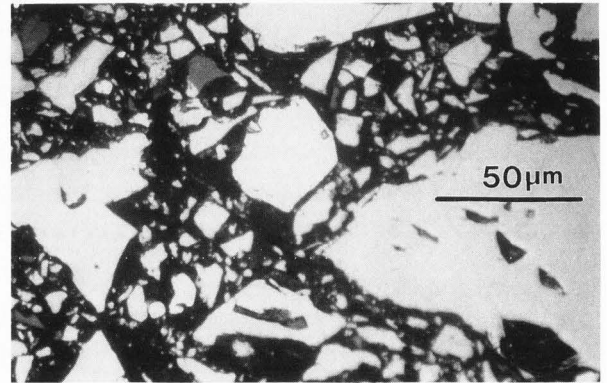


Fig. 13 Trout Lake (Manitoba) ore sample. Inclusions (bright areas) vary in size from 10 to about 250 μm . The gold in the inclusions of this sample is "invisible" to SEM techniques.

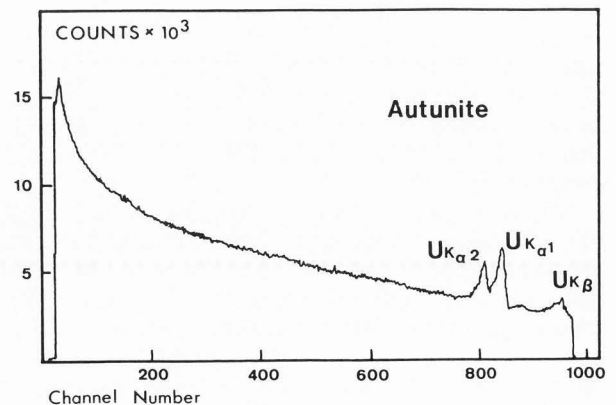


Fig. 14 X-ray spectrum from a thick autunite ore sample illustrating the clear signal from Uranium, the detection of three of the four K x-ray peaks. Cut-offs at high and low channel number are due to electronic instrumentation.

High Energy Proton Induced X-Ray Emission

Au-Ag-Hg alloy and 'invisible' gold in a sulphide sample obtained from Trout Lake, Manitoba. The inclusions shown are 250 μm in size and indicate in this case that for gold ore analysis, a millimetre beam of protons is well enough defined to yield a useful picture. A microbeam of protons if available would have been useful to study elemental concentration within an inclusion, but would not have yielded a figure for the global concentration of Au in the ore sample. Both studies however are interesting, and HEPP can be tailored to either. Finally, a thick slice of autunite gave an instant spectrum as shown in Figure 14. The cut-off is electronic and the spectrum was not optimised. It merely indicates that in principle all elements from As to U can be determined simultaneously from one sample using the same intrinsic germanium crystal as the detector.

Applications of HEPP to biological, medical and geological sciences should prove invaluable in the future.

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Discussion with Reviewers

U. Lindh: The overall characteristics of HEPP seem promising. Could you make some estimate of the accessibility and some comments on the expected proliferation of the technique?

Authors: Today, HEPP is indeed restricted to accelerator laboratories where a cyclotron of suitable energy is available. Protons of energies from 40 MeV to 70 MeV seem ideal. In traditional nuclear and sub atomic physics laboratories, however, competition for accelerator time is generally not as keen as in the past, and many such facilities are actively seeking alternative projects in applied areas that may extend their long term viability. At the same time, a plethora of medical accelerators has arrived upon the scene, many with a 42 MeV capability for neutron therapy. Such facilities can also be appropriate for analytical work of the kind described in our paper.

K. Malmqvist: You state in the section on minimum detectable limits that HEPP is "...two orders of magnitude better than low energy PIXE results for elements above Germanium." Apart from the fact that the conditions [X ray lines, matrix etc] under which the comparison is made are not given, I would like to question whether this is in fact at all true. Assuming a minimum detection limit of 1 $\mu\text{g/g}$ in HEPP, that would correspond to MDL's of hundreds of mg/g in ordinary PIXE for elements heavier than Ge. Since the detection limits of, for instance, lead in organic matrices is normally below 1 $\mu\text{g/g}$ this is as a general statement wrong. I would like the authors comments and ask them to elaborate slightly more on this point.

U. Lindh: HEPP is claimed to be two orders of magnitude more sensitive than low energy PIXE for elements of atomic number $Z > 31$. Johansson and Johansson (1976) observe MDL of roughly 1 ppm for $Z=46-92$. If these cited MDL are true, than your claimed limits are not two orders of magnitude less, but similar. I think this situation requests clarification.

