#### Feasibility Study and Design of a Proton Microprobe

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#### ABSTRACT

A proton microprobe can be thought of as a scanning proton microscope. It is a precision tool. The microprobe described in this paper consists of a set of four high quality quadrupoles which focus a proton beam to a spot of 10µm diameter. This facility, when used in conjunction with existing proton induced x-ray equipment, will make possible the monitoring of particulate and fibrous matter as well as the measurement of environmental composition. It is intended to establish the instrument for use in conjunction with the University of Manitoba 20-50 MeV proton Cyclotron. The feasibility in terms of design and cost of the system is assessed. Effects of aberrations are discussed. Uses of the microprobe in the fields of environmental toxicology, materials science and metallurgy are outlined.

### 1. Introduction

Particle induced x-ray emission (P.I.X.E.) has become a widely used tool in the microanalysis of biological, environmental and metallurgical samples. There is no need to engage in a lengthy description of the technique in this report, as detailed accounts can be found elsewhere in the literature (e.g. 1,2). P.I.X.E. is a powerful tool for a variety of reasons: It is non-destructive, and has a high sensitivity in terms of ppm and even ppb due to the development of high resolution x-ray detectors. Inherent difficulties with the technique are: The requirement of an accurate knowledge of x-ray production cross sections as a function of incident particle energy for all elements under consideration, and at low energies, difficulty in unfolding peaks in the observed spectra.

The University of Manitoba sector-focused cyclotron produces proton beams in the energy range 20-50 MeV with currents of the order of a few microamps at all energies. Such beams have been used for P.I.X.E. studies. Two main objectives are identified with this work: i) An understanding of K-shell ionization processes and of proton induced x-ray emission process; ii) The use of x-ray fluorescence as a tool in the microanalysis of environmental samples. The first theme involving as it does the intercomparison of theory and experiment gains some motivation from the second.

Most laboratories interested in P.I.X.E. work use proton beams of 1-5 MeV in energy from Van de Graaff accelerators. This is due more to the relative abundance of such accelerators than to any suggestion that their energy range is the optimum for x-ray studies. It is, in fact, believed that the energy range 10-40 MeV is a much more suitable one for the precise study of medium and heavy elements. Results obtained in this laboratory support this statement<sup>3</sup>).

So far bulk samples only have been investigated. The next step in the development of an analysis system is to establish a beam focusing device which can focus the incident proton beam to a diameter of the order of 10µm. A beam of this size can then be scanned across a sample to obtain positional information on the distribution of elements across the sample and to establish physical form. Such a beam would be used in microstructure studies. Applications will be discussed in a later section of this paper. The focusing system is called a proton microprobe and the purpose of this report is to examine the feasibility of establishing such a system at the Cyclotron Laboratory of the University of Manitoba.

# 2. The Production of a Proton Microbeam

The proton beam leaves the cyclotron through a slit of dimensions 5 mm x 12 mm in x- and y- coordinate respectively and propagates along the z-axis. It is then focused by a quadrupole doublet (QD 1,2) to produce a 4 mm x 4 mm spot 5.75 m downstream (Fig. 1).



The field strengths required as a function of proton energy are presented in columns 3 and 4 of Table 1.

