The development of integrated chemistry-based experiments implications for the teaching of environmental chemistry in the Vietnamese context

> By Hoa Thi Tran, BSc Hanoi, 1995

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The candidate confirms that the work submitted is her own work and the appropriate credit has been given where reference has been made to the work of others.



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## ABSTRACT

A survey of environmental chemistry laboratory experiments in the Journal of Chemical Education (ACS) and Education in Chemistry (RCS) has been carried out from 1969 to the present. The experiments have been categorized as being related to the areas of either air, water or soil. Over the same period of time, a similar survey has been carried out for commonly used environmental chemistry textbooks, whereby the relative number of pages have been assessed which are devoted to the same areas. The data obtained from both of these analyses indicate that the area of soil is seriously underrepresented in the environmental chemistry curriculum. Further analysis of this material also highlights the failure of many experiments to reflect "real world" laboratory processes such as sample preparation, data analysis and quality control/assurance. To help alleviate these deficiencies, a strategy was designed to develop several soil-related laboratory experiments which would not only reflect real world practices in a developed country such as Australia, but which would also be appropriate for educational programs in a developing country such as Vietnam. In order to gain the necessary skills and experience, the author undertook a five month work experience program at the Australian Government Analytical Laboratories (AGAL). The program focussed on the routine analysis of hydrocarbons in soil samples. A programmed schedule allowed the author to experience all aspects of the procedure, from sampling through to reporting. Following the work experience program, the author undertook a visit to her home country of Vietnam in order to conduct a modified Dephi survey of leading Vietnamese educators, considered expert in the area of environmental chemistry education. The survey was constructed to assess the requirements for the design and implementation of undergraduate environmental chemistry experiments in the Vietnamese context. The Delphi study successfully converged after two rounds. Both the AGAL experience and the results of the Delphi survey have been used to formulate environmental chemistry laboratory experiments which help address the deficiency of soil-related experiments in the current curriculum, reflect the professional skills of real world environmental scientists, and which are well-suited for implementation in developing countries such as Vietnam. Attention has also been paid to international requirements for environmental education and to the advantages of the integrated approach with respect to the different areas of chemistry.

## ABBRIVIATIONS

EE	Environmental Education
AAS	Atomic Absorption Spectroscopy
AGAL	Australian Government Analytical Laboratories
ASEAN	Association of South East Asia Nations
DCM	Dichloro Methane
EC	Environmental Chemistry
ECE	Environmental Chemical Education
EPA	Environmental Protection Agency
FTIR	Fourier Transform Infrared
GC	Gas Chromatography
GC/MS, FID, PID	Gas Chromatography with Mass Spectroscopy, Flame
	Ionization Detector, Phosphorus Ionization Detector
H soil	Homogenized soil
HC	Hydrocarbons
HPLC	High Performance Liquid Chromatography
ICP/MS	Inductive Coupled Plasma Emission/Mass Spectroscopy
IHC	Individual Hydrocarbons
IR	Infrared Spectroscopy
IUCN	International Union for the Conservation of Nature and Natural
	Resources
LFB	Laboratory-Fortified Blank
LFM	Laboratory-Fortified Matrix
MOSTE	Ministry of Science, Technology and Environment
MS	Mass Spectroscopy
NH soil	Non-Homogenized soil
РН	Petroleum Hydrocarbons
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percentage Difference of Duplicates
Stdev	Standard deviation
TLC	Thin Layer Chromatography
ТРН	Total Petroleum Hydrocarbons
UK	United Kingdom
UNEP	United Nations for Environmental Program
USA	United State of America
UV-VIS	Ultraviolet-Visible spectroscopy

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#### INTRODUCTION

The implications and consequences of environmental problems have been widely recognised. Thus, a new strategy "to defend and improve the environment for present and future generations has become an imperative goal for mankind" (1). Together with innovation in science and technology, education, as identified by the world's first International Conference on Environmental Education (EE), has a vital role to play in helping the public to become aware of and to better understand environmental problems (1).

Regarding EE at university level, the creation of integrated courses on environmental issues for all students, regardless of their specific fields, has been stated to be in high demand (2). Science educators in general and chemical educators, in particular, have worked toward the development of appropriate curriculum with regard to environmental issues (3). A significant number of environmental chemistry (EC) textbooks are now available worldwide accompanied by the development of appropriate EC experiments suitable for undergraduate EC courses. Commonly, these books and experiments have been developed in the context of the resources and educational/ scientific background of developed Western countries.

EE has now become an important part of the curriculum in the Vietnamese Education and Training system, particularly at university level (4). Thus, the development of environmental programs in Vietnamese universities is now a high priority. As part of the undergraduate EC curriculum development, accessible experimental programs need to be designed. Three problems, however, confront the development of the EC curriculum in Vietnam.

- <u>Current international EC programs</u>. EC programs, and experimental EC programs in particular, tend to be designed and more applicable for developed countries. Thus, developing countries, such as Vietnam find it difficult to transpose the strategies and resources/equipment from such existing programs. Any introduction of new environmental-focused content, therefore, will require innovation in course design, availability of resources/equipment, and the acceptance of national goals for EC experimental programs in Vietnam as a whole.
- 2. <u>The "gap" in the existing EC program</u>. The soil issue is seriously under-represented in both coursework and experimental programs. Soil quality is an important factor for the economic development of a developing country such as Vietnam. Moreover, the current international laboratory programs in EC courses have serious drawbacks in terms of inefficiency and failure to provide students with opportunities to practice vital procedures in the environmental science. Thus, it is essential for an EC program in Vietnam to address this issue.
- <u>The cost of the development of new courses</u>. The introduction of EE will need to be cost effective in a developing country such as Vietnam. Particularly, experiments in EC will need to be developed which are within the means of the science faculties in Vietnamese universities.

This project is aimed at developing a set of criteria for effective experiments for EC courses in Vietnamese universities. Specific attention will be paid to the design of innovative EC experiments in soil analysis, an aspect relatively under-represented in existing courses. These experiments will also take into account the limitations of the

current international EC curriculum. The adaptation of such experiments is intended to be a preparatory step in the development of a desirable experimental program for EC suitable for the laboratory conditions existing in developing countries in general and in Vietnam in particular.

### CHAPTER 1 AIMS OF THE STUDY AND METHODOLOGY

#### 1.1 The Aims of the Study

The aims of this study are as follows:

- (i) To identify the appropriate approach for the teaching of EE worldwide.
- (ii) To critically survey the international educational literature and textbooks, in order to identify factors that are considered to be essential for the success of EC teaching- EC education (ECE) laboratory experiments. Also, the literature review will identify areas that need to be addressed in the development of ECE laboratory experiments, which, though important in the practice of environmental science worldwide, may be underrepresented in the educational curricula.
- (iii) Having identified such areas, to undertake a five-month work experience program with a relevant employer in order to gain up-to-date, "real-world" professional experience for translation of EC experiments into material for the educational curriculum.
- (iv) Using the elements that are considered to be essential in the ECE laboratory from the international perspective, to construct and conduct a survey of Vietnamese higher education staff in order to determine the requirements for laboratory teaching experiments in EC in the Vietnamese context.
- (v) Using the above information, to design a number of EC laboratory teaching experiments, which will address any curriculum deficiency and be relevant to the requirements of developing countries.

#### **1.2 Design and Methods**

Towards achieving aim (i), a thorough survey of the literature on EE has been undertaken. The main objectives and guiding principles from the international perspective have been clarified. Guiding principles and limitations for EE at the higher education level, in particular, have also been identified. These are discussed in more detail in **Chapter 2**.

In order to achieve aims (ii) and (iii) a thorough survey of available environmental chemistry textbooks and international educational literature review has been carried out. For the design and implementation of sound ECE laboratory experiments, it is imperative to combine the conventional wisdom on EE programs with the requirements of the chemical education curriculum. These led to the "theory" criteria, which suggest issues are in need of coverage in a good ECE laboratory experiment. Based on such criteria, the 5-month-work experience program in the real world environmental laboratory will provide a framework for the strengthening of the critical review of the literature on ECE laboratory experiments.

Aims (iv) and (v) of this study intend to develop actual environmental chemistry-based experiments which are feasible in Vietnamese universities. The experiments themselves must be developed in a laboratory with suitable equipment and which is staffed by teachers with professional expertise in environmental chemistry (EC). Such conditions are not available in Vietnamese universities. Thus, the researcher is undertaking the investigation in Australia, which has organisations relatively well-endowed with both the necessary equipment and professional expertise. In this regard, a five-month work

experience program was carried out in order to gain real-world professional experience and to access materials and facilities for the study.

However, there is another aspect of the problem facing the researcher. To evaluate with certainty the suitability of experiments designed in Australia for inclusion in chemistry courses in Vietnam would normally require extensive trialing. Unfortunately, the time required in undertaking such an evaluation is well beyond the scope of a Master of Education thesis. It is understandable that to help develop a good experiment it is vital for the developer to understand factors that make the experiment work best in the specific situation, which in this study, is Vietnamese universities. Unfortunately, the lack of availability of appropriate guidelines for EC experimental development is another obstacle facing the researcher. There is no such material in Vietnam at the time of conducting this study. The researcher has had to start from the beginning. Therefore, in order to achieve aim (v), aim (iv) must be achieved in order to find out what are the criteria for an undergraduate EC experimental program in the Vietnamese situation. In order to have a valid and valued answer to this question, there may be no source other than Vietnamese EC experts. Therefore, the Delphi survey method, which is a tool for research using experts' opinions, was chosen and modified to use as the most appropriate methodology in this study. The Delphi method is discussed in Chapter 4. The details of the application of the Delphi survey method in this specific study are covered in Chapter 5, and the finding of this survey is reported and discussed in Chapter 6. Chapter 7 presents the two proposed experiments, which are the fruits of the research.

### CHAPTER 2 ENVIRONMENTAL EDUCATION

#### **2.1 International Perspectives**

Environmental Education (EE) has been a term in common usage for several decades. It was first used in the meeting of the International Union for the Conservation of Nature and Natural Resources (IUCN) in Paris in 1948 (5). Further attention has been paid to EE in subsequent international meetings, including IUCN in 1965 and the Biosphere Conference (held by UNESCO) in 1968. However, it was not until 1970 that the term "Environmental Education" was officially clarified by IUCN/UNESCO, who officially named a new aspect of EE in the International Working Meeting on Environmental Education School Curriculum. The so-called "classic definition" of EE was understood as:

"The process of recognizing values and clarifying concepts in order to develop skills and attitudes necessary to understand and appreciate the inter-relatedness among man, his culture, and his biophysical surroundings. Environmental education also entails practice in decisionmaking and self-formulation of a code of behavior about issues concerning environmental quality".

(Palmer, J. A. 1998) (6)

Indeed, it has been argued that EE has always accompanied the interaction of human beings with the environment (5, 7). The implicit reason behind the study of the environment, however, has changed over time according to the state of humans' attitude towards the environment. The purposes of EE in each period of time, therefore, have been marked by significant differences. In the past, it was to understand and to explore the surrounding world in order to live in harmony with the environment. In this regard, it may latter be classified as "education *in* the environment" and/or "education

*about* the environment". That rationale has been changed, however, when humans worldwide have faced many negative, unforeseen natural disasters and extremes in weather. EE thus has come to mean work for the improvement and protection of the environment (7). The concept now refers to "education *for* the environment", as well as "education *in* the environment" and "education *about* the environment".

#### 2.1.1 The Goals, Objectives and Guiding Principles of EE Programs

The first international meeting on the environment, The United Nations Conference on the Human Environment, was held in 1972 (Stockholm). It emphasized the rapid increase in awareness of environmental issues worldwide and the need for the practice of EE. As a result, three years later, in 1975, an international program, the so-called IEEP (International Environmental Education Program), was launched through the cooperation between UNESCO and UNEP (United Nation Environmental Program) (5). The guidelines for this new field of education were subsequently formulated in the International Environmental Education Workshop, held in 1975 in Belgrade. The outcome of this workshop was the Belgrade Charter, which has been reported as the historic basis of EE (8). It created a draft framework of principles and guidelines for worldwide EE programs, which were redrafted at the next workshop. Indeed, the new framework, two years later, was endorsed in the world's first Intergovernmental Conference on Environmental Education, held in 1977 at Tbilisi, Georgia (USSR) (1). The result of the above meeting was the Tbilisi Declaration, in which the goals, objectives and guiding principles for EE were specified. Though published decades ago, the Tbilisi Declaration still plays a dominant role in guiding the development of worldwide EE programs (5, 9). It has been considered as a primary standard for everyone who seeks to undertake EE at all levels: local, national, regional, international, and for all age groups in both formal and informal education (9-12). Ten years latter, in 1987, the UNESCO/UNEP International Environmental Education Program Conference or "Tbilisi Plus Ten" held in Moscow, formally endorsed the principles of EE which were launched in the Tbilisi Declaration (5). Given the recognition internationally as the guidelines for the development of environmental education program, the Tbilisi Declaration will be, therefore, used as the key criteria for the evaluation of the validity of an EE program, in this study. The researcher will also use these worldwide guidelines in developing specific applications of EE in chemistry: that is in environmental chemistry.

Three goals of EE that have been specified in the Tbilisi Declaration are as follow (1):

- To foster clear awareness of, and concern about, economic, social, political and ecological interdependence in urban and rural areas.
- (ii) To provide every person with opportunities to acquire the knowledge, values, attitudes, commitment and skills needed to protect and improve the environment.
- (iii) To create new patterns of behaviour of individuals, groups and society as a whole towards the environment.

The five interrelated categories of objectives of EE include (1):

- Awareness: to help social groups and individuals acquire an awareness of and sensitivity to the total environment and its allied problems.
- **Knowledge**: for social groups and individuals to gain a variety of experiences in and acquire basic understanding of the environment and its associated problems.

- Attitude: to help social groups and individuals acquire a set of values and feelings of concern for the environment and the motivation for actively participating in its improvement and protection.
- Skills: to help social groups and individuals acquire the skills for identifying and solving environmental problems.
- **Participation**: to provide social groups and individuals with an opportunity to be actively involved at all levels in working toward resolution of environmental problems.

Twelve principles for the development of the EE program have been advocated (1):

- 1. Environmental education should consider the environment in its totality-natural and built, technological and social (economic, political, cultural-historical, moral, ethical).
- 2. Environmental education should be a continuous life-long process, beginning at the pre-school level and continuing through all formal and non-formal stages.
- 3. Environmental education should be interdisciplinary in its approach, drawing on the specific content of each discipline in making possible a holistic and balanced perspective.
- 4. Environmental education should examine major environmental issues from local, national, regional and international points of view so that students receive insights into environmental condition in other geographical areas.
- 5. Environmental education should focus on current and potential environmental situations while taking into account the historical perspective.

- 6. Environmental education should promote the value and necessity of local, national and international cooperation in the prevention and solution of environmental problems.
- 7. Environmental education should explicitly consider environmental aspects in plans for development and growth.
- 8. Environmental education should enable learners to have a role in planning experiences and provide an opportunity for making decision in the prevention and solution of environmental problems.
- 9. Environmental education should relate environmental sensitivity, knowledge, problem-solving skills and values clarification to every age, but with special emphasis on environmental sensitivity to the learner's own community in early years.
- 10. Environmental education should help learners discover the symptoms and real causes of environmental problems.
- 11. Environmental education should emphasize the complexity of environmental problems and thus the need to develop critical thinking and problem-solving skills.
- 12. Environmental education should utilize diverse learning environments and a broad array of educational approaches to teaching/ learning about and from the environment with due stress on practical activities and first-hand experience.

#### 2.1.2 Environmental Education Approaches

Interdisciplinary and multidisciplinary are two approaches, which have been considered to be critically important procedures in EE processes. The interdisciplinary approach is understood as the co-ordination of many different disciplines towards the study of a particular subject. It is not a brand new "thread" in the educational methodology. It has been, indeed, used and considered as a renovation approach (7). The holistic characteristic of the environment, which the education is aimed at, demonstrated the feasibility of applying this approach. As the Tbilisi declaration suggested that the environment is a complex system, which consists of natural, built and social components (1), therefore, it might be impossible for one single discipline to cover the totality characteristic of the environment. Thus, in order to achieve effectively the goals of EE, an interdisciplinary approach has been strongly suggested. In other words, in order to achieve success in solving environmental problems and managing the quality of the environment, EE requires the contribution of all disciplines (e.g. natural science, social science, humanities, applied sciences, and technologies) by fusing, combining, and co-ordinating with one another (1). To a lesser extent, when the environmental concepts are integrated into each discipline, it is the so-called the multidisciplinary approach (7).

In focusing on the details of approach of EE in each discipline, three threads can be referred to as follows:

- Education *about* the environment.
- Education *in* (other words have also been alternatively used include *through*, *from* or *with*) the environment.
- Education *for* the environment.

They will be described in the following parts:

#### 2.1.2.1 Education *about* the Environment

It is considered that education *about* the environment is the most popular mode of EE (11). In this approach, students are provided with fundamental knowledge and concepts to help them understand how the natural system works and the impact of human activities upon them. However, the drawback of the education *about* the environment approach is that its progress often has neglected the connection between natural systems and social systems. The integration between EE, natural and social system is considered to play an important role in solving environmental issues from local, national to international aspects and their responsibility for the management of the environment (11). Nevertheless, through the *about* approach, educators can provide students with useful knowledge and skills. For example, students can understand the concepts, facts and figures, and based on informed knowledge, are able to evaluate environmental issues (5).

#### 2.1.2.2 Education in the Environment

This approach relates to the use of the environment as a means for education. Through direct contact with the environment, students can acquire appropriate knowledge and important skills (e.g. data collection, field investigation, observation, sketching, photography, using scientific instrument, etc.). In gaining such knowledge students may, therefore become aware of the environmental problems and have more concern about environmental issues. However, the ultimate purpose of the EE could not have been met if EE uses this approach alone.

#### 2.1.2.3 Education for the Environment

Education *for* the environment is developed based upon two critically important aspects: international trends in EE and the development in critical curriculum theorizing and critical pedagogy. It, therefore, is considered to play a vital role in EE and to be used most widely and as one important orientation approach of EE (5, 11).

The target of this approach is more sophisticated than that of two previously introduced approaches, namely education *in* and *about* the environment. It is not only simply teaching environmental facts and concepts as in the education *about* the environment or just experiential learning in nature as in the education *in* the environment approach. Above all, education *for* the environment aims at engaging students in the active resolution of environmental questions, issues and problems based on both *in* and *about* approaches. By doing that, students are not only required to acquire skills by working on finding solutions for environmental problems, but also to become concerned with an environmental, moral and political education. It is, therefore, considered as a "counterhegemonic" process or the related "dangerous knowledge" (11).

Following are the detailed characteristics of education for the environment (13):

 Education for the environment emphasises the development of a critical environmental consciousness based upon: (i) A holistic view of the environment as a totality of the interdependent relationships between natural and social systems; (ii) A historical perspective on current and future environmental issues; (iii) The study of the causes and effects of environmental problems, and alternative solutions to them, through an examination of: (a) The relationships between ideology, economy and technology; and (b) The linkages between local, regional, national and global economies and governments.

- 2. Education *for* the environment emphasizes the development of critical thinking and problem-solving skills through a variety of practical and interdisciplinary learning experiences which focus on real-world problems and involve the study of a wide range of sources and types of information.
- 3. Education *for* the environment emphasizes the development of an environmental ethic based upon sensitivity and concern for environmental quality.
- 4. The development of the understandings, attitudes and skills of political literacy which promote participation in a variety of forms of social action to help improve and maintain environmental quality.
- 5. Education *for* the environment requires teaching strategies that are consistent with its goals.

These characteristics of the different forms of EE have strong relationships with each other. The researcher considers that the ultimate and the highest purpose of environmental education is the *for* approach. It does not, however, mean that the other two approaches *in* and *about* are "valuable only in so far as they are used to provide skills and knowledge to support the transformative intentions of education *for* the environment", argued by Fien, 1993 (11). Depending on the level of students, EE may be constructed in different approaches (5). The *in* and *about* approaches may be suitable for the students at the lower level and education *for* the environment approach approaches of *in* and *about* approaches for higher level of students. Indeed, in the following section, EC experiments will be examples of the use of education *for* the

#### environment (Chapter 7).

#### 2.2 Environmental Education at the University Level

#### 2.2.1 General Trend

Universities and higher education play an important role in graduating people who will have an impact on society and the environment. They include policy and decision-makers, engineers, architects, administrators, lawyers, teachers, environmental specialists, etc. (14). Therefore, EE programs at this level play an important part in the improvement of the Earth (14). EE at the university level is different from EE programs designed for lower levels of education, in which EE is primarily introduced as fundamental concepts and principles (in the category of education *about* the environment). For higher education students, however, the emphasis is on education *for* the environment. Indeed, the process must be to educate how to analyze environmental problems, and to find the specific causes and effects of such problems on the quality of life. Moreover, it also requires students to propose possible solutions for such environmental problems (14).

An EE course at the university level will need to consider (i) the physical planet (atmosphere, hydrosphere, lithosphere, their physical and chemical laws); (ii) the biosphere; and (iii) the technosphere and sociosphere (14). The most feasible approach in EE is likely to be one with a multidisciplinary basis. In other words, it is the best to incorporate the relevant environmental knowledge into the existing discipline. It is also important to use real-world problems examples and case studies. Such an approach is supported by the accepted international trends in EE, informed by the Tbilisi Declaration.

#### 2.2.2 In ASEAN Countries

Well aware of the important role of EE and the urgent need for the development of EE programs, some environmental courses/ programs have been offered at some universities in ASEAN countries. In these courses, the goals, objectives, and guiding principles for the development of EE from the Tbilisi declaration have been adopted. Also, the development of EE in ASEAN has followed the pattern in developed countries, including (i) the addition of the environmental component into the existing programs; (ii) the creation of new specialised institutes and (iii) the establishment of environmental materials (12). Interestingly, the integration between developments in science, technology, social factors and EE in examining vital environmental issues from local, national, regional and international perspectives has been strongly emphasised (12, 15). Thus, EE programs need to illustrate real world environmental issues and working closely with the environment:

"The critical role of field work and project engagement throughout students' courses of study may be the most positive approach being reported in those institutions that have as yet accepted the challenge. The importance of students' engagement in "real" environmental project work with a true social dimension is thus reinforced".

(Fensham, et al. 1997 (16))

EE in higher education in the ASEAN countries in general and in Vietnam in particular is considered to be particularly important in the educational system. EE not only plays a crucial role in generating environmental expertise, but is also regarded as a solution to help overcome existing difficulties of current environmental programs and their lack of effectiveness (15, 17). The most important task of EE is that of responsibility in training and retraining of people, who may have an effect on environmental protection activities, such as professional and managerial staff and environmental educators. Moreover, higher education is where the creation, exchange and application of knowledge take place, thus to help. Such knowledge may then be transferred to lower educational areas such as schools.

In ASEAN countries, the multi-sectorial and interdisciplinary approach has been suggested to be a key success for EE in a region where there is a complexity of environmental problems. Similar to the general approach of EE, at the higher education level in particular, the key element for success is a close working relationship with environmental activities. The combination of education with environmental legislation and participation in real environmental projects is highly recommended (15). However, recent evaluation suggests ASEAN EE programs did not work effectively because of the lack of communication between relevant government departments. This led to EE programs being divorced from real-world environmental activities (15). This needs to be taken into consideration for the development of an EE program in Vietnam.

#### 2.2.3 In Vietnam

#### 2.2.3.1 Environmental Issues

Environmental pollution has become an issue of great concern to Vietnam. Some recent reports have shown that environmental problems including air, water, soil pollution, and wastewater and solid disposal have become matters of great concern (18, 19). The reasons for such serious problems may be accounted for by the rapid development of industry (18, 19) in the period of modernization and industrialization in Vietnam. The intensive exploitation of land resources and the overuse of chemical fertilizers and pesticides in agriculture have created soil and water pollution (18). Marine pollution, on the other hand, is the consequence of the development of oil and gas exploitation,

transportation and processing (18). In addition, many environmental problems exist in Vietnam as a legacy of war (17).

#### 2.2.3.2 Environmental Education

For the reasons mentioned above, it is an essential issue for Vietnam to strengthen environmental protection in order to sustain national development during this industrializing and modernizing period (20). Teaching environmental issues in the national education system is argued to be one of the important tasks of the country in this regard (19-21). The recognition of the vital role that education can play in the environmental protection movement and sustainable development in the country has been emphasized in a large number of governmental documents. These include: The Law on Environmental Protection, which has been passed by the Vietnamese National Assembly on December 27th 1993; Politburo Instruction 36-CT/TW; and Prime Minister Decree 1320/CP-KG (22, 23). Most recently, on 5<sup>th</sup> August 1998, in the conference of "A national strategy for environmental protection and sustainable development to 2020", EE has been reaffirmed in its key role. Teaching environmental issues in the national education system is considered to be one of the vital tasks in this national effort (20). Most importantly, the Vietnamese Ministry of Education and Training has included EE into the nation's educational programs at all levels. Currently, one of the most important tasks of the Education and Training Ministry is to develop environmental curricula for universities (4).

In the 1990s, there is an increase in environmental problems in Vietnam across all sectors urban, rural, industrial and agricultural. Natural resources have become seriously undermined. The Vietnamese government has responded by pointing out various potential solutions (22). The government and related authorities have acknowledged the key role of EE and the urgent need for its development in the educational system of the country. Consequently, through the cooperation between two sectors, the National Education System (under the supervision of Ministry of Education and Training); and Environmental Protection Sector (supervised by the Ministry of Science, Technology and Environment- MOSTE), an EE program has been gradually formulated (22). In 1991, adopting the National Plan for Environment and Sustainable Development (suggested by UNESCO/UNEP), the Vietnamese plan for EE clearly asserted that (24):

"This curriculum (integrated environmental and sustainable development curriculum) should specifically focus attention on the basic concepts of sustainable development. It should also establish the need for specialized degree courses in the field and the training of teachers in this field. Both men and women should have equal access to all training programs.... Development of curricula, syllabus and textbooks should be given high priority for the introduction of environmental education at all levels and the establishment of specialized degree courses in the field of environmental sciences".

EE programs in Vietnam are developed in the light of the Tbilisi Declaration, in which the goals, objectives and methods of EE have been adopted (23). At the higher education level, in particular, on 12 September 1995 MOSTE issued a decision, that study of the environment will be the subject of a national textbook, namely "Environment and Men". Use of this textbook is compulsory for all students in all branches of learning, from natural sciences, humanities, social sciences, agriculture, industry technologies, economic to business management (23). However, there remains substantial work to be done in order to develop more sophisticated and effective EE curricula at all levels in Vietnam.

#### 2.2.3.3 Weaknesses of EE Programs in Vietnam

As in many countries, the educational system in Vietnam faces many challenges (25). EE is one component of this and although EE programs have existed for a decade, it is recognized that current EE programs in Vietnam are not completely satisfactory. The reasons for this are as follows:

- (i) Educational approach: A large part of efforts in EE is oriented to "Education about the Environment", rather than "Education for the Environment".
- (ii) The interdisciplinary approach: Efforts for EE development are segmented in the education and training system, as well as in individual educational institutions. Efforts for the establishment of the teaching of a separate environmental subject are stronger than that for integration of EE into all disciplines at all levels.
- (iii) Administrative problems: There is not adequate national organisation for the coordination of the interdisciplinary, multisectoral activities of EE, and the establishment of the necessary links between EE, environmental training and environmental awareness.
- (iv) Exchange of information: There is a serious lack of information on progress and experience in international developments in EE, and only weak links with international and regional networks of EE (22).
- (v) EE staff, facilities and laboratory equipment for teaching and research: there is a lack of teaching and research staff with strong background in environmental science. In addition, there was reported to be a severe lack of information, references, facilities, and laboratory equipment. Therefore, teaching and researching activities are limited (23).

Acknowledging such unsatisfactory factors in the development of EE program, this study will attempt to design some experiments for use in the Vietnamese situation, so that the perceived weakness of EE in Vietnam will be taken into a large account.

## **CHAPTER 3 ENVIRONMENTAL CHEMISTRY EDUCATION**

Environmental chemistry education (ECE) is a division of chemical education, the laboratory program of which seeks to provide students with the opportunity for "handson" experience of the work of scientists in the professional laboratory. Therefore, this chapter considers the work performed by environmental chemists in the laboratory. The goals of environmental improvement and protection are also discussed. These aims, together with a consideration of international guidelines for environmental education (EE) programs and ECE, form the basis for a critical literature review of past and current ECE laboratory experiments and curriculum. A major objective of this exercise is to expose what ECE laboratory experiments have achieved to date and what remains to be done.

#### 3.1 Environmental Chemistry

It has been argued that the discipline of EC has implicitly paralleled the development of the discipline of chemistry itself (26). However, as with EE generally, changing attitudes relating to EC have resulted in this discipline being regarded differently in more recent times than in the past. Previously, the environmental aspects of chemistry simply referred to the use of the knowledge and skills of chemistry as an underpinning to understand the surrounding environment or in the use of the environment for human purposes. In this regard, EC may be considered to have been initiated by the 17<sup>th</sup> century-French-born chemist, Antoine Lavoisier, who pioneered our understanding of the chemical composition of the atmosphere. Other important discoveries in chemistry of "environmental" significance occurred in the early 19th century (26), such as the

breakthrough in synthesizing organic substances and the use of the valuable "gift" of ammonia from the environment to produce materials of benefit to mankind (26).

