Comprehensive Analysis Center



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URL http://www.sanken.osaka-u.ac.jp/labs/cac/

The Institute of Scientific and Industrial Research 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), 3 technical staffs, 3 part-time staffs, 5 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, physical organic 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 Nanotechnology Research Building 4 Comprehensive Analysis Center (CAC) (5) Administration Building (6) Factory Building (7) Accelerator and Quantum Beam Laboratory, Scientific and Industrial Nanotechnology Center (8) Accelerator and Quantum Beam Laboratory, Scientific and Industrial Nanotechnology Center (Linac Building) (9) Electronic Processing Laboratory, Scientific and Industrial Nanotechnology Center (10) Kusumoto-Kaikan Hall 1 Incubation Building 2 Field Emission High Resolution Electron Microscope Room (S-102, I-103, S-105 and S-107) (B) Chemistry Laboratory (244 and 246) (I) Electron Microscope Room for Biology (192 and 194) (I) Nuclear Magnetic Resonance Room (428 and 507)

(via JR Ibaraki Station)

(via Banpaku Kinen Koen Station)

Floor Map













T. Matsuzaki









H. Yoshida

H. Haneoka

Staff : Nao Eguchi, Center for Scientific Instrument Renovation and Monufactuaring Support



D. Zhou



K. Asano





M.Nishino



T. Goto



T. Takehara



T. Ishibashi



N. Eguchi



T. Tanaka



E. Tani

Insutrument List

Instrument Name	Model (Maker) Name	Room No.	Staff	
	Avance III 600 (BRUKER)	104		
	Avance III 700 (BRUKER)	105	D.Zhou H.Haneoka	
Nuclear Magnetic Resonance (NMR)	ECA-600 (JEOL)	106	п.панеока	
	ECS-400 (JEOL)	F428 ^{*1)}	K. Takenaka	
	ECS-400 (JEOL)	F507 ^{*1)}	M. Sako	
	JMS-700 (JEOL)	303		
	JMS-600H (JEOL)	303		
	AccuTOF-DART (JEOL)	303	K. Asano	
Mass Spectrometer (MS)	Ultraflex III (BRUKER)	304		
	micrOTOF II (BRUKER)	304	T. Matsuzaki	
	Orbitrap XL (THERMO)	304		
	ITQ1100 (THERMO)	304		
Infrared Spectrophotometer (IR)	FT/IR4100 (JASCO)	302		
	React-IR45m (METTLER)		T. Suzuki H. Haneoka	
Ultraviolet-visible Spectrophotometer (UV)	V-770 (JASCO)			
Polarimeter	P-2300 (JASCO)			
Inductively Coupled Plasma Spectrometer (ICP)	ICPS-8100 (SIMADZU)	301	N. Eguchi H. Haneoka	
Secondary Ion Mass Spectrometer (SIMS)	SIMS4100 (ATOMIKA)	102	T. Suzuki	
X-ray Microanalyzer (EPMA)	JXA-8800R (JEOL)	102	Y.Murakami	
Scanning Electron Microscope (FE-SEM)	JSM-6330F (JEOL)	S107 ^{*2)}	N.Eguchi	
	JEM-ARM200F (JEOL)	S104*2)	Y.Murakami	
Transmission Electron Microscope (TEM)	JEM-2100 (JEOL)	F192 ^{*1)}	T. Ishibashi M.Nishino H. Yoshida	
X-ray Diffractometer (XRD)	SmartLab (RIGAKU)	101	T. Takehara	
Fluorescent X-ray Diffractometer (XRF)	ZSX100e (RIGAKU)	101	T. Goto	
X-ray photoelectron spectroscopy(XPS)	JPS-9010MX (JEOL)	101	H. Haneoka	
X-ray Diffractometer for Single Crystals	AFC-7RCCD,AFC-7R4CXD (RIGAKU) FR-E-IP,FR-E-AXIS IV (RIGAKU)	203	T. Takehara	
CHN Element Analyzer	2400 (PERKIN-ELMER) JM10 (J-SCIENCE)	302	T. Matsuzaki	
Differential Themogravimetric Analyzer	TG8120 (RIGAKU)	302	T. Takehara	
Differential Scanning Calorimeter	DSC8270 (RIGAKU)	302	T. Takehara	
Nanoscale Hybrid Microscope	VN-8010 (Kryence)	S107 ^{*2)}	Y.Murakami	