# TABLE 1

	Tp	(MeV)	P(MeV/c)	-Q <sub>D1</sub> (mT)	(mT)	±Q <sub>0</sub> (mT)	*Q <sub>I</sub> (mT)		
		20	194.76	154.23	197.29	252.14	545.78		
		25	218.03	174.90	220.85	282,27	610,99		
		30	239.16	192.03	242.34	309.62	670.21		
		35	258.66	207.69	262.10	334,87	724.85		
		40	276.88	222.10	280.47	358.46	775.91		
		45	294.06	236.12	297.97	380.70	824.05		
		50	310.37	249.09	314.12	401,81	869.76		
т.,		proto	n energy (He	(Y)					
P	1	proton momentum (MeV/c)							
( QD1	1	field strength of the "first quad" used to focus the beam on the collimator slit (-ve; defocuses in $x_\star$ focuses in $y)$							
( aDS	z	I field strength of the "2nd quad" used to focus the beam on the collimator slit +ye; focuses in x, defocuses in y)							
( Q0	z	field of the first and fourth (outer) quadrupoles of the quadruplet							
101	I.	field	of the seco	nd and thi	rd (inner)	quadrupo	oles of the	quadruplet	
	$T_p p P$ $\left\{\begin{array}{c} Q_{D1} \\ Q_{D2} \\ Q_{D2} \\ Q_{1} \end{array}\right\}$	т <sub>р</sub> = р = а ( с с с с с с с с с с с с с	$T_{p}(hev) = \frac{1}{20}$ 20 25 30 35 40 45 50 40 45 50 60 7 a proto $\left\{ \frac{9}{00} + \frac{1}{2} + \frac{1}{20} + \frac{1}{2$	T <sub>p</sub> (HeV) P(HeV/c) 20 194.76 25 218.03 0 239.16 35 258.66 40 276.88 45 294.06 50 310.27 T <sub>p</sub> # proton energy (f 70 2 16.64 strength of 3116 (vs; defco) 0 2 2 16.64 strength of 3116 strength of 311	T <sub>p</sub> (MeV)         P(MeV/c)         -G <sub>g1</sub> (m1)           20         194.76         156.23           25         218.03         174.90           30         239.16         192.03           35         258.66         207.67           40         276.88         222.10           45         294.06         236.12           50         310.37         248.09           T <sub>p</sub> # proton emergy (MeV)         P           9.111         (rws) efficients in a. def           102         # field strength of the "first market in a."           9.2         # field strength of the "strength of the "strength" of the strength of the "strength of the "strengt strength of the "strengt strength of the "strength of	Tp(HeV)         P(MeV/c)         -Qp1 (m7)         Qp2 (m7)         Qp2 (m7)           20         194.76         154.23         197.29           25         218.03         174.50         220.46           30         239.16         192.03         242.34           30         239.16         192.03         242.34           40         276.48         227.10         280.47           40         276.48         227.10         280.47           45         294.06         236.12         29.797           50         310.37         249.09         314.12           Tp         #         proton meantum (MeV/C)         201           {0         # frid is trength of the "frist outsd" usilit (-ws) decourses in s., decourses in f.11         202           0         # frid is trength of the frist doutsd' usilit (-ws) decourses in f.a., decourses in	$ \begin{array}{c} T_p(\text{HeV}) & P(\text{HeV}/c) & -Q_{0,1} & Q_{0,2} & zQ_{0} \\ (mT) & (mT) & (mT) \\ \hline 20 & 194,76 & 135,22 & 197,28 & 252,14 \\ 25 & 218,03 & 174,50 & 220.66 & 282,27 \\ 30 & 239,16 & 192,03 & 244,34 & 099,62 \\ 35 & 258,66 & 207,68 & 262,10 & 334,87 \\ 40 & 276,88 & 222,10 & 280,47 & 358,46 \\ 45 & 254,66 & 235,12 & 297,97 & 380,70 \\ 50 & 310,37 & 249,09 & 314,12 & 401,81 \\ \hline T_p & z & proton emergy (MeV) \\ P & z & proton emergy (MeV) \\ P & z & proton emergy (MeV) \\ Q_1 & z & friel a trength of the "first quad" used to for $11t (-ve; defocuses in $x, defocuses in $y] \\ \hline Q_0 & z & friel of the might of the "first quad" used to for $11t (-ve; defocuses in $x, defocuses in $y] \\ \hline Q_1 & z & friel of the friet for the forth (stor) quadrup; $100 might of the the the forth (tinner) quadrup; $100 might of the the the the forth (tinner) quadrup; $100 might of the the the the forth (tinner) quadrup; $100 might of the the the the forth (tinner) quadrup; $100 might of the the the the forth (tinner) quadrup; $100 might of the the the the the forth (tinner) quadrup; $100 might of the the the the the the the the the the$	$ \begin{array}{c} T_{p}(\text{NeV}) & P(\text{NeV}/c) & -Q_{0.1} & Q_{0.2} & xQ_{0} & xQ_{1} & (\text{mT}) \\ \hline & (\text{mT}) & (\text{mT}) & (\text{mT}) & (\text{mT}) \\ \hline & 20 & 194,76 & 154,23 & 197,29 & 252,14 & 845,78 \\ \hline & 20 & 128,16 & 192,02 & 242,34 & 507,62 & 670,27 \\ \hline & 30 & 239,16 & 192,02 & 242,34 & 507,62 & 670,27 \\ \hline & 30 & 239,16 & 192,02 & 242,34 & 507,62 & 670,27 \\ \hline & 35 & 258,66 & 207,69 & 262,10 & 334,87 & 724,485 \\ \hline & 40 & 276,48 & 222,10 & 260,47 & 358,46 & 775,81 \\ \hline & 45 & 294,06 & 236,12 & 297,97 & 380,70 & 824,05 \\ \hline & 50 & 310,37 & 249,09 & 314,12 & 401,81 & 669,76 \\ \hline & T_{p} & x & proton momentum (MeV/C) \\ \begin{cases} 0_{11} & x & relation simple (MeV) \\ p & x & proton momentum (MeV/C) \\ \hline & 0_{12} & x & frick thereight of the "first quad" used to focus the beam is lift 'weight focus in x, defocus in x) \\ \hline & 0_{12} & x & frick thereight of the "That duad" used to focus the beam is lift or the first and duad' used to focus the beam is lift weight of the first duad" used to focus the beam is lift of the first and fourth (user to quadrupoles of the \\ \hline & q_{1} & x & frick of the exceed and third (Inner) quadrupoles of the thereight of the first quadrupoles of the th$	$ \begin{array}{llllllllllllllllllllllllllllllllllll$

