Comprehensive Analysis Center



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SANKEN Osaka University

Introduction

Material Analysis Center (MAC), the predecessor of Comprehensive Analysis Center (CAC), had been established in 1977 as an affiliated facility of the Institute of Scientific and Industrial Research (ISIR). CAC, with the reorganization of MAC integrating the former Electron Microscope Room, was established in 2009 as a common comprehensive facility to support a wide range of basic and applied research fields in ISIR. CAC staffs consist of 1 associate and 2 assistant professors (as full-time professors), 5 technical staffs, 1 part-time staffs, 5 associate and assistant professors (concurrent post) and 1 director (concurrent post).

Fortunately, immediately after the renewal, CAC could update many of decrepit instruments to globally advanced instruments by obtaining a supplementary budget in 2010 to be equipped with observation instruments including component and surface analyzers, spectrometers and electron microscopes, shown in this booklet, to comprehend various research fields in ISIR. The instruments are maintained and managed by CAC staffs to allow users to utilize at any time. CAC staffs support analyses by utilizing instruments required expertise and each researcher can utilize easily operable instruments all day. Lectures for instrument users, including instrumental analysis lectures for new students, is vigorously held every year. Fully utilize the instruments in CAC if you read this booklet and/or CAC users' guide.

CAC is an ISIR-affiliated common facility and primarily positioned as a research support facility in ISIR. Additionally, CAC enhances cooperation with collaborative research facility networks centering on Center for Scientific Instrument Renovation and Manufacturing Support, Osaka University. CAC users consisting of intramural and extramural researchers and Incubation Building-located company workers are currently increasing. CAC was highly evaluated by external evaluations held in 2012 as "an open facility which is a model case in Japan and Osaka University should be proud of".

Above-mentioned performance related to university-wide supports of education and research was recognized in 2015. An expenditure of CAC-proposed project "promotion of university-wide education for material state analysis" has been supported by presidential discretionary expenditure (intensive promotion expenditure for education and research). All CAC staffs make further efforts to effectively utilize the expenditure to allow all users to give excellent research results. The CAC full-time professors conduct their original research related to organic, organometallic and analytical chemistry by fully utilizing instruments in CAC. Additionally, CAC staffs vigorously participate in opening CAC to the public (Icho Festival), tours for high school students and introduction activity of advanced instruments and research.

CAC staffs would appreciate the understanding and cooperation of all users' for maintenance and development.

 $\langle Map \& Access \rangle$





① 1st Research Building ② 2nd Research Building ③ Nanoscience and Nanotechnology Center Building (4) Comprehensive Analysis Center (CAC) (5) Administration Building (6) Common Laboratory (7) Cobalt Building Center (8) Linac Building (9) Anechoic Room (10) Accommodation KUSUMOTO (11) Incubation Building (12) Open Innovation Building (3) Electron Microscope Room (S-104, I-107, S-113) (4) Chemistry Laboratory (F244 and F246) (b) Electron Microscope Room for Biology (F192 and F194) (b) Nuclear Magnetic Resonance Room (F428 and F507)





[Monorail] 15 minute-walk from Handai Byoin Mae Station on Osaka Monorail (via Banpaku Kinen Koen Station)

Floor Map



Staff List

Director * : Akira Oiwa Associate Prof : Takeyuki Suzuki Assistant Prof : Dayang Zhou Assistant Prof : Kaori Asano Associate Prof * : Mitsuko Nishino Associate Prof * : Tomoyo Goto Assistant Prof * : Yasunobu Yamashita Assistant Prof * : Seihou Jinnail Staff : Tsuyoshi Matsuzaki Staff : Hitoshi Haneoka Staff : Yosuke Murakami Staff : Tsunayoshi Takehara Staff : Takuya Yamanaka Staff : Nao Eguchi † Staff : Hsieh, Ming-chun Assistant Administrative Staff : Tomoko Wada * Concurrent post [†] Center for Scientific Instrument Renovation and Monufactuaring Support