Authors: As a general comment, perhaps our statement concerning MDL is too strong. Johansson and Johansson (1976) documented that the MDL for transmission targets in low energy PIXE can be 1 ppm, and as the referee suggests, concentrations of elements at this level have been measured. Those authors were, however, careful to point out at the time that the sensitivities quoted for K X-rays or L X-rays alone are not attainable in many real

samples in which interferences between K X-rays from one element and the L X-rays of another occur. This interference is in fact, a much more common occurrence than is indicated in their paper, and a frequent feature of mineralogical samples. Thick sample analysis, on other hand, in low energy PIXE has been discussed exhaustively by Teesdale et al. (1988). Here, again, sensitivities of 1 ppm are attainable, but the element under consideration, the matrix in which it is found, the absorber, and the beam energy are important factors in achieving such a MDL. Detection of many rare earth and higher Z elements is shown in their paper to be at the 100 ppm, rather than the 1 ppm level, and complex mixtures of heavier elements do not in practice yield a 1 ppm result. A recent paper by Przybylowicz et al. (1990) which describes the analysis of trace elements in black shales shows clearly that the limits of minimum detectability are in practice well above the ppm level. For comparison, they show that the limits of detection for an electron microprobe lie in the range from 300 to 500 ppm, and those for low energy PIXE are in excess of 100 ppm for some elements, while being at the several tens of ppm level for others. Our position would be that 1 ppm is attainable for HEPP routinely in such samples for all elements above Ge. We do agree, however, that similar sensitivities can be reached in certain selected low energy PIXE experiments where particular elements are concerned, but the universal capabilities of HEPP are not attainable in low energy analysis.

U. Lindh: If your MDL are achieved by using stopping thick targets, what are the advantages of micro-HEPP?

Authors: We emphasize that we have used the HEPP technique only in conjunction with thin, transmission targets. The speed of the 40 MeV proton, for example, matches the speed of the K-shell electrons in the moderate to heavy atomic species, so that the ionization process is particularly efficient. We can then use thin samples that allow isolation of particular features in the target. Furthermore, because the 40 MeV protons deposit small amounts of energy at the surface of the target compared to 3 MeV protons, the integrity of the target is not degraded by the bombardment process, and the observed ablation of samples common to low energy PIXE just does not occur in HEPP. Taken with the MDL information and the simplicity of the observed spectra, exploitable advantages of micro HEPP are many.

T. Cahill: I would like to see some quantitative examples of standard reference materials, or pre-analyzed ores with known concentrations investigated by the HEPP technique. This would make the reader comfortable with statements regarding MDL values of 1 ppm, mentioned in the text. Does such work exist?

Authors: Our practice has been to determine concentrations of elements by comparison with suitably chosen doping materials. Such materials have been reliably inserted and detected at the 1 ppm level in a number of different types of samples.

U. Lindh: Do you think your approach to spectrum analysis using Fourier transform techniques could be

successfully adapted to low-energy PIXE spectrum analysis?

Authors: In so far as the "broad spectral features" which contain many interfering K and L X-ray peaks, common to low energy PIXE spectra, are not reconstructed out of the few low frequencies associated with the background events, this could be done. As long as the low energy PIXE analysis restricts itself to measuring elemental concentrations in an energy regime where only K X-ray lines are excited, success would be assured.

U. Lindh: You state that microbeams of high-energy protons are developed to probe materials at the micron level. Do you mean 1 μm ? in that case, with what current?

Authors: Our proton microprobe, which is currently under development, is capable of measuring pixels roughly $10 \mu^2$, with a current of a few hundred picoamps. We emphasize that the Manitoba facility is unique, no other high energy microprobe yet exists.

K. Malmqvist: In discussing the use of a HEPP microbeam for analyzing geological samples, you claim that such a beam would have been useful for a detailed investigation within an inclusion. Since you are using a very penetrating ion beam to study mainly heavy elements, don't you think it would be very difficult to use a high lateral resolution when the depth variation could be significant and deep layers of different composition would add significantly to the signal from a particular irradiated pixel?

Authors: You are quite right in observing that our transmission beam could certainly excite features interior to a sample, if care were not taken to ensure that the sample were thin enough to eliminate such overlapping inclusions. Let us remind you that the HEPP technique eliminates the problem of surface ablation, so that damage to the sample is not a problem that leads to a lower limit to the thickness of the prepared sample. Stability and high lateral resolution will, of course, be extremely important, if depth profiling of a sample is required. The detection of scattered protons, in coincidence with X-rays in order to supply depth information may make experiments not only technically difficult but extremely time consuming. They appear possible, however.

T. Cahill: I would like to see a comparison of some sample run on both low-energy PIXE and HEPP. Many labs would be delighted to cooperate as we are all interested in this process, even if we don't have a HPGe detector.

Authors: Yes, such data do exist, and they were summarized in Durocher et al. (1988). There, HEPP and low energy PIXE spectra (Rodgers, et al. 1984) were compared for Durango apatite. The task of analysis of the unambiguous HEPP spectrum (Figure 1 of Durocher et al. 1988) is straightforward. We draw your attention to the broad spectral feature in the low energy PIXE spectrum of Rodgers, et al. (1984) (Figure 2 of Durocher et al. 1988), where no less than 20 individual L X-ray peaks have been introduced to completely "fill" the

High Energy Proton Induced X-Ray Emission

observed spectral feature. While auxiliary information may be present in other parts of the spectrum that could be used to verify some of the included peaks, it is clear that heroic efforts are necessary to identify the elements present in the sample from the observed low energy PIXE spectrum.

U. Lindh: You mention an experiment concerned with Parkinson's disease and central nervous system (CNS) uptake of Manganese. Do you by homogeneous brain samples mean homogenised samples? In that case, how do you separate the striatum?

Authors: Yes they are homogenised. The striatum is dissected out prior to homogenisation.

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