Highlights of JST-SENTAN 2014

Development of Systems and Technologies for Advanced Measurement and Analysis

JST

Contents

Environmental Science

Rediation Measurement

Overview

1. About JST

The Japan Science and Technology Agency (JST) is one of the core institutions responsible for the implementation of science and technology policy in Japan, including the government's Science and Technology Basic Plan. From knowledge creation—the wellspring of innovation—to ensuring that the fruits of research are shared with society and Japan's citizens, JST undertakes its mission in a comprehensive manner.

2. About "JST-SENTAN (Development of Advanced Measurement and Analysis Systems)" Program

● Objectives

Creative and original R&D is a key to generating innovation. To boost these activities, JST promotes the development of systems and technologies for advanced measurement and analysis.

Our program offers suitable support types to match different needs of each development phase.

Since FY2012, in addition to our original support types, priority development areas are set after scientific, technological and social issues. To produce breakthrough on recognized problems in these areas, development teams are required to cooperate closely with users.

As the priority development areas, "Radiation Measurement" and "Green Innovation" have been set for FY2012, then "Life innovation area" for FY2013.

●Program Scheme

MEXT (Ministry of Education, Culture, Sports, Science & Technology in Japan)

↓ Policy, Budget

Japan Science and Technology Agency

JST-SENTAN Program

"Development of System and Technology for Advanced Measurement and Analysis"

- "Program Director" manages whole program.
- "Review Committee" select proposals.
- "Program Officers" manages each project.
- ✓Both industry and academia participants are required to create collaborative team.

Technology development type

Creating new technologies which dramatically improve measurement and analysis concept.

System development type

Developing prototypes of novel measurement and analysis apparatus and systems.

Practical Realization type

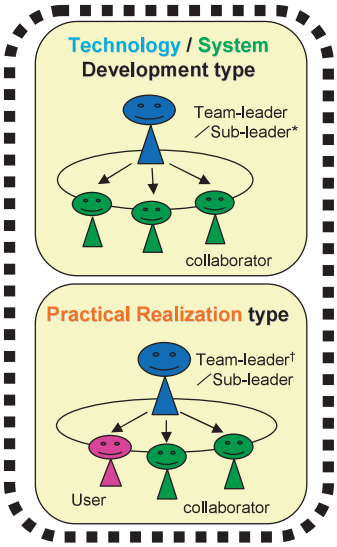
Realizing commercial products by reflecting user's expectations, under strong industry leadership.

✓Industry members are required to share team budget for development.

Call for Proposals →

Contract ↔

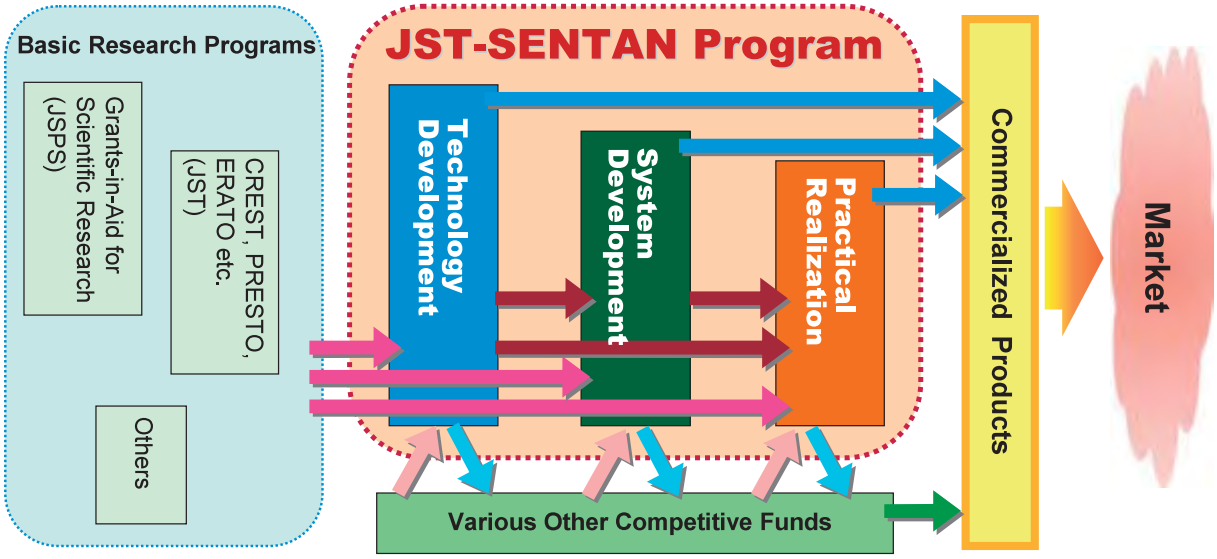
Promotion of Development →



＊ Sub-leader may not be assigned in Technology type.

† In Practical Realization type, team-leader must be affiliated to Industry.

●Scope of JST-SENTAN Program



Result No.1

Fully automated glycan analyzer for the development of disease early diagnostic system

Team Leader

Kazuyuki Hamada

System Instruments Co.,Ltd.

Sub Leader

Shin-ichiro Nishimura

Hokkaido University

Participating organization

System Instruments Co.,Ltd., Hokkaido University, Bruker Daltonics K. K., Science & Technology Systems, Inc.

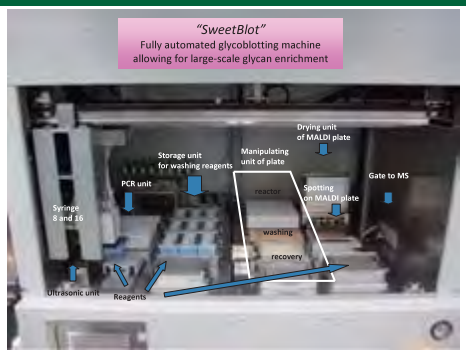
Keywords

Glycoblotting method, Human serum glycoproteins, Disease-relevant glycans, High throughput glycomics, Biomarkers, Early diagnosis

Abstract

It is thought that glycans can be potential candidates for novel diagnostic and therapeutic biomarkers because drastic structural changes in human serum glycoproteins are often observed during cancer cell differentiation and progression. Although there have been substantial advances in our understanding of the effects of glycosylation on some biological systems, we still do not fully understand the significance and mechanism of glycoform alteration detected widely in many human diseases due to their highly complicated structures and extremely tedious and time-consuming processes for glycan enrichment analysis. We have developed for the first time a fully automated glycan analyzer based on “glycoblotting method” known as only one method allowing rapid and large-scale clinical glycomics of human whole serum glycoproteins. We discovered novel glycan biomarkers from serum samples of patients suffering from various cancers such as hepatocellular carcinoma (HCC), pancreatic cancer, renal cancer, and so on.

Breakthrough: Discovery of the glycoblotting method



Apparatus name

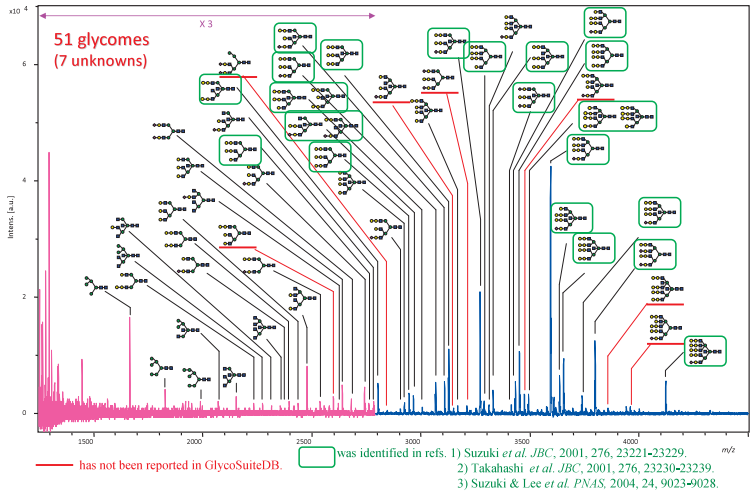
Fully automated glycan analyzer

Technical Overview

We demonstrated that the "SweetBlot" machine, an automated glycoblotting platform, can be combined with MALDI-TOFMS by a designated robot for the transportation of MALDI plate carrying labeled serum glycans. The "Sweetblot" greatly accelerated whole serum glycan-enrichment and subsequent labeling in an all-in-one protocol for 96-well filter-plate format. It requires very little material (human serum, 10˜100 micro L) and takes only ˜14 hours to complete whole glycan profiling of 96 samples when combined with glycomics using general mass spectrometry.

For example, use of automated glycan analyzer established herein allowed for rapid and quantitative *N*-glycan profiling of 103 human serum samples (83 HCC patients and 20 normal donors) concurrently. To identify the essential features to optimally classify the sera between the two relevant classes, disease and normal, we applied a sequential forward-selection algorithm that sequentially selected a better combination of *N*-glycan peaks based on leave-one-out (LOO) error rates of a *k*-nearest neighbor classifier ($k = 3$). When we chose the ratio of every two peaks among the acquired that show significant difference (two-sided *t*-test, $P < 0.001$) between disease and control, the algorithm finally selected three combinations of *N*-glycan ratio features that distinguished HCC samples from normal controls with 99% accuracy. In a similar manner, we have discovered a variety of potential prognostic biomarkers for renal cancer and HCC as well as early diagnosis of pancreatic cancer.

Most common theme in the glycan analysis has long been considered to be the need to develop simplified and cost effective techniques that could be used by non-specialists. In addition, it seems likely that the expense and complexity of many of the existing tools limits the ability of the broader biology community to address questions in carbohydrate research. Thus, the automated glycan analyzer based on glycoblotting method was demonstrated to provide a convenient, noninvasive diagnostic tool for many diseases that have previously been difficult in early diagnosis or differentiation.



Whole *N*-glycomes of pigeon egg white revealed by "glycoblotting"
M. Amano & K. Hirose *et al., in prep.*

Technical Performance

1) Whole *N*-glycan structural characterization of 96 human serum samples/24 hours
2) Quantitation of 50˜60 glycan structures
3) High repeatability and reproducibility
4) Flexibility in the labeling reagents and structural profiling methods such as HPLC-, LC-ESIMS, and MALDI-TOFMS platform

Publications

1) Nishimura S-I, "Toward automated glycan analysis" *Adv. Carbohydr. Chem. Biochem.* 65, 219-271 (2011).
2) Miura Y. *et al.* "BlotGlycoABC: An integrated glycoblotting technique for rapid and large-scale clinical glycomics" *Mol. Cell. Proteomics* 7, 370-377 (2008).
3) Nishimura S-I. *et al.* "Glycomics for drug discovery: Molecular perturbation in androgen-independent prostate cancer cells induced by unnatural hexosamine mimics" *Angew. Chem. Int. Ed.* 51, 3386-3390 (2012).
4) Kamiyama T. rt al. "Identification of novel serum biomarkers of hepatocellular carcinoma using glycomic analysis", *Hepatology* 57, 2314-2325 (2013).
5) Nouso K. et al. "Clinical utility of high-throughput glycome analysis in patients with pancreatic cancer", *J. Gastroenterology* 48, 1171-1179 (2013).

Contact
Person in charge : Shin-Ichiro Nishimura
Affiliation : Hokkaido University
E-mail : shin@sci.hokudai.ac.jp

Result No.2

Development of two-dimensional multi-confocal Raman microscope

Team Leader

Kenichi Kawamura

Tokyo Instruments, Ltd.

Sub Leader

Koichi Iwata

Gakushuin University

Participating organization

Tokyo Instruments, Ltd., Gakushuin University

Keywords

Raman spectroscopy, Confocal microscopy, 3D imaging, Bio-imaging, Fast imaging

Abstract

A two-dimensional multi-point confocal Raman microscope "Phalanx-R" that enables one to obtain Raman microscopic image of living cells and tissues in real time. "Phalanx-R"is based on the prototype developed and demonstrated by Prof. Hamaguchi within JST-SENTAN program (system development type). In the Raman microscope, an excitation laser beam is split into 21 by 21, totally 441 beamlets and shined on a sample. Consequently, two-dimensional Raman image is obtained without raster scan of the excitation beam or the sample. This makes the measurement 441 times faster than conventional confocal Raman microscopes with a single excitation beam.

Fig. 1 Appearance of inverted-type two-dimensional multi-point confocal Raman microscope. The left unit consists of an optical microscope, a multi-point confocal optic unit, and an excitation laser. The right one consists of a transmission-grating spectrograph and a CCD detector. The two units are linked with a fiber bundle.

Apparatus name

Two-dimensional multi-point confocal Raman microscope (Product name: Phalanx-R)

Technical Overview

The two-dimensional multi-point Raman microscope "Phalanx-R" can obtain a Raman image in a moment without scanning an excitation spot or a sample. The laser beam is split into 21 by 21, totally 441 beamlets of square matrix array and is shined onto a sample. Interval of the spots is 560 nm in the focal plane when a 100x objective is used. Consequently, the field of view covers an area of 11 by 11 square microns in the case. The Raman scattering from each spot is collected with the objective lens and transferred through a confocal optical system and a fiber bundle. The fiber bundle is used for rearranging two-dimensionally distributed Raman scattering beamlets into a one-dimensional line to be introduced to the spectrometer. At its input end, the element fibers are arranged in square-matrix array with unprecedentedly high precision, so that the Raman scattering from each spot is coupled to the corresponding fiber element. At the other end, element fibers are rearranged into dual one-dimensional arrays of 221 and 220. The dual arrays lie in parallel with each other, which results in dual series of spectra lying side by side in a CCD image. Thus, totally 441 spectra are recorded simultaneously at once. Each pair of excitation beamlet and its corresponding fiber element conforms to confocal optical system, which enhances contrast and spatial resolutions. Therefore, one can observe the interior of a transparent sample three-dimensionally as well as minimize the hindrance by background auto-fluorescence.

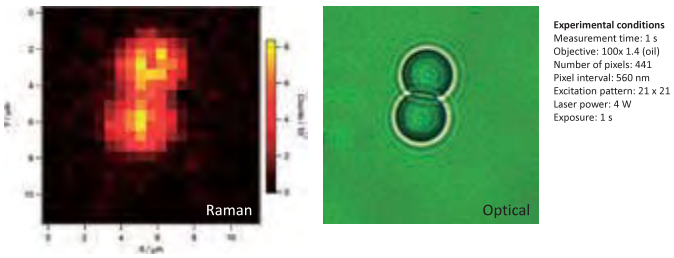


Fig. 2 Raman image of polystyrene beads. The image is of ring breathing mode at 1000 cm^{-1}. The excitation pattern here is a square matrix of 21 by 21, totally 441 spots with the interval of 0.56 mm. The image was measured in one second without sample scan.

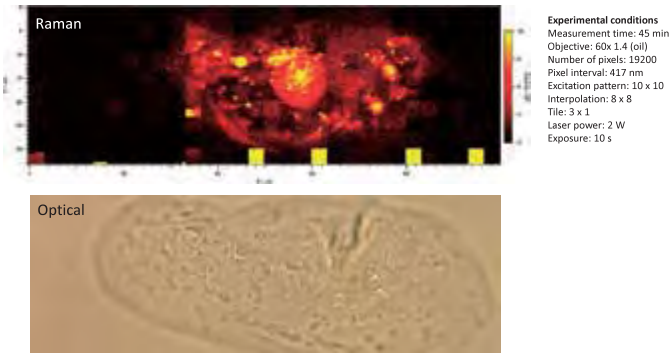


Fig. 3 Raman image of a human epithelial cheek cell. The Raman image is of CH stretching mode at 2950 cm^{-1}. The excitation pattern here is a square matrix of 10 by 10, totally 100 spots with the interval of 3.3 mm. The image was measured by scanning the sample so as to interpolate the interval of the excitation spots (interpolation) and to expand the field of view (tile).

Technical Performance

Excitation wavelength	532 nm. Customizable.
Number, formation of spots	21x 21 square matrix. Customizable.
Interval of spots	560 nm (@ 532 nm). Customizable
Dimensions (W x D x H)	1,500 x 700 x 800 mm.
Weight	Approx. 100 kg.
Spatial resolution	X-Y: 350 nm, Z: 900 nm (@532 nm, 100x NA1.4). Near diffraction limit.
Spectral range	400 – 2400 cm^{-1} (1300-lines/mm grating, 441 spots). 150 – 3100 cm^{-1} (1300-lines/mm grating, 100 spots). Customizable.
Spectral resolution	>7 cm^{-1} (@532 nm, 1300-lines/mm grating). Customizable.