Thus, although environmental associations of chemistry have long been recognized, the more recent term "environmental chemistry" was not commonly used until the 1960s. This was when the possibility of adverse effects of chemical discharge into the environment became a significant issue. Indeed, concern for the large-scale negative effects of chemical discharge into the environment were undoubtedly responsible for the initial popularity of EC (27). However, the subsequent development of EC moved far beyond chemical pollutants and became more holistic in nature. EC is now defined as the study of the sources, reactions, transport and the fate of chemical species in all three physical environments namely: air, water and soil; and the effects of these chemical species on the natural environment and humans (26, 28).

#### **3.2 Environmental Issues**

Environmental issues are often driven by public health concerns, which the world was becoming more aware of in the 1960s (29). Of the physical environments of the Earth, the environmental pollution of water and air in particular (rather than soil) were recognized early on. Such public recognition was marked by the introduction of environmental laws and regulations to protect the environment and human health. In the United States for example, the US Public Health Service first established a drinking water standard in 1914. As late as 1963 the Clean Air Act was passed. Only since the 1980s, has the contamination of the soil environment been generally recognized to be a potential health risk for humans (30). Thus, it may be said that, only recently, has the problem of the contamination of soil been given equal status to that of air and water pollution (31). It must be stated, however, that in the educational arena, a consideration of the soil environment still lags behind water and air; as this project demonstrates.

#### 3.2.1 Environmental Issues Relating to Water and Air

It is not surprising that the educational curriculum reflects the above trends. In line with the above development, this project has found that the environmental issues of the water and air environments have been given the major emphasis in EC textbooks and in the educational literature. In particular, the sources of contaminants, effects of pollutants on human health and solutions to reduce the level of such pollutants have been thoroughly discussed (28, 32).

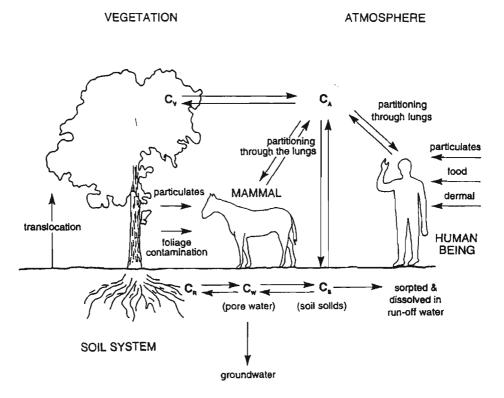
#### **3.2.2 Environmental Issues Relating to Soils**

Soil is the most important part of the geosphere (lithosphere) for human beings and most terrestrial organisms, although it is generally taken for granted. It plays a vital role in agriculture, primarily as a mean to produce food, which is required by most living things, including humans (28). Soil is also a sink for the degradation and disposal of biogenic and anthropogenic wastes. As such it is the receptor of many hazardous chemical wastes including landfill leachate, lagoons, and tanks.

Chemical contamination of soil is extremely diverse and may be categorized into various types (26, 33) (**Appendix 1**). Among the contaminants in soil, petroleum hydrocarbons are among the most common and are currently of major environmental concern. This particular area will be a focus of study in this project and will be further discussed in a later section (3.2.2.1).

Such contaminants may be hazardous for human health when people are directly or indirectly exposed to them through the intake of food grown in contaminated soil, when potentially toxic substances reach ground water used for drinking water supplies, or the volatility of contaminants into the atmosphere (33). Various contamination pathways are summarised in **Appendix 2**. The process of distribution of soil contaminants and<sup>\*</sup>its interrelationship with other environments is shown in the **Schematic** (26):

Atmosphere (C<sub>A</sub>)  $\downarrow \uparrow$ Soil solids (C<sub>S</sub>)  $\downarrow \uparrow$ Pore water (C<sub>w</sub>)



The distribution of soil contaminants in the environment

As alluded to above, a major concern of soil contamination is subsequent water contamination. Thus, the conservation of soil and the protection of water resources are strongly interrelated. Because most fresh water falls initially on soil, to a large extent the condition of the soil determines the fate of the water and how much it is retained in a useable condition (28). Natural disasters, such as floods, can also be the means by which contaminants from soil reach and contaminate water resources and, consequently, constitute a potential risk to human health. A particular example of this is heavy metal contamination of soil after flooding (34, 35).

In summary, the recognition of the interrelationship between air, water and soil is contributing to an increased interest in soil contamination. There is a growing realization of the importance of soil science and soil contamination. Researchers are working towards an understanding of the sources, effects and fates of chemical species in soil as well as the effect of contaminated soil on human health. There is also an increasing interest in the development of remediation solutions for contaminated sites. These concerns are yet to be fully reflected in educational curriculum. In this regard educationalists are lagging behind the scientific professional community as will be revealed by the present study (**3.4.1** and **3.4.2**).

#### 3.2.2.1 Soil Contaminated with Petroleum Hydrocarbons

Of the wide range of organic contaminants in soil, petroleum hydrocarbons are particularly common. These arise from the extensive use of petroleum products in the chemical, petrochemical and transportation industries (26, 36) which give rise to incidental spills and leaking of underground storage tanks. The number and complexity of such contaminated sites are increasing (37).

In the 1980s, the increasing risk to human health of hydrocarbon contaminated soil (38) has stimulated research in this area. Soil contaminated with petroleum hydrocarbons can be hazardous from three different perspectives (37):

- 1. Fire and explosive hazards
- 2. Toxicity with respect to site usage
- 3. Environmental damage, especially to drinking water.

Humans may be at risk when dealing with soil that is contaminated with petroleum hydrocarbons in several ways (38):

- 1. Potential exposure onsite/offsite via ingestion of dirt, inhalation of airborne contaminants, and/ or absorption through skin after dermal contact with contaminated soil.
- 2. A potential risk may also be created when people use water (both surface and ground water) from onsite/offsite, where they may be exposed from ingestion, inhalation and/or dermal contact with the contaminated water.
- 3. Consumption of foods that may be associated with contaminated sites may also pose a risk as the result of bioaccumulation through the food chain.

Details of the toxicity and the health effects of some hydrocarbons which may contaminate soil are given in the table (37) in the Appendix 3.

Because of the potential adverse effects of soil contaminated with petroleum hydrocarbon, risk assessments are often conducted in order to establish if remediation is necessary. Towards this end, the determination of the level of contaminants in soil has become an important task for professional environmental chemists. For these reasons, it is clearly beneficial for students to gain hands-on experience with the analysis of such contaminants in soil in the EC laboratory.

#### **3.3 Environmental Chemists in the Laboratory**

Environmental chemists play a key role in the improvement of the quality of the environment by providing other scientists and decision-makers with information about the level of chemicals and other species in the environment (28, 29, 39, 40). Therefore, having good skills in chemical analysis is considered to be essential for an environmental chemist (28, 41). As with any analytical chemistry procedure, environmental analytical chemistry includes the following key elements: sample preparation, data collection, data analysis, data interpretation and data reporting (29, 42). Environmental chemical analysis is considered to be a particular challenging task because it often requires working with very low concentrations and complex matrices. Furthermore, raw environmental samples are often non-homogeneous (31). In order to produce reliable and defendable laboratory data, which is the ultimate task of environmental analytical chemistry, every aspect of laboratory procedure needs to be carried out scrupulously by highly skilled scientists (29). Therefore, those who aspire to be environmental chemists need to be given the opportunity to be trained in, and to practice these underpinning skills.

In an analytical procedure, the competent use of state-of-the-art instruments has become essential for environmental chemists to help speed up the collection of accurate data (28, 41). However, it must be emphasized that obtaining data is only the beginning. An environmental chemist must also be expert in the processing and interpretation of such data, especially with respect to the application of statistical methods. It must be emphasised that "reportable" data, which is used for scientific and political decisions on environmental issues, is not the raw data obtained from reading instruments. Indeed, raw data may be meaningless to other non-environmental analysts. They are useful and comprehensible only when they are processed (analysed, tabulated, displayed, etc.) using statistical methods (43).

Other important procedures to be learned include quality assurance and quality control (QA/QC), which encompass the whole procedure of the analytical process, from sampling to reporting (31, 44). Evidence of QA/QC may be required to support the quality of the data (29, 31, 45, 46).

#### 3.4 Environmental Focii of ECE Materials

The following investigation is aimed at analyzing the ECE curriculum in order to assess the relative emphasis, which has been placed on the three environments of water, air, and soil. The two main sources of information are EC textbooks used worldwide and the international journals: *Journal of Chemical Education* and *Chemistry in Education*. These journals are considered to be the highest profile mainstream publications, which incorporate the discipline of chemical education.

#### **3.4.1 Environmental Chemistry Textbooks**

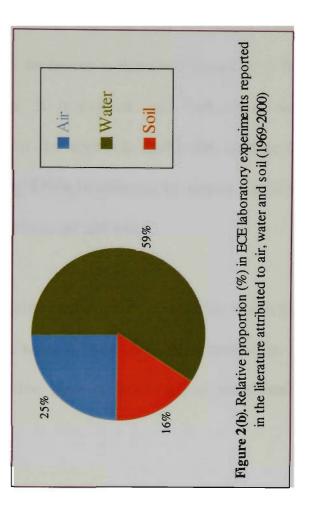
A survey of prominent internationally available EC textbooks for undergraduate students (from 1972-2000) has been conducted. Twenty-nine such textbooks have been reviewed (**Appendix 4**). An evaluation has been made of each textbook by counting the number of pages, which are devoted to each environmental domain in such material. An analysis of this survey is given in, and **Figure 1(a)** and **(b)**.

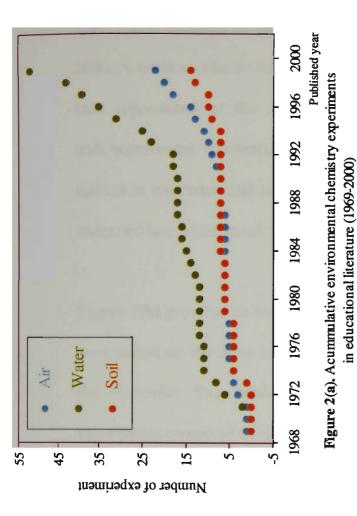
Figure 1(a) shows that since 1972 there has been a significant increase in published textbook material in the area of EC. However, it can be seen that the attention, which

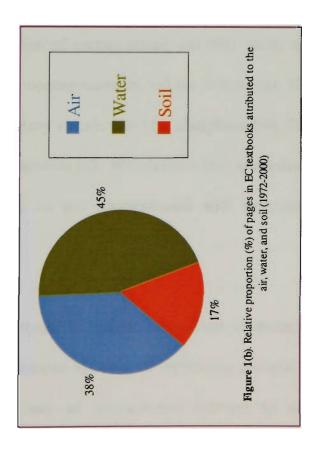
has been paid to the soil environment, has lagged behind that of the other two environments (air and water). Figure 1(b) provides an alternative representation of the data. This data is consistent with a general failure to recognise that contaminated soil poses as great a health risk as contaminated air or water. Indeed, such concern for contaminated soil only started to emerge in the 1980s and this is reflected, perhaps, by the climb in the soil curve of Figure 1(a) from the early 1990s. Figure 1(a) also shows an escalation of interest in the water and air environments since 1991. This reflects the upsurge of general interest in environmental issues in the 1980s.

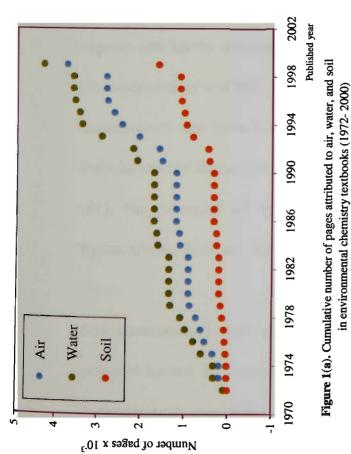
#### 3.4.2 Environmental Chemistry Education Laboratory

EC experiments play an important role in giving students a sound understanding of the environment. Indeed, it may be argued that one cannot appreciate environmental processes properly without doing practical laboratory-based work (47). Therefore, EC laboratory programs are considered to be a vital component of EC courses (48). Whereas a large number of undergraduate EC textbooks have been published since 1972, educational material for EC laboratory-based work is insufficient and therefore needs to be expanded. Much of the development in this area to date has been reported in the international literature, primarily in the *Journal of Chemical Education* and to a lesser extent in *Education in Chemistry*. A thorough survey of these journals over the period 1969-2000 has been conducted as part of this study. The results are shown in **Figure 2(a)** and (b).









**Figure 2(a)** presents an analysis of EC experiments reported over the period 1969-2000. A trend similar to that observed for EC textbooks is apparent. Thus soil is the least represented of the three environments. It is evident here that air is also underrepresented compared to water. As with the textbook study, the upsurge of interest in environmental issues from the early 1990s is reflected by a steep climb in water and less pronounced, but significant, climbs in air and soil.

**Figure 2(b)** provides an alternative representation of the relative emphasis, which has been placed on the three environments. Here, water is even more represented than in the textbooks. This could reflect the relative ease of constructing water-based experiments compared to air and soil.

#### 3.4.2.1 Soil Related Experiments in ECE Laboratory

Fourteen individual soil-related EC experiments and three laboratory courses have been reported in the educational literature since 1972. The primary goals of the laboratory experiments are to demonstrate the remediation of contaminated soil (49), study the characteristics of soil (48), and to analyze the contaminants in soil (or sediments). The contaminants that have been analyzed are heavy metals (50-54), phosphate (54, 55), Polychlorinated Biphenyls-PCBs (56, 57), pesticides (58, 59), atrazine (60) and sulfide (61). No examples of experiments relating to soil contaminated with petroleum hydrocarbons, however, have been reported.

Soil contaminated with petroleum hydrocarbons is widely recognized as being a potential hazard for human health and has become an issue of increasing concern to environmental scientists. The current absence of experiments relating to soil contaminated with petroleum hydrocarbons may be considered a deficiency in the international EC curriculum. Therefore, there is a need to develop such experiments for students in EC courses. In particular, there is a distinct need to develop such experiments for developing countries in general and for Vietnamese universities in particular. The Vietnamese context is a particular consideration for this study. In Vietnam, the petroleum industry is developing rapidly. The increasing transportation and storage of petroleum products are likely to lead to contamination of both water and soil. The current study, therefore, will attempt to develop EC experiment(s) for use in the chemistry laboratory that will specifically demonstrate the analysis of soil contaminated with petroleum hydrocarbons. These experiment will be based on the experience gained by the author during a five month work experience program at Australian Government Analytical Laboratories (AGAL), and will also draw on a survey of Vietnamese academics in order to place the experiments in a Vietnamese context.

#### 3.5 Practical Work in the Chemical Education Laboratory

# **3.5.1** Objectives of a Chemical Education Laboratory

The science laboratory has been considered to be an important place for the teaching of "real" science. Science subjects in general and chemistry, in particular, are unthinkable without practical work. The role of the laboratory in chemical education has been viewed as so critical that "chemistry without lab work was seen as a body of factual information and general laws, which conveyed nothing of lasting power to the mind. Its educational potential could only be realized in the lab" (62). One may even say, "the chemistry teacher without a lab was like a Jewish mother without chicken soup" (63). The primary aims of the practical laboratory which have been clarified are (62, 64, 65):

- 1. Conceptual learning: Laboratory work is used extensively to develop students' conceptual learning and understanding of science.
- 2. Techniques and manipulative skills: Laboratory work provides students with the opportunity to gain hands-on experience, using appropriate instruments and apparatus and associated techniques.
- 3. Investigation skills: in the context of laboratory work, investigation skills include: planning an investigation, processing and interpreting data, and evaluating findings.
- 4. Affective objectives: the affective objectives of laboratory work can be divided into two main categories:
  - Attitudes to science include interest, enjoyment, satisfaction, confidence and motivation toward the study of a subject. Its important role is evidenced by the study of what factors affect students' attitudes to science. Two of the important factors were (i) the availability of the facilities in the laboratory; and (ii) the time allowed for students to perform the experiment.
  - Scientific attitudes refer to styles of thinking such as objectivity, criticalmindedness, scepticism, and willingness to consider the evidence.

From the literature, the two important approaches, which have been favorably implemented in the existing chemical education laboratory in order to achieve the above goals, were: the scientific approach and the integrated approach. They will be further discussed as follows.

# **3.5.2 Chemical Education Laboratory Approach**

#### 3.5.2.1 The "Scientific" Approach

Traditionally, a science teaching laboratory has been considered to be a place where teaching of "real world" science was undertaken (66). Such an approach is not only

designed to develop practical scientific skills but is also intended to engender more enthusiasm and to improve investigative and problem solving skills (63, 67-70). Thus, science educators in general and chemical educators in particular have strived to introduce experiments into the laboratory which reflect what the students will be likely to encounter in a professional environment (62, 70). However, the criticism that practical work for science undergraduates fails to reflect the way scientists work in practice still prevails (62, 71), and it has been suggested that this contributes to a major lack of effectiveness in science education (62).

The above consideration relating to the real world applicability of scientific education is termed the "scientific" approach. This approach, in the chemistry laboratory in particular, may be characterised as follows (63):

- Experimental design and execution, including, selection use and optimisation of apparatus and instrumentation as well as observation, measurement, and data management.
- 2. Scientific thinking skills: data analysis, making inference.
- 3. Direct objective knowledge of the natural world.

#### 3.5.2.2 The "Integrated" Approach

The conventional format of chemistry laboratory courses has involved dividing the disciplines into the areas of organic, inorganic, physical and analytical. The recognition of the many limitations in running such laboratory programs separately has led to the proposal of a different format, the so-called "integrated experiment" (72, 73). Integrated experiments (unified laboratory or integrated laboratory), in contrast with the traditional style, bring together several chemical branches into one experiment and

therefore, enable it to be used in any individual relevant chemical laboratory program (74). The development of the integrated laboratory program was considered to be a reform and an innovation in the chemistry curriculum (74, 75). Indeed, two careful surveys have been conducted to examine the perspective of the integrated approach in the chemical laboratory. The results of these surveys agree on the benefits of the new laboratory approach (74, 76). Such advantages are discussed below:

Compared with traditional experiments, the integrated approach provides students with a more holistic view of chemistry and a more realistic picture of what chemists actually do in the real world (thus encompassing the scientific approach). This has the advantage of better preparing a student for his/her professional role. As the integrated laboratory covers practical aspects and principles of more than one branch of a discipline, students are able to gain more realistic experience and knowledge from a given experiment. The integrated nature of this new style of laboratory program may also provide students with enhanced problem-solving skills. These aspects will help students gain enhanced knowledge and confidence for their future chemical employment. The integrated approach may also impart benefits to the institution by providing a more efficient utilization of resources.

Some disadvantages, however, have been discovered in using the integrated laboratory approach. These include the time commitment required to develop appropriate experiments, and the difficulties for students with insufficient background, since a broader background in chemistry may be required (74, 76).

Although some difficulties are expected in running an integrated laboratory program, its long-term advantages to the students in particular are considered to outweigh these (76). There is now a significant number of integrated laboratory programs that have been developed. Some examples, which replace traditional laboratory programs, have been conducted successfully. For example, the integration of physical and organic chemistry has been developed for first-year courses (77). The combination of three areas of chemistry, physical, analytical and organic has also been undertaken (78). An advanced program that integrates the four branches of chemistry has also been developed (79). The promise of the integrated laboratory approach is reflected in the support given by the National Science Foundation, United States of America-USA, to develop such integrated laboratory experiments (80, 61), and a recent report suggest the potential of such programs (82). Therefore, it is advisable when developing new chemistry-related laboratory courses to take the integrated approach into account.

#### 3.6 A Critical Review of the Literature on ECE Laboratory Experiments

This review of the literature on ECE laboratory experiments is also based upon the principle that EC is in part a chemistry subject. Therefore, the objectives of the ECE laboratory are taken to model those of the chemical education laboratory. The two approaches, namely the integrated and scientific approaches, which have been used in the chemical education laboratory, are therefore also applicable in the ECE laboratory. Moreover, the ECE program is also a part of the broader EE program. Thus, the objectives of the EE program should also be taken into consideration in the design of ECE laboratory experiments.

A critical review of ECE laboratory experiments in the international literature has been undertaken in order to find out what previous ECE experiments have focused on, and what are the limitations of such programs. The first answer will play an important role in the formulation of the "initial" criteria for the survey of Vietnamese EC educators to find out the "consensus criteria" for the development of the ECE laboratory program in Vietnam (**Chapter 5**). The second answer will help identify the need for improvements for the current ECE laboratory programs as well as the need for a basis in development of experiments for use in the Vietnamese context (**Chapter 8**).

# 3.6.1 Towards Reflecting Objectives of Chemical Education Laboratory

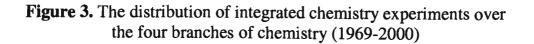
#### 3.6.1.1 The Use of "Integrated" Approach

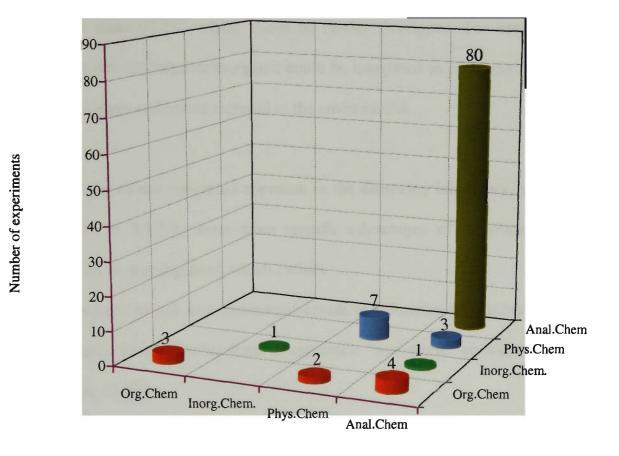
EE in the educational context in general, and EC within the subject of chemistry in particular, are considered to be among the most integrated of disciplines. EC is considered a holistic subject compared to traditional chemistry, which has strictly defined areas of organic, physical and analytical chemistry (26). The successful application of the integrated approach in traditional chemistry has heralded certain possibilities that can also apply in the EC laboratory.

A careful survey of existing EC experiments and EC laboratory programs reported in the international chemical education literature shows that a large majority of such experiments have employed the integrated approach. For example, combining EC with one traditional area such as inorganic or organic. In some experiments the integration of three branches has also been developed such as the integration of EC with two areas: (i) physical and analytical chemistry; (ii) inorganic and analytical chemistry; (iii) organic and analytical chemistry; or (iv) organic and physical chemistry (**Figure 3**). The detailed survey is illustrated in **Figure 3**. Raw data is given in **Appendix 5**. Of the 116 reported experiments 87% have been used in combination with other traditional chemistry branches. Among them, analytical chemistry has been used with the highest frequency, with 88 experiments or almost 80%. Analytical chemistry appears to be the most versatile as it can be combined with any of the other the chemistry branches. Indeed, it has been also used in connection with inorganic, organic and physical chemistry; even creating the triple-function experiments (environmental-analytical-inorganic chemistry; environmental- analytical- organic chemistry; environmental-analytical- organic chemistry).

Unlike analytical chemistry, the integration of EC with any one of the three remaining branches of chemistry, namely organic, inorganic, and physical chemistry constitutes only a small proportion of the published experiments. Of these, the largest number of integrated experiments has occurred between environmental-physical chemistry branches and the least number has occurred between environmental-inorganic chemistry branches.

The prominent use of analytical chemistry in combination with EC is not surprising, given that analytical chemistry for measurement of levels of contaminants in the environment is the primary tool of professional environmental chemists (3.3).





Abbreviations:

Org.Chem: Organic chemistry; Inorg.Chem: Inorganic chemistry; Phys.Chem: Physical chemistry; Anal.Chem: Analytical chemistry. The survey has also considered the international experience in the development of EC laboratory programs at the undergraduate level. To a large extent, the aims have been to develop and improve students' practical skills in environmental analytical analysis, in which the primary focus is in the use of instrumentation to determine the concentration of chemical species in the environment (**Appendix 6**). Figure 3 can provide some useful insights into areas of EC, which could be further developed. For example, organic/inorganic or physical/organic/inorganic could be integrated in EC experiments on the importance of clays and humic material in the environment.

Besides the advantages of the integrated approach in the chemistry laboratory, which have been discussed in **3.5.2.2**, some other specific advantages of integrated EC experiments have also been recognized, which include:

- (i) Enabling students to use and understand chemical principles and techniques, and at the same time to enable them to appreciate "real world" environmental problems (56, 83-85).
- (ii) Increasing students' interest in environmental issues without increasing the time commitment of teaching staff (56).
- (iii) Creating more motivation and enthusiasm in students, since the chemical principles are more related to the "real world", whilst maintaining the relevancy and vitality of the program (86).

Thus there are advantages for both the student and the institution in adopting the integrated approach.

#### 3.6.1.2 The Use of the Scientific Approach

In an early chapter (3.3), it was argued that an important component of environmental chemistry is chemical analysis. Professional laboratory work related to this includes sample preparation, data measurement, analysis, interpretation, and reporting. More importantly, QA/QC is always conducted in any procedure to ensure they are all under control. The following discussion considers the extent to which ECE laboratory programs have included these tasks.

In this regard, the author of this thesis has had a valuable five-month work experience program in a professional environmental chemistry laboratory, in order to gain exposure to and experience of the work undertaken by professional chemists. This experience, together with the review of literature (reported above) provides the required comprehensive background necessary to evaluate the existing curriculum in ECE laboratory programs and to further develop this curriculum.

#### (a) Sample Preparation and Using Real Samples

For the production of reliable and defendable environmental measurements, which is the crucial role of environmental chemists (**3.3**), the sample preparation process contributes substantially to the uncertainty in analytical results (86). Given the importance of sample preparation in professional environmental practice, it is of concern that this procedure is often ignored in the educational laboratory (88). Although some recent textbooks have covered this issue, traditionally students have been only told of the importance of sample preparation and have rarely had a chance to practise this in laboratory classes (89, 90). Indeed, in the traditional undergraduate analytical laboratory the sample is normally provided as a matrix spiked with a high concentration of a single analyte. Thus, there is no need for the student to refer to any sample preparation to ensure that the sample is representative (88, 91). With regard to environmental samples, the problem is even more complex as environmental samples usually have relating low levels of pollutant, whose behaviour may be influenced by the background (41). Therefore, the use of the traditional approaches cannot reflect real environmental issues in this regard.

A recent study has shown that the failure to use real-world examples in the science laboratory is a reason for students' decreased interest in science courses (92). Given such observations, it is strongly suggested that the ECE laboratory use real-world samples and include sample preparation procedures so that students have the chance to practise these challenging skills.

Although the use of real-world samples may provide great benefits for students, some disadvantages may also be expected. They are as follows:

#### Advantages

The inclusion of real-world issues that requires the accessibility of real-world samples provides great benefits for students. It helps engage students with the realm of the real life applications rather than just with "pure" theoretical study. It has been found that by dealing directly with real-world samples and real-world issues students become more interested and curious about what they are studying and doing in the laboratory (67, 68). More importantly, it gives students an appreciation of the challenges involved (68). It therefore, increases the students' confidence in working with these and related issues upon graduation.

#### Disadvantages

The disadvantages of using real-world samples are: (i) they involve having to deal with matrix effects and low-levels of chemical species; and (ii) they may create a challenge for the instructors as, unlike synthetic samples, the content of analyte is unknown. However, the benefits to students in using real-world samples outweighs any disadvantages of "real" scientific processes and connect the student with real-world issues rather than "cookbook" methods and theoretical reflection. Indeed, there is an increasing trend in the use and application of real-world samples in the ECE laboratory reported in the chemical education literature (92-95).

#### (b) Using Instrumentation in Analysis

In the EC laboratory, the use of state-of-the-art instrumentation has become an important tool to help environmental chemical analysts produce the most reliable results in the shortest possible time (3.3). In the education laboratory, the utilization of modern instruments has become one of the main concerns of chemical educators in general, and of environmental chemical educators in particular (see the results of the instruments survey in **Appendix 6**. Indeed, it is argued (96) that because modern science uses instrumental methods so extensively, students should be encouraged to experience this aspect of science as much as possible. Moreover, it is modern instrumental techniques that can help students have favourable expectations about the subject that they study. In this way students may attain a better attitude towards the subject when their expectations are met. Environmental investigation, in particular, requires the employment of relatively advanced techniques. The employment of instruments enables students not only to be exposed to the technology, but it can also provide them with a good opportunity to practice dealing with data: in particular,

analyzing data, presenting data in a comprehensible manner and consequently interpreting the data. It is not surprising, therefore, that a significantly large number of EC experiments in the literature have utilized instruments, particularly in the area of environmental analytical chemistry. The result of the survey is in **Appendix 6**.

