

Chapter 7 Analytical Research Core for Advanced Materials

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1. Introduction

This document reports on the twelve months of activities at the Analytical Research Core for Advanced Materials from January to December 2004, subsequent to the previous report that covered the two-year period from January 2002 to December 2003.

2. Organization

The Research Core's organizational structure and its constituent members are shown in Table 1: the staff for analytical work consists of 11 members, who belong to the Technical Services Office's materials test team or the Evaluation Office's analysis team within the Technical Services Division, with a decrease in the total head count over 2003 of 1.5 persons. There was a reduction of one assistant and one technical staff, with one technical assistant on a six-month contract now upgraded to a full year's employment contract and therefore counted as an increase of 0.5.

3. Overview of Activities in 2004

Services at the Core include chemical analysis – and structural analysis by transmission electron microscopy – of various materials being studied and developed at our own laboratory as well as those in collaboration with others. Following is a status report on the analysis services made available on request.

3.1 Analysis Services

(1) Chemical Analysis

The numbers of analyzed elements grouped by the test process are shown along with the percentage distribution in Table 1. In chemical analysis, samples are dissolved in acid and then the quantity of each element is determined, except when determining C, S, O, N and H. Sample dissolution is an essential process for high-accuracy analyzing devices, most of which require test samples to be prepared in the form of a liquid solution. The chemical analysis must be conducted by the same personnel throughout the process from acid dissolution to the conclusion of the

analysis. Researchers should choose the most appropriate test processes for each material, after assessing its component elements and their composition ratios. The total number of elements analyzed turned out to be approximately 30% fewer than for the previously reported years of 2002 and 2003. This may be primarily due to: (1) an increase in the number of requests for sample testing that require a more time-consuming chemical dissolution process; and (2) a decrease in the number of staff available for analytical work.

Table 1. Methods of chemical analysis and numbers of analyzed element

Affiliation of requester	Numbers of analyzed element						Total
	ICP-OES	C, S	O, N	H	Spectrophotometry	Gravimetric and Volumetric	
IMR	632	479	392	107	9	5	1320
External parties	343	18	45	2	0	5	413
(Subtotal)	971	197	437	109	9	10	1733
(Ratio)	56.0 %	11.4 %	25.2 %	6.3 %	0.5 %	0.6 %	100 %

From the percentage distribution of the analyzed elements grouped by the process adopted, it can be observed that the most frequently-required process is Inductively Coupled Plasma (ICP) - Optical Emission Spectrometry, which exceeds 50% of the total. The group of gas components, including carbon, sulfur, oxygen, nitrogen and hydrogen, accounts for slightly more than 40%. The trend featuring those two groups appears to be consistent with the previously-reported period from 2003 through 2004.

Major external parties requesting our analysis services include the Engineering Department and the Science Department of the University's Graduate School, altogether accounting for nearly 25% of all our service operations in terms of the number of analyzed elements, a 10 to 15% increase over the periods for 2002 and 2003. Their actual contribution may be even larger, since it is not possible to determine the actual number of requests including those apparently brought in by the Institute's internal researchers working on collaborative programs.

(2) Transmission Electron Microscopy

Transmission electron microscopes are generally designed so that researchers can operate the equipment themselves as they work to obtain the required sample data. An ion milling device, used to form test samples as thin slices, is a crucial part of the equipment for that operation.

The operating hours for the transmission electron microscopy devices are shown in Table 2. While the total length of time remains almost the same as for the 2002 and 2003 periods, the proportion of our services allocated to external parties is growing and is almost equivalent to the proportion for the Institute's internal use. Such external users include other organizations in the

University, including the Engineering Department and the Science Department of the Graduate School, the Institute of Multidisciplinary Research for Advanced Materials and the Center for Interdisciplinary Research.

Table 2. Time for operation of transmission electron microscopy (TEM)

Affiliation of requester	Time for operation、 Hour/Year					
	TEM	2000 EX	TEM	2000 EX-2	Ion milling device	Total
IMR		283		541	4846	5670
External parties		247		397	374	1018
(Subtotal)		530		938	5220	6688

3.2 Services for Internal and External Parties Associated with Chemical Analysis and Transmission Electron Microscopic Analysis

(1) Activities Related to Test Method Development and Experimental Evaluation

Publications on newly-developed analytical methods and evaluation experiments required for implementing our chemical testing services on request, as well as the release of data on ingenious devices invented for transmission electron microscopic observation, are shown in the tables below.

See Table 3 for the number of papers appearing in domestic and international academic journals and Table 4 for the number of inventions verbally presented during the period. Table 5 shows the numbers of technical lectures and sessions given by our staffers at relevant seminars hosted by the Japan Society for Analytical Chemistry (JSAC) and Table 6 likewise shows the status of participations in collaborative research groups related to analytical chemistry.