Contact
Person in charge : Kenichi Kawamura
Affiliation : Tokyo Instruments, Inc.
E-mail : k_kawamura@tokyoinst.co.jp

Result No.3

Development of a diagnostic device based on differential phase contrast by X-ray Talbot-Lau interferometry

Team Leader

Sumiya Nagatsuka

Konica Minolta, Inc.

Sub Leader

Junji Tanaka

Saitama Medical University

Participating organization

Konica Minolta, Inc., Saitama Medical University, Tohoku University,

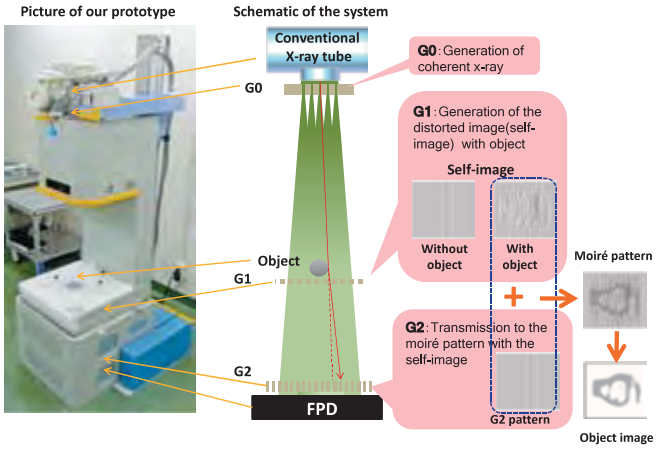
Keywords

X-ray interferometry, Phase contrast,
New imaging technology

Abstract

We've developed a diagnostic system based on differential phase contrast by an X-ray Talbot-Lau interferometry, which has high sensitivity to depict soft tissue such as tendon and cartilage in human body. With Talbot-Lau interferometry, three different types of images can be simultaneously obtained by calculations: an attenuation image, a small-angle-scattering (dark-field) image, and a differential phase image. We examined its feasibility for clinical diagnoses of joint diseases with the differential phase image. The joints of healthy volunteers were imaged, and the results indicated that the developed device had sufficient sensitivity to image cartilages.

Therefore, this diagnostic device based on X-ray Talbot-Lau interferometry is very promising and will offer better diagnosis.



Abstract of the device

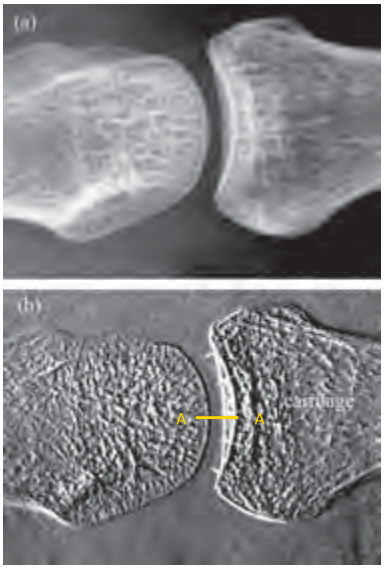
Apparatus name

X-ray Talbot-Lau interferometry for clinical use

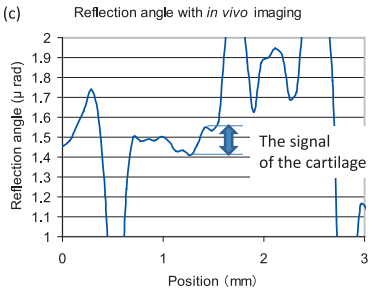
Technical Overview

The system consists of three X-ray gratings, a conventional X-ray tube, an object holder, an X-ray image sensor and a computer for image processing. The gratings were named G0, G1 and G2 respectively. An X-ray generator of a tungsten anode was operated with a tube voltage of 40 kVp, and a tube current of 100 mA. The mean X-ray energy was 28 keV. The pitches of G0, G1 and G2 were 22.8 μm, 4.3 μm, and 5.3 μm, respectively. The opening width of G0 was 7 μm, and the duty cycle of G1 and G2 was 0.5. G1 and G2 were located 1.1 m and 1.36 m from G0, respectively. G1 was a π/2 phase grating for 28 keV X-rays, and G2 was an amplitude grating, whose gold pattern height was 43 μm. The object to be imaged was placed in front of G1. The area size of G1 and G2 was 60 mm x 60 mm, and the effective field of view was 49 mm x 49 mm taking account of the magnification of the image. All gratings were fabricated with X-ray lithography and gold electroplating. A flat panel detector was located behind G2 and its pixel size was 85 μm.

The in vivo image was obtained with this system with a three-step fringe-scan.



Example of the image data



The example of the image data
(a) Attenuation image of the metacarpophalangeal joint of the third finger of a healthy volunteer.
(b) Differential phase image of (a). The cartilage was depicted.
(c) The line profile of A-A in (b). The signal of the cartilage was shown in (c) and was approx. 0.14 μ rad.

Technical Performance

The cartilage of metacarpophalangeal joint of the third finger of a healthy volunteer was clearly depicted. The refraction angle of the surface of the cartilage was 0.14 μrad.

Publications

1) Momose A *et al*, Phase Tomography by X-ray Talbot Interferometry for Biological Imaging, *Jpn J Appl Phys*, 45, 5254-5262, 2006
2) Yashiro W *et al*, Efficiency of Capturing a Phase Image Using Cone-Beam X-ray Talbot Interferometry, *J Opt Soc Am A*, 25, 2025-2039, 2008
3) Kido K *et al*, Bone cartilage imaging with X-ray interferometry using a practical X-ray tube, *SPIE Proc*. 7622 76224O, 2010

Contact
Person in charge : Sumiya Nagatsuka
Affiliation : Konica Minolta, Inc.
E-mail : sumiya.nagatsuka@konicaminolta.jp

Result No.4

Development and Optimization of Mass Microscope

Team Leader

Kiyoshi Ogawa

SHIMADZU Corporation

Sub Leader

Mitsutoshi Seto

Hamamatsu University School of Medicine

Participating organization

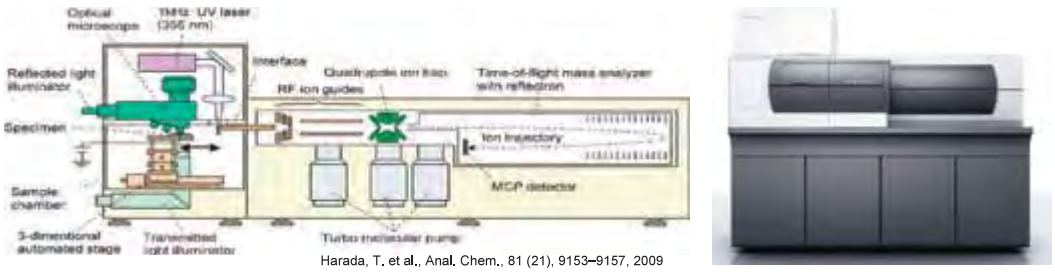
SHIMADZU Corporation, Hamamatsu University School of Medicine, Keio University

Keywords

Mass Imaging, Microscope, Lipid, Metabolite, Life Science

Abstract

We have developed a “Mass Microscope” which can investigate bio-molecules such as contributors of diseases using technology of mass spectrometry. The Mass Microscope can observe morphology of biological tissues with a high-resolution optical microscope, as well as analyze the molecule distribution in tissues with a high spatial resolution imaging mass spectrometry. This combination of precise morphology observation and molecule distribution analysis with high spatial resolution is quite a unique technology. The distributions of lipids, sugar chains, drugs and unknown materials can be investigated at single-cell size level. The Mass Microscope is expected to contribute to various fields such as rapid diagnosis, drug discovery and developments of novel therapy.



Harada, T. et al., Anal. Chem., 81 (21), 9153–9157, 2009

Fig. 1 Schematic diagram and photograph of the Mass Microscope (iMScope)

Apparatus name

Mass Microscope (iMScope)

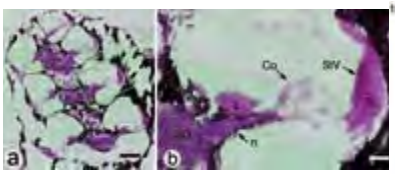
Technical Overview

We have developed a Mass Microscope which enables us to observe morphology of a tissue by high resolution optical microscope, and simultaneously enables us to analyze the distribution of bio-molecules in the tissue using technology of mass spectrometry. The optical microscope equips high magnitude objective lenses, so users can observe the morphology of samples in detail. Mass analysis is performed with AP-MALDI (Atmospheric Pressure Matrix-Assisted Laser Desorption / Ionization) method. Sample is coated or mixed with substance called "Matrix", and it is irradiated by focused laser light under atmospheric pressure for ionizing molecules. Matrix plays a role to assist the soft ionization process. The laser light of the Mass Microscope is focused less than 5 micrometers, so it enables us to obtain information of single-cell level molecular distributions.

The Mass Microscope also equips ion-trap which enables MS/MS or MS^n analysis. The MS/MS or MS^n analysis is a very powerful tool to identify unknown molecules detected in the tissues.

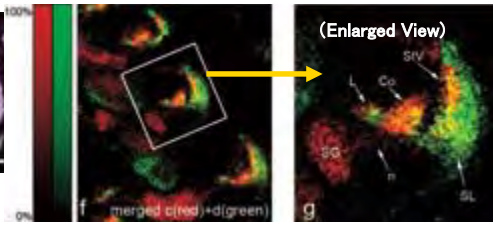
Fig. 2 shows the distribution of lipids in cochlea of guinea pig. It indicates that the Mass Microscope can clearly visualize the distributions of bio-molecules in a very small organ such as cochlea.

The Mass Microscope has started to be utilized for researches of medical and pharmaceutical field, for example it has contributed to discover specific molecules for serious diseases. And also it is expected to be a useful tool in the industrial fields such as organic material analysis.



Micrograph of HE stain sample

a. Bar=500 µm
b. enlarged view Bar=100 µm
(SG : spiral ganglion、n : osseous spiral lamina、
L : spiral limbus Co : Corti organ、
StV : stria vascularis 、SL : spiral ligament)



Images of Mass microscope 10 µm pitch

Distribution of biomolecules.
Green: PC(16 : 0/18 : 1) Red: PC(16 : 0/18 : 2)
PC: Phosphatidylcholine

Takizawa, Setou et al., Audiology & Neurotology Vol. 16, No. 5, , 315-322, 2011

Fig. 2 Molecule distribution in Cochlea of guinea pig

Technical Performance

Items	Specifications
Sample chamber	Atmospheric pressure
Resolution of optical microscope	1μm
Spatial resolution of MS imaging	Less than 5μm
Analysis speed	0.2sec/Pixel
Mass range	50～3000 Da
Mass resolving power	10,000 (@ *m/z* 1,000)
MS^n analysis	Available

Publications

1) Harada, T. *et al.*, Visualization of Volatile Substances in Different Organelles with an Atmospheric-Pressure Mass Microscope, *Anal. Chem.*, 81 (21), 9153–9157, 2009
2) Kubo, A *et al.*, Semi-quantitative Analyses of Metabolic Systems of Human Colon Cancer Metastatic Xenografts in Livers of Superimmunodeficient NOG Mice, *Anal. Bioanal. Chem.*, 400, (7), 1895-1904, 2011.
3) Takizawa, Y. *et al.*, Specific Localization of Five Phosphatidylcholine Species in the Cochlea by Mass Microscopy, *Audiology & Neurotology,* 16, No. 5, 315-322, 2011

Contact
Person in charge : Kiyoshi Ogawa
Affiliation : SHIMADZU Corporation
E-mail : ogawak@shimadzu.co.jp

Result No.5

Automated 2D Electrophoresis and Electro-Blotting Device

Team Leader

Yutaka Unuma

SHARP Corporation

Sub Leader

Norie Araki

Kumamoto University

Participating organization

SHARP Corporation, Kumamoto University

Keywords

2D Electrophoresis, Proteome, IEF, SDS-PAGE, Western Blotting

Abstract

Protein analysis that combines a two dimensional (2D) electrophoresis and a western blotting method has been used for drug discovery and clinical research. In these methods, proteins are separated by an isoelectric focusing and SDS-PAGE, and transferred to a membrane electrically, followed by the detection with immunological reaction. This method consists of a lot of complicated processes on many different instruments. Therefore, the results are poorly reproducible.

1) We have developed an automated 2D electrophoresis device. It can automatically execute all the 2D electrophoresis process including soaking up sample, IEF, chemical treatments, connecting 1st gel to 2nd gel, and SDS-PAGE in 100 minutes. We commercialized this product in September, 2011.

2) We have stepped into the next stage of automation including electro-blotting process. We have developed an automation device that was able to perform 2D electrophoresis, electro-elution of protein, combined with protein blotting to a PVDF membrane. We achieved high transfer efficiency >90% and high reproducibility CV<5%.

Fig. 1 Automated 2D electrophoresis device (Auto2D BM-100)

Fig. 2 Automated 2D electrophoresis and electro-blotting device

Apparatus name

Automated 2D-Electrophoresis Device (Auto2D BM-100)
Automated 2D-Electrophoresis and Electro-Blotting Device

Technical Overview

1) Automated 2D-Electrophoresis Device (Auto2D BM-100, Fig.1)

Conventional method of 2D-Electrophoresis requires carefully elaborated manual operation and has problems of analysis time, reproducibility and resolution. We have developed "Auto2D", 2D electrophoresis device by achievement of accurate robotic manipulation. This machine provides users easy setting of the sample and user-friendly touch panel operation. And also, it eliminates manual operation by skilled technicians, and shortened analysis time greatly. Automatic analysis with first dimension of isoelectric point: 0.02pH resolution and second dimension of molecular weight: 2kDa resolution. The phosphorylation shift is clearly separated and detected. Long waited device is now available. Auto2D is useful for protein analysis in pharmaceuticals and food industry as well as biochemical study.

2) Automated 2D-Electrophoresis and Electro-Blotting Device (Fig.2)

We have stepped into the next stage of automation including electro-blotting process. Conventional electro-blotting process consists of a lot of complicated processes based on many different instruments. Especially, researchers have to take out gel from the electrophoresis device, and set gel and membrane to electro-blotter manually, and these procedures always lack reproducibility of the protein transfer efficiency and resolution, and also are complicated and time consuming. Therefore, we developed an automation system that was able to perform 2D electrophoresis, electro-elution of protein, combined with protein blotting to a PVDF membrane. The separated proteins in the second-dimensional electrophoresis (i.e. SDS-PAGE) step are eluted continuously from the edge of gel, and then the eluted proteins can be transferred continuously on the moving membrane (Fig.3).

We developed a prototype device of fully automated 2D-electrophoresis and electro-blotting system. It took about 3 hours for total analysis. To confirm whether the prototype can detect post-translational modifications of protein, samples derived from human brain tumor was analyzed. As the results, the 2DE-WB pattern that showed the proteinpost-translational modification was obtained immediately with the high resolution (0.02pH)(Fig.4).

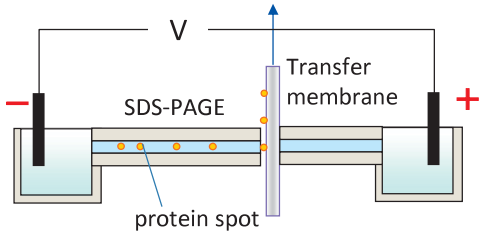


Fig. 3 Scheme for automated electro-blotting

Fig. 4 Western blotting result of brain tumor specific proteins (Vimentin)

Technical Performance

Fully automated 2D electrophoresis and electro-blotting device

	Auto2D BM-100	Conventional Devices
Operation	Fully automated	Manual
Analysis time	2~3 hours	3~4 days
Transfer efficiency	>90%	<70%
Reproducibility (CV)	<5%	>30%

Publications

1) Araki, N, Integrated proteomics for studying cellular mechanism of neural tumor formation. *Connective Tissue Research*, 2012 in press

Contact

Person in charge : Yutaka Unuma
Affiliation : Research Dept. II, Healthcare Systems Laboratories, Corporative Research and Development, Sharp Corporation
E-mail : unuma.yutaka@sharp.co.jp

Result No.6

Development of high-temperature thermophysical property measurement system using electromagnetic levitation technique in dc magnetic field

Team Leader

Hiroyuki Fukuyama

Tohoku University

Sub Leader

Yoichi Takasaki

ULVAC-RIKO, Inc.