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

X-ray Diffractometer for Single Crystals IP



Element Analyzer CHN



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This diffractometer using Cu as an X-ray source enables to determine the absolute structure of organic low molecules, which is also suitable for crystals having a large lattice constant. High brightness mirrors attached to the diffractometer enables to secure a 10 or more time higher bright X-ray source than an X-ray source of rotary anticathode X-ray generator (18kW) and to collect data of fine crystals. The diffractometer has a curved IP detector unit having a resolution of more than 150° in terms of 2θ and RAPID-AUTO as software.

Antipyrine Acetanilide		4-Nitro		anili Ph		enacetin		holestero	種類	標準試料一覧		
	No.P 🗵		試料名	SP	試料量			いっかった		н	С	N
1	B ^*					1005.3	2625	3464	3577			
2	1 4 捨					1005.2	18209	30888	13216			
3	B A*					1005.2	2779	3523	3688			
1	2 A 捨				1092	1005.1	16240	25816	11497			
	B \\'					1005.0	2776	3520	3676			
			Acetanilide	- 1	1089	1005.0	16285	25769	11468	0.53820	3.52907	1.4878
	B ^'					1004.8	2766	3530	3688			
			Acetanilide	1		1004.8	16069	25279	11341	0.53609	3.52692	1.4776
	B ^'					1004.9	2849	3536	3680			
0			Acetanilide	1		1004.8	15320	23901	10841	0.53337	3.52678	1.4807
1	B ^*					1005.0	2795	3530	3699			
2			4-Nitroaniline	18		1004.9	12267	20657	19697	4.45	52.21	20.2
3	B ^*					1005.0	2770	3531	3741			
4			4-Fluorobenzoic	25	1094	1004.9	10315	22298	3737	3.71	59.85	0.0
5	B ^'					1005.0	2763	3510	3685			
6	8 4 未		C6			1004.8	12397	18841	3783	5.12	52.94	0.0
7	B 11					1004.8	2767	3526	3693			
B	9 A 未		012			1004.7	14521	21297	3825	5.78	58.65	0.0
9	B ^*					1004.7	2788	3533	3733			
0			FBBCN		1028	1004.9	11569	24006	12032	4.59	69.47	11.7
1	B ^*					1004.9	2817	3516	3678			
2	11 A 未		ATBON			1004.7	10488	22094	10789	4.18	65.52	10.4
3	B ^*	-7				1004.7	2786	3530	3662			

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 and determination of the structure of natural compounds. Stable and highly pure samples afford highly accurately analytical values. Measurable elements are carbon, hydrogen and nitrogen. Ash content is also quantifiable.

X-ray Diffractometer XRD

Fluorescent X-ray Diffractometer ZSX-100e





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).

Nuclear Magnetic Resonance 600MHzNMR (solid)



This wavelength dispersion X-ray diffractometer (WDX) was transferred to CAC, through a courtesy of prof. Tohru SEKINO, ISIR. A vacuum tube (Rh) of 4kW used as an X-ray source in the WDX diffracts fluorescent X-rays generated from samples to 5 analyzing crystals (LiF1, PET, Ge, RX-25 and RX-75). Two detectors (scintillation counter and gas flow proportional counter) in the WDX enable highly sensitive qualitative and quantitative analyses of various elements (from F to U). The WDX corresponds to powder, bulk and liquid samples. Continuous measurement (turret-type measurement) enables simultaneous measurement of up to 12 samples. The WDX has SQX program performing semi-quantification by FP method without standard samples through qualitative analysis results, and EZ scanning mode allowing users including inexperienced users to perform SQX analysis without instruction manuals.



