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# The Materials Characterization Central Laboratory: An Open-Ended Laboratory Program for Fourth-Year Undergraduate and Graduate Students

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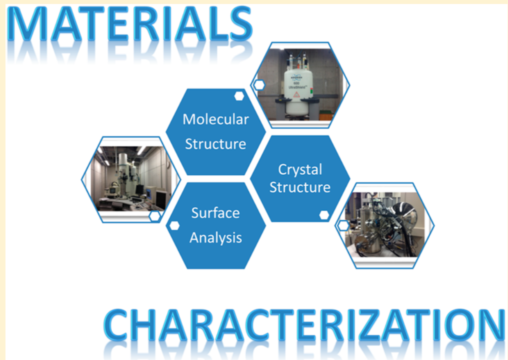
Supporting Information

**ABSTRACT:** Teaching materials characterization to support student research projects requires a systematic educational approach, because characterization involves a combination of analysis instruments. As analytical instruments are expensive, it is difficult to provide multiple sets simultaneously. An effective educational program allows students to select their own research materials to characterize and apply their personal strategies of instrumental analysis. These strategies are designed around the purposes of the analytical instruments, e.g., molecular structure analysis, crystal structure analysis, morphology assessment, surface analysis, elemental analysis, and thermal analysis. An open-ended laboratory complements this educational purpose. Here, we report on an open-ended laboratory program for fourth-year undergraduate and graduate students at the Materials Characterization Central Laboratory at Waseda University (Tokyo, Japan). The goals of our open-ended laboratory program are to enable students to (1) conduct instrumental analysis, (2) operate analytical instruments, and (3) interpret their data. A team led by a supervisor and laboratory staff offers students a flexible program. This flexibility can be applied to various research fields, such as macromolecular chemistry, inorganic chemistry, organic chemistry, physical chemistry, electrochemistry, physics, catalyst chemistry, biomaterials science, and chemical engineering. These diverse research fields demonstrate the feasibility of applying our open-ended laboratory program to student research projects.




**KEYWORDS:** *Graduate Education/Research, Upper-Division Undergraduate, Analytical Chemistry, Laboratory Instruction, Laboratory Equipment/Apparatus, Hands-On Learning*

## INTRODUCTION

Instrumental analytical chemistry is an important educational topic and offers many projects for upper-level undergraduate students.[1−4] These projects introduce modern instrumental analysis and further the development of student skills. One example is the Western Washington University Instrument Center, which introduces instrument training programs for new students.[5] Teaching materials characterization requires a systematic educational approach, as characterization requires a combination of analysis instruments. To be effective, a program should permit students to analyze their own research materials and conduct instrumental analysis by applying their personal strategies. Thus, an open-ended laboratory is best suited to this educational purpose, and allows students to advance their individual projects in an optimal way.

In this report, we introduce an open-ended analytical instrument laboratory, namely, the Materials Characterization Central Laboratory at Waseda University. The mission of the MCCL is to offer state-of-the-art analytical instrumentation and support for fourth-year undergraduate and graduate students. The goals of our open-ended laboratory program are to enable students to (1) conduct instrumental analysis, (2) operate analytical instruments, and (3) interpret their data. Our systematic team teaching may be a useful resource for similar analytical facilities.

## MATERIALS CHARACTERIZATION CENTRAL LABORATORY

The MCCL has been in operation since 1993 as part of the Graduate School of Science and Engineering at Waseda University (Tokyo, Japan). The MCCL is an analytical instrument laboratory designed to support the research and education of students. The MCCL currently houses the following analytical instruments (additional information about the instruments is available in the Supporting Information):

(1) Transmission electron microscope
(2) Scanning electron microscope
(3) X-ray photoelectron spectrometer