Participating organization

Tohoku University, Gakushuin University, Chiba Institute of Technology, ULVAC-RIKO, Inc., System House Inc.

Keywords

Electromagnetic levitation, DC magnetic field, High-temperature melts, Thermophysical property

Abstract

Thermophysical properties of high-temperature melts are indispensable for numerical simulations of materials processes. However, crucial obstacles make measurements of thermophysical properties difficult at elevated temperatures because of high chemical reactivity and fluidity of melts. From the background mentioned above, we have developed a thermophysical property measurement system for heat capacity, thermal conductivity, emissivity, surface tension and density of high-temperature metallic melts using an electromagnetic levitator in a dc magnetic field. The oscillation and convection of the droplet are suppressed because of the Lorentz force, which enables true thermal conductivity measurement. Numerical simulation was also conducted to justify the thermal conductivity measurement.

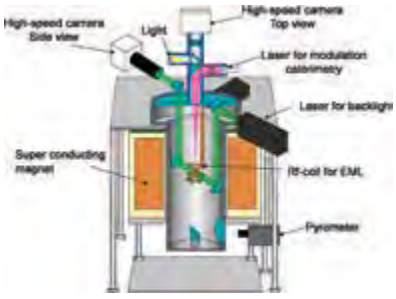


Fig. 1 PROSPECT

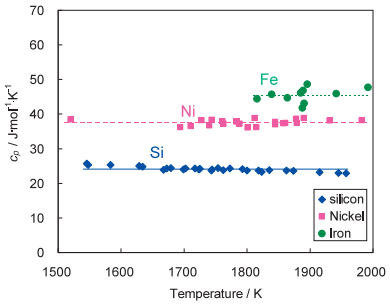


Fig. 2 Molar heat capacities of liquid Fe, Ni and Si

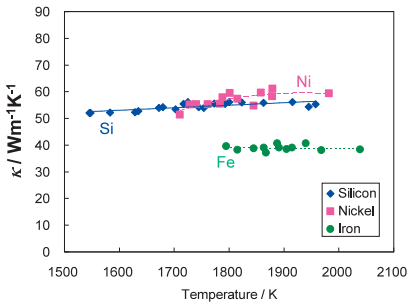


Fig. 3 Thermal conductivities of liquid Fe, Ni and Si

Apparatus name

PROSPECT (Properties and Simulations Probed with Electromagnetic Containerless Technique)

Technical Overview

This high-temperature thermophysical property measurement system (PROSPECT) consists mainly of an electromagnetic levitator incorporating a superconducting magnet, laser heating system, high-speed video camera, data-logging system and gas-controlling system including an oxygen sensor and oxygen pump. The electromagnetic levitator consists of a radio-frequency power source (max power of 10 kW and automatic tuning ranging from 150 to 400 kHz). A superconducting magnet with a bore diameter of 120 mm was used to generate a dc magnetic field. The magnet coils are made of Nb_3Sn and NbTi, and generate max magnetic field of 10 T. A fiber-coupling type CW laser diode was equipped on the system for laser modulation calorimetry. The laser wavelength is 807 ± 3 nm, and its max power is 140 W. The PROSPECT also has an integrated measurement and control system constructed using LabVIEW. The system includes user-friendly simulation applications software (electromagnetic field, heat and mass flow, and surface oscillation analysis) and thermophysical property analysis applications software (heat capacity, emissivity, thermal conductivity, density and surface tension measurements).

Using the PROSPECT,

(1) Sample melts can be kept noncontact, which provides contamination-free measurements.

(2) Surface oscillation and convection of sample droplet can be suppressed in a dc magnetic field, which enables true thermal conductivity measurements.

(3) Measurements can be conducted even at a deep supercooling state, which enables wide-temperature measurements and non-equilibrium processes.

(4) Measurements can be conducted under vacuum condition or reducing ambient or controlled oxygen partial pressure using a gas/liquid equilibrium method.

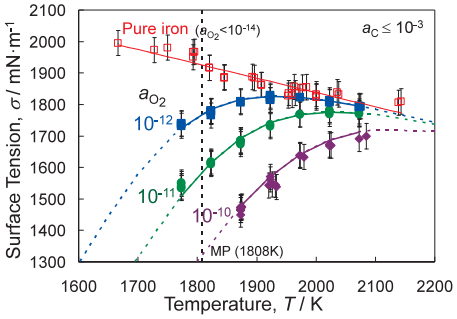


Fig. 4 Surface tension of liquid Fe vs Temp. under various oxygen activities

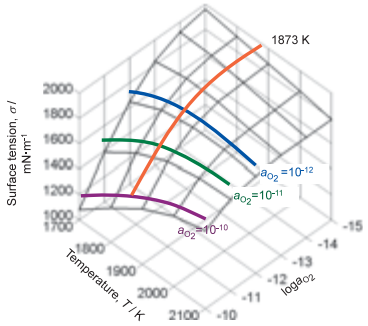


Fig. 5 Surface tension of liquid Fe in 3D diagram

Technical Performance

The following table shows the thermophysical properties with uncertainty measured using PROSPECT.

Thermophysical property	Element	Value at melting point	Uncertainty/%	Temperature range/ K
Density	Si	2585 kg/m^3	±1.0	1440-1700
Heat capacity	Si	24.1 J/ (mol·K)	±2.9	1550–1960
	Fe	45.4 J/ (mol·K)	±3.5	1848–1992
Thermal conductivity	Si	56.0 W/ (m·K)	±2.6	1550–1960
	Fe	39.1 W/ (m·K)	±3.2	1794–2050
Total hemispherical emissivity	Si	0.27	±3.8	1750–1910
Normal spectral emissivity at 807 nm	Si	0.227	±1.8	1660–1790
Surface tension	Fe	1925 mN/m	±3.4	1666-2120

The experimental uncertainty used here is the value of the standard deviation.

Publications

1) H. Kobatake, H. Fukuyama, T. Tsukada, S. Awaji
Noncontact modulated laser calorimetry in a dc magnetic field for stable and supercooled liquid silicon
Meas. Sci. Technol. 21 (2010) 025901

2) K. Morohoshi, M. Uchikoshi, M. Isshiki, H. Fukuyama
Surface Tension of Liquid Iron as Functions of Oxygen Activity and Temperature
ISIJ International, Vol. 51 (2011), No. 10, pp. 1580–1586

3) K. Sugie, H. Kobatake, M. Uchikoshi, M. Isshiki, K. Sugioka, T. Tsukada, H. Fukuyama
Noncontact Laser Modulation Calorimetry for High-Purity Liquid Iron
Jpn. J. Appl. Phys., 50 Nov. (2011) pp.11RD04-1-6

Contact
Person in charge : Hiroyuki Fukuyama
Affiliation : Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University
E-mail : fukuyama@tagen.tohoku.ac.jp

Result No.7

Development of ultra fast magic angle spinning module in the solid state NMR: a practical solution to characterize mass-limited samples with a rotor of 1mm diameter.

Team Leader

Katsuya Hioka

JEOL RESONANCE Inc.

Sub Leader

Tetsuro Asakura

Tokyo University of Agriculture and Technology

Participating organization

JEOL RESONANCE Inc., Tokyo University of Agriculture and Technology

Keywords

Solid-state NMR, Nano volume analysis, Ultra fast magic-angle-spinning

Abstract

A ultra tiny magic-angle-sample spinning (MAS) system in solid-state NMR has been developed. The diameter of the sample tube is only 1 mm, which enables a very fast MAS rate of 80 kHz, very strong rf field irradiation and nano volume sample analysis of less than 800 nL. The ^{1}H high resolution NMR spectrum can be obtained from only ultra fast MAS, giving the intra- and inter-molecular structural information from the ^{1}H NMR spectra easily. And highly sensitive ^{1}H-^{14}N two dimensional (2D) correlation NMR spectra of peptide and inorganic material analysis of volume limited samples have been reported.

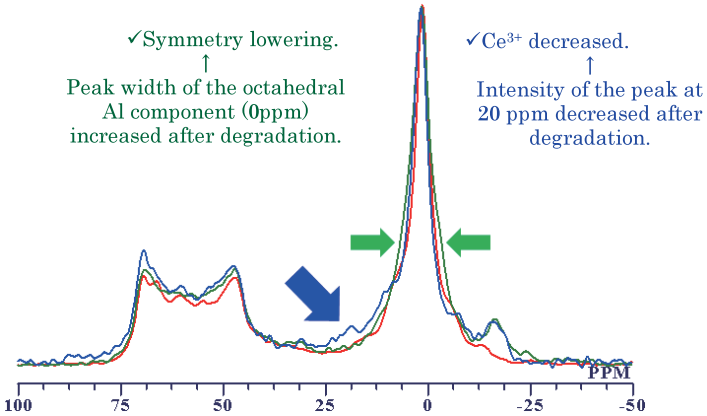


Fig. 1 ^{27}Al MAS NMR spectra of LED phosphors before (green), after (blue) degradation and non-Ce ion doped YAG (red).

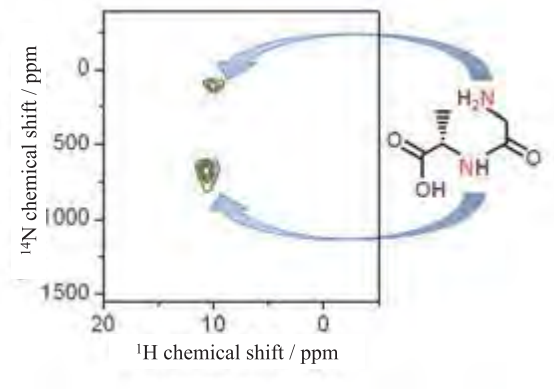


Fig. 2 ^{1}H-^{14}N 2D correlation HMQC spectrum of glycyl-L-alanine at 70 kHz MAS

Apparatus name

Ultra fast magic angle spinning probe

Technical Overview

A ultra tiny magic-angle-sample spinning (MAS) system in solid-state NMR has been developed. The diameter of the sample tube is only 1 mm, which enables a very fast MAS rate of 80 kHz, very strong rf field irradiation and nano volume sample analysis of less than 800 nL.

Figure 1 shows a typical application of its high sensitivity per unit volume. The 1 mm MAS system is applied to investigate the degradation of a very small amount of phosphors (Ce-doped YAG) in phosphor-based white LEDs. Although the sample weight of the phosphors taken from a few LEDs is only 1 mg, ^{27}Al MAS NMR spectra of Ce-doped and non-doped YAG has successfully revealed the change of valence state of Ce ions in the degraded LED phosphors. This example illustrates its promising ability to characterize other tiny materials in electronic devices.

The ^{1}H high resolution NMR spectrum can be obtained from only ultra fast MAS, giving structural information from the 1H NMR spectra easily. This was applied to determine the structures of peptide and proteins.

The ^{1}H-^{14}N 2D correlation measurement is a novel application of combining ultra fast MAS and strong rf field as shown in Figure 2. The spectrum is obtained within a few minutes despite of very small sample volume of 800 nL. This opens a new way to high throughput analysis of ^{14}N nuclei.

Thus, ultra tiny and ultra fast magic-angle- sample spinning (MAS) system developed here promise to expand new application of solid state NMR.

Technical Performance

Specifications

Sample tube outer diameter:	1 mm
Main observation nuclei:	^{1}H, ^{31}P, ^{7}Li, ^{11}B, ^{23}Na, ^{27}Al, ^{13}C, ^{79}Br, ^{207}Pb, ^{29}Si, ^{6}Li, ^{15}N, ^{14}N
Irradiation nucleus:	^{1}H
Maximum RF intensity	
^{1}H:	360 kHz
90° pulse width	
^{1}H:	≤ 0.7 μs
	≤ 1.0 μs
^{13}C:	≤ 0.7 μs
	≤ 1.0 μs
Sensitivity:	(≥19 Unlabeled Gly ^{13}C-α, 32 scans, reference value)

Publications

1) Koji Yazawa, Furitsu Suzuki, Yusuke Nishiyama, Takuya Ohhata, Akihiro Aoki, Katsuyuki Nishimura, Hironori Kaji, Tadashi Shimizu and Tetsuo Asakura, "Determination of accurate ^{1}H positions of an alanine tripeptide with anti-parallel and parallel β-sheet structures by high resolution ^{1}H solid state NMR and GIPAW chemical shift calculation", *Chem. Commun*., *48*, 11199-11201. 2012

2) Yusuke Nishiyama, Yuki Endo, Takahiro Nemoto, Hiroaki Utsumi, Kazuo Yamauchi, Katsuya Hioka, Tetsuo Asakura, "Very fast magic angle spinning ^{1}H-^{14}N 2D solid-state NMR: Sub-micro-liter sample data collection in a few minutes", *J. Magn. Reson*., *208*, 44-48. 2011

3) Riko MIYOSHI, Yuko MIWA, Masanobu YOSHIKAWA, Katsuya HIOKA, and Tetsuo ASAKURA, Phosphor Research Society of Japan The 388th Meeting Technical Digest (June 3, 2011) 13

Contact

Person in charge : Yusuke Nishiyama
Affiliation : JEOL RESONANCE Inc.
E-mail : yunishiy@jeol.co.jp

Result No.8

Technology development of innovative viscoelasticity measurement system

Team Leader

Shinsuke Ishihara

Kyoto Electronics Manufacturing Co., Ltd.

Sub Leader

Keiji Sakai

University of Tokyo

Participating organization

Kyoto Electronics Manufacturing Co., Ltd, University of Tokyo

Keywords

Viscoelasticity measurement, Quadrupole electromagnet, Electro-Magnetically Spinning method, Rheometer

Abstract

Viscoelasticity measurement technology has made less progress in recent years. Commercially available viscometers with conventional methods still require complicated and skillful operations, such as adjustment and washing. In addition, the sample should necessarily be exposed to the open air. To settle these problems, we have developed a novel measurement system based on the Electro-Magnetically Spinning (EMS) technique. This system has unique features: It is quite easy to use, requires very small amount of sample, and keeps hermetically sealed condition. In addition, sample tubes are disposable, and it is possible to measure over wide viscosity range. The EMS system extends the variety of the rheology measurements to, for example, the medical and biological fields.

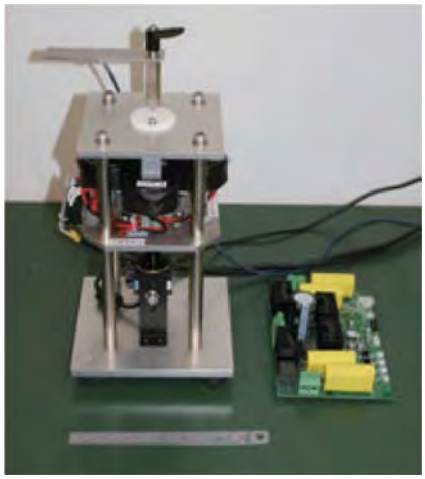


Fig. 1 Photograph of QEMS.

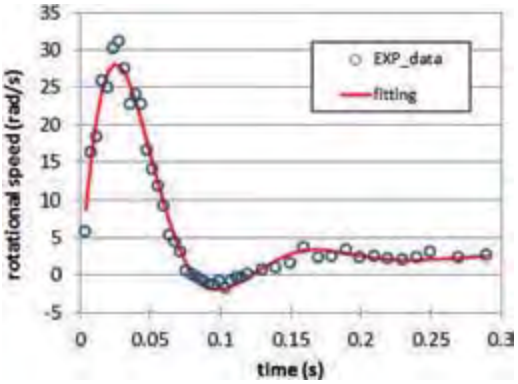


Fig. 2 Time-series variation of rotational speed of sphere in warm like micellar solution after induction of electromagnetic torque with a constant value.

Apparatus name

Quadrupole Electro-Magnetically Spinning Rheometer

Technical Overview

The Electro-Magnetically Spinning method for the viscosity measurement was devised by Prof. Sakai of the University of Tokyo. The principle of the measurement is as follows; The sample is placed into a small sample tube together with a metal sphere. A rotating magnetic field is then applied to the sphere and the Lorentz interaction between the magnetic field and the current induced in the sphere generates torque that rotates the sphere in a non-contact manner. By measuring the rotational speed of the sphere relative to that of the external magnetic field, we can determine the viscosity of the sample.

We have remarkably improved the technology by introducing the Quadrupole Electro-Magnetically Spinning (QEMS) technique to the system.

