Conference Paper

Analysis of Implementation and Result of Analytical Chemistry Instrument Labwork

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Abstract
The purpose of this research was to obtain an overview about the implementation of analytical chemistry instrument practicum and its learning outcome. The subject of this research was the students of chemistry education program of FMIPA UNNES who took Analytical Chemistry Instrument Practice course. Observations, interviews and field notes were carried out on practicum related to the method of assessment, the activities of lecturer and students during class, as well as the practical manual. Meanwhile, the instruments that used for learning outcome were verbal pretest, an observation sheet to assess the practicum result, verbal questions when students reported observational data, and assessment sheet of practicum report and its rubric. The assessment was carried out in writing containing the mastery of the concept and knowledge of the procedure, the results of practicum observation data, and the practicum report. The practicum report was assessed without using criteria that are known to students, so that it lacks continuous feedback. In general, students do not understand the basic concepts related to practicum, were less able to explain what was done and were less able to explain the observed tendency, and even did not realize that the measurement results were far from expectation. In the case of writing report, there was a weakness in correlating the data of observations, discussions, and conclusions. It was necessary to pursue a practicum activity that in addition to developing basic skills in conducting experiments and improving understanding of concepts, also develops problem-solving skill, and the formation of scientific attitudes, all of which are netted in well-prepared assessments.

Keywords: Practicum, analytical chemistry instrument, result of practicum learning, practicum assessment.

1. Introduction

Chemistry as science grew and developed on an experimental basis, therefore chemistry contained both declarative knowledge and procedural knowledge. Declarative knowledge is studied by students as a chemical theory and procedural knowledge is studied through chemical practicum [1]. In relation to learning, practicum activities are needed so that students gain concrete learning experiences and as a means of confronting the misconceptions that the students have, and in their efforts to construct
new knowledge [2]. Through experiments in a practicum, it gives students the opportunity to gain knowledge of events, propositions, imaginations, thinking skills and motoric skills. With one’s own experience one will get a memory of event, a picture of experience that has a long-term effect [3]. Some reasons for practical activities include: generating motivation to learn, aiming at understanding material, developing basic skills in carrying out experiments, and being a vehicle for learning scientific approaches [4, 5]. Furthermore, practicum activities provide wider opportunities for competency development, however, in order to obtain good learning outcomes in the learning process, good planning, preparation and evaluation tools are needed [6].

Practicum or laboratory work has cognitive, psychomotor and affective goals. Cognitive goals include: promoting intellectual development, increasing learning scientific concepts, developing problem solving skills, developing creative thinking, increasing understanding of science and scientific methods. Psychomotor/Practice or procedural objectives include: developing skills in the appearance of scientific investigations, developing skills in analyzing data findings, developing skills in communication, developing skills in working with others. Affective goals include: improving scientific attitudes, promoting positive perceptions to understand and influencing the environment [7].

A Practicum could take the form of: training, for example the use of tools; can be in the form of experience, for example in the form of verification or induction; and can be in the form of investigation or experiment. Through practicum methods students have the opportunity to experience / conduct practicum activities themselves, follow a process, observe an object, analyze, prove and draw conclusion.

Practicum implementation should pay attention to aspects of implementation, as described above. The next question is how the implementation pattern and how to assess analytical chemistry practicums, especially in universities. This research is important because student competencies, especially teacher candidates, were influenced by the teaching strategies of their lecturers [8]. The American National Science education standard [9] suggested that in lectures preparing prospective science teachers, teacher candidates’ briefing on decision-making technical skills, mastery of theory, reasoning, and practicum activities should be given more attention. The problem that will be revealed is how the pattern of implementation and how to assess the analytical chemistry practicum especially in the FMIPA UNNES chemistry department? This can provide a useful idea for the improvement of the learning process of the next chemical practicum.
2. Research Method

This research is a descriptive research which revealed the learning process of analytical chemistry instrument practicum in the Department of Chemistry, FMIPA UNNES. The research involved 1 class of students who took analytical chemistry practicum course. The analytical chemistry practicum material covers the substance of the study: Determination of acid dissociation constants in potentiometry (I), Determination of hydrolysis constants (Kh) of salt (Pb(NO$_3$)$_2$) and Constants Results of solubility (Ksp) of PbSO$_4$ and PbI$_2$ (II) salts, Conductometry titration (III), Determination the number of moles of CNS ligands in Fe(CNS)$_6^{3+}$ complex spectrophotometrically (IV), Determination of permanganate and chromate in a mixture spectrophotometrically (V), and determination of iron content in water with AAS (VI).