Of the 88 environmental analytical chemistry experiments surveyed, 75 actually utilize analytical instruments. Of these, 52 have employed relatively modern and sophisticated instruments and were the most frequently used such as gas chromatography (GC) with different types of detectors (mass spectrometry-MS; electronic chemical detector-ECD; flame ionization detector-FID; pulsed discharge photoionization detector-PDPD; thermal conductivity detector-TCD and nitrogen phosphorous detector-NPD). Atomic absorption spectroscopy (AAS) is also another instrument that has been used extensively (13 of the experiments). Other modern instruments used include UV-Visible spectrophotometer and infrared (IR) (4 experiments); high performance liquid chromatography (HPLC) (3 experiments); Fourier transform infrared spectroscopy-FTIR (2 experiments), and a fluorescence and spectrofluorimenter in one experiment.

#### (c) Data Analysis and Interpretation

The important role of data analysis and interpretation of data in producing comprehensive and reliable results in the EC laboratory has been discussed (3.3). The undergraduate chemistry laboratory, however, has been criticized on the grounds that being deficient in these areas, it does not reflect the normal industrial laboratory (69, 71). Indeed, the procedures that students follow do not always illustrate the procedures that professional chemists actually use in the laboratory. In the undergraduate laboratory, while the concept of statistical analysis may be introduced as an important

part of the evaluation of experimental data (97), students are normally instructed to collect data but are not required to interpret and understand the meaning of the results (71, 98). The lack of practice in the analysis and interpretation of data in the undergraduate laboratory, therefore, limits a deeper understanding of the usefulness of the experiments.

Given the important role of data analysis and data interpretation processes in the environmental chemistry laboratory, the lack of consideration of these issues in the undergraduate analytical laboratory suggests that much more attention should be paid to these issues including creating an awareness in students as to why they are important (e.g. relevance to decision making).

#### (d) Quality Assurance and Quality Control in EC Laboratory

The important role and the regulated use of QA/QC in the environmental analytical laboratory, to ensure that data are valid and legally defendable, have been discussed (3.3). However, no chemical education experiment has been reported to date, which demonstrates the importance of this process to students (99). Given the intensive practice of QA/QC in the real science laboratory, the lack of consideration on this issue in the chemistry teaching laboratory has been criticised as being a serious deficiency in the chemistry curriculum in general and in the EC laboratory in particular (93, 99). Insufficient demonstration of procedure in the undergraduate chemical and EC laboratories have been blamed for the misunderstanding of the pivotal role of this process by students (46, 100). The occurrence of this problem in the EC laboratory must be considered to have negative effects on students' future research and/or employment (93).

Several attempts have been made to try to integrate QA/QC into existing programs (46, 99) by emphasising new experiment design (100), or even by creating a new course focusing on this issue (93). It has been found that doing QA/QC in the laboratory helps students improve their problem solving skills, as QA/QC enables students to use data analysis to identify the source of the uncertainty and evaluate the quality of their own results. This has also been observed to make students more enthusiastic about their laboratory work (46). Moreover, students become more confident in related QA/QC work when they come to apply their knowledge (46, 93), rather than having to build this up "on the job".

Therefore, for the sake of the students, it is suggested that more attention should be paid in providing students with opportunity to practice QA/QC in the chemical education laboratory in general and in the EC education laboratory specifically.

#### 3.6.2 Towards Reflecting the Objectives of EE

As ECE is one part of EE, the objectives of EE also need to be considered in the ECE laboratory. The five objectives of the EE program, which are set by the UNEP-UNESCO in the Tbilisi Declaration for international use in the development of any EE program, have been presented in **Chapter 2**. They include:

- 1. *Awareness*: to help students become aware of the environmental issues and its allied problems.
- 2. *Attitude*: to help students to become concerned for the environment and to be willing to participate in the improvement and protection of the environment.
- 3. *Knowledge*: to help students gain a variety of experiences in and acquire basic understanding of the environment and its associated problems.

- 4. *Skills*: to help students acquire the skills for identifying and solving environmental problems.
- 5. *Participation*: to provide students with an opportunity to be actively involved at all levels in working toward resolution of environmental problems.

This chapter has considered the components of the ECE program needed to achieve the objectives of the undergraduate chemistry curriculum, namely: learning of concepts, investigation skills, technique and manipulation skills and the development of affective qualities. The integrated and real-world scientific approach in the ECE laboratory provides students with opportunities to: (i) practice with real world environmental samples; and (ii) use instrumentation to measure chemicals in the environment.

It has been found that by doing so, students not only gain basic knowledge of the environment in the context of chemistry, they also gain experience on how to use their knowledge in identifying environmental problems and using their knowledge and practical skills to solve them. It has also found that by dealing with real environmental problems, students are more enthusiastic and are more confident when they graduate to participate in the related environmental science. Therefore, if the real-world scientific and the integrated approaches are applied effectively in the ECE laboratory, its goals will coincide with the goals of EE programs (objectives 2-5, **Chapter 2**).

Towards achieving objective 1, the appropriate guidelines, which have been given among the 12 guiding principles (Chapter 2), are:

"EE should be interdisciplinary in its approach...; examine major environmental issues from local, national, regional and international points of view so that students receive insights into

environmental condition in other geographical areas; and focus on current and potential environmental situations ...".

The current environmental issues have been discussed in **3.2**, in which the major worldwide problems are soil contamination, water and air pollution. Therefore, the following section will consider international, national and local environmental issues in the ECE laboratory program. The three environmental problems of air, water and soil will also be discussed.

# 3.6.2.1 International, National and Local Environmental Issues in the EC Laboratory

In the review of the literature on ECE laboratory experiments, some experiments have suggested the use of local environmental problems as interesting materials for further development (51, 56). The inclusion of such issues in an experiment enables educators to increase the students' interest as they realize the relevance of chemical knowledge for understanding current and familiar environmental issues (86). Also, it encourages students to use their knowledge to propose possible solutions for improving their local environment (86). That is it enables to enhance students' problems solving skills.

#### 3.6.2.2 The Inclusion of Environmental Issues of Air, Water and Soil

Regarding the environmental focus (e.g. air, water and soil environment) in the ECE materials, a thorough survey of all available ECE experiments since 1969 has been carried out and reported (3.4.2). This has shown the serious under-representation of the soil environment in ECE laboratory programs in comparison to air and water. Given that soil contamination is an environmental issue of major concern to environmental scientists and society, this finding has been argued to be a serious deficiency in the

current EC curriculum. In other words, an ECE laboratory program designed to achieve the objectives of EE program, needs to be balanced and pay more attention to soil environmental issues.

#### 3.7 Conclusions from the Literature

From the survey of the literature, it has been found that, there is an increasing interest in the development of ECE laboratory experiments by environmental educators. The two approaches: integrated and scientific, which have been used in the chemical education laboratory, have been used with great success and can be suggested for inclusion in ECE laboratory programs. The successful use of these two approaches toward achieving the objectives of the chemical curriculum only meets the objectives of the EE program when it considers the current environmental issues of water, air, and soil at all levels (international, national, and local). In other words, in order to reflect both EE and environmental chemistry in ECE laboratory programs consideration must be given to:

- 1. The environmental issues of international, regional, national and local.
- 2. The environmental issues of air, water and soil environments.
- 3. Use of an integrated approach.
- 4. Providing students with opportunities to practice appropriate instruments and apparatus.
- 5. Use of real samples and scientific processes.
- 6. Helping students with opportunities to improve the following environmental chemistry skills: sample preparation, measurement, data collection and analysis, interpretation and report data, along with the QA/QC procedure.

Regarding the objectives of EE and the objectives of chemical education to be incorporated into ECE laboratory programs, the literature has found two major problems, which are considered to be serious drawbacks for the current EC curriculum:

- 1. The serious lack of material for the study of the soil environment among the three physical domains (soil, air and water), particularly, with regard to the inclusion of the environmental soil contamination in teaching laboratory experiments.
- 2. Insufficient practical materials relating to sample preparation, data analysis and interpretation, and QA/QC.

These findings provide valuable information for the development of the following parts of this study. The identified objectives of the ECE laboratory are used as the "initial" criteria in order to find out the specific requirement for the development of ECE laboratory in Vietnamese universities in **Chapter 5**. The two approaches to ECE laboratory program will be used, coupled with the results from **Chapter 5** to develop satisfying ECE experiments. In the development of the experiments, the researcher will ensure that the reported problems of EC curriculum will be excluded from the proposed experiments.

#### **CHAPTER 4** THE DELPHI METHOD

In Chapter 2, it was noted that the inclusion of appropriate laboratory experiments was an important requirement in the development of a curriculum in general and environmental chemistry suitable for Vietnam. There is an increasing interest in the development of such experiments among environmental chemistry educators internationally (Chapter 3). Experiments designed for developed countries, however, may not be suitable for the use in the Vietnamese context. In this research, in-person testing of the applicability of particular experiments for the Vietnamese context is not possible. Thus, an alternative is to employ a strategy, which seeks to generate a set of criteria for the development of experiments suitable for Vietnam. The Delphi research method has proved to be a useful approach in tasks such as this.

#### 4.1 Description of the Delphi Method

The Delphi technique was first used by the Rand Corporation in the USA in the 1950s as a new and interesting tool for scientific and technological forecasting (101, 102). Since then, this method has been significantly developed and widely implemented in other areas such as industry, government, defence and education (101-103). The Delphi technique is described in terms of its procedure. It can generally be defined as: "a method for the systematic solicitation and collection of judgements on a particular topic through a set of carefully designed sequential questionnaires interspersed with summarised information and feedback of opinions derived from earlier responses" (104).

Depending upon the method of conducting Delphi research, two different forms of the Delphi technique have been classified. The most popular one is the paper-and-pencil version or "Delphi exercise", also named the "conventional Delphi" (105). In this form, the directors prepare questions relating to the issues of concern and send them to each expert anonymously and confidentially. Participants' responses are then analyzed and summarized by the researcher who then prepares questionnaires for the next round. In the second step, the list of the group's viewpoints is re-circulated to the respondents for further consideration. The process is repeated until consensus (or the convergence of opinions) is gained. Generally, a consensus is reached after two rounds, "feedback sessions" (or "iterations") (103). A newer form, is the so-called "Delphi conference", in which the manual questionnaire distribution, summarizing and analyzing of the conventional Delphi have been replaced by a computer program. Because of its speed, it is also referred to as "real-time Delphi". Real-time Delphi, however, has not been used extensively because of its complexity and its inflexibility in terms of tailoring the computer software to the project (105).

#### 4.2 The Delphi Processes

# 4.2.1 Expert Panel Selection

## **4.2.1.1 Definition of Expert**

It is suggested that great care should be taken in selecting the panel of experts, in order to avoid a weakening in research validity which may result from an unequal level of expertise of the participants (102, 106). The "expert" may be distinguished from the non-expert by applying the following three criteria (107):

(i) Experts are people who have sufficient knowledge and experience to have mastered the advanced skills of a particular domain of knowledge

or experience.

- (ii) Not only do experts have a special skill, they are proficient in their actions and they have special ways of applying their knowledge to a task in their area of expertise.
- (iii) Experts are also proficient at identifying problems in their areas and then being able to tell if the problems are solvable. When the problems are solvable, the experts can propose appropriate solutions.

#### 4.2.1.2 The Size of the Panel

The optimal number of experts to make up a Delphi panel is not clearly specified in the literature. It is understandable that the larger the panel size the more likely a better result will be achieved. Thus, there should be no upper limit to the panel size. The largest panel recorded since 1993 was 1685 (102). The lower limit of the panel, however, is of a concern. The minimum number of participants is, indeed, somewhat unclear in the literature. It has been suggested that the panel size should be no less than ten (104). However, a panel of as few as eight has been reported to produce valuable information in the development of the content of a course in clinical pharmacology in the United Kingdom-UK (108).

#### 4.2.2 Questionnaire Preparation

#### 4.2.2.1 First Round Questionnaire

Traditionally, the Delphi method employs a set of open-ended questions in the first round. The result of this will be the basis for follow-up questionnaires. This traditional format has the advantage that it provides participants with the freedom to express their opinions (109). The most common modification of the traditional Delphi method is the use of the structured questionnaire, in which participants are asked to modify, add or delete items in a prepared list (107, 109), instead of providing answers to open-ended questions. This format is sometimes preferred because it can help save time and expense (104). More importantly, it enables the participants to focus their attention immediately on the issues (104, 109). The structured questionnaire may be prepared with reference to a literature review (107, 109) and/or by careful consultation with selected members of the panel (106).

#### 4.2.2.2 Second and Following Rounds

In the second round, participants are asked to rank their agreement on each item, considered by the group in the first round. They are also invited to add further comments. A multi-point (4, 5, 7 or 9 points) Likert scale is typically used for the rating responses (104). If a subsequent round is needed, the panel members are normally sent a questionnaire together with a report of the previous round responses and the comments of the whole group. They are then asked to review the report and re-rate or re-rank each item.

#### 4.2.3 Data Analysis and Interpretation

The meaning of "consensus" is arbitrary from study to study. The dictionary definition for consensus is "a general or widespread agreement" of opinion (110). "Consensus" therefore, may normally be considered to be achieved when it has greater than 50% support on a specific issue. Indeed, a setting for consensus varies dependent upon on the researcher(s). Consensus criteria ranging from as low as 55% to 100% have been reported (102). The final result of a specific Delphi study is a list of items, which reach an adopted consensus.

#### 4.2.4 Final Step

When the final round of the Delphi study is completed, an analysis and summary is prepared followed by a final report. It is strongly recommended that the researcher send the final report to each participant (104, 107). By doing so, the participants will be acknowledged for the time they have devoted to providing their professional knowledge and insights to the study. Thus, they are more likely to contribute their intellectual commitment to further studies (107). In addition, serious errors or discrepancies might be exposed.

#### 4.3 Advantages of the Delphi Technique

Compared to other techniques, which utilize face-to-face discussion, such as committee meetings or group discussion, the Delphi approach enables the elimination of certain psychological aspects. It is considered to be a quiet, thoughtful conversation, in which every one gets a chance to listen to their peers instead of working alone. It therefore, encourages freedom for the expression of individuals' opinions without group pressure (101, 105). The Delphi survey method is particularly worthwhile in the case where face-to-face discussions cannot be organised. It is not always possible to have all interested participants in one place due to either their time availability or the cost of organizing the meeting. With the purpose of gathering experts' opinions, the Delphi method enables the organizers to reduce costs as it eliminates travelling expenses of the participants in order to attend meetings. Mail, the alternative way to communicate with the expert panel, is relatively cheap. The Delphi technique is also well known for its time effectiveness. It helps save time, not only for the participants, but also for the researchers because it eases administrative tasks (101). The Delphi method is also flexible and versatile in its application. It can be employed in any situation as long as there are experts in particular field of study who are available (101, 102).

The quality of the results is one of the strengths of the Delphi method. It is assumed that the decisions made by a group are normally more valid than that made by a single person, "several heads are better than one" (107). Another factor that the participation in a Delphi study is by people who are experts in the field, therefore, research validity is enhanced. It has been reported that results from Delphi studies are more likely to be accurate than ones achieved from face-to-face discussion (107).

#### 4.4 Limitations of the Delphi Technique

Some criticisms of the Delphi method relate to the expert panel, which is considered to be a potential source of bias (101). As the method involves the collection of participants' opinions and their ideas are equally treated, the size of the panel, sampling method and the relative quality of members are, therefore, important factors which might affect the correctness of the final result. Regarding the expert factor, it is claimed that the lack of making clear the criteria for the inclusion of panel members prior to conducting the survey is a source of bias in the final result. Indeed, it has been found that not many previous studies have paid attention to this (102). Therefore, it may lead to a distortion of the result. Other concerns, including the number of participants and how they have been chosen have been thoroughly reviewed (102). It has been found that there is no specific regulation in this regard. In other words, the researchers may struggle to decide what is the best number for a panel and what to do with a larger number of available experts than they can handle in order to generate the most reliable result. It has been considered, to a large extent, that the Delphi method enables reduce administrative time compared to face-to-face meetings. However, some Delphi users might find, to a lesser extent, that the time they spend for collecting and distributing questionnaires is significant because of the sequential of the Delphi study (111, 112). It may be expected to take up to 5 months to finish the necessary rounds (104).

Some other potential difficulties of the Delphi method include (104):

- 1. The panel members may be influenced by the prepared questionnaire.
- The expertise of the participants may not be fully utilised because of the absence of face-to-face discussion.
- 3. Panel members may not fully understand the purpose of the study.
- 4. The lack of participants' effort in fully taking part in the study may lead to distortion of the chosen panel.

#### 4.5 Questionnaire Distribution Methods

Traditionally, the Delphi questionnaire is distributed by mail (101). However, the personal interview, and recently at the interactive level, on-line computer, electronic mail (e-mail) and facsimile (fax) have also been employed. The use of mail has been popular because of its simplicity and cost effectiveness. However, it is a relatively slow method of communication between researchers and participants. It normally, takes four weeks to complete a round. The alternative methods, which are significantly faster than post, are e-mail and fax. They have been used successfully in some recent studies (107, 113). While in the developed countries, fax or computer with fax modems are very commonly set up in industries and universities, they are not, however, widely available in developing nations such as Vietnam. Therefore, the above alternative methods are not yet applicable in the case of Vietnam.

# 4.6 Applications of the Delphi Technique in Educational Research

### 4.6.1 Context for Application

The Delphi technique has various applications and can be particularly useful (105) when:

- A question does not lend itself to precise analytical techniques but can benefit from collective subjective judgements.
- There is a requirement for individuals to take part in the study of a complex problem, which has not to be subjected to prior research and which may require the participation of experienced people with a diversity of expertise.
- Time and cost make frequent group meetings difficult or impossible.
- The potential for disagreement makes it desirable that the communication process is refereed and/or anonymity assured.
- The heterogeneity of the participants plays an important role in ensuring the validity of the results.
- There are difficulties in organizing a face-to-face group meeting, especially when the group is large.

The Delphi method has been used in a number of educational studies such as educational planning, administration, curriculum evaluation, and to develop curriculum criteria (102-104). It can also be a useful tool in the following areas (107):

- To determine or develop a range of possible program alternatives.
- To explore or expose underlying assumptions or information leading to different judgments.
- To seek out information which may generate a consensus on the part of the respondent group.

- To correlate informed judgement on a topic spanning a wide range of disciplines.
- To educate the respondent group as to the diverse interrelated aspects of the topic.

#### 4.6.2 Educational Curriculum Development

For the development of educational curricula, a significant number of studies employing the Delphi method have been reported (106, 108, 115). In particular, it has been applied widely in the development of nursing and medical educational programs. For example, Walley, *et al.* (108), have conducted a successful Delphi survey in order to develop a core curriculum in clinical pharmacology and therapeutics for universities in the UK. Such a specific program was not available in UK at the time of the authors' study. The researchers, therefore, prepared structured questionnaires based on a program that had been done in USA. Questionnaires were administered by using post and fax to all expert panels. In this study, participants were asked to rank their agreement on each point in the list. After four Delphi rounds, with the participation of eight experts in the field, who were the heads of academic departments of clinical pharmacology and therapeutics around UK, the core curriculum was formulated and suggested to be particularly relevant to the UK's context (108).

Another example of the application of the Delphi method to curriculum development is in chemical education (111). In response to the immediate need for a new chemical program for the graduate and upper undergraduate level, researchers at the University of Texas applied the conventional Delphi technique to curriculum development. Their ultimate task was to obtain consensus among chemical educators, administrators in industrial laboratories and chemistry students nationwide to formulate a new chemical program. As a result of the Delphi process, a valuable guiding framework for the development of such a program was successfully achieved. Indeed, information from the first round Delphi was evaluated to be particularly useful for such program development. However, the long length of time needed to complete the study was one of the University of Texas researchers' main concerns.

Although, the Delphi technique has been used widely as an effective tool in curriculum research, it has not, to the author's knowledge, been previously applied to the determination of criteria for the development of laboratory programs or in environmental chemistry. In terms of time and cost effectiveness, the Delphi technique is an appropriate tool for curriculum innovation in developing countries in particular. That is a compelling reason for choosing the Delphi technique for the development of criteria for an EC experimental program in Vietnamese universities.

# CHAPTER 5 APPLICATION OF THE DELPHI TECHNIQUE IN THE STUDY

In this chapter, the international objectives of the environmental chemistry education (ECE) laboratory, which have been identified in **Chapter 3**, will be used extensively as the "initial" criteria in the construction of the first round Delphi questionnaire. These will be evaluated and modified by the Vietnamese EC educators to seek their consensus the criteria for the ECE laboratory in the context of Vietnamese universities.

#### **5.1 Expert Panel Selection**

Choosing the expert panel was given careful consideration in this study as it was acknowledged that this step might affect the validity of the result. The criteria for the participants were set prior to the study. Participants were those who met one of the following criteria:

- (i) People who have experience in teaching EC courses.
- (ii) People who are responsible for the administration of EC programs at universities or institutes, where such courses are carried out as independent subjects or independent programs.

With the above criteria, a group of 13 Vietnamese experts from 7 universities and 1 institute were invited to take part in the study. The names and allocations of the EC experts who accepted the invitation to join the expert panel are given in **Appendix 7**.

#### **5.2 Questionnaire Construction**

#### 5.2.1 First Round Questionnaire

Some modifications from the more traditional Delphi method have been made in this study. Instead of using the standard open-ended questionnaire, which was considered to risk a more scattered response to the questions of this survey (in the initial round), a structured questionnaire has been prepared. Indeed, such a structured questionnaire, which was formulated using current knowledge, has been used in a significant number of previous Delphi studies with the aim of enabling panel members to concentrate on the central research questions (109). As described previously (**Chapter 4, 4.2.2.1**), there are two main ways of preparing the framework for a first round structured questionnaire: by interview and through a literature review. In this study, a review of the international literature was chosen as the most appropriate strategy.

The aim of the applying the Delphi technique to this study is to help find out the criteria for the development of ECE experiments in Vietnamese universities. The international literature review and analysis is reported in **Chapter 3**. This suggested that in order to encompass the goals of the EE program, the following elements should be included in the ECE laboratory:

- Consideration of the environmental issues at international, regional, national and local levels.
- 2. The inclusion of environmental issues of air, water and soil environments.
- 3. The use of an integrated approach to learning about chemistry and environmental science.
- 4. Provision of opportunities for students to practice with appropriate instruments and apparatus.

- 5. The use of real samples and real scientific processes.
- 6. Helping students to improve the following environmental chemistry (EC) skills: Sampling, measurement, data collection and analysis, interpretation and reporting.

Regarding the goals of the chemical education laboratory, one important target is the inclusion of the "affective objectives", of which, to help students have a good attitude to science is the major concern (**Chapter 3**). The time allowed for laboratory work, which is dependent on the time allocation and the time allowed for a laboratory session, was shown to have an effect on students' attitudes to science (116). Moreover, it is considered that time allowed for a laboratory session also affects conceptual learning and investigative skills of students. Indeed, it was argued that if students have not enough time in the laboratory, they do not fully understand the results and the relevance to other contexts (71). Therefore, a good ECE experiment should also consider this element in its content.

To ensure a chemistry experiment has the appropriate pedagogical content, the guidelines for the design of chemical education experiments, set out by an internationally accepted journal for chemical educators (*Journal of Chemical Education*), recommends that experiments should be suitable for the level of the students. In other words, a teaching laboratory experiment needs to consider the students' background. Therefore, a good ECE experiment should also be expected to address this issue.

From the review of the international literature, which focused on both the scientific and the pedagogical content of the experiment, the following factors are considered to be important:

- 1. The reflection of environmental issues of international, regional, national and local.
- 2. The inclusion of environmental issues of air, water and soil environments.
- 3. The use of the integrated approach.
- 4. Providing students with opportunities to practice appropriate instruments and apparatus.
- 5. The use of real samples and real scientific processes.
- 6. Helping students with opportunities to improve the following EC skills: Sampling, measurement, data collection and analysis, interpretation and report data.
- 7. Identification of students' background.
- 8. Identification of the suitable allowed time for an experiment.

These issues were used as the framework for the development of the first round questionnaire or the so-called "initial" criteria. From there, the Delphi technique asks experts in the field to evaluate and amend and, finally, to agree on the "consensus" criteria. This questionnaire, although structured, does not limit participants' freedom to express other ideas, which they may consider to be pertinent to the issue because the questionnaire provides space for them to explain and expand on their answers, and to add other remarks.

Enclosed with the questionnaire, each member of the panel was sent a package, which fully described the project together with a letter of introduction and explanation from the candidate and her supervisors. In this first round, participants were asked to give their opinions by answering "yes" or "no" to each listed item. The full questionnaire is in Appendix 8.

#### 5.2.2 Establishing a Consensus Level for the First Round

The level of consensus in the first round was set equal to or greater than 50%. In other words, items with 50% or more "**yes**" answers will be used to formulate the second round questionnaire. This is considered to be a low setting compared to many such studies. However, it has been adopted here to place more emphasis on the second round, where a level of consensus of 75% has been set.

In some cases, panel members did not answer all items. Such unanswered items have been assumed to have "yes" replies. The result of such items will then be calculated as normal. In other words, the total number of "yes" answers will be the sum of "yes" replies and number of non-replies. By doing this (rather than assuming that they have been given "no" answers), participants who have not evaluated the items from the first round are more likely to have an opportunity to give their opinion in the following-up. Alternatively, the analysis may be based only on the actual responses (109). In the opinion of the researcher this approach is likely to weaken the validity of the second round.

#### 5.2.3 Second Round Questionnaire

Second round questionnaires were prepared on the basis of the experts' opinions from the first round. Items from the first round with the total number of "yes" answer equal or greater than 50% were used to compose the second round questionnaire. At this stage, participants were asked to rate their agreement on each item by using a 4-point-Likert scale: **strongly agree (4)**, **agree (3)**, **disagree (2)** to **strongly disagree (1)**. Panel members were also encouraged to provide further explanations by adding their comments and suggestions. The details of the second round questionnaire are in

#### Appendix 9.

The second round questionnaires were sent to individuals, who had returned the first round replies. Included with each questionnaire, which also consisted of instructions for replying to questions, was a letter of thanks from the researcher to participants in the first round Delphi survey, a brief report of the first round result and an invitation to contribute to the next round.

# 5.2.4 Establish a Consensus Level for the Second Round

In the second round, the consensus was set at 75% ( $\frac{3}{4}$ ). In other words, items have agreement equal to or greater than 75% (in two scales: **strongly agree** and **agree**) will be considered as consensus and they will compose a list of criteria for the ECE laboratory program.

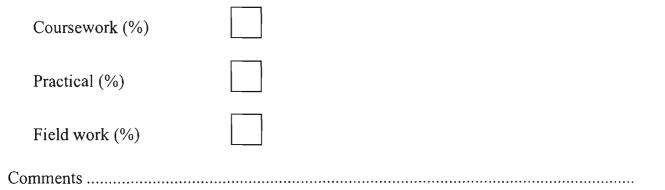
The level of the agreement on each item among the experts, however, will be calculated as the mean of the 4-point Likert scale. The higher the mean, the higher degree of agreement achieved. In contrast to the first round analysis, items which received no responses, will be ignored. The mean value has been calculated from the number of actual responses only. In this event, the mean value does not reflect the level of agreement of the whole group. A more accurate result may be generated if a further Delphi round is to be taken.

# **5.3 Data Collection and Analysis**

Questionnaires were sent to 13 panel members in the first round. The researcher received 11 replies after 6 weeks (yield 85%). All 11 participants indicated a willingness to participate in the second round. The detailed result of each question from the first round, the analysis of the first round result and the formulation of the second round are presented in this section.

#### **QUESTION 1**

By putting a percentage weighting (in terms of time allocation) in each box please indicate the components of a current environmental chemistry (EC) program in your university:



Answer for question 1

Percentage weighting (in terms of time allocation) in a current EC program:

												Average
Coursework (%)	70	70	60	70	65	85	85	90	No	60	60	71.5
Practical (%)	20	20	35	20	30	10	10	0	answer	30	25	20.0
Field work (%)	10	10	5	10	5	5	5	10		10	15	8.5

This finding showed that the current practical component is low in most Vietnamese universities. The largest time portion is allocated to coursework. One university, surprisingly, has no practical work. As indicated previously, the aim of this question is for information and therefore, it is not necessary to include it in the next Delphi round.

#### **QUESTION 2**

Within an EC program in your University, what do you consider to be the most appropriate time allocation for the following three areas?

Coursework (%)	
Practical (%)	
Field work (%)	
Comments	

#### Answer for question 2

The panel responded with following preferred proportions for EC coursework, practical and fieldwork components:

												Average
Coursework (%)	70	60	60	60	60	75	75	40	40	60	60	60.0
Practical (%)	20	25	35	30	35	15	15	30	40	30	25	27.3
Field work (%)	10	15	5	10	5	5	10	30	20	10	15	12.7

Comment: None

In comparison to replies from Question 1, a large number of experts considered that the current allocation time for the practical component in EC coursework at their

universities needs to increase. From the 11 replies, 7 considered the time allocation for practical work should be in the range 20-35%. The second round, therefore, will ask panel members to give their opinions on this proposition.

# Question for the second round

1. In Environmental Chemistry (EC) course, the most appropriate allocation time is

Coursework	60%-70%	4	3	2	1
Practical	20%-35%	4	3	2	1
Fieldwork	5%-10%	4	3	2	1
Comments		••••	••••		

(Where 4 is strongly agree, 3 is agree, 2 is disagree and 1 is strongly disagree)

# Result of the second round

		Ranking						
Items	4	3	2	1	Ν	Mean		
Coursework: 60%- 70%	4	6	1		11	3.3		
Practical 20%- 35%	3	7	1		11	3.2		
Fieldwork 5%- 10%	2	7	2		11	3.0		

# Comment:

- If the university had adequate facilities, the practical work should increase up to 50%.
- Should increase the allocation time for practical work for the final year students.