The QEMS generates computer-controlled arbitrary magnetic field with electromagnets. Figure 1 shows an external view of the QEMS prototype. The QEMS creates a measuring system that is totally free of moving parts for enhanced reliability.

Since the QEMS allows control over the magnitude and direction of the magnetic field, it enables rapid and precise measurements of viscoelasticity spectrum. Figure 2 shows the dynamic response of the probe sphere against the applied torque in a step function measured for the surfactant solution.

The behavior is well described by a mechanical model of the viscoelastic relaxation shown in Figure 3. The QEMS system would be a powerful tool for the variety of rheology measurements including medical and biological fields.

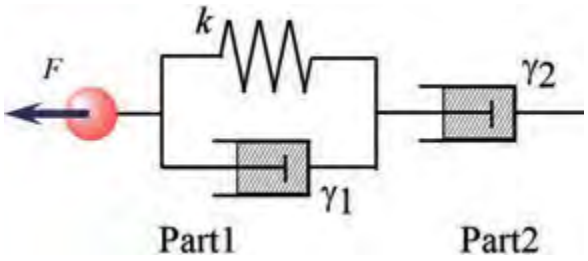


Fig. 3 Schematic image of a mass-spring-damper model.

Technical Performance

The advantages of QEMS over conventional measurement systems are shown in Talble.1.

Table 1. Comparison with QEMS and the conventional method

Method	QEMS	Conventional Methods
Sample volume	0.3 ml	More than 30 ml
Sample cell	Possible to be sealed	Difficult to prevent exposure the air
Operation	Easy and quick	Troublesome adjustment and preparation
Moving part	NO High reliability and high MTBF	YES low MTBF and need preventive maintenance
Cleaning	Not required (could be disposable)	Required

Publications

1) Keiji Sakai, *et al*,"Electromagnetically Spinning Sphere Viscometer", *Appl. Phys. Express,* 3, 016602 1-3, 2010
2) Maiko Hosoda, *et al*,"Low-Viscosity Measurement by Capillary Electromagnetically Spinning Technique", *Japanese Journal of Applied Physics,* 50, 07HB03 1-3. 2011
3) Taichi Hirano, *et al*,"Spontaneous Ordering of Spherical Particles by Electromagnetically Spinning Method", *Appl. Phys. Express,* 5, 027301 1-3. 2012

Contact
Person in charge : Masanori Yasuda
Affiliation : Kyoto Electronics Manufacturing Co., Ltd.
E-mail : yasuda.masanori@kyoto-kem.com

Result No.9

Development of an Real-Time Stereo SEM

Team Leader

Sukehiro Ito

Hitachi High-Technologies Corporation

Sub Leader

Tatsuo Ushiki

Niigata University

Participating organization

Hitachi High-Technologies Corporation, Niigata University, Shizuoka University, EIZO Corporation

Keywords: 3D, SEM, Real-Time, Naked Eye

Abstract

Scanning Electron Microscope (SEM, hereinafter) is useful for observing the specimen surface three-dimensionally. However, stereoscopic (3D) information has not been utilized effectively because simple SEM images contain monocular information.

3D observation with a SEM usually requires obtaining a stereo-pair image by tilting mechanically the specimen stage, combining the two views, and finally viewing them with a 3D glass (e.g., red-cyan glass). These 3D images cannot be observed in real time because of the mechanical tilt of samples.

To overcome this problem, we developed a Real-Time Stereo SEM which allows observing 3D images in real time.

In this project, we developed novel techniques including tilted-beam control technology, 3D display technology and off-axis aberration reduction technology for 3D SEM imaging.

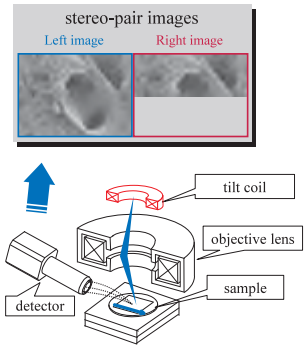


Fig. 1 Principle of beam tilt and control technology

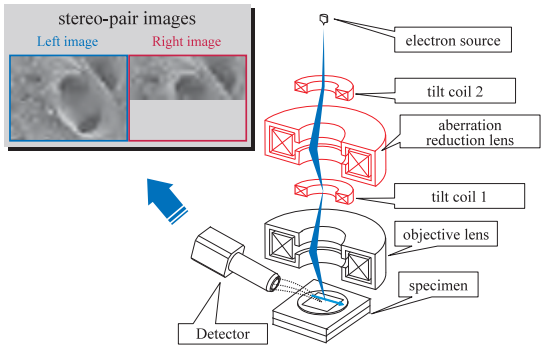


Fig. 2 Reduction technology of the off-axis aberration

Apparatus name

Real-time Stereo SEM (Hitachi SU3500)

Technical Overview

1. Tilted beam control technology (Fig. 1)

To observe 3D images in real time, we have succeeded in getting a stereo-pair of SEM images by tilting electron beam.

In this technique, the beam can be controlled from right to left by lines or frames with the specific tilt coil.

2. 3D display technology

A newly developed data-conversion software enabled the stereo-pair images to be outputted for commercial 3D monitor. On the other hand, conventional 3D viewing techniques using glasses (e.g., deflection or anaglyph methods) are unsuitable for the long operation because these methods cause brightness decrease.

Though some other methods such as the parallax barrier method and the lenticular method do not need glasses, these methods cause resolution decrease, moiré and pseudoscopic images.

Thus, we developed the novel stereoscopic image display device which can execute real-time 3D observation with high quality by the naked eyes. For this purpose, we adopted directional backlight with an elliptical mirror, achieving space saving with the appearance of 262mm long.

This device has been released as FDF2301-3D since fiscal 2011.

3. Reduction technology of the off-axis aberration (Fig. 2)

To acquire parallax images by electron beam tilt, the Real-Time Stereo SEM should prevent the beam from going through the axis of objective lens so that off-axis aberration and low-resolution occur. Therefore, off-axis aberration reduction lens is added to the side of the electron source seen from the objective lens.

Aberration reduction at the objective lens, by making use of aberration at the off-axis aberration reduction lens, executes 3D observation at high magnification.

Technical Performance

Fig. 3 shows the relation between beam tilt angle and resolution. The full line indicates the effect after aberration reduction, and the dotted line shows the value of aberration and resolution before aberration reduction. Comatic aberration and chromatic field aberration shown in Fig. 3 cause low resolution at beam tilting.

The aberration reduction optical system is designed as resolution is 15 nm when the beam tilt angle is 3.0 degrees, and observation magnification is 20,000 times.

Fig. 4 is the image where the beam tilt angle is about 3.0 degrees and observation magnification is about 20,000 times. The aberration reduction is successfully confirmed as shown in Fig. 4 (after).

This development contributes to the practical use of real-time stereo observation function as an option of Model SU3500 made by Hitachi High-Technologies Corporation, which was already released in 2012. The appearance to illustrate a combination of Model SU3500 SEM and FDF2301-3D are shown in Fig. 5.

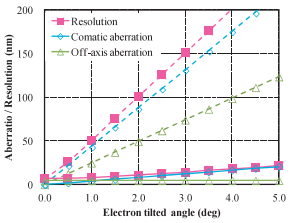


Fig. 3 Relation between beam tilt angle and resolution

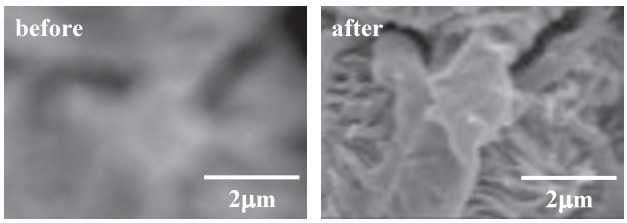


Fig. 4 Effect of the aberration reduction

Fig. 5 Real-time Stereo SEM Setup

Publications

1) Akinori Hayashi, Akira Sakai, Tomohiro Kometani, Hiroshi Ito : Cross-talk Caused by Light Reflected on a Back-face of a LCD glass in Auto-stereoscopic Display with Field-sequential Method and Directional Backlight System: SID Display Week 2011/5/15-20 Digest P-2, pp.1098-1011

2) F. Iwata, Y. Mizuguchi, H. Ko, T. Ushiki : Nanomanipulation of biological samples using a compact atomic force microscope under scanning electron microscope observation: Journal of Electron Microscopy, 60(6) (2011) 359–36

Contact

Person in charge : Hideki Tsuboi
Affiliation : Marketing Dept.2, Science Systems Sales & Marketing Div., Science & Medical Systems Business Group, Hitachi High-Technologies Corporation
E-mail : tsuboi-hideki@nst.hitachi-hitec.com

Result No.10

Atomic Resolution Scanning Probe Microscope Working in Air/Liquid

Team Leader

Ryohei KOKAWA

SHIMADZU Corporation

Sub Leader

Hirofumi YAMADA

Kyoto University

Participating organization

SHIMADZU Corporation, Kyoto University, Osaka University, Kobe University, Japan Advanced Institute of Science and Technology (JAIST), Kanazawa University

Keywords: SPM, AFM, FM detection, Atomic or molecular scale

Abstract

We developed the high resolution microscope that can be evaluated the structural and functional properties of metal, semiconductor, insulator and organic material at the atomic and molecular scale operating in air or liquid. Scanning probe microscopy (SPM) is a high resolution imaging technique applied for a variety of samples. In ultrahigh vacuum, by detecting the change of the resonant frequency of the cantilever (Frequency modulation method: FM method), it is possible to observe the structure of the atomic or molecular scale. However, there was a lack of stability and resolution in air or liquid. The purpose of this project is to establish a high-resolution observation technology by FM-AFM in air-liquid.

Fig. 1 SPM-8000FM

Apparatus name

High Resolution Scanning Probe Microscope, SPM-8000FM

Technical Overview

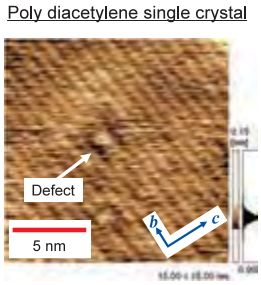
1. Capable of Ultra-High Resolution Observations in Air and in Liquids

By heightening the efficiency of the optical condenser system that detects the cantilever vibrations, and developing laser light non-interference techniques, the noise in the optical beam deflection system that detects cantilever displacement has been significantly reduced, to about 1/20th of conventional levels. As a result, this instrument provides ultra-high resolution observations in air and in liquids, which has been difficult for conventional SPMs. Examples include the molecular structure of thin films of lead phthalocyanine crystals in air, and the atomic structure of sodium chloride (NaCl) in water. Since this instrument can evaluate the reactions and functionality of organic molecules, which demonstrate specific reactions in solution, it will also be useful in the development of organic devices. The SPM-8000FM is an HR-SPM that for the first time transcends the ultra-high vacuum limitation.

2. Provides Not Only Surface Observations But Also Measurements of the Localized 3D Structure of Solid-Liquid Interfaces

Solid-liquid interfaces are known to be structured into complicated layers by interactions between the solutes and water (the solvent). This is referred to as hydration/solvation. Hydration/solvation is known to have a significant impact on chemical reactions at the solid-liquid interface, as well as charge transfer, lubrication, and heat conduction. However, since the layers are extremely thin, they are not easily measured. In particular, observations of hydration/solvation, with its 3 dimensional structure, non-uniform horizontally with respect to the surface, have been impossible to date.

With the SPM-8000FM, it is now possible to measure localized hydration/solvation structures, thank to ultra-sensitive force detection. The liquid structure at solid-liquid interfaces can be observed by operating the probe at the interface, and taking accurate measurements of the force as a function of the probe position. Furthermore, by adopting a new scanning method, not only 2D but also 3D structures can be analyzed for the first time. With behavioral observations in liquids of biological interfaces, surfactants, polymers, and electrodes, application of this instrument will extend not only to surface observations, but also to structural measurements of solid-liquid interfaces.



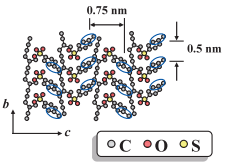
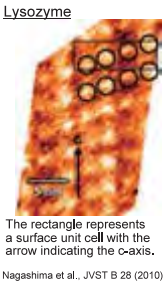


Fig. 2 High Resolution Topography in Air



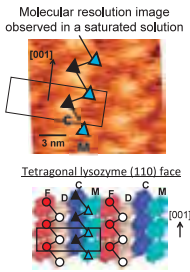


Fig. 3 Molecular Resolution Observation in Liquid

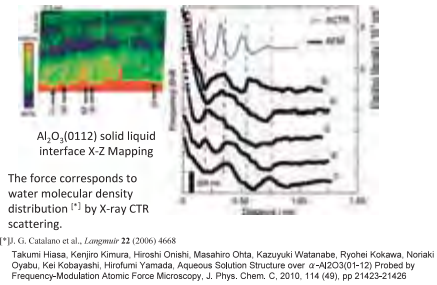


Fig. 4 Water Structure at Interfaces

Technical Performance

The noise level of the optical beam deflection sensor: 20fm/√Hz
The sensitivity of FM detection method: 0.01Hz/√Hz
Thermal drift: 1nm/min

Publications

1) Ryohei Kokawa, Masahiro Ohta, Akira Sasahara, Hiroshi Onishi, Kelvin Probe Force Microscopy Study of a Pt/TiO_2 Catalyst Model Placed in an Atmospheric Pressure of N_2 Environment, Chemistry-An Asian Journal, 7, 1251-1255 (2012).
2) Kei Kobayashi, Noriaki Oyabu, Kenjiro Kimura, Shinichiro Ido, Kazuhiro Suzuki, Takashi Imai, Katsunori Tagami, Masaru Tsukada and Hirofumi Yamada, Visualization of hydration layers on muscovite mica in aqueous solution by frequency-modulation atomic force microscopy, Journal of Chemical Physics, 138, 184704 (2013).
3) Takumi Hiasa, Kenjiro Kimura, Hiroshi Onishi, Masahiro Ohta, Kazuyuki Watanabe, Ryohei Kokawa, Noriaki Oyabu, Kei Kobayashi, Hirofumi Yamada, Aqueous Solution Structure over α-Al_2O_3 (0112) Probed by Frequency-Modulation Atomic Force Microscopy, J. Phys. Chem. C, 2010, 114 (49), pp 21423-21426.

Contact
Person in charge : Ryohei KOKAWA
Affiliation : SHIMADZU Corporation
E-mail : kokawa@shimadzu.co.jp

Result No.11

Development of AFM tip characterizers and evaluating the tip shape.

Team Leader

Hisataka Takenaka

NTT advanced technology corp.

Sub Leader

Hiroshi Ito

National Institute of Advanced Industrial Science and Technology (AIST)

Participating organization

NTT Advanced Technology Corporation, National Institute of Advanced Industrial Science and Technology (AIST), Olympus Corporation, Daiken chemical Co. Ltd., National Institute of Materials Science (NIMS), Tokyo University of Science

Keywords

Atomic force microscopy, Tip, Cantilever, Probe characterizer and CD measurement

Abstract

Atomic force microscopy (AFM) images are strongly affected by the shape of the AFM probe used for imaging. In this project, we aimed for establishing a technology to fabricate an AFM tip characterizer also to evaluate the tip shape. We established a process to deposit multilayer and succeed in fabricating tip characterizers which have multiple trench patterns with from 5 to 100 nm and a narrow ridge with 1.5-nm curvature. Using the tip characterizer, AFM users can evaluate the tip shape. Besides that, we developed an algorithm to correct the AFM image. These results are contributing to an international standardization for AFM.

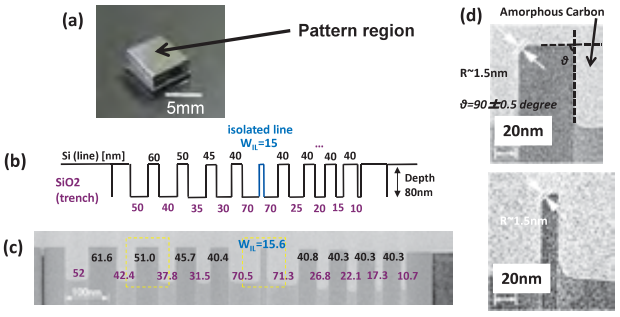


Fig. 1 Cross sectional TEM image of a tip characterizer
(a) Outlook of the proto type, (b) Designed value,
(c) Measured value using TEM image
(d) TEM image of a 40 nm-width ridge and the 15 nm-width ridge.