Table 3. Numbers of papers appeared in academic journal

Academic journal	Number of paper
Material Transactions, Vol.45	1
Materiaux & Techniques-No. HORS SERIE-December 2003	3
Appl. Phys. Letters, Vol.84 & Vol.85	2
Bunseki Kagaku, Volume 53	3
Technical Research Report of Technical Services Division in IMR, Tohoku University, Volume 21	1
Total	10

Table 4. Numbers of oral presentation on international and domestic analytical meeting

Academic Society	Number
Internat. Confer. UHPM-2004 (December, NIST, USA)	3
Asian Internat. Symposium on Instrumental Analysis of Various Matyerials, AISIA (July, Sendai)	1
Drip X (September-October, Batz sur mer, France)	2
Meeting of The Japan Society for Analytical Chemistry, No.53 (September, Chiba)	2
Meeting of The Japan Institute of Metals, No.134 (March, Tokyo)	2
The Iron and Steel Institute of Japan Meeting, No.147 (March, Tokyo)	2
Others	4
Total	16

Table 5. Technical lecture of seminar for chemical analysis

Seminar	Number
Lecturer: Chemical Analysis Seminar of Metals and Alloys in The Japan Society for Analytical Chemistry	1
Lecturer: Chemical Analysis Seminar of Ceramics, Ores and Cement in The Japan Society for Analytical Chemistry in The Japan Society for Analytical Chemistry	1
Lecturer: Fundamental Technical Seminar for Analytical Chemistry in The Japan Society for Analytical Chemistry	3
Experimental: Fundamental technique for chemical analysis in Tohoku Branch, The Japan Society for Analytical Chemistry	1
Total	6

Table 6. Participation in collaborative research groups related to analytical chemistry

Commission	Number
Commission for Japanese Iron and Steel Certified Reference in The Japan Iron and Steel Federation	1
Sectional meeting for Analytical Technique in The Iron and Steel Institute of Japan	1
Workshop for High Precise Analysis with ET-AAS	1
Commission for Chemical Analysis of Impurity Elements in Scrapped Steel in The Iron and Steel Institute of Japan	1
Forum for Development of Advanced Chemical Analysis in The Iron and Steel Institute of Japan	1
Total	5

(2) Committee Membership in Academic Societies

Table 7 shows activities of committee member in research groups hosted by academic societies of several kinds focusing on analytical chemistry.

Table 7. Committee Membership in Academic Societies

Name of Academic Society	Number	Note
Commission for Japanese Iron and Steel Certified Reference in The Japan Iron and Steel Federation	1	Since 1984/4
Sectional meeting for Analytical Technique in The Iron and Steel Institute of Japan	1	Since 1995/6
Workshop for High Precise Analysis with ET-AAS	1	Since 2000/10
Commission for Chemical Analysis of Impurity Elements in Scrapped Steel in The Iron and Steel Institute of Japan	1	Since 2001/5
19-th Committee in Japan Society for the Promotion of Science	1	Since 1991/4
Fundamental Technical Seminar for Analytical Chemistry in The Japan Society for Analytical Chemistry	1	Since 2002/4
Committee for analytical equipment and operation	1	Since 2003/4
Total	7	

(3) Acceptance of Research Staff

A privately-endowed research project at the Laboratory for Advanced High Temperature Nano-Metallurgy will require chemical testing services for materials studies and is accepting a research staff in that area.

(4) Collaborative Research in the Institute's Laboratories

A collaborative program for studies on grain boundary structures and properties is underway using transmission electron microscopes.

(5) Visitors on Tour

The numbers of visitors touring the Core are 30 persons of 6 parties (Domestic parties 5 and international parties 1).

3. Assignments, Qualifications, Recognitions and Training

(1) Qualifications for Assignments

In order to carry inventories of chemicals to be used for research experiments, the person in question must be registered as a "Safety Manager for Hazardous Materials". The Core has appointed a certified staffer to this assignment.

(2) Academic Titles

One of the Core's staff members received a doctor's degree from the University's Graduate School of Environmental Studies in February 2004. This was a result of the person's cumulative achievements in experiments to develop, improve and review chemical testing methods for the purpose of fulfilling analytical services on request, which were recognized through presentations at academic societies and publications in relevant journals in the form of data or papers.

(3) Recognitions

A staffer who acquired a certificate required for safe operation in the Institute was commended by management.

(4) Training

An Analytical Core staff member was trained on exhaust evolved by chemical operation in a draft.

4. Others

The Core's staff members of 17 persons participate in the 13 committees set up within the University, the Institute for Materials Research (IMR) and the Technical Services Department in which Core staffers are members.