Murakami Takehara Yamanaka



Eguchi

-2 -

Hsieh



Wada

Insutrument List

Instrument Name	Model (Maker) Name	Room No.	Staff	
	Avance III 600 (BRUKER)	104	71	
	Avance III 700 (BRUKER)	105	Zhou Haneoka	
Nuclear Magnetic Resonance (NMR)	ECA-600 (JEOL)	106	Hundoku	
	ECS-400 (JEOL)	F428 ¹⁾	Yamashita	
	ECS-400 (JEOL)	F507 ¹⁾	Jinnai	
	JMS-700 (JEOL)	303		
	AccuTOF-DART (JEOL)	303	Asano	
	Ultraflex III (BRUKER)	304		
Mass Spectrometer (MS)	micrOTOF II (BRUKER)	304		
	Orbitrap XL (THERMO)	304	Matsuzaki	
	ITQ1100 (THERMO)	304		
Infrared Spectrophotometer (IR)	FT/IR4100 (JASCO)			
	React-IR45m (METTLER)		Suzuki	
Ultraviolet-visible Spectrophotometer (UV)	V-770 (JASCO)	302		
Polarimeter	P-2300 (JASCO)		Haneoka	
Circular Dichroism spectrometer	J-1500 (JASCO)			
Inductively Coupled Plasma Spectrometer (ICP)	ICPS-8100 (SIMADZU)	301	Eguchi Haneoka	
Time of Flight Secondary Ion Mass Spectrometry	M6 (IONTOF)	102	Eguchi	
Electron Prove Microanalyzer (EPMA)	JXA-8800R (JEOL)	102	Eguch	
Scanning Electron Microscope (FE-SEM)	JSM-F100 (JEOL)	S107 ²⁾	Murakami	
Transmission Electory Missesson (TEM)	JEM-ARM200F (JEOL)	S104 ²⁾	Nishino	
Transmission Electron Microscope (TEM)	JEM-2100 (JEOL)	F192 ¹⁾	Murakami	
Forcused Ion Beam	FB-2100 (JEOL)	S113 ²⁾	Murakami	
Nanoscale Hybrid Microscope	VN-8010 (Keyence)	S107 ²⁾	Murakami	
Fluorescent X-ray Diffractometer (XRF)	ZSX100e (RIGAKU)	101	Goto	
X-ray photoelectron spectroscopy (XPS)	JPS-9010MC (JEOL)	101	Haneoka	
X-ray Diffractometer (XRD)	SmartLab (RIGAKU)	101	Takehara	
X-ray Diffractometer for Single Crystals	XtaLAB PRO (RIGAKU)	203	Takehara	
CHN Elemental Analyzer	2400 (PERKIN-ELMER) JM10 (J-SCIENCE)	302	Matsuzaki	
Thermogravimetric/differential temperature analyzer	TG8120 (RIGAKU)	302	Takehara	
Differential Scanning Calorimeter	DSC8270 (RIGAKU)	302	Takehara	



Elemental Analyzer EA



¹⁾ Installed in the 1st Building of ISIR. ²⁾ Installed in the 2nd Building of ISIR.

X-ray Diffractometer for Single Crystals SC-XRD





This single crystal X-ray structure analyzer is equipped with a rotating anti-cathode type high-intensity X-ray source and a 1-photon detection type hybrid pixel detector. The X-ray source can be selected from two sources, Mo and Cu. The detector with zero noise and a wide dynamic range can detect weak and strong reflections at the same time with a high S/N, and also enables high-speed shutterless measurement. Not only for metal complexes and low molecular weight compounds, but also for protein crystals are applicable.

Ar	tipy	rine	Acetanilide		4-Nitro	anili	Phe	nacetir	0	holestero	標準	試料一覧
	No.	P区分	試料名	SP	試料量	気圧	89914	いかが	Nシウィナル	н	С	N
1	1	3 1'-7				1005.3	2625	3464	3577			
2	1.	1 拾焼				1005.2	18209	30888	13216			
3		3 11-2				1005.2	2779	3523	3688			
4	2 .	4 括規			1092	1005.1	16240	25816	11497			
5		3 11-2				1005.0	2776	3520	3676			
6	3	4 標準	Acetanilide	1	1089	1005.0	16285	25769	11468	0.53820	3.52907	1.48780
7		3 11-2				1004.8	2766	3530	3688			
8	- 4	標準	Acetanilide	1	1064	1004.8	16069	25279	11341	0.53609	3.52692	1.47766
9		3 1-2				1004.9	2849	3536	3680			
0	5	4 標準	Acetanilide	1	997	1004.8	15320	23901	10841	0.53337	3.52678	1.48075
11		8 12				1005.0	2795	3530	3699			
12	6	4 未知	4-Nitroaniline	18	1144	1004.9	12267	20657	19697	4.45	52.21	20.25
13		8 12				1005.0	2170	3531	3741			
14		未知	4-Fluorobenzoic	25	1094	1004.9	10315	22298	3737	3.71	59.85	0.00
5		8 4 4				1005.0	2763	3510	3682			
6	8	1 未知	C6		1008	1004.8	12397	18841	3783	5.12	52.94	0.00
17		3 1 - 2				1004.8	2767	3526	3693			
18	9 /	未知	C12		1091	1004.7	14521	21297	3825	5.78	56.65	0.00
19		5 7 - 6	500.00			1004.7	2788	3533	3/33	1.50	00.17	
20	10	「不知	FODUN		1028	1004.9	11569	24006	12032	4.59	08.47	11.73
21		1.00	170.00		0.00	1004.9	2817	3516	3678	4.10	05.50	10.44
22	-11	木刀	ATECN		989	1004.7	10488	22U94	10/89	4.18	05.52	10.44

Elemental analysis is a classical and important quantitative analysis and purity test, which determines a weight percentage of elements composing pure samples including organic compounds by combustive and oxidative decomposing the pure samples. The analysis is mainly applied to confirmation of synthetic chemicals. Measurable elements are carbon, hydrogen and nitrogen. Ash content is also quantifiable.