Instrument used for data collection were observation sheets, interviews, verbal tests, and rubrics to assess the practicum report. The results of the research data were qualitative and quantitative data that presented descriptively. The observation format was used as a survey guide, interviews were used to complete the observation data. To find out the readiness of students in conducting the practicum, a verbal pretest was conducted. The questions given in the pretest have included the basic principles of the method, the benefits of the equipment/instrument components, and the purpose of the steps in the procedure. Meanwhile, while the student was carried out the practicum and when reported the observation data, the researcher asked verbally related to the basic concept of practicum, the observed tendencies, and the observational data produced. Student explanatory weaknesses in answering verbal questions have been analyzed and summarized in Table 1.

3. Result and Discussion

3.1. Practicum implementation

Data on practicum implementation was obtained from observation, field notes, and interviews with lecturer and students as a complement to the observation data. The first met discussed the lecture contract and the group division. The met was to generally inform the basics of the material to be practiced that were related to the material of analytical chemistry instruments that had been obtained in the previous semester. Students were divided into odd groups (6 groups) and even groups (6 groups), accorded to the number/type to be practiced. Odd groups practiced first after pre-test held, even
groups were a week later. The pre-test conducted could be written or verbal. The next met, the students were prepare the instruments to be used for each groups, trained to use the instrument, and how to calibrate it. When setting the instrument and then continued by instrument utilization training, almost all of students were actively involved. Practicum implementation began with attendance check, practicum accorded to the instruction that was prepared, observational data record, and report to lecturer, followed by made report that submitted one week later. Preparation of reagent solution was prepared at the second met as well as at the time of the practicum prepared by students and with the assistance of assistants and technicians. In each practicum there is also a group who was on duty that tasked to overcome if there is a lack of reagents and tidy up/clean tools and materials related to the practice. The members were generally two lecturers, one technician, and one assistant. The assistant was assigned to assist when preparing the reagent, as well as assisting students during the practicum.

The practicum assessments were based on the results of initial tests, observation data on practicum results, practicum reports, and post-test conducted at the end of the practicum (not necessarily done). Pre and post-test are revealed regarding mastery of concept and knowledge of procedures. The practicum report was assessed without using criteria that are known to students, so that it lacks continuous feedback. Performance evaluation to this date has not been conducted.

The work procedures in the practicum manual book help practicum lecturer in preparing and carrying out laboratory activities. Work procedures in the form of sequential work steps described in detail as a recipe (cookery book type) and an observation table format was provided. This type tended to follow the verification model, where students were not given the opportunity to find something new, or were not given the opportunity to build knowledge [10]. Verification laboratory activities with the detailed guides were often make students bored, and did not encouraged students to solve problems so that students could truly able to find facts, concepts as their own findings [11].

Based on the results of observations on lecturer activities during the practicum, namely: (1) conveying the objectives of the practicum, (2) guiding during practicum activities, (3), evaluating the steps in the procedure during the practicum, (4) evaluating the mistakes made by students, (5) guiding the discussion on the results of the practicum. Of the five items, what the lecturer did was step (1), in the form of general explanation at the first met. Activities (2) to (4) could not be carried out properly because the lecturer was busy marking the practicum report that students wrote in the book, so it must be returned immediately for writing the next report. When reporting observational data,
errors were just appeared, and students generally did not know if the observation data deviated from what they should be.

### 3.2. Result of practicum learning

The readiness of students, especially the knowledge of procedures was very lacking. Generally, they were not able to explain the purpose of the steps in the experiment. This seemed in the way of answering the questions posed, apparently only 8 out of 30 students were able to answer well. The questions given in its entirety include the basic principles of the method, the benefits of equipment/instrument components, and the purpose of the steps in the procedure. Knowledge about how to prepare standard solutions and reagents were also very lacking, especially about how to calculate, and how to implement them. As an example, to prepare 100 ppm Cu standard solution, as well as a series of standard solutions, generally the calculations could not yet be calculated and did not take into account the amount / volume requirements that must be made. Questions regarding the main components and the basic principles of how the equipment work in general, the students’ explanations were not good enough even though this course was given after the Analytical Chemistry Instrument course.