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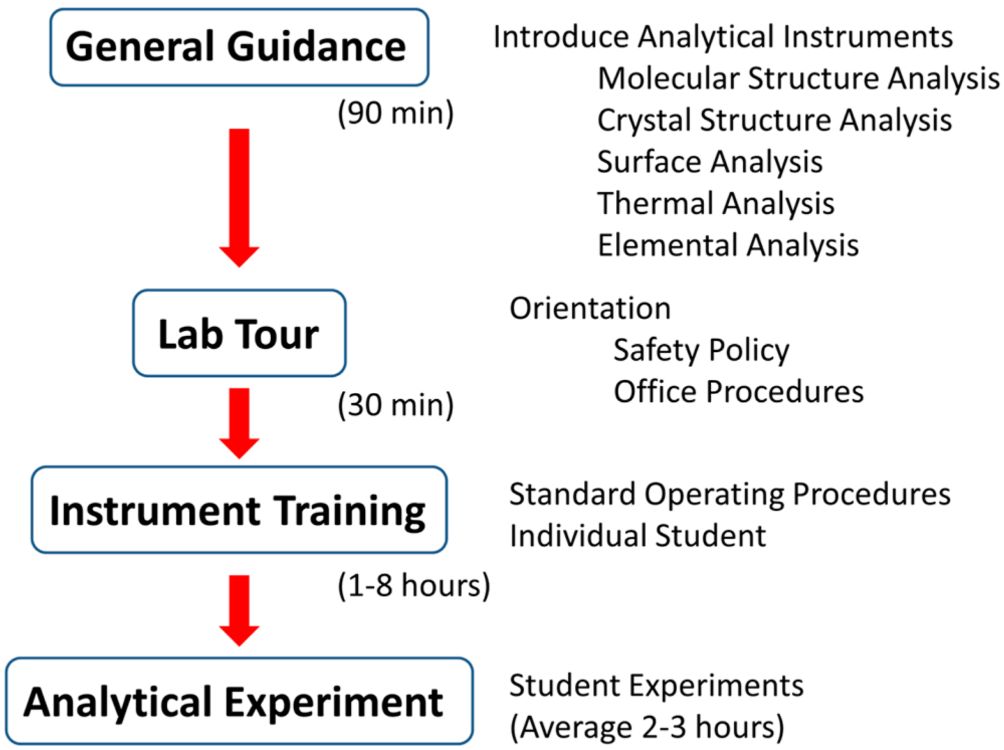


**Figure 1.** Laboratory workflow chart.

(4) Scanning probe microscope
(5) Nuclear magnetic resonance spectrometer
(6) Mass spectrometer
(7) Infrared spectrometer
(8) Raman spectrometer
(9) Electron spin resonance spectrometer
(10) X-ray diffractometer
(11) X-ray fluorescence spectrometer
(12) Glow discharge optical emission spectrometer
(13) Thermal analyzer
(14) Magnetic property measurement system

These analytical instruments are essential for the development, understanding, and study of new materials. Many research-oriented university instrument centers possess similar analytical instruments. Therefore, systematic education programs are an important and relevant topic. As an example, we report on our open-ended laboratory program.

## ■ OPEN-ENDED LABORATORY PROGRAM

To assist students in their studies, the MCCL provides an open-ended laboratory program. This program starts with a general overview (90 min) for new fourth-year undergraduate and graduate students. This overview briefly introduces the analytical instruments of the MCCL (Figure 1). The laboratory staff explain the purposes of the analytical instruments, e.g., for molecular structure analysis, crystal structure analysis, morphology, surface analysis, elemental analysis, and thermal analysis. At this stage, students register as MCCL users and attend a laboratory tour (30 min). The laboratory tour provides an orientation to the MCCL and includes a discussion of safety policies and office procedures. Instrument operation training is then arranged by laboratory staff.

During the instrument operation training session, which is provided to students singly or in pairs, the staff explain the standard operating procedures of relevant instruments. In many cases, students then select the sample related to their research project. The length of the training session depends on the analytical instruments covered and ranges from 1 to 8 h. When instrument operational training is completed, students can start their analytical experiments. Though analytical experiment time depends on the instrument, experimental conditions, and number of samples, the average experiment time is approximately 2−3 h. If students comply with the safety policies, they can use the MCCL as an open-ended laboratory. The MCCL currently accommodates as many as 300 fourth-year undergraduate student users and 500 graduate student users. We carry out approximately 628 instrument operation training sessions each year. While we cannot provide 628 instrument training programs to students simultaneously, our flexible and on-demand open-ended program can adjust experiment schedules easily, and streamline the instrument training and student experiment schedules (Table 1).

