National Observatory for Environment and Sustainable Development (ONEDD)
Ministry of Land Planning and Environment (MATE)
The People's Democratic Republic of Algeria

The Project for Capacity Development of Environmental Monitoring (Phase 2)

Project Completion Report

September 2012

JAPAN INTERNATIONAL COOPERATION AGENCY (JICA)

OYO INTERNATIONAL CORPORATION
OAFIC CO., LTD.

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Annexes

Abbreviation (Abréviations)

AAS: Atomic Absorption Spectrometer (Spectrométrie d'Absorption Atomique)
ADE: Algerian Company of Water (Compagnie Algérienne des Eaux)
ANRH: National Agency of the Hydraulic Resources (Agence Nationale des Ressources Hydrauliques)
BOD: Biochemical Oxygen Demand (Demande Biochimique d'Oxygène / DBO)
COD: Chemical Oxygen Demand (Demande Chimique d'Oxygène / DCO)
C/P: Counterpart (Interface/Homologue Algérien)
CRL: Central Regional Laboratory (Laboratoire Régional Central / LRC)
DEWA: Direction of the Environment of Province of Algier (Direction de l'Environnement de la Wilaya d'Alger)
DEWB: Direction of the Environment of Province of Blida (Direction de l'Environnement de la Wilaya de Blida)
EQS : Environmental Quality Standard (Norme de Qualité Environnementale)
FEDEP: Nationals Funds for the Environment and Depollution (Fonds National pour l'Environnement et la Dépollution)
FOREMHYD : State Enterprise of Realization of Hydraulic Drilling and Electromechanical Works (Entreprise Publique de Réalisation de Forages Hydrauliques et de Travaux Électro-Mécaniques)
FTIR: Fourier Transform Infrared Spectrophotometer (Spectrophotomètre Infrarouge à Transformée de Fourier)
GC: Gas Chromatograph (Chromatographe à Gaz)
GCMS: Gas Chromatograph Mass Spectrometer (Chromatographe en Phase Gazeuse-Spectrométrie de Masse)
GLP: Good Laboratory Practice (Bonnes Pratiques de Laboratoire)
GTZ: German Technical Cooperation (Coopération Technique Allemande)
HURBAL: Establishment of the Urban Hygiene of the Town of Algiers (Établissement d'Hygiène Urbaine de la Ville d'Alger)
ISMAL: Institute for Marine Science and Coastal Management (Institut National des Sciences de la Mer et de l'Aménagement du Littoral)
ISO: International Organization for Standardization (Organisation Internationale de Normalisation)
JCC: Joint Coordinating Committee (Comité de Coordination Conjoint / CCC)
JET: JICA Expert Team (Equipe d'experts de la JICA)
JFY: Japanese Fiscal Year: from April to March (Année Budgétaire Japonaise: d'Avril à Mars)
JICA: Japan International Cooperation Agency (Agence Japonaise de Coopération Internationale)
MATE: Ministry of Land Planning and Environment (Ministère de l'Aménagement du Territoire et de l'Environnement)
M/M: Minutes of Meeting (Procès-Verbal)

NA: Algerian Standard (Normes de l'Algérie)
NAPE-SD: National Action Plan for Environment and Sustainable Development (Plan d'Action National pour l'Environnement et le Développement Durable)
OEH: El Harrach River (Oued El Harrach)
ONA: National Office of Environment (Office National de l'Assainissement)
ONEDD: National Observatory for Environment and Sustainable Development (Observatoire National de l'Environnement et du Développement Durable)
PAH: Policyclic Aromatic Hydrocarbon (Hydrocarbure Aromatique Polycyclique)
PCM: Project Cycle Management (Gestion Cyclique du Projet)
PDM: Project Design Matrix (Matrice de Conception du Projet)
PNAE-DD: Action Plan for the Environment and Sustainable Development (Plan National d'Action pour l'Environnement et le Développement Durable)
PNAGDES: National Plan for the Management of Special Wastes (Plan National de Gestion Intégrée des Déchets Spéciaux)
PO: Plan of Operation (Plan de l'Opération)
QA/QC: Quality Assurance/ Quality Control (Assurance Qualité/ Contrôle de Qualité)
R/D: Record of Discussions (Compte rendu des Discussions)
RNE 2000: National Report on the Status of Environment in 2000 (Rapport National sur l'état et l'avenir de l'Environnement en 2000)
SNE: National Environmental Strategy (Stratégie Nationale de l'Environnement)
SNIE: National Environmental Information System (Système National d'Information Environnementale)
SOP: Standard Operating Procedure (Procédure Opératoire Normalisée)
TOC: Total Organic Carbon (Carbone Organique Total)
USTHB: Houari Boumediene University of Science and Technology (Université des Sciences et de la Technologie Houari Boumediene)
VOC: Volatile Organic Compound (Composé Organique Volatil)
XRF: X-ray Fluorescence Analyzer (Spectromètre de Fluorescence X)

1 Outline of the Project

1.1 Background of the Project

The environmental management system in the People's Democratic Republic of Algeria (hereinafter referred to as Algeria), such as development of laws and regulations, policy making, and coordination among related organizations has been developed by the Ministry of Land Planning and Environment (hereinafter referred to as "MATE"). The department of prefectures, a subsidiary organization of MATE has conducted inspection, given instructions and taken measures for preservation of the environment aiming at the pollution sources such as factories and industries. In order to implement environmental monitoring, the National Observatory of Environment and Sustainable Development (hereinafter referred to as "ONEDD") have also been established under MATE. However, because of insufficient management system and limited capacity for investigating the state of pollution, Algeria requested Japan for technical cooperation. Responding to the request, Japanese expert was dispatched. It was revealed that there are heavy metals including mercury in El Harrach River flowing through the major industrial area of Alger city.

In order to cope with such an emergency, JICA dispatched three (3) Japanese experts to investigate the pollution of mercury in El Harrach River basin and the Algeria Bay. They have conducted training on basic analysis for mercury and related parameters for ONEDD Central Regional Laboratory (hereinafter referred to as "CRL") staff.

Organizing open seminars and publishing news in environmental journals, news coverage in national news papers and television, the pollution of mercury in El Harrach basin has attracted the national attention, and developed public awareness regarding issues of pollution and environment. With activities of this time as a turning point, lead MATET and ONEDD as recognized authorities for the enforcement of environmental monitoring, and responsible for formulation of rational measures against environmental pollution.

For the purpose of comprehensive and basic capacity development of environmental monitoring for ONEDD as a counterpart (C/P), JICA has already carried out a Japanese technical cooperation program titled "The Project for Capacity Development of Environmental Monitoring" (hereinafter referred to as "Phase 1") during the past three years starting from December 2005. As a result of the Phase 1, ONEDD/CRL has learnt a great deal of basic technology in the field of environmental monitoring such as sampling technology, analyses of organic and inorganic chemistry, microbiology (coliforms) and so on. This result also has induced the public to recognize the position of ONEDD/CRL as a national institute for environmental monitoring, and has increased orders for various analyses from outside. In addition, the law of Algerian government introduced in 2007 indicated that the analytical data of industrial effluents from ONEDD should be the most reliable basis

for imposition of surcharge, and has lead ONEDD/CRL to play an important role as an unique governmental institute for environmental monitoring.

However, since ONEDD/CRL is still at the basic level of technology, issues such as capacity development in laboratory management for producing reliable data constantly, introduction of a system of quality control using advance analytical technology including GCMS, FTIR and XRF are need to be introduced. The other remaining issues need to be addressed include comprehensive interpretation, risk assessment of monitoring results and formulation of measures for minimizing environmental pollution. Since basic capacity development has been achieved already through Phase 1, ONEDD/CRL as an advanced institute is expected to disseminate their gained knowledge and technology to ONEDD regional laboratories (Oran and Constantine) and monitoring stations, and to support them in the field of monitoring technology.

In order to deal with the above issues, based on request of cooperation from MATET and ONEDD in February 2008, JICA conducted a preparatory study for the formulation of the Japanese technical cooperation program regarding “The Project for Capacity Development of Environmental Monitoring (Phase 2)" (hereinafter referred to as "the Project" or "phase 2") in March 2009. As a result of the discussions and in accordance with the framework defined in R/D and M/M, the agreement on technical cooperation between the Government of Japan and the Government of Algeria was signed on April 28, 2009.

1.2 Objectives and Scope of the Project

(1) Objectives of the Project

Based on Project Design Matrix agreed between the Algerian and the Japanese sides in March 2009, this project was implemented with the aim to enhance ONEDD's capacity to generate environmental information for effective environmental management including inspection and pollution prevention. The objective and expected output of the Project are shown in Table 1-1.

Table 1.1 Purposes and Expected Output of the Project

Overall Goals	ONEDD establishes environmental monitoring system based on the National Environmental Strategy under the well-organized network of laboratories and stations where CRL plays a leading role.
Project Purpose	ONEDD's Capacity to generate environmental information for effective environmental management including inspection, enforcement and pollution prevention is strengthened.
Output	**Output 1:** CRL acquires advanced analytic technique for GCMS, FTIR and XRF. **Output 2:** Quality of environmental monitoring capacity of CRL is upgraded through the environmental monitoring activities including effluent monitoring in the Model Site. **Output 3:** CRL enhanced quality control capacity of lab analytical works. **Output 4:** Environmental monitoring technologies possessed by CRL are disseminated to other ONEDD regional laboratories, monitoring stations and other relevant organizations.

(2) Project Area

The Project area is as follows.

- Alger, Blida, Oran and Constantine Provinces
- Model Site in the Project: Oued El Harrach (hereinafter referred to as “OEH”) basin in Alger and Blida Provinces and coastal area in Alger Province.

(3) Implementation Structure

ONEDD is nominated as counterpart organization, and MATE, governing agency of ONEDD, is deployed as supporting organization. Persons in charge of management of the Project are as follows:

- Project Director: Mr. Abdelkader BENHADJOUDJA - Chief of Minister’s Cabinet, MATE
- Project Manager: Mr. Tayeb TIRECHE - Director General, ONEDD

Besides the above, to secure the smooth implementation of the Project, following Joint Coordinating Committee (JCC) was established based on Record of Discussion (R/D).

Organization of JCC

1) Chairperson: Project Director - Chief of Minister’s Cabinet, MATET
2) Vice Chairperson: Project Manager - Director General, ONEDD
3) JCC Member

《Algerian side》

- MATET
- ONEDD Headquarter
- ONEDD CRL Alger
- ONEDD ERL Constantine
- ONEDD WRL Oran
- Environment Direction, Wilaya of Alger (DEWA)
- Environment Direction, Wilaya of Blida (DEWB)

《JICA》

- JICA Experts
- Representatives of JICA

《Others》

Ministry of Foreign Affairs of Algeria and the Embassy of Japan in Algeria may attend JCC as observer(s), when necessary.

Although the number of assigned counterpart personnel including General Director of ONEDD was 21 at the commencement of the Project, it has increased to 25 at the closing stage of the project (displaced: 4, reseated: 3, newly employed: 11). Table 1-2 shows the list of counterpart personnel assigned during the period of the Project.

(4) Project Implementation Period

Implementation period of the Project is 3 years from October 2009 to September 2012.

2 Output of the Project

2.1 Overview of Technology Transfer

Technology transfer by JET in the phase 2 was conducted focusing on acquisition of advanced analytic technique (output-1) using equipment of GCMS, FTIR and XRF. Those equipment were installed in the CRL laboratory in the phase 1. In addition to the above (output-1), technology transfer of the Project consists of remaining three (3) components such as upgrading of quality of environmental monitoring (output-2), enforcement of quality control capacity of lab analytical works (output-3) and dissemination of environmental monitoring technologies to other ONEDD regional laboratories, monitoring stations and other relevant organizations (output-4).

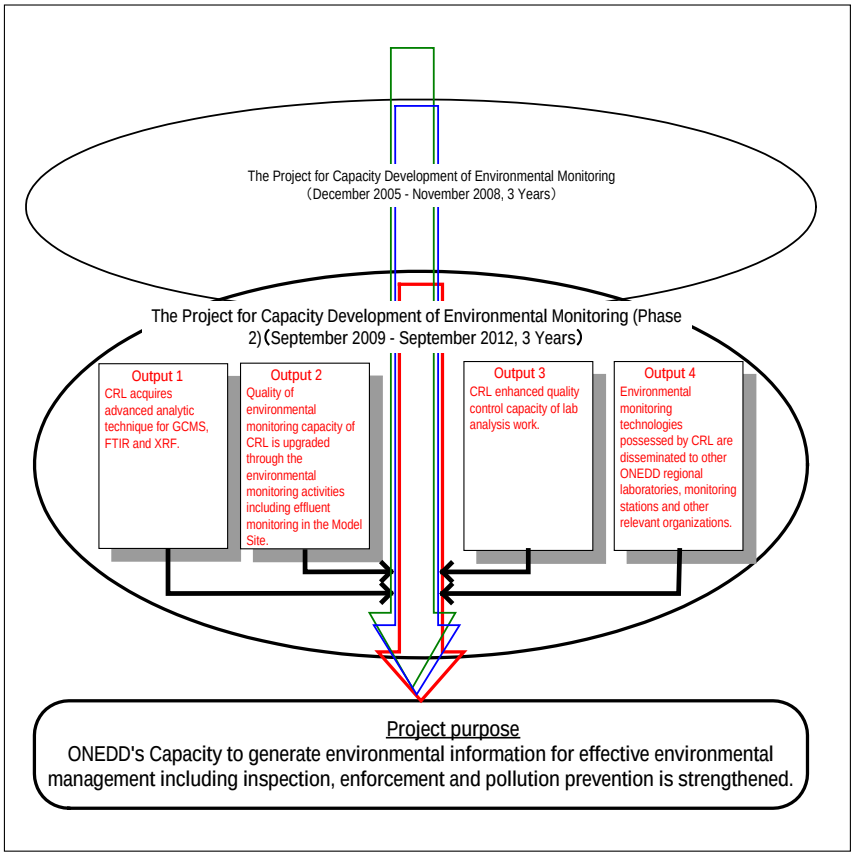


Figure 2.1.1 Concept of the Project

Figure 2.1.2 indicates the flowchart on technology transfer of the Project, where each output affects and complements each other for realizing the Project purpose of PDM.

Table 2.1.1 shows chronology of technology transfer during three (3) years in phase 2.

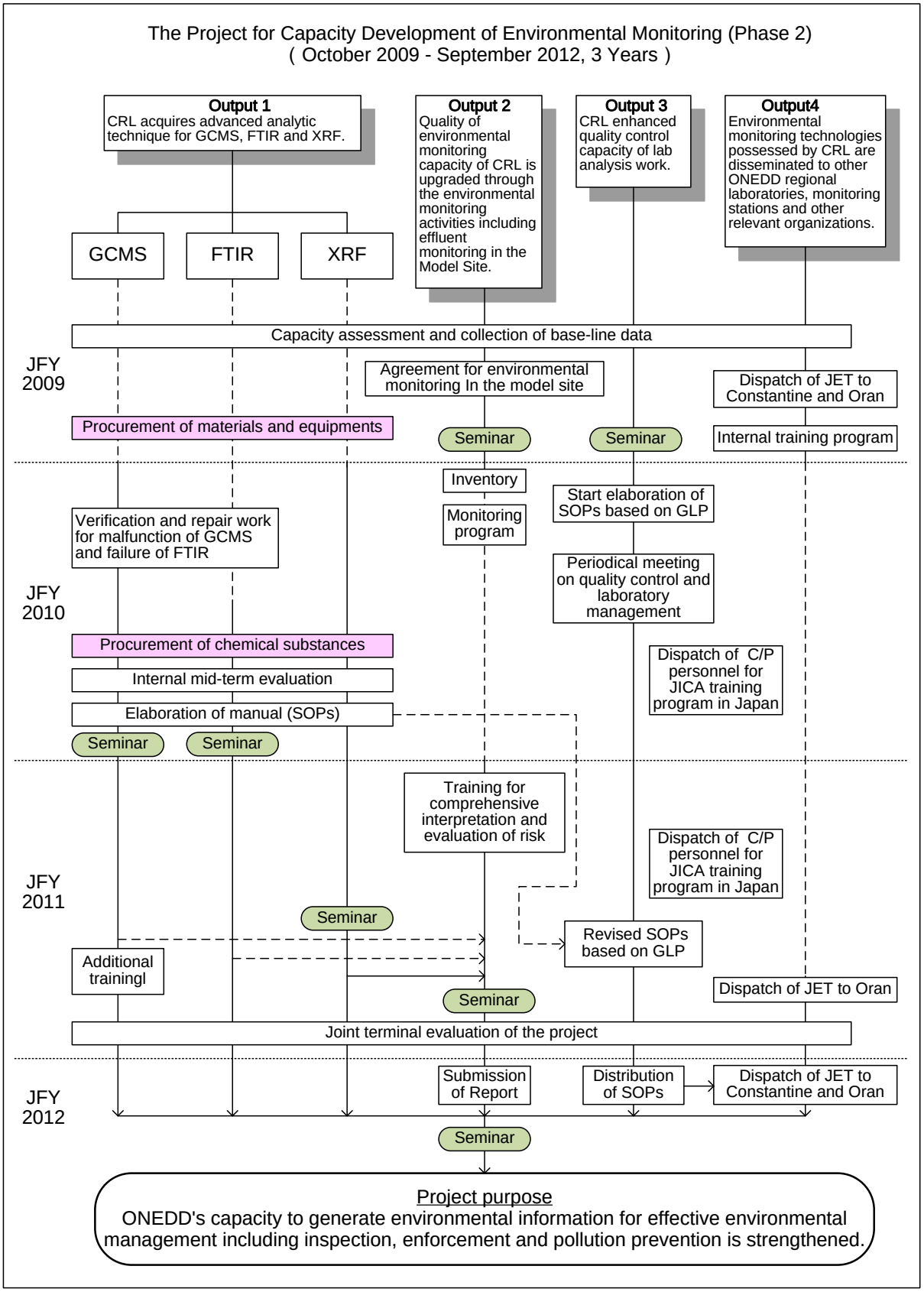


Figure 2.1.2 Flowchart of Technology Transfer

Table 2.1.1 Chronology for Technology Transfer of the Project

Year	Activities	Results of the Project
JFY 2009	Dispatch of JICA consultation team and JICA experts team (October)	1,2,3,4
	The 1st JCC (October)	1,2,3,4
	Conclusion of agreement for environmental monitoring between CRL-ONEDD, DEWA and DEWB (October)	2
	Capacity assessment and collection of base-line data (October - November)	1,3
	Dispatch of JET to Constantine regional laboratory for investigation of needs for internal training program (October)	4
	Verification and repair work for malfunction of GCMS and failure of FTIR (October - February)	1
	Dispatch of JET to Oran regional laboratory for investigation of needs for internal training program (February)	4
	The 1st (Environmental monitoring) and the 2nd (Quality control) seminar for technology transfer (February)	2,3
	Start environmental monitoring (river water and industrial wastewater) of the model site (2010-2012)	2
	Procurement of materials and equipment for GCMS, FTIR and XRF from Japan(March)	1
JFY 2010	The 4th Algeria - Japan joint seminar (April)	4
	Meeting on verification of technology transfer for analysis using GCMS, FTIR and XRF (June)	1
	The 2nd JCC (June)	1,2,3,4
	Dispatch of C/P personnel (Azouani Sophia） for JICA training program in Japan (May-August)	4
	Start elaboration of SOP based on GLP (May 2010 - June 2012)	3
	Repair works for FTIR and adjustment for GCMS by SHIMAZU (June)	1
	Periodical meeting on quality control and laboratory management (June 2010 - June 2012)	3
	Dispatch of C/P personnel (Mokhatari Hanifa） for JICA training program in Japan (August-September)	4
	Dispatch of C/P personnel (Daouadji Nassima) for JICA training program in Japan (September-October)	4
	Procurement of chemical substances for GCMS、XRF from France (October)	1
	Establish of the system for industrial effluents monitoring between MATE-ONEDD and D. E. Wilaya (November)	2
	Completion of technology transfer for FTIR (February)	1
	Internal mid-term evaluation of transfer technology on advanced equipment (GCMS, FTIR and XRF) (February)	1
	Elaboration of manual for operational and maintenance on GCMS, FTIR and XRF (February)	1

	The 3rd seminar (GCMS) and the 4th seminar (FTIR) for technology transfer (February)	1
JFY 2011	Dispatch of JICA consultation team (April)	1,2,3,4
	The 5th Algeria - Japan joint seminar (April)	4
	Dispatch of C/P personnel (Guerfi Lynda） for JICA training program in Japan (August-October)	4
	Training for comprehensive interpretation and evaluation of risk in the model site (June 2011 - November 2012)	2
	The 5th seminar for technology transfer on XRF (October)	1
	Additional training for GCMS analysis (November)	1
	The 3rd JCC (November)	1,2,3,4
	Dispatch of JET to Oran regional laboratory for training (November)	4
	Print and distribution of SOPs (November)	3
	The 6th seminar for technology transfer on comprehensive interpretation and evaluation of risk (February)	2
	Additional training and completion of technology transfer for GCMS (February)	1
	Joint terminal evaluation of the Project (February)	1,2,3,4
JFY 2012	The 6th Algeria - Japan joint seminar (April)	4
	Dispatch of JET to Oran and Constantine regional laboratories for training of GLP (May)	3,4
	Completion of technology transfer for XRF (June)	1
	The 7th seminar for technology transfer on the summary of activities of the Project (June)	1,2,3,4
	Print and circulation of supplemental SOPs (June)	3
	The 4th JCC (July)	1,2,3,4
	Termination of dispatch of JET(July)	1,2,3,4
	Submission of final report of the Project (September)	1,2,3,4

2.2 Output-1

Based on PDM of the Project, expected output-1 and its verifiable indicators are shown in Table 2.2.1. In order to achieve the Project purpose and expected output, CRL-ONEDD conducted their activities by instructions of JET.

Table 2.2.1 Detailed Output with Verifiable Indicators in Output-1 of PDM

Output of the Project	Detailed Output with Verifiable Indicators	Means of Verification
Output-1 CRL acquires advanced analytic technique for GCMS, FTIR and XRF.	1. Reliable analytical results on hydrocarbon, organo-chlonine, BTX, PAH and agrochemicals (pesticides and insecticides) are generated using GCMS. 2. Reliable analytical results on non-volatile organic chemicals are generated using FTIR and its data library. 3. Reliable results of quantitative XRF analysis are generated. 4. SOPs for advanced analytical methods for GCMS, FTIR and XRF are developed.	1, 2, 3 Records of analyses 4. SOPs

Practicing the technology transfer for advance analytic technique, application for GCMS, FTIR and XRF in the Project were examined in the meeting between CRL-ONEDD and JET in June 2010. As a result of discussion, according to the agreement by JET and CRL-ONEDD as shown in Table 2.2.2, activities of technology transfer for three equipment were started.