There was an increase in the degree of agreement in the second round compared to the first round on the allocation of time to practical work. While in the first round, only 7

among 11 replies agreed that 20-35 % of time should be allocated to the laboratory, in the second round, 10 replies were in favour of such idea. Consensus, therefore, has been achieved in the second round for this item.

# **QUESTION 3**

What do you consider to be the most appropriate length of time for an EC laboratory session?

hour (s)

Comments .....

#### Answer for question 3

The panel's responses to the question on the most appropriate length of time for an EC laboratory session were:

5 2 3 3 3 8 4 4 4 No answer No answer

Comment: unclear question

Of the 9 replies, 8 indicated that the suitable length for one laboratory session should be between two and five hours. This finding is somewhat similar to the previous works such as (94, 116, 117) in the international literature, which shows that laboratory sections of 3-4 hours are normal in EC courses.

#### Question for the second round

2. The most appropriate length of time for an EC laboratory session is 2-5 h

4 3 2 1

Comments .....

# Result of the second round

<u> </u>		Ran	king			
Items	4	3	2	1	Ν	Mean
2-5 h	4	7			11	3.4

Comment: The most suitable length time should be 4 h

In this round, both the number of replies and the agreement level have been achieved. All 11 panel members answered instead of only 9 in the first round and 100% of the answers agreed that the length time of an EC laboratory session should be 2-5 h.

# **QUESTION 4**

What do you expect students to achieve in the practical component?

	Yes No
4.1 Hands-on experience with standard EC techniques involving	
equipment and instrumentation.	
4.2 Awareness of international environmental problems and issues.	
4.3 Awareness of local environmental problems and issues.	
4.4 An understanding of the processes involved in:	
Sample preparation	
Measurement	
Data processing	
Interpreting data	
Reporting	

Other.....

# Answer for question 4

The aims of the EC practical component:

				No
	Aims	Yes	No	answer
4.1	Hands-on experience with standard EC techniques	11	_	
	involving equipment and instrumentation.			
4.2	Awareness of international environmental problems and issues.	4	3	4
4.3	Awareness of local environmental problems and Issues.	8		3
4.4	An understanding of the processes involved in:			
	Sample preparation	11		
	Measurement	11		
	Data processing	6	3	2
	Interpreting data	11		
	Reporting	10		1

# Comment: None

Using the pre-set consensus for the first round, all items will go to second round for further consideration. Item 2 (4.2), however, gained very low support "**yes**", only 4 among 7 responses (4 unanswered). Based on the regulation set for non-replied items, it was also put into second round.

# Question for the second round

3. In the practical component students can achieve:

3.1 Hands-on experience with standard EC technique involving equ	3.1 Hands-on experience with standard EC technique involving equipment and								
instrumentation.	4	3	2	1					
3.2 Awareness of international environmental problems and issues.	4	3	2	1					
3.3 Awareness of local environmental problems and issues.	4	3	2	1					
3.4 Understanding of the process involved in:									
Sample preparation	4	3	2	1					
Measurement	4	3	2	1					
Data processing	4	3	2	1					
Interpreting data	4	3	2	1					
Reporting data	4	3	2	1					
Comments	•••••		•••••						

# Result of the second round

		Ran				
Items	4	3	2	1	Ν	Mean
4.1 Hands-on experience with standard EC technique	2	9			11	3.2
involving equipment and instrumentation.						
4.2 Awareness of international environmental	1	3	7		11	2.5
problems and issues.						
4.3 Awareness of local environmental problems	1	10			11	3.1
and issues.						
4.4 Understanding of the process involved in:						
4.4.1 Sample preparation	2	8	1		11	3.1
4.4.2 Measurement	4	7			11	3.4
4.4.3 Data processing	3	7	1		11	3.2
4.4.4 Interpreting data	3	8			11	3.3
4.4.5 Reporting data	2	9			11	3.2

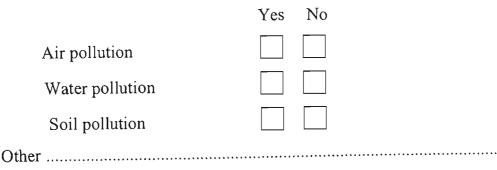
Comment: None

The items: [4.1]; [4.4.2]; and [4.4.4] gained the highest agreement in both round all at 100%. The level of agreement, though, was evaluated to be the highest for the first item based on the second round result. It showed that, EC/ES experts considered that the most important aim of the EC laboratory experiment is to help students practice the process of measurement.

Item [4.2] gained very low response in the first round (4 panel members did not give their opinions). In the second round, the reply level increased greatly (all 11 made their ranking). Of the 11 replies, 7 disagreed that this item should be one of the aims of the EC laboratory experiment. This finding, however, contradicts the Vietnamese guidelines for environmental education programs, which states that one of the purposes of the EE program should be that it refers to international environmental problems and issues (23). This matter will be discussed further in **Chapter 6**. The remaining items all reached the consensus setting in the second round.

#### **QUESTION 5**

In the practical component, please indicate which of the environmental problems listed below *should be included* in the EC experimental program?



# Answer for question 5

In the practical component, the environmental problems *should be included* in the EC experimental program:

	Should be included								
Environmental issues	Yes	No	No answer						
Air pollution	11								
Water pollution	11								
Soil pollution	11								

*Comments*: A panel member suggested that the issue of hazardous waste in the environment should be included in the EC laboratory program. This comment was put into the second round for the group comment. Other comments suggested the inclusion of environmental issues in Vietnam, toxic processes in the environment, and the effect of industrial pollution in the environment. As these were considered to be already covered in the questionnaire, they were not put into the second round.

All replies were "yes" answers, therefore, all 3 items were included in the second round.

#### Question for the second round

4. In the practical component, the following environmental problems should be included:

Air pollution	4	3	2	1
Water pollution	4	3	2	1
Soil pollution	4	3	2	1
Hazardous wastes	4	3	2	1
Comment				

		Ran	king			Mean	
Items	4	3	2	1	Ν		
Air pollution	5	6			11	3.5	
Water pollution	7	4			11	3.6	
Soil pollution	4	7			11	3.4	
Hazardous wastes	3	7	1		11	3.2	

#### Result of the second round

Comments: In EC, it is vital to regard all the above issues.

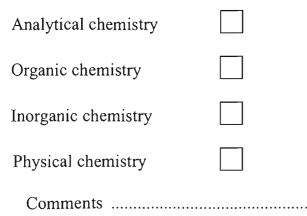
All three proposed items had a high level of agreement in both rounds (100%). This indicated the importance of considering of the environmental issues associated with air, water and soil environments in ECE laboratory experiments. The additional item formulated from a comment from the expert panel also gained a consensus, though with lower level (10 among 11 members agreed).

# **QUESTION 6**

Do you think that an EC experiment should also teach basic chemical principles?



If "yes", which of the following areas of chemistry are most appropriately taught within the context of an EC experiment ?



# Answer for question 6

Should an EC experiment be integrated with teaching other chemistry principles? 10 replies with "**yes**" and 1 "**no**" (the explanation was that: EC is an independent subject having its own textbook, practical book, therefore, should be taught independently). This item, therefore became a component in second round questionnaires.

The areas of chemistry the panel considered were most appropriately taught in an integrated EC experiment were:

10	
3	
3	
3	
	3

*Comments*: EC is related to all branches of chemistry. Analytical chemistry is the most suitable but it does not mean that it is the only single subject to be included in teaching basic chemistry.

With these replies, analytical chemistry was evaluated to be the most suitable subject in the integration context. Only analytical chemistry, therefore, went to second round, as organic, inorganic and physical chemistry had very low support (only 3 among 11 replies were in favor) in the first round.

# Question for the second round

5. EC experiments should be taught in combination with analytical chemistry

4 3 2 1

Comments .....

Result of the second round

		Ran	king			
Items	4	3	2	1	N	Mean
Analytical chemistry	7	4			11	3.4

#### Comments:

Four members made comments that EC should be integrated not only with analytical chemistry but also with other chemistry branches including physical, organic, inorganic, and also with technological chemistry.

A higher level of agreement has been gained in the second round answer than in the first round. 100% respondents (compared to 10/11 in the first round) agreed that analytical chemistry is the most suitable chemistry field to be included in the EC laboratory experiment. Some panel members were also in favour of the integration of EC experiment with chemistry branches besides analytical chemistry.

. . . . . . . . . . . . . . .

# **QUESTION 7**

To what extent do you think that an EC experiment should reflect the work carried out by professional environmental scientists ?

	A lot	Some	Not important	
Sample preparation				
Measurement				
Data processing				
Data interpretation				
Reporting data				
Other		•••••	••••••	•••

# Answer for question 7

Extent an EC experiment should reflect the work carried out by professional environmental scientists:

Processes	A lot	Some	Not important	No answer
Sample preparation	5	3	2	1
Measurement	7	3	1	
Data processing	1	8	2	
Data interpretation	2	9		
Reporting data	5	5		1

# Comment: None

The items concerning "sample preparation" and "data processing" gained less support compared with other items. For both, 2 panel members disagreed that they are important skills for practice in EC laboratory experiments. However, all items met the setting consensus, and therefore were put into the second round for further consideration.

# Question for the second round

6. An EC experiment should reflect mainly the work carried out by professional

environmental scientists which include

Sample preparation	4	3	2	1
Measurement	4	3	2	1
And to some extend should	l in	clu	de:	
Data processing	4	3	2	1
Data interpretation	4	3	2	1
Reporting	4	3	2	1
Comments		••••		

# Result of the second round

		Ranl				
Items	4	3	2	1	Ν	Mean
Sample preparation	3	7	1		11	3.2
Measurement	4	7			11	3.4
Data processing	1	9	1		11	3.0
Data interpretation	1	10			11	3.1
Reporting	2	9			11	3.2

# Comment: None

There was an increase in the level of agreement in two items *sample preparation* and *data interpretation*. Both had 10 from 11 replies agreeing that they should be included in the EC laboratory, compared with 9 in the first round. The remaining items

(*measurement*, data interpretation and reporting data) all had 100% support. The highest level of agreement belonged to the item *measurement*.

# **QUESTION 8**

Please indicate the background of EC students in your Institution, which you consider to be the most suitable background for undertaking EC courses?

	Yes	No
General chemistry background		
Students with basis knowledge in organic chemistry		
Students with basis knowledge in inorganic chemistry		
Students with basis knowledge in physical chemistry		
Students with basic knowledge in analytical chemistry		

Other .....

# Answer for question 8

Background	Yes
General chemistry background	10
Students with basis knowledge in organic chemistry	6
Students with basis knowledge in inorganic chemistry	6
Students with basis knowledge in physical chemistry	7
Students with basic knowledge in analytical chemistry	9

Comment: None

All items met the setting level of consensus therefore, are put in the second round for further consideration.

# Question for the second round

7. EC should be taught to students who possess basic knowledge in

General chemistry	4	3	2	1
Inorganic chemistry	4	3	2	1
Organic chemistry	4	3	2	1
Analytical chemistry	4	3	2	1
Physical chemistry	4	3	2	1
Comments	••••			

		Ran	king				
Items	4	3	2	1	No answer	Ν	Mean
7.1 General chemistry	4	7				11	3.4
7.2 Inorganic chemistry	3	8				11	3.3
7.3 Organic chemistry	3	8				11	3.3
7.4 Analytical chemistry	5	6				11	3.5
7.5 Physical chemistry	4	6			1	10	3.4

# Result of the second round

#### Comment:

Three members considered that because EC is highly interrelated with other chemistry branches, in order to help students achieve better outcomes, an EC course should run after all branches of chemistry have been taught. Indeed, the more background in chemistry the students have the better.

# **QUESTION 9**

Please indicate which instruments and apparatus are available at your institute and whether you consider the listed instrument to be an essential hands-on items for an EC

course?

	Available	Not available	Essential	Not essential
GC/MSD				
(Gas Chromatography/ Mass Selective Detector)				
GC/MS				
(Gas Chromatography/ Mass Spectrometry)				
GC/FID				
(Gas Chromatography/ Flame Ionisation Detector)				
GC/ECD				
(Gas Chromatography/ Electron Capture Detector)				
TLC				
(Thin Layer Chromatography)				
Ion Chromatography				
ICP/MS				
(Inductively Coupled Plasma Emission- Mass Spectromet	ter)			
MS				
(Mass Spectrometer)				
AAS				
(Atomic Absorption Spectrometer)				
HPLC				
(High Performance Liquid Chromatography)				
UV-VIS				
(Ultraviolet Visible Spectrophotometer)				<b>—</b> ——
FTIR				
(Fourier Transform Infrared Spectrophotometer)				<b>—</b> —
Fluorometer				
Voltammeter				
Polarographic instrument				

	Available	Not available	Essential	Not essential
Ion Selective Electrodes				
Computers and software				
Turbidimeter				
Vortex mixer				
Centrifuge				
Soxhlet apparatus				
Conductivity instrument				
pH meter				
Shaker				

Other .....

# Answer for question 9

The panel provided the following rating instrument availability those considered to be essential hands-on item for an EC course.

Instrument/Apparatus	Available	Essential	No answer
GC/MSD	4	9	2
GC/MS	5	11	1
GC/FID	3	6	4
GC/ECD	2	6	5
TLC	1	4	5
Ion Chromatography	3	6	3

Instrument/Apparatus	Available	Essential	No answer
ICP/MS	1	7	3
MS	1	5	5
AAS	4	6	4
HPLC	1	6	5
UV-VIS	4	7	4
FTIR	2	5	6
Fluorometer	2	6	4
Voltammeter	6	5	4
Polarographic instrument	3	8	2
Ion Selective Electrodes	5	6	3
Computers and software	7	8	1
Turbidimeter	6	7	2
Vortex mixer	6	6	3
Centrifuge	4	7	2
Soxhlet apparatus	4	6	4
Conductivity instrument	6	7	2
pH meter	6	7	2
Shaker	6	7	2
Total	92	158	

Comment: None

This was the lengthiest question and had the lowest response rate. Based on the setting consensus and the regulation, all items met the requirement to be included in the second round questionnaire.

# Question for the second round

8. To help students have effective EC laboratory, it is necessary to have the following items in the laboratory.

GC/MSD	4	3	2	1
GC/MS	4	3	2	1
GC/FID	4	3	2	1
GC/ECD	4	3	2	1
IC	4	3	2	1
ICP/MS	4	3	2	1
MS	4	3	2	1
AAS	4	3	2	1
HPLC	4	3	2	1
UV-VIS	4	3	2	1
FT-IR	4	3	2	1
Fluorometer	4	3	2	1
Voltammeter	4	3	2	1
Polarographic instrument	4	3	2	1
Ion Selective Electrodes	4	3	2	1
Computers and Software	4	3	2	1
Vortex mixer	4	3	2	1
Centrifuge	4	3	2	1
Soxhlet apparatus	4	3	2	1
Conductivity instrument	4	3	2	1
pH meter	4	3	2	1
Shaker	4	3	2	1

		Ranl	king			<u> </u>	
Items	4	3	2	1	No answer	Ν	Mean
GC/MSD	3	5	1		2	9	3.0
GC/MS	4	5	1		1	10	3.3
GC/FID	4	7				11	3.4
GC/ECD	5	6				11	3.5
IC	3	8				11	3.3
ICP/MS	2	8	1			11	3.1
MS	3	6		1	1	10	3.1
AAS	2	9				11	3.2
HPLC	3	7			1	10	3.3
UV-VIS	3	8				11	3.3
FT-IR	2	7			2	9	3.2
Fluorometer	5	5			1	10	3.5
Voltammeter	2	8			1	10	3.2
Polarographic instrument	3	6	1			11	3.2
Ion Selective Electrodes	4	6			1	10	3.4
Computers and Software	3	8				11	3.3
Vortex mixer	4	6			1	10	3.4
Centrifuge	3	6	2			11	3.1
Soxhlet apparatus	3	7			1	10	3.3
Conductivity instrument	4	7				11	3.4
pH meter	5	6				11	3.5

<u>Result of the second round</u>

Comment:

- Two members considered that instrument and apparatus for the measurement of BOD (Biochemical Oxygen Demand) and COD (Chemical Oxygen Demand) are also needed in the EC laboratory experiment.
- Two members commented: some sampling apparatus such as air, water and soil sampling to help students with hands-on experience with sampling procedures.

In the second round, there was a significant increase in the number of replies compared to the first round. The result has shown the important need for students to practice on specific instruments and apparatus in EC laboratory experiments. It has also demonstrated that, there was a significantly higher demand for sophisticated equipment than is currently available in most Vietnamese universities.

# **QUESTION 10**

In your opinions what are the difficulties in conducting EC experiments ?

Time constraints	
Financial	
Staffing	
Facility availability	
Other	

# Answer for question 10:

Difficulties	Yes	No answer
9.1 Time constraints	6	1
9.2 Financial	9	1
9.3 Staffing	4	1
9.4 Facility availability	10	1

Difficulties in conducting an EC experiment

# Comment: None

In this question, 3 items were included in the second questionnaire. The item [9.3] was not in the second round questionnaire, because it had very low level of support in the first round (only 5 from 11 replies).

# Question for the second round

9. In conducting EC experiments, rate the following as difficulties:

Time constraints	4	3	2	1
Financial	4	3	2	1
Facility availability	4	3	2	1
Others				

	Ranking						
Items	4	3	2	1	No answer	$\mathbf{N}$	Mean
9.1 Time constraints	2	5	2		2	9	3.0
9.2 Financial	5	6				11	3.5
9.4 Facility availability	5	6				11	3.5

# Result of the second round

*Comment*: One commented: In Vietnamese universities, the lack of staff with well trained EC staff is still one of the difficulties they have.

Two items: [9.2] and [9.4] gained the highest level of agreement in both rounds. Their mean value of 3.5 was also highest mean in this Delphi study. In other words, Vietnamese experts considered that the most serious difficulties in running an EC laboratory are financial and facility availability. In this study, the response to the "staffing" item differed from those in national studies. One of the reported difficulties in running EE programs in Vietnam was the lack of knowledgeable staff (1), this study, however, showed that staffing is not an issue in running EC/ES laboratory programs. In order to find out clear reasons for this discrepancy, a more specific **Question 10** (in the first round), with regard to the "staff", should be made. For example, this question could be... number of staff and number of qualified staff.

# **QUESTION 11**

What do you perceive will be the benefits to students of conducting EC experiments that incorporate the teaching of chemical principles?

•	Provide students with a broader perspective on chemistry	
•	Help to link with different branches of chemistry	
٠	Help the students to understand how chemistry is applied	
	to environmental problems	
•	Increase the appeal of chemistry as a discipline	
٠	Provide students with a more scientific understanding of the environment	
	Other	

# Answer for question 11

	Yes	No	
11.1 Provide students with a broader perspective on chemistry	7	4	
11.2 Helps to link with different branches of chemistry	8	3	
11.3 Helps the students to understand how chemistry is applied	11	0	
to environmental problems			
11.4 Increase the appeal of chemistry as a discipline	6	5	
11.5 Provides students with a more scientific understanding	11	0	
of the environment			

# Comment: None

All items met the setting consensus for the first round (except item [11.4]). Therefore all items were put into the second round. For the purpose of the study (as explained before), all items were included in the second round questionnaire.

# Question for the second round

10. In conducting EC experiment that incorporate the teaching of chemical principles,

students will have the following benefits:

	Gives a broader perspective on chemistry	4	3	2	1	
	Helps to link with different branches of chemistry	4	3	2	1	
	To understand how chemistry in applied to environmental problems	4	3	2	1	
	Increases the appeal of chemistry as a discipline	4	3	2	1	
	To have more scientific understanding of the environment	4	3	2	1	
Сс	omments	•••••		•••••		

# Result of the second round

		Ran	king			
Items	4	3	2	1	Ν	Mean
11.1 Having a broader perspective on chemistry	4	7			11	3.4
11.2 Having a strong link with different branches	4	6	1		11	3.1
of chemistry						
11.3 Understand how chemistry is applied to	6	5			11	3.5
environmental problems						
11.4 Increase the appeal of chemistry as a	4	7			11	3.4
discipline						
11.5 Having more scientific understanding of the	4	7			11	3.4
environment						

*Comment*: One commented that for the teaching of EC it is desirable for it to be integrated with other branches of chemistry.

Except for the item [11.2], all items gained a very high level of agreement (100%). Significantly, to help students [11.3] has the highest degree of agreement, with 6

strongly agreeing and 5 agreeing. In the second round, the [11.2], which should have been eliminated in the first round, also received more support from the panel members. Only 6 replies were in favour in the first round, while 10 supported the proposition in the second round. Therefore, all items in this question had reached the criterion for consensus of the second round.

# 5.4. Second Round Delphi Result

The summary result of the second round is shown in the table below:

			Ranking	ting		No		
Answers	Items	4	e	7	-	answer	Z	Mean
	Coursework: 60%- 70%	4	9	-			11	3.3
1. The most appropriate allocation time is	Practical 20%- 35%	ю	5	-			11	3.2
the following	Fieldwork 5%- 10%	7	2	7			11	3.0
2. The most appropriate length of time for	2-5 h	4	7				11	3.4
an EC laboratory session is								
	Hands-on experience with standard EC	7	6				11	3.2
	technique involving equipment and							
	instrumentation.							
	Awareness of international environmental	-	ς	2			11	2.5
	problems and issues							
3. The aims of the practical component are:	Awareness of local environmental problems	1	10				11	3.1
	and issues							
	Understanding of the process involved in							
	Sample preparation	5	$\infty$	-			11	3.1
	Measurement	4	7				11	3.4
	Data processing	ω	7	-			11	3.2
	Interpreting data	ω	$\infty$				11	3.3
	Reporting data	7	6				11	3.2
		1	1					

Answers	Items		Ranking	ing		No	Z	Mean
		4	e	7	1 ans	answer		
	Air pollution	S	9				11	3.5
4. In the practical component, the following	Water pollution	2	4				11	3.6
environmental problems should be	Soil pollution	4	2				11	3.4
included	Hazardous wastes	с С	2	-			11	3.2
11. EC experiment should be best integrated	analytical chemistry	7	4				11	3.4
with								
	Sample preparation	ŝ	7				11	3.2
12. An EC experiment should reflect the	Measurement	4	7				11	3.4
work carried out by professional	Data processing	-	6				11	3.0
environmental scientists which include	Data interpretation		10				11	3.1
	Reporting	7	6				11	3.2
	General chemistry	4	7				11	3.4
13. EC should be taught for student with the	Inorganic chemistry	m	×				11	3.3
basic knowledge in	Organic chemistry	m	~				11	3.3
	Analytical chemistry	S	9				11	3.5
	Physical chemistry	4	9			1	10	3.4

Answers	Items		Ranking	ing		No	Z	Mean	
		4	e	7	-	answer			
	GC/MSD	ю	5			2	6	3.0	
	GC/MS	4	5			-	10	3.3	
	GC/FID	4	2	-			11	3.4	
	GC/ECD	S	9				11	3.5	
	IC	e	~				11	3.3	
	ICP/MS	7	$\infty$	-			11	3.1	
	MS	Э	9		-	1	10	3.1	
14. To help students have effective EC	AAS	7	6				11	3.2	
laboratory, it is necessary to have the	HPLC	Э	7				10	3.3	
following instrumentations and	UV-VIS	33	~				11	3.3	
apparatus in the laboratory	FT-IR	2	5			5	6	3.2	
	Fluorometer	S	5	-		1	10	3.5	
	Voltammeter	2	∞			-	10	3.2	
	Polarographic instrument	3	9				11	3.2	
	Ion Selective Electrods	4	9				10	3.4	
	Computers and Software	ε	$\infty$				11	3.3	
	Vortex mixer	4	9			1	10	3.4	
	Centrifuge	ω	9	10			11	3.1	
	Soxhlet apparatus	ю	7				10	3.3	
					-		1		

ing No	2 1 answer N Mean	11 3.4	11 3.5	11 3.5	2 2 9 3.0	3 2 9 2.8	11 3.5	11 3.5		11 3.4		1 11 3.1		11 3.4	
Ranking	3	2	9	9	5	5	9	5		~		9		~	
	4	4	5	5	12		S	to 6		4		f 4		4	-
	Items	Conductivity instrument	pH meter	Financial	Time constraints	Staffing	Facility availability	chemistry is applied	environmental problems	Having more scientific understanding of the	environment	Having a strong link with different branches of	chemistry	T the amount of chamictry as a disciplin	Increase the appeal of culturations as a survival statement of the sur
	Answers			15. In conducting EC experiment, the	following difficulties will be expected	)		16. In conducting EC experiment that	incorporate the teaching of chemical	principles, students will have the	following benefits:	3			

### CHAPTER 6 DELPHI FINDINDGS AND DISCUSSION

#### 6.1 Delphi Findings

After the second round Delphi survey, the following criteria for the development of environmental chemistry education laboratory experiments (in the Vietnamese context) are suggested:

1) Time allocation

Practical work should constitute 20-35% of the course.

2) The length of time for a laboratory session

The suitable length of time of one laboratory session is from 2-5 hours.

- 3) Aims of the ECE experimental program
- To help students with hands-on experience of standard EC techniques involving equipment and instrumentation including the following: GC/ECD Fluorometer; pH meter; GC/FID; ion selective electrodes; vortex mixer; conductivity; GC/MS; IC; HPLC; UV-VIS; computers and software; Soxhlet apparatus; AAS; FT-IR; voltameter; polarography; ICP/MS; MS; centrifuge; and GC/MSD.
  - To help students become aware of local and regional environmental problems and issues.
  - To help students understand the process involved in: sample preparation; measurement; data processing; interpreting data; and reporting data.
- Environmental issues water pollution; air pollution, soil contamination, and hazardous wastes should be included in the ECE experiments.
- 5) Specific skills to be practiced included sample preparation, measurement, data collection, data analysis and interpretation, and reporting data.
- 6) Students' background

Environmental chemistry should be taught to students with basic knowledge in analytical chemistry; and/or (general chemistry; physical chemistry; organic chemistry).

7) Real world professional practices

An EC experiment, to a large extent should reflect the work carried out by professional environmental scientists e.g. Sample preparation, measurement; data collection; data analysis and interpretation; and reporting data.

- 8) Using the concept of integration in EC experiments
  - EC experiments should integrate with other branches of chemistry. Analytical chemistry is recommended to be the most suitable area to be integrated with EC experiments.
  - Integrating EC with other chemistry branches should aim to help students:
    - Understand how chemistry is applied to environmental problems.
    - Have a more scientific understanding of the environment.
    - Establish a strong link-up between different branches of chemistry.
    - Increase the appeal of chemistry as a discipline.
    - Have a broader perspective on chemistry itself.

#### 6.2 Discussion of Method

## 6.2.1 The Implications of the Delphi Technique

Use of the Delphi method in this study has highlighted the versatility of this method and suggests that it may be utilized effectively in other areas of education in countries such as Vietnam. Currently the Vietnamese Government is developing an EE curriculum for all levels of the educational system, which is expected to be completed and enacted by 2005 (23). The Delphi approach may serve as an effective means to create more applicable and suitable programs by including as many educators as possible who are interested in this significant new field. One of the characteristics of the Delphi technique is the use of a group of people, who are expert in their specific field.

In Vietnam, EE is becoming increasingly important in the educational system (19-21). EC has been taught in universities and some textbooks are available nationally. However, there is no practical program available at the time of conducting this study. The set of criteria for the development of an EE/ES experimental program, found here by using the Delphi method, was a response to this need.

The process of having this set of criteria itself says that it is not value free. The panel for the study was made up of leading experts in EC in Universities across Vietnam. Moreover, the questionnaire, which was sent to the experts to evaluate, was formulated based on the international literature in this field, including international guidelines for the development of EE programs, and journals on chemical education. In addition, the evaluation of the literature on the ECE laboratory was completed after the researcher had spent a 5-month work experience program in a professional EC laboratory. For all the above reasons, it can be suggested that the set of criteria will be trustworthy as a set of national guidelines for the development of EC experimental program at the university level.

#### **6.2.2 Interpretation and Discussion of the Findings**

#### 6.2.2.1 International Environmental Issues in the EC Laboratory

The necessity to consider the international environmental issues in EE has been emphasized in the Tbilisi Declaration, which set out the international guidelines for the development of EE programs (1). The development of EE programs in Vietnam are based on national guidelines, which are inturn, use the groundwork of the Tbilisi Declaration (23). Therefore, the inclusion of international environmental issues is one of the aims in the development of EE programs in Vietnam.

#### The Tbilisi Declaration suggests (1):

"Environmental education should examine major environmental issues from local, national, regional and international points of view so that students receive insights into environmental conditions in other geographical areas".

In this regard, the contradiction between this clear recommendation and the findings of the Delphi survey should be recognized. The Delphi findings, which represent EC Vietnamese educators' points of view, were that the inclusion of international environmental issues is unnecessary in EC education. This difference may be explained by the current difficulties in accessing international information in most Vietnamese universities, which has been defined to be one of the weaknesses of the EE program in Vietnam (22). The difficulties in accessing international resources in environmental issues in general and incorporating these into EC courses may be the reason for the lack of importance accorded to international environmental issues by EC Vietnamese educators. It may, however be an issue of interest to further research to have a clearer explanation for this difference between the 'practical' EC curriculum and the 'theory' EC curriculum at the university level in Vietnam.