When students reporting the results of observational data and while doing the practicum, researchers ask verbally related to the basic concepts of practicum, observed tendencies, and the observational data produced. The weaknesses of students’ explanations during the practicum are summarized in Table 1.

The inability of students in mastering concepts that have been received in the previous semester, as well as being less capable in explaining what was done and the observed tendencies as listed in Table 1, probably caused by laboratory activities that were still based on verification which is proving the concept is less emphasized on mastery that give students the opportunity to organized knowledge as scientists do. Conditions related to chemical practicum problems including analytical chemistry that relevant are also stated, among others [12–15]. According to these experts that through practicum, in addition to understanding concepts, psychomotor skills can be developed, as well as the ability to explain what is done and the symptoms observed. Emphasis on this concept is in accordance with what is stated in the core curriculum of the competency points of analytical chemistry practicum courses, namely being able to develop chemical concepts by utilizing technology and art, and using chemical equipment in the development of chemical concepts [16]. Both of these competency items indicate that the development of chemical concepts can be achieved through
| No | Substance of Study                                                                 | Result of Data Collected                                                                                                                                                                                                                                                                                                                                                     |
|----|----------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1  | Determination of acid dissociation constants in potentiometry                    | a. Could not explain why the potentiometric titration being worked out should be reached at pH > 7   
b. The results of Ka prices that were obtained through 4 different ways, in general, students did not try to discuss which method was the most thorough, and not many students have compared the result with Ka from the literature.  
c. Not able to explain the purpose of each step in the procedure such as:  
1. Why need to find an equivalent point 
2. What is the purpose of making titration curve 
3. Why pH that sought after was at half the equivalent point. |
| 2  | Determination of hydrolysis constants (Kh) of salt (Pb(NO₃)₂) and Constants Results of solubility (Ksp) of PbSO₄ and PbI₂ (II) salts | a. Students did not know that there was an error in pH data of (Pb(NO₃)₂) solution, which was caused by inaccuracy when making the solution. 
b. Students did not know that inaccurate (Pb(NO₃)₂) solution resulted in inaccuracies of Kh and Ksp prices.  
c. Students did not know the reason why the pH area of (Pb(NO₃)₂) solution should be lower than 7  
d. Students did not know the reason why pH of PbSO₄ and PbI₂ should be on pH Pb(NO₃)₂ area. |
| 3  | Conductometry titration                                                          | a. Students did not know when the equivalence point was reached, because they were not able to predict from the concentration of the solution used, so often the titration has ended even though the equivalent point has not been reached.  
b. Graph seemed to be carelessly made, the abscissa and ordinate were not given names and scales were less noticed so that the location of the equivalence point is less precise. |
| 4  | Determination of the number of moles of CNS ligands in Fe(CNS)₆³⁺ complex spectrophotometrically | a. Students could not calculate the number of mole fraction of Fe³⁺  
b. Students did not know that the observational data of absorbances were less appropriate because of inaccuracy in solutions making.  
c. Students could not explain the purpose of adding HNO₃  
d. Students could not explain the correlation between mole fraction and absorbance |
| 5  | Determination of permanganate and chromate in a mixture spectrophotometrically   | a. Observation data of absorbance both in permanganate and chromate mixtures and on their own were often reversed, but students did not know that the data was clearly wrong. |
| 6  | Determination of iron content in water with AAS                                 | a. Students could not explain the purpose of adding HNO₃  
b. Students did not know the purpose of blanko solution and how to prepare it  
c. Students did not know that calibration curve was not linear enough  
d. Students did not know that there is correlation between sample's absorbance data and calibration curve  
e. Students could not distinguished the difference between calibration curve method and addition standar method |
well-planned laboratory activities. The planning is in addition to the practicum guide, practicum implementation pattern, as well as how to conduct performance assessments during the learning process including in terms of affective. The evolving physical and mental activities in an effort to construct new knowledge through primary data obtained using all five senses, well-practiced chemical practicum activities will give students the opportunity to develop chemical concepts, ways of thinking, attitudes, and communication skills, and equipment manipulation skills [17, 18]. Besides that it will also encourage students to become more directed both mentally and intellectually, so that they will become more active in asking questions, discussing with friends, and also more responsible for cleanliness and work safety.