Apparatus name

AFM tip characterizer and the software to reconstruct the AFM image

Technical Overview

AFM is a powerful tool to measure the shape of nanometric structures. However, AFM images are strongly affected by the shape of the AFM probe used for imaging. So AFM users need to know the shape of AFM probe tip to measure nanostructures. Therefore, we developed the tip characterizers to evaluate the probe tip shape. The developed characterizers have a narrow-ridge structure, multiple-trench structure and a grating scale. Besides that, we developed the procedure to reduce the effect of the tip shape for the AFM image as well as a software to reconstruct the AFM image using the determined tip shape.

We also developed a detection method for the phase change, and then succeeded in tracing the true shape. Figure 2 shows an example to measure the diameter of a multi-wall carbon-nano tube (MW-CNT) after the AFM tip had been evaluated using the tip characterizer. The widen AFM image of MW-CNT was corrected with the tip shape, and then we could measure the diameter of MW-CNT within an error of 1 nm. Using the tip characterizer, we can evaluate not only the tip shape by measuring the 15-nm-narrow ridge structure, but also the detection limit by measuring the multiple-trench depth. In figure 2(b), the probe shape characteristics are drawn using the effective probe shape characteristic method combined with the measured data of the multiple-trench structure. In order to measure the tip diameter in detail, we made a CNT type characterizer whose CNT is bridged on the multiple-trench structures. Using the CNT type characterizer, the tip shape is measured easily.

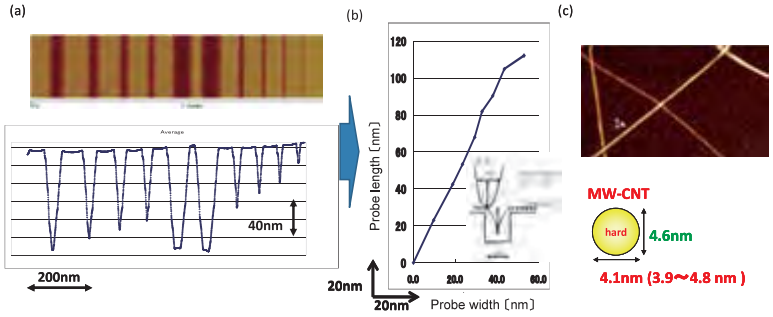


Fig. 2
(a) AFM image and line profile of the characterizer.
(b) Probe shape characteristic from the effective probe shape.
(c) Example to measure a diameter of a multi-wall carbon nano-tube after the AFM probe shape was evaluated.

Technical Performance

The developed depositing process can make a 5-nm-width-narrow ridge structure and 3-nm-width trench structure. The developed tip characterizer contains 15-nm-width ridge structure, multiple-trenches with 10 nm to 50 nm and 25-nm-pitch scale, also being practically mass-producible. The corner radius of the ridge pattern is less than 2 nm, typically 1.5 nm, the angle of inclination is less than 0.5°. Using the tip characterizer, AFM users can evaluate their tip shape within an errorrange of 1~2 nm.

Publications

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2) S. Ichimura *et al.,* "Current standardization activities for the measurement and characterization of nanomaterials and structures", *J. Phys. Conf. Series* 159, 012001, (2009).
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Contact
Person in charge : Akio Takano
Affiliation : NTT-AT Corporation
E-mail : akio.takano@ntt-at.co.jp

Result No.12

Portable Total Reflection X-Ray Fluorescence Elemental Analyzer

Team Leader

Jun Kawai

Kyoto University

Participating organization

Kyoto University

Keywords

Analytical chemistry, Trace elemental analysis

Abstract

In X-ray fluorescence (XRF) analysis, trace elemental analysis was usually performed using a stationary X-ray analyzer with a high power X-ray source. Measurements at synchrotron radiation facility made it possible to detect elements in the fg range (1 fg = 10^{-15}g). On the other hand, we have developed a light weight (less than 6 kg) portable total reflection X-ray fluorescence (TXRF) elemental analyzer with a several watts X-ray tube. Although a low power X-rays, elements in the pg range can be detected.

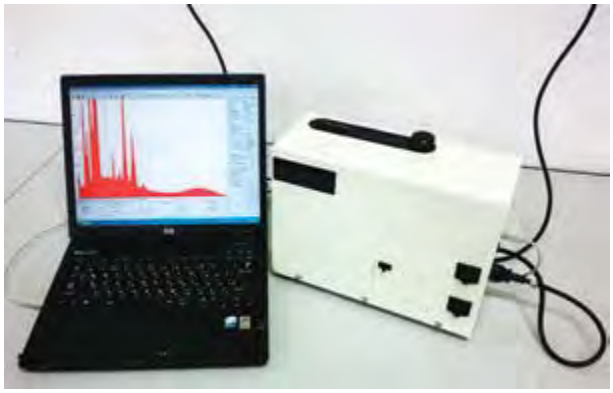


Fig. 1 Portable total reflection X-ray fluorescence elemental analyzer.

Apparatus name

OURSTEX 200TX (Ourstex Co., Ltd., Neyagawa, Japan)

Technical Overview

The portable TXRF elemental analyzer mainly consists of a naturally air-cooled X-ray tube, a semiconductor X-ray detector, an X-ray waveguide as a collimator, and an X-ray reflector as a sample holder. This portable analyzer is less than 6 kg, and it is possible to carry by hand. An X-ray tube is usually operated at 25 kV and 200 μA (5 W), and detection limits down to 5 pg have been achieved by 5 W X-ray tube.

Analysis of ppb (μg/L) concentrations of elements in water solution samples can be performed by using the present portable analyzer, and the total volume of a sample solution needed for a measurement is typically 10 μL. For example, trace elemental analysis of river water, commercial bottled drinking water, wines, a leaching solution of soil, a leaching solution of toy, and a leaching solution of metallic material were performed without pre-treatment. Using sample preparation techniques leads to improvement in detection limits. Microwave decompositions for blood analysis, solid-phase extraction for seawater analysis, and a combination of acid decomposition of steel and separation of iron for steel analysis were performed, and a few nanograms of elements were detected after using these sample preparation techniques. Powder samples can be analyzed with the present portable analyzer after preparing suspensions. The present portable analyzer can be used for monitoring environmental pollution, safety evaluation of foods and toys, and manufacturing process control of metallic materials.

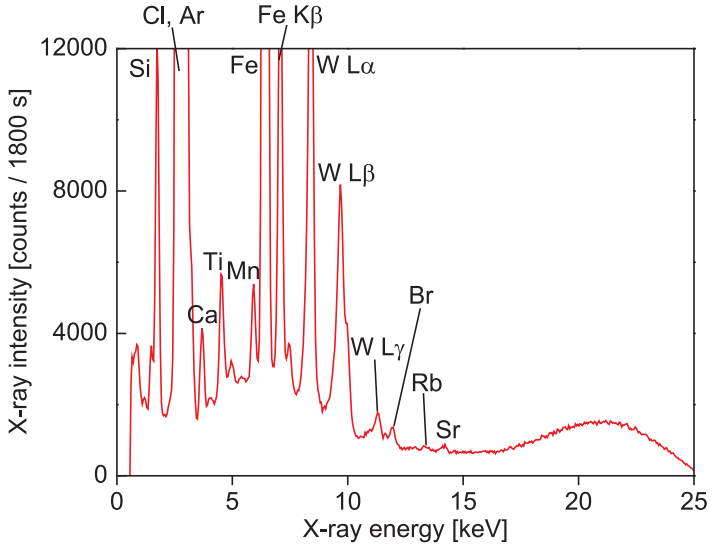


Fig. 2 Total reflection X-ray fluorescence spectrum of a leaching solution of soil. To prepare this leaching solution, the soil sample was immersed into 1 M HCl.

Technical Performance

The portable TXRF elemental analyzer is used for analysis of elements with an atomic number greater than 14 (Si). Detection limits of representative elements are shown in Table 1.

Table 1. Detection limits of representative elements.

	Cr	As	Y	Cs	Cd	Pb
Detection limit (ng)	0.005	0.035	0.06	0.3	1	0.15
Measurement time (s)	1800	1800	1800	600	1800	1800
Anode material of X-ray tube	W	W	W	W	W	W
Tube voltage (kV)	25	25	25	25	40	25
Tube current (μA)	200	200	200	200	200	200

Publications

1) S. Kunimura and J. Kawai, Polychromatic Excitation Improves Detection Limits in Total Reflection X-Ray Fluorescence Analysis Compared with Monochromatic Excitation, *Analyst*, 135, 1909-1911, 2010.
2) S. Kunimura, D. P. Tee, J. Kawai, Analysis of Nanograms of Cadmium Using a Portable Total Reflection X-Ray Fluorescence Spectrometer, *Tetsu-to-Hagané*, 97, 81-84, 2011.
3) S. Kunimura and J. Kawai, Application of a Portable TXRF Spectrometer to Determine Trace Amounts of Toxic Elements, *Advances in X-Ray Analysis,* 53, 180-186, 2010.

Contact
Person in charge : Shinsuke Kunimura, Jun Kawai
Affiliation : Tokyo University of Science (SK), Kyoto University (JK)
E-mail : kunimura@ci.kagu.tus.ac.jp (SK), kawai.jun.3x@kyoto-u.ac.jp (JK)

Result No.21

Development of an advanced gamma-ray imaging system with an ultra-wide angle field of view and a high sensitivit

Team Leader

Tadayuki Takahashi

Japan Aerospace exploration Agency (JAXA)

Sub Leader

Yoshikatsu Kuroda

Mitsubishi Heavy Industries (MHI), Ltd.

Participating organization

JAXA, Mitsubishi Heavy Industries (MHI), Ltd., Nagoya University

Keywords

Gamma-ray imaging, Si/CdTe Compton camera, radioactive substances.

Abstract

A gamma-ray imaging system equipped with a wide-angle vision and high sensitivity is desired to visualize radioactive substances (Cesium 137 and 134) released from Fukushima Daiichi nuclear power plant. We have developed a new Compton camera consisting of silicon (Si) and cadmium telluride (CdTe) semiconductor detectors, which is originally proposed by ISAS/JAXA group for the next-generation gamma-ray astrophysics. This camera covers almost 180 degree (2π steradian), hence it can be utilized to obtain images of the radioactive substances over a large area of ground or a house that are difficult to survey using an investigation method by human with a survey meter. Mitsubishi Heavy Industry released the first commercial Compton camera, "ASTROCAM 7000 HS".

Fig. 1 Photograph of ASTROCAM 7000 HS. The Si/CdTe Compton camera and an optical fish-eye camera are included.

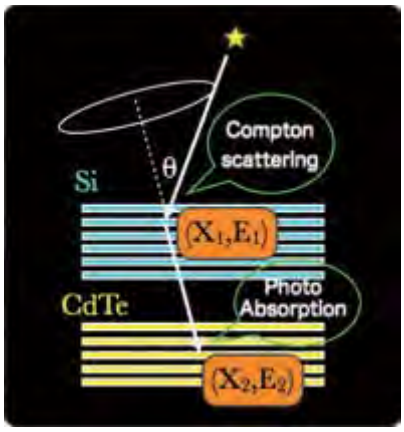


Fig. 2 Conceptual view of Si/CdTe Compton camera consisting of many layers of Si and CdTe semiconductor imaging devices. Gamma-ray scattered in Si part and then absorbed in CdTe part are recoded by the detectors, and used image reconstruction.

Apparatus name

Ultra-wide angle Compton Camera and ASTROCAM-7000HS

Technical Overview

Gamma-rays emitted from Cesium 137 and 134 have energies ranging 600 to 800 kilo electron volts (keV). A Compton camera is a visualization technology that constraints the direction of incident gamma-rays by using Compton scattering process. The Compton camera can locate the radioactive substances without using a pinhole collimator and lead shields that are used in conventional gamma cameras,

We have developed a "Ultra-wide-angle Compton Camera", which has a wide field of view, almost 180 degrees (2π steradian) and an angular resolution of several degrees in the energy range of 500 to 800 keV. The camera is based on a concept to use densely stacked Si and CdTe semiconductor imagers (Si/CdTe Compton camera), The concept is originally proposed by ISAS/JAXA for the ASTRO-H X-ray astronomy satellite. The combination of Si and CdTe is thought to be ideal to achieve both good angular resolution and high efficiency. Since the effect of the Doppler broadening is smaller in the Si devices than other semiconductor devices, ambiguities in determining the direction of incident gamma-ray is small in our camera. CdTe semiconductoris good for absorption part thanks to its high probability of photo-absorption and high density.

"ASTROCAM 7000 HS" is the first commercial Compton camera released by Mitsubishi Heavy Industry, based on our data taken in Fukushima by using the prototype Si/CdTe cameras developed in this project. Its large of view and a high-contrast image give great advantages in hotspot survey over a large area of ground or a house. We performed imaging tests at the evacuation zone, and demonstrated abilities to detect hotspots on the environmental radiation level of even below 1μSv/h.

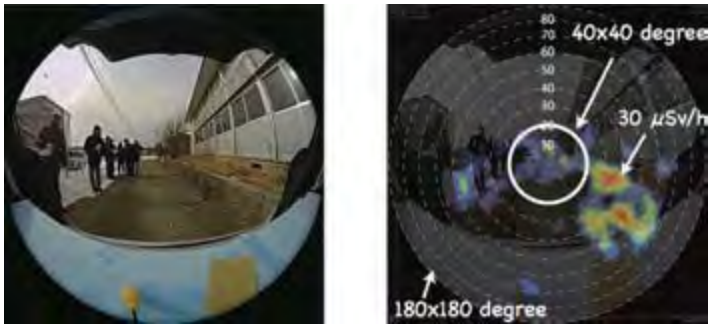


Fig. 3 Visualization of radioactive substances. Optical image (left) taken by the fish-eye camera is overlaid with the gamma-ray image by the Compton camera (right). Ultra-wide vision covering almost 180×180 degree is achieved, while that of typical gamma camera is about 40×40 degree (bold white line).

Technical Performance

Following is the specifications of ASTROCAM 7000 HS.

Dimensions	445L×340W×235H (mm)
Weight	Approximately 8-13kg (Camera Unit) <depending on specifications>
Power Source	AC 100-240V and Battery
Operating temperature	0 to 40 degree Celsius
View angle	180 degree (2π steradian)
Energy resolution	Approximately 2% (FWHM) @ 662keV
Angular resolution	Approximately 5degree @ 662keV
Efficiency	0.16cps/MBq @ 1m, 137-Cs (standard configuration) 2.8cps/MBq @ 1m, 137-Cs (expanded configuration)

Publications

1) Tadayuki Takahashi et al. "High resolution CdTe detectors for the next generation multi-Compton gamma-ray telescope", Proc. SPIE, vol.4851, pp.1228-1235, 2003
2) Shin Watanabe et al. "A Si/CdTe semiconductor Compton camera", IEEE Trans. Nucl. Sci, vol.52, no.5, pp.2045-2051, 2005
3) Shin'ichiro Takeda et al. "Applications and imaging techniques of a Si/CdTe Compton gamma-ray camera", Physics Procedia, vol.37, pp.859-866, 2012

Contact
Person in charge : Tadayuki Takahashi
Affiliation : ISAS/JAXA
E-mail : takahasi@astro.isas.jaxa.jp

Result No.13

Development of automated asbestos counting system based on bio-fluorescence method

Team Leader

Akio Kuroda

Hiroshima University

Participating organization

Hiroshima University, INTEC Inc., Siliconbio inc.

Keywords

Asbestos, Bioimaging, Fluorescence microscope, Image analysis

Abstract

Using a protein that can specifically bind to asbestos, we previously developed an easy and highly sensitive bio-fluorescence method to detect asbestos fibers under fluorescence microscopy (JST Development of Systems and Technologies for Advanced Measurement and Analysis, 2007-2010). However, analysts still need to follow complex rules when identifying and counting asbestos fibers, making asbestos analysis a complicated and time-consuming task. Asbestos fibers on the sampling filter membrane may cross or get entangled with each other, adopt a twisted shape, and overlap with non-fibrous dust particles, leading to large variability in the resulting asbestos counts among different analysts. To address this problem, we have developed automated asbestos detection and counting system that would enable anyone, and not just experienced analysts, to reliably identify and count asbestos fibers.

