Introduction to PIXE Analysis

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Catchphrases of our PIXE

1. We’ll give you the results of analyses in the evening, if you take them to our laboratory in the morning.

2. We perform quantitative analysis of any kind of samples.
Inner-shell Ionization and X-ray Detection

N-shell
M-shell
L-shell
K-shell
Nucleus

Incident Proton from accelerator

Atom in the Sample

Electron ejection

Inner-shell Ionization

PIXE Ion (Proton)

EPMA Electron

X-ray Fluorescence Analysis

X-ray

Si(Li) Detector

Production and Detection of K-x rays
Particle Induced X-ray Emission Analysis

Aerosol
An example of the method of quantification

1. Internal-standard method
   (for the samples easy to be homogenized)

\[
X_i = X_s \times \frac{Y_i}{Y_s} \times \frac{\sigma_s^x \times A_s \times \text{eff}_s}{\sigma_i^x \times A_i \times \text{eff}_i}
\]

Quantitative analysis by the internal standard method

- **Number of atom**
- **Number of atom of the standard element**
- **Ratio of peak yields**
- **Ratios of three physical quantities**

Internal-standard method

- Weight measurement of the sample and the internal standard
- Addition of the internal standard and homogenization
- Drying
- Measurement

Beam

Target

X-ray

4μm Mylar film

Si (Li)
Analytical Attributes of PIXE

1. Simultaneous analysis of all elements (Na ≤) in a short time (3-10 min).

2. High- and almost equal- sensitivity for all elements.

3. Quantitative analysis of extremely small amount of samples (≤ 1 mg).

4. Quantitative and non-destructive analysis of untreated samples. (Hair, Urine, Nail, Blood, Organ etc.)

5. Analysis of liquid samples like an oil, bio-samples staying in alive, large samples such as an ancient pottery, powdered samples such as soil, ash and aerosol etc.

6. Micron-order elemental mapping using a micro-beam (e.g. for a single cell) and beam channeling (e.g. for metals and alloys) etc.
Our method doesn’t require any target preparation procedure except for washing before irradiation.

It is possible to quantitatively analyze as little as one hair of 1 cm in length.
In-Air non-destructive analysis

- Beam in vacuum in air
- Living plants
- Living animals
- Arts
- Archaeological excavations
Micro PIXE camera (MOLT-4)

Elemental mapping in a single cell.

No Radiation

Fe(Iron)

Ca(Calcium)

Zn(Zinc)

6 hours

Occurrence of Apoptosis
Nishina Memorial Cyclotron Center (NMCC)

Nation-wide common utilization facility for
PET : Positron Nuclear Medicine
PIXE : Particle Induced X-ray Emission
since 1995

Takizawa Laboratory

Ultra-high field MRI

Takemi Memorial Hall

NMCC
NMCC and Mt. Iwate
Snow scene of NMCC
Small-size Cyclotron at NMCC
Ultra-compact cyclotron developed for PIXE analysis

By Ion Accelerator Co., Ltd.

Cyclotron

The whole system for PIXE analysis
Frequency of PIXE experiments by each research field

Integration from 1990 to the present time

Fields

Frequency of PIXE experiments
A THREE-DETECTOR MEASURING SYSTEM
USING A PURE-Ge DETECTOR
(for Fluorine analysis)

Fluorine is an essential element from the viewpoint of public health and hygiene medicine.
Simultaneous Measuring System of In-Vacuum and In-Air PIXE

4-Detector 2-Sample Simultaneous Measuring System
Chemical-Ashing Method Using an Electric Range

- Weight measurement of the sample
- Adding an internal standard and nitric acid
- Heating in an electric range
- Dropping and drying

In 1,000ppm HNO₃
External-standard method for uniform and dotted sample

Uniform and dotted aerosol samples

Beam distribution

Sample distribution on the target

External standard

Uniform-beam method

Dotted aerosol collected with an impactor etc.

Uniform-sample method

Uniform aerosol collected with a sampler etc.
Example of Target Preparation for Uniform Aerosol Samples (collected with a step sampler)
Example of Target Preparation for Doted Aerosol Samples (collected with an impactor)

Doted sample collected with an impactor
Measurement of aerosol samples

Spectrum analysis using SAPIX code (Sera:1989)
Changes of elemental concentration in aerosol samples successively collected in a suburban dwelling in Akita city.
Powdered Internal-Standard method
(for soil, ash, sediment, deposit, tailings, smashed rock etc.)