Table 2.2.2 Practical Application for GCMS, FTIR and XRF in the Project

Equipment	Analytic Substance	Parameters of industrial wastewater (Décret exécutif No. 06-141)	Application to the Project (Technology Transfer)				Period of application for environmental monitoring
			Industrial wastewater	Surface water	Groundwater	Sediment of river (Soul)	
GCMS/ P&T	BTX; Benzene, Toluene, Xylene	For designated industries (type of activities)	Applicable	Applicable	Applicable	Impossible in LRC	2010 - Analyze some of the substances
	PAH, aromatic hydrocarbon	For designated industries (type of activities)	Application very difficult	Application possible	Application possible	Application possible	2011 - Analyze some of the substances
	Volatile organochlorine	Application to all of industrial wastewater	Applicable	Applicable	Applicable	Impossible in LRC	2011 – Analyze some of the detergents and others
	Organochlorine pesticide	Application to all of industrial wastewater	Not applicable	Applicable	Not applicable	Not applicable	2011 – Analyze some of the representative residual insecticides
FTIR	Non-volatile organic compound	No standard value	Applicable	Applicable	Applicable	Applicable	2011 – Qualitative analysis of oil, such as heavy oil, wax, and others
XRF	Heavy metals (Cd, Pb, As, Cr)	Application to all of industrial wastewater	Not applicable	Not applicable	Not applicable	Applicable	2011 – Use AAS simultaneously depending on concentration
	Hg	Application to all of industrial wastewater	Not applicable	Not applicable	Not applicable	Applicable	2011 – Hg analyzer used simultaneously depending on concentration

(1) GCMS

Products of technology transfer and records of training/activities in the field of GCMS analysis are listed respectively in Table 2.2.3. The major products which will be revised by CRL-ONEDD after the Project are collected in the annex of the final report as follows:

- Annex 2-1-1-1 Record of training for GCMS analysis
- Annex 2-1-1-2 Mid-term evaluation of technology transfer on advanced equipment (GCMS)
- Annex 2-1-1-3 Summary of GCMS analysis
- Annex 2-1-1-4 Standard operation procedures (SOP) for GCMS analysis

Table 2.2.3 Products of Technology Transfer for GCMS

Year	Products	Source		**Annex in the final report**
		Report	Annex	
2009	Record of training	Progress Report (I)	3-3-1-1-1	**2-1-1-1**
	SOP of the analysis using P&T-GCMS		3-3-1-1-2	
	Manual of maintenance (preliminary draft of SOP)		3-3-1-1-3	
	Record of daily checklist of GCMS		3-3-1-1-4	
2010	Summary of technological transfer for GCMS	Progress Report (II)	3-3-1-1-1	
	Summary of training results - Analytical results of BTX in underground waters - Calibration curve and Total Ion Chromatogram of VOCs - Drawing up the list of Compounds Information of VOCs - Detection limits and Determination limits of VOCs Calibration curve and Total Ion Chromatogram of PAH - Results of separation test of PAH by silica gel column - Analytical results of PAH		3-3-1-1-2	
	Record of training in 2010		3-3-1-1-3	**2-1-1-1**
	Record of daily checklist of GCMS in 2010		3-3-1-1-4	
	SOP of the analysis for VOCs including BTX using P&T-GCMS		3-3-1-1-5	
	SOP of the analysis for PAH in the water using GCMS		3-3-1-1-6	
	SOP of the analysis for PAH in the soil using GCMS		3-3-1-1-7	
	SOP of operation manual for GCMS - Operation procedure to change from P&T to direct injection		3-3-1-1-8	
	SOP of maintenance manual for GCMS		3-3-1-1-9	
	Mid-term evaluation of technology transfer on advanced equipment (GCMS)		3-2	**2-1-1-2**
2011	Analytical results of PAH - Results of recovery tests - Analytical results of PAH in certified standard materials and biota sample - Report of the analysis MEDPOL - Detection limits and Determination limits of PAH in water	Progress Report (III)	3-2-1-1-1 3-2-1-1-2 3-2-1-1-3 3-2-1-1-4	
	Record of training in 2011		3-2-1-1-5	**2-1-1-1**
	Summary of technology transfer on GCMS analysis in 2012		3-2-1-1-6	**2-1-1-3**
	Standard operation procedures (SOP) for GCMS analysis		Separate Volume	**2-1-1-4**

(2) FTIR

Products of technology transfer and records of training/activities in the field of FTIR analysis are listed in Table 2.2.4. All the products such as manuals, analysis methods and result of analysis using FTIR are included in the Annex 2-1-2-2 of the final report as follows:

- Annex 2-1-2-1 Record of training for FTIR analysis
- Annex 2-1-2-2 Mid-term evaluation of technology transfer on advanced equipment (FTIR)
- Annex 2-1-2-3 Standard operation procedures (SOP) for FTIR analysis

Table 2.2.4 Products of Technology Transfer for FTIR

Year	Products	Source		**Annex in the final report**
		Report	Annex	
2009	Record of training	Progress Report (I)	3-3-1-2-1	**2-1-2-1**
	Program of cooperative guide		3-3-1-2-2	
	Standard operating procedure for FTIR spectroscopy		3-3-1-2-3	
2010	Operation manual (SOP) of equipment and materials	Progress Report (II)	3-3-1-2-1	
	Analysis of non-volatile organic compounds by KBr technique and SOP		3-3-1-2-2	
	Analysis of non-volatile organic compounds by ATR technique and SOP		3-3-1-2-3	
	Analytical method of FTIR spectra of non-volatile organic compounds		3-3-1-2-4	
	Management of samples and spectral data collected		3-3-1-2-5	
	Record of Training		3-3-1-2-6	**2-1-2-1**
	Recommendation of target substances for FTIR		3-3-1-2-7	
	Operation manual (SOP) of equipment and materials		3-3-1-2-1	
	Mid-term evaluation of technology transfer on advanced equipment (FTIR)		3-2	**2-1-2-2**
2011	Standard Operation Procedures (SOP) for FTIR analysis	Progress Report (III)	Separate Volume	**2-1-2-3**

(3) XRF

Products of technology transfer and records of training/activities in the field of XRF analysis are listed in Table 2.2.5. The major products which will be revised by CRL-ONEDD after the Project are collected in the annex of the final report as follows:

- Annex 2-1-3-1 Record of training for XRF analysis
- Annex 2-1-3-2 Mid-term evaluation of technology transfer on advanced equipment (XRF)
- Annex 2-1-3-3 XRF results of Pb content from sediment of Oued Harrach
- Annex 2-1-3-4 List of XRF analysis during training in 2009-2012
- Annex 2-1-3-5 Standard operation procedures (SOP) for XRF analysis

Table 2.2.5 Products of Technology Transfer for XRF

Year	Products	Source		**Annex in the final report**
		Report	Annex	
2010	Record of training	Progress Report (II)	3-3-1-3-1	**2-1-3-1**
	Maintenance manual (SOP) for minipal4		3-3-1-3-2	
	Sample preparation Manual (SOP) for XRF		3-3-1-3-3	
	Determination of Plomb (Pb) in rice by XRF		3-3-1-3-4	
	Preparation of background sediment “Oued El Harrach”		3-3-1-3-5	
	Mid-term evaluation of technology transfer on advanced equipment (XRF)		3-2	**2-1-3-2**
2011	Record of training	Progress Report (III)	3-2-1-2-1	**2-1-3-1**
	Resume of XRF Seminar		3-2-1-2-2	
	XRF results of Pb content from sediment of Oued Harrach		3-2-1-2-3	**2-1-3-3**
	Manual (draft) of "Analysis of liquid sample using XRF in CRL		3-2-1-2-4	
	Manual (draft) of "Screening of Pb in environmental sample"		3-2-1-2-5	
	List of XRF analysis during training in 2009-2012		3-2-1-2-6	**2-1-3-4**
	List of element and equation for standard curves		3-2-1-2-7	
	Records of log book		3-2-1-2-8	
	Standard Operation Procedures (SOP) for XRF analysis (draft)		Separate Volume	
2012	Record of training	Japanese Annual Report (IV)	3-1-1	**2-1-3-1**
	Standard Operation Procedures (SOP) for XRF analysis		3-1-2	**2-1-3-5**

2.3 Output-2

Based on PDM of the Project, expected detailed output-2 and its verifiable indicators are shown in table 2.3.1. In order to achieve the Project purpose and expected output, CRL-ONEDD conducted their activities by instructions of JET.

Table 2.3.1 Detailed Output with Verifiable Indicators in Output-2 of PDM

Output of the Project	Detailed Output with Verifiable Indicators	Means of Verification
Output-2 Quality of environmental monitoring capacity of CRL is upgraded through the environmental monitoring activities including effluent monitoring in the Model Site.	1. Pollution inventories including pollution loads are developed. 2. Comprehensive monitoring plan including effluent monitoring plans is developed. 3. Collaborative effluent monitoring activities with DEWA and DEWB are conducted periodically. 4. Types/kinds of analysis parameters are increased. 5. Comprehensive interpretation and risk assessment of the monitoring results are publicized.	1. Pollution inventories 2. Comprehensive monitoring plan 3. Records of effluent monitoring activities 4. Records of analysis 5. Presentation documents, reports, publication

Products of technology transfer and records of training/activities on output-2 are listed in Table 2.3.2. The major products which will be revised by CRL-ONEDD after the Project are collected in the annex of the final report as follows:

- Annex 2-2-1 Agreement of environmental monitoring
- Annex 2-2-2 Inventory of pollution sources
- Annex 2-2-3 Record of meeting on working group
- Annex 2-2-4 Plan of environmental monitoring in the model site
- Annex 2-2-5 A Guideline for detailed environmental monitoring plan
- Annex 2-2-6 Record of training on output-2
- Annex 2-2-7 A Guideline for comprehensive interpretation and risk assessment
- Annex 2-2-8 Summary of environmental monitoring in 2012
- Annex 2-2-9 Summary of the pollution in the Oued El Harrach river basin

Regarding collected data and created lists and maps for comprehensive interpretation and risk assessment, the report as a separate volume including electric data was submitted separately to CRL-ONEDD.

Table 2.3.2 Products of Technology Transfer for Output-2

Year	Products	Source		**Annex in the final report**
		Report	Annex	
2009	Agreement of environmental monitoring	Progress Report (I)	3-3-2-1	**2-2-1**
	Inventory of point pollution sources		3-3-2-2	**2-2-2**
	Record of meeting on working group		3-3-2-3	**2-2-3**
	Plan of environmental monitoring in the model site		3-3-2-4	**2-2-4**
2010	A Guideline for detailed environmental monitoring plan	-	-	**2-2-5**
	Summary of environmental monitoring in 2010	Progress	3-3-2	

		Report (II)		
2011	Summary of environmental monitoring in 2011	Progress Report (III)	3-2-2-1	
	Summary of comprehensive interpretation and risk assessment in the model site		3-2-2-2	
	Record of training on Output-2		3-2-2-3	**2-2-6**
	Resume of Seminar on Output-2		3-2-2-4	
	A Guideline for comprehensive interpretation and risk assessment		Separate Volume	**2-2-7**
2012	Summary of environmental monitoring in 2012	Japanese Annual Report (IV)	3-2-1	**2-2-8**
	Summary of pollution in the Oued El Harrach river basin		3-2-2	**2-2-9**

2.4 Output-3

Based on PDM of the Project, expected detailed output-3 and its verifiable indicators are shown in table 2.4.1. In order to achieve the Project purpose and expected output, CRL-ONEDD conducted their activities by instructions of JET.

Table 2.4.1 Detailed Output with Verifiable Indicators in Output-3 of PDM

Output of the Project	Detailed Output with Verifiable Indicators	Means of Verification
Output-3 CRL enhanced quality control capacity of lab analytical works.	1. More than 16 staff in CRL work for quality control for inorganic/organic/microbiological analysis. 2. More than 16 staff in inorganic/ organic/ microbiological analysis section in CRL joined trainings on quality control. 3. Quality control system of analytic works is established in CRL.	1. Hearing from CRL 2. Training records Hearing from CRL 3.2 QC reports and log books in CRL

Products of technology transfer and records of training/activities on output-3 are listed in Table 2.4.2. The major products which will be revised by CRL-ONEDD after the Project are collected in the annex of the final report as follows:

- Annex 2-3-1 Good laboratory practice
- Annex 2-3-2 Organization: list of staff
- Annex 2-3-3 Procedures for management of analytical sample
- Annex 2-3-4 Specimen documents of laboratory
- Annex 2-3-5 SOP verion 1.01
- Annex 2-3-6 Supplemental SOP
- Annex 2-3-7 Record of QC meeting

Regarding SOP (Standard Operating Procedure) as listed in Annex 2-3-5 and Annex 2-3-6, these reports were published with 200 copies respectively and distributed widely in Algeria.

Table 2.4.2 Products of Technology Transfer for Output-3

Year	Products	Source		**Annex in the final report**	Remark
		Report	Annex		
2009	List of SOPs	Progress Report (I)	3-3-3-1		
	List of certified reference material		3-3-3-2		
	Logbook of water purifier		3-3-3-3		
	Logbook of chemical balance		3-3-3-4		
	Organization structure for quality control system in CRL		3-3-3-5		
	Responsibility of the staff, section chief, and director for quality control system in CRL		3-3-3-6		
2010	List of revised SOPs	Progress Report (II)	3-3-3-1		
	List of certified reference material		3-3-3-2		
	Responsibility of the staff, section chief, and Director for quality control system in CRL		3-3-3-3		
	List of equipment logbooks prepared in CRL		3-3-3-4		

2011	Good Laboratory Practice (GLP)	Progress Report (III)	3-2-3-1	**2-3-1**	
	Organization: List of staff		3-2-3-2	**2-3-2**	
	Procedures for management of analytical sample		3-2-3-3	**2-3-3**	
	Record of QC meeting		3-2-3-4		
	List of client agreement		3-2-3-5		
	List of daily clients (from Nov.13. 2011)		3-2-3-6		
	Specimen documents of laboratory		3-2-3-7	**2-3-4**	
	SOP ver.1.01		3-2-3-8	**2-3-5**	**Published**
	Supplemental SOP (draft: pH, DO, Conductivity)		3-2-3-9		
	Safety protocol		3-2-3-10		
2012	Supplemental SOP	Japanese Annual Report (IV)	3-3-1	**2-3-6**	**Published**
	Program of seminar for regional laboratory		3-3-2		Output-4
	Record of training for regional laboratory		3-3-3		
	Record of QC meeting		3-3-4	**2-3-7**	

2.5 Output-4

Based on PDM of the Project, expected output-4 and its verifiable indicators are shown in table 2.5.1. In order to achieve the Project purpose and expected output, CRL-ONEDD conducted their activities by instructions of JET.

Table 2.5.1 Detailed Output with Verifiable Indicators in Output-4 of PDM

Output of the Project	Detailed Output with Verifiable Indicators	Means of Verification
Output-4 Environmental monitoring technologies possessed by CRL are disseminated to other ONEDD regional laboratories, monitoring stations and other relevant organizations.	1. Training team by ONEDD(HQ) and CRL is formulated. 2. Training plan for regional laboratories and monitoring stations is developed. 3. Training courses for regional laboratories and monitoring stations are conducted by twice a year. 4. Various stakeholders including industries, academics and NGOs participated in ONEDD-MATET-JICA Joint Seminar. 5. The workshops for regional laboratories are held as a dissemination of Project contribution.	1. Hearing from ONEDD 2. Training plan 3. Training records 4.1 Records of joint seminars 4.2 Proceedings of the seminars 5. Records of workshops

Products of technology transfer and records of training/activities on output-4 are listed in Table 2.5.2. Records of training program are collected in the annex of the final report as follows:

- Annex 2-4-1 Internal plan of training
- Annex 2-4-2 Program of training for regional laboratory
- Annex 2-4-3 Program of seminar for regional laboratories and monitoring stations

The SOP based on GLP which was elaborated in output-3 was mainly used as a text book in the training. Regarding ONEDD-MATET-JICA Joint Seminar, as described in the section 2.6, proceedings were published and distributed widely.

Table 2.5.2 Products of Technology Transfer for Output-4

Year	Products	Source		**Annex in the final report**	Remark
		Report	Annex		
2009	Internal plan of training	Progress Report (I)	3-3-4-1	**2-4-1**	
2010	Program of training in Japan (2010)	Progress Report (II)	3-3-4	4-1	Chapter 4
2011	Program of training in Japan (2011)	Progress Report (III)	3-2-4-1	4-1	Chapter 4
	Program of training for regional laboratory		3-2-4-2	**2-4-2**	
2012	Program of seminar for regional laboratories and monitoring stations	Japanese Annual Report (IV)	3-3-2	**2-4-3**	

2.6 Seminars and Workshops

(1) Seminar for Technology Transfer

All the materials presented in the seminar for technology transfer are collected in the Annex 2-5-1.

Outlines of seminars held in the course of this project can be summarized as follows:

1. The 1st Seminar on Environmental Monitoring
   - Date: February 25th, 2010
   - Place: Conference room Hotel du Port in Alger city
   - Participants: All members related to the Project, CRL- ONEDD, DEWA, DEWB and others.
   - Theme of Presentation:
     1) An approach for environmental monitoring (Kenji Fukushima, JET)
     2) Pollution of the Oued El Harrach River in DEWA (Khelifi Fatiha, DEWA)
     3) Pollution of the Oued El Harrach River in DEWB (Hammouda Rachid Fethi, DEWB)

2. The 2nd Seminar on Quality Control
   - Date: February 25th, 2010
   - Place: Conference room Hotel du Port in Alger city
   - Participants: All members related to the Project, CRL-ONEDD, DEWA, DEWB and others.
   - Theme of Presentation:
     1) Guide of Good Practice Laboratory in CRL (Moali Mohamed, CRL(
     2) Sampling (Lakhdari Mohamed, CRL)
     3) Quality assurance for procedures of inorganic analysis (Azouani Sophia, CRL)
     4) Data base (Hanifa Mebrek, CRL)
     5) Quality control in laboratory (Ryo Ishimoto, JET)

3. The 3rd Seminar on advanced analytic technique for volatile organic chemicals using GCMS
   - Date: February 21st, 2011
   - Place: CRL meeting room (Ben Aknoun, Alger city)
   - Participants: All members related to the Project, CRL-ONEDD and others.
   - Theme of Presentation:
     1) Application of GCMS to environmental monitoring in Algeria (Tomoko Fukaya, JET)
     2) Analysis of volatile organic chemicals using Pruge and Trap (Kimri Leila, CRL)
     3) Analysis of polycyclic aromatic hydrocarbons in water (Nechaouni Leila, CRL)

4. The 4th Seminar on advanced analytic technique for non-volatile organic chemicals using FTIR
   - Date: February 21st, 2011
   - Place: CRL meeting room (Ben Aknoun, Alger city)
   - Participants: All members related to the Project, CRL-ONEDD and others.
   - Theme of Presentation:
     1) FTIR Spectroscope for environmental monitoring (Masamichi Tsuji, JET)
     2) Analysis by FTIR using ATR method (Anane Radhia, CRL)
     3) Analysis by FTIR using KBr pellet method (Bensouilah Ouhahiba, CRL)

5. The 5th Seminar on advanced analytic technique for heavy metals using XRF
   - Date: October 24th, 2011
   - Place: CRL meeting room (Ben Aknoun, Alger city)
   - Participants: All members related to the Project, CRL-ONEDD and others.