Fig. 1 Bio-fluorescent asbestos testing kit (A) and asbestos detection and counting system (B)

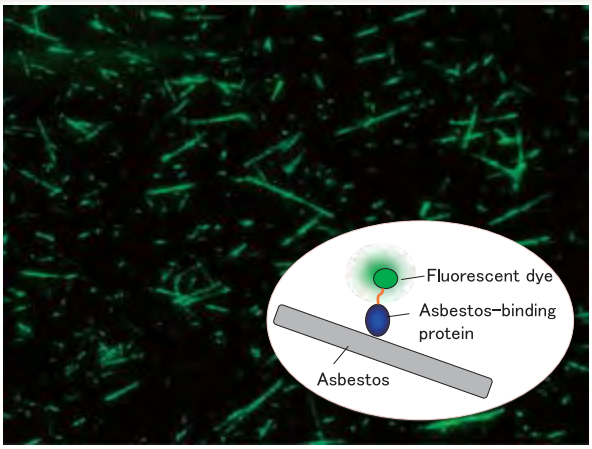


Fig. 2 Fluorescent image of asbestos obtained by bio-fluorescent asbestos testing kit

Apparatus name

Automated asbestos detection and counting system

Technical Overview

The amount of asbestos-containing construction materials in Japan has been estimated at 40 million tonnes. During demolition of the old buildings, which is expected to peak in the near future, it is necessary to check whether any asbestos fibers are released into the environment. We previously developed a novel bio-fluorescence method that solved the problems of the conventional methods, such as insufficient rapidity and selectivity, presenting a unique opportunity to greatly simplify on-site asbestos testing (figure 1 and 2).

However, asbestos analysis still remains a complicated and time-consuming task, as analysts need to follow complex rules when identifying and counting asbestos fibers on up to 100 fields of view per each sample. Therefore, we developed a software algorithm for counting such fibers according to the officially-endorsed "Asbestos counting rules". The software can automatically correct for the differences in fluorescent background and fiber brightness, which arise due to diverse sampling and imaging conditions. Since some non-fibrous particles are naturally fluorescent, we also developed a method for selective image correction (removing particle image areas) followed by localized background adjustment, leading to more accurate fiber identification. The use of software greatly simplified asbestos analysis, and the test results were well correlated with the results of the conventional asbestos testing method, confirming the effectiveness of the developed software for rapid asbestos testing (figure 3). This automated asbestos-counting system enabled even non-experienced analysts to perform reliable and rapid on-site detection or inspections for airborne asbestos.

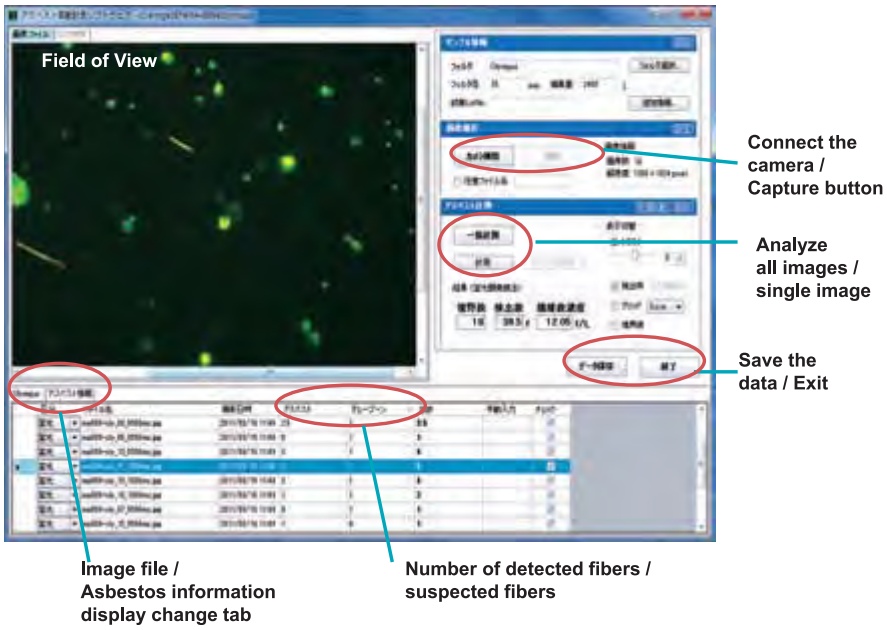


Fig. 3 Automated asbestos detection and counting software screenshot

Technical Performance

The developed system automatically detects and counts asbestos fibers according to the "Asbestos counting rules." The system is capable of detecting asbestos fibers with diameter above 30 nm. Asbestos testing using the system takes only one hour, including the time necessary for sample preparation, examination of the filter under fluorescent microscope, image acquisition, and automated fiber counting by the software. Portable fluorescence microscope can be used to acquire images, making it possible to conduct on-site analysis. Automated fiber counts generally fall within 10% of average fiber counts by experienced analysts.

Publications

1) T. Ishida, A. Kuroda *et al*, Selective detection of airborne asbestos fibers using protein-based fluorescent probes, *Environ Sci Technol.*, 44 (2), 755-9, 2010.
2) T. Ishida, A. Kuroda *et al*, Evaluation of Sensitivity of Fluorescence-based Asbestos Detection by Correlative Microscopy, *J. Fluorescence*, 22, 357-363, 2012

Contact
Person in charge : Prof. Akio Kuroda
Affiliation : Department of Molecular Biotechnology, Graduate School of Advanced Sciences of Matter, Hiroshima University
E-mail : akuroda@hiroshima-u.ac.jp

Result No.14

Development of Lidar System for Simultaneous Measurements of CO_2 Density, Wind and Temperature Profiles

Team Leader

Chikao Nagasawa

Tokyo Metropolitan University

Sub Leader

Makoto Tsukamoto

EIKO Instruments Co., Ltd.

Participating organization

Tokyo Metropolitan University, EIKO Instruments Corporation

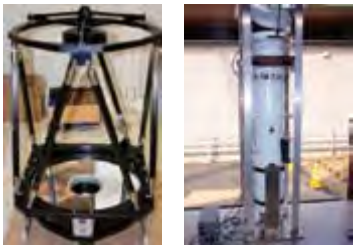
Keywords: Lidar, CO_2, Wind

Abstract

We have developed a direct detection 1.6μm differential absorption lidar (DIAL) to perform range-resolved measurements of vertical CO_2 concentration profiles in the atmosphere. And also, a scanning 1.6μm DIAL and an incoherent Doppler lidar system have been developed to perform simultaneous measurements of CO_2 concentration and wind speed profiles in the atmosphere. Our 1.6μm DIAL system consists of the Optical Parametric Generator (OPG) transmitter, the receiving optics including the near-infrared photomultiplier tube in the photon counting mode, and the telescope with large aperture. Laser beams of three wavelengths around a CO_2 absorption line are transmitted alternately to the atmosphere for measurements of CO_2 concentration and temperature profiles. A fiber Bragg grating (FBG) filter is used to detect Doppler shift for measurement of wind profiles.

CO_2-DIAL System Installed in Trailer

Transmitter of CO_2-DIAL



Compact 60 cm telescope and 25 cm telescope with scanning mirror

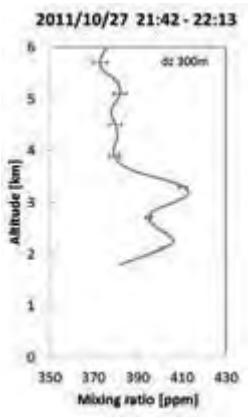
Apparatus name

Lidar System for Measurements of CO_2 Density, Wind and Temperature Profiles

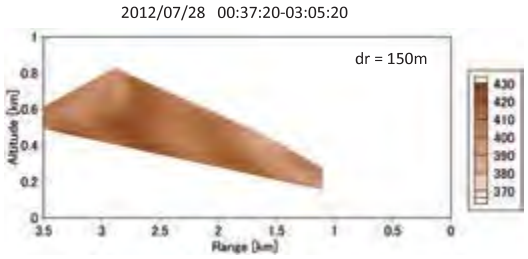
Technical Overview

The accurate vertical CO_2 profiles in the troposphere are increasingly required to improve quantification of the global CO_2 budget and also to understand global climate changes. In comparison with the ground-based monitoring network, CO_2 measurements for vertical profiles in the troposphere have been conducted by dirigible airplanes and commercial airlines so as spatial and temporal coverage are limited. Horizontal CO_2 distribution and wind profiles are important information to understand the regional sink of CO_2 and its source. The differential absorption lidar (DIAL) and the renge resolved Doppler lidar are expected to bring several advantages over passive measurements.

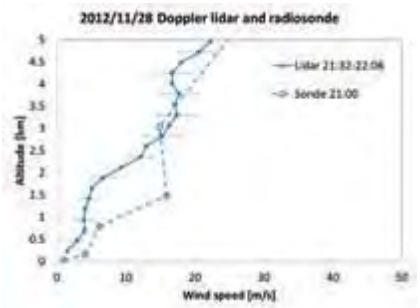
Our 1.6 μm DIAL system consists of the Optical Parametric Generator (OPG) transmitter excited by the LD pumped Nd:YAG laser with high repetition rate. It also consists of the receiving optics with a near-infrared photomultiplier tube of high quantum efficiency in the photon counting mode. This system also includes a telescope with larger aperture than that of the coherent detection method. Laser beams of three wavelengths around a CO_2 absorption line are transmitted alternately to the atmosphere for measurements of CO_2 concentration and temperature profiles. Moreover, retrieval algorithms of CO_2-DIAL are also performed to improve measurement accuracy. The scanning 1.6 μm DIAL and the incoherent Doppler lidar system can perform simultaneous measurements of CO_2 concentration and wind speed profiles in the atmosphere. Laser beam is transmitted coaxially, and then a motorized scanning mirror system scans the laser beam and field of view 0-360deg horizontally and 0-52deg vertically.



An Example of Vertical CO_2 Profile



An Example of Vertical Scanning Measurement of CO_2 (Range-Height Indication)



An Example of Horizontal Wind Speed Profile

Technical Performance

The vertical CO_2 concentration profiles from ground to an altitude of 10km can be obtained with 200m (at the lower altitude range) and 500m (at the upper altitude range) altitude resolutions by integration time of 30 minutes within 1% standard deviation. Moreover, this CO_2 DIAL system can be used as the scanning lidar in the atmospheric boundary layer. We can obtain vertical wind vector profiles up to 5 km altitude with 1km altitude resolution by measuring line-of-sight wind profiles at two azimuth angles with a fixed elevation angle.

Publications

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2) C. Nagasawa et al., Direct detection 1.6 μm DIAL for measurements of CO_2 concentration profiles in the troposphere, *Proc. SPIE* 8182, 81820G, 2011.

Contact
Person in charge : Chikao Nagasawa
Affiliation : Tokyo Metropolitan University
E-mail : nagasawa@tmu.ac.jp

Result No.15

Development of high accuracy and high stability pH combination electrode equipped with ionic liquid salt bridge

Team Leader

Satoshi Nomura

HORIBA Ltd.

Sub Leader

Takashi Kakiuchi

Kyoto University

Participating organization

HORIBA Ltd., Kyoto University, Akita University, Nihon-HELS Co. Ltd.

Keywords

pH sensors, Ionic liquid, Salt bridge, Dilute aqueous solutions, Environmental analysis, Pharmaceutical research

Abstract

Conventional pH electrodes have intrinsic problems such as the unstable electrode response in dilute solutions and the contamination of the sample due to the leakage of concentrated KCl. These problems originate from the working principle of the salt bridge (SB) used in the reference electrode, which relies on the diffusion of concentrated KCl. In this project, a new type of SB based on the distribution of ions constituting an ionic liquid (IL) into water was improved so that it can be applied to the reference electrode for accurate pH measurement. The new type of pH electrode equipped with new type of SB has enabled us to determine more accurate pH of low ionic strength sample solutions than conventional pH electrode.

Fig. 1 pH combination electrode equipped with ILSB, “PUREIL”

Apparatus name

pH combination electrode equipped with liquid salt bridge. “PUREIL”

Technical Overview

The new type of SB uses a hydrophobic IL, which generates thermodynamically stable liquid junction potential difference between IL and dilute aqueous solutions. The new type of SB can also eliminate the change in pH due to the leakage of concentrated electrolyte from SB into the sample solution. These advantages of the new pH electrode equipped with ionic liquid salt bridge (ILSB) over the conventional pH electrodes that have been used over a century are distinctive.

It is noteworthy that more accurate pH values could be obtained with this electrode in environmental water samples such as rain water and surface water. Further, accurate pH values of these environmental water samples are expected to contribute to more precisely and quantitatively understanding environmental phenomena depending on pH, including the toxicity of natural water to animals.

Another application of the new pH electrode is the pH measurement of solutions in which the addition of concentrated electrolytes must be avoided. For example, in case of the medical injection solution, the contamination of concentrated electrolyte will change the characteristics of the sample solutions. Since pH is an important parameter that characterizes drug efficiency, the new pH electrode is expected to contribute deeply in research initiatives taken toward discovering new drugs. This advantage is also expected to be applied to enhancing research studies on macromolecule electrolytes, which form the basis of numerous advanced materials.

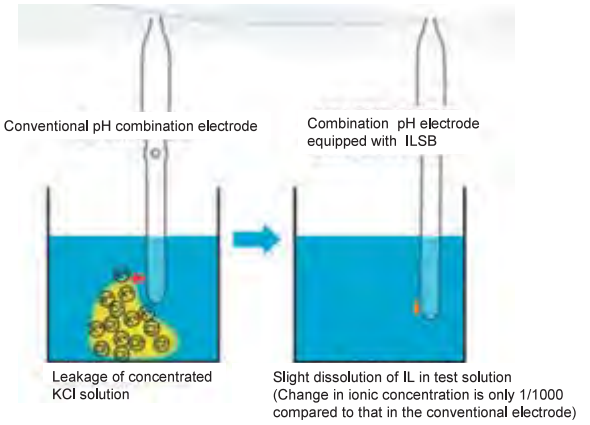

Fig. 2 Advantages of the composite pH electrode incorporating IL salt bridge.

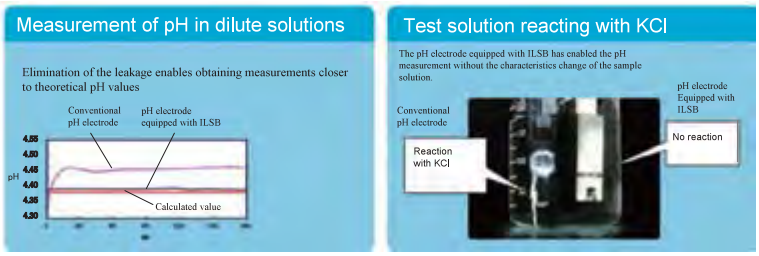

Fig. 3 Examples of applications of the combination pH electrode equipped with ILSB

Technical Performance

pH of sulfuric acid solutions with concentrations in the range of 20 μM–200 μM could be measured.

Precision of pH measurements: 0.003

Deviation from the calculated pH: < 0.03

In the case of a conventional pH electrode, the precision of measurements and the deviation from the calculated pH are reported as 0.055 and 0.055, respectively in R. C. Metcalf, *Analyst*, 112, 1573-1577, 1987.

Publications

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2) Stability of a Ag/AgCl Reference Electrode Equipped with an Ionic Liquid Salt Bridge Composed of 1-Methyl-3-octylimidazolium Bis (trifluoromethanesulfonyl) -amide in Potentiometry of pH Standard Buffers., M. Shibata, M. Yamanuki, Y. Iwamoto, S. Nomura, T. Kakiuchi, *Anal. Sci.,* 26, 1203, 2010
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4) Ionic Liquid Salt Bridge Based on Tributyl (2-methoxyethyl) phosphonium Bis (pentafluoroethanesulfonyl) amide for Low Ionic Strength Aqueous Solutions, Yousuke Fujino and Takashi Kakiuchi, *J. Electroanal. Chem.*, 651, 61-66, 2011

Contact
Person in charge : Dr. Manabu Shibata
Affiliation : HORIBA Ltd.
E-mail : manabu.shibata@horiba.com

Result No.16

Development of an Online Aerosol Particle Combined Analysis System

Team Leader

Nobuyuki Takegawa

University of Tokyo

Sub Leader

Noritomo Hirayama

Fuji Electric, Co., Ltd.