The ability of students to correlate between observational data, discussion, and conclusions were captured through analysis of practicum reports [19–21]. Throughout the experience of researchers regarding practicum reports, students generally follow the example of their seniors’ report without paying attention to the differences in the correlation between each component that is written in the class report. Student weaknesses that stand out in general in the discussion that less able to correlate between the results of observational data with literature reviews that have been written.

To find out the description of the practicum result, it is also measured through student activities that are captured through observation sheets. Student activities observed included: (1) preparing practical equipment, (2) designing practical work, (3) preparing tools and materials, (3) observing, (4) writing observations, (5) answering lecturer questions, (6) discussion with friends, (7) asking lecturers (feedback), and (8) tidying up and cleaning equipments and materials. Of all the items observed, item (2) has not been implemented/has not been seen, while other activities appear evenly [23-28].

4. Conclusion

1. Practicum began with attendance check and collection of reports, followed by practicum with verification based manual, and ended with reporting of observation data.

2. The assessment included the pre-test data carried out in writing or verbal contained the mastery of the concept and knowledge of the procedure, the results of the practicum observation data, and the practicum report. Performance evaluation during the learning process could not be carried out yet, and its instrument is not yet available.
3. Guidance and evaluation of the steps in the procedure during the learning process could not be carried out properly, if the lecturers were focus more on correcting the practicum report that must be returned immediately.

4. In general, students did not understand the basic concepts related to practicum, less able to explain what has to be done and were less able to explain the observed tendencies, and even did not realize that the measurement results were far from expected.

5. The practicum report was assessed without using criteria that were known to students, so that it lacking continuous feedback. In the case of writing report, there was a weakness in correlating the data of observations, discussions, and conclusions.

5. Suggestion

1. There is need to be an effort for providing an instrument during practicum activities which in addition help to develop basic skills in conducting experiments and improving the understand of concepts, also develops problem-solving skills, and the formation of scientific attitudes, which are compatible with the purpose of practicum.

2. To implement analytical chemistry instruments practicum based on projects base learning, it is necessary to pay attention to mastery of concepts, skill of equipment mastery, and analysis of the results of observational data.

References

[1] Dahar, R dan Liliasari. (1986). Pengelolaan Pengajaran Kimia. Modul Universitas Terbuka. Depdikbud: Universitas Terbuka

[2] Ergul, R., Y. Simsekli, S. Calis, Z. Ozdilek, S. Gocmencelebi, and M. Sanli, 2011. The Effect of Inquiry-Based Science Teaching on Elementary School Students’ Science Process Skills and Science Attitudes. Bulg. J. Sci. Educ. Policy, 5(1): 48–68.

[3] White, R. T. 1996. The Link between the Laboratory and Learning, International J. Science Education. 18(7), 761-774.

[4] Nuryani, Y Rustaman. 2002. Perencanaan dan Penilaian Praktikum di Perguruan Tinggi. Makalah disiapkan untuk Program Applied Approach Bagi Dosen UPI. Unpublished.
[5] Tobing, dan Rangke, L. 1981. *Model Pembelajaran IPA di Sekolah Lanjutan*. Jakarta: P3G Depdikbud.

[6] Hackathorn, J., Solomon, E. D., & Blankmayer, K. L. (2011). Learning by Doing: An Empirical Study of Active Teaching Techniques. *The Journal of Effective Teaching, 11*(2):40-54.

[7] Pabellon J.L. and A. B. Mendoza. 2000. *Sourcebook on Practical Work for Teacher Trainers: High School Physics Volume 1*. Science and Math Education Manpower Development Project (SMEMDP). University of The Fillipin, Quezon City.

[8] Prudente, M. S. & Aguja, E.S. 2002. “Science Teaching and Learning Process in Preservice Teacher Education: The De Salle University-Manila Experience”. *Electronic Journal of Science Education, Vol 1, September*. [Online]. Tersedia: http://unr.edu/homepage. [17 Oktober 2018].