#### 6.2.2.2 Time Allocation for EC Laboratory Work

Time allowed for the laboratory work refers to the length time for one laboratory session and the time allocation for the laboratory program. It has been found that the time in the laboratory has an effect, not only on learning concepts and thinking skills, but also on students' attitudes to science. The Delphi finding, in this regard, enables the curriculum planner to construct the program effectively by providing the recommended time for a laboratory session.

The result of the brief survey of the current time allocation for practical work in EC course in some Vietnamese universities agrees with previous reports (25). A criticism of the educational curricula in Vietnam has been that too much attention is given to providing students with academic theory rather than with practical skills and knowledge (25). This drawback of the educational curricula suggests that the quality of education can be improved by reducing emphasis on theory and enhancing the level of practical work (25). In this study, the survey of Vietnamese environmental educators has found that the current time allocation to practical work is considered to be too small, with most universities devoting no more than 30% of course time to EC laboratory work. Although, a significant number of the expert panel did consider that current programs need to be changed (i.e. to increase the practical work), their proposed time allocation for laboratory work does not seem to be significantly different from current practice. Panel members agreed that practical work should compose between 20-35% of the time available for EC courses. In comparison time spent in practical work from universities outside Vietnam, for example, was 75% of course hours (118, 54). The small time allocation to practical work in the EC laboratory proposed by the Vietnamese environmental educators, correlates with their acknowledgement of current difficulties in budget and time constraints and the lack of facilities in their universities. Such difficulties are challenging the whole Vietnamese educational system (25) not just in EC courses. These difficulties have led to weaknesses in the educational curricula, one of which is the seriously inadequate time for practical work in educational courses (25).

#### 6.2.2.3 Using Instrumentation in the EC Laboratory

Similar to the international environmental educators' point of view, Vietnamese experts in the field have expressed their acknowledgement of the great benefits of using appropriate instruments in the EC experiments. There is therefore a need to equip the environmental chemistry laboratory with such instruments.

The survey has shown that there is a serious shortage of facilities in the EC laboratories in most Vietnamese universities. It has been acknowledged that the lack of facilities in the laboratory is the reason for the ineffectiveness of science laboratories in general and in EC laboratories in particular (116). Yet, a science laboratory is expensive, and often a major issue for a faculty's budget (63, 116). Therefore, it is essential to find out what are the most needed facilities for an undergraduate EC laboratory (116). Depending on the available budget of the faculty, one may consider equipping the EC laboratory with affordable facilities. The finding of the Delphi survey in this study provides a list of instruments and apparatus that EC experts in Vietnam considered necessary for students to have hands-on experience. The three most needed instruments include GC/ECD; Flourometer; and GC/FID. The four most needed pieces of apparatus were pH meter; Ion Selective Electrodes; Vortex mixer; and conductivity apparatus. Other relatively important instruments include GC/MS; IC; HPLC; UV-VIS. Soxhlet apparatus and computers and software were also considered to be relatively important.

#### **Proposed Solutions to Overcome the Shortage of Facilities**

In order to improve the quality of science education in general and in EC in particular, the difficulties with time, budget and facilities should not lead to the reduction of practical work in courses. This study has re-emphasised the important place of laboratory work in science teaching and chemistry in particular.

The ultimate aim of the EC courses is, therefore, to provide more experiments for students to practice. In circumstances where there are some obstacles, innovative ways must be found to solve these problems. Interestingly, in the design of chemistry experiments in general and EC experiments in particular, the use of the integrated approach has been used widely and with great success internationally (**Chapter 3**, **3.6.2**). The reasons that this approach has been favored by chemical educators are that, it enables them to use instruments more efficiently, and to reduce the time commitment of staff (**Chapter 3**, **3.5.2.2**). The other benefit of the integrated approach is for the students of which all Vietnamese experts have also strongly agreed that using integrated experiments in EC lab will:

- (i) Help students understand how chemistry is applied to environmental problems.
- (ii) Help students have more scientific understanding of the environment.
- (iii) Help students establish a strong link with between different branches of chemistry.
- (iv) Increase the appeal of chemistry as a discipline.

For all of these reasons, it can be said that one promising way to help Vietnamese universities overcome the current difficulties is through the application of the integrated approach in designing experiments. This study, therefore, recommends the development of more integrated experiments in Vietnamese universities rather than single-purpose ones.

An emphasis on integrated experiments in EC laboratory work is promoted by the results of the Delphi study reported here, and by the survey of international publications outlined in **Chapter 3**. The representative Vietnamese educational experts in EC all consider that analytical chemistry is the most suitable chemical branch to be included in the EC laboratory. The Delphi panel also recommended that other chemistry branches such as organic, inorganic and physical chemistry are also worthy of integration within EC laboratory experiments.

Some other possible solutions to help overcome the above mentioned difficulties may include:

- (i) Cooperation between the university and industrial laboratories or with research centers, in order to access laboratory equipment.
- (ii) Seeking external funding, which Steehler (96) has recommended.

#### 6.2.2.4 Soil Environmental Issues in EC Laboratory

The Delphi study strongly suggests that water, air and soil should be included in EC programs in Vietnamese universities. The international survey, on the other hand, has found that issue of soil environment has lagged far behind the focus on water and air. This is one of the problematic aspects of current international EC programs (**Chapter** 

**3**). There is a significant difference of attitude towards soil environmental issues between international environmental educators and Vietnamese environmental educators. In other words, environmental experts in Vietnam have recognised the specific environmental issues relevant to Vietnam and the need for their inclusion in EC laboratories. This significant recognition can only be fulfilled when such issues are demonstrated to students through both textbooks and practical work. In terms of the development of EC laboratory programs, it is clear that there is a need for more experiments relating not only to air and water but, importantly, to the soil environment.

#### 6.2.2.5 The Scientific Approach in the EC Laboratory

Findings from the Vietnamese EC educators demonstrated that, according to the experts, it is necessary to illustrate the scientific practice of environmental chemists in the EC laboratory (e.g. the demonstration of not only measurement, collecting data and reporting processes, but also sample preparation, data analysis and interpretation procedures). One of the strong criticisms of the current EC curriculum is the lack of demonstration of the scientific process in the ECE laboratory, in terms of the sample preparation, data analysis and interpretation, and QA/QC (**Chapter 3**). Thus, in the Vietnamese context, any development of the ECE laboratory for use at the undergraduate level should take this issue into account. In this study, this issue will be clearly demonstrated in the design of experiments.

#### 6.3 Method Validation and Discussion

#### 6.3.1 Expert Panel

The size and the quality of the participants are often a source of bias of the Delphi technique (**Chapter 4, 4.4**). In this study, steps have been taken to minimise this bias.

These steps have included the setting of criteria for the Delphi study in advance of the establishment of the panel and the use of a panel with a sufficient number of members. Indeed, as discussed previously, the minimum number of panel members is not yet well-determined (**Chapter 4**, **4.4**), however, a large number of previously successful Delphi studies have used panel numbers in the range between 10-1685 (102). The panel size used in this study was 11, and the criteria of the participants were clearly indicated before choosing experts, which is listed in **Chapter 5** (**5.1**).

Variation in the level of experts' participation is another possible threat to the accuracy of the Delphi result (**Chapter 4, 4.4**). In this study, the willingness to participate by all panel members can be seen when 100% of the respondents from the first round participated in the second round with all returning their second round replies.

#### 6.3.2 Questionnaire Construction

#### 6.3.2.1 The Formulation of the First Round Questionnaire

The Delphi questionnaire was specifically constructed to develop criteria for the establishment of EC laboratory practice in undergraduate chemistry programs in Vietnam. There are no clear international guidelines on this matter. In this study, the starting point has been the reported objectives and content of EE programs and of chemistry course in general. In the ECE laboratory, EC experiments are used for teaching purposes. Therefore, besides the science content, pedagogical content is another issue of concern. In this case, chemical education guidelines set by the *Journal of Chemical Education* have been employed. Hence, it can be said that, the "initial" criteria have been formulated after consideration of both the scientific and the pedagogical context, using reliable sources. In addition, the construction of the

questionnaire was completed after the researcher had undertaken a five-month work experience program in a real-world environmental laboratory, where the researcher had the opportunity to understand and undertake the procedures used by professional environmental chemists. The care taken in the construction of the initial survey could explain the highly satisfactory convergence of the Delphi process at the second round.

#### 6.3.2.2 The Length of Question and Its Influence

The study was time-consuming due to the sequential rounds of questionnaires and the difficulty in accessing panel members. In agreement with a previous study (107), this project suggests that the quantity and the quality of the results may be enhanced if the Delphi study is conducted with the active backing of a significant person or organization. In this case, it is suggested that the survey's yield could not have been as high as 87% if there were not a letter from a key supporter: the Dean of the Chemistry Department, Hanoi Teachers' Training University, who is well known to his chemistry colleagues in Vietnamese universities.

The time demands on the participants may explain the incompletion of some items in the first round questionnaire. Indeed, the lengthiest question (Question 9) had the highest number of unanswered replies. Rephrased in the second round, this question subsequently gained more answers from panel members. It is suggested that in designing a questionnaire in a Delphi study a researcher must seek to be as succinct as possible. The length of the questionnaire could explain the difficulties faced by chemical educators in a 1977 study, which employed the traditional Delphi method, with a first round questionnaire of 70 questions (111).

#### 6.3.2 Distribution Method

In terms of questionnaire distribution, the researcher has made a modification from the conventional Delphi method. Instead of administering the questionnaire by mail, the researcher distributed the questionnaire in person to the Delphi panel, although this created some difficulties for the researcher. However, the advantage of being able to complete the survey quickly far outweighed the problem which personal distribution caused. Above all, the researcher experienced direct contact with the members of the expert panel leading to an increased willingness of the members to participate. Also, conversations with the panel members helped the researcher to understand their particular concerns about the specific issues, which could not have been done in using written replies. The most interesting example related to Question 3 of the first round Delphi. The purpose of the question was to ask the experts their opinion about the most appropriate length time for an EC laboratory session. Because of a minor mistake in translation, the participants were led to believe that the question was asking for the length time for one experiment instead of one session of experiments. Many of the experts replied using the unintended meaning of the question. Some were confused and therefore did not reply. The latter group explained the reason for not answering the question was that it was unclear. By having a direct conversation with the panel members during the distribution process the researcher recognized this problem in the questionnaire design. Consequently, the mistake was not repeated in the second round and as result, the question in the second round was answered by all panel members.

#### CHAPTER 7 EXPERIMENTAL DESIGN

The results of the international literature review of **Chapter 3** have revealed an increasing interest in the development of experiments for use in the environmental chemistry education (ECE) laboratory. These experiments also illustrate the successful implementation of both the integrated approach and the scientific approach in the ECE laboratory. The six primary goals of an ECE laboratory program have been identified as follows: (i) consideration of international, regional, national and local environmental issues; (ii) inclusion of environmental issues of air, water and soil environments; (iii) the use of the integrated approach; (iv) providing students with opportunities to use appropriate instruments and apparatus; (v) the use of real samples and scientific processes; and (vi) providing students with opportunities to improve practical skills, such as sampling, measurement, data collection, data analysis and interpretation, reporting data and the procedure of quality assurance/quality control (QA/QC).

However, the two main criticisms of the above program, which are considered drawbacks of the current international EC curriculum, are that: (i) there is a serious lack of soil-related experiments in ECE laboratories that help demonstrate concerns about environmental problems; and (ii) the demonstration of ECE experiments is often removed from the actual procedure used by environmental chemists, in terms of the insufficient inclusion of some critical environmental analysis processes. These include sample preparation, data analysis and interpretation, and the use of QA/QC.

Chapters 5 and 6, where the Delphi technique was employed, identified criteria for the design and implementation of ECE laboratory experiments in Vietnamese universities.

The results suggest that the above-mentioned primary aims of the international ECE laboratory were also the goals, which Vietnamese EC educators considered should be targeted. In other words, the environmental issue of soil should be one of the concerns in the ECE laboratory. Furthermore, Vietnamese EC educators have recommended that the most suitable time for an experimental period is between 2-5 h.

For these reasons, this study will attempt to design two soil-related EC experiments. The criteria for the Vietnamese ECE laboratory, which are also reflected in the international goals, will be used as guidelines. Acknowledging the current shortcomings of the international EC curriculum in terms of its practical work component, such issues were taken into account in designing the experiments. A brief description of the two experiments is as follows:

**EXPERIMENT 1:** Uses real soil samples and aims at (i) practicing the sample preparation process; and (ii) practicing data analysis and interpretation.

**EXPERIMENT 2:** Uses real soil contaminated with petroleum hydrocarbons; and aims to demonstrate the whole environmental analysis processes, which will help students improve the following practical skills: (i) sample preparation; (ii) data analysis; (iii) data collection; (iii) data interpretation; (iv) data reporting; and (v) QA/QC procedures.

#### Format of the Experiment

In seeking the best format to use in the designed experiments, guidelines set by the Journal of Chemical Education were chosen as being the most appropriate. The Journal of Chemical Education was first published in 1926 by the American Chemical Society, Division of Chemical Education, and ever since has been accessed by chemical educators worldwide. One of its important sections is the "Teaching Laboratory", and its associated laboratory experiments, which aim at "providing chemistry teachers with materials for helping them in their teaching and in their effectiveness in developing the talents of students" (119). To a lesser extent, experiments in this study are for EC teachers.

The following broad guidelines have been given for the formatting of laboratory experiments:

- 1. *The lab summary*: is where the essential information is provided to enable prospective adopters to consider whether it is suitable for their program, before heading to the additional materials. It suggested that the following information should be given in the *lab summary*:
- (i) The rationale for adopting the experiment and an indication of where it fits into the curriculum (course, level).
- (ii) A clear, brief statement of the procedures, techniques, facts, and concepts students will learn and an explanation of "how" and "why" the experiment helps the students learn.
- (iii) A summary of results from any evaluation studies of the efficacy of the experiment in achieving its goals.
- (iv) A list of equipment, chemicals, and/or instruments used in the experiment that are not expected to be available in a typical chemistry department.

- (v) Any other information that would help a potential adopter of the experiment to decide whether or not to put in the effort needed to adapt it for use at another institution.
- 2. *Student materials*, which include the written directions, experimental procedures, handouts, report forms, etc., used by students.
- 3. *Instructor notes* which include: (i) background information; (ii) laboratory preparation and equipment needs; (ii) troubleshooting; (iv) tips for success; and (v) CAS registry numbers for all chemicals.

This format and such criteria were imposed so that the teaching experiments have both pedagogical and technical content, and enable the adopters to be able to apply them in their situation quickly and easily. Some rearrangements, however, were made in this study and below are given the intended guidelines used in this study:

 Proposed experiment, which includes (i) a clear, brief description of the procedures, techniques, facts, and what concepts, techniques and manipulative skills, investigative skills, and affective objectives that will be transferred to students in the experiment; (ii) a list of equipment, chemicals, and/or instruments used in the experiment that are not expected to be available in a typical chemistry department; (iii) recommendation of the suitable background of students; and (iv) the time needed to run the experiment.

- 2. *Experimental explanation* which addresses: "what", "how" and "why" concepts, techniques and manipulative skills, investigative skills, and affective objectives that will be transferred to students in the experiment.
- 3. *Discussion* which demonstrates procedures, "how" concepts, techniques and manipulative skills, investigative skills, and effective objectives studied by the students.

Both the "experimental explanation" and "discussion" will address the rationale for adopting the experiment, and refer to the results from previous studies of the efficacy of the experiment in achieving its goals.

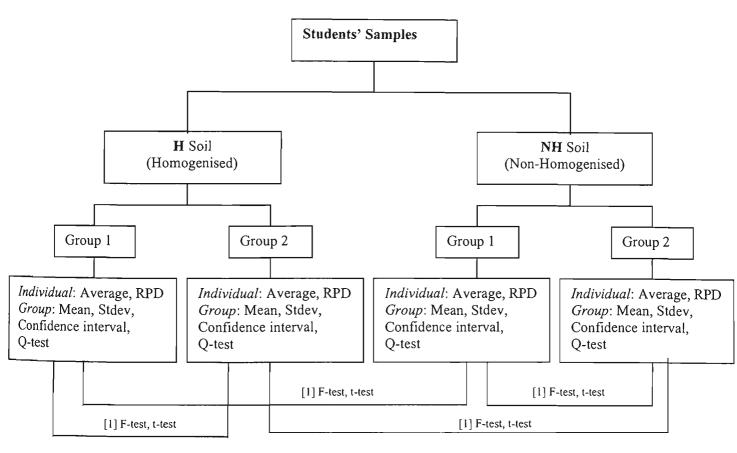
- 4. *Instructor notes*, which include (i) background information; (ii) laboratory preparation, which includes a list of chemicals, equipment, and/or instruments used in the experiment that are not expected to be available in a typical chemistry department; (iii) troubleshooting; (iv) tips for success; and (iv) CAS registry numbers for all chemicals.
- 5. *Materials for students* which include the written directions, experimental procedures, handouts, report forms, etc.

In this study, *instructor notes* and *materials for students* will be placed in Appendix 10-11 for Experiment 1 and Appendix 12-13 for Experiment 2. The details of two experiments will be demonstrated in the following sections.

#### 7.1 PROPOSED EXPERIMENT 1

# The use of a real soil sample to demonstrate interrelationship between sample preparation and sample analysis

The main aims of this experiment are to help students understand the important role of sample preparation in environmental analysis. Towards this goal, two soil samples of similar origin are prepared. One is under good treatment towards the homogenous state and labeled **H**, and the other is not homogenized and labeled **NH**. By applying statistical methods to evaluate the precision of the relative moisture content of two soil samples, students can learn about the effects of sample preparation on the analytical result. The following diagram describes the procedure that students need adopt for this experiment:



RPD: Relative percentage difference of duplicates

Stdev: Standard deviation

In this experiment, students are required to collect their own soil sample (approximately 300-400 g). They will be asked to divide their samples into two portions and place these in two pre-labeled jars, namely jar **H** and **NH**. Students will be given instructions for the treatment of the two soils. Each student in a group of five (or more) is asked to obtain duplicates of two soil samples. They then determine the moisture content in each soil by using a gravimetric method (instructions are given). After gathering group and class data, students are required to apply some important statistical methods and to answer set questions to help them understand and interpret the results. For example, students will use the Q-test to examine whether any suspect value(s) should be rejected or not. They also need to use the F-test and t-test for the evaluation of the precision of their results. From this information, students can gain a sound understanding of the effect of the sample preparation on the analytical data, which in this case is the moisture content.

For this experiment to be successful there must be a distinct difference between the homogenization levels of the two soil samples, **H** and **NH**. Students will need specific advice on (i) the type of soils they should collect. Soil texture with a clay content of greater than 20%, such as loam, silty loam, sandy clay loam, clay loam, light clay or medium clay are advised to be used. The reasons for the choice of such soils, but not sand, loamy sand, clay sand or sandy loam, which all has clay content of less than 20%, is that water in soils with low clay content is often easily distributed throughout (120). In other words, the sample preparation for these specific types of soil does not greatly affect the analytical result. These, particularly, are not suitable for this experiment, since the objective is to help students to recognise the effect of sample preparation; and (ii) how to obtain the **H** soil with considerable level of homogenization.

This experiment can help students not only enhance their practical skills, in terms of the use of the gravimetric method for the determination of moisture content in soil and the procedure employed to obtain the representative sample from the bulk sample, but can also provide students with the opportunity to gain experience with the use of statistical methods in a real situation. It can be used for students with a general chemistry background, who have a basic knowledge of statistical analysis. It is suitable for use in a three-hour ECE laboratory or in a three-hour analytical laboratory after the statistical method is introduced. The experiment uses facilities that are readily available, and can therefore be implemented in a typical chemistry laboratory. **Table 1** represents a typical set of moisture content data for a clay loam. Here, the results were obtained by five students per group.

Moisture content (%)				
Gro	Group 1		oup 2	
H soil	NH soil	H soil	NH soil	
12.31	11.95	12.51	11.54	
12.50	12.32	12.35	9.69	
12.68	15.21	12.40	12.25	
12.45	11.25	12.28	12.01	
11.12	14.21	12.41	11.01	

Table 1

#### 7.1.1 Experimental explanation

#### 7.1.1.1 Sample Preparation

For NH soil, students are asked to shake the jar several times and then leave it to stand ready to use. For H soil, they are required to homogenize the soil by vigorously shaking, mixing and then spreading it on a clean steel tray before cutting it into quarters, in accordance with the so-called "cone and quarter" method (48). This provides students with the opportunity to gain hands-on experience with an acceptable procedure for obtaining a representative sample from a bulk sample.

#### 7.1.1.2 Moisture Content Determination - Data Analysis

Each student is asked to take duplicates of each soil and analyze these for moisture content. Their data are recorded on the class data sheet (provided) and they are asked to evaluate the reproducibility of the measurements from two soils. To do this, students have to be familiar with the concept of relative percentage difference of duplicates (RPD) (93).

The students then are asked to pool their group data to determine the mean, standard deviation (Stdev) and confidence interval of the mean (95% confidence) for both soils. Before doing the comparison, students are required to identify if there is any "suspect" value. Toward this task, students have to use the Q-test. They are then asked to gather class data to evaluate the precision of individual group data. In this task, students have the opportunity to use the F-test and t-test.

In a group and for each soil, students are required to identify any suspect result. It has been observed that, as "analyst learners", results that students obtain from an analytical procedure can be significantly different (121). In this experiment, students can test for suspect values in both the **NH** and **H** samples. In both cases, the use of the Q-test can help them to examine whether such data should be rejected or not. For example, in **Table 2**, the suspected values are shown in bold font. If students find that Q-calculated is greater than Q-critical, then they can conclude that the suspected data should be rejected (122). In this case, students are required to recalculate the mean and the standard deviation. From this, students can recognise how the outlier affects the final result. Of course, if the Q-test is passed, it means that the suspect value should be kept.

## Table 2 Q-test of typical group data

	Group 1		Group 2	
	H soil	NH soil	H soil	NH soil
	12.31	11.95	12.51	11.54
	12.50	12.32	12.35	9.69
	12.68	15.21	12.40	12.25
	12.45	11.25	12.28	12.01
	11.12	14.21	12.41	11.01
Mean	12.21	12.99	12.39	11.30
Stdev	0.62	1.66	0.08	1.02
Range	0.78	2.06	0.11	1.26
95% Confidence Interval	$12.21 \pm 0.78$	$12.99 \pm 2.06$	$12.39 \pm 0.11$	$11.30 \pm 1.26$
Q-test	<u>.</u>		J	
Q-critical value ( $P = 0.05$ )	0.717	0.717		0.717
Q-calculated	-0.763	0.253		-0.516
Conclusion	Q-test failed	Q-test passed		Q-test passed
Results after Q-test				
Mean	12.49	12.99	12.39	11.30
Stdev	0.15	1.66	0.08	1.02
n	4	5	5	5

*Note*: In this case, group 2 did not conduct Q-test on **H** soil due to the insignificance difference among data.

In a group, students are asked to compare which result from the two soils is more precise and whether the moisture content they found in the two soils is the same or not. To do this, students are exposed to the use of the null hypothesis in the F-test and t-test. Two situations that students can encounter are:

- (i) An F-test pass which mean there is no significant difference between the precision of the two moisture contents obtained from the H and NH soils. This rarely happens for soil with a clay content greater than 20% (as is suggested to be used in this experiment).
- (ii) An F-test fail is very likely to happen with the use of the suggested soil and where H soil is considerably homogenized. In this case, students can conclude that the data from NH soil are less precise than those from H soil. This can, therefore, illustrate to students the effect of sample preparation on the analytical results.

	Group 1		Group 2	
	H soil	NH soil	H soil	NH soil
Mean	12.49	12.99	12.39	11.30
Stdev	0.15	1.66	0.08	1.02
n	4	5	5	5
F-test	ay, constanting <b>a</b> second s		0907-000-000-000-000-000-000-000-000-000	
F-critical value ( $P = 0.05$ )	15	.10	9	.61
F-calculated	117.42		161.73	
Conclusion	F-test failed		F-test failed	

Table 3 F-test for the comparison of the precision between two soils in each grou	Table 3 F-test for the	comparison of the	precision between	two soils in each grou
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After using the null hypothesis for the F-test to find out whether the two sets of data have the same precision, students may find that the F-calculated value is greater than the F-critical value. This leads them to the conclusion that the data derived from **H** soil are more precise than from the **NH** soil. This is a clear case, which helps students recognize how sample preparation affects the quality of the analytical results and how careful data analysis may help to reveal this.

In the class, for each soil, two groups are required to pool their data and compare which group's data are more precise and whether the mean moisture value they found is different in each soil. Again, students have to use the null hypothesis for the F-test and t-test. As the experiment is conducted using one sample (i.e. **NH** sample due to its non-homogenous state) the F-test can fail or pass. For the **H** soil, however, the result obtained from the two groups should be similar but the data from which the result has been derived will clearly be different. In both cases, students are provided with a good opportunity to experiment with the use of statistical methods in order to evaluate and interpret their results. In the case where students find the value from one group is more precise than that of the other, it will help them to realize that the sample preparation procedure can have an effect on the analytical result.

In this experiment, when students accumulate their data, they may obtain the following results similar to the following:

	H soil		NH soil	
	Group 1	Group 2	Group 1	Group 2
Mean	12.49	12.39	12.99	11.30
Stdev	0.15	0.08	1.66	1.02
n	4	5	5	5
F-test		<u></u>		
F-critical value ( $P = 0.05$ )	9.979		9.605	
F-calculated	0.527		2.649	
Conclusion	F-test passed		F-test passed	
t-test				
t-critical value ( $P = 0.05$ )	2.36		2.31	
t-calculated	0.909		1.472	
Conclusion	t-test passed		t-test passed	

Table 4 F-test and t-test in class data for two soils

In both soils, the two groups have performed the F-test and passed the data. This helps students to understand that for each soil, they have achieved the same precision (though the value may be different). Therefore, students can use the pooled standard deviation to calculate t for the t-test in order to examine whether the mean value of the moisture in each soil is, for two groups, different or not. In this example, students would find their data to have the same precision because the t-test is passed in both soils.

Students may also be interested in pooling all results from the two groups to calculate the mean and standard deviation for each soil, and then use the F-test and the t-test to find out whether the number of data used in the tests affects the precision of the value.

#### 7.1.2 Discussion

#### 7.1.2.1 Sample Preparation

Sample preparation, which involves obtaining a representative sample from the bulk sample or the gross sample, is the first step of any analytical process (42). Its important and challenging role in EC analysis has been discussed in **Chapter 3**. The difficulties are due to the non-homogeneity of environmental samples in general and soil samples in particular. It has also been recognized, however, that sample preparation is very rarely undertaken in the chemical education laboratory (**Chapter 3**).

The reflection of the professional process in the ECE laboratory is one of the ultimate goals of EC educators (Chapter 3). The importance of sample preparation in the EC laboratory, and the lack of inclusion of this issue in the ECE laboratory to date, highlight a deficiency in the EC curriculum. In some analytical chemistry and environmental analytical chemistry textbooks, sample preparation has been described (31, 41, 42). Therefore, students may have some knowledge of this from classroom activities. However, the question to be raised here is how to communicate this issue effectively to students in the laboratory. An attempt has been made, for example, to demonstrate to students the importance of the sample preparation procedure (48). It is presented through showing the students how challenging the job is in order to obtain a representative sample from a real sample. In the current study, the proposed experiment presents another approach toward helping students to understand the vital role of sample preparation in determining the quality of laboratory data. This has been achieved by taking advantage of the rapidity and simplicity of the gravimetric method for the determination of moisture in soil (as the analytical method) and by using real soil samples and subsequent statistical analysis of the data.

#### 7.1.2.2 Gravimetric Method - Moisture Content

The moisture content of solid samples such as soils is a common parameter and is used in reported analytical results on a "free dry" basis in analytical laboratories as well as in EC laboratories (31, 41). Moisture content is often analyzed by using a gravimetric method (31, 41). It relies on the use of an analytical balance, which is an important and frequently used instrument, to measure mass with high precision (41, 123, 124). It is beneficial for students to gain hands-on experience with this procedure in the ECE laboratory.

#### 7.1.2.3 Data Analysis

The pivotal role of data analysis and interpretation in chemical analysis, particularly in the EC laboratory has been discussed in **Chapter 3**. One of the reasons for the negligence in attempting to reflect these procedures in the chemical curriculum is time constraint (71). It is often considered to be too time-consuming to obtain valid sets of data for analysis (97). Efforts have been made by chemical educators using illustrative examples, but it can be argued these have failed to help students to thoroughly understand the principles. Therefore, students are uncomfortable when they have to apply statistics in real situations (125). Innovative solutions that have been proposed to overcome this problem involve the use of such simple experiments to enable students to access data quickly (97, 125). However, a criticism of such simple experiments is their inability to raise students' enthusiasm, due to the failure of using real-world samples (97, 126). Therefore, to effectively enhance data analysis in the teaching laboratory, an experiment is desirable, which has the following two features: (i) quick access to data; and (ii) use of real-world samples. In this experiment, these concerns have been addressed by the taking advantage of the gravimetric method to rapidly obtain data on the moisture content of real soil samples.