Participating organization

University of Tokyo, Fuji Electric, Co., Ltd., Japan Agency for Marine-Earth Science and Technology (JAMSTEC)

Keywords

Aerosol, Climate change, Air pollution, PM2.5

Abstract

Atmospheric aerosols have significant impacts on air pollution and climate change. We have developed a new analysis system based on laser and mass spectrometric techniques for online measurements of aerosol properties (size distribution, chemical composition, mixing state, etc.) relevant to the environmental issues. The system may also be applicable to particle monitoring in clean rooms or industrial processing sections.

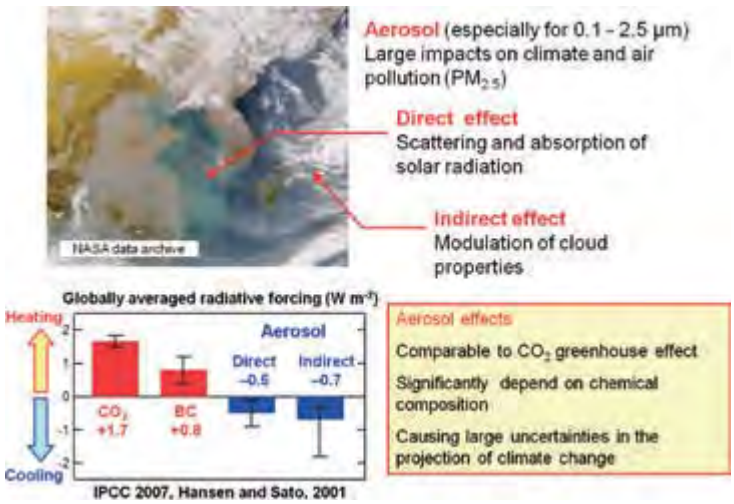

Fig. 1 (a) Importance of aerosols in atmospheric environment

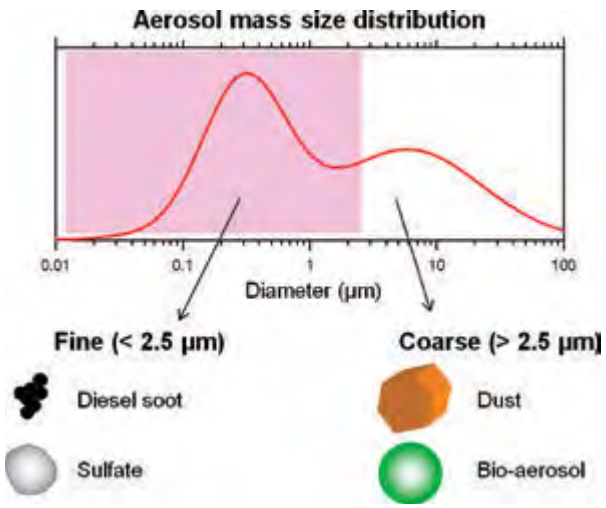

Fig. 1 (b) Typical aerosol mass size distribution

Apparatus name

Aerosol Particle Combined Analysis System

Technical Overview

Introduction

Aerosols (suspension of particles in air) play important roles in air pollution and climate change from local to global scales. Aerosol particles can efficiently scatter or absorb solar radiation (direct effect) and also alter cloud properties as cloud condensation nuclei (indirect effect). These effects can significantly affect the radiation budget of the Earth's atmosphere (Figure 1a). In addition, high concentrations of aerosols due to diesel exhaust and photochemical smog in urban air have adverse influences on human health.

Figure 1b represents a typical aerosol mass size distribution. Aerosol particles with diameters smaller than 2.5 μm are referred to as PM2.5. Aerosols consist of a number of compounds including black carbon and sulfate from anthropogenic origins and biogenic materials from natural sources. Development of instruments for online measurements of aerosol properties including size, composition, and mixing state is one of the research frontiers of atmospheric science. The purpose of our development is to meet these demands.

Instrument description

The concept of the Aerosol Combined Analysis System is to use multiple analysis methods to characterize variety of aerosol properties in sample air. Figure 2 shows a picture of the system. The major components include a laser-induced fluorescence (LIF) detector, laser-induced incandescence (LII) detector, and mass spectrometer (MS). A combination of these components provides a new insight into chemical composition and mixing state of aerosols. Figure 3 illustrates an example of time series of sulfate aerosols obtained by the system. Sulfate is one of the most relevant compounds of atmospheric aerosols and often contributes to the major fraction of PM2.5. Highly time-resolved measurements of aerosol composition would improve our understanding of sources and processes of aerosols.

Fig. 2 Prototype of Aerosol Particle Combined Analysis System

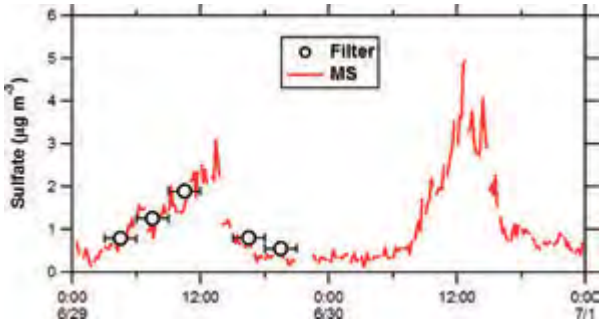

Fig. 3 Comparison of online prototype data with off-line filter analysis for sulfate (June 2012)

Technical Performance

Table. 1 Technical data

	Specifications (nominal)	Remarks
Diameter range	0.1-2.5μm	Approximately $PM_{2.5}$
Time resolution	5–20min or continuous	Depends on ambient aerosol concentrations
Sample flow	120cc min^{-1}	16.7L min^{-1} with cyclone
Main products		
Number size Distribution	Concentration range 0-5000cm^{-3} Number of size bins 20ch	LII
Black carbon mass concentration	Concentration range 0-100μg m^{-3} LOD (10s) <0.01μg m^{-3}	LII
SO_4^{2-}, NO_3^-, OC mass concentration	Concentration range 0-100μg m^{-3} LOD (SO_4^{2-}・5 in) 0.04μg m^{-3}	MS
Bio-aerosol	Excitation 266 nm, Fluorescence 3ch, Diameter >0.5μm	LIF
Mixing state	SO_4^{2-}, NO_3^-, OC mass concentration classified by BC mixing state	LII+MS
Size and weight	W1400・D800・H1500, 300kg	
Power supply	AC100V, 1.5kW	

Publications

1) Moteki, N., N. Takegawa, K. Koizumi, T. Nakamura, and Y. Kondo, Multiangle Polarimetry of Thermal Emission and Light Scattering by Individual Particles in Airflow, *Aerosol Science and Technology,* 45, 1184-1198, 2011.
2) Takegawa, N., T. Miyakawa, T. Nakamura, Y. Sameshima, M. Takei, Y. Kondo, and N. Hirayama, Evaluation of a New Particle Trap in a Laser Desorption Mass Spectrometer for Online Measurement of Aerosol Composition, *Aerosol Sci. Technol.*, 46, 428-443, 2012.
3) Taketani F., Y. Kanaya, T. Nakamura, K. Koizumi, N. Moteki, N. Takegawa, Measurement of Fluorescence Spectra from Atmospheric Single Submicron Particle Using Laser-induced Fluorescence Technique, *J. Aerosol. Sci.,* 58, 1-8, 2013.

Contact

Person in charge : Noritomo Hirayama
Affiliation : Fuji Electric, Co., Ltd.
E-mail : hirayama-noritomo@fujielectric.co.jp

Result No.17

Ultra compact measurement system for carbon dioxide monitoring

Team Leader

Shoichi Uematsu

Yazaki Corporation

Sub Leader

Gen Inoue

University of Tokyo

Participating organization

Yazaki Corporation, Research Institute for Humanity and Nature, Kyoto University, Hokkaido University, Nagoya University, Meisei Electric Co., Ltd.

Keywords

Carbon dioxide, CO_2, Sonde, High accuracy, Measurement system

Abstract

Carbon dioxide gas (CO_2) is considered as one of the primary global warming gases. More accurate measurement of vertical distribution of atmospheric CO_2 enables more precise evaluations of CO_2 emissions and absorptions. A CO_2 sonde has been used to investigate the vertical distribution of CO_2, but conventional apparatuses demonstrate lower performance than the required level.

We developed the ultra-small optical CO_2 sensors that contribute to more accurate measurement CO_2 concentration by sondes. Furthermore, we succeeded in the mass production of, the CO_2 sensors. The sensor can be used not only in meteorological balloon (sonde) for researchers but also in environmental education for students. The latter establishes the environmental education network to provide regional CO_2 concentration distributions.

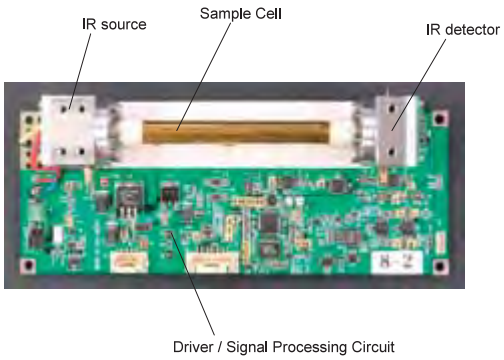

Fig. 1 CO_2 Sensor (Prototype)

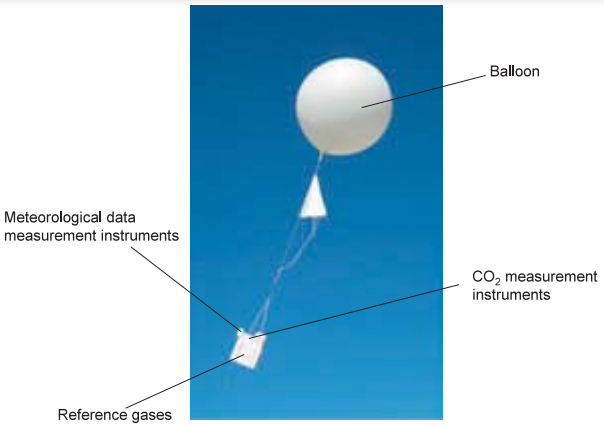

Fig. 2 CO_2 Sonde (Prototype)

Fig. 3 CO_2 meter for education (Prototype)

Apparatus name

- Balloon-borne CO_2 measurement system (CO_2 sonde)
- CO_2 meter for educational purposes

Technical Overview

Developments of two types of CO_2 meters have been carried out. One is balloon-borne CO_2 measurement system (CO_2 sonde) for its measuring vertical distributions and the other is a ground level CO_2 meter for environmental educations. We also aim to develop compact apparatus with high accuracy and low cost.

As the CO_2 sensing element, we adopt non-dispersive infrared (NDIR) method. The optimizations on IR source, IR detectors, cells, driving methods, signal processing method and temperature compensations have been carried out to develop a CO_2 sensor which meets the requirements on the accuracy, cost and size of the system. The developed CO_2 sensor is applied to CO_2 sonde and CO_2 meters for meteorological research, and also to those for environmental education.

During ascent, the CO_2 sonde measures CO_2 concentration, temperature, humidity and altitude, and transmits the data to the ground. The sonde carries two types of CO_2 reference gases (370ppm and 400ppm). The sensor in the sonde monitors the reference gases and external air alternately to perform accurate CO_2 measurement. In the launch test, the altitude resolution of approximately 250m was archived together with the CO_2 concentration measuring accuracy of approximately 1ppm. And in various tests, high stability of the measurement was constantly obtained.

A CO_2 meter developed for environmental education is equipped with a display, and the data can be stored to a SD memory card or a personal computer. Data transmission tests through a phone line were successfully carried out by using personal computer connected to the meter. And the CO_2 meter installed in a classroom detected the CO_2 concentration changes related to the daily school activities within an error range of approximately ±1ppm.

Technical Performance

Specifications of CO_2 sonde prototype

Measurement items		CO_2 Concentration, Temperature, Humidity, Atmospheric pressure, Wind Direction, Wind Speed, Altitude, Position
CO_2	Measurement Range	0ppm～1000ppm
	Accuracy	1ppm
	Altitude Range	0m～10000m
	Altitude Resolution	250m～300m
	Time Resolution	60sec～80sec
Reference gases		Equipped (2 types of CO_2 reference gases)
Size		approx. 280×270×140mm (w/o reference gas containers)
Weight		approx. 1000g
Data Transmissions	Transmission Range	250km
	Measurement Interval	1sec

Publications

1) Y. Matsumi et al. (T4-073) Development of balloon-borne CO_2 instruments, 8th International Carbon Dioxide Conference (Jena, Germany), 13-19 September 2009.
2) S. Takegawa et al. AAS001-P12 Improvements of Balloon-borne CO_2 instrument for the high accurate measurement of CO_2 vertical profile, Japan Geoscience Union Meeting 2010 (Makuhari, Chiba), May 23-28, 2010.

Contact

Person in charge : Shoichi Uematsu
Affiliation : Yazaki Research and Technology Center, Yazaki Corporation
E-mail : s-uematsu@ytc.yzk.co.jp

Result No.18

Development of Handy-type Smart Radiation Becquerel Counter.

Team Leader

Shigeo Okubo

Shinnihondenko Co., Ltd.

Sub Leader

Ryohei Sato

Osaka University

Participating organization

Shinnihondenko Co., Ltd., Osaka University, Mie University

Keywords

Cesium iodide crystal, Handy-Type, foods and soils.

Abstract

We develop a small and light weight γ-ray detector, named "H-SRBC" which has a large volume of CsI crystal (about 7 times larger than the conventional one).

This device realizes high speed measurement of radioactivity concentration and space radiation dose of foods, soils, forests, for both cesium-137 and cesium-134 nuclide with high accuracy and sensitivity.

Our cost effective and compact γ-ray detector demonstrates high competitiveness compare to other existing products.

By the present results of this development, it will realize a simple measurement for the radioactivity concentration in the foods and the spatial radiation dose.

1.Portable size & Easy measuring

H-SRBC is 17.5x20.0x16.0cm³ and 13kg.
Easy to be carried by single person.

By using a general plastic container,you can measure easily.

H-SRBC has a lead-shielding plates.

Bottom lid	:20.0mm
Upper lid	: 7.5mm
Side lid	:10.0mm

A commercial battery(Lithium-ion Battery DC5V 30Wh) installed in the counter enables the H-SRBC to be operated over 5 hours without commercial electricity power supply.

Fig. 1 H-SRBC (Handy type Smart Radiation Becquerel Counter)

I.S.C.Lab,inc. is the only company which manufactures cesium iodide(Thalium addition type)cyystals in Japan.
The CsI(Tl)scintillator is of 3.8x3.8x2.5cm³.

MPPC(Multi Pixel Photon Counter) show high stability of the performansce under the severe environmental circumstances such as atmospheric temperature, vibration, electric and magnetic disturbance.

Fig. 2 Large CsI (Tl) & MPPC (Multi Pixel Photon Counter)

Apparatus name

Handy type-Smart Radiation Becquerel Counter (H-SRBC).

Technical Overview

We develop a handy type smart radiation Becquerel counter (H-SRBC) for on-the-spot radiation contamination assessment of foods and environment in affected area by radioactive substances, originated from the Fukushima dai-ichi nuclear power plant accident.

Our previously reported mobile smart Becquerel counter (M-SBC) (1) has been supersensitized by adopting multi-pixel photon counter (MPPC) to the photodiode (PD). This has assured high energy resolution in γ-ray spectroscopy (～8%) as seen in Fig. 3. Highly stabile radioactivity measurement has been performed even under the severe environmental circumstances such as atmospheric temperature, vibration, electric and magnetic disturbance etc., which the M-SBC had been faced during on-the-spot inspection of γ-ray emitting substances.

The CsI (Tl) scintillator of $3.8\times3.8\times2.5cm^3$ is supplied from I.S.C. Lab. Co., Ltd. and connected to the MPPC (S11830-3344MF) (Fig.2) of HAMAMATSU PHOTONICS K.K. Whole body of the H-SRBC is $17.5\times20\times16cm^3$ with 13kg weight (Fig.1), which allows to be carried by one person. Furthermore, H-SRBC is able to be operated over 5 hours from build-in Lithium-ion Battery (DC5V 30Wh).

A typical example of the measurement of Cs standard sample provided by Japan Society of Analytical Chemistry is shown in Fig. 4.

The practical use of the H-SRBC for hot spots screening in the farms and/or forest is scheduled to be exemplified under the guidance of the Recovery Measures for Farms and Forest Unit, Fukushima Prefectural Government.