Nuclear Magnetic Resonance 700MHzNMR





AVANCE III 700, the latest NMR spectrometer of Bruker BioSpin, is an advanced digital NMR device at the highest 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, the latest solid NMR spectrometer of Bruker BioSpin, has a wide bore magnet, which enables measurement at $-140 \sim +150$ °C. Combination of 4-mm CPMAS probes and superhigh speed rotary 1.3-mm CPMAS probes enables proton, multinuclear and 2D NMR measurements difficult for conventional spectrometers. The solid NMR spectrometer at the highest level is applicable to the various fields including material science, life science etc. and determination of solid catalysts.

Nuclear Magnetic Resonance 600MHzNMR

Mass Spectrometer FAB-MS (JMS-700, JMS-600H)





JNM-ECA 600, developed by Japan Electron Optics Laboratory (JEOL), is an FT-NMR spectrometer using the advance digital technology and high frequency. Automatic tuning and matching of JNM-ECA 600 MHz NMR 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 down to rhodium and has an MICCS instrument enabling reaction tracing measurement.



Scanning Electron Microscope FE-SEM+EDS

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This spectrometer has a virtual-imaged stereoscopic and double focusing ion optical system, which enables easy operations including setting-up, ion source tuning, resolution adjustment, data measurement and data processing. The spectrometer as a FAB ionization instrument is specially applied to high resolution measurement of hardly volatile samples.





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.



Scanning electron microscope (SEM) for analysis (JSM-6335F) has resolution of ~1 nm (15 kV) and acceleration voltage of 1~30 kV. A characteristic X-ray detector in an element analyzer in the SEM is required no liquid nitrogen supply and cooled by a Peltier element. The SEM has detectability of Be~U and energy resolution of 138 eV or less. Appropriately using carbon coater or osmium coater according to purposes enables to acquire clear images. Attending a lecture on FE-SEM+EDS are reguired to use.

X-ray Microanalyzer EPMA





EPMA (Electron Probe Micro-Analyzer) finely narrows generated electron beams from an LaB₆ 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 linear analysis are also performable. The EPMA has measurability of B \sim U, 8 analyzing crystals and 4 detectors. Measurability of cathode luminescence is characteristic of the EPMA. The EPMA exhibits remarkable effect on evaluation of metals, minerals, ceramics, semiconductor materials etc.

Transmission Electron Microscope JEM-ARM200F





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 field emission electron gun by heating at the high temperature of 1800 K. 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 DART-SVP-MS



Mass Spectrometer FT-MS



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This mass spectrometer has a dedicated DART (Direct Analysis in Real Time) ion source attached to a high-resolution time-of-flight mass spectrometer developed by JEOL. 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. Combination of AccuTOF and DART enables accurate estimation of element composition based on precise mass measurement. The spectrometer is applicable to gases, liquids and solids. Instantaneous measurability of samples without any pre-treatments (wiping, solvent extraction etc.) of the surface of the samples is characteristic of the spectrometer. The spectrometer is effective for screening and high-throughput analysis.



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.

Mass Spectrometer MALDI-MS





Ultraflex III, developed by BRUKER, has smartbeam as a laser beam to largely enhance sensitivity and resolution. The focal point size $(10\mu$ m~ 80μ m) of the laser is controllable by PC. Applying minimal laser focuses to samples enables to scan tissue 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





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) developed by BRUKER, which enables CryoSpray-ESI-TOF-MS measurement and measurement under cooled ionization conditions. The spectrometer is suitable for samples with unstable structures at room temperature: organometallic complexes, supramolecular complexes and reactive intermediates etc.

Inductively Coupled Plasma Spectrometer ICP



X-ray photoelectron spectroscopy XPS · UPS





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.



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.