Therefore, by using real samples and the gravimetric method to analyse the moisture content of soils, students are provided with an opportunity to practice data analysis. In doing this experiment, students not only enhance their practical skills but also improve their problem solving skills.

#### 7.2 PROPOSED EXPERIMENT 2

## The analysis of total petroleum hydrocarbons (TPH) in soil: an integrated environmental chemistry experiment

Petroleum hydrocarbon products are complex mixtures of hundreds of individual components, including straight-chained, branched and cyclic alkanes, alkenes, as well as monocyclic and polycyclic aromatic hydrocarbons (37). They can be divided into volatile and semivolatile fractions. The volatile fraction includes  $C_{1}$ - $C_{5}$  and  $C_{6}$ - $C_{9}$  hydrocarbon (petrol). The semivolatile fraction includes  $C_{10}$ - $C_{14}$  hydrocarbon (kerosene);  $C_{15}$ - $C_{28}$  hydrocarbon (heavy kerosene and lubricating oils); and  $C_{29}$ - $C_{36}$  hydrocarbon (asphalt).

The contamination of soil by petroleum hydrocarbons is a worldwide problem, which is increasing in frequency and complexity (127). Using the traditional methods such as gravimetry and infrared spectroscopy for the determination of total petroleum hydrocarbons (TPH) is not possible to identify the presence of specific/group compounds. Instead, gas chromatography with flame ionisation detection (GC/FID) has been used broadly as one of the most effective instruments to help identify and quantify individual and/or group hydrocarbons in TPH components (127). One of the first steps of the analytical procedure is to convert the sample into a suitable form, which often involves the use of extraction techniques (42). Sonication, which has been approved by the environmental protection agency (EPA) to be used for the extraction of semivolatile organic compounds, has been used largely as an extraction technique for the determination of TPH in soil (127).

A government environmental analytical laboratory method for the analysis of petroleum hydrocarbons in soil (128) was adopted, which requires the use of the sonication for the extraction and GC/FID (BPX5, 25 m  $\times$  0.22 mm I.D.  $\times$  0.25  $\mu m$  F.T. column) for the quantification of the petroleum hydrocarbons. The proposed experiment will provide students with the opportunity to experience a method used in a professional environmental research laboratory. By doing the proposed experiment, students have the opportunity to enhance their practical skills with the use of GC/FID for analysis of semivolatile organic compounds. They also have the experience of using a sonicator to extract semivolatile organic components from solid samples. Significantly, the QA/QC approach is integrated into the procedure to provide students with the opportunity to learn the important steps required in this approach and how to apply these in the evaluation of analytical results. The proposed experiment can be carried out in two sessions each of four to five hours duration in a quantitative analytical or environmental chemistry laboratory. Students will require a basic background in GC and statistics.

Towards the success of this experiment, providing the students with areal a soil sample containing petroleum hydrocarbon contamination is an important element. Fortunately, it is not difficult to find such contaminated soils. They are very likely to be found in car parking areas, areas near petrol service stations, industrial refinery plants or even in areas near railways. It could also a good idea to ask for samples of contaminated soil which are no longer required by industrial analytical laboratories or environmental analytical laboratories. For example, in Australia, The Australian Government Analytical Laboratories (AGAL) are organisation from where such samples might be obtained. One, however, can also produce the petroleum-contaminated soil by spiking

petrol or diesel into a normal soil sample that can be taken from any garden.

#### 7.2.1 Experimental Explanation

#### 7.2.1.1 Session 1

Students are briefly introduced to the application of the GC/FID for analyzing petroleum hydrocarbons. They are also introduced to the use of QC analysis to evaluate the condition of the measurement system (analytical instrumentation) and the effect of a sample matrix on analytical data. This leads to the introduction of the use of control charts. Students should be aware of the use of control charts for QC analysis and the use of QC samples such as reagent blanks, duplicates of samples or laboratory-fortified blank (LFB), laboratory blank matrix (LBM), surrogate standard. Such an introduction also leads students to understand that, in order to plot a control chart, they need to gather class data.

Particularly, in this session, students should be introduced to the use of QC analysis and its associated samples (e.g. LFM) to evaluate the effect of a matrix on their results. Students are encouraged to discuss possible factors that can affect the percentage recoveries of the LFM, such as the effectiveness of the extraction (solvents and techniques) and the nature of the matrix. Therefore, they may investigate to what extent such factors affect the result by proposing an appropriate experimental design.

After the students given a clear idea as to what samples they will need for the QC procedure they will then prepare a sample for GC/FID analysis in readiness for the next session. In doing this, they are provided with the opportunity to improve their practical skills such as: (i) spiking technique in preparation for LFM and LBM; and (ii) using

sonication for the extraction of petroleum hydrocarbons from soil.

#### 7.2.1.2 Session 2

In the laboratory, students are required to quantify the petroleum hydrocarbons by using GC/FID. They are asked to answer some prepared questions, which aim at helping them to: (i) understand the use of QC procedures for the evaluation of their results; and (ii) to identify the source of the contaminants from the sample they analyzed. Towards these tasks, students are required to be familiar with some basic statistics. For example, they should find out the RPD of the duplicate, and the percentage recoveries for the LBM and LFM samples. Particularly, when the class has completed these experiments, students will be provided with further opportunities: (i) to gain hands-on experience with the use of statistical methods for the interpretation of suspect values, etc. (all associated questions are given in the **Appendix 13**); and (ii) to extend their understanding about the use of QC analysis for the evaluation of the analytical system by requiring them to specify the method of detection and to evaluate reagent effect(s) on the analytical result, etc.

Figure 4 and 5 show the typical chromatograms of the petroleum hydrocarbons standard and a contaminated sample.

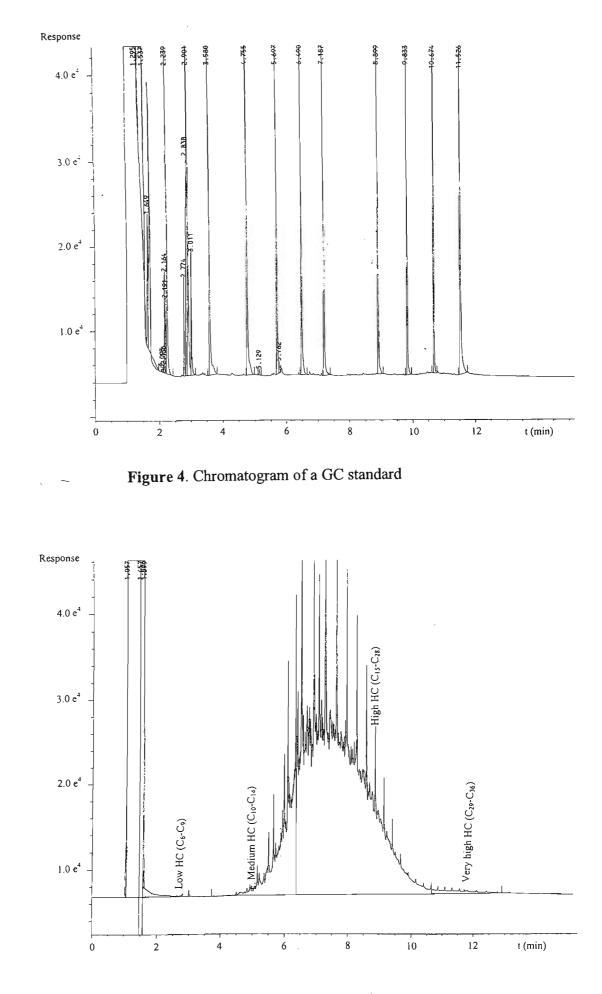


Figure 5. Chromatogram of the sample

Based on the area of the standard and the area of sample, the typical concentrations of the TPH fractions are calculated and the results are as follows:

TPH range	TPH
	(mg kg <sup>-1</sup> )
Low HC C <sub>6</sub> -C <sub>9</sub>	0
Medium HC C <sub>10</sub> -C <sub>14</sub>	1,193
High HC C <sub>15</sub> -C <sub>28</sub>	5,484
Very high HC C <sub>29</sub> -C <sub>36</sub>	1,403
ТРН	8,079

Table 5 Result of petroleum hydrocarbons in contaminated soil

The level of each petroleum hydrocarbon range can help students predict the source of contaminants in soil. For example, from the results in the **Table 5** students can conclude that a significantly large amount of contamination is due to high HC or heavy kerosene and lubricating oils.

By comparing the percentage recovery calculation for LFM and LBM samples, students can evaluate the matrix effect on the analytical measurement. Having reached such a conclusion, they should understand that having QC samples is important as they can use these to evaluate the quality of the measurement. Having the percentage recoveries of the LFM can help students understand that the value "TPH concentration in soil" is not the precise term to use, as it does not indicate the true amount of TPH in soil; it is more accurately the extractable amount. Students should understand that the percentage recoveries of LFM could tell how close the measurement is to the real amount of TPH in soil. Therefore, an increase in these recoveries is likely to indicate a better reliability of the measurement. Students will learn that the solvents, the techniques used in extraction, and the characteristics of the matrix, may all affect the

percentage recoveries.

A good exercise is for students to test their hypothesis by experimental design. For example, in this experiment the typical percentage recovery of LFM is 81% and the percentage recovery of LBM is 98%. These data suggest that the matrix has an effect on the recoveries of the analytes of interest. Only 81% of the analyte of interest is measured, the rest is not. It leads students to understand that the data they obtained is not the actual amount of TPH in the soil. It is only likely to be 81% of the true value. In other words, students can estimate how close the real TPH value in soil is to the data they have. It can also help students to understand that while the analytical measurement is not the true value, the percentage recoveries from the LFM must be included in the report to make the measured data meaningful.

Finally, when the class work for this experiment is completed, the students can be asked to gather class data together to produce the final analytical result. This will provide further opportunities to practise data analysis, and to evaluate the accuracy and precision of their results within the context of the class's data. They can also be provided with further QC activities by asking them to plot the control chart from all data of: (i) TPH concentration in the reagent blank; (ii) RPD of duplicates; (iii) percentage recoveries of the LBM; and (iv) concentration of the standard.

Therefore, in doing this experiment, students are provided with the opportunity to practice the entire procedure for determining TPH in soil. It enables students to improve their practical skills, and concept learning. Particularly, the integrated QC in the procedure enables students to identify the problems associated with environmental

analysis and also to help students to find the solution to such problems. It thus helps students to enhance their problem solving skills.

### 7.2.2 Discussion

The environmental issue of soils in general and soil contaminated with petroleum hydrocarbons in particular has been found to be insufficiently demonstrated in the ECE laboratory (**Chapter 3**). This is in spite of the fact that petroleum hydrocarbons are collectively one of the most common contaminants in soils. The potential hazard these present to human health has become an issue of concern for environmental scientists (**Chapter 3**). Therefore, in order to keep abreast of the current environmental problems, the EC curriculum in general and the ECE laboratory in particular need to address this issue. The proposed experiment is a response to the shortage of such practical material in the EC curriculum.

Whilst it is recognised that soil contaminated with petroleum hydrocarbons is quite complex, and the use of traditional methods (e.g. FTIR or gravimetry) are insufficient for the purpose of tracing for the source of the contaminants, the use of GC/FID is becoming prominent in the scientific laboratory (127). In the EC curriculum, the use of instruments in the ECE laboratory and its benefits to students has been discussed in **Chapter 3**. The major advantages to teachers of using instrumentation at the undergraduate level are that it enables them to: (i) provide students with a good opportunity to practice appropriate technical skills; (ii) improve students' investigative skills; (iii) provide students with a good opportunity to learn new concepts and revise old concepts; and (iv) improves students' attitudes to science.

Given (i) the routine use of GC/FID in analyzing petroleum hydrocarbon contaminants in soil; (ii) the wide availability of GC equipment in most undergraduate chemistry laboratories; (iii) the benefit that students may have by using such instruments; and (iv) the surprising finding that there are seemingly no reported teaching experiments in the ECE laboratory that are concerned with the analysis of petroleum hydrocarbons in soils, the proposed experiment is expected to meet the needs of the EC curriculum.

One of the important steps in environmental analysis is to transfer the sample into the "analytical state". In other words, the analytes have to be extracted into a suitable solvent. For the extraction of petroleum hydrocarbons from soils, there are several extractive methods, which include Soxhlet and sonication extraction. Though Soxhlet extraction is considered to be beneficial to students in gaining hands-on experience (121, 129), it is time-consuming and perhaps inappropriate for this kind of experiment, given that one laboratory session should not exceed five hours (**Chapter 6**). One needs to seriously consider the availability of time if this method is conducted as extraction periods of as much as 24 h are often required (121). With such long extraction time, it is concerned that Soxhlet extraction is not likely to be workable in the educational laboratory experiment.

In the proposed two-session laboratory experiment, students will prepare all appropriate samples in the first session. In the second session, they will use GC/FID for analysis and process the data. Consideration must be given to how the work to be done by the students is divided between the two sessions. It has been argued that sample preparation and analysis should be included in one laboratory period (121). Consideration must also be given to use of the GC since interruption of an experiment

for more than one or two hours is likely to change the instrument response (56). Thus, students should not plot the calibration curve in one session and run the samples in the other (121). This suggests that, for the multiple laboratory session experiments, instructors should carefully consider the breakdown of tasks between sessions to ensure the scientific and pedagogic aspects.

One of the serious problems of the EC curriculum, which has been recognized in the ECE laboratory, is insufficient consideration of QA/QC. This is considered to be the main reason for students' misunderstanding the important role of this procedure in the scientific laboratory. This is also blamed for the students having little confidence when they conduct QA/QC procedures (**Chapter 3**). In this experiment, the QA/QC procedure is particularly emphasized. One of the most important parts of QA is the use of QC samples (both field QC samples, and laboratory QC samples) to evaluate the performance of the measurement system and the matrix effects on the produced data. In the teaching laboratory, the preferable and perhaps most suitable way of introducing the concept of QA/QC, is via the use of laboratory QC samples as it relates directly to the use of "in house" samples.

Indeed, a survey of the literature has found that control charts, which is one way of using laboratory QC samples to evaluate the performance of the instrumentation, have been extensively used in the analytical teaching laboratory (46, 99, 100). In order to introduce the concept of QC through the use of control charts, a large number of data are needed, and this may take a significantly long period of time, typically one semester (99, 100). Experiments, which are often used to demonstrate this concept, are simple analyses such as titrations, or conductance measurements. Here, students are asked to

repeat the experiments using sample duplicates, reagent blanks, and surrogate standards. The inclusion of QA/QC in such teaching laboratories has produced great benefits to students. These include: the enhancement of the student's identification and problem solving skills, an increase in the students' excitement and interest in the laboratory, and more importantly students are found to be more confident in their related work when they graduate (**Chapter 3**).

A control chart is one of the important components of OC. Adopting this procedure helps students gain hands-on experience in creating and using control charts to evaluate the validity of the laboratory data based on QC samples. Therefore, it is worthwhile to address this issue in the ECE laboratory. However, another important role of the use of QC samples is to evaluate the matrix effect on the result. This is particularly important in environmental analytical analysis due to the "matrix nature" of environmental samples (31). The proposed experiment, therefore, has focused on providing students with the opportunity to practice QC for this specific purpose. Interestingly, the introduction of QC to students by using QC samples for the evaluation of matrix effects can be done after they finish their laboratory work. In this regard, it has the advantage over introducing students to the use of QC samples through control charts, which normally takes several weeks, months or terms to have enough data to plot the chart and understand how it is used. Nevertheless, this does not mean that in the ECE laboratory students should only be introduced to the use of QC to evaluate the matrix effect on results. Instead, they should be exposed to both procedures, as these are what graduates have to be familiar with in associated environmental analytical work.

Soil contaminated with petroleum hydrocarbons is a worldwide environmental issue, particularly in Vietnam. The increase in oil and gas exploitation, its transportation and

processing, are potential sources of these contaminants for not only the marine but also for the soil environment (18). Moreover, the Delphi findings from the Vietnamese EC educators have suggested that GC/FID is one of the four most desirable instruments to be used in the EC laboratory in Vietnamese universities (**Chapter 6**). Therefore, this experiment is also suitable for use in the Vietnamese context.

### **CHAPTER 8 SIGNIFICANCE OF THE RESEARCH**

### 8.1 New Knowledge

The survey of the literature on EE has shown the appropriate approach that should be followed in the development of EE curricula internationally and in Vietnam in particular; that is education *for* the environment following the guidelines of the Tbilisi Declaration (1).

From the literature review of EE and chemical education laboratory programs, which are the two interrelated subjects of EC, together with the critical review of the available ECE laboratory programs in the literature, the "ideal" criteria for the ECE laboratory have been identified. The review suggests a good ECE experiment should:

- 1. Consider the international, regional, national and local environmental issues.
- 2. Include the environmental issues of air, water and soil environments.
- 3. Use the integrated approach.
- 4. Provide students with opportunities to practice appropriate using instruments and apparatus.
- 5. Use real samples and scientific processes.
- 6. Help students with opportunities to improve the following environmental chemistry skills. sample preparation, measurement, data collection and analysis, interpretation and report data, along with the QA/QC procedure.

This set of criteria would be useful information for international EC educators in the development of such programs, as there are no agreed guidelines yet available. More importantly, the research has also identified current shortcomings of the international EC curriculum, in which there is a serious lack of consideration of the environmental

issue of soil in students' EC materials (including textbooks and practical work). Additionally, it has identified the drawbacks of ECE laboratory programs in terms of the insufficiency and ineffectiveness in providing students with opportunities to be exposed to the important procedures, which are normally done in the professional EC laboratory. Such drawbacks include deficiency in students' practical work related to processes such as sample preparation, data analysis and interpretation, and most seriously the QA/QC procedure. These have led to negative effects on EC undergraduate students, in terms of the improvement of practical skills, concept learning and attitudes to science. These findings, therefore, would provide international EC curriculum planners in general and EC educators in particular with a basis for the development of more effective programs.

The survey of the literature on chemical education has clearly identified that the EC curriculum is in a relatively advanced state in developed countries such as Australia. Great attention has also been paid to up-dating textbooks and in the development of new EC experiments. These provide Vietnamese EC educators with an overview of the increased interest in the development of the ECE laboratory programs internationally. The survey will help them towards recognising the need to pay more attention to international trends in their field of interest, while they acknowledge the current proposals for development of EC curriculum in Vietnam.

Yet, to keep abreast with the international trend in the development of the ECE laboratory, Vietnamese universities have great difficulties in accessing facilities. However, the studies have also clarified feasible solutions for the equipment problem, of which the most suitable is the use of the integrated laboratory experiment. Indeed,

this approach has been used broadly in the EC curriculum and has also been considered to be useful and beneficially workable in the Vietnamese situation. Of particular importance in this regard is that, from the application of the modified Delphi technique, the above-mentioned international "ideal" criteria have been given consideration by the leading Vietnamese EC educators. They have come to the consensus that such internationally accepted criteria will be applicable in the ECE laboratory in Vietnamese universities. Therefore, this provides useful guidelines for the national Vietnamese EC educators, who are interested in development of such programs.

The literature provided an overview of the relative importance accorded to particular environmental problems (air, water and soil) in reported undergraduate chemistry courses. Surprisingly, the environmental issue related to soil has been identified as being seriously under-represented in the international EC curriculum. The reading of the literature strongly suggests a need for more soil-related materials in environmental chemistry textbooks and experiments. Particularly, in developing countries such as Vietnam, where soil quality is an important aspect of that country's economic development, the soil question should therefore, be accorded a critically focus in the development of ECE laboratory programs.

### **8.2 Practical Laboratory Experiment**

The research has identified specific weaknesses in the EC curriculum and EC laboratory program. The two experiments tried and evaluated in the research directly address the curriculum and laboratory deficiencies in the research. The problems of the sample preparation and data analysis, which are two important procedures in the EC laboratory, are addressed in **Experiment 1**. The other issue of soil contaminated with

petroleum hydrocarbons and the ignorance of QA/QC to ensure defendable data in the international EC curriculum are both addressed in the **Experiment 2**.

The development of such experiments based on guidelines for the development of ECE laboratory in Vietnamese universities, which also address the international interest, therefore would serve as a useful curricular source for EC educators. In the development of such experiments, the most important issue is how to design experiments so that they can be not only used in the well-equipped laboratory but also can be adaptable to Vietnamese circumstances. Therefore, such experiments would need to be feasible in the Vietnamese University laboratories' conditions.

The two proposed experiments have presented the integration of the two approaches in EE: education *in* the environment was used as the foundation for the ultimate purpose, the *for* approach. The two experiments have both used real environmental samples as the predominant ECE materials. **Chapter 3** has shown that by doing so, students are provided with unique opportunities to practice with challenging problems, that are often associated with work in the environmental science area. Significantly, students are often found to be more interested in such work, as their curiosity toward understanding the environmental problems are met. In other words, by using real-world environmental samples, in EC laboratory programs, students can obtain appropriate knowledge and skills. These can help them have more awareness of environmental problems. Thus, the use of real environmental samples is an example of the education *in* the environment approach, which has been discussed in **Chapter 2**.

Interestingly, previous studies have shown that through the challenge of using realworld environmental samples, students are enable to understand the complexity of the environment (**Chapter 3**). This helps enhance their problem solving skills and therefore enables them to use their knowledge for the improvement and protection of the environment more effectively. By doing this, these processes can be referred to as the education *for* the environment approach, which is the ultimate purpose of EE of which EC is one component (**Chapter 2**).

Indeed, in **Experiment 1**, a real soil sample is used to provide students with the context to improve their practical skills such as the use of gravimetric methods to analyse moisture content in soil. From these, they have an opportunity to practice with data analysis. From the deep understanding of their results, students are able to identify problems associated with the sample preparation. In doing this experiment students can also enhance their problem solving skills.

In Experiment 2, by using soil contaminated with petroleum hydrocarbons, students are provided with the opportunity to improve their practical skills and problem solving skills. From using GC/FID students are enable to identify the source of TPH contaminants in soil, and therefore can help them predict the cause of the soil contamination. In addition, by having a chance to practice using QA/QC procedure, students are able to understand and identify associated problems in environmental analytical procedures. Students are also able to recognise that QC can be used as the solution to the uncertainty in the environmental analytical procedure due to the complexity of the natural matrix of typical environmental samples. These problem solving skills, which the proposed EC experiments enable students to gain, are likely to

help them participate in solving associated environmental problems and contributing to the improvement and protection of the environment confidently and effectively.

Therefore, it would be said that in the ECE laboratory, the use of the *in* approach provides a useful context toward the ultimate goal of EE, education *for* the environment. From working with real environmental problems, students are able to identify the causes, propose appropriate solutions, and develop problem-solving skills. With such knowledge and skills students can effectively participate in the improvement and protection of the environment. In other words, the *in* approach is strongly suggested in use with the *for* approach to achieve the target of EE and EC courses.

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### THE GROUPS OF CONTAMINANTS IN SOILS

- 1. Heavy metals: The most important heavy metals with regard to potential hazards and occurrence in contaminated soils are: As, Cd, Cr, Hg, Pb, and Zn. They are mainly from tanneries, wood-preserving activities, mining wastes, cattle dips, smelters, etc.
- 2. Hydrocarbon pollutants: These include alkanes, aromatic hydrocarbons, and organic constituents of some petroleum deposits. They may appear at the sites of petrol stations, fuel depots and may also be due to the accidental spillage and disposal of waste chemicals, etc.
- 3. Toxic organic micropollutants: The most common are polycyclic aromatic hydrocarbons (PAHs), polyheterocyclic hydrocarbons (PHHs), polychlorodibenzodioxin (PCDD) and polychlorodibenzofuran (PCDF) which may appear in petroleum hydrocarbons and coal gas plants. Others include pesticides such as 1,1-di(4-chlorophenyl)-trichloroethane (DDT), dieldrin, etc., which are residues from agricultural chemical wastes such as cattle dips.
- 4. Other industrial chemicals such as solvents, polychlorinated biphenyls (PCBs), acids, alkalis, etc. which come from the disposal of waste chemicals and industrial operations
- 5. Nutrient-rich wastes.
- 6. Radionuclides.
- 7. Pathogenic organisms.

### SUMMARY OF THE SOIL CONTAMINATION PATHWAY

- Direct ingestion of contaminated soil (by gardeners, children, animals and on unwashed vegetables). In such cases compounds of As, Cd, Pb, CN-, Cr+ 6, Hg, coal tars (PAHs), PCBs, dioxins, phenols, pathogenic bacteria, viruses, eggs of parasites are the main contaminants.
- 2. Inhalation of dusts and volatiles from contaminated soil. Organic solvents (toluene, benzene, xylene) are often associated with this.
- 3. Uptake by crop plants of pollutants hazardous to animals and humans through the food chain. These include heavy metals (As, Cd, <sup>137</sup>Cs, Hg, Pb, <sup>90</sup>Sr, Tl), PAHs, and various pesticides.
- 4. Phytotoxicity by compounds of SO<sub>4</sub><sup>2-</sup>, Cu, Ni, Zn, CH<sub>4</sub>, Cr and B may be envolved.
- 5. Toxicity to soil microbial biomass by metals such as species of Cd, Cu, Ni and Zn.
- 6. Deterioration of building materials and services. Contaminants such as  $SO_4^{2-}$ ,  $SO_3^{2-}$ , Cl<sup>-</sup>, tar, phenols, mineral oils, and organic solvents may be included.
- Fires and explosions. Species such as CH<sub>4</sub>, S, coal, and coke dust, petroleum oil, tar, rubber, plastics, high calorific value wastes (from old landfills) can be produced.
- Contact of people with contaminants during demolition and site development: Species such as Tars (PAHs), phenols, asbestos, radionuclides, PCBs, TCDs, pathogenic bacteria and viruses may be involved.
- 9. Contamination of water. CN<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, metal salts, hydrocarbons, solvents, surfactants, sewage and farm waste pesticides are examples.

### SUMMARY OF THE TOXICITY AND THE HEALTH EFFECTS OF SOME REPRESENTATIVE PETROLEUM HYDROCARBONS

Product	Compound	TLV	Potential health effects
Gasoline	benzene	10	Nausea, vomiting, known human
			carcinogen
	toluene	100	CNS depression, fatigue,
			weakness, eye irritant
	xylenes	100	CNS depression, nausea,
			vomiting, skin irritant
	ethylbenzene	100	CNS depression, nausea, vomiting
			liver/kidney damage.
	n-hexane	50	Nausea, dizziness, vomiting,
			severe but reversible paralysis
	other hexane isomers	500	Skin, mucous membrane irritant
	octane	300	Weakness, fatigue, headache,
			nausea, vomiting, anorexia,
			diarrhea.
Middle	PAHs naphthalene	10	Weakness, tremors, dizziness,
distillates			vomiting
	benzo(a) anthracene	0	Probable human carcinogens
	benzo(a) pyrene	0	Probable human carcinogens
Residual	PAHs benzo(a) anthracene	0	Probable human carcinogens
fuels	benzo(a) pyrene	0	Probable human carcinogens
	crysene	0	Probable human carcinogens

TLV: Threshold limit values, values are time-weighted average exposures in ppm for vapors CNS: Central Nervous System.