The MCCL provides technical seminars (Table 2), during which the staff review the hardware, theory, and application of the instruments. These seminars help students to understand the technical background of instrument operation.

At this stage, the supervisor may assign the students the next step of a research project. The supervisor discloses the purpose of the project, but not the analytical procedure. Therefore, the students must decide on a plan for their analytical experiments to complete the project. MCCL staff members play a supportive role. Because each material will be characterized using a combination of analysis instruments, staff members advise students on the different analytical methods and their limitations. Figure 2 illustrates a systematic education approach for teaching materials characterization at the MCCL. Each Friday, MCCL users and staff meet and discuss the next week's experimental plans. Each student decides which instrument to


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**Table 1. Frequency of Instrument Training and Student Experiment Sessions**

| Month | Number of Instrument Training Sessions | Number of Student Experiments | Student Experiments, h |
|---|---|---|---|
| April | 61 | 1,173 | 2,711 |
| May | 72 | 850 | 2,075 |
| June | 67 | 1,064 | 2,770 |
| July | 76 | 1,439 | 3,579 |
| August | 29 | 536 | 1,158 |
| September | 63 | 1,099 | 2,837 |
| October | 81 | 1,245 | 3,395 |
| November | 53 | 1,521 | 4,409 |
| December | 42 | 1,443 | 3,679 |
| January | 33 | 1,097 | 3,306 |
| February | 8 | 713 | 1,833 |
| March | 43 | 665 | 2,073 |
| Total | 628 | 12,845 | 33,825 |

**Table 2. Synopsis of Materials Characterization Seminars**

| Seminar Title | Instrument Abbreviation | Seminar Length, min |
|---|---|---|
| Transmission electron microscopy | TEM | 120 |
| Scanning electron microscopy | SEM | 90 |
| X-ray photoelectron spectroscopy | XPS | 180 |
| Scanning probe microscopy | SPM | 180 |
| Nuclear magnetic resonance spectroscopy | NMR | 120 |
| Mass spectrometry | MS | 90 |

use, and the schedule of the experiment. Laboratory staff provide help if customization of instrument settings is required.

After the analytical experiment and data analysis are complete, the student provides a report to the supervisor on the progress of the research project. This results in valuable feedback for the students. The supervisor can suggest elements of experimental design for future experiments. This systematic educational feedback provides students with interesting and thoughtful insights. This mode of hands-on experience allows students to devise their own strategies and further their instrumentation skills continuously.

The research projects are published[6−17] in many different fields, such as macromolecular chemistry,[6] inorganic chemistry,[7,8] organic chemistry,[9,10] physical chemistry,[11] electrochemistry,[12] physics,[13] catalyst chemistry,[14] biomaterials science,[15] and chemical engineering.[16,17] These diverse research fields demonstrate the feasibility of applying our open-ended laboratory program to student education.

## ■ EXAMPLE PROJECT

In this section, we demonstrate a specific example of one research project to help understand our open-ended program. K.A. is a student in the Graduate School of Applied Chemistry at Waseda University. His research project involves functional polymer design for an organic device. His supervisor, Prof. Takeo Suga, assigned him to synthesize functionalized block copolymers, which have radical/ionic sites. These functionalized block copolymers are designed for organic resistive memory. He entered the open-ended program at the MCCL to analyze the molecular structure of the functionalized block copolymers. At this stage, he has experimental background knowledge of nuclear magnetic resonance spectroscopy (NMR) and infrared spectroscopy (IR) from the analytical chemistry laboratory course he took in the second year of his undergraduate degree. He also has experimental background knowledge of differential scanning calorimetry (DSC) from the physical chemistry laboratory course he took in his third year of his undergraduate degree.