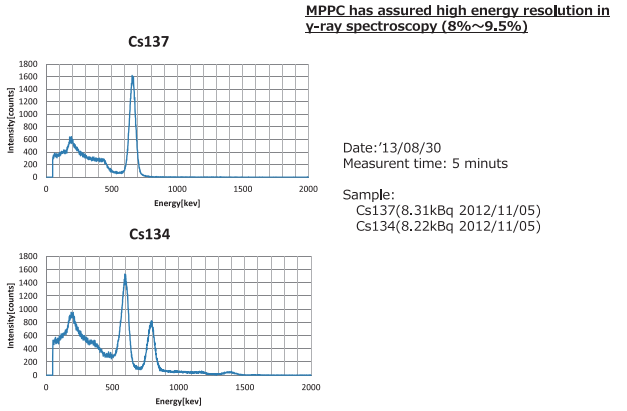

Fig. 3 Large CsI (Tl) & MPPC to achieve high resolution-①

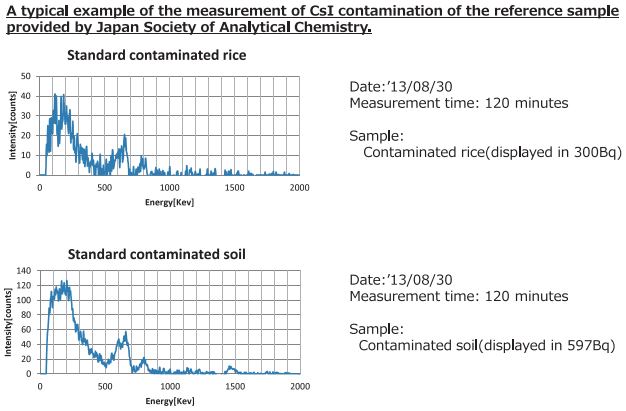

Fig. 4 Large CsI (Tl) & MPPC to achieve high resolution-②

Technical Performance

Equipment：H-SRBC (Handy type smart Radiation Becquerel counter)
Model number：H-SRBC3600A
Scintillation crystal：CsI (Tl) scintillator is of $3.8\times3.8\times2.5cm^3$ (thallium addition type cesium iodide crystal)
Detection element：MPPC (Multi Pixel Photon Counter) 2×2ch
Energy resolution：Less than 10% (Cs137)
Energy range：50kev～2000kev
Measurement range detection limit minimum：50Bq/kg ※100Bq/kg/20 minutes or less

Publications

1) Hideji Yumen, Shigeo okubo, Ken Kawamura, Yuuichiro Manabe, Yoshihide Kimura, Isao Murata, Kenzo Ikeda, Kyousiro Imagawa, Kensuke Yasuda, Ryohei Satou, Ryuichi Shimizu:
A Smart Becquerel Counter for On-site Inspection, Proc. 8th International Workshop on Ionizing Radiation Monitoring (Dec.1-2, 2012, Oarai, Japan) pp312-313.
2) Patent No2013-37315/Measuring device
3) J. Yoshii, R. Ikeda, Y. Kimura, K. Kawamura, Y. Manabe, R. Sato, S. Nagai, S. Okubo, R. Shimizu;
Software Platform for Smart Becquerel Counter,JSPS-141 Committee Technical Repoort#149th Meeting (2012.9.27-Nagoya) pp1-9

Contact
Person in charge： Goken Hino
Affiliation： Shinnihondenko Co.,Ltd.
E-mail： hino@snd.co.jp

Result No.19

Development of a method for rapid and simultaneous monitoring of particulate and dissolved radiocesium (^{137}Cs) in water

Team Leader

Yasuhiro Ito

Japan Vilene Company, Ltd.

Sub Leader

Tetsuo Yasutaka

National Institute of Advanced Industrial Science and Technology (AIST)

Participating organization

Japan Vilene Company, Ltd., National Institute of Advanced Industrial Science and Technology (AIST), Fukushima Agricultural Technology Centre

Keywords

Radiocesium in water, Nonwoven fabric, Prussian Blue, Particulate form, Dissolved form.

Abstract

Rapid and simultaneous radiocesium (^{137}Cs) monitoring method is developed for both suspended and dissolved ^{137}Cs in water. This method uses pleated polypropylene nonwoven fabric filter to collect ^{137}Cs particulate, and nonwoven fabric impregnated with Prussian blue (PB) to absorb water dissolved radiocesium. Each fabric was placed into cylindrical plastic cartridges (SS-cartridge and PB-cartridge).

Conventional monitoring methods require time consuming pre-processing such as evaporative concentration. Our method demonstrates shorter pre-processing time before the detection.

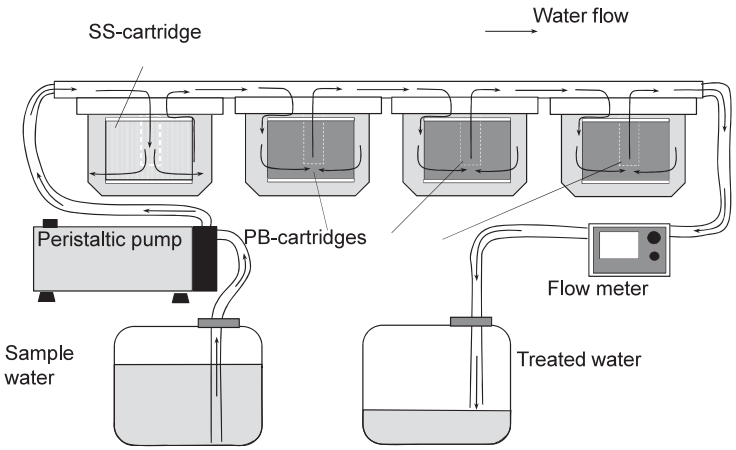

Fig. 1 Water flow in "JINSOKU-kun"

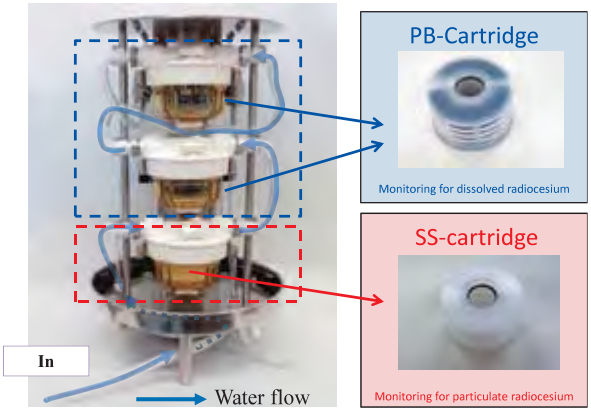

Fig. 2 Monitoring device "JINSOKU-kun"

Apparatus name

JINSOKU-kun, PB-Cartridge, SS-Cartridge

Technical Overview

An effective and simultaneous radiocesium monitoring system was developed for natural water. This system named "JINSOKU-KUN" consists of a feed-water tank, peristaltic pump, housing component with plain filter, flow meter, and drain tank. To monitor radiocesium in natural water, one only needs passing water through the system and measure trapped radiocesium concentration of each cartridge.

Two types of cartridge filter were designed to collect radiocesium in water. SS-cartridge is housed with the plain nonwoven fabric to collect water suspended solids including radiocesium particulates. This fabric is made of polypropylene fibers with a pore size of 1μm. PB-cartridge is made to absorb water dissolved radiocesium on Prussian Blue (PB) impregnated nonwoven fabric filter. PB is made up of potassium ferrocyanide (II) potassium oxide iron (II) KFe [Fe $(CN)_6]_3 \cdot zH_2O$), and its radiocesium absorbent property has been discovered by our previous study.

By using this system, the separation time for particulate and dissolved ^{137}Cs is reduced by 60 times compared to the conventional evaporative concentration method. Based on these results, "JINSOKU-KUN" present monitoring method exhibits remarkable efficiency for simultaneously monitoring particulate and dissolved radiocesium concentration in the field.

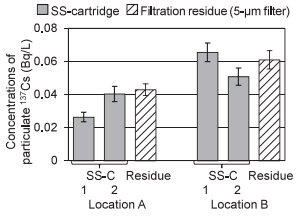

Fig. 3 Concentrations of ^{137}Cs of SS-cartridges(SS-C) and filtration residues by 5-μm filter. The concentrations of ^{137}Cs from the 0.45-μm membrane filters was less than the detection limit (0.005-0.009 Bq/L) Error bars indicate the counting error by Ge semiconductor detector

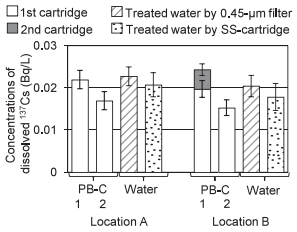

Fig. 4 ^{137}Cs of residues, treated water from SS-cartridges, and PB-cartridges(PB-C). Error bars indicate the counting error.^{137}Cs radioactivity of the second PB-cartridges was less than detection limit (0.004-0.006 Bq/L) except in Fukushima, and the third PB-cartridges were not served to the detector because of low concentration of the second PB-cartridges. Error bars indicate the counting error by Ge semiconductor detector

Technical Performance

The applicability of this system to the natural water monitoring was verified in laboratory by using environmental water mimic sample. Over 99 % of the suspended solids were collected in one SS-cartridge. Furthermore, two PB-cartridges could collect more than 92% (filtration rate; 2.5L/min) and 97% (filtration rate; 0.5L/min) of water dissolved ^{137}Cs under neutral pH in any given temperature. Monitoring of the Abukuma River by this method was evaluated and compared with conventional filtrating and evaporative concentration methods. The detected suspended solids concentrations, particulate and dissolved radiocesium concentrations measured by this method corresponded well with the results obtained by other methods.

	Pre-concentration of dissolved radiocesium		Pre-concentration of particulate radiocesium	
Method	Using two PB-Cartridges	Conventional evaporation methods	Using a SS-cartridge	Conventional filtering methods
Pre-concentration time for 20L of environmental water	10-40 minites	420-600 minutes (7-10 hours)	10-40 minites	120-180 minutes (2-3 hours)
Recovery rate	Over 92% (FR: 2.5L/min) Over 97% (FR: 0.5L/min)	100%	Over 99%	100%
Detection limits (Measuring time of Ge semiconductor detector)	0.006-0.008Bq/L (43200s)	0.011～0.014Bq/L (43200s)	0.007～0.011Bq/L (9000s)	0.010～0.019Bq/L (4000s)

Publications

1) Hideki Tsuji, Yoshihiko Kondo, and Yasukazu Suzuki, Tetsuo Yasutaka, (2014) Development of a method for rapid and simultaneous monitoring of particulate and dissolved radiocesium in water with nonwoven fabric cartridge filters, *Journal of Radioanalytical and Nuclear Chemistry,* 2014, Volume 299, Issue 1, pp 139-147

2) Tetsuo Yasutaka, Hideki Tsuji, Yoshihiko Kondo, and Yasukazu Suzuki (2013), Developemnt of Rapid Monitoring for Dissolved Radioactive Cesium with a Cartridge Type of Prussian blue-impregnated Nonwoven Fabric, *BUNSEKI KAGAKU* Vol.62, No.6, pp.499-506 (in Japanese)

3) Tetsuo Yasutaka, Tohru Kawamoto, Yoshishige Kawabe, Toshio Sato, Mutsuto Sato, Yasukazu Suzuki, Kimihito Nakamura, Takeshi Komai (2013) Rapid measurement of radiocesium in water using a Prussian blue impregnated nonwoven fabric, *Journal of Nuclear Science and Technology.* 50 (7), pp.674-681.

Contact

Person in charge : Yoshihiko Kondo, Tetsuo Yasutaka
Affiliation : Japan Vilene Company, Ltd., National Institute of Advanced Industrial Science and Technology
E-mail : y-kondo@vilene.co.jp, t.yasutaka@aist.go.jp

Result No.20

Certified reference materials for determination of environmental radioactivity

Team Leader

Yoshitaka Minai

Musashi University

Sub Leader

Hiroshi Iwamoto

Environmental Technology Service Co. Ltd.

Participating organization

Musashi University, Environmental Technology Service Co. Ltd., National Institute of Advanced Industrial Science and Technology (AIST), Saitama University, Japan Institute of International Affairs, Japan Society for Analytical Chemistry

Keywords

Quality control, Cs-134, Cs-137, Food

Abstract

Application of proper reference material is an indispensable element for ensuring the reliability of determination of chemical constituents. Our project focus on establishing production technology of reference materials for determination of radioactivity in foods and agricultural products. The reference material of brown rice, having the certified values for radioactivity of radiocesium, was the first product of this program; it was released before the fall harvest in 2012. The certified values based on the analytical data from ca.10 collaborative institutes have been confirmed by some foreign organizations. Consequently, the reference materials of beef, soybean, and mushroom were developed in the year 2012-2013.

Fig. 1 Certified reference material of brown rice (granular): (left) 100 mL bottle and (right) 1 L bottle packages, respectively.

Apparatus name

Certified reference materials for determination of environmental radioactivity

Technical Overview

The reference materials were prepared from foodstuffs and agricultural products contaminated by radionuclides possibly released from the Fukushima Daiichi Nuclear Power Plant. Our group successfully develops the reference materials of brown rice, beef, soybean, and mushroom, having the certified values of radioactivity for ^{134}Cs, ^{137}Cs, and ^{40}K.

In the case of the development of brown rice reference material, over hundred kilograms of the brown rice granules, having several hundreds Bq/kg of radiocesium, harvested from a village in the Eastern Japan was used as the source of the reference material developed. The brown rice granules were gently mixed to achieve homogenous distribution, packed into two types of brown bottles. After γ-irradiation for sterilization, several bottles were chosen for homogeneity test. This indicated low uncertainty in each certified value for inhomogeneity. Finally, following the procedures noted in JIS based on ISO, the inter-laboratory collaborative test (Round-Robin test) was carried out to obtain the certified values for radioactivity of ^{134}Cs, ^{137}Cs, and ^{40}K. The thorough reviewing on development procedures and certification with the experts in the Japan Society of Analytical Chemistry allows to distributing the reference materials as one of the composite reference materials having certified values issued by the society. The other standard materials have developed in similar manner.

The reference materials would be used to control the quality of food analysis in food production and distribution system. Developers of analytical instruments would be interested in the reference materials to ensure the performance of the developing apparatus.

CRM Code	Nuclides	Certified Valus±*U* (*k*=2) (Bq/kg)
JSAC 0731* JSAC 0732**	^{134}Cs	141 ± 9
	^{137}Cs	210 ± 13
	^{40}K	75 ± 7

* : JSAC 0731 100 mL, ca. 90 g
** : JSAC 0472 1 L, ca. 900 g

Table. 1 Certified values of radioactivity in the brown rice (granular) reference materials. Reference Date: June 1. 2012, JST 00:00:00.

Technical Performance

Certified values for concentrations of radionuclides in the reference materials of beef, soybean, and mushroom are listed as follows.

CRM Code (JSAC)	Material	^{134}Cs (Bq/kg)	^{137}Cs (Bq/kg)	^{40}K (Bq/kg)	Reference date
0751 0752	Beef	174±12	297±20	276±46	11/19/2012
0761 0762 0763	Soybean	37.1±2.6	68.2±4.6	619±60	2/1/2013
0753 0754	Beef	63±6	106±9	283±54	11/19/2012
0764 0765 0766	Soybean	190±11	345±19	613±40	2/1/2013

※Certified reference material for mushroom is in prepation

Publications

1) The measurement comparability of ^{134}Cs and ^{137}Cs in foodstuff samples in Japan-result of inter-laboratory experiment for certification of certified reference material, Tsutomu Miura et al., Yoshitaka Minai, Shoji Hirai, Hiroshi Iwamoto, Chushiro Yonezawa, Yoshinobu Uematsu, Akira Okada, Masami Shibukawa, Koichi Chiba, Kiyoshi Kitamura, Takahiro Yamada, Kazutoshi Kakita, and Isao Kojima, Journal of Radioanalytical and Nuclear Chemistry, in press.

Contact
Person in charge : Ken-ichi Abe
Affiliation : Japan Society for Analytical Chemistry
E-mail : crmpt@ml.jsac.or.jp

Keywords Index

Name Index

Institution Index

Office of Advanced Measuring Technology, Japan Science and Technology Agency

K's Gobancho, 7, Gobancho, Chiyoda-ku, Tokyo, 102-0076 Japan
E-mail: sentan@jst.go.jp http://www.jst.go.jp/sentan/

2014.03

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