## LISTS OF ENVIRONMENTAL CHEMISTRY TEXTBOOKS PUBLISHED OVER THE PERIOD 1972 TO 2000

Published	Textbook		Numbe	Number of pages	Sa
year		Air	Water	Soil	Total
1972	Stephen, H. S.; Seager, S. L. Environmental chemistry: Air and water pollution, 1 <sup>st</sup> ed. Scott, Foreman and Company, USA.	88	95	0	184
1973	Manahan, S. E. Environmental chemistry, 1 <sup>st</sup> ed. Willard Grant Press, Boston, USA.	106	225	17	392
1975	Manahan, S. E. Environmental chemistry, 2 <sup>nd</sup> ed. Willard Grant Press, Boston, USA.	145	287	24	532
	Stephen, H. S.; Seager, S. L. Environmental chemistry: Air and water pollution, 2 <sup>nd</sup> ed. Scott, Foreman and Company, USA.	113	114	0	213
1976	Moore, J. W.; Moore, E. A. Environmental chemistry, Academic Press, New York, USA.	72	61	23	500
1977	Brockris, J. O'M. Environmental chemistry, Plenum Press, New York, USA.	90	192	17	795
1978	Bailey, R. A. Chemistry of the environment, Academic Press, New York, USA.	107	105	42	575
1979	Manahan, S. E. Environmental chemistry, 3 <sup>rd</sup> ed. Willard Grant Press, Boston, USA.	147	231	34	490
	Raiswell, R. W.; Brimblecomble, P.; Dent, D. L.; Liss, P. S. (Eds.), Environmental chemistry, the earth-air-water factory, Edward				
1980	Arnold, London, UK.	34	39	47	184
1984	Manahan, S. E. Environmental chemistry, 4 <sup>th</sup> ed. Willard Grant Press, Boston, USA.	189	254	51	612
1985	O'neil, P. Environmental chemistry, 1st ed. G.Allen & Unwin, Boston, USA.	16	28	L	232
1986	Hester, R. E. (Ed.), Understanding our environment, Royal Society of Chemistry, Bristol, UK.	72	59	56	333
	Bunce, N. J. Environmental chemistry, 1 <sup>st</sup> ed. Wuerz Publishing Ltd, Canada.	182	192	99	339
1991	Manahan, S. E. Environmental chemistry, 5 <sup>th</sup> ed. Willard Grant Press, Boston, USA.	139	198	36	583

Dubliched	Textbook	Π	Number of pages	of pages		
vear		Air	Water	Soil	Total	南
•	Harrison R M (Ed.) 2nd ed. Understanding our environment: An introduction to chemistry and pollution, Royal Society of					
1007	Chemistry Cambridge UK	68	96	30	326	147
7//1	Manahan S. F. Fundamentals of environmental chemistry, Lewis Publishers, Boca Raton, USA.	106	122	45	844	in i
	Bunce. N. J. Introduction to environmental chemistry, 2 <sup>nd</sup> ed. Wuerz Publishing Ltd, Canada.	51	66	0	559	
1993	O'Neil, P. Environmental chemistry, 2 <sup>nd</sup> ed. Chapman Hall, New York, USA.	94	86	73	268	
	Tolgeessy, J. (Ed.), Chemistry and biology of water, air, and soil environmental aspects, Elsevier, Czechoslovakia.	214	461	234	832	
	Bunce, N. J. Environmental chemistry, 2 <sup>nd</sup> ed. Wuerz Publishing Ltd, Canada.	209	225	80	376	T
1994	Manahan, S. E. Environmental chemistry, 6 <sup>th</sup> ed. Lewis Publishers, Boca Raton, USA.	192	235	69	811	— T
1995	Baird, C. Environmental chemistry, W.H.Freeman and company, New York, USA.	180	64	48	484	- r
	Andrews. J. E.: Brimblecomble; Jicklls, T. D.; Liss, P. S. (Eds.), An introduction to environmental chemistry, Blackwell Science,					
	TISA.	72	46	68	209	—— 1
1996	Spiro. T. G.: Stigliani, W. M. Chemistry of the environment, Prentice Hall, USA.	86	62	0	356	
1997	Connell. D. W. Basic concepts of environmental chemistry, Lewis Publishers, USA.	26	45	23	506	
	Raird, C. 1999. 2 <sup>nd</sup> ed. Environmental chemistry, W. H. Freeman, USA.	331	207	168	544	
	Harrision, R. M. 1999, 3 <sup>rd</sup> ed. Understanding our environment: An introduction to environmental chemistry and pollution, Royal	230	197	142	437	
1999	Chemical Society, Cambridge, UK	163	181	104	470	
	Manahan, S. E. 1999, 7 <sup>th</sup> ed., <i>Environmental chemistry</i> , Lewis Publishers, Boca Raton, USA.	215	145	89	875	<b></b> -

# SURVEY OF ENVIRONMENTAL CHEMISTRY EXPERIMENTS IN EDUCATIONAL LITERATURE (1969-2000)

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Year	Air	Water	2011
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	<b>52</b> , 12, 811-822.		
1976			
1977		Lambert, J. L.; Meloan, C. E. 1977, 'A simple qualitative analysis scheme for several environmentally important elements', <i>J</i> .	
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1978			
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1980			
1981			
1982		Ruzinki, W. E.; Beu, S. 1982, 'GC determination of environmentally significant pesticides', <i>J. Chem. Educ.</i> <b>59</b> , 7, 614-615.	

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<ul> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of</li> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of</li> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of</li> <li>Seasholtz, M. B.; Pence, J. Chem. Educ. 65, 9, 820-823.</li> <li>Betterton, E. A. 1991, 'Acid rain experiment and onstruction of a simple tubiditymeter', J. Chem. Educ. 68, 3, 254-256.</li> </ul>	Year	Air	Water	Soil
<ul> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of</li> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of</li> <li>CO in automobile exhaust by FTIR spectroscopy- An instrumental analysis laboratory experiment', <i>J. Chem. Educ.</i> <b>65</b>, 9, 820-823.</li> <li>Betterton, E. A. 1991, 'Acid rain experiment and construction of a simple tubiditymeter', <i>J. Chem. Educ.</i> <b>68</b>, 3, 254-256.</li> </ul>	1983		Cirsp, P. T.; Eckert, J. M.; Gibson, N. A. 1983, 'The determination of anionoic surfactants in natural and waste waters', J. Chem. Educ. <b>60</b> , 3, 236-238.	
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<ul> <li>Seasholtz, M. B.; Pence, L. E.; Moe, O. A. 1988, 'Determination of CO in automobile exhaust by FTIR spectroscopy- An instrumental analysis laboratory experiment', <i>J. Chem. Educ.</i> <b>65</b>, 9, 820-823.</li> <li>Betterton, E. A. 1991, 'Acid rain experiment and construction of a simple tubiditymeter', <i>J. Chem. Educ.</i> <b>68</b>, 3, 254-256.</li> </ul>	1987		Sanderson, P. L. 1987, 'Environmental chemistry in action', Educ. Chem. 24, 1, 16-18.	
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1994	83.	Educ. 71, 6, 521-523.	
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1995		Butala, S. J.; Zarrabi, K.; Emerson, D. W. 1995, 'Sampling and analysis lead in water and soil samples on a university campus- A student research project', J. Chem. Educ. 72, 5, 441-444.	
1996	Ramachdran, B. R.; Allen, J. M.; Helpern, A. M. 1996, 'Air- Water partitioning of environmentally Important organic compounds- An environmental	Quigley, M. N. Vernon, F. 1996, 'Determination of trace metal ion concentrations in seawater', J. Chem. Educ. 73, 7, 671-675.	Kegley, S. E.; Hansen, K. J.; Cunningham, K. L. 1996, <i>J. Chem. Educ.</i> 73, 6, 558-562.
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	Measuring Exnosure to Environmental Tobacco	Chem. Educ. 76, 9, 1256-1258.	Stehman, C. F. 1999, 'Chemical
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### INTEGRATED APPROACH AND THE USE OF INSTRUMENTATIONS IN ENVIRONMENTAL CHEMISTRY EDUCATION LABORATORY

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#### **THE DELPHI PANEL**

- 1. Associate Professor Viet Hung Pham, Center for Natural and Environmental Studies- Vietnam National University (CRES-VNU).
- 2. Associate Professor Hieu Tu Tran, Center for Natural and Environmental Studies-Vietnam National University (CRES-VNU).
- Associate Professor Thuong Van Pham, Faculty of Chemistry, Hanoi Teachers' Training University.
- 4. Associate Professor Chi Kim Dang, Environmental Technology Institute, Hanoi University of Technology.
- 5. Mr Anh Tuong Vu, Environmental Technology Institute, Hanoi University of Technology.
- 6. Dr Uyen Huu Tran, Department of Environmental Technology, Hanoi University of Construction.
- 7. Dr Ha Duc Tran, Department of Environmental Technology, Hanoi University of Construction.
- 8. Dr Thanh Tho Bui, Dean of Faculty of Chemistry, Natural Science University, Vietnam National University at HoChiMinh.
- 9. Professor Nhuan Duc Hoang, Scientific and Educational Institute, Vietnam Ministry of Education and Training.
- 10. Mr Thang Van Nguyen, Faculty of Hydrography and Environment, Hanoi University of Water Resource and Development.
- 11. Associate Professor Tuc Tat Do, Faculty of Hydrography and Environment, Hanoi University of Water Resource and Development.

# QUESTIONNAIRE FOR THE FIRST ROUND DELPHI SURVEY

1. By putting a percentage weighting (in terms of time spending) in each box please indicate the components of a current environmental chemistry (EC) program in your university,

Coursework (%)	
Practical (%)	
Field work (%)	

Comments

2. Within an EC program in your University, what do you consider to be the most appropriate time allocation for the following three areas?

Coursework (%)	
Practical (%)	
Field work (%)	

(	Commen	nts	 						
• • • • • • •	••••••	•••••	 		•••••	•••••		••••••	
•••••	•••••	• • • • • • • • • • • • • • • • • •	 ••••••	•••••		•••••••••••••••••	••••••	••••••	
•••••	•••••	•••••	 						

3. What do you consider to be the most appropriate length of time for an EC laboratory section?

hour (s)
Comments

- 4. What do you expect students to achieve in the practical component?
  - 4. What do you expect students to achieve in the practical component?

		Yes No
4	.1 Hands-on experience with standard environmental chemistry	
	(EC) techniques involving equipment and instrumentation.	
4	.2 Awareness of international environmental problems and issues.	
4	.3 Awareness of local environmental problems and issues	
4	.4 An understanding of the processes involved in:	
	Sample preparation	
	Measurement	
	Data processing	
	Interpreting data	
	Reporting	

Other .....

5. In the practical component, please indicate which of the environmental problems listed below should be included in the EC experimental program?

Yes	No
Air pollution	
Water pollution	
Soil pollution	
Other	
6. Do you think that an EC experiment	nt should also teach basic chemical principles?
Yes	No
If "yes", which of the following	ng areas of chemistry are most appropriately taught within
the context of an EC experiment	
Analytical chemistry	
Organic chemistry	
Inorganic chemistry	
Physical chemistry	
Comments	
	•

7. To what extent do you think that an EC experiment should reflect the work carried out by professional environmental scientists?

	A lot	Some	Not important
Sample preparation			
Measurement			
Data processing			
Data interpretation			
Reporting data			
Other	••••••	•••••••••••••••••••••••••••••••••••••••	

8. Please indicate the background of EC/ES students in your Institution which you *consider* to be the most suitable for undertaking EC/ES courses?

	Yes
General chemistry background	
Students with basis knowledge in organic chemistry	
Students with basis knowledge in inorganic chemistry	
Students with basis knowledge in physical chemistry	
Students with basic knowledge in analytical chemistry	

es	No

Other	 	••••••	••••••	
••••••	 ••••••			
••••••	 			
••••••	 	•••••		

9. Please indicate which instruments and apparatus are available at your institute and whether you consider the listed instrument to be essential hands-on items for an EC course?

	Available	Not available	Essential	Not essential
GC/MSD				
(Gas Chromatography/ Mass Selective Detector)				
GC/MS				
(Gas Chromatography/ Mass Spectrometry)				
GC/FID				
(Gas Chromatography/ Flame Ionisation Detector)				
GC/ECD				
(Gas Chromatography/ Electron Capture Dectector)				
TLC				
(Thin Layer Chromatography)				
Ion Chromatography				
ICP/MS				
(Inductively Coupled Plasma Emission- Mass Spectrometer	·)			
MS				
(Mass Spectrometer)				
AAS				
(Atomic Absorption Spectrometer)				
HPLC				
(High Performance Liquid Chromatography)				<b></b>
UV-VIS				
(Ultraviolet Visible Spectrophotometer)	<b>—</b> —–	<b>—</b> —–	<b>—</b> —–]	<b></b> 1
FTIR				
(Fourier Transform Infrared Spectrophotometer)	<b>[</b> ]	<b>[</b> ]		
Fluorometer				
Voltammeter				
Polarographic instrument				
Ion Selective Electrodes				
Computers and software				
Turbidimeter				

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Vortex mixer	Available	Not available	Essential	Not essential
Centrifuge				
Soxhlet apparatus				
Conductivity instrument				
pH meter				
Shaker				
Other	g EC experi	ments?		
11. What do you perceive will be the benefits to student incorporate the teaching of chemical principles?	s of conduc	cting EC expe	riments t	hat

inc	corporate the teaching of chemical principles?	
•	Provide students with a broader perspective on chemistry	
•	Help to link with different branches of chemistry	
•	Help the students to understand how chemistry is applied	

• Increase the appeal of chemistry as a discipline	
• Provide students with a more scientific understanding of the environment	
Other	

## **QUESTIONNAIRE FOR THE SECOND ROUND DELPHI SURVEY**

In the following questions, the answers are ranked from strongly agree, agree, disagree, and strongly disagree or 4, 3, 2, 1 appropriately. Could you please circle the number you consider being most suitable.

1. In Environmental chemistry (EC) course, the most appropriate allocation time is

Coursework: 60%-70%	4	3	2	1
Practical 20%-35%	4	3	2	1
Fieldwork 5%-10%	4	3	2	1

Comments.....

2. The most appropriate length of time for an EC laboratory section is 2-5 h.

4 3 2 1

# Comments

- 3. In the practical component students can achieve:
  - 3.1 Hands-on experience with standard EC technique involving equipment and instrumentation.

	4	3	2	T
3.2 Awareness of international environmental problems and issues.	4	3	2	1
3.3 Awareness of local environmental problems and issues.	4	3	2	1
3.4 Understanding of the process involved in				
Sample preparation	4	3	2	1
Measurement	4	3	2	1

### Appendices 192

Data processing	4 3 2 1
Interpreting data	4 3 2 1
Reporting data	4 3 2 1

Comments	
	•••••
	•••••
	••••••
	• • • • • • • • • • • • • • • • • •

4 In the practical component, the following environmental problems should be included:

Air pollution	4	3	2	1
Water pollution	4	3	2	1
Soil pollution	4	3	2	1
Hazardous wastes	4	3	2	1

Comments....

#### 5 EC experiment should combine teaching analytical chemistry 4 3 2 1

Comments.....

6 An EC experiment should reflect mainly the work carried out by professional environmental scientists which include

Sample preparation	4	3	2	1
Measurement	4	3	2	1

In some extend should include:

Data processing	4	3	2	1
-----------------	---	---	---	---

Data interpretation	4	3	2	1
Reporting	4	3	2	1

omments	
	••
	••

7 EC should be taught for student with the basic knowledge in

General chemistry	4	3	2	1
Inorganic chemistry	4	3	2	1
Organic chemistry	4	3	2	1
Analytical chemistry	4	3	2	1
Physical chemistry	4	3	2	1

omments	•••••
	•••••

8 To help students have effective EC laboratory, it is necessary to have the following items in the laboratory

GC/MSD	4	3	2	1
GC/MS	4	3	2	1
GC/FID	4	3	2	1
GC/ECD	4	3	2	1
IC	4	3	2	1
ICP/MS	4	3	2	1
MS	4	3	2	1
AAS	4	3	2	1
HPLC	4	3	2	1
UV-VIS	4	3	2	1
FT-IR	4	3	2	1
Fluorometer	4	3	2	1
Voltammeter	4	3	2	1

Polarographic instrument	4	3	2	1
Ion selective electrodes	4	3	2	1
Computers and software	4	3	2	1
Vortex mixer	4	3	2	1
Centrifuge	4	3	2	1
Soxhlet apparatus	4	3	2	1
Conductivity instrument	4	3	2	1
pH meter	4	3	2	1
Shaker	4	3	2	1

Comments

9 In conducting EC experiments, rate the following as difficulties:

Financial	4	3	2	1
Time constraints	4	3	2	1
Staffing	4	3	2	1
Facility availability	4	3	2	1

omments	

10	In conducting EC experiment that incorporate the teaching of chemical princip	les,	sti	ıde	nts
	will have the following benefits:				
	Understand how chemistry in applied to environmental problems	4	3	2	1
	Having a more scientific understanding of the environment	4	3	2	1
	Having a strong link with different branch of chemistry	4	3	2	1
	Increase the appeal of chemistry as a discipline	4	3	2	1
	Having a broader perspective on chemistry	4	3	2	1

Comments
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	• • • • • • • • • • • • • • • • • • • •		•••••	
***************************************	••••			
		• • • • • • • • • • • • • • • • • • • •		• • • • • • • • • • • • • • • • • • • •
***************************************				

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# APPENDIX 10 EXPERIMENT 1 - INSTRUCTOR NOTES

#### **1. Background Information**

Acquiring data for students to practice data analysis is often an issue of concern because of the limited time available in one laboratory session. In this experiment, advantage is taken of the rapidity at which data can be obtained using a gravimetric method to determine the moisture content in soils. Individual students will analyse two soil samples. One is homogenised and the other is not. From interpretation of the data, it is expected that students can draw their own conclusions about the effects of the sample preparation process on the analytical results, and therefore understand its critical role in the analytical procedure.

To achieve the above goals, students will be required to use some statistical methods, such as mean, standard deviation, relative percentage difference of duplicate (RPD), null hypothesis, Q-test, F-test, and student t-test. Two good references in this regard appear in the literature (1, 2).

#### 2. Lab Preparation

The experiment uses a large number of soil containers. Each student uses four, therefore, the experiment needs about 40 or more, depending on the number of students in the class. Aluminium dishes or pans are suitable containers.

#### 3. Tips for Success

- A class of ten or more is ideal for this experiment. They should work in two groups, therefore, students are able to have more data to practice analyse.
- (2) It is suggested that instructors should review with students steps (1)-(6) of Section 2.3.1 "Materials for Students". The success of the experiment is significantly dependent upon the different levels of homogeneity of the two soils. Therefore, it is important that students have H soil considerably homogenized.
- (3) Instructors should advise students to decant any supernatant water before dividing the sample into two jars, since if the moisture content is greater than 25%, it will take a longer time its determination and thus affect the time available for data analysis.

<sup>1.</sup> Harris, D. C. 1999, 5th ed. Quantitative chemical analysis, W.H. Freeman, USA. Chapter 4.

<sup>2.</sup> Miller, J. C.; Miller, J. N. 1988, 2<sup>nd</sup> ed. Statistics for analytical chemistry, John Wiley & Sons, UK.

- (4) Students can also use a computer-generated spreadsheet for quick data analysis, once the data are available. However, it is also possible for them to use a pocket calculator to calculate results.
- (5) The use of two different soils such as a clay-base soil and a sandy soil could be a good exercise to help students investigate the effect of the soil type on the analytical results in regard to the sample preparation procedure.

#### **EXPERIMENT 1 - MATERIALS FOR STUDENTS**

#### **1. Introduction**

#### **1.1 Sample Preparation**

Sample preparation, which in this case refers to as obtaining a representative sample from the bulk sample or gross sample, is the first important step in any chemical analysis. In environmental analysis, solid samples are often heterogeneous. Therefore, the sample preparation procedure often has a large effect on the analytical result. In this experiment, you will use the moisture in soil as an example of a target analyte to find out how the soil sample preparation process affects the analytical results.

#### **1.2 Moisture Content**

The determination of water content is often required in any solid sample analysis (e.g. soil) in order to report subsequently the analysis results on a "dry basis". The difference in mass of a soil sample before and after it is dried in an oven at 105°C to constant mass is used to calculate the moisture content. It can be calculated from the following equation:

% moisture = 
$$\frac{m_1 - m_2}{m_1 - m_0} \times 100\%$$
 (1)

Where  $m_0$  is the weight of the container,  $m_1$  is the weight of the sample and its container, and  $m_2$  is the weight of dried sample and its container.

In this experiment, the use of a gravimetric method will enable you to quickly obtain data for the purpose of statistical analysis.

#### 1.3 Data Analysis

Statistical methods will be used in this experiment to help analyse the data and to help you understand your results. It is suggested that you should revise some important terms in statistics such as mean, standard deviation, confidence intervals, null hypothesis, relative percent difference (RPD), Q-test, F-test and student t-test. These will be useful for you to complete your tasks (Section 3).

#### 2. Experimental Section

#### 2.1 Materials

You will need to collect about 300-400 g of your own soil sample. Divide it into two parts and transfer these into two pre-labelled glass jars, which is provided by your instructor to contain the whole class sample. One of these jars has been labelled "H" (meaning homogenized) and the other "NH" (meaning non-homogenized).

#### 2.2 Preparation

You will need to prepare 4 containers. Label two of these with an H and the other two with a NH. To avoid any confusion between your samples and your friends' samples, you need to label in the container. For example, the label will contain 3 items: your name (or initial name), soil status (H or NH), and the number to distinguish duplicates. For example, one could have labels: A-H<sub>1</sub> and A-H<sub>2</sub>, where, A is the student's initial name, H<sub>1</sub> and H<sub>2</sub> present the two portions taken from the jar H.

#### 2.3 Procedure

Significant contaminants may be released in the laboratory from drying a heavily contaminated sample in the open. The drying oven should be contained in a hood or be vented. The use of a fume hood is desirable for all sample preparation activities.

#### 2.3.1 Treatment for H soil and NH

In the following steps (1)-(6) you should participate in the class activities to help answer questions in **Section 3** well.

- (1) For the jar **NH** shake it several times, then, do not attempt to further homogenise the contents. Set the jar **NH** aside for later analysis.
- (2) For jar H shake the jar vigorously to mix the sample well.
- (3) Spread the sample H on a clean, steel tray. Then, use a steel spatula to further mix and homogenize the sample.
- (4) Spread the sample evenly into a circle. Use the spatula to cut it into quarters and remove the two opposite quarters.
- (5) Repeat step (4) several times (eight repetitions should be used). Step (4) and (5) are the socalled "cone and quarter" technique, which is often used in obtaining a representative soil

sample from a bulk sample.

(6) Keep the final two quarters for the use to determine the moisture content, follow instructions below.

#### 2.3.2 Moisture Content Determination

- Use a top-loading balance to weigh to the nearest 0.01 g the four pre-labelled containers. Record the mass of each (m<sub>0</sub>) on the data sheet provided.
- (2) Weigh approximately to the nearest 0.01 g 10 appropriate g of H soil (duplicate) and NH soil (duplicate) in containers. Record the mass (m<sub>1</sub>) in the data sheet.
- (3) Place your contains in step (2) together with others' sample containers in a large steel tray. Put the tray in the oven, maintained at a temperature of 105 ± 5°C for 1 h. Whilst the samples are heating, you should gather with whole class and instructor to further discuss about the statistic methods use in this study.
- (4) Remove the samples from the oven. Handle the hot sample tray with leather or thick cotton gloves. Allow the samples to cool in a desiccator for 30 min.
- (5) Remove samples from the desiccator and reweigh each sample to the nearest of 0.01 g. Record the mass (m<sub>2</sub>) in the data sheet.

#### 3. Suggested Questions and Calculations

- Using equation (1) (Section 1.2), calculate the percentage moisture content of each sample in both H soil and NH soil and the average value in each soil.
- (2) Calculate the RPD of the duplicates of two soils. Comment on the precision of the two results.
- (3) Exchange the average value calculated in Question (1) with your group to obtain a set of group data. Using the group data, calculate the mean and the standard deviation of the moisture content for H soil and NH soil.
- (4) Is there any suspect result in the group data? If so, use the Q-test to find out whether it should be rejected. Give possible reasons for the observed discrepancy of any rejected data.

- (5) If result(s) from Question (3) is/are rejected, recalculate the mean and the standard deviation of the new set of data. If not, use the data from Question (3) for the subsequent work.
- (6) Using the F-test, with significant level of 5%, comment on the precision of the two results obtained from H soil and NH soil in your group. Which one is more precise? Explain reasons for any difference in the precision of moisture values obtained from the two soils.
- Using data from Question (3) (or Question (4) if appropriate), calculate the 95% confidence intervals of the means of the two soil samples.
- (8) Exchange your group data with the other group (mean, standard deviation, and n, the number of valid results) to obtain the full class data of two soil samples.
- (9) Use the F-test and the t-test to find out whether the results of your group for each soil (mean and standard deviation) are significantly different. Explain your findings.
- (10) Estimate the true value of the moisture content in each of the two soils (at 95% confidence. Comment on the level of uncertainty in each of the two results and state your conclusion about possible factors that affect the analytical results.

## STUDENT DATA SHEET

Student name			H So	il			NH S	oil
	m <sub>0</sub>	<b>m</b> <sub>1</sub>	m <sub>2</sub>	% moisture	m <sub>0</sub>	mı	m <sub>2</sub>	% moisture
	RPD	)			RPD	) }		
	Ave	rage			Aver	age		
								_
	Mea	n =			Mea	n =		
	Stde	ev =			Stde	v =		
	% n	noisture	:=		% m	oisture	=	
	(959	% confi	dence)		(95%	∕₀ confi	dence)	
Group #	Mea	an =		<u> </u>	Mea	n =		
	n	=			n	=		
	Stde	ev =			Stde	ev =		
Class Result	% n	noisture	;=		% n	noisture	;=	
	(95	% confi	dence)		(95%	% confi	dence)	

#### **EXPERIMENT 2 - INSTRUCTOR NOTES**

#### **1. Background Information**

#### **1.1 Petroleum Hydrocarbons Analysis**

Methods for the analysis of petroleum hydrocarbons (PH) can be classified into two types: (i) non-target compound methods e.g. gravimetric and infrared spectroscopic method; and (ii) target compound methods, which use gas chromatography (GC) with different detectors such as the flame ionisation detector (FID), phosphorus ionisation detector (PID), and mass spectroscopy (MS). Generally, with the increase in the complication of the contaminated site and sources of the contaminants, there is a need to trace the source of the contaminants. GC/FID is used preferably in this regard. It enables one to determine effectively the individual components (e.g. benzene, toluene, etc.) or the range of PH in the sample (e.g. petrol, kerosenes, heavy kerosenes, lubricating oils, and asphalt). Quantification is based on the comparison of a standard chromatogram to the chromatographic responses of the samples (1). It is therefore beneficial for students to gain "hands-on" experience with the use of GC/FID to analyze the range of PH in petroleum-contaminated soils.

#### **1.2 Petroleum Hydrocarbons Extraction**

The use of Freon-113 as a solvent for the extraction of PH in soil contaminated PH has been gradually replaced by the use of other solvents which are less harmful to the environment (2). **Table 8.7** is a brief summary of the current extraction methods which have been approved by EPA for semivolatile organic compounds from solid samples in general and for PH in soil alike. It would be helpful to consider the available methods so that the most suitable method can be chosen to apply to the EC laboratory.

Currently, common methods for the extraction of semivolatile and nonvolatiles compounds in general and PH in particular from solid samples (e.g. soil), are: Soxhlet or Soxtec, sonication,

<sup>1.</sup> Fan, C. J.; Krishnamurthy, S.; Chen, C. T. 1994, 'A critical review of analytical approaches for petroleum contaminated soil', O'Shay, T. A.; Hoddinott, K. B. (Eds.), *Analysis of soils contaminated with petroleum constituents*, ASTM, 61-75.

Eckert-Tilotta, S. E.; Hawthorne, S. B.; Miller, D. J. 1993, 'Supercritical fluid extraction with carbon dioxide for the determination of total petroleum hydrocarbons in soil', *Fuel*, 72, 7, 1015-1023.

supercritical fluid extraction (SFE), accelerated solvent extraction (ASE) or pressurised fluid extraction (PFE) (3). Of these, sonication is a comparatively quick method and therefore is commonly used. The extraction techniques and their characteristics are summarised in Table 7.

<sup>3.</sup> EPA, Method 3500B, Organic extraction and sample preparation, Revision 2, December 1996.

Table 7 Extraction tee	Table 7 Extraction techniques as discussed in reference (4)	rence (4)			
	Soxhlet	Automated Soxhlet (Soxtec)	Ultrasonic Extraction	Accelerated solvent	Supercritical Fluid extraction
			(Sonication)	extraction (ASE)	(SFE)
				(Presssureized Fluid	
				Extraction)	
Method	EPA 3540	EPA 3541	EPA 3550	EPA 3545	EPA 3560/3561
Description of system	Uses cooled condensed	Sample is placed into the	Sample is covered with	Sample is extracted under	SFE uses supercritical CO <sub>2</sub> and/or
	solvents to pass over the	boiling solvent before	organic solvent, then its	high pressure (2000 psi)	organic modifier to extract
	sample contained in an	retrieving it and then	containers is put in a	and temperature (100°C).	analytes. The system is under high
	extraction thimble to	flushing clean solvent over	water bath, where a sonic	A small volume of fresh	pressure (up to 680 atm) and
	extract the anlytes. Uses	it. It is much quicker than	horn is placed inside.	solvent and a $N_2$ purge are	temperature up to 150°C. Liquid
	specialised glassware and	Soxhlet.		then used to flush solvent	solvent or a solid phase trap are
5	heating apparatus			and analytes from the	often used to collect extracts. The
				extraction vessel. It is	subsequent step is to desorb these.
				fully automated.	
Acceptability	Used as the benchmark	Considered to be the	Considered to be less	It is used later than the	Considered difficult to develop
	extraction technique by	automated version of Soxhlet	reliable than Soxhlet	availability of	reliable methods based on SFE
	which others are judged.	extraction.	extraction.	microwaves.	
Extraction time	4-24 h are commonly used	Reduced time compared to	Initiated for periods of 3-	It is a significantly rapid	Relatively short extraction times
		Soxhlet extraction i.e. 4-5 h	15 min. Typically, three	extraction method (15	(30-60 min).
			time periods might be	min)	
			used.		
Solvent usage	250-500 mL per extraction	40-50 mL per extraction	150-300 mL	Minimal solvent usage	Minimal solvent usage (10-30 mL)
			per extraction	(15-25 mL) per extraction	per extraction

4. Dean, J. R. 1998, Extraction methods for environmental analysis, John Wiley & Sons, USA. 212-215.

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	Soxniet	Automated Soxhlet (Soxtec)	Ultrasonic Extraction	Accelerated solvent	Supercritical Fluid extraction
			(Sonication)	extraction (ASE)	(SFE)
				(Pressureized Fluid	
				Extraction)	
Cost	Very inexpensive	Inexpensive	Relatively inexpensive	High cost	High cost
Ease of operation	Easy to use	Easy to use	Relatively easy to use.	Easy to use	Considered to be difficult to use.
Sample size	>10 g	>10 g	Approximately 5-10 g	>10 g possible	>1 g
Main disadvantages	Large solvent usage	Large solvent usage; more	Cannot be automated.	High capital cost; new	Matrix effects identified; high
		expensive than Soxhlet		technique	capital cost
		extraction.			