Weight measurements of a sample and palladium-carbon

Mixing (Powdered as they are)
Standard-free method for Biomedical and Environmental Samples

Standard-free Method of Quantitative Analysis for Bio-Samples.
K. Sera, S. Futatsugawa, K. Matsuda and Y. Miura
Int’l Journal of PIXE Vol. 6-3,4 1996 (467-481)

Quantitative Analysis of Bio-Medical Samples of Very Small Quantities by the Standard-Free Method.
K. Sera, S. Futatsugawa, S. Hatakeyama, Y. Saitoh and K. Matsuda
Int’l Journal of PIXE Vol. 7-3,4 1997 (157-169)

Quantitative Analysis of Powdered Bio-Medical Samples by the Standard-Free Method.
K. Sera and S. Futatsugawa
Int’l Journal of PIXE Vol. 8-1 1998 (19-32)

Quantitative Analysis of Untreated Bio-Samples.
K. Sera, S. Futatsugawa and K. Matsuda
Nuclear Instr, and Meth, in Phys, Res. B150 1999 (226-233)
Standard-free Method
(Quantitative Analysis without Standard)
Our method doesn’t require any target preparation procedure except for washing before irradiation.

It is possible to quantitatively analyze as little as one hair of 1 cm in length.

Quantitative analysis of untreated hairs

Elemental concentration in hairs reflects that in a human body.
Standard-free Method for Untreated Nails

Detector No.1
Ep = 2.9 MeV
with a 300 μm Mylar

Counts/Channel

Channel Number

Nail
Tape

Target holder
Beam spot
Application of a Standard-free Method to Quantitative Analysis of Urine Samples

K. SERA, Y. MIURA and S. FUTATSUGAWA

Int'l Journal of PIXE  Vol.11-3,4  149-158  (2001)

Human Urine Net Spectrum

Target is prepared only by dropping 5μl of urine onto a backing film.
The standard-free method for sweat samples

The total yield of continuous x-rays between 4.4 and 6 keV is used for determining the conversion coefficient for the standard-free method.
Standard-free method for saliva samples

Ep=2.9MeV
With a 300μm Mylar
X-ray spectra of tear samples taken from a 26 years-old woman

a) Si(Li) No.1
   with a 500 μm Mylar absorber

b) Si(Li) No.2
   without absorber
The effect of specially designed absorber (for geological and mineralogical samples)

Fig. 6. a) X-ray spectrum for a soil sample obtained with a conventional 300μm-Mylar absorber.

b) Same as Fig. 6-a) but with the absorber whose structure is illustrated in the figure.

c) Same as Fig. 6-a) but with the specialized absorber shown in Fig. 1-c). The typical least-squares fittings are also shown.
Method of determining transmission curves

Determination of Physical Quantities for PIXE by Means of PIXE 1. Absorption Curve
Sera, K., Futatsugawa, S., Hatakeyama, S. and Saitou, Y.
Int'l Journal of PIXE Vol. 4-2, 3, 165-179 (1994)
Examination of lymphatic vessels as a drug delivery route
A. Fujimura a, K. Sera b, S. Futatsugawa c and Y. Nozaka a

Tumor: 1 week after transplantation Before injection of Cisplatin

Tumor reduced: after injection of Cisplatin

VX2+E7820

3D reconstruction images in tongue cancer

CDDP(Cisplatin)

Peripheral injection of CDDP around the tumor

Cl- NH3

Pt

Cl- NH3
Result & Discussion

Explantation of VX2 cancer cells
Growing and metastasis
Injection of Cisplatin
Diffusion and movement of Cisplatin

Peri-tumor injection 0.1mg/ml, 100 μl = 10 μg/2.5kg

Intra-venous injection 4mg/kg

Whole body 4μg/g

0.004μg/g × 1/100

Tongue 4μg/g

100μg/g × 25

Deep cervical lymph node 4μg/g

2μg/g × 1/2

Graph:
- X-axis: R-SML, L-SML, R-DC, L-DC
- Y-axis: 0, 1, 2, 3, 4, 5, 6
- Colors:
  - 1mg/ml
  - 0.1mg/ml
  - 0.01mg/ml

Legend:
- 1mg/ml
- 0.1mg/ml
- 0.01mg/ml
Effect of Mineral-Rich Water Spraying

Soybeans

spraying → no spraying

← spraying  no spraying →
We’ll give you the results of analyses of your sample in the evening, if you take them to our laboratory in the morning.
Thank you for your attention

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