- Theme of Presentation:
  1) Outline of XRF analysis (Ryo Ishimoto, JET)
  2) Preparation of sample for XRF analysis (Djoghlaf Hada, CRL)
  3) XRF analysis for contaminated soil with heavy metals (sediments from Oued El Harrach) (Azouani Sophia, Guerfi Lynda, CRL)
  4) Methodology for identification of element's spectre using XRF (Houas Omar, CRL)
  5) Introduction of XRF analysis as an example of Japan (Ryo Ishimoto, CRL)

6. The 6th Seminar on detailed interpretation and evaluation of risk based on monitoring results in the model site of the Project
   - Date: February 6th, 2011
   - Place: CRL meeting room (Ben Aknoun, Alger city)
   - Participants: All members related to the Project, CRL-ONEDD and others.
   - Theme of Presentation:
     1) Outline for understanding our works (Kenji Fukushima, JET)
     2) General background of pollution in the model site (Benboudjema Meriem , Hannachi Naila, ONEDD-HQ)
     3) Collection and treatment of data (Mebrek Hanifa, ONEDD-HQ)
     4) A trial for interpretation, evaluation of risk, conclusion and recommendation (Boulekraouet Souhila,ONEDD-HQ)

7. The 7th Seminar on summary of results on the Project for capacity development of environmental monitoring (Phase2)
   - Date: June 26th, 2012
   - Place: Mouflon D'or in Alger city
   - Participants: All members related to the Project, CRL-ONEDD and others.
   - Theme of Presentation:
     1) Output 1: CRL acquires advanced analytic technique for GCMS, FTIR and XRF (Omri Lynda, Bensouilah Ouhahiba, Azouani Sophia, CRL)
     2) Output 2: Quality of environmental monitoring capacity of CRL is upgraded through the environmental monitoring activities including effluent monitoring in the Model Site (Mebrek Hanifa, ONEDD-HQ)
     3) Output 3 CRL enhanced quality control capacity of lab analytical works. (Houas Omar, CRL)
     4) Output 4 Environmental monitoring technologies possessed by CRL are disseminated to other ONEDD regional laboratories, monitoring stations and other relevant rganizations. (Moali Mohamed, CRL)

(2) Algeria-Japan Joint Workshops and Seminars

All the materials presented in the Algeria-Japan joint workshops and seminars were distributed widely as a publication of proceedings every year (see Annex 2-5-2). Outlines of the Algeria-Japan joint workshops and seminar held in the course of this project can be summarized as follows:

1. Algeria-Japan Joint Workshops and Seminar (4th) on Water Environmental Protection 2010
   - Date: April 26th and 27th, 2010
   - Place: Auditorium of MATE
   - Participants: All members related to the Project – ONEDD, DEWA, and others.
   - Theme of Presentation:
     1) Introduction of the fourth Algeria-Japan joint seminar on water environmental protection

(Tayeb Tireche (ONEDD), Mitsuo Yoshida (JICA))

2) Historical background of Minamata issue, Japan
(Naoki Ikeda, (Kwansei Gakuin Univ. Japan))
3) Environmental water quality standards regulation of water evaluation and integrated indicator of water environment in Japan (Mitsuo Yoshida, JICA)
4) Water environment in the province of Aichi (Japan), current situation and planned and taken measures (Mayumi Otani (Aichi Prefecture, Japan))
5) The use of Algerian bentonite clay for the removal of heavy metals from aqueous solution: Application in wastewater treatment (Soraya DIB, Makhlouf Boufatit, U.S.T.H.B)
6) Application of natural materials to the environmental protection from the contamination by toxic metals (Mitsuo Yoshida, JICA)
7) Essay of treatablility of turbide in case of "Beni Amrane" Dam, using biofloculant "Chitosane" (H. Zemmouri, H. Lounici, N. Mameri (L.B.E.G.P - E.N.S.P)
8) Study of the pollution in the Oued El Harrach basin (Moali Mohamed, ONEDD-CRL)
9) Legal and regulatory framework ruling the industrial liquid wastes (A. Bechari, MATE)

2. Algeria-Japan Joint Workshops and Seminar (5th) on Solid Waste and Pollution 2011
- Date: April 19th and 20th, 2011
- Place: Auditorium of MATE
- Participants: All members related to the Project – ONEDD-CRL and others.
- Theme of Presentation:
1) Introduction of the fifth Algeria-Japan joint seminar on solid wastes and pollution
(Tayeb Tireche (ONEDD), Mitsuo Yoshida (JICA))
2) Management of toxic wastes in Japan (Shoichi Hayami, JICA)
3) Environmental pollution problems caused by the solid wastes (Mitsuo Yoshida, JICA)
4) Development of environmental monitoring capacities (Moali Mohamed, ONEDD-CRL)
5) Application of the XRF (X-ray analyser) for measuring the heavy metals in sediments
(Sophia Azouani, ONEDD-CRL)
6) Analysis of volatile organic compounds using Gas-chromaotgraph-MS Spectrometer
(Leila Kimri, Leila Nechauouni, ONEDD-CRL)

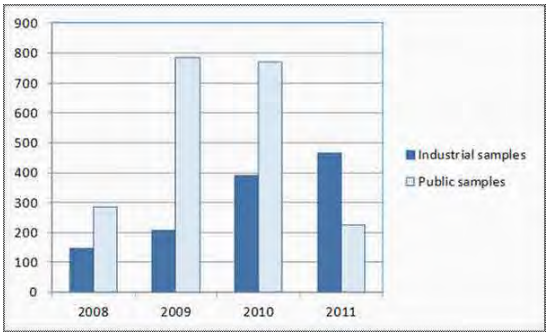
3. Algeria-Japan Joint Workshops and Seminar (6th) on marine pollution due to activities on land
- Date: April 24th - 25th, 2012
- Place: Conference room of Hotel Abassydes Palace, Palm Beach (Tipaza)
- Participants: Members related to the Project – ONEDD,-CRL and others.
- Theme of Presentation:
1) Introduction of the sixth Algeria-Japan joint seminar 2012 and summary of technical cooperation between JICA and ONEDD in the framework of environmental monitoring
(Tayeb Tireche (ONEDD), Mitsuo Yoshida (JICA))
2) Present state of marine environment pollution and regulations in Japan
(Mitsuo Yoshida, JICA)
3) Considerations related to restoration of environmental damages in a circumstance of marine farm, a study of Osaka Bay (Koji Otsuka, University of Osaka Prefecture, Japan)
4) Effects of marine pollution in Algeria on the loose bottoms: causes, effects and mitigations (Samir Grimes, E.N.N.S.M.A.L)
5) Integration of multisource data for the study of the state of pollution in the Bay of Algiers
(Houma Fouzia (E.N.N.S.M.A.L), Bachari Nour el islam (U.S.T.H.B))
6) Development of a GIS for mapping of biocenological maps using remote sensing at very high spatial resolution and submarine diving
(Bachari Nour el islam(U.S.T.H.B), Lamouti Soad (C.R.D.P.A)
7) Contribution to the impact study of the chemical pollution on the posidonius herbarium
(Guendouzi Y., Ghalmi R. (E.N.N.S.M.A.L))
8) Remote sensing of the marine flora by passive optical radiometers: a case of Algiers coast
(A. Hassini (U.S.T.O.M.B), M.A. Benmostefa (I.M.S.I-U.O)

9) Measuring the organic and metal contamination in the large ports
(Moali Mohamed, ONEDD-CRL)
10) Evaluation of the contamination of Oued El Harrach by the heavy metals
(Boulekraouet Souhila, ONEDD)
11) Water quality of bathing beach (Hanifa Mebrek, ONEDD)
12) The threat of the Algerian west coast line by urban and industrial effluents
(Krachai N, Hadjel M, Bendraoua A (L.S.T.G.P.- U.S.T.O.-M.B)
13) Control of water quality of Oran coast line case of industrial wastes from Arzew petrochemical industrial zone (Sahnoun F., Hadjel M. (L.S.T.G.P.- U.S.T.O.-M.B))

2.7 Achievement of Project

Achievement of PDM indicators based on the joint terminal evaluation of the Project can be summarized in Table 2-7. As mentioned in the previous sections 2.2 to 2.6, detailed supporting evidence related to following indicators is collected in annexes of the report.

Table 2-7 Achievement of PDM Indicators

Narrative Summary	Indicators	Achievement
Project Purpose ONEDD's Capacity to generate environmental information for effective environmental management including inspection, enforcement and pollution prevention is strengthened.	1. The Central Regional Laboratory (Alger) is able to response to the requisition about the environmental monitoring from various clients 2. Number of disclosed information related environmental pollution is increased. 3. Number of effluent monitoring is increased. 4. Number of contract on industrial wastewater monitoring is increased.	As show in the following Figure 2-7-1, the number of clients is increasing since the commencement of the Project. It means CRL-ONEDD is able to respond to the requisition about the environmental monitoring from various clients, according to the human and installed analytical instruments (Indicator P-1). The information related to environmental pollution is disclosed three times in the occasions of joint Seminar in 2010, 2011 and 2012, by the CRL staff. It is expected to disclose the results of monitoring program of the model site through ONEDD website (Indicator P-2). The number of effluent monitoring in the model site is also increased as shown in the Figure 2-7-1 (Indicator P-3). Lastly, the number of contract on industrial wastewater monitoring is increased as summarized in the Table 2-7-1 (Indicator P-4).  **Figure 2-7-1 The number of samples analyzed by CRL-ONEDD (source CRL-ONEDD)** **Table 2-7-1 The number of clients on industrial wastewater monitoring (source CRL-ONEDD)** Fiscal Year / Number of clients 2008 / 40 2009 / 54 2010 / 69 2011 / 82 Judging from the above figures, the ONEDD's capacity to generate environmental information for effective environmental management including inspection, enforcement and prevention is undoubtedly strengthened. The number of monitoring services for industrial units is steadily increased since the commencement of the course of the Project, which indicates that CRL-ONEDD is gradually recognized as an environmental monitoring

		institute for effective environmental management. Therefore, the Project Purpose could be said as **"mostly achieved"**. However, in order to sustain the current level of achievements, continuous efforts to expand environmental monitoring including effluent monitoring, which requires coordination among stakeholders, are needed.
Output 1 CRL acquires advanced analytic technique for GCMS, FTIR and XRF.	1. Reliable analytical results on hydrocarbon, organo-chlonine, BTX, PAH and agrochemicals (pesticides and insecticides) are generated using GCMS. 2. Reliable analytical results on non-volatile organic chemicals are generated using FTIR and its data library. 3. Reliable results of quantitative XRF analysis are generated. 4. SOPs for advanced analytical methods for GCMS, FTIR and XRF are developed.	Owing to the limitation of present prefabricated lab infrastructure, toxic organic chemicals (organo-chlorine, pesticide, insecticide, etc.) cannot be analyzed. BTX also cannot be analyzed due to malfunction of P&T unit of GCMS. However other volatile organic compounds can be analyzed using GCMS. The results of test analysis of masked standard sample in the terminal evaluation showed that the reliability of GCMS analysis of the volatile compounds is satisfactory level (Indicator 1-1). The results of test analysis of masked standard sample in the Terminal evaluation showed that the reliability of FTIR analysis of non-volatile compounds is satisfactory level (Indicator 1-2). As for the reliability of XRF qualitative analysis, the results of test analysis of masked standard sample in the Terminal evaluation showed that it is satisfactory level. However, regarding the quantitative analysis, it has been developed basically (Indicator 1-3). Apart from these analytical techniques, SOPs for advanced analytical methods for GCMS, FTIR and XRF have been successfully developed, which are practically applicable in present conditions of ONEDD. SOPs for other analytical instruments also developed. A handbook of SOPs (preliminary version) is firstly published by ONEDD under the financial support of JICA (Indicator 1-4). It is expected that CRL-ONEDD will keep on acquisition of advanced analytical technique for GCMS, FTIR and XRF, even after completion of the Project. However due to malfunction of P&T device of GCMS, one of advanced techniques cannot be practically utilized. In summary, the Output 1 could be said as **"mostly achieved".**
Output 2 Quality of environmental monitoring capacity of CRL is upgraded through the environmental monitoring activities including effluent monitoring in the Model Site.	1. Pollution inventories including pollution loads are developed. 2. Comprehensive monitoring plan including effluent monitoring plans is developed. 3. Collaborative effluent monitoring activities with DEWA and DEWB are conducted periodically. 4. Types/kinds of analysis parameters are increased. 5. Comprehensive interpretation and risk assessment of the monitoring results are publicized.	Inventories of industrial unit (potential polluters) are developed for the Model Site (Oued El Harrach and Oued Smar area). However DEWA did not give the necessary support to CRL-ONEDD for the sampling in these industrial units. This prevented to make detailed inventories of the pollution sources (Indicator 2-1). Monitoring plan including effluent monitoring plan is developed within the framework of available inventory data (Indicator 2-2). Collaborative effluent monitoring activities with DEWA and DEWB have been conducted five (5) times. Collaborative effluents monitoring with DEWB is frequent. As a whole, it is hard to say the monitoring has been conducted "periodically" (Indicator 2-3). 4 types and more than 39 kinds of analysis parameters are increased in the course of the Project as summarized in the following Table 2-7-2 (Indicator 2-4). Before the joint terminal evaluation of the Project, preliminary interpretation has been already attempted in CRL-ONEDD in-house workshop (Indicator 2-5). According to the plan of operation (see Annex 3-1), a comprehensive interpretation and risk assessment of the monitoring results in model site was presented in the final

<table>
<tr><td></td><td></td><td>seminar and the final report.

Table 2-7-2 Increased number of analytical parameters in the Project (source JET)
<table>
<tr><th>Type</th><th>GCMS</th><th>GCMS/ P&T</th><th>FTIR</th><th>XRF</th></tr>
<tr><td>Analytical Parameters</td><td>15</td><td>24</td><td>Non-volatile organic compound analysis</td><td>Quick element analysis of solid sample</td></tr>
</table>
It is expected that CRL-ONEDD will upgrade the environmental monitoring capacity even after completion of the Project if planned effluent monitoring activities in the model site will be successfully implemented in close collaboration with DEWA/DEWB under the coordination of MATE. Therefore, it was confirmed that the Output 2 could be said as “partly achieved”.</td></tr>
<tr><td>Output 3
CRL enhanced quality control capacity of lab analytical works.</td><td>1. More than 16 staff in CRL work for quality control for inorganic/organic/microbiological analysis.
2. More than 16 staff in inorganic/ organic/ microbiological analysis section in CRL joined trainings on quality control.
3. Quality control system of analytic works is established in CRL.</td><td>Total 20 staffs in CRL-ONEDD are participating the quality control works (Indicator 3-1). The number of the in-house workshops counts 29 times with participation of majority of staff (Indicator 3-2). Quality control system of analytical works is established on the basis of GLP (Good Laboratory Practice) principle and being managed by three core staff trained by the JICA expert (Indicator 3-3).
Therefore, it is expected that CRL-ONEDD will enhance the quality control capacity of lab analytical works even after completion of the Project. The framework of quality control is based on GLP concept which is firstly introduced new concept for ONEDD. In summary, the Output 3 could be said as “successfully achieved”.</td></tr>
<tr><td>Output 4
Environmental monitoring technologies possessed by CRL are disseminated to other ONEDD regional laboratories, monitoring stations and other relevant organizations.</td><td>1. Training team by ONEDD(HQ) and CRL is formulated.

2. Training plan for regional laboratories and monitoring stations is developed.

3. Training courses for regional laboratories and monitoring stations are conducted by twice a year.

4. Various stakeholders including industries, academics and NGOs participated in ONEDD-MATET-JICA Joint Seminar.

5. The workshops for regional laboratories are held as a dissemination of Project contribution.</td><td>A trainer team of ONEDD had been set-up with the support of JET as shown in the Table 2-7-3 (Indicator 4-1).

Table 2-7-3 Name of the engineer trainers from CRL (source CRL-ONEDD)
<table>
<tr><th></th><th>Target Parameters</th></tr>
<tr><td>MOALI Mohamed</td><td>Laboratory Management</td></tr>
<tr><td>ANANE Radia</td><td>Cyanide, Nitrogen Kjeldahl</td></tr>
<tr><td>AZOUANI Sophia</td><td>Heavy metals</td></tr>
<tr><td>BENSOUILAH Ouahiba</td><td>BOD5 and Total nitrogen</td></tr>
<tr><td>Lakhdari Mohamed</td><td>Sampling</td></tr>
<tr><td>DJOGHLAF Hadda</td><td>COD, oil and grease, SS</td></tr>
<tr><td>HOUAS Omar</td><td>Heavy metals</td></tr>
<tr><td>MEBREK Hanifa</td><td>COD, oil and grease, SS</td></tr>
<tr><td>NECHAOUNI Leila</td><td>Total phosphorus</td></tr>
<tr><td>TIBECHE Amel</td><td>COD, oil and grease, SS, florides, chlorides</td></tr>
</table>
A draft training plan for ONEDD regional laboratories and monitoring stations was developed (Indicator 4-2). As for training courses for ONEDD regional laboratories and monitoring stations, only two training courses were conducted by ONEDD owing to a lack of budget (see Table 2-7-4).</td></tr>
</table>

Table 2-7-4 Internal training course made by CRL engineers to their colleagues (source CRL-ONEDD)

Unit	Training duration	Year of the training	Number of trainees	Place of the traning
Monitoring station of Bordj Bou Aréridj	3 days	2009	3	Station of BBA
	3 days	2010	2	CRL
Monitoring station of Ain Edefla	3 days	2009	3	CRL
	6 days	2010	3	CRL
Monitoring station of Djelfa	3 days	2009	4	Station of Djelfa
	4 days	2010	2	CRL
Monitoring station of Annaba	4 days	2010	2	CRL
Eastern Regional Laboratory of Constantine	4 days	2010	2	CRL

Moreover, visiting consultations of JICA expert to Western Regional Laboratory Oran and Eastern Regional Laboratory Constantine were carried out (see Table 2-7-5). Laboratory staff of Oran and Constantine also participated in training programs conducted by JET in CRL-ONEDD (Indicator 4-3). Algeria-Japan Joint Seminar on Environmental Issue was organized three times, 2010, 2011 and 2012 in Alger, by MATE, ONEDD and JICA, according to the initial plan. The seminar topics were water pollution (2010), waste pollution (2011) and marine pollution (2012). More than 200 professionals, researchers, NGOs and government officers attended the Joint Seminars (Indicator 4-4).
JET-led workshops for ONEDD regional laboratories and monitoring station have been held six (06) times (Constantine (2009, 2012), Oran (2010, 2011, 2012), and Annaba (2012)), as summarized in the following Table 2-7-5 (Indicator 4-5).

Table 2-7-5 Record of workshops and training made by the Project supported by JET (source JET)

Year/ Month	Venue	Participants	Workshop	Training Course
Nov. 2009	Constantine	2 JICA experts 1 ONEDD HQ Officer 8 Regional lab staff	Discussion on environmental issues with industry and local government	
Feb. 2010	Oran	2 JICA experts 1 ONEDD HQ Officer 5 Regional lab staff	Discussion on laboratory issue	
Nov. 2011	Oran	1 JICA expert 1 CRL-ONEDD Director 1 ONEDD HQ Officer 6 Regional lab staff 5 Station staff	Discussion on laboratory issue	Handling of analytical data

May 2012	Constantine	1 JICA expert 1 ONEDD HQ Director General 1 CRL-ONEDD Director 7 Regional lab staff	GLP/SOP	GLP/SOP
May 2012	Annaba	1 JICA expert 1 ONEDD HQ Director General 1 CRL-ONEDD Director 10 Monitoring station staff	GLP/SOP	GLP/SOP
May 2012	Oran	1 JICA expert 1 CRL-ONEDD engineer 4 Regional lab staff	GLP/SOP	GLP/SOP

It is expected that CRL-ONEDD will disseminate the environmental monitoring technologies acquired by the Project to other ONEDD regional laboratories, monitoring stations and other relevant organizations after completion of the Project if planned training courses and workshops are successfully organized by the ONEDD headquarters. In summary, the Output 4 could be said as **"partially achieved"**.

3 Project Schedule and Achievement

Plan and achievement of project works are shown in Plan of Operation (PO) by bar chart as per attached in Annex 3-1 and Annex 3-2.

4 Input

4.1 Japanese Experts dispatched to Algeria

Assignment period of Japanese experts in the Project is summarized as shown in Table 4-1.

Table 4-1 Japanese Experts dispatched to Algeria

#	Field	Name	Assignment Period	M/M	Responsible Output and/or Activities of PDM
1	Leader/Environmental Management (Comprehensive Analysis, Risk Assessment, Laboratory Management)	Mr. Kenji FUKUSHIMA	16/10/2009 - 14/11/2009 (30)	1.00	1 to 4
			21/01/2010 - 06/03/2010 (45)	1.50	
			01/06/2010 - 30/06/2010 (30)	1.00	
			01/10/2010 - 30/10/2010 (30)	1.00	
			12/01/2011 - 10/02/2011 (30)	1.00	
			07/06/2011 - 30/06/2011 (24)	0.80	
			15/10/2011 - 19/11/2011 (36)	1.20	
			12/01/2012 - 10/02/2012 (30)	1.00	
			01/06/2012 - 30/06/2012 (30)	1.00	
2	Sub-Leader/X-Ray Fluorescence(XRF)/ Quality Control	Dr. Ryo ISHIMOTO	16/10/2009 - 14/11/2009 (30)	1.00	1-1, 1-3, 1-4, 2-4, 3-1, 3-2, 3-3, 4-3, 4-4
			05/02/2010 - 06/03/2010 (30)	1.00	
			18/05/2010 - 16/06/2010 (30)	1.00	
			26/10/2010 - 24/11/2010 (30)	1.00	
			17/01/2011 - 15/02/2011 (30)	1.00	
			20/05/2011 - 18/06/2011 (30)	1.00	
			03/10/2011 - 16/11/2011 (45)	1.50	
			09/01/2012 - 07/02/2012 (30)	1.00	
			15/05/2012 - 20/06/2012 (37)	1.23	
3	Gas Chromatography Mass Spectrometer (GCMS)	Ms. Tomoko FUKAYA	16/10/2009 - 09/11/2009 (25)	0.83	1-1, 1-2, 1-5, 2-4, 4-3, 4-4
			05/02/2010 - 06/03/2010 (30)	1.00	
			18/05/2010 - 16/06/2010 (30)	1.00	
			26/10/2010 - 24/11/2010 (30)	1.00	
			30/01/2011 - 28/02/2011 (30)	1.00	
			24/10/2011 - 29/11/2011 (37)	1.23	

			19/01/2012 - 17/02/2012 (30)	1.00	
4	Fourier Transform Infrared Absorption Spectrometry(FTIR)	Dr. Masamichi TSUJI	16/10/2009 - 04/11/2009 (20)	0.67	1-1, 1-3, 2-4, 4-4
			05/02/2010 - 06/03/2010 (30)	1.00	
			01/06/2010 - 08/06/2010 (8)	0.27	
			01/10/2010 - 22/11/2010 (22)	0.73	
			30/01/2011 - 23/02/2011 (25)	0.83	
5	Coordinator	Ms. Hiromi NONAKA	16/10/2009 - 14/11/2009 (30)	1.00	1 to 4
			05/02/2010 - 06/03/2010 (30)	1.00	
		Mr. Kenji FUKUSHIMA	11/02/2011 - 02/03/2011 (20)	0.67	
			11/02/2012 - 01/03/2012 (20)	0.67	
			01/07/2012 - 20/07/2012 (20)	0.67	

4.2 Counterpart Training in Japan

Trainings of counterpart in Japan were made as following Table 4-2, and their repots are shown in Annex 4-1.

Table 4-2 Counterpart Training

#	Name	Title/Responsibility	Title of Training Course	Training Period
1	Ms. Azouani Sophia	Engineer (CRL)	Pollution Control for Hazardous Substances in the environment	31 May 2010 to 7 July 2010
2	Ms. Mokhatari Hanifa	Engineer (CRL, ONEDD)	Urban Environmental Management	29 August 2010 to 15 September 2010
3	Mrs. Daouadji Nassima	Engineer (CRL)	Water Environmental Monitoring	5 September 2010 to 23 October 2010
4	Ms. Guerfi Lynda	Engineer (CRL)	Pollution Control and Local Environmental Management	20 August 2011 to 6 October 2011

4.3 Materials and Equipment Supplied by JICA

Materials and equipment supplied by JICA can be summarized as follows. Detailed list of supplied apparatuses and chemicals is shown in Annex 4-2.

（1）JFY 2009

【Brought from Japan】

- Materials and equipment for FTIR (6 items)
- Materials and Glassware for GCMS (38 items)
- Materials and equipment for XRF (13 items)

（2）JFY 2010

【Procured from France】

- Reagent and certified standard for GCMS (37 items)
- Certified standard for XRF (6 items)

【Brought from Japan】

- Equipment and Glassware for GCMS (7 items)
- Materials for XRF (1 item)

4.4 Local Cost by Japanese Side and Algerian Side

Local cost disbursed by Japanese side to implement the Project can be summarized as shown in Table 4-3. Expenditure of Algerian side (CRL-ONEDD) related to the Project in 2010 - 2011 is shown in Table 4-4.