[9] National Research Council. 1996. *National Science Education Standards*. Washington, DC: National Academy Press.

[10] Zhang, S & X. Zhang. 2014. Teaching Analytical Chemistry in China: Past, Present, and Future Perspectives. *Anal. Bioanal. Chem.*, 406(17): 4005–4008.

[11] McComas, W. 2005. Laboratory Instruction in The Service of Science Teaching and Learning " The Science Teacher. 72 (7): 24-29.

[12] Jalil, P.A. 2006. A Procedural Problem in Laboratory Teaching: Experiment and Explanation, or Vice-versa? *Journal of Chemical Education:* 83(1).

[13] Adani, G. A. 2006. New Project-Based Lab for Undergraduate Environmental and Analytical Chemistry. *Journal of Chemical Education, 83*(2), 253-256.

[14] Amarasiriwardena, D. 2007. Teaching Analytical Atomic Spectroscopy Advances In An Environmental Chemistry Class Using A Project-Based Laboratory Approach: Investigation Of Lead And Arsenic Distributions In A Lead Arsenate Contaminated Apple Orchard. *ABCS of Teaching Analytical Science*.

[15] Kipnis, M dan Hofstein, A. 2007. The Inquiry Laboratory As A Source for Development of Metacognitive Skills. *International Journal of Science and Mathematics Education*.

[16] Frederick, K. A. 2013. Using forensic science to teach method development in the undergraduate analytical laboratory. *Anal. Bioanal. Chem., 405*(17):5623–5626.

[17] Arifin, M. 2005. *Kegiatan Praktikum Dalam Proses Pembelajaran Kimia Untuk Mendukung Pengembangan Kompetensi Calon Guru*. Laporan Penelitian.

[18] Chan, J. Y. K. & Bauer, C. F. 2016. Learning and Studying Strategies used by General Chemistry Students with Different Affective Characteristics. *The Royal Society of Chemistry, 1*(1):1-28.
[19] Demirel, M. & Dagyar, M. 2016. Effects of Problem-Based Learning on Attitude: A Meta-analysis Study. *Eurasia Journal of Mathematics, Science & Technology Education*. 12(8): 2115-2137.

[20] Downing, K. 2010. Problem-Based Learning and Metacognition. *Asian Journal Education & Learning*, 1(2):75-96.

[21] Ferreira, M.M. & Trud, A.R. 2012. The Impact of Problem-Based Learning (PBL) on Student Attitudes toward Science, Problem-Solving Skills, and Sense of Community in the Classroom. *The Journal of Classroom Interaction*. 47(1): 23-30.

[22] Urena, S.S., Melanie M. C., and Ron S. 2012. Effect of Cooperative Problem-Based Lab Instruction on Metacognition and Problem-Solving Skills. *Journal of Chemical Education*, 1(89):700-706.

[23] Haryani, S., Prasetya, A.T., & Wardani, S. 2010. Peningkatan Metakognisi Mahasiswa Calon Guru Kimia Melalui Simulasi Laboratorium Virtual Berbasis Masalah pada Materi HPLC. *Proceeding Himpunan Kimia Indonesia*.

[24] Haryani, S. 2011. Praktikum Kimia Analitik Instrumen Berbasis Masalah pada Spektrometri UV-Vis untuk Meningkatkan Metakognisi Calon Guru. *Laporan Penelitian*.

[25] Hicks, R.W., & Bevsek, H. M. 2011. Utilizing Problem-Based Learning in Qualitative Analysis Lab Experiments. *Journal of Chemical Education*, 1(89): 254–257.

[26] Günter, T. & Alpat, S. K. 2013. The Effects of Problem-Based Learning (PBL) on the Academic Achievement of Students Studying ‘Electrochemistry’. *The Royal Society of Chemistry*, 1(1): 2-19.

[27] Tosun, C. & Senocak, E. 2013 The Effects of Problem-Based Learning on Metacognitive Awareness and Attitudes toward Chemistry of Prospective Teachers with Different Academic Backgrounds. *Australian Journal of Teacher Education*, 38(3):61-73.

[28] Mataka, L. M. & Kowalske, M.G. 2015. The influence of PBL on students’ self-efficacy beliefs in chemistry. *The Royal Society of Chemistry*. 1(1):1-10.