Fluorescent X-ray Diffractometer XRF





A vacuum tube (Rh) of 4kW used as an X-ray source in the XRF diffracts fluorescent X-rays generated from samples with 6 analyzing crystals (LiF, PET, Ge, RX-25, RX-75 and TAP). Two detectors (scintillation counter and gas flow proportional counter) in the WDX enable highly sensitive qualitative and quantitative analyses of various elements (from B to U). The XRF corresponds to powder, bulk and liquid samples. Continuous measurement (turret-type measurement) enables simultaneous measurement of up to 12 samples. The XRF has SQX program performing semi-quantification by FP method without standard samples through qualitative analysis results, and EZ scanning mode to perform SQX analysis.

Nuclear Magnetic Resonance 700MHz NMR



Nuclear Magnetic Resonance 600MHz NMR



X-ray Diffractometer XRD



This multipurpose diffractometer measures scattered and diffracted X-rays generated from solid samples by irradiating strong X-rays (45kV, 200mA, Cu), which exhibits remarkable effect on film samples. The diffractometer is applicable to in-plane, film thickness, orientation, particle and void size distribution, and rocking curve measurements. Following guidance function allows users including beginners to easily obtain data. Choosing an incident X-ray source from Ge double crystals or Ge quadruple crystals enables high resolution measurement. The diffractometer has a scintillation detector, a one-dimensional detector enabling fast measurement within several minutes, and ICDD (Ver2. 1102) .





AVANCE III 700, NMR spectrometer of Bruker BioSpin, is an advanced digital NMR device at the high level. Combination of cryoprobes enables supersensitive NMR measurement. The spectrometer has supersensitive triple resonance probes for ¹H, ¹³C and ¹⁵N, which optimizes ¹H and ¹³C nuclear measurements, enables high sensitive and rapid 2D and 3D measurements, and remarkably shortens measurement time. The spectrometer has automatic tuning/matching functions and enables fully automatic measurement with rapid and high resolution by simultaneously using sample changers, which is applicable to the various fields including pharmaceutics, biotechnology, chemistry, material science etc.



AVANCE III 600WB, solid NMR spectrometer of Bruker BioSpin, has a wide bore magnet, which enables measurement at -140 \sim +150°C. Combination of 4mm CPMAS probes and superhigh speed rotary 1.3mm CPMAS probes enables proton, multinuclear and 2D NMR measurements difficult for conventional spectrometers. The solid NMR spectrometer at the high level is applicable to the various fields including material science, life science and determination of solid catalysts.

Nuclear Magnetic Resonance 600MHz NMR





JNM-ECA 600 (JEOL), is an FT-NMR spectrometer using the advance digital technology and high frequency. Automatic tuning and matching enables gradient shim. The spectrometer easily provides NMR spectra with high reproducibility and quality. The water signal elimination and differential spectrum measurements are also easily measurable. Low frequency probes are also equipped. The spectrometer is applicable to rhodium element and has an MICCS instrument enabling reaction tracing measurement.



Transmission Electron Microscope 3D-TEM







This mass spectrometer has a dedicated DART (Direct Analysis in Real Time) ion source attached to a high-resolution time-of-flight mass spectrometer. The DART ion source is a novel ion source and applicable to samples under atmospheric pressure and at ground potential, which enables contactless and rapid analysis. The spectrometer is applicable to gases, liquids and solids. Instantaneous measurability of samples without any pre-treatments of the surface of the samples is characteristic of the spectrometer. The spectrometer is effective for screening and high-throughput analysis.



Scanning Electron Microscope FE-SEM



The scanning electron microscope (JSM-F100) can set the acceleration voltage of the electron beam from 10V to 30kV, and can handle samples that are vulnerable to heat damage and samples of insulation. It also has an option to observe at a lower vacuum than usual. In addition, the performance of elemental analysis using EDS has been greatly improved. Since this JSM-F100 can acquire data while switching between observation and analysis with a simple operation, it is possible to analyze the sample surface efficiently.