Table 4-3 Local Cost by Japanese Side

Budget Item	Currency Unit	JFY 2009 (Sep.2009 - Mar.2010)	JFY 2010 (Apr.2010 - Mar.2011)	JFY 2011 (Apr.2011 - Mar.2012)	JFY 2012 (Apr.2012 - Sep.2012)	Total
Exchange rate (Euro/DZD)		97.077	98.987	99.325	100.147	
Exchange rate (DZD/Yen)		1.248	1.120	1.077	1.000	
Employment Cost	DZD	1,780,392	2,138,119	1,972,396	829,217	6,720,124
	Yen	2,221,929	2,394,693	2,124,270	829,217	7,570,109
Consumable goods	DZD	73,126	62,697	79,033	64,671	279,527
	Yen	91,261	70,220	85,118	64,671	311,270
Travel and Transport	DZD	41,121		14,690		55,811
	Yen	51,319	0	15,821	0	67,140
Communication	DZD		5,200	6,200	1,000	12,400
	Yen	0	5,824	6,677	1,000	13,501
Publishing materials	DZD	16,973	5,962	12,565	82,200	117,700
	Yen	21,182	6,677	13,532	82,200	123,591
Rental and Employment	DZD	1,228,500	1,621,620	1,719,900	617,760	5,187,780
	Yen	1,533,168	1,816,214	1,852,332	617,760	5,819,474
Office maintenance	DZD					
	Yen	0	0	0	0	
Local Consultant	DZD					
	Yen	0	0	0	0	
Seminars and Workshops	DZD	89,410	32,968	147,602	85,445	355,425
	Yen	111,583	36,924	158,967	85,445	392,919
Others	DZD	215,452				215,452
	Yen	268,884	0	0	0	268,884
Total	DZD	3,444,974	3,866,566	3,952,386	1,680,293	12,944,219
	Yen	4,299,326	4,330,552	4,256,717	1,680,293	14,566,888

Table 4-4 Expenditure of CRL related to the Project in 2010-2011

Item	Amount (in Dinar)
Gas and reagent	7,500,000
Repairing and maintenance of equipment	850,000
Procurement of equipment (microwave digester, multi-parameter suitcase)	2,500,000
Workshop and seminar expenses	350,000
Maintenance and repayment of cars	500,000
Communication expense	250,000
Total	11,950,000

5 Attempts and Lessons Learned

5.1 Actual Situation and Problems in the Project

For the purpose of comprehensive and basic capacity development of environmental monitoring for ONEDD as a counterpart (C/P), JICA has already carried out a Japanese technical cooperation program titled “The Project for Capacity Development of Environmental Monitoring” (hereinafter referred to as “Phase 1”) for three years starting from December 2005 until November 2008. However, since ONEDD/CRL is still at the basic level of technology, and in order to deal with the remaining issues based on the request from Algerian side, the Japanese technical cooperation program titled “The Project for Capacity Development of Environmental Monitoring (Phase 2)" (hereinafter referred to as "the Project" or "phase 2") has been carried out during the past three years starting from October 2009 until September 2012

In the course of phase 2, thanks to the proper leadership of newly appointed director general of ONEDD and untiring efforts by C/Ps personnel, the capacity on the environmental monitoring has been strengthened, as seen in increased number of laboratory staff and strengthened laboratory management with expanded analytical ability. In addition, some of issues recommended by phase 1 have been solved during phase 2. However there are still some difficult problems to be solved as mentioned follows:

(1) Problems in deteriorated laboratory facilities

The current prefabricated laboratory is a temporal facility constructed using worn out materials in June 2007 for meeting the purpose of implementation of phase 1, and the necessity of a new laboratory has been recommended from phase 1. Due to some constraints such as vibrations, dust, uncontrolled temperature and risks of contaminations, the deteriorated current prefabricated laboratory may disrupt the analytical works and pretreatment of samples that require precision control and proper maintenance of equipment (e.g. GCMS, FTIR and XRF).

In consideration of the above situation, in phase 2, JET has requested the construction of a new laboratory to the Algerian-side. Meanwhile, the Algerian-side informed that the public announcement of the bid on the study of basic design for the new laboratory related to the plan of new city construction project in Bouguezoul city (prefectural capital of Medea) has been made in October 2011. It is assumed that the construction of the new laboratory and move to the new facilities may take at least a few years, current situation of the laboratory may hamper the proper laboratory management such as maintenance of equipment.

With respect to the laboratory facilities, issues encountered in phase 2 and verification of the issues recommended in phase 1 are as follows:

- Repair work for cracked wall by the earthquake in 2003:

As indicated in phase 1, there was a fear of the collapse of cracked wall located near from the room of existing laboratory where Atomic Absorption Spectrophotometer (AAS) is installed. In order to deal with this problem, ONEDD has made a repair work of cracked wall in 2011.

- Installation of Draft Chamber (Fume Food):
  As pointed out in phase 1, installation of a draft chamber in the laboratory was required to prevent health damage due to volatile and/or toxic chemicals. In order to deal with this problem, ONEDD has installed the draft chamber in the renovated storage building in 2011.

- Air Conditioner:
  Air conditioner has already been installed in every room of the prefabricated laboratory, some of them are out of order. For prolonging life of air conditioners and analytical equipment used of analysis, regular management and maintenance are necessary.

- Expansion of Power Supply Capacity:
  Various kinds of equipment in the laboratory consume a large amount of electricity. In case of phase 2, additional equipment was purchased such as microwave oven that is used for pretreatment of samples for AAS analysis. There is an urgent issue for CRL to secure power supply at the time of power cut. Since the failure of FTIR has been caused by the failure of UPS, it is necessary to purchase UPS as a back-up power supply, and expansion of power supply should be considered.

- Clean Room:
  As also pointed out in phase 1, clean room is still not available in the current laboratory. In order to comply with requirements of an environmental laboratory to conduct microbiological analysis, installation of clean room should be considered.

(2) Problems in laboratory equipment and chemicals

Technology transfer by JET in phase 2 could make a contribution to the analysis of volatile organic compounds using GCMS and qualitative analysis using FTIR. However, existing GC has become unusable due to repairing error, further Purge and Trap (accessory of GCMS) is not available to use because there is no skilled engineer to repair this equipment in Algeria. On the other hand, inventory control related to the glass ware and chemicals had not been made satisfactorily as pointed out in phase 1. One of the main causes of the above constraints is that most equipment, apparatus and chemicals are forced to import from abroad, and the import of such commodities requires at least several months in Algeria. Therefore, even accessories and chemicals have been directly imported from Japan and France in phase 2, it took a lot of time for custom clearance because this matter is beyond the control of MATE. Such situations were entirely same as the case of phase 1.

As for laboratory equipment, periodical maintenance has not been made in CRL. In general, C/Ps has no habit to read attached user's manual of analytical equipment. Furthermore, there is no technician familiar with operating machine and electrical equipment in CRL. In order to make a proper maintenance of the laboratory equipment that might be increased in the future, CRL-ONEDD needs to strengthen the relationship with the skilled local agent in the field of

laboratory equipment. In addition, new employment of mechanic/technician in CRL-ONEDD should be considered.

(3) Problems in laboratory staff

More than seven teen (17) laboratory staff have been assigned during phase 2, and most of them have had a fundamental knowledge and experience related to their fields at the starting time of phase 2. Since those who resigned from CRL were very few during phase 2, the number of laboratory staff and their assignment were appropriate for the Project activities. However, staff having experience in a leadership position and extensive knowledge is limited in CRL-ONEDD, and important/high level work such as elaboration of the report could be entrusted only on few of the competent staff. Since there are few opportunities to share the knowledge and experience among the staff in CRL-ONEDD, it is not easy to train newly employed staff (young generation).

The shortage of fundamental knowledge as pointed out in phase 1 may be caused by shortage of the time for self-learning. Since most the staffs are not familiar with writing and reading of reports, it is not easy to develop their extensive capacity other than the analytical skill.

Thanks to the leadership of Director General of ONEDD, the managing function of CRL has been strengthened in compared with phase 1. Since the acting laboratory director in phase 1 has been promoted to the director of CRL in phase 2, his performance as a laboratory director has been more aggressive in accordance with his awareness of mission and responsibilities. As for the management of laboratory, the director of CRL has a lot of responsibility to lead daily work in laboratory operation such as control of analytical work and sampling, coordination and negotiation with the client, logistics of laboratory and others. Except human affairs and budget implementation, these are belonged to the laboratory director and performed by the direction of the director general of ONEDD, and their appropriate roles are properly shared as of the present.

As pointed out in phase 1, some staff was not punctual in the working hour of ONEDD due to child care and other reasons. This situation has been gradually improved in phase2.

(4) PC and internet environment in CRL

The internet environment has been gradually improved in Algeria compared with phase 1. The personal computers belonging to ONEDD are limited in number and some of them are old. Therefore, some privately owned note book computer is sometimes used for daily work. Since computer is an essential tool for analytical work in the sense of expansion of knowledge and information on environmental monitoring, needs of computer will be increasing greatly in the future. In order to develop the capacity of laboratory staff, appropriate distribution of note-book computer is an urgent matter.

In the course of phase 2, some computers in CRL and ONEDD headquarter were infected with computer virus due to the lack of recognition to the menace. Furthermore, it was informed that the computer connected to high advance analytical equipment has been infected by computer virus. Appropriate management and maintenance of computers as well as equipment is also urgent issue

for CRL and ONEDD headquarter.

(5) Internal training to the other regional laboratories and monitoring station

During the period of phase 2, internal training for other regional laboratories and monitoring stations has not been successively done due to lack of budget and other reasons. In order to deal with this problem, securing the budget with systematic training program by ONEDD is necessary.

5.2 Current Situation and Problems in Related Fields

(1) Environmental laws and enforcement

Since the establishment of ONEDD on the basis of "Decree No. 02-115 of 3 April 2002", laws and regulations related to the control of industrial effluents which is one of the primary missions of CRL-ONEDD have been developed steadily in Algeria. Quality of industrial effluents in Algeria has been regulated based on "Decree No. 06-141 of 19 April 2006 on setting limit values for discharged industrial effluents". According to "Decree No. 07-300 of 27 September 2007 on setting modalities of application of the complementary tax on industrial wastewater", identification of discharging of pollution required for calculation of charges (taxation) of industrial effluents is available according to the analysis of industrial effluents executed by ONEDD.

However, monitoring of industrial effluents based on the above law has not been smoothly executed due to the lack of concrete implementing method and agreement between related organizations. In order to cope with this, MATE (Minister of Land Planning and Environment) had issued the circular "370/SPM/10; 28th November 2010 on procedures of execution of the Executive Decree No. 07-300 for industrial wastewater monitoring"(see Annex 5-1). The intention of this circular was to promote joint inspection/joint effluent monitoring activities of ONEDD, Wilaya Environmental Departments and industrial units. The circular paved the way for joint monitoring and regarded as a promoting factor.

With respect to the analysis cost, payment by industrial units was stipulated in the circular according to the convention of monitoring and analysis between ONEDD and industrial units. However, DEWA did not give the necessary support to CRL-ONEDD for sampling in the industrial units. This prevented to make detailed inventories of the pollution sources.

As pointed out in phase 1, environmental water quality standards at public water body are not established yet in Algeria. In order to make an evaluation of environmental risk such as degradation of living environment and ecological system caused by the pollution of industrial effluents, environmental water quality standards is indispensable.

(2) Current situation on environmental pollution

As mentioned in phase 1, the state of pollution at public water body such as river, lake, and sea water in Algeria is very serious in a certain place due to discharging of untreated effluents from factories, domestic water and others. Especially the pollution in the El Harrach basin, where environmental monitoring has been executed as a model site of phase 2, is not improved yet, and bad smell due to the accumulated organic substances on river bed is becoming still serious problems. Although monitoring data in the Project is limited, it is still apparent that pollution in the river is not improved during the Project period. The cause of pollution is not only industrial effluents but also long-term accumulation of pollutants derived from untreated discharging of domestic water and illegal dumping along the river side. Under the above situation, there is a limit to improve the water pollution only by enforcement the regulation to the industrial units in

accordance with environmental monitoring, and drastic de-pollution measures are expected. However, accumulation of monitoring data is indispensable for effective de-pollution measures. Even under any circumstances, environmental monitoring should be continued form a long-term perspective.

(3) Environmental monitoring

Historically ANRH has been a leading organization in environmental monitoring for public water body in Algeria. Nation-wide periodical monitoring related to surface water and ground water has been executed in each water system. Measuring parameters of surface water are composed of BOD、COD、DO、NH_4、NO_2、NO_3、PO_4 and flow rate. From the view point of water resource management and pollution control, the results of monitoring has been reported with pollution map based on the water quality indicators classified with into four (4) categories (index of organic water pollution). Regarding El Harrach water system related to the Project, one (1) monitoring point has been set up at Baraki.

On the basis of regulation on bathing water, water quality monitoring at beach has been conducted with cooperation of ONEDD and related organizations. During bathing season from June to August, CRL-ONEDD has been executing the analysis of physico-chemical parameters, and monitoring points in 2010 reached eighty (80) beaches in Alger city and its neighbor Wilayas.

Other than the above monitoring, requests for analysis to CRL-ONEDD related to the pollution investigation such as port, lake and others on the basis of the Project has been increasing recently.

In the course of phase 2, environmental monitoring of river water and industrial effluents in the El Harrach basin was made periodically. Sampling for river water and sediments was executed twice a year in dry and rainy seasons. At the beginning of phase 2, implementation of industrial effluent monitoring was scheduled based on the conclusion of monitoring agreement between ONEDD, DEWA and DEWB, and inventories of industrial units offered by DEWA and DEWB.

However, environmental monitoring in phase 2 has not been always conducted as scheduled. Because the monitoring of industrial effluents was conducted only in the case that monitoring convention was concluded between ONEDD and industrial units. Furthermore, DEWA as the authority responsible for supervising industrial units did not involve in monitoring activities of industrial effluents such as sampling and site inspection. As for environmental monitoring of El Harrach, sampling and analysis has not always made as scheduled because ONEDD must bear the expenses of analysis and request of the client must be given priority to the environmental monitoring.

Even though monitoring data related to river and industrial effluents has been accumulated since phase 1, identification of the state of pollution in the El Harrach basin is still difficult due to limited monitoring data. In order to solve this problem, strengthening of cooperative relation between ONEDD and DEWA under the sufficient support from MATE is needed.

(4) Inadequate disclosure of environmental information

As pointed out in phase 1, progress in disclosure of environmental information is still slow in Algeria, as well as the monitoring results of public water bodies executed by ANRH. With respect to the sea bathing monitoring, ONEDD has not been informed the data in coliform analysis that was made by other institute. The disclosure of environmental monitoring results in the governmental organizations does not take place in general, and very less or no information has been disclosed to the public so far. In the course of phase 2, even though JET has requested repeatedly ONEDD to disclose the report of monitoring results of El Harrash basin based on comprehensive interpretation and evaluation of risk to the relevant organizations, no concrete action has taken until now. In consideration with this situation, development of legal framework in disclosure of environmental information is necessary in the future.

(5) Coordination among relevant organizations

In recent years, opportunities in relation to environmental issues required environmental monitoring has been increasing in many organizations in Algeria. Furthermore, specific project dealing with international or borderless environmental issues that includes environmental monitoring has been gradually increasing in Algeria. Under such circumstances, opportunities for CRL-ONEDD to participate in the above project, as a leading laboratory in environmental monitoring of Algeria, have been increasing. However, due to hands full by requested analytical works in CRL-ONEDD, close relationship between CRL-ONEDD and relevant organizations has not made so much. In consideration with the role and function of ONEDD as a national leading organization to deal with environmental issues, not only laboratory analysis but also close relationship and coordination among relevant organizations would be more important in the future.

5.3 Attempts for Smooth Implementation of the Project

In consideration the issues mentioned in 5.1 and 5.2, following measures and attempts for effectiveness, achievements, relevance and sustainability of the Project were taken by ONEDD and JET as stated bellow:

(1) Attempts and efforts by ONED

Since fourteen (14) technical staff have been assigned at the end of phase 1 in 2008, the number of technical staff in phase 2 have been always keeping with at least seventeen (17) for all the duration of the Project. In addition, most staff trained in phase 1 have been continuously employed in phase 2 so that the problem such as drain of human resources has not happened in the Project. According to the base-line study related to the capacity of CRL at the beginning of phase 2, choosing of suitable technical staff in the field of advanced analytical technique for three (3) equipments has been made smoothly. On the other hand, most staff related to the field of data interpretation at ONEDD headquarter were transferred in the halfway of phase 2 due to various reasons. In order to deal with this situation, new staffs were assigned at the second half of phase 2 so that training of comprehensive interpretation and evaluation of risk could be made without trouble. In view of the above, the number of C/P was sufficient for project implementation so that the technology transfer program has been made efficiently implemented. Such efforts for the above mattes are admirable.

Newly appointed director general of ONEDD in 2009 has brought a great change in the laboratory management of phase 2. The director general has made efforts to visit CRL as far as circumstances permit so as to give detailed instruction to the laboratory director and all other staff. Such frequent visits of the director general have made a positive impact on motivation of laboratory staff and increased communications to raise productivities at CRL. Furthermore JET could have a close communication timely with him. On the other hand, direct coordination (command) by the director general to meet requirements of the client and daily analytical works has led a large increase of analytical results. Such efforts are also admirable.

(2) Dispatch schedule of JET

Assignment of four (4) experts has been focused on the first half of phase 2. This could enable to execute various kinds of activities such as adjustment and repair work for three (3) equipment and procurement of necessary accessories in advance, furthermore this also could enable to avoid the delay of technology transfer caused by failure of equipment and lack of accessories. In considering continuity of the training and motivation of staff, sea bathing monitoring in summer and in comply with the request of CRL-ONEDD, dispatch schedule of JET was modified and implemented to minimize the absence period of JET.

(3) Measures in the absence period of JET

During the absence period of JET, lowering of motivation and negligence in disseminating the

acquired skill were anticipated. In order to deal with this matter, JET prepared a recommendation letter, in which tasks to be done by C/Ps during the absence period of JET were pointed out (see Annex 5-2). The recommendations have been confirmed in the meeting between CRL-ONEDD and JET at the end of work period of JET in Algeria every time. The execution of their tasks based on recommendations by JET has been checked as well in the next work period of JET in Algeria.

(4) Periodical meeting between CRL staff and JET

Before starting of phase 2, periodical internal meeting has not been held at CRL, and only the meeting according to the request from the laboratory director or the director general of ONEDD has been held irregularly. In order to develop QA/QC and exchanging information at CRL, regular meeting attended by all staff have been organized every Sunday morning since July 2010 in phase 2, and general tasks at CRL as well as QA/QC has been discussed in the meeting. From July 2010 until July 2012, in total twenty-nine (29) regular meetings related to QA/QC held, and the number of average attendants for one meeting reached about thirteen (13) (one hundred-thirteen (113) in total) (see Annex 2.3.7). Since eighteen (18) regular meetings were held during the assignment period of JET, gradually it has become a regular habit at CRL. Major topics in the meetings were weekly reports and QA/QC (SOPs based on GLP), report on sea bathing inspection, safety control, human affairs and requirements of consumable materials and so on. Other than the above, it should be noted that some presentations related to QA/QC seminar in Oran/Constantine and report of international training program in Greece by the laboratory director have been made in the regular meeting. In view of the communication at CRL, the regular meetings have been favorably received by C/Ps, even though some arguments caused by strong requests from the staff to the administrator happened occasionally. In consideration of the above situation, JET has recommended to continue the regular meetings even after the Project.

(5) C/Ps **training at small group**

Trainees for three (3) equipments such as GCMS, FTIR and XRF were selected as a small group composed of a few trainees on the basis of the base-line study on human resources and state of equipment at the beginning of the phase 2. From the viewpoint of efficiency and effectiveness of training, the small group could have advantages to avoid the trouble due to the absence and/or resignation of any trainee, and to encourage their responsibility and team work.

(6) Encouragement of record and documentation management

From the view point of sustainability of the laboratory after the Project, QA/QC and ensuring of traceability are indispensable. Through the OJT and instruction by JET in phase 2, record of logbook related to analytical work and documentation management has been started by C/Ps. Especially operational records (logbook) on three (3) equipment of advance analytical technology were useful not only for appropriate maintenance of equipment but for identification of analytical procedures in the absence period of JET. As for documentation management of the laboratory, the

method based on GLP has been introduced to CRL. That is to say, documents produced by activities of the laboratory were classified according to the item number of GLP, and they were controlled centrally and universally with the format by fixed form (see Annex 2-3-1). For example, this also includes Standard Operational Procedures (SOPs) based on GLP, client's lists of agreement for environmental analysis, and client's lists of laboratory analysis and the safety protocol. These documents are indispensable for the laboratory management as well as QA/QC, and it is expected that these documents will be updated by C/Ps even after the Project.

(7) Elaboration of documents written in French by C/P

Some of C/Ps is good at English so that JET could communicate with them even in the training except in case of some complicated subjects. However their willingness to read books and website in English are not so strong in general and their opportunities to learn English are still limited. On the other hand, most of JET is not good at French. Under such situation, JET has made an effort to communicate with C/Ps directly in English or French as much as possible without only relying on interpreters. Since C/Ps has not accustomed to write a report even in French so far, JET has urged C/Ps to write all the products related to the Project activities in French.

(8) Encouragement of presentation by C/P

Through the implementation of technology transfer aimed to achieve each output of phase 2, opportunities to present what C/Ps had learned in their specific field were provided to C/Ps in the seven (7) times of seminars and three (3) times of joint seminars and workshops. Since basic skill for the presentation has been already acquired in phase 1, their capacity related to the presentation skill has been strengthened further in phase 2.

(9) Safety management for laboratory staff and maintenance of equipment

From the viewpoint of safety management for the laboratory staff and appropriate maintenance of laboratory equipment, following measures have been taken by ONEDD and JET in phase 2:

- In order to avoid the contamination derived from outdoor shoes, use of sandals was introduced to enter the prefabricated laboratory.
- In order to avoid an accident resulting from ingesting of VOC and acid gas, draft chamber was installed in the renovated stock house by ONEDD.
- White robe for analytical works, acid resistance gloves and gas mask have been purchased.
- In order to avoid the contamination of analytical equipment due to analytical sample itself, JET has made a request to pay close attention in case of handling of highly concentrated analytical sample.

(10) Strengthening of relationship between CRL-ONEDD and local agents

In order to maintain the analytical equipment properly and to avoid constraints of analytical works, relationship between CRL-ONEDD and local agent/supplier of equipment and materials should be

strengthened in the future. In addition, consumable materials such as chemicals, gases and accessories consumed in daily analytical works should be provided to CRL timely and definitely. Therefore, JET has urged CRL-ONEDD to establish good communication between CRL-ONEDD and local agents as much as possible.