K.A. required information on the concentration of radical sites and radical/ionic ratio in the functionalized block

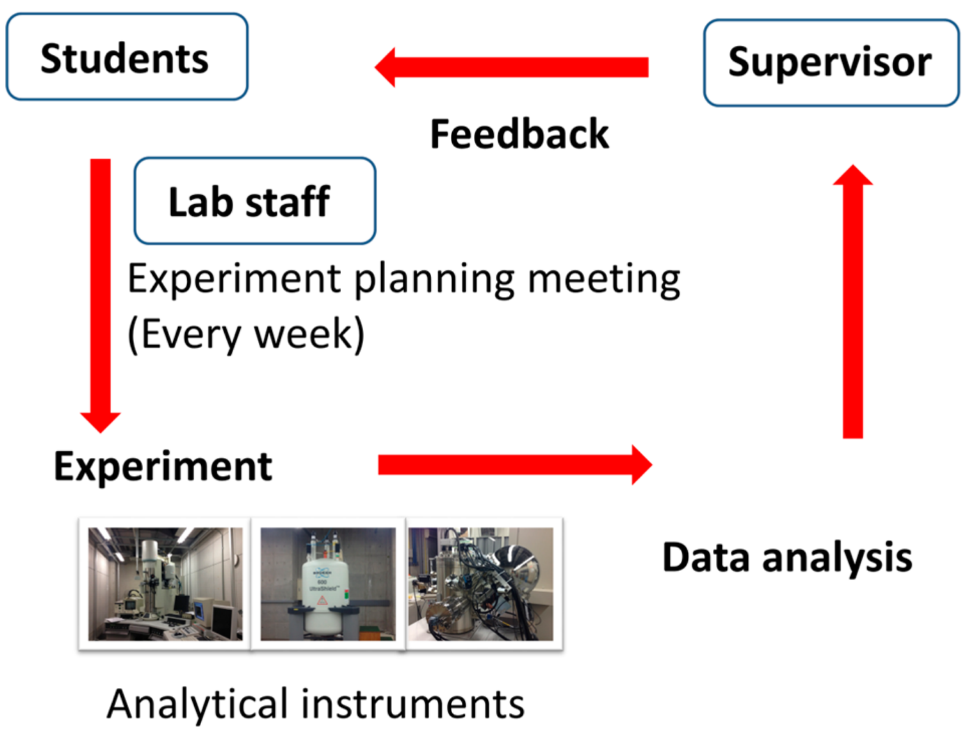


**Figure 2.** Framework of team teaching for material characterization.


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copolymers. Then, laboratory staff advised him to use electron spin resonance spectroscopy (ESR) for radical concentration, and differential scanning calorimetry (DSC) for radical/ionic ratio. In addition, as the molecular weight of the polymerized block copolymers is too great for mass spectrometry (MS), the laboratory staff advised him to use diffusion-ordered spectroscopy (DOSY) in NMR for measuring polymerization. If the proton peaks of two segments of the polymer appear to have the same diffusion coefficient on the DOSY spectrum, they are generally regarded as being connected chemically. When he first synthesized a functionalized block copolymer, the lab staff arranged his operation training in $^1$H NMR (1 h), DOSY (1 h), IR (1 h), ESR (1 h), and DSC (2 h). After training, he could operate those analytical instruments on his own. He continued to synthesize and analyze functionalized block copolymers.

He successfully obtained a series of synthesized functionalized block copolymers designed for organic resistive memory, which have good current−voltage characteristics. Then he consulted his supervisor regarding the next step of his project, and decided to investigate the morphological characterization of the functionalized block copolymers. He took a 3 h training session in atomic force microscopy (AFM). He also asked the laboratory staff to help him with an experiment involving high angle annular dark-field scanning transmission electron microscopy (HAADF-STEM). The analytical data from his experiments are provided in his group's published report and Supporting Information.[18,19]

This is one specific example of our systematic laboratory teaching program, which offers students a flexible platform for research and learning.

## SUMMARY

We reported on our open-ended laboratory program as a teaching tool in materials characterization. An open-ended laboratory is a useful systematic educational approach to teaching materials characterization in research projects of fourth-year undergraduate and graduate students. Instead of following a generic laboratory manual, an open-ended laboratory program allows students to design their own research experiments. A team led by a laboratory supervisor and staff offers a flexible, customizable program. This flexibility can be applied to various research fields in both graduate and undergraduate study.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.6b00161.

Safety policy and instrumentation list (PDF, DOCX)

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### Notes

The authors declare no competing financial interest.

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