Each quadrupole has a half aperture of 52.7 mm and an effective length of 31.1 cm. The first quadrupole defocuses the beam in x- and focuses it in y- while the second focuses in x- and defocuses in y-. The combined effect of the doublet produces a beam spot of the dimensions quoted above.

At a point C (Fig. 1), where the beam is 4 mm in diameter, a system of collimators with diameters ranging from  $20-70\mu m$  is placed. These allow a very small fraction of the beam to continue upon its journey

to the target. Beam currents incident upon the target are tabulated as a function of collimator diameter in Table 2. This calculation has been based on the

TABLE 2						
(µm)	E	Ι <sub>τ</sub> (pA)				
20	4.0 x 10*	25				
30	1.8 x 10*	56				
40	1.0 x 10*	100				
50	6.4 x 10 <sup>3</sup>	160				
60	4.4 x 10 <sup>3</sup>	230				
70	3.3 x 10 <sup>3</sup>	300				

d ≡ diameter of collimator slit (μm)

d

F ≡ factor by which beam current is reduced

I  $_{\tau}~\equiv~$  current on target based on  $\underline{1\mu A}$  emanating from 30  $^{o}$  slit and incident on the collimator slit

assumption of lµA of proton beam current emerging from the cyclotron. The current density produced at the target is approximately  $(3.8\pm1\%) \text{ pA}(\mu\text{m})^{-2}$ . The rest of the design is based on a 70µm diameter collimator. A system of four quadrupoles is placed 3.5 m from

A system of four quadrupoles is placed 3.5 m from the collimator slit to focus the beam down to 10µm diameter. This system is commonly known as the "Russian quadruplet". The properties of the system have been studied by Dymnikov et al<sup>4</sup>). The main advantage of this arrangement is that the fields can be arranged in such a way that aberrations are cancelled or at least minimized. A system of this sort has been successfully employed in conjunction with a 3 MeV proton beam<sup>5</sup>).

Each of the four quadrupoles has an aperture diameter, pole tip to pole tip, of 5.4 cm and is 18.05 cm in length (21.15 cm effective length). Neighbouring quadrupoles are separated by 4.5 cm. The first and fourth (outer doublet) have equal but opposite fields, as have the second and third (inner doublet). The inner doublet is oriented at 90° relative to the outer doublet. Field strengths as a function of proton energy are tabulated in columns 5 and 6 of Table 1. Field stability is a very important factor, as is the relative orientation of the quadrupoles. These problems are briefly discussed below.

A system such as that described above will focus a 35 MeV proton beam emerging from a collimator slit of 70µm diameter to a spot of 10µm diameter at a distance of 3.1 cm from the exit of the fourth quadrupole. This magnification of 1/7 is expected to be smaller for particle energies less than 35 MeV, and for smaller collimator slit diameters. The reason for this is that the smaller the energy, the less are the field strengths; and the smaller the collimator slit, the smaller the maximum diameter of the beam.