(11) Communication with ONEDD headquarter

From the experience in phase 1, difficulty of communication with ONEDD headquarter has been feared at the beginning of the Project. Because ONEDD headquarter is located about a half hour's drive from CRL, it was assumed that JET would be under physical constraint in daily communication with general director and staff in charge of data interpretation. However, the director general of ONEDD has visited CRL twice a week at least and staff of data interpretation has also accepted to JET's request for the training in CRL so that most of the activities in phase 2 has been executed timely. Such efforts and good coordination by the director general are to be highly praised.

(12) Enhancement of internal information sharing

With regards to information sharing between JET and C/Ps, following measures were taken:

- In case the absence of the director general of ONEDD, JET always gave a message to the director of CRL so as to avoid the delay of making decision on the program.
- JET has made efforts to communicate directly with C/Ps in English or in French as much as possible, so that some of C/Ps could improve their English speaking capacity.
- Through the regular meeting in every Sunday morning, information sharing between C/Ps and JET has been improved.

(13) Exchange of information with external institute

In the sense of sharing information related to the environmental monitoring, exchange of information with external organizations is indispensable for CRL-ONEDD. In that sense, CRL-ONEDD and JET have made an effort as much as possible to communicate with related external organization such as DEWA, DEWB, ANRH, GTZ, USTHB, IAEA, and so on.. Further, in order to diffuse and exchange the knowledge and experience related to the results of the Project, JET and C/Ps staff had often visited regional laboratories such as Oran and Constantine and the monitoring station in Annaba in phase 2. By way of the most effective way in information sharing with external organizations, joint seminars and workshops could help creating an opportunity for discussing the related field among various stake holders.

5.4 Recommendations to Algerian Side

Since the achievement level in phase 1 (from December 2005 to November 2008) was as basic level in capacity of environmental monitoring, further capacity development to the following subjects was made in phase 2 (from October 2009 to September 2012).

- Capacity development of advanced analytical technology for GCMS, FTIR, and XRF
- Capacity development for comprehensive analysis based on the monitoring results and its reporting
- Introduction of QA/QC system that can provide reliable data to customers
- Dissemination of environmental monitoring technologies possessed by CRL to other ONEDD regional laboratories, monitoring stations and other relevant organizations.

As a result of activities of phase 2, the Project purpose which is "ONEDD's capacity to generate environmental information for effective environmental management including inspection, enforcement and pollution prevention is strengthened." was mostly achieved. However, if CRL-ONEDD desires the sustainability of current level of achievements, efforts for further enforcement of environmental monitoring including effluent monitoring, which requires coordination among stakeholders, are needed. In addition, the above subjects were not fully achieved in some ways in phase 2, further efforts by Algerian-side is expected.

Recommendations to the Algerian-side have been reported by JET in each work period in Algeria, and Algerian-side has made a lot of efforts for their solution in some ways. These past recommendations also include some issues needed to be dealt with in the future (see Annex 5-2).

In order to secure sustainability and further capacity development of CRL-ONEDD, JET would like to make following recommendations:

(1) Construction of new laboratory

The largest constraint for efficient implementation of the Project was the delay of new laboratory construction. Laboratory facilities of CRL are consisted of following buildings.

- Prefabricated temporal laboratory which was built in 2007
- Old laboratory which was built more than thirty years ago
- Old temporal laboratory（for pre-treatment of analysis sample）which was renovated in 2011
- Two old office buildings which were built more than thirty years ago

These laboratory facilities which are located at a short distance each other, may become a factor of constraints for sustainable activities of CRL after the Project from following reasons:

- Inefficiency and danger due to hand-carrying of samples for analysis and reagent between

laboratory facilities

- Possible machine trouble of analytical equipment due to vibration of floor in the prefabricated temporal laboratory and declined floor of old laboratory
- Lack of proper water supply and drainage system
- Difficulty of temperature control for analytical equipment and samples
- Contamination of analytical equipment and samples by dust and sample itself
- Lack of enough work space for smooth analytical works
- Possible accidents may caused by fire and handling of dangerous substances due to difficulty in appropriate placement of equipment in the small work space
- Lack of sufficient air intake and exhaust system

The lack of proper laboratory has been already pointed out in phase 1. This situation in phase 2, other than the above, also limited certain advanced chemical analysis for toxic organic chemicals (organic-chlorine, pesticide, insecticide, etc.), and affected the condition of analytical equipment. Therefore, it is strongly recommended that ONEDD would continue its effort for construction of new laboratory.

(2) Laboratory management based on GLP

All the laboratory facilities including current prefabricated temporal laboratory have negative effect on analytical equipment procured in phase 1. During the Project period, delay of development of fragile laboratory infrastructure made some interference to the technology transfer that requires accurate chemical analysis and safe work environment. However, as the laboratory management based on GLP (Good Laboratory Practice) has started in this difficult situation, a certain result was brought in phase 2. Although some difficulties are anticipated till the construction of new laboratory, JET would like to make following recommendations for appropriate laboratory management.

- Capacity development for QA/QC should be continued based on GLP. Especially SOPs based on GLP needs to be rigorously applied in the actual analytical works, and C/Ps should make continuous efforts to develop SOPs by themselves.
- In order to improve the precision in analytical results, repeated training using standard reference materials is effective. In some cases, proficiency testing using the known standard sample in cooperation with the external laboratory shall be considered from the view point of QA/QC for CRL.
- Taking every opportunity, CRL-ONEDD should inform the clients about the reliable of CRL's data based on SOPs and proper QA/QC system. The client will realize the importance of environmental monitoring through providing reliable data.
- With regard to laboratory documents, documentation management of GLP has made from

view point of QA/QC and proper laboratory management. In order to secure QA/QC and traceability in CRL, it is necessary for CRL to make a continuous effort to revise these documents and secure the traceability

- Because the samples used for analysis in CRL is mostly highly-concentrated samples such as industrial waste water and sediments, unexpected results of analysis are not uncommon. This may cause contamination of equipment (e.g. mercury analyzer) or damage human health. In order to deal with this kind of accident, CRL needs to investigate background information of samples before the analytical works. In case that unexpected value was obtained, re-sampling and re-analysis should be considered.

- In order to avoid the health hazard in the laboratory, where highly concentrated pollutants are treated, it is recommended that masks/dust respirators and gloves should be equipped in the laboratory.

- Regular meeting has been held twenty-nine (29) times for two years in phase 2. This meeting was very effective for strengthening of information sharing among all staff of CRL. It is expected that the weekly meeting at CRL will be held continuously so as to provide an opportunity not only for QA/QC but for sharing of knowledge and technology to CRL staff.

- Staff of CRL, regional laboratories and monitoring stations should strengthen the skills through training and internal/external seminar. In that case, enhancement of relationship with other analytical laboratory and invitation of external human resource should be considered.

(3) Securing the periodical maintenance of analytical equipment

Even under the situation of fragile infrastructure of laboratory facilities, keeping the good condition of analytical equipment is essential for continuing environmental monitoring activities. In particular, the advanced analytical techniques using GCMS, FTIR and XRF require appropriate maintenance of equipment. JET would like to make following recommendations for maintenance of analytical equipment. Since the same can be said of all other equipment in CRL, periodical maintenance based on the bundled manual has not been made so far. It is quite common that equipment has been used until out of order. ONEDD should secure necessary budget for the periodical maintenance of equipment, and keep its effort for seeking maintenance supports from the engineering firms.

- Due to the difficulties of repair work for Purge and Trap (accessory of GCMS), analysis on VOCs including BTX has not been made more than half a year in phase 2. Since the repair work of this equipment is not available in Algeria, ONEDD shall ask the engineer from abroad (Produced by Teledyn Tekma) to repair it as soon as possible.

- With regard to the analysis of PAH using GCMS, in order to secure the acquired technique, practicing repeatedly using standard and real sample is necessary.

- Since the data library software of FTIR was procured in phase 2, training by JET on the use of this software and analysis using real sample has not been made sufficiently. For effective development of the qualitative analysis for non-volatile organic compound and inorganic

pollutants in various samples using FTIR, it is necessary for C/Ps to practice (self-training) repeatedly using known sample and to expand knowledge and experience in this field. In addition, in order to avoid failure of FTIR, UPS shall be purchased as a backup power supply.

- Training on inorganic chemical analysis using XRF has successfully conducted in phase 2. Analysis by XRF should be made focusing on sediment sample that contains high concentration of heavy metals, and it will be expected effective use of XRF together with analysis by AAS.

(4) Comprehensive interpretation and evaluation of risk on pollution of El Harrach

Environmental monitoring in the El Harrach was initiated in 2004, and its frequency is twice a year in average. Since then the periodical monitoring are taking place at the representative points among seven-teen (17) points in total, it is still difficult to identify the change and the tendency in water quality and the spreading of pollution in the river. On the other hand, the monitoring of industrial effluents has started in 2005, and forty-seven (47) industrial units have been inspected as of 2011. This covers about thirty (30) % of industrial units indicated in the inventory of industrial units, and actual number of industrial unit in the El Harrach basin is estimated more than five hundred (500). As a result of the above situation, comprehensive interpretation and evaluation of risk in the model site were not successfully made due to insufficient data in quality as well as in quantity, and other reasons. Therefore, JET would like to make following recommendations:

- It is necessary to continue the environmental monitoring of El Harrach twice a year at least.
- From the viewpoint of water quality control, it is recommended to continue the industrial effluents monitoring and to increase the number of target industry units.
- CRL-ONEDD should strengthen the system of industrial effluents monitoring including on–the –spot inspection of industry units with the involvement and cooperation of DEWA and DEWB.
- For the smooth implementation of industrial effluents monitoring, ONEDD should ask DEWA and DEWB to promote the convention between ONEDD and industrial units.
- Only a few measurements of flow rate in El Harrach and industrial effluents has made in the environmental monitoring so far. Therefore, it is recommended to measure the flow rate of El Harrach with cooperation of NARH, and ONEDD should ask industrial units for the data of flow rate with cooperation of DEWA and DEWB.
- With reference to the results of processing of the data base (monitoring inventory by Excel file) elaborated by activities in phase 2, it is necessary for ONEDD staff to observe a monitoring and accumulation (up-dating) of measured data from the viewpoint of water quality control.
- With regard to the evaluation of risk on pollution, ONEDD should make efforts to collect various kind of information related to the context of industrial effluents, other than monitoring data. Because it is difficult to identify the state of pollution by using only monitoring data, in addition, data itself may show a wrong interpretation from scientific point of view,.

- It is recommended that CRL-ONEDD to investigate the state of industrial effluents (sampling point). In addition, CRL-ONEDD should investigate possible pollution sources such as solid waste along the river side and inflow of domestic effluents with cooperation of DEWA and DEWB. The above information should be stored in the database and prepared an inventory by ONEDD' staff.

(5) Measures to enhance the human resources of ONEDD headquarter and CRL

Because some of the data management staff in ONEDD headquarters were transferred to MATE and other section in the first half of phase 2, acquired knowledge and experience in terms of data interpretation in phase 1 was not accumulated which hampered the activities of comprehensive data interpretation and evaluation of risk in phase 2. In order to deal with the above situation, new staff were assigned to ONEDD headquarter and trained by JET in the second half of phase 2. However, their capacity including knowledge and experience in the field of data management and reporting for environmental monitoring has not always reached to a satisfactory level. On the contrary, as far as human resources of CRL is concerned, there are some staff who are good at data management and reporting related to environmental monitoring other than analytical works. In consideration of the above situation, it is recommended that the strengthening of human resources is necessary. In order to raise the ability of environmental laboratory comprehensively, following additional employment is an urgent matter for CRL:

- Researchers or engineers in the field of environment with much experience and with report writing skills, and at least with a qualification of master's degree.
- Mechanical engineer for repair and maintenance works of equipment with experience of electrical engineering.
- Experienced administrator for management of document and accounting works with ability of instruction.
- I.T. technician for operation of computer and database

It is not so easy for analytical engineers or assistant administrators to handle the above tasks concurrently. Since the work range (category) is classified clearly in Algeria, a range of experience and knowledge about the job is relatively narrow compared with Japan. Even though their potential is at high level, under the poor habit of self-learning and the specific work-environment、employees do not share the knowledge and experience among themselves, which hampers their improvement. This is due to lack of incentive such as a raise in salary and promotion in the administrative organization. While the number of highly educated employees in the administrative organization has been increasing in Algeria, opportunity to improve their ability is limited. Although seminar and training program has been increasing, opportunity to learn job skill is limited other than OJT where more effective and practical training is available.

(6) Dissemination of knowledge and skills

Knowledge and skill acquired by ONEDD/CRL in phase 2 are not only advanced technologies of chemical analysis but also techniques and experience accumulated though the routine work including laboratory management and SOPs based on GLP and the comprehensive interpretation of monitoring results, and others. In order to disseminate the acquired knowledge, experience and skill to other ONEDD regional laboratories and monitoring stations, more practical and systematic program such as a long-term OJT at CRL should be considered. For capacity development of the trainer, increasing the opportunity to participate the external training program would be more effective other than self-training.

(7) Budget allocation and strengthening of regional laboratories and monitoring stations

Since analytical equipment in the regional laboratories and monitoring stations are very limited, quite a few numbers of parameters for environmental monitoring are available in compared with CRL. For example, only one-set of the multi-parameters water quality probe has been used in each regional laboratory so that the in-situ test is not available in case of failure of equipment. Regional laboratories and monitoring stations also suffer from chronic shortages of reagents, accessories for instrumental analysis. Furthermore, they have no means of communications and work environment such as computer with internet infrastructure, printer, telephone and facsimile. Under the above poor situation, it is very difficult for CRL-ONEDD to disseminate knowledge and experience acquired by the Project. Even though the procurement of basic equipment including chemicals is an urgent matter for the regional laboratories prior to the training, ONEDD could not meet this requirements so long. In order to strengthen the capacity of environmental monitoring for regional laboratories and monitoring stations, it is strongly recommended to secure/allocate budget for maintenance of instruments and training activities.

5.5 Lessons Learned

(1) Procurement of chemicals and accessories

In the course of phase 2, accessories and chemicals required for advanced analytical technology were procured from Japan and France. In case of purchase in Algeria, it would have been difficult to obtain them on time with guarantee. In case of implementation of a project related to environmental laboratory, sufficient investigations on the supply system and the ability of agents to procure consumable materials such as reagents, chemical accessories and gases are necessary. In addition, sufficient stock for at least one year should be secured so as to avoid disruption in analytical works and activities of the Project due to unavailability of materials. It is strongly noted that smooth procurement of chemicals and accessories should be the precondition for the Project of this kind or similar projects.

(2) Development of laboratory infrastructure

In most cases of technical cooperation project for enforcement (capacity development) of the environmental laboratory, with dispatching of Japanese experts, JICA provides necessary equipment and apparatus including reagents. Counterpart organization provides laboratory facilities such as buildings and required expense for logistics. However, the delay of development of laboratory infrastructures and lack of required logistics even during the Project can be seen not only in Algeria but also in other countries. Any delay of development of laboratory infrastructures, failure of equipment and induction of accidents are not only the matter of concern, but this situation may become a factor to hinder the practice of acquired technologies by the project.

In case of the similar project as enforcement of the analytical laboratory, and/or relocation of new laboratory, identification of basic design of the laboratory is very important. As learned from the Project, it is strongly noted that the Project should not be started until the completion of laboratory infrastructures and purchase of necessary reagents, chemical accessories, gases etc. are confirmed. In addition, selection and introduction of the analytical equipment should be made stage by stage considering the capacity of the C/P's.

(3) Total ability of laboratory and human resources for laboratory management

The capacity development only for the analytical engineers through transfer of technology is not enough for achievement of the Project goals. In general, human resources in the analytical laboratory are mostly composed of analytical engineers with only few administrative staff. This has caused the back-up system required for laboratory management very fragile in most cases and ultimately weakened the total performance. Such an imbalanced assignment of human resources in an analytical laboratory may hinder the daily analytical works as well as other activities of the Project. When a similar kind project is to be implemented, it is necessary to consider the availability of enough and balanced human resources required for laboratory management.

(4) Dispatch schedule of Japanese experts

In the course of phase 2, the total assignment of JET was not enough; moreover, short-term dispatch (one month in average) has been repeated. Under such circumstances, in order to avoid the delay of technology transfer even in case of absence of JET, activities of the Project has been conducted with the best management and coordination. However, JET could not always give enough instructions of each analytical technique to C/Ps due to limited time for one to one guidance. Furthermore, as a matter of fact, CPs have been doing analysis in their own ways, therefore, it is impossible to say that the most engineers could receive sufficient instructions for each procedure of analysis from JET. The limits related to transfer of technologies in the field of analytical laboratory were experienced from the Project. Therefore, when a similar project is planned in the future, even a few (2 or 3 persons) engineers who can instruct detailed procedures of analysis should be dispatched for long-term.

(5) Ways of organizing seminars and workshops on analytical technique

Seminars and workshops can help C/Ps to improve the level of technology, disseminate the acquired technology, and share know-how and experience. There are also certain advantages for C/Ps to have extensive and general knowledge especially in the field of environmental monitoring of various kinds of pollutions with their regulation and control. The participation in seminars and workshops will also lead C/Ps to stimulate their curiosity, and strengthen their incentives. However, with respect to the field of analytical laboratory required a very high level of professionalism, so far only a few seminars hosted by universities and related research institutes in Algeria. In recent years, ways of organizing seminars and workshops are slightly changing, small and internal seminars with only working-level participants has been increasing instead of one-sided presentation with limited discussion in the big hall as before. Considering the original purpose of seminars, cost and its effectiveness/efficiency, this changing tendency may be more rational in the present day. Therefore, the internal seminars specialized for "analytical techniques for analytical engineers of analytical laboratory", which was different from the seminars and workshops repeatedly organized so far in the field of "environment", has been held several times in the course of phase 2. Unfortunately the attendants from other laboratories were very few in such kind of seminars. As far as analytical laboratory and analytical equipment are concerned in Algeria, the number of engineers in this field is limited due to its high level of professionalism. In consideration of the above situation, it is necessary to promote the interaction of specialized engineers in this field beyond the administrative agencies, and it is also recommended to increase the opportunities for small and specialized seminars and workshops. The ways of organizing seminars as mentioned above should be considered in the similar projects.

6 Modification of PDM

Modification of PDM in this project can be summarized as follows:

(1) Original PDM-1 in March 2009 (refer to Annex 6-1);

(2) Modified PDM-2 in November 2009 (refer to Annex 6-2);

(3) Modified PDM-3 in April 2011 (refer to Annex 6-3).

In the modified PDM-2, followings are added. This added PDM was effectively implemented in the Project.

- Objectively Verifiable Indicators for Output3:
  1. More than 16 staff in CRL work for quality control for inorganic/organic/microbiological analysis.
  2. More than 16 staff in inorganic/ organic/ microbiological analysis section in CRL joined trainings on quality control.

In the modified PDM-3, followings are added and modified. This modified PDM was effectively implemented in the Project.

- Objectively Verifiable Indicators of Project Purpose:

  4. Number of contract on industrial wastewater monitoring is increased.

- Objectively Verifiable Indicators of Output4:
  2. Training plan for regional laboratories and monitoring stations is developed.
  3. Training courses for regional laboratories and monitoring stations are conducted by twice a year.
  5. The workshops for regional laboratories are held as a dissemination of Project contribution.
- Activities for Output4:
  2. ONEDD develops the plans for supporting regional laboratories and monitoring stations under the support of JET.
  3. ONEDD organizes training courses for regional laboratories and monitoring stations under the support of JET.

7 Records of JCC

Joint Coordinating Committees (JCC) held in the course of the Project is shown in Table 7-1.

Table 7-1 Record of JCC

Date	Subject	Minutes
2009		
October. 20	Discussions with MATET-ONEDD, DEWA and DEWB on project in 1st year (Japanese Project Consultation Team)	Annex 7-1
November 11	Discussions with MATET-ONEDD, DEWA and DEWB on project in 1st year (Japanese Expert Team)	Annex 7-2
2010		
June 22	Discussions with MATET-ONEDD, DEWA and DEWB on project in 2nd year (Japanese Expert Team)	Annex 7-3
2011		
April.13	Discussions with MATET-ONEDD on project in 3rd year (Japanese Project Consultation Team)	Annex 7-4
October. 29	Discussions with MATET-ONEDD, DEWA and DEWB on project in 3rd year (Japanese Expert Team)	Annex 7-5
2012		
February 21	Discussions with MATET-ONEDD on terminal evaluation of the Project (Terminal Evaluation Team)	Annex 7-6
July 15	Discussions with CRL-ONEDD on completion of the Project (Japanese Expert Team)	Annex 7-7

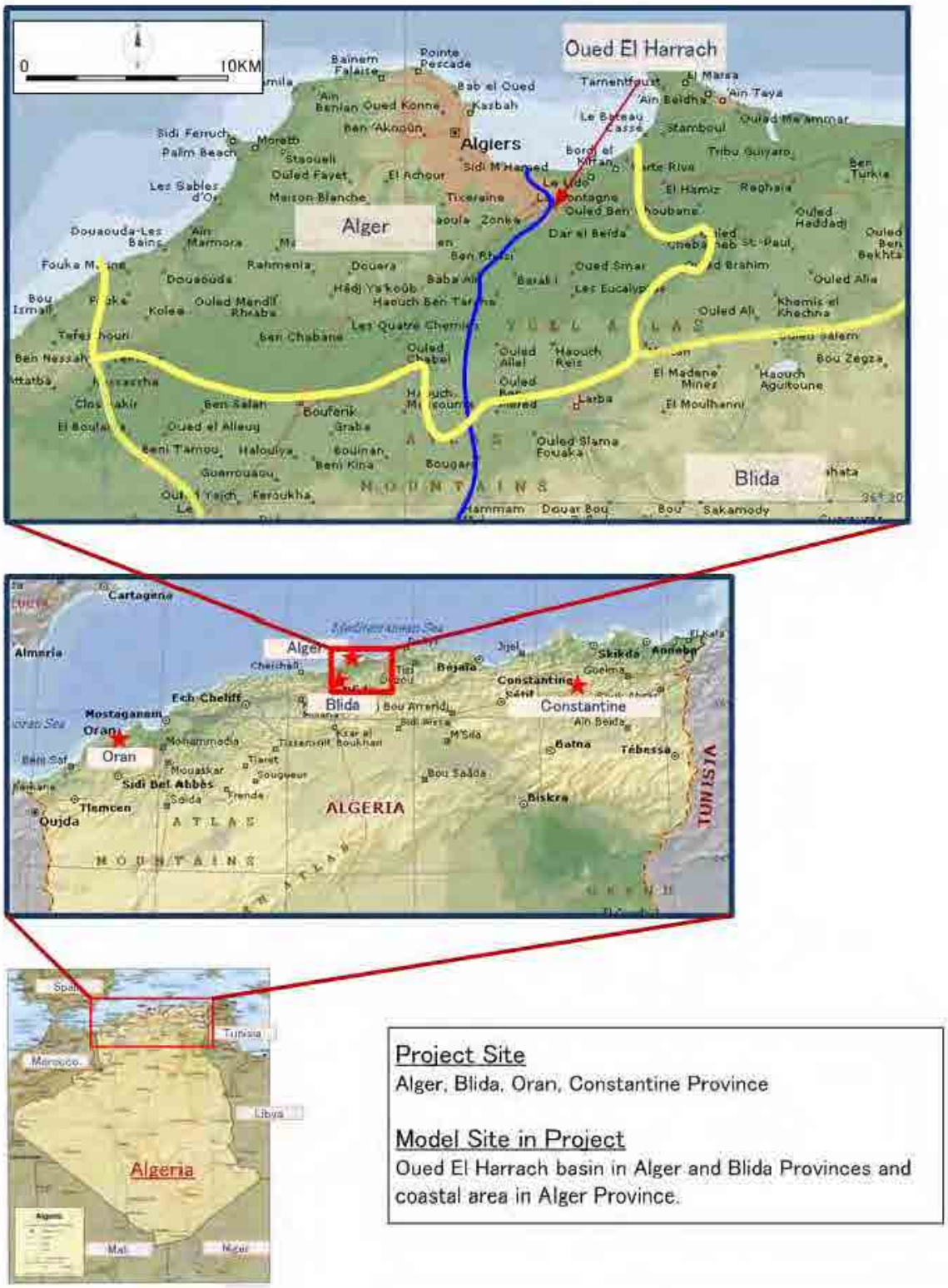
8 Report elaborated by Algerian Side

Report of Algerian side is shown in Annex 8.