LaB₆ electron gun-equipped transmission electron microscope (200 kV) (JEM-2100) enables high resolution and high contrast observation, which is suitable for observation of biological samples. CCD camera enables to photography TEM images as digital data. High-tilt holder enables to set the tilt angle of samples to up to $\pm 80^{\circ}$. TEM tomography system enables to automatically acquire continuously tilted images. 3D reconstruction of samples and visualization of 3D structures are performable by PC.

Electron Probe Microanalyzer EPMA





EPMA (Electron Probe Micro-Analyzer) finely narrows generated electron beams from an W electron gun to tens nanometers and accelerates the beams to up to 40 kV to irradiate the surface of solid samples. Determination of elements composing the samples and quantitative analysis of the samples are performable based on the wavelength of characteristic X-rays generated from the samples. Mapping measurement indicating element distribution states and line analysis are also performable. The EPMA has measurability of B \sim U, using 8 analyzing crystals and 4 detectors. Measurability of cathode luminescence is characteristic of this EPMA.

Transmission Electron Microscope FE-TEM





This microscope is a Schottky emission electron gun equipped with a ZrO/W emitter as a cathode, which has higher brightness and a smaller electron source than a thermionic electron gun and affords higher current stability and larger probe current than a normal field emission electron gun. Negative spherical aberration coefficients obtained by an attached spherical aberration corrector (Cs corrector) counteract positive spherical aberration coefficients obtained by a condenser lens having a symmetrical axis of a magnetic field, which enables to acquire smaller and higher intensity probes and to analyze elements with higher resolution. Detecting electrons transmitted through film samples acquires STEM images affording scattering/absorption, diffraction and phase contrasts.

Mass Spectrometer FT-MS



Mass Spectrometer MALDI-TOF-MS





This hybrid electric field Fourier-transform mass spectrometer (FT-MS) has Orbitrap and high speed and high sensitive LTQ XL equipped with linear ion trap. The spectrometer has high performances with high resolution (100,000 of resolution) and accuracy (3 ppm), which enables not only structural analysis of low molecules but also identification of complicated proteins by multistep MS/MS. Accelerating scanning speed and shortening a cycle time enable a very short measurement time of a few to 5 minutes per sample. ESI, APCI and APPI ionizations are selectable and a wide range of samples is measurable at the highest level.

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Ultraflex III, developed by BRUKER, has smartbeam as a laser beam to largely enhance sensitivity and resolution. The focal point size (10µm~80µm) of the laser is controllable by PC. Applying minimal laser focuses to samples enables to scan sample regions with very high pixel resolution, very high sensitivity and very high resolution by MARDI imaging experimental instrument. The spectrometer exhibits a wide mass range of 1 - 500,000 and high resolution of 25,000 by using PAN (Panoramic) technology.

Mass Spectrometer CSI-MS

X-ray photoelectron spectroscopy XPS · UPS





This spectrometer has a cryogenic ion source (CryoSpray) with micrOTOF II (mass accuracy: 1-2 ppm, mass resolution: 16,500 and measurable mass range: 50~20,000 m/z), which enables CryoSpray-TOF-MS measurement and measurement under cooled ionization conditions. The spectrometer is suitable for samples with unstable at room temperature: organometallic complexes, supramolecular complexes and reactive intermediates etc.



Time of Flight Secondary Ion Mass Spectrometry TOF-SIMS



X-ray photoelectron spectroscopy (XPS), one of the surface analysis instruments, is utilized for the analysis of wide materials such as metal, a semiconductor, organics and ceramics. This method supplies the chemical states of a wide range of elements. About 6nm depth from the surface and several cm² area of the sample can be analyzed. Ion etching enables a depth direction analysis. Ultraviolet photoelectric spectroscopy (UPS) is also applicable.



Inductively Coupled Plasma Spectrometer ICP



This top-class ICP emission spectrometer has 2 sequential scanning spectrometers, which exhibits both high resolution and high speed. Plasma energy from the spectrometer excites component elements in samples. The spectrometer measures emission rays of the excited elements transiting to the lower energy levels. The spectrometer enables a wide range of highly accurate analyses including ppb-level analysis of trace elements in a solution and high concentration analysis such as composition analysis. Rapid simultaneous quantitative analysis of multiple elements is also performable.



TOF-SIMS irradiates the sample surface with an accelerated ion beam and detects the generated secondary ions with a time-of-flight mass spectrometer. Unlike dynamic SIMS, TOF-SIMS can perform surface analysis using pulsed ions. Since this device is equipped with a spatter gun, it is possible to perform depth direction analysis while spattering materials. It is possible to obtain MS spectra and depth profiles for all elements and molecules. In addition, since it has excellent spatial resolution compared to other surface analysis, it is possible to analyze minute areas and perform MS imaging.