The overall design is shown in Fig. 2 with a calculated beam profile. The target position is



marked. Target scanning can be achieved with the aid of a stepping motor for coarse target displacement (> beam diameter) while a fine scanning of the beam across small portions ( $\simeq$  beam diameter) of the target can be achieved by a system of electrostatic deflection plates placed inside the fourth quadrupole. Beam parameters at important stages of propagation are tabulated in Table 3. These, together with the data

#### TABLE 3

#### Beam Parameters\*



 X and Y represent the 1/2 size of the beam spot in X and Y directions

 $\boldsymbol{\theta}_{\chi}$  and  $\boldsymbol{\theta}_{\psi}$  represent the 1/2 angles of the beam in  $\chi$  and  $\gamma$ 

of Table 1 and Figs. 1 and 2, look promising. Technical difficulties are now briefly discussed. Chromatic aberrations require  $\Delta p | p_0 \approx 10^{-4}$ . In terms

of energy this is  $\approx$  7 keV. This can be achieved on a 30-45° line. A variation of beam momentum of this magnitude contributes < 20% to the size of the beam spot at the target position. Geometrical alignment problems behave linearly with field stability<sup>6</sup>). In this particular case their effects are less serious than those of chromatic aberrations. Third order aberrations can be corrected for by a suitably chosen octupole field at the quadrupole aperture<sup>7</sup>).

Beam detection can be performed by placing a screen 10 cm downstream from the target position. At this point the beam has a diameter of 7 mm. A preferable method of "viewing" however is to place a microchannel electron multiplier<sup>8</sup>) at the target position. These devices can handle beam sizes of 15µm. It is also possible to use a digital matrix. Detailed studies of the design and evaluation of a digital matrix have still to be performed.

# 3. Applications

It will be impractical to include all the applications of the microprobe here. It is sufficient to outline a few selected examples which are of particular interest to a coupled P.I.X.E.-microprobe setup. The field is discussed in detail in references 9 and 10.

An ideal application of P.I.X.E. is to the investigation of small environmental samples, preferably as thin foils, containing 10-15 elements in amounts of 10-.01 ng. Aerosol samples present such a case. For atmospheric aerosols, the natural constituents as well as anthropogenic contributions have already been studied in several cases. The aerosol contamination of work places, especially associated with welding operations, has been the subject of increasing interest as has the deposition of inhaled aerosols in the human respiratory tract. Size distribution information is of considerable medical importance since deposition and removal of aerosol particles in the respiratory system is strongly size dependent. The size of such particles

can be examined by means of the microprobe technique. The number of samples obtained from aerosol investigations is large, while the amount of material involved is quite small. Therefore, an analytical method with large throughput and high sensitivity must be used. The coupled microprobe-P.I.X.E. technique is ideal for this purpose.

The industrial environment often involves situations where the concentration of air pollutants is very high. An exploratory investigation into the nature of air pollution near welding arcs has been carried outll). It has been found that aerosol concentration near the welding arc was 3-4 orders of magnitude above that in the ambient air. Both the composition and particle size distribution of the aerosol depend upon the welding technique and the materials being used. The size distribution data enable a separation of welding generated aerosol components from general background in the room.

An investigation of solid state materials has also been made. In particular, studies of thin films of semiconducting chalcogenide glasses12) as well as the purity of thin boron films. Another example in this field is the use of P.I.X.E. to determine the zinc concentration in an epitaxial layer of InSb13).

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# \*\* DISCUSSION \*\*

M. CHAUDHRI: You mentioned that spot sizes down to 4 µm are easily obtainable. However, our experience with the Melbourne University Microprobe indicates that for spot sizes of much below 10 µm a great deal of care is needed, and that even then it is difficult to obtain such small spots. Secondly, small currents are a problem, as the irradiation time with the available beam current of some tens of pA is many hours for obtaining any reasonable statistics. So one should try to get as much beam current as possible (some tens of nA) in the micron size spot.

M.S. AL-GHAZI: For our energy range the yield of the K x-ray cross-section is quite high, since the proton velocity is about the same as that of the orbital electron velocity. In consequence, we need low currents. For the first half of the question, the spot size is determined by phase space area. One has to choose the size of collimator which is appropriate, keeping the beam divergence within acceptable limits. This is clearly a very tricky problem.

C. FOSTER: Bremstrahlung is the normal background limiting the sensitivity of PIXE at lower energies. How does this affect the technique at higher energies? What are the limits of sensitivity for your microprobe?

M.S. AL-GHAZI: The K x-ray yield enables fairly clean spectra to be obtained. Our detection limit is 2 ppm for Rb. At present we are in the process of acquiring a Compton suppression unit (recently funded by the University of Manitoba Research Board). This will enable a reduction of the background by a factor of 10, thus improving sensitivity.