ANNEXES

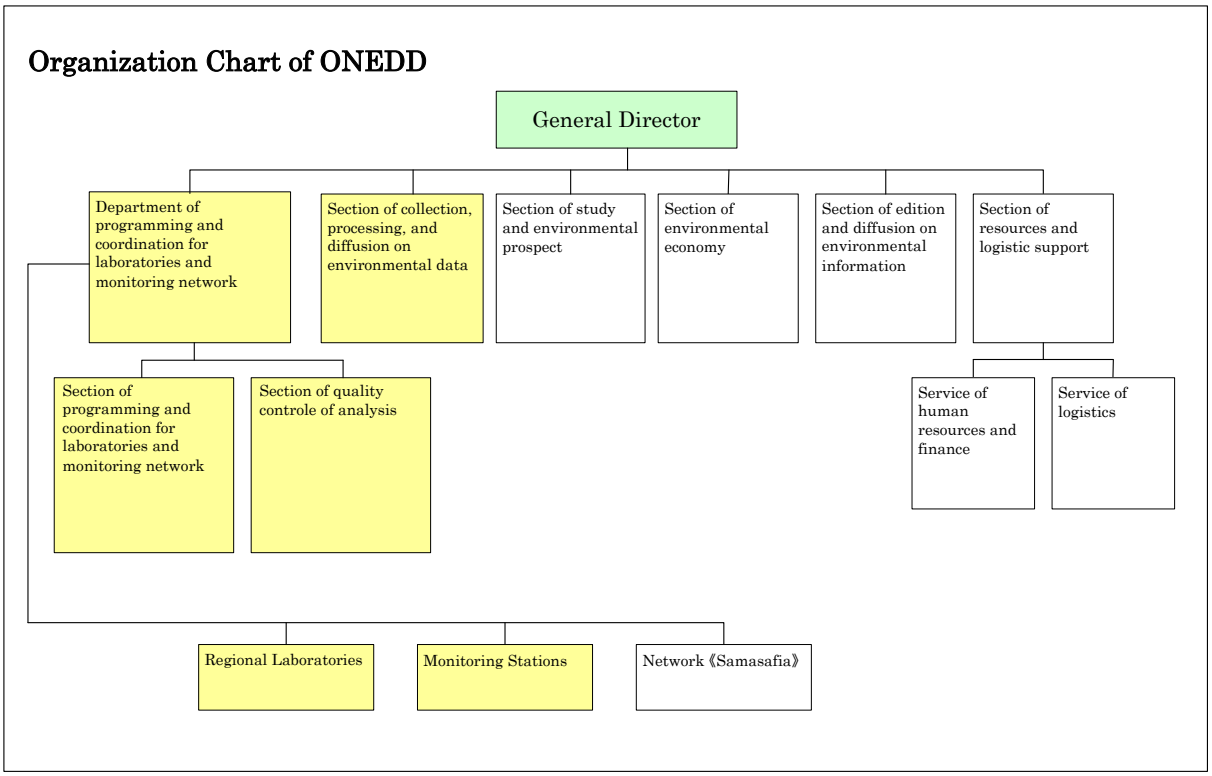
Annex 1　Outline of the Project

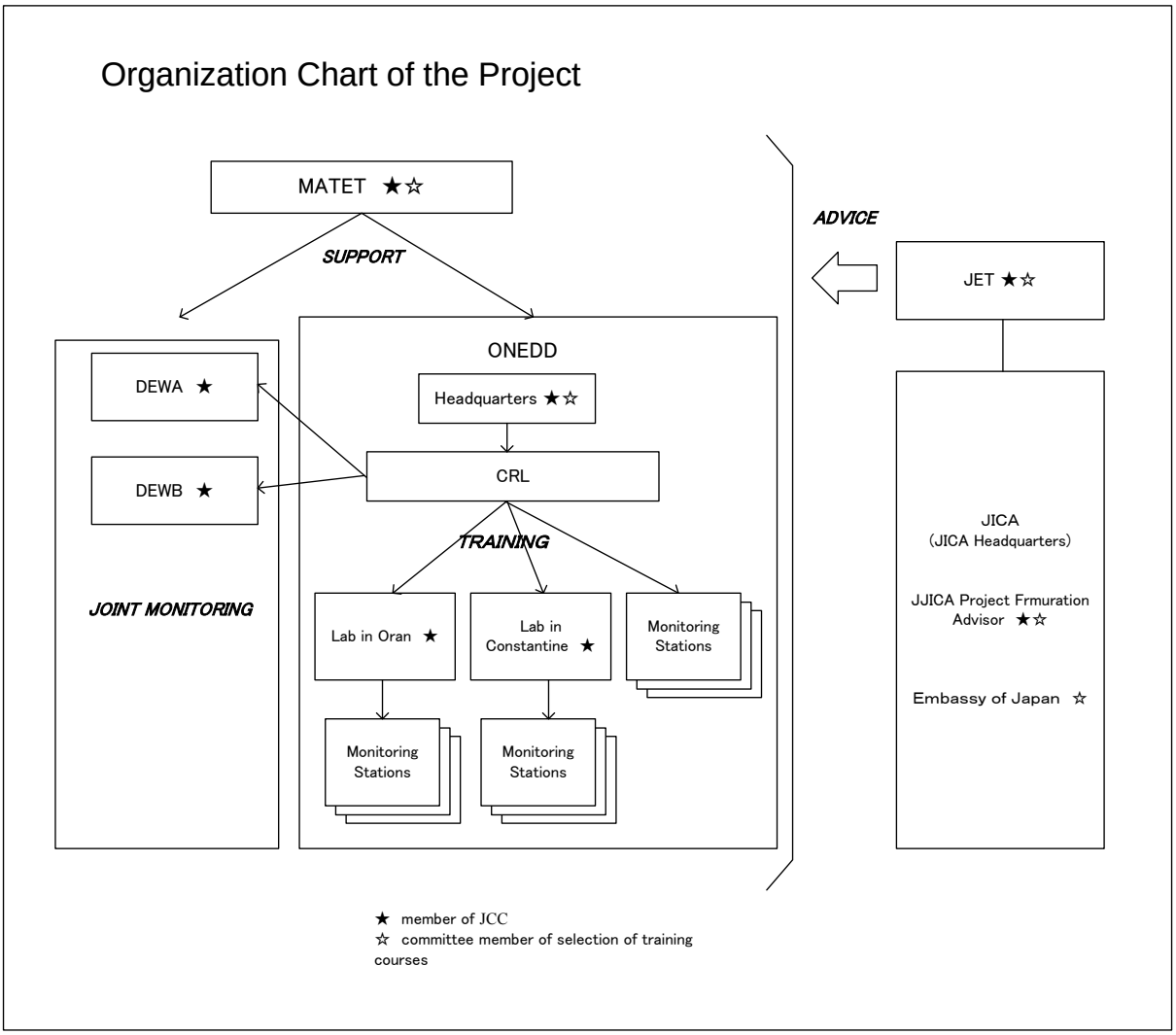
Annex 1-1 Location map



Location Map of the Project Area

Annex 1-2　Organization chart


Organization Chart of ONEDD
General Director
Department of programming and coordination for laboratories and monitoring network
Section of collection, processing, and diffusion on environmental data
Section of study and environmental prospect
Section of environmental economy
Section of edition and diffusion on environmental information
Section of resources and logistic support
Section of programming and coordination for laboratories and monitoring network
Section of quality controle of analysis
Service of human resources and finance
Service of logistics
Regional Laboratories
Monitoring Stations
Network《Samasafia》


Organization Chart of the Project
MATET ★☆
SUPPORT
ADVICE
JET ★☆
ONEDD
Headquarters ★☆
DEWA ★
DEWB ★
CRL
TRAINING
JOINT MONITORING
Lab in Oran ★
Lab in Constantine ★
Monitoring Stations
Monitoring Stations
Monitoring Stations
JICA
(JICA Headquarters)
JJICA Project Frmuration Advisor ★☆
Embassy of Japan ☆
★ member of JCC
☆ committee member of selection of training courses

Annex 1-3　List of Algerian counterpart personnel

	Name	M/F	Position	Organization		JICA project								Field (Output) in charge				Equipment in Charge		Training in Japan	Assignment		Experience			
				CRL	ONEDD	JICA		CD phase 1			CD phase 2										Assignmnet in CRL/ONEDD	Transfer/ Resignation	Organic	Inorganic	Analysis	Microbiology
						2004	2005	2006	2007	2008	2009	2010	2011	2012	1	2	3	4	Phase 1	Phase 2						
1	Moali Mohamed	M	Director of Labboratoy	●											●	●	●	●	Lab. management		2007 ONEDD	2001.01	-	○	○	○	
2	Houas Omar	M	Engineer	●											●		●		SAA	XRF	2004 MATE	1989.06	-		○		
3	Lakhdari Mohamed	M	Engineer	●												○	●		Sampling		2007 ONEDD	1987.03	-			○	
4	Nechaouni Leila	F	Engineer	●											●		●		UV, FTIR	GCMS	2006 MATE	1991.11	-	○			
5	Smai Mohamed	F	Technician of Laboratory	●													●		Sampling			1989.06	-			○	
6	Anane Radhia	F	Engineer	●											●		●		Kjeldahl, CN	FTIR		1990.05	-			○	
7	Tibeche Amel	F	Engineer	●													●		GC, DCO			2006.05	-			○	
8	Bensouilah Ouahiba	F	Engineer	●											●		●		GC, DCO	FTIR		2007.03	-	○			
9	Djoghlaf Hadda	F	Engineer	●											●		●		DBO,TOC	XRF		2007.07	-			○	○
10	Azouani Sophia	F	Engineer	●											●		●		SAA	XRF	2010 ONEDD	2007.1	-		○		
11	Mebrek Hanifa	F	Engineer	●											●	●	●		Microbio.	XRF	2010 ONEDD	2007.11	-			○	○
12	Kimri Leila	F	Engineer	●											●		●		GCMS FTIR	GCMS		2008.04	-	○			
13	Guerfi Lynda	F	Engineer	●											●		●		SAA	XRF	2011 ONEDD	2008.01	-		○		
14	Bouadi Fatima Zohra	F	Engineer	●											○					FTIR		2008.02	Resign (Mar 2010)			○	○
15	Daouadji Nassima	F	Engineer	●													●				2010 ONEDD	2009.04	Resign (Apr 2011)			○	○
16	Kamel Nawel	F	Assistant of Administration	●													●					2008.03	-				
17	Assia Chatal	F	Engineer		●											○						2005.09	-				
18	Abdallah Ahlem	F	Assistant of Administration	●													●					2009.03	-				
19	Salima Oussalem	F	Engineer		●											○						2002.12 MATE & ONEDD	Transfer to MATE (Jul 2011)				
20	Sarah Oudjal	F	Engineer		●											○						2006.06	Transfer to MATE (Dec 2010)				
21	Remini Louisa	F	Assistant of Administration	●																		2008.1	Trasnfer to Univ (Dec 2010)				
22	Naasse Saadjia	M	Engineer	●												○	●					2005. 09 from ONEDD/HQ	-				
23	Omri Lynda	F	Engineer	●											●		●			GCMS		2010	-			○	
24	Boulekraouet Souhila	F	Engineer		●											●						ONEDD/HQ 2009.12	Trasnfer to CNL (May 2012)				
25	Hannachi Naila	F	Engineer		●											●						ONEDD/HQ 2011.06	-				
26	Benboudjema Meriem	F	Engineer		●											●						ONEDD/HQ 2010.05	-				
27	Tirechi Zabaria	M	Accountant	●													●					2010.03	-				
28	Tillou Soulayman	M	Principle Engineer	●													●					2011.07	-				
29	Saoud HADDA	F	State Engineer	●													●					2011.07	-				
30	Lakheira Kenza	F	Assistant of Administration	●													●					2011.07	-				

Annex 2　Output of the Project

Annex 2-1 Output-1

Annex 2-1-1-1　Record of training for GCMS analysis

Record of Training for GCMS (JFY 2009)
Enregistrement de Formation sur 1er année
2ème Travail

7 Fev	D	Vérification des fuites He, changement de la colonne(DB624), changement du linear et du septum,
8 Fev	L	Connexion du Purge and trap, mise en service du GC/MS
9 Fev	M	Sop de maintenance ,tunning (H2O/N2)
10 Fev	M	Sop de maintenance, tunning (H2O/N2) , changement de férule coté MS
11 Fev	J	Changement bouteille d'He, vérification des fuites, tunning (H2O/N2)
14 Fev	D	Résultat auto tunning, mise en service de purge and trap
15 Fev	L	essai avec le système purge and trap avec échantillon d'eau, Arrêt quotidien de la machine,
16 Fev	M	vérification des fuites, résultat tunning (PFTBA), essais avec de l'eau, tableau de maintenance
17 Fev	M	Remplissage de la bonbonne avec de l'eau pure, vérification de fuites,
18 Fev	J	Préparation des standards BTEX, standard interne, essai avec un std 0.05ppm
21 Fev	D	Essais avec des standards de BTX (5, 10, 20,50) ppb avec une nouvelle méthode (changement de
22 Fev	L	Nouvelle configuration de GC-MS avec le noueau injecteur installé (SPL1, SPL2)
23 Fev	M	Analyses de deux échantillons d'eau potable (eau de LRC, eau de l'hôtel de Hydra)
24 Fev	M	Etablir la liste de contrôle du GCMS et les SOP
25 Fev	J	Seminar
27 Fev	S	(Echantillonnage d'un échantillon réel)
28 Fev	D	Préparer les solutions standards (4points pour chaque interface et mesure des STD et des
1 Mars	L	Vérification des résultats des mesures, établir le SOP
2 Mars	M	Préparer la solution standard (1point) et mesurer autant qu'échantillon, Vérification des résultats
3 Mars	M	Control de qualité, exercice pendant l'absence de la JET

Record of Training for GCMS (JFY 2010)
Enregistrement de Formation sur 2ème année
1er Travail

20 Mai	J	Montage de l'environnement de travail laboratoire, arrangement des documents Présentation du personnel, passage du gaz hélium, test d'étanchéité, mesure contre la détérioration de la cuve d'étalon interne
23 Mai	D	Maintenance du système de production d'eau ultra-pure Millipore Révision de la POS
24 Mai	L	Démarrage de la CGSM, mise en service du système Millipore Nettoyage et séchage des vaisselles telle que la fiole jaugée
25 Mai	M	Explication sur la méthode sandwich (usage de microseringue) Explication sur le traitement du chiffre significatif, exercices
26 Mai	M	Recalcul de la dilution pour améliorer la précision de la concentration de l'étalon Préparation de la solution mère de l'étalon (25 COVs), bon fonctionnement du SM
27 Mai	J	Redémarrage suite d'une mauvaise communication entre appareils, renouvellement de l'eau de lavage, mesurage de l'eau témoin Réglage automatique, préparation du témoin (1), mesurage
30 Mai	D	Vérification du temps de rétention, élaboration du tableau des composés Changement d'heure du commencement de mesurage, remesurage de l'étalon
31 Mai	L	Elaboration du tableau des composés (suite), mise en oeuvre de l'identification des pics du mélange Etablissement de la méthode de quantification, préparation et mesurage des 7 séries d'étalons
1 Juin	M	Vérification de la courbe d'étalonnage, mise en oeuvre de l'identification manuelle des composants dont le pic n'a pas été détecté Préparation et mesurage des échantillons pour mesurage de la limite de quantification et de la limite de détection
2 Juin	M	Cours plénier CRL et exercices sur le traitement du chiffre significatif dans le cadre du contrôle d'exactitude Confirmation des résultats de mesurage
3 Juin	J	Arrivée de la cuve d'étalon interne, passage à l'ajout automatique Remesurage de la limite de quantification, de la limite de détection et des échantillons réels
6 Juin	D	Confirmation et sortie des résultats de mesurage, explication sur les techniques de requantification à l'aide du fichier séquentiel Remplissage du tableau de calcul de la limite, détermination de la limite de quantification et de la limite de détection
7 Juin	L	Explication sur la pratique de la recherche par index, identification et impression du spectre de masse de tous les composants, explication du numéro CAS, etc. Elaboration du profil des composés à l'Hôtel Hydra, car l'internet est inaccessible à CRL
8 Juin	M	Explication de la méthode d'évaluation de la toxicité, et de la pratique de la recherche d'informations sur les composés Arrêt des appareils dû à la panne d'électricité, recherche d'usages des composés
9 Juin	M	Révisions à partir du démarrage des appareils, préparation des échantillons pour la courbe d'étalonnage Mesurage, explication sur le choix de la masse cible
10 Juin	J	Mesurage des échantillons réels, révisions sur le rapport des teneurs isotopiques et sur la forme des spectres de masse Révisions sur la manière de confirmation des résultats, identification des composants détectés, recherche d'usages des composés
13 Juin	D	Explication sur le rapport S/N, les sous-produits de désinfection, etc. Explication sur la manière de résumer les résultats de quantification
14 Juin	L	Analyse des échantillons demandés, explication sur les exercices pratiques durant l'absence Rapport des résultats d'analyse à la conférence ONEDD-CRL-JET

Record of Training for GCMS (JFY 2010)
Enregistrement de Formation sur 2ème année

2ème Travail

8 Oct	J	Présentation du personnel, renouvellement de l'eau de lavage, vérification des réactifs fournis, passage du gaz hélium, test d'étanchéité, démarrage de la CGSM en confirmant la POS
31 Oct	D	Vide du SM en bon état, mesurage de l'eau témoin P&T→mauvais écha ntillonnage, nettoyage et séchage des vaisselles telle que la fiole jaugée, achèvement de la vérification des réactifs fournis
2 Nov	M	Revérification P&T, abandon, arrêt du SM, remplacement de la colonne, redémarrage, changement de la configuration de l'orifice d'injection, préparation de la solution étalon PAH, installation de la tourelle longue (équipement portable), cuisson de la colonne
3 Nov	M	Fourniture du hexane, élaboration du tableau des conditions de mesurage de PAH, explication et mise en oeuvre de la configuration "Auto tune" de l'injection directe, explication sur la méthode "surrogate", arrangement de la préparation des réactifs
4 Nov	J	Répétition de l'identification des pics du mélange, correction du tableau des composés organiques volatils, préparation des solutions étalons internes pour PAH, préparation des solutions étalons PAH surrogate, mesurage des solutions étalons pour élaboration du tableau des composés, investigation de l'endroit défectueux de P&T
7 Nov	D	Réunion du contrôle de qualité, réglage de l'installation du four électrique, cuisson des réactifs à utiliser, confirmation du temps de rétention, élaboration du tableau des composés
8 Nov	L	Achèvement de l'élaboration du tableau des composés, aménagement de la salle de prétraitement, préparation et déplacement des instruments d'expérience
9 Nov	M	Préparation des échantillons étalons additifs, traitement avant extraction par solvant, traitement avant extraction par solvant, installation de l'appareil d'enrichissement en azote
10 Nov	M	Installation de l'appareil de distillation sous pression réduite, distillation sous pression réduite, transfert aux éprouvettes
11 Nov	J	Enrichissement en azote, préparation des échantillons d'analyse, élaboration de la méthode de mesurage, mesurage à la CGSM
14 Nov	D	Confirmation des résultats de mesurage (mesurage des échantillons témoins et des échantillons étalons additifs impossible, contamination d'huile de l'appareil de distillation sous pression réduite étant jugée comme cause), préparation de la colonne de séparation pour nettoyage. Réunion du contrôle de qualité, essai de séparation des échantillons étalons par colonne pour vérifier la position d'élution (jusqu'à la collection des fractions)
15 Nov	L	Enrichissement en azote de chaque fraction, préparation des échantillons d'analyse, mesurage à la CGSM (c/p rentraient avant l'heure pour préparation de l'Aïd al-Adha)
18 Nov	J	Confirmation des résultats d'analyse, remplacement de la pompe de l'appareil de distillation sous pression réduite, installation du piège, mesurage à la CGSM du hexane de haute pureté après distillation sous pression réduite et enrichissement en azote, confirmation des résultats d'analyse (mesurage impossible)
21 Nov	D	Explication des résultats de l'essai de séparation par colonne, détermination du flux d'analyse, seconde explication de la méthode "surrogate" à l'aide des schémas, nettoyage et arrangement des instruments utilisés
22 Nov	L	Projection des vidéos éducatifs sur les opérations d'extraction liquide-liquide, de distillation sous pression réduite et de séparation par colonne, explication sur les devoirs durant l'absence et sur le contenu du prochain programme Recommandation au directeur M. Moali et au personnel