Mechanical shaking, which utilises a tumbler or shaker, has also been used and shown to give a comparable result to Soxhlet extraction (5, 6). Furthermore, soaking, a method that does not require the use of any apparatus to accelerate the extraction process, though considerably time-consuming, has also been used for the extraction of PH in soil (5).

Traditionally, PH are extracted by using trichlorotrifluoroethane (Freon-113) as the extraction solvent (7). However, an effort has been made to reduce or even to cease the use of this solvent due to environmental concerns (7). Alternative solvents are organic solvents such as methanol, methylene chloride (dichloromethane-DCM), a mix of acetone/hexane, toluene/methanol (8) and dichloromethane/acetone (9). Inorganic solvent such as liquid  $CO_2$  has also been used (9).

#### 1.3 Quality Assurance and Quality Control

An important aspect of any environmental analytical laboratory is quality assurance and quality control (QA/QC). This is used as evidence for the quality and acceptability of the laboratory data. Quality control is an important part of QA, which is used to evaluate the performance of the measurement system (i.e. both the method and the analytical instrumentation) and to estimate matrix effects on the analytical results. In this experiment, students gain experience using laboratory QC samples, which include the reagent blank, laboratory fortified blank (LFB), laboratory fortified matrix (LFM), and a sample duplicate. They will then be asked to evaluate their results based on such QC data.

#### 2. Laboratory Preparation and Equipment Needs

• Soil contaminated with PH, which is normally collected in an area near petrol service stations, car-parking areas, industrial refinery plants or even in an area near railways, should be prepared (in jars). Industrial analytical laboratories or environmental analytical laboratories may have suitable contaminated samples, which have been tested and which they would be willing to dispose of. These may be used as alternative sources. Spiking normal soil with petrol or diesel fuel of producing "real" samples for students.

<sup>5.</sup> AGAL Public Interest Project, No. 98-013, 'Optimization of extraction procedures for the analysis of total petroleum hydrocarbons-TPH'.

<sup>6.</sup> Schwab, A. P.; Su, J.; Wetzel, S.; Pekarek, S.; Banks, M. K. 1999, 'Extraction of petroleum hydrocarbons from soil by mechanical shaking', *Environmental Science and Technology*, 33, 1940-1945.

<sup>7.</sup> Eckert-Tilotta, S. E.; Hawthorne, S. B.; Miller, D. J. 1993, 'Supercritical fluid extraction with carbon dioxide for the determination of total petroleum hydrocarbons in soil', *Fuel*, **72**, 7, 1015-1023.

<sup>8.</sup> Fan, C. J.; Krishnamurthy, S.; Chen, C. T. 1994, 'A critical review of analytical approaches for petroleum contaminated soil', O'Shay, T. A.; Hoddinott, K. B. (Eds.), *Analysis of soils contaminated with petroleum constituents*, ASTM, pp. 61-75.

- Clean soil
- 2 mL GC vials and crimp caps to fit the vials (rubber/PTFE).
- Crimper to seal caps.
- Micro-pipette, 500 µL.
- A GC/FID with a BPX5 (25 m × 0.22 mm I.D. × 0.25 μm F.T.) column. In this experiment, the determination of TPH was conducted on a Hewlett-Packard 5890 Series II GC/FID, equipped with a 7673A auto sampler, and Hewlett-Packard computer control software.

# 2.1 Chemicals

- Anhydrous granular sodium sulfate. This can be calcined at 400°C for 4 h in a shallow tray or by precleaning with methylene chloride. If methylene chloride is used, a method blank must be analyzed, demonstrating that there is no interference from the treated sodium sulfate.
- Extraction solvent: DCM/acetone (1/1: v/v)
- C<sub>6</sub>-C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub>, C<sub>24</sub>, C<sub>28</sub>, C<sub>30</sub>, C<sub>32</sub>, and C<sub>36</sub> hydrocarbon standards (AR grade).

# 2.2 Standard Preparation

# Hydrocarbons Working Standard

- (1) Hydrocarbons for the standard include: individual hydrocarbons (IHCs) ranging from C<sub>6</sub> to C<sub>36</sub> carbon chain length: i.e. C<sub>6</sub>- C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub>, C<sub>24</sub>, C<sub>28</sub>, C<sub>30</sub>, C<sub>32</sub>, and C<sub>36</sub>.
   Note: C<sub>6</sub>- C<sub>10</sub> represents all IHCs between C<sub>6</sub> and C<sub>10</sub> iin chemistry.
- (2) Stock hydrocarbon standards of the following IHCs: C<sub>6</sub>-C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub>, C<sub>24</sub>, C<sub>28</sub>, C<sub>30</sub> are prepared by weighing 1.00 g of each IHCs and making up to 100 mL in DCM to give 10,000 ng/μL solutions.
- (3) A C<sub>32</sub>-C<sub>36</sub> compounds present solubility difficulties in DCM are prepared by weighing 1.0 g and making up to 100 mL in CHCl<sub>3</sub> to give 10,000 ng/µL solutions.
- (4) IHCs intermediate standards are prepared by diluting 10 mL of each of solutions in step
  (2) to 100 mL with DCM to give 1000 ng/μL.
- (5) The C<sub>32</sub>-C<sub>36</sub> intermediate standard are prepared by diluting 10 mL of the solution in step
   (3) to 100 mL with CHCl<sub>3</sub> to give 1000 ng/μL.
  - 9. Australian Government Analytical Laboratories (AGAL), Mehtod No VL237, issue No 4, 1998.

- (6) A mixed, intermediate standard Intermediate mixed IHCs standard is prepared by transferring 1 mL of each of the stock standards in step (4) to a 100 mL volumetric flask and making it up to the mark with DCM to give 100 ng/μL of each IHCs component.
- (7) A mixed C<sub>32</sub> and C<sub>36</sub> intermediate standard is prepared by transferring 1 mL of each C<sub>32</sub> and C<sub>36</sub> in solutions in step (5) to 100 mL volumetric flask and making it up to the mark with CHCl<sub>3</sub> to give 100 ng/μL of each component.
- (8) The working standard for mixed IHCs is prepared by transferring 20 mL of the solutions from step (6) and (7) into 100 mL volumetric flask and making it up to the mark with DCM to give 20 ng/µL of each component.

#### Hydrocarbon Spiked Standard

(9) The TPH spiked standard is prepared by mixing 5 mL of the IHCs intermediate standard solution of step (4) with 5 mL of C<sub>32</sub>-C<sub>36</sub> intermediate standard solution of step (5) into a 50 mL volumetric flask and making it up to the mark with DCM to give 100 ng/µL of each component.

#### 2.3 GC/FID Conditions

Column: BPX5, 25 m × 0.22 mm × 0.25  $\mu$ m film thickness; injector temperature: 320°C; detector temperature: 355°C; injector: splitless; detector: flame ionisation; purge: on 0.2 minutes; head pressure: 150 kPa; sample size: 1  $\mu$ L; sample washes: 5; sample pumps: 5; viscosity delay: 1 s; solvent: A washes 5 times with DCM; solvent B washes 5 times with DCM.

### **Oven Temperature Program:**

Initial temperature: 4°C; initial time: 0.8 min; rate (1): 27°C/min up to 100°C; rate (2): 35°C/min up to 350°C; hold time (2): 5 min; total run time: 15.17 min.

#### 3. Procedure

#### 3.1 First session

Depending on the organisation of the laboratory, an individual, or a group of two or three, should perform this experiment. The students are asked to prepare samples for analysis. Instructors should:

(1) Explain how to achieve a representative sample. The instructor should also show students how to use the sonicator.

- (2) Advise students on how to store samples for other groups to use. Soil samples are often kept at 4°C to reduce the level of evaporation of the target analytes.
- (3) When students have all of their vials containing extracts ready for GC/FID analysis, they will also need to be shown how to store these samples. They are normally kept at 0°C-4°C.

#### 3.2 Second Session

Student(s) will use GC/FID to determine the TPH concentration. Instructors should:

- (1) Set the GC/FID conditions (the suggested conditions are given above).
- (2) Advise students on how to set up the sequence of samples to analyse. Normally, a sequence based on an increasing concentration is used. For example:(i) one or two solvents are run to clean any contaminants from the column and to stabilise the baseline; (ii) reagent blank; (iii) standards; (iv) sample; (v) sample duplicate; (vi) LFB; (vii) LFM; (viii) a further number of solvent to leave the column free of contaminants.

It would be useful for students to perform the whole process of laboratory QC. This would require experimentation using the same sample and, when all experiments are finished, a class discussion and exchange of data could be carried out. Thus, students would have a large set of data for analysis and can evaluate their findings compared to the whole class in terms of its accuracy and precision. They may also construct a control chart and determine both the limit of detection and whether or not their results are affected by the reagent blank.

#### 4. Tips for Successes

- (1) If the level of TPH in the soil sample is high, dilution should be performed; otherwise the column will be adversely affected by the accumulation of the PH. It is advised to run a solvent blank, normally DCM after high levels of contaminants are observed. At the end of the analysis, a column cleaning procedure should also be carried out to avoid the accumulation of PH in the column. This is particularly required for a soil contaminated with very high petroleum hydrocarbons.
- (2) In this experiment, a sonicator is proposed for use in the extraction procedure because of its time effectiveness and therefore its suitability for use in the teaching laboratory. However, other methods, which have been listed (Table 7) may also be suitable. Alternatives to sonication are the Soxhlet apparatus, tumbler, and twist shaker. Experiments can also be done without using any of these extraction apparatus. For example: soaking overnight can be used as well. Although, the use of the Soxhlet

apparatus is beneficial for students' learning experience, it is time-consuming. Thus, it is suggested that this should not be used in this experiment, particularly since QA/QC has been integrated, and many extractions are needed in a limited time.

- (3) If there is a difficulty in obtaining all of the standard hydrocarbons, a standard that is less representative of PH can be substituted. For example, the standard may contain :
  - Low HC (C<sub>6</sub>-C<sub>9</sub>): C<sub>6</sub>-C9 hydrocarbon
  - Medium HC ( $C_{10}$ - $C_{14}$ ):  $C_{10}$ ,  $C_{12}$  and  $C_{14}$  hydrocarbon
  - High HC ( $C_{15}$ - $C_{28}$ ):  $C_{15}$ ,  $C_{18}$ ,  $C_{24}$  and  $C_{28}$  hydrocarbon
  - Very high HC (C<sub>29</sub>-C<sub>36</sub>): C<sub>29</sub> and C<sub>36</sub> hydrocarbon

The determination of TPH may be limited to the range of  $C_6$ - $C_{14}$ ;  $C_{10}$ - $C_{28}$ ;  $C_6$ - $C_{28}$ . In these cases, the required components of the standard are reduced significantly. If there are time constraints, checking for very high HC can be omitted, so that the running time should take 12 min. If analysing a hydrocarbon range of  $C_6$ - $C_{14}$ , the running time should take 9 minutes.

- (4) The use of different solvents and extraction methods and the evaluation of their effectiveness by using percentage recoveries is also a good exercise for students to perform. Comparing the use of different solvents and different techniques can help students to understand the factors that may affect analytical results.
- (5) Students are required to use sample duplicates to evaluate the precision of the method. However, if the sample has little PH, students should be asked to use LFM duplicates for that purpose.

Chemical name	Molecular formula	CAS No
n-C <sub>6</sub> hydorcarbon (Hexane)	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub>	110-54-3
n-C <sub>7</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub>	142-82-5
n-C <sub>8</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>6</sub> CH <sub>3</sub>	111-65-9
n-C <sub>9</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>7</sub> CH <sub>3</sub>	111-84-2
n-C <sub>10</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>8</sub> CH <sub>3</sub>	124-18-5
n-C <sub>12</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>10</sub> CH <sub>3</sub>	112-40-3
n-C <sub>14</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>12</sub> CH <sub>3</sub>	629-59-4
n-C <sub>16</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>14</sub> CH <sub>3</sub>	554-76-3
n-C <sub>18</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>16</sub> CH <sub>3</sub>	630-02-4
n-C <sub>24</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>22</sub> CH <sub>3</sub>	646-31-1
n-C <sub>28</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>26</sub> CH <sub>3</sub>	630-02-4
n-C <sub>30</sub>	CH3(CH2) <sub>28</sub> CH <sub>3</sub>	111-01-3
n-C <sub>32</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>30</sub> CH <sub>3</sub>	544-85-4
n-C <sub>36</sub>	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>34</sub> CH <sub>3</sub>	630-06-8

# 6. CAS Numbers of Chemicals

### **APPENDIX 13**

# **EXPERIMENT 2 - MATERIALS FOR STUDENTS**

#### **1. Background Information**

Soil contamination has become a major environmental concern worldwide. There is a wide range of contaminants in soils, of which the most common is petroleum hydrocarbons (PH). Incidents during transportation, and leaking during storage are the main causes for such problems. Soil contaminated with PH is recognized to have bad effects on human health. Risk assessment of contaminated sites with PH is, therefore, becoming an important task. The accurate assessment of the level of PH plays a critical role in this regard. The analysis of total petroleum hydrocarbons (TPH) in soil has been conducted largely in industrial and governmental environmental analysis laboratories for this purpose. In this experiment, you will perform an analytical procedure to determine the TPH level in a sample of soil.

Petroleum hydrocarbon products are complex mixtures of hundreds of individual components, including straight-chained, branched and cyclic alkanes, alkenes, monocyclic and polycyclic aromatic hydrocarbons. The physical properties of these components range from highly volatile gases (e.g. methane, CH<sub>4</sub>) to high boiling point solids (e.g. hexatriacontane,  $C_{36}H_{74}$ ). Petroleum hydrocarbon products consist of groups (or fractions) of components that have been separated based on their boiling point ranges. These groups can be divided into volatile and semivolatile fractions. The volatile fraction incorporates the C<sub>1</sub>-C<sub>5</sub> and C<sub>6</sub>-C<sub>9</sub> (petrol) hydrocarbons; the semivolatile fractions incorporates the C<sub>10</sub>-C<sub>14</sub> (kerosenes), C<sub>15</sub>-C<sub>28</sub> (heavy kerosenes and lubricating oils), and C<sub>29</sub>-C<sub>36</sub> (asphalt) hydrocarbons. Because of the increase in the number of contaminated sites and the complexity of these sites, it is necessary to identify the range of the contaminants or even specific components of PH from contaminated soil samples. Therefore, TPH is often determined based on the analysis of different ranges of PH. In this experiment, the TPH is defined as the fraction (C<sub>6</sub>-C<sub>9</sub>); (ii) medium HC fraction (C<sub>10</sub>-C<sub>14</sub>); (iii) high HC fraction (C<sub>15</sub>-C<sub>28</sub>); and (iv) very high HC fraction (C<sub>29</sub>-C<sub>36</sub>).

#### **1.2 Extraction Techniques**

Currently, common methods for the extraction of semivolatile and nonvolatiles compounds in general and PH in particular from solid samples (e.g. soil), are sonication, Soxhlet extraction, supercritical fluid extraction (SFE), accelerated solvent extraction (ASE) or pressurised fluid

extraction (1). Sonication has been used widely in the extraction of PH from solid samples in general and soil samples in particular.

Traditionally, trichlorotrifluoroethane (Freon-113) has been used as the extraction solvent for PH from solid samples (2). However, efforts have been made in order to reduce or even to cease the use of this solvent due to environmental regulations on using this compound (2). Alternative organic solvents that have been used are methanol, methylene chloride (dichloromethane, DCM), mixtures of acetone/hexane, toluene/methanol (3), or dichloromethane/acetone (4); and inorganic "solvents" such as liquid  $CO_2$  (3).

### Suggested additional reading

Dean, J. R. 1998, Extraction methods for environmental analysis, John Wiley & Sons, New York, USA.

### 1.3 GC/FID

Gas chromatography (GC) has been broadly used as an effective instrument for analyzing PH (3). The most frequent detectors used in conjuration with this technique are the photoionization detector (PID), flame ionization detector (FID) and mass spectroscopy (MS). The PID and MS respond quite well to aromatic compounds but are not as good as the FID in terms of their response to hydrocarbons in general (3). In this experiment, the GC/FID is used to quantify the following groups of PH: (i) low HC fraction:  $C_6$ - $C_9$ ; (ii) medium HC fraction:  $C_{10}$ - $C_{14}$ ; (iii) high HC fraction:  $C_{15}$ - $C_{28}$ ; and (iv) very high HC fraction:  $C_{29}$ - $C_{36}$ .

<sup>1.</sup> EPA, Method 3500B, Organic extraction and sample preparation, Revision 2, December 1996.

<sup>2.</sup> Eckert-Tilotta, S. E.; Hawthorne, S. B.; Miller, D. J. 1993, 'Supercritical fluid extraction with carbon dioxide for the determination of total petroleum hydrocarbons in soil', *Fuel*, 72, 7, 1015-1023.

<sup>3.</sup> Fan, C. J.; Krishnamurthy, S.; Chen, C. T. 1994, 'A critical review of analytical approaches for petroleum contaminated soil', O'Shay, T. A.; Hoddinott, K. B. (Eds.), *Analysis of soils contaminated with petroleum constituents*, ASTM, 61-75.

<sup>4.</sup> Australian Government Analytical Laboratories (AGAL), Mehtod No VL237, issue No 4, 1998.

## Suggested additional reading

- (i) Skoog, D. A.; Holler, F. J; Nieman , T. A. 1998, 5<sup>th</sup> ed. *Principles of instrumental analysis*, Sounder College Publishing, Philadelphia, USA, pp. 701-716.
- McNair, H. M.; Miller J. M. 1998, Basic gas chromatography, John Wiley & Sons, New York, USA, pp. 105-119.
- (iii) Hill, H. H. 1992, Detectors for capillary chromatography, John Wiley & Sons, New York, USA, pp. 7-21.

# Use of GC/FID for the PH Quantitative Analysis

The quantification of unknown samples, is normally based on known quantities of analytes that are present in the so-called standards. Methods of using standards may be divided into: (i) addition standards in which known quantities or target analytes are added to the unknown components; (ii) internal standards where a known quantity of compounds which is different to the components in the analytes is added to the sample; and (iii) external standards where a known amount of target analyte is used together with the sample.

In this experiment, an external standard is used. By comparing the chromatographic response area of the sample with the response area of the standards, the unknown amounts of analytes are determined.

The measurement of the TPH ( $C_6$ - $C_{36}$ ) in this experiment is explained as follows:

- TPH is the sum measured from the start of the benzene peak to the end of the C<sub>36</sub> peak in the GC working standard as measured in the GC/FID trace.
- The C<sub>6</sub>-C<sub>9</sub> (low HC fraction) is measured on the GC/FID trace from the start of the benzene peak to the midpoint between the C<sub>9</sub> and C<sub>10</sub> peaks in the GC working standard.
- C<sub>10</sub>-C<sub>14</sub> (medium HC fraction) is measured on the GC/FID trace from the midpoint between the C<sub>9</sub> and C<sub>10</sub> peaks to ¼ distance between C<sub>14</sub> and C<sub>16</sub> peaks in the GC/FID working standard.
- C<sub>15</sub>-C<sub>28</sub> (high HC fraction) is measured on the GC/FID trace at ¼ distance between the C<sub>14</sub> and C<sub>16</sub> peaks and ¼ distance between C<sub>28</sub> and C<sub>30</sub> peaks in the GC working standard.
- C<sub>29</sub>-C<sub>36</sub> (very high HC fraction) is measured on the GC/FID trace at ¼ distance between the C<sub>28</sub> and C<sub>30</sub> peaks and the end of the C<sub>36</sub> in the GC working standard.
- TPH is the total of low, medium, high and very high HC fractions.

For the calculation of TPH, the formula below is used:

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$$R = \frac{a}{s} \times \sum c \times \frac{V_{I}}{W}$$
(1)

where R is the TPH residue (mg kg<sup>-1</sup>); a is a peak area of sample (total of IHCs), s is the peak area of standard (total of IHCs),  $\Sigma c$  is the sum of concentration of individual component used in standard (ng/µL), V<sub>1</sub> is the final volume of sample extract (mL), and W is the weight of sample taken for analysis (g).

# 1.4 Quality Assurance and Quality Control in the EC Lab

Producing defendable data is the ultimate role of environmental analysis. Quality assurance and quality control (QA/QC) are the associated processes that ensure the laboratory data are reliable and are under control (5). It is often a regulatory requirement that environmental analytical laboratories document and include in their reports the QA/QC procedures adopted as the evidence of the acceptable quality of the reported data (5).

Quality assurance is the definitive program for laboratory operation that specifies the measures required to produce defensible data of known precision and accuracy. An important part of QA is the control of the measurement system, which includes the analytical instruments and the laboratory methods. The performance of the measurement system, which is based on the measurement system control and the extent to which the sample matrix affects the laboratory data, is evaluated by quality control (QC) samples. Quality control samples are comprised of: (i) a set of "field" of samples, which are additional samples to the target sample collected from the field during the sampling procedure, and (ii) laboratory QC samples.

In this experiment, you will use the following "laboratory" QC samples, which are typical of some QC samples often used in the scientific laboratory (5, 6), to help you evaluate your result:

(a) *Reagent blank*: to help determine the contribution of the reagents and the preparative analytical steps to the error in the measurement.

<sup>5.</sup> Clescerl, L. S.; Greenberg, A. E.; Eaton, A. D. (Eds.), 1998, 20<sup>th</sup> ed. Standard methods for the examination of water and wastewater, pp. (1-4)-(1-11).

<sup>6.</sup> Liabastre, A. A.; Carlberg, K. A.; Miller M. S. 1995, 'Quality assurance for environmental assessment activities', *Methods of environmental data analysis*, Elsevier Science Publishers, USA, pp. 273-280.

- (b) Laboratory-fortified blank (LFB) or "blank matrix": is the blank sample. In the case of soil samples, clean soil containing a known amount of the analyte will be used for this purpose. It is used to evaluate laboratory performance and analyte recoveries from the blank matrix. The results obtained for the LFB are used to evaluate batch performance, calculate recovery limits and plot control charts.
- (c) Laboratory-fortified matrix (LFM) or use sample matrix: involves the addition of a known amount of analyte to the sample before sample preparation. The LFM is used to evaluate analyte recoveries from the sample matrix. It is normally used at the same concentration as the LFB to enable the analyst to separate the effect of the matrix from laboratory performance. Recoveries of the LFM are also used to evaluate the accuracy of the method.
- (d) Duplicate sample or LFM duplicates: These are used to evaluate the precision of the analytical procedure. Duplicate samples are used when there is a high likelihood that the sample contains the analyte of interest. LFM duplicates are used when there is little likelihood the sample contains the compounds of interest.

In the QC report the following results may need to be included: For the LFM, the result is reported as the percentage recovery:

% Recovery = 
$$\frac{\text{LFM sample result - sample result}}{\text{known LFM added concentration}} \times 100\%$$
 (2)

For the LFB, the result is reported in the percentage of recovery:

$$\% \text{Recovery} = \frac{\text{LFB sample result - sample result}}{\text{known LFB added concentration}} \times 100\%$$
(3)

In the cases of duplicate samples or duplicate LFM, the result is reported as relative percentage difference of duplicates (RPD):

$$RPD s_{ample} = \frac{sample \ result \ - \ duplicate \ result}{(sample \ result \ + \ duplicate \ result)/2} \times 100\%$$
(4)

$$RPD_{LFM} = \frac{\text{sample result - duplicate result}}{(\text{sample result + duplicate result})/2} \times 100\%$$
(5)

# 2. Experimental Section

CAUTION: Many of the substances used in this experiment, including the samples being analyzed can be potentially hazardous (flammable, toxic, corrosive or carcinogenic). The use of eye protection and protective clothing such as gloves and coats is highly recommended. The use of a fume hood is also most desirable.

# Materials

You will be provided with a soil sample, some clean soil, solvents and sodium sulfate (granular, anhydrous). The GC working standards and GC spike standard, which contain the range of IHCs:  $C_6-C_{10}$ ,  $C_{12}$ ,  $C_{14}$ ,  $C_{16}$ ,  $C_{18}$ ,  $C_{24}$ ,  $C_{28}$ ,  $C_{30}$ ,  $C_{32}$ , and  $C_{36}$  with the concentration of 20 and 100 ng/µL appropriately, are also provided.

# 2.1 Session 1: Extraction

# 2.1.1 Preparation

The following steps will require the use of several jars and vials. It is necessary to label the glassware clearly before doing any further work. Items of glassware need to be labelled include: 125 mL jars and vials for "sample", "duplicate sample", "matrix sample", "matrix blank", and "reagent blank" (solvent).

# 2.1.2 Extraction

All the operations in this experiment should be performed as quickly as possible to avoid any loss of analytes during the sample preparation.

- (1) Use a top-loading balance to weigh two approximately 10 g soil samples to the nearest 0.01 g in two (tarred) 125 mL glass jars, which have been labelled "sample" and "matrix sample".
- (2) Weigh out approximately 10 g clean soils to the nearest 0.01 g in the (tarred) 125 mL glass jars, which have been labelled "matrix blank".
- (3) Take one of the jars prepared in step (1) that is labelled "matrix sample" and use a micropipette to spike 500 μL GC spike standard into the surface of the sample. Quickly cover the jar with its lid.

- (4) Take one of the jars from step (2) that is labelled "matrix blank", and spike the surface of the sample with 500 μL of the spike standard (try to avoid contact between the spike solution and the edge of the jar). Quickly cover the jar with its lid.
- (5) Add 20 mL of the solvent mixture (1/1) DCM/acetone all the jars with samples in steps (1)-(4). Use clean glass rods to mix the contents of all jars well so that samples are completely submerged in the solvent. Tighten all the lids after mixing. Place all jars in the sonicator for 10 min.
- (6) Remove all the jars from the sonicator and add approximately 10 g of anhydrous sodium sulfate to each jar. Mix well and replace them in the sonicator for a further 10 min.
- (7) Remove all the jars from the bath, allow the soil to settle and examine the DCM/acetone fraction. If the solvent is cloudy, add and mix 10 g lots of anhydrous sodium sulfate until it turns clear. Otherwise, continue with the next step.
- (8) Allow the soil to separate and settle from the solvent fraction. If the extracts contain particulate or floating, insoluble material, it is necessary to centrifuge the sample to help the solvent to separate. In such cases, centrifuge a portion of the extract at 13,000 RPM for 5 min.
- (9) Carefully transfer approximately 1.5 mL of the clear extracts into pre-labelled 2 mL GC vials and seal the tops immediately using a crimp cap and crimper to avoid evaporation.
- (10) Transfer approximately 1.5 mL of the solvent into the vial labelled "reagent blank". Crimp the cap.
- (11) The instructor will show you where to store all the vials in a refrigerator ready for use in the next session.

# 2.2 Session 2: Petroleum Hydrocarbons Analysis

- (1) Recover all the sample vials prepared in **Session 1**. Slightly shake vials then allow them to equilibrate to room temperature. When all the necessary analytes are ready which include reagent blanks (solvent blank), standards, matrix blank, sample and its duplicate, and the sample matrix, your instructor will show you how to operate the GC/FID.
- (2) Consult with your instructor on how to place all of the samples in proper order and how to use the GC/FID for this task.
- (3) Consult with your instructors to create a suitable table for recording data.
- (4) Record appropriate data in your data table.

### 2.3 Disposal

When you finish the experiment, the following steps should be taken:

- (1) All waste solvent should be collected in the appropriate laboratory solvent waste bottles.
- (2) Soil waste should be placed in the fume hood to allow any remaining solvent to evaporate before disposal in a vessel provided for this purpose.

### 3. Suggestion Questions and Calculations

- Using equation (1), calculate the TPH content (on group HC, such as low, medium, high and very high basis, and then take the sum) in the soil sample and its duplicate based on equation (1).
- 2. Using equation (4), calculate the RPD of the sample duplicate. Comment on the reproducibility of your result. Give reasons for any differences and if appropriate, propose a means to increase the reproducibility.

- 3. Use equations (2) and (3) determine the percentage recoveries of LFB and LFM. Comment on the matrix effect.
- 4. Based on the percentage recoveries of LFM, comment on TPH value obtained in question 1. Is the latter the actual concentration of TPH in the soil sample? How close are your results to the actual value? What factors do you consider may have affected the closeness of the measured value and the actual value?
- 5. In environmental analytical laboratories, besides the report of the sample results they are also required to report the RPD of sample duplicates and the percentage recoveries of the LFM. To what extent do you agree?
- 6. Based on your average TPH in each range of HC, give your opinion on the possible source of contaminants.

The following questions can be answered when you have a complete set of class data for this experiment:

- 7. Calculate the mean value of the TPH content, and its standard deviation.
- 8. Are there any suspect values? If so, use the Q-test to find out whether these values should be rejected.
- 9. Calculate the 95% confident interval of the mean of the TPH content for each range of HC.
- 10. What is the lowest level of analyte you can reliably detect, with this method?
- 11. What is the highest level of analyte you can reliably detect, in this method?
- 12. Based on your value of the TPH in the reagent blank and the answer to question (10), give your evaluation of the influence of reagents (solvent and soil) on your result for the target analyte.