Record of Training for GCMS (JFY 2010)
Enregistrement de Formation sur 3ème année

3ème Travail

2 Fev	M	Préparation de l'eau ultra pure Démontage des parties du rotavapeur et nettoyage avec une solution de lavage de concentration (200 ml/10 l) dans l'ultrason Rinçage des différentes parties avec de l'eau distillée, acétone puis chauffer a l'étuve à 70 °C Remplissage de la bombonne avec de l'eau pure Correction du S.O.P des COV L expert japonaise va essayer de réparer le purge et trappe
2 Fev	M	Mettre le standard interne (p-Bromofluorobenzene) dans le tube VESSEL Changement du mode SPL1 à SPL2 dans le GC Changement de la bouteille d'Hélium Lancement du vacuum pour une nuit
3 Fev	J	nettoyage de la verrerie pour le prétraitement des PAH dans les sédiments Correction du S.O.P des PAH dans l'eau Remise de la norme ISO (détermination des 16 PAH dans les sédiments)
6 Fev	D	Réunion (évaluation de la formation de GCMS) Préparation des solutions standards
7 Fev	L	préparation des solutions standards pour le test du taux de récupération Mettre le gel de silice dans l'étuve à 130°C pendant 16h. Mettre le chlorure de sodium dans le four à 300°C pendant 8h.
8 Fev	M	Préparation des solutions : (éthanol/hexane) ,5% de NaCl, gel de silice,
9 Fev	M	Procédure de prétraitement du sédiment
10 Fev	J	Procédure de séparation des phases
13 Fev	D	Nettoyage de la verrerie réunion de contrôle de qualité, Tuning, autotuning, analyse d'hexane concentré
14 Fev	L	Analyse d'hexane pure, concentration des échantillons par rotavapeur et par un jet d'azote, préparation des standards de calibration, analyse des standards
16 Fev	M	Préparation des présentations
17 Fev	J	Préparation des présentations
20 Fev	D	Préparation des présentations
21 Fev	L	Séminaire, préparation de la colonne de purification
22 Fev	M	Purification des échantillons, concentration avec rota vapeur, et par un jet d'azote, préparation des standards de calibration, analyse des standards
23 Fev	M	Analyse des échantillons
24 Fev	J	Vérifier les résultats, recomendations

Record of Training for GCMS (JFY 2011)
Enregistrement de Formation sur 3ème année

2nd visit of 2011

C/P : NECHAOUNI Leila, KIMRI Leila, Omri Linda

Date	Record
10/26～10/27	Identify the cause of problem (Exchange from P&T to Direct injection), Update the maintenance SOP, Start up of GCMS
10/30～10/31	Start up of GCMS(continued), Training of how to calculate the rate of recovery , Wash and dry of glass tool
11/2～11/3	Revise the SOP, Prepare the standard solutions of PAH, Dry of samples
11/8～11/10	Pretreatment of certified standard materials and collaborative experiment sample from IAEA
11/13～11/17	Pretreatment of certified standard materials and collaborative experiment sample from IAEA (continued), Measurement by GCMS
11/20	Determine quantify for PAH by GCMS, Dilution and re-measurement for high concentration samples, Calculate the rate of recovery
11/21～11/23	(Training for the regional laboratory and monitoring stations (in Oran))
11/24	Determine quantify for PAH by GCMS , Re-dilution and re-measurement for high concentration samples, Calculation for results
11/27	Q&A about the method of quantification and the report of IAEA sample Discussion and recommendation to Training for the regional laboratory and monitoring stations (to Moali)

3nd visit of 2011

C/P : NECHAOUNI Leila, KIMRI Leila, Omri Linda

Date	Record
1/22～1/24	Verify the point of repair of P&T, confirm the movement of P&T in the presence of business agent of the Shimazu in Algeria and Moali (Leak check is failed), Check the all measurement of in the absence of expert, Change the method of GSMS solution (soft-wear) for retention time of benzo-fluoreanthene (Identification is in failed in the result of 2nd training) , Recalculate the measurement of certified standard materials.
1/25	Research the leaking point of P&T, Confirm the all results of certified standard materials
1/26	Recalculate the measurement of blank test(work in the absence of expert), Give an explanation how to calculate the diluted samples using internal standard method
1/29～1/30	Revise the SOP, Comment to the report of collaborative experiment sample from IAEA
1/31～2/2	Give an explanation how to calculate detection limit and quantification limit, Check measurement for stability of GCMS, Make a suggestion about calculation of diluted samples using internal standard method (using another way, complicated but easy to understand)
2/6～2/7	Seminar, Specify the leak point of P&T, Find the same problem seen a year ago, Revise the SOP,
2/8～2/9	Prepare the standard solutions, Measurement of calibration curve and standard sample to calculate detection limit and quantification limit, Give an explanation for QC of GCMS measurement, Conclude detection limit and quantification limit
2/12～2/15	Revise the SOP

Annex 2-1-1-2　Mid-term evaluation for GCMS analysis

Algérie - Projet de développement de la capacité de suivi de l'environnement (Phase 2) ; Tableau d'évaluation de l'avancement du transfert technique (Output 1:GCMS)

Rédigé le 22 septembre 2010

■ Evaluation de l'avancement du transfert technique au niveau organisationnel

▼ Début de Projet ▼ A mi-période de Projet (A la fin du transfert technique) ▼ A la fin du Projet

Noms du équipement	Eléments de transfert technique	Contenu d'exécution du transfert technique	Année / Mois / Période de séjour des experts sur place	Perspectives à la fin du Projet
			2009: 10 11 12 | 2010: 1 2 3 4 5 6 7 8 9 10 11 12 | 2011: 1 2 3 | 2011: 4 5 6 7 8 9 10 11 12 | 2012: 1 2 3 4 5 6 7 8 9	
GCMS	Connaissances de base concernant l'analyse de la GCMS	Compréhension sur le principe de l'appareil	Contenu exécuté en Phase 1 vérifié / Quelques cours réalisés / Quelques cours réalisés / Quelques cours réalisés / Confirmation des connaissances par séminaire	Nécessite d'une compréhension approfondie pour établir et multiplier des mesures de nouveaux éléments d'analyse
		Compréhension sur le spectre de masse	Quelques cours réalisés / Quelques cours réalisés / Confirmation des connaissances par séminaire	
		Connaissances des coposants a analyser	Réalisé y compris la méthode de recherche / Confirmation des connaissances par séminaire	
	Utilisation des matériels	Contrôle quotidien et maintenance des matériels	Absence de l'idée du contrôle quotidien / Tableau de contrôle quotidien rédigé, contrôle réalisé quotidiennement / Contrôle quotidien réalisé / Contrôle quotidien réalisé / Contrôle quotidien réalisé	Maintenance periodique du fabricant souhitable
		Manipulation des appareils (y compris le logiciel)	Pas réalisée à cause de dysfonctionnement du matériel / Une série de manipulation de P&T(Purge & Trap) réalisée / P&T réalisée répétitivement et acquise / Formation sur injection directe / Réalisée répétitivement et acquise	
		Méthode de maintenance des matériels	Pas de conscience suffisante concernant le bon moment d'exécution / Maintenance réalisée, SOP rédigée / Maintenance d'un appareil de production d'eau ultra-pure réalisée.	
	Traitement des valeurs d'analyse	Totes methodes de calcul de limites de detection et de limites de quantitatives	Pas réalisé / Réalisé / Réalisée lors de nouveaux éléments / Réalisée répétitivement et acquise	Capacité d'instruire également les personnes chargées des autres éléments
		Evaluation des valeurs d'analyse (les valeurs obtenues sont-elles correctes?)	Fiablité des données existantes douteuse / Compréhension pas encore suffisante / Compréhension approfondie / Methode d'utilisation du standard interne et du standard d'injection / Réalisée répétitivement et acquise	
		Méthode de gestion de précision des valeurs d'analyse (A)	Pas réalisé / Réalisé, compréhension pas encore suffisante / Réalisé / (A) to add in SOP	
	Analyse des BTX (Benzène, Toluène, Xylène)	Etablissement de méthodes de mesures des matières standard et de mesures avec un appareil	Révision de la méthode existante / Etablissement d'une nouvelle méthode / Methode BTX combinée aux VOCs	Atteindre un niveau de capacité pour réaliser un service d'analyse Necessite de reparer le Purge & Trap Necessite de le verifier avec un nouveau standard
		Lavage des récipients, manipulation d'échantillons (destinés aux composés organochlorés volatils) (B)	Pas réalisée à cause de dysfonctionnement du matériel / Réalisée et acquise / (B) to add in SOP	
		Rédaction et révision de la SOP	SOP existante vérifiée / rediger une nouvelle SOP / Methode BTX combinée aux VOCs	
		Nombre d'analyses des échantillons réels	Pas réalisée à cause de dysfonctionnement du matériel / Environ 15 / Methode BTX combinée aux VOCs	
	Analyse des composés organochlorés volatils	Etablissement de méthodes de mesures des matières standard et de mesures avec un appareil	Ajouté par la nécessité d'ajuster aux critères d'évacuation / Méthode de BTX élargie et rédigée / Confirmation selon nouvelle standard	Atteindre un niveau de capacité pour réaliser un service d'analyse Necessite de reparer le Purge & Trap Necessite de le verifier avec un nouveau standard
		Rédaction et révision de la SOP	rediger une nouvelle SOP / revision de la SOP / Harmonisation aux normes du LRC / revision de la SOP	
		Nombre d'analyses des échantillons réels	Environ 15	
	PAH: Analyse des hydrocarbures aromatiques polycycliques	Etablissement de méthodes de mesures des matières standard et de mesures avec un appareil	Méthode existante vérifiée / Etablissement de méthodes / Mesure de ka courbe standard	Aquitateur necessaire pour divers analyses. L'analyse de l'évacuation de la cokerie est techniquement difficile. Il est plus réalisable d'utiliser d'autres indices pour l'évaluation. Il s'agit d'un élément qui sera important dans l'avenir.
		Technique de conditionnement et de prétraitement d'échantillons (destinée aux pesticides organochlorés)	Utilisation de "clean-up" pas réalisée / Bonne technique pour tracer la courbe de calibration / Manipulation de seringue réalisée / Extraction des échantillons des eaux, méthode de "clean-up" realisée / Méthodes de extraction des échantillons du sol	
		Rédaction et révision de la SOP	SOP existante vérifiée / SOP rédigée / revision de la SOP	
	Analyse de pesticide organochloré	Etablissement de méthodes de mesures des matières standard et de mesures avec un appareil	Méthode existante vérifiée / Acheter et stoker les matières standard	Aquitateur necessaire pour divers analyses. Necessite d'avoir la bibliotheque pour pesticides. Pas besoin d'exécuter lors de l'analyse de l'évacuation. Il s'agit d'un élément qui sera important dans l'avenir.
		Rédaction et révision de la SOP	SOP existante vérifiée / Harmonisation aux normes du LRC	

■ **Evaluation de l'avancement du transfert technique au niveau individuel**		Critères d'évaluation (par les experts japonais)		+ : niveau d'initiation (quasiment pas de connaissance ni expérience) ++ : niveau débutant (Capable de comprendre une série de SOP mais pas suffisamment) +++ : niveau intermédiaire (capable de faire une analyse ordinaire sous l'instruction d'un instructeur) ++++ : niveau supérieur (ayant la compétence technique suffisante et capable d'instruire les autres)					
Personne en charge	Période d'évaluation (année, mois)	Connaissances de base concernant l'analyse de la GCMS	Utilisation des matériels	Traitement des valeurs d'analyse	Analyse des BTX(Benzène, Toluène, Xylène)	Analyse des composés organochlorés volatils	PAH: Analyse des hydrocarbures aromatiques polycycliques	Analyse des pesticides organochlorés	Perspectives à la fin du Projet
Kimri Leila	Au début du Projet (octobre 2009)	+	+/++(**logiciel**)	+	+	+	++	+	
	A la fin du transfert technique (mars 2011)	++	+++	++	+++	+++	++/+++	+	
	A la fin du Projet (mai 2012)								
Nechaouni Radia	Au début du Projet (octobre 2009)	++	+/++(**logiciel**)	+	+	+	++	+	
	A la fin du transfert technique (mars 2011)	+++	+++	++	+++	+++	++/+++	+	
	A la fin du Projet (mai 2012)								
Omri Linda	Au début du Projet (octobre 2009)	-	-	-	-	-	-	-	
	A la fin du transfert technique (mars 2011)	+	+	-	-	-	+	-	
	A la fin du Projet (mai 2012)								

Annex 2-1-1-3　Summary of GCMS analysis

Résultat du transfèrt de la technique analytique avancée pour des composants organiques volatiles en utiltsant le GCMS

Substances d'Analyse			Equipments Nécessaires		Analyse	SOP	Application du Projet (Transfert de techniques)			
			GCMS				Eaux usées	Eau de rivière	Eau souterraine	Sédiment de rivière (Sol)
			Liquid-liquid extraction	P&T						
COV (composés organiques volatils)	**BTX**	benzene	-	●	Élaboré	Élaboré	Application possible	Application possible	Application possible	Impossible en LRC
		toluene	-	●						
		p-xylene	-	●						
		m-xylene	-	●						
		o-xylene	-	●						
	1.1-dichloroethlene		-	●	Élaboré		Application possible	Application possible	Application possible	Impossible en LRC
	dichlomethane		-	●						
	trans-1,2-chloroethylene		-	●						
	tert-butylmethyl ether		-	●						
	cis-1,2 chloroethylene		-	●						
	1,1,1-trichloroethane		-	●						
	tetrachloromethane		-	●						
	1,2-dichloroehane		-	●						
	trichloroethylene		-	●						
	1,2-dichloropropane		-	●						
	1,1,2-trichloroethane		-	●						
	tetrachloroethylene		-	●						
	Trihalomethanes (disinfection bi-products)	trichloromethane	-	●						
		bromodichloromethane	-	●						
		dibromochloromethane	-	●						
		tribromomethane	-	●						
	Pesticide	cis-1,3 -dichloropropene	-	●						
		trans-1,3 dichloropropene	-	●						
		1,4--dichlorobenzene	-	●						
PAH (hydrocarbures aromatiques polycycliques)	Acenaphthene		●	-	Élaboré	Élaboré	Application très difficile	Application possible	Application possible	Application possible
	Fluolene		●	-						
	Phenanthrene		●	-						
	Anthracene		●	-						
	Fluoranthene		●	-						
	Pyrene		●	-						
	Benz(a)anthracene		●	-						
	Chrysene		●	-						
	Benzofluoranthene		●	-						
	Benzo(a)pyrene		●	-						
	Indeno(1,2,3-cd)pyrene		●	-						
	Octachlorostyrene		●	-						
	Benzo(a)pyrene		●	-						
	Dibenz(a,h)anthracene		●	-						
	Benzo(g,h,i)perylene		●	-						
Pesticide (composés organochlorés)	Persistent Organic Pollutants		○	-	Élaboré	Élaboré, mais nécessiter de réviser	Pas d'application	Application possible (*formation indépendante par la même procédé de PAH)	Application possible (*formation indépendante par la même procédé de PAH)	Application possible (*formation indépendante par la même procédé de PAH)

Annex 2-1-1-4 Standard operation procedures (SOP) for GCMS analysis

Standard Operating Procedure (SOP)

GSMS

February 2012

M^{me} NECHAOUNI Leila • M^{me} KIMRI Leila • M^{me} OMRI Linda

Détermination des composés organiques volatils (COV) dans l'eau Dosage par « Purge et Trappe »couplé à un chromatographe en phase gazeuse et à un spectromètre de masse

Norme internationale EPA : 524	
Objectifs **Analyse des composes organique volatils**	**Présentés par** M^me NECHAOUNI Leila M^me KIMRI Leila M^me OMRI Linda
Date de préparation **Juillet 2010**	**Responsable** M^me NECHAOUNI Leila M^me KIMRI Leila M^me OMRI Lynda
Date d'approbation **Juin 2010**	**Approuvé** **M^r Mohamed MOALI**

1. Elément à analyser

La présente méthode est applicable pour la détermination des composés organiques volatils dans l'eau potable, l'eau de surface et les eaux souterraines par un système « Purge et Trappe »couplé à un chromatographe en phase gazeuse et à un spectromètre de masse.
Ce sont des composés organiques volatils qui présentent généralement une faible solubilité dans l'eau et une forte pression de vapeur.
Ils sont nocifs pour la santé humaine et se trouvent dans l'environnement émis par différentes sources comme la combustion du bois, du carburant et différentes industries chimiques et de synthèse.

2. Mesure de sécurité

Les standards utilisés sont très nocifs pour la santé et l'environnement des gants et des lunettes de protection sont conseillés, travailler sous la hotte.
Les déchets de standards et de solvant sont stockés.

2. Principe

L'analyse des composés organiques volatils s'effectue en deux étapes. La première consiste à transférer ces composés de l'échantillon aqueux par un entrainement gazeux à l'aide d'un système « purge et trappe » et qui sont ensuite piégés sur un adsorbant pour être finalement désorbés thermiquement sur un chromatographe en phase gazeuse couplé à un spectromètre de masse où ils sont analysés et quantifiés.

(Voir annexe A)

3. Appareillages, Matériels, Produits Chimiques et Réactifs

3.1. Appareillages

- Système de « purge et trappe » de marque TELEDYNE Tekmar, modèle Velocity XPT
- Echantillonneur automatique de marque TELEDYNE Tekmar, modèle Aquatek 70
- Chromatographe en phase gazeuse de marque SHIMADZU, modèle GCMS 2010
- Spectromètre de masse de marque SHIMADZU, modèle GCMS 2010
-Millipore pour préparer de l'eau ultra pure

3.2. Matériels

- Fioles jaugées (10ml, 50ml, 100ml),
- Micro seringues (50µl, 100 µl)
- Viales de 44 ml avec des Bouchons contenants des septums
-- Trappe VOCARB 3000
- Colonne chromatographique capillaire d'une longueur de 30 m x 0.32 mm Di, d'épaisseur 1.8µm et de type DB-624

3.3. Produits Chimiques et Réactifs

Nom des composés	Formule Chimique	N°de Cas	Marque	pureté
1,1-dichloroethène	$C_2H_2Cl_2$	75-35-4		99%
Dichloromethane	CH_2Cl_2	75-09-2		99%
Trans-1,2-dichloroethène	$C_2H_2Cl_2$	156-60-5		99%
tert-butylmethyl ether	$C_5H_{12}O$	1634-04-4		-
Cis-1,2-dichloroethène	$C_2H_2Cl_2$	156-59-2		99%
Trichloromethane	$CHCl_3$	67-66-3		98%
1,1,1-trichloroethane	CCl_3CH_3	71-55-6		99%
Tetrachloromethane	CCl_4	56-23-5		99%
Benzène	C_6H_6	71-43-2		99%
1,2-dichloroethane	$C_2H_4C_{l2}$	107-06-2		
Trichloroethène	C_2HCl_3	79-01-6		99%
1,2-dichloropropane	$C_3H_6Cl_2$	78-87-5		99%
Bromodichloromethane	$CHBrCl_2$	75-27-4		99%
cis-1,3-dichloropropène	$C_3H_4Cl_2$	10061-01-5		99%
Toluène	C_7H_8	108-88-3		99%
trans-1,3-dichloro-propène	$ClCH_2CH{=}CHCl$	10061-02-6		99%
1,1,2-trichloro-ethane	$Cl_2CH\text{-}CH_2Cl$	79-00-5		99%
Tetrachloroethene	$Cl_2C{=}CCl_2$	127-18-4		99%
Dibromochloromethane	$CHBr_2Cl$	124-48-1		99%
m,p-xylène	C_8H_{10}	108-38-3		99%
o-xylène	C_8H_{10}	95-47-6		99%
Bromoforme	$CHBr_3$	75-25-2		99%
4-bromoflurobenzene	C6H4BrF	460-00-4		99%
1,4-dichlorobenzene	$C_6H_4Cl_2$	106-46-7		99%
Methanol	CH_3OH	67-56-1	Panreac	99.9%

3.4. Réactifs et étalons

(1) Eau ultra pure utilisée pour la préparation des solutions de calibration et pour le remplissage de la bombonne du système (purge et trappe)

(2) Méthanol, CH_3OH (67-56-1) utilisé pour la préparation des solutions standards (qualité chromatographique).

(3) Solution étalon de 1000 µg/ml
Ampoule de 2 ml contenant 1000 µg/ml de 25 composés organiques volatiles.

(4) Solution étalon interne de 1 mg/ml
Ampoule contenant 1 mg/ml de p-Bromofluorobenzene (BrC_6H_4F)

(5) Gaz d'Hélium 99.999 %

4. Echantillonnage et Prétraitement de L'Echantillon

Prélever les échantillons dans des flacons en verre complètement remplis et fermés avec des bouchons en silicone contenant des septums en téflon et les conservés à 4°C. Le délai de conservation entre le prélèvement et l'analyse ne doit pas excéder 7 jours.

5. Lavage de la verrerie

Placer les tube dans un bêcher et laver dans l'ultrason pendant 20 min, jeter l'eau puis répéter l'opération pendant 5 min puis chauffer à 110°c pendant 3 h, laisser refroidir dans un dessiccateur

6. Préparation des solutions standard

6.1. Préparation des solutions standards

A partir de la solution mère (3.4.3) on prépare une série de dilution avec le méthanol pour des concentrations comprises entre 2 ppb et 50 ppb dans des fioles de 10 ml et à l'aide de micro seringues appropriées (Tableau 1).

Note
Ces solutions sont conservées à -25°C et utilisées pendant un mois. Elles sont préparées pour chaque série d'échantillon analysé, et elles ne peuvent pas être réutilisées.

6.2. Préparation des solutions de calibration

A partir des solutions standard préparées (6.1) on réalise une série de dilution avec de l'eau ultra pure pour des concentrations comprises entre 0.002 ppm et 0.05 ppm dans des fioles de 50 ml à l'aide de micro seringues appropriées.

6.3. Préparation de la solution interne

A partir de la solution (3.4.4) on prépare une dilution avec le méthanol dans une fiole de 100 ml.

Note
Cette solution est conservée à -25 °C.

Tableau 1. Tableau récapitulatif des différentes concentrations réalisées

Volume prélevé de 1000 µg/ml (µl)	**Dilution avec Methanol (ml)**	**Concentrations des standards (ppm)**	**Volume prélevé des standards (µl)**	**Dilution avec eau pure (ml)**	**Concentrations finales (ppb)**
20	**10**	0.002	100	**100**	2
50	**10**	0.005	100	**100**	5
100	**10**	0.010	100	**100**	10
200	**10**	0.020	100	**100**	20
500	**10**	0.050	100	**100**	50

Protocole d'analyse

1- Introduire les échantillons dans des viales de 44 ml accompagnés d'un témoin (blanc) afin de vérifier la présence de contaminants dans la verrerie ou dans le système. De plus, lorsqu'un échantillon très contaminé est injecté, il faut toujours vérifier s'il y a encore des traces de celui-ci dans le système à l'aide d'un échantillon témoin.
(Le témoin dans notre cas est de l'eau pure)

2- Introduire les viales des échantillons et des solutions de calibration (6.b) dans l'échantillonneur automatique Aquatek 70 (les viales de solutions de calibration sont classés avant les viales du témoin et de l'échantillon ; voir l'exemple de batch table annexe***)

3- Remplir le réservoir de l'échantillonneur de solution étalon interne (5.4) diluée

4- Vérifier les conditions d'opération des différents systèmes, **voir annexe B**

5- Effectuer l'analyse.

6- Les résultats d'analyse sont obtenus à l'aide d'un système informatisé de traitement de données.

7- Les résultats sont exprimés en ppb de chacun des composés organiques volatils.

9. La mise en service du système« Purge et Trappe » couplé à un « GC/MS »

Remplir la bombonne (eau pure)
Ouvrir la bouteille de gaz He (deuxième cadran à 50psi)

« GC/MS » — « Purge and Trap »

Mettre en marche **stabilisateur**, **GC**, **MS** et **PC** → Mettre en marche les trois unités de **«P** et **T »**

Vérifier la pression du cadran (20 psi)

Vérifier les fuites d'hélium dans tout le système de raccordement avec un détecteur
(En cas de fuites au niveau de la colonne serrer avec une clé)

Appuyer sur [On flow] pour mettre le gaz
d'He en circulation dans la colonne

- Cliquer sur le programme **GCMS Real Time Analysis**
- Vérifier le système de configuration
- Démarrer le système **Vacuum** (attendre une nuit avant d'effectuer d'autres procédures)

-Vérifier les fuites avec le **Tuning** (vérifier le ratio de 18 m/z et 28 m/z.si la hauteur du pic 28 m/z est deux fois plus petite que la hauteur du pic 18m/z aucune fuite n'est détectée
-un échantillon d'eau et introduit dans le système avant de réalisé l'autotuning

- Réaliser l'**Autotuning** avec un courant d'émission de 60µA (mettre les conditions opératoire puis procéder à l'autotoning ; lancer acquisitions Download initial paramètre)

Introduire les échantillons dans des viales de 44 ml accompagnés d'un témoin (blanc) afin de vérifier la présence de contaminants dans la verrerie ou dans le système. De plus, lorsqu'un échantillon très contaminé est injecté, il faut toujours vérifier s'il y a encore des traces de celui-ci dans le système à l'aide d'un échantillon témoin. (Le témoin dans notre cas est de l'eau pure)

- Créer un nouveau dossier (Project Folder)
- Copier la méthode (conditions opératoires)
- Créer la table séquentielle (Batch table)

Cliquer sur **Start** (batch table) →

- Cliquer sur le programme **velocity XPT tek Link**
- Créer une méthode **(conditions opératoires)**
- Créer un nouveau **Schedule**
- Cliquer sur la barre **make active**
- Attendre l'ajustement des conditions
- Cliquer sur **Set**
- Cliquer sur **Start**

Analyse en cours

10-La mise en arrêt du système « Purge et Trappe » couplé à un « GC/MS »

- Arrêter le système **Vacuum** (shutdown)
- Quitter le programme **GCMS Real Time Analysis**
- Quitter le programme **Velocity XPT tek Link**
- Eteindre le pc, les trois unités de « PetT », le MS, le GC et le stabilisateur
- Fermer la bouteille du gaz
  Ouvrir la valve de la bouteille d'eau pour éviter l'effet ventouse en éliminant l'hélium
  Ce refera aux instructions du manuel

11- Contrôle de qualité

Les échantillons sont analysés en double pour confirmer le résultat au cas où il contient des composes organique volatils.

On fait passé un standard de 20ppb si les résultats est supérieur à 70% l'analyse est fiable dans le cas contraire l'analyse doit être refaite.

11.1. Limite de détection

Nom des composes	Temps de rétention (mn)	Limite de quantification (ppb)	Limite de détection (ppb)
1.1-dichloroethène	2.61	7	2
Dichloromethane	3.01	5	2
trans-1,2-dichloroethène	3.23	5	2
tert-butylmethyl ether	3.23	5	2
cis-1,2-dichloroethène	3.98	5	2
Trichloromethane	4.21	5	2
1,1,1-trichloroethane	4.35	5	2
Tetrachloromethane	4.47	5	2
Benzène	4.62	5	2
Trichloroethène	5.07	5	2
1,2-dichloropropane	5.23	5	2
Bromodichloromethane	5.42	5	2
cis-1,3-dichloropropène	5.73	5	2
Toluene	5.98	5	2
trans-1,3- dichloro-propène	6.12	5	2
1, 1,2-trichloro-ethane	6.26	5	2
Tetrachloroethene	6.37	5	2
Dibromochloromethane	6.56	5	2
m,p-xylène	7.15	7	2
o-xylène	7.45	5	2
Bromoforme	7.60	5	2
1,4-dichlorobenzene	8.74	5	2

Tableau 2 : tableau récapitulatif des composés organiques volatils.

Nom des composés	Target (m/z)	Ion de référence (m/z)	Temps de rétention (mn)
1.1-dichloroethène	61	96	2.61
Dichloromethane	84	86	3.01
trans-1,2-dichloroethène	96	61	3.23
tert-butylmethyl ether	73	57	3.23
cis-1,2-dichloroethène	61	96	3.98
Trichloromethane	83	85	4.21
1,1,1-trichloroethane	97	99	4.35
Tetrachloromethane	117	119	4.47
Benzène	78	77	4.62
1,2-dicloroethane	62	64	4,63
Trichloroethène	130	132	5.07
1,2-dichloropropane	63	62	5.23
Bromodichloromethane	83	85	5.42
cis-1,3-dichloropropène	75	49	5.73
Toluène	91	92	5.98
trans-1,3-dichloropropène	75	49	6.12
1,1,2-trichloro-ethane	97	83	6.26
Tetrachloroethene	166	164	6.37
Dibromochloromethane	129	127	6.56
m,p-xylène	91	106	7.15
o-xylène	91	106	7.45
Bromoforme	173	171	7.60
4-bromofluorobenzéne	174	176	7.84
1,4-dichlorobenzene	146	148	8.74

12. Référence

ISO 15680
EPA **524**

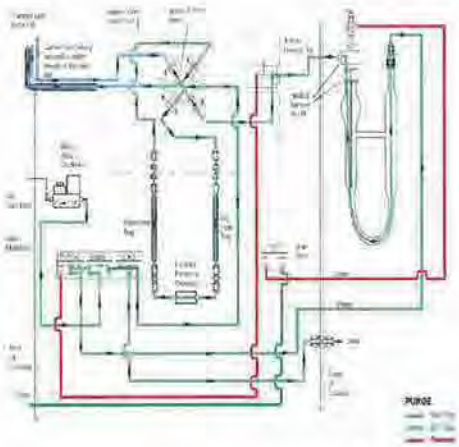
13. Enregistrement et les Révisions des Modes Opératoires Normalisés

October 1, 2010: First Revision was made by M.

Annexe A
Principe « purge et trappe »

1. La Purge

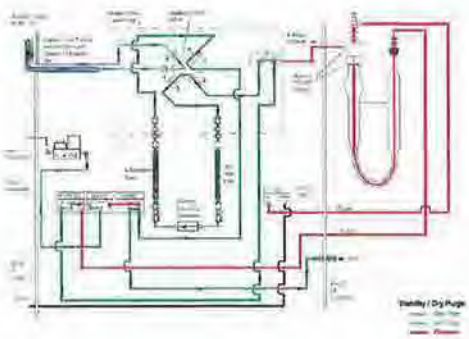
Flux de gaz : Tube de la purge → Trappe du flux sec → Trappe d'adsorption → Ventilation



L'échantillon est aspiré et barboté avec de l'Hélium dans le tube en U pendant 11min
Les composés organiques volatiles sont piégés dans la trappe d'absorption.

2. La purge sèche

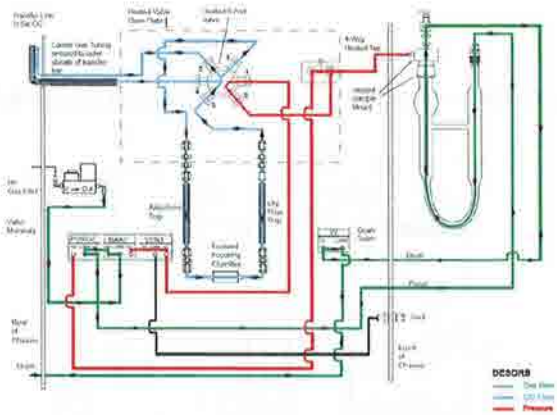
Flux de gaz : Trappe du flux sec → Trappe d'adsorption→ Ventilation



Un flux de gaz circule

3. La désorption

Flux de gaz : Trappe d'adsorption → Trappe du flux sec

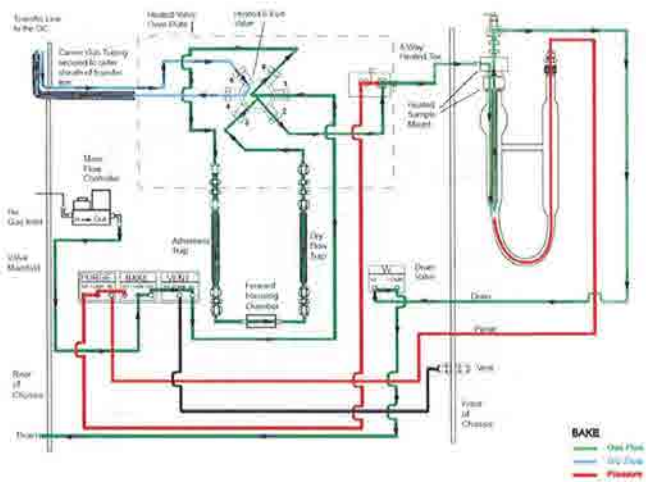


- La trappe d'adsorption est chauffée à 200°C pendant 2 mn

- Les composés organiques volatiles sont adsorbés dans la trappe du flux sec puis envoyés au GC /MS.
- L'eau est éliminée dans la trappe du flux sec

4. Le chauffage

Flux de gaz : Trappe d'adsorption→ Trappe de flux sec → Montée d'échantillon→ Drainage



Les composés organiques volatiles et l'eau sont éliminés par chauffage à 270°C pendant

Annexe B

a. Les paramètres chromatographiques

Colum oven température	40°C
Injection température	200°C
Injection mode	Split
Flow control mode	Linear Velocity
Pressure	31.1 KPa
Total Flow	61.7 ml/min
Column Flow	1.99 ml/min
Linear Velocity	50.9 cm/sec
Purge Flow	0.0 ml/min
Split Ratio	30 ml/min
High Pressure Injection	OFF
Carrier Gas Saver	OFF
Splitter Hold	OFF

- Oven Temperature Program

Rate	Température (°C)	Hold Time (min)
-	40.0	2.00
20.00	230.0	5.00

Equilibrium Time 1.0 min

b. Les paramètres du spectromètre de masse

Ion Source Température	200.00 °C
Interface température	230.00 °C
Solvent Cut Time	2.0 min
Detector Gain Mode	Relative
Detector Gain	-0.20 KV
Threshold	0
Start Tim	2.10 min
End Time	16.00 min
ACQ Mode	Scan
Event Time	0.50 sec
Scan Speed	476
Start m/z	35.00
End m/z	260.00

c. Les paramètres du « purge et trappe »

Variable	valeur	Variable	valeur
Valve Oven Température	120°C	Dry Purge Température	20°C
Transfer Line Température	120°C	Dry Purge Flow	200ml/min
Sample Mount Température	90°C	GC Start	Start of desorb
Purge Ready Température	45°C	Desorb Preheat Température	200°C
Dry Flow Standby Température	120°C	Desorb Drain	ON
Standby Flow	40mL/min	Desorb Drain	2.00min
Pressurize Time	0.30min	Desorb Température	250°C
Fill I.S Time	0.10min	Desorb Flow	300 ml/min
Sample Transfer Time	0.50min	Bake rinse	on
Pre-Purge Time	0.50min	Number of Bake Rinses	3
Pre-Purge Flow	40mL/min	Bake Drain Time	0.50 min
Sample Heater	Off	Bake Drain flow	400ml/min
Sample Preheat Time	1.00min	Bake Time	4.50min
Preheat Température	40°C	Bake temp	270°C
Purge Time	11.00min	Dry flow Bake Temp	220°C
Purge Température	0°C	Bake Flow	400ml/min
Purge Flow	40mL/min	Focus Temperature	/
Purge Rinse Time	0.25min	Inject Time	/
Purge Line Time	0.25min	Inject Temperature	/
Dry Purge Time	1.00min	Standby Temperature	/

Exemple de batch table

Vial No.	Sample Name	Sample I.D.	Sample type	Analysis type	Method file	Data file
1	Water	UNK-0001	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-001.qgd
2	STD1	STD-0001	1:standard(I)	IT QT	10.05.30 voc25.qgm	20100602-002.qgd
3	STD2	STD-0002	1:standard	IT QT	10.05.30 voc25.qgm	20100602-003.qgd
4	STD3	STD-0003	1:standard	IT QT	10.05.30 voc25.qgm	20100602-004.qgd
5	STD4	STD-0004	1:standard	IT QT	10.05.30 voc25.qgm	20100602-005.qgd
6	STD5	STD-0005	1:standard	IT QT	10.05.30 voc25.qgm	20100602-006.qgd
7	STD6	STD-0006	1:standard	IT QT	10.05.30 voc25.qgm	20100602-007.qgd
8	Water	UNK-0002	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-008.qgd
9	BLK	UNK-0003	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-009.qgd
10	Sample-1	UNK-0004	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-010.qgd
11	Sample-2	UNK-0005	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-011.qgd
12	Sample-3	UNK-0006	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-012.qgd
13	Sample-4	UNK-0007	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-013.qgd
14	Sample-5	UNK-0008	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-014.qgd
15	STD5	UNK-0009	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-015.qgd
16	Water	UNK-0010	0:unknown	IT QT	10.05.30 voc25.qgm	20100602-016.qgd

Titre : Dosage des Hydrocarbures Aromatique polycyclique dans l'eau par GCMS

Norme internationale : ISO/ DIS 28540

Les objectifs : **Analyse des HAP dans l'eau**	**Fait par :** **M^{me} NECHAOUNI Leila** **M^{me} KIMRI Leila** **M^{me} OMRI Linda**
Date de préparation **Octobre 2010**	**Responsable** **M^{me} NECHAOUNI Leila** **M^{me} KIMRI Leila** **Mme OMRI Linda**
Date d'approbation	

1. Elément analysé

Les hydrocarbures aromatiques polycycliques (HAP voir tableau 4.2)

2. Mesures de sécurité

Les standards utilisés sont très nocifs pour la santé et l'environnement des gants et des lunettes de protection sont conseillés.
Les déchets de standards et de solvant sont stockés.

3. Appareillage et matériel

-Flacons en verre brun de 1l de type pyrex

-Ampoules à décanter de 2l

-Erlenmeyer de 250ml

-Support d'ampoules

-Ballon SPC29- de 300 ml

-Entonnoirs

-Colonne chromatographique (pour la purification d'extrait)

-Dessiccateur

-Tubes à essai (20ml ,10 ml).

-fioles jaugées (10ml, 100ml), micro seringues (25µl, 50 µl), pipettes (1ml), et des pipetes pasteur.

-Viales de 1ml

-Ajustable de bouchon de viales

-Evaporateur rotatif LABOROTA 4000, Heidolph.

-Chromatographe en phase gazeuse de marque SHIMADZU, modèle GCMS 2010

-Un échantillonneur automatique AOC-20i

-Un détecteur de spectromètre de masse de marque SHIMADZU, modèle GCMS 2010

-Logiciel permettant l'acquisition et le traitement des données provenant de l'instrument.

-Colonne chromatographique capillaire d'une longueur de 30 m x 0.25 mm Di, d'épaisseur 0.25µm et de type HP5ms

4. Réactifs et étalons

4.1 Réactifs

Noms de réactif	Numéro de CAS	Pureté	Formule chimique	Marque
Sulfate de sodium	77-82-6		Na2SO4	Kanto chemical
Chlorure de sodium	7647-14-5	99.5%	NaCl	PROLABO
Hexane	110-54-3	PESTINORM ≥95%	C6H14	PROLABO
Acétone	016-00346	99.5%	C3H6O	WAKO
Gel de silice 40	63231-67-4			Sigma Aldrich

-Sulfate de sodium, Na2SO4, anhydre, purifié préalablement par un chauffage à 300 °C pendant 8 h

-Chlorure de sodium, NaCl, anhydre, purifié préalablement par un chauffage à 300 °C pendant 8 h

-Gaz d'Hélium (He) pour la chromatographie en phase gazeuse 99,99%

-Gaz d'Azote (N_2) pour évaporer les extraits.

4.2 étalons

-Solution standard mère de 2000 ppm RESTEK (mixture de 19HAP) voir (tableau 4.2)
Pour ces composés : Naphtalène ; 1-Methylnaphthalene ; 2-Methylnaphthalene; Acenaphthylene le taux de récupération est faible donc, difficile de les analysés avec cette méthode)

-Solution standard interne Phénanthrène-D10

-Solution standard interne Fluoranthene-D10

-Solution standard interne Benzo (a) pyrène-D12

-Solution standard d'injection p-Terphenyl-D14

Noms	Numéro de CAS	Pureté	Formule chimique	Marque
Naphtalène	91-20-3	99%	$C_{10}H_8$	RESTEK
1-Methylnaphthalene	90-12-0	99%	C11H10	RESTEK
2-Methylnaphthalene	91-57-6	97%	C11H10	RESTEK
Acenaphthylene	208-96-8	99%	C12H8	RESTEK
Acenaphthene	83-32-9	99%	C12H10	RESTEK
Fluorene	86-73-7	99%	C13H10	RESTEK
Phénanthrène	85-01-8	99%	C14H10	RESTEK
Anthracène	120-12-7	99%	C14H10	RESTEK
Fluoranthene	206-44-0	98%	C16H10	RESTEK
Pyrène	129-00-0	98%	C16H10	RESTEK
Benz[a]anthracène	56-55-3	99%	C18H12	RESTEK
Chrysene	218-01-9	99%	C18H12	RESTEK
Benzo[b]fluoranthene	205-99-2	99%	C20H12	RESTEK
Benzo[k]fluoranthene	207-08-9	99%	C20H12	RESTEK
Benzo[a]Pyrène	50-32-8	99%	C20H12	RESTEK
3-Methylcholanthrene	56-49-5	99%	C21H16	RESTEK
Indeno (1, 2,3-cd) Pyrène	193-39-5	99%	C22H12	RESTEK
Dibenzo [a,h]anthracène	53-70-3	99%	C22H14	RESTEK
Benzo[ghi]perylene	191-24-2	99%	C22H12	RESTEK
Phenanthrene-D10	1517-22-2		C14D10	SUPELCO
Fluoranthene-D10	93951-69-0		C16D10	SUPELCO
Benzo(a) pyrene-D12	.63466-71-7		C20D12	SUPELCO
P-Terphenyl-D14	1718-51-0		C18D14	SUPELCO

5. Prélèvement et conservation

Prélever les échantillons dans des flacons en verre brun ayants un volume de 1000 ml et fermés avec des bouchons contenant des joints en téflon et les conservés à 4°C. Le délai de conservation entre le prélèvement et l'extraction ne doit pas excéder 7 jours afin d'éviter les pertes.

Remplir la bouteille d'échantillonnage au ménisque.

6. principe

La détermination des hydrocarbures polycycliques aromatiques dans un échantillon d'eau se fait par extraction liquide-liquide à l'aide d'un solvant organique (hexane) et après purification si nécessaire et concentration avec le rotavapeur puis avec un jet d'azote l'extrait est analysé par chromatographe en phase gazeuse couplé à un spectre de masse.

7. Solutions standards

7.1 Préparation des solutions standards

Le domaine d'application utilisé pour le dosage par chromatographie en phase gazeuse couplé à un spectromètre de masse se situe entre 0.5 et 3 mg/l de HAP.

On prépare une série de dilutions avec l'hexane dans des fioles de 10 ml à partir de la solution mère de 2000 ppm selon les proportions suivantes :

	Concentration de solution étalon (ppm)	Volume prélevé de solution mère (µl)	Volume final (ml)
(a)	0	0	10
(b)	0.5	2.5	10
(c)	1	5	10
(d)	1.5	7.5	10
(e)	2	10	10
(f)	2.5	12.5	10
(g)	3	15	10
(h)	10	50	10

Note - Ces solutions sont conservées à -25°C et utilisées pendant 6 mois

7.2 Préparation de solution standard d'injection

A partir de la masse pesée (0.01g) du standard d'injection p-Terphenyl-D14, on prépare une dilution avec l'hexane dans une fiole de 100 ml. **(A)**

Note - Ces solutions sont conservées à -25°C.

7.3 Préparation de solution standard interne

-A partir de la masse pesée (0.01g) du standard interne Phénanthrène-D10, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

-A partir de la masse pesée (0.01g) du standard interne Benzo (a) pyrène-D12, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

-A partir de la masse pesée (0.01g) du standard interne Fluoranthene-D10, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

On prépare une mixture de 30 ppm de standard interne en prélevant 30 ml des trois solutions préparées dans une fiole de 100 ml et on complète avec de l'hexane. **(B)**

Note - Ces solutions sont conservées à -25°C

Préparation de la solution du standard interne ajouté aux échantillons
-A partir de la mixture de 30ppm du standard interne **(B)** on prend 10 ml et on prépare une dilution avec l'acétone dans une fiole de 100 ml (3ppm) **(C)**

Note - Cette solution est ajoutée aux échantillons d'eau

Préparation de la solution standards pour le calcule du taux de récupération

A partir de la solution (h) de 10 ppm, on prend 1ml et on prépare une dilution avec l'acétone dans une fiole de 10ml (1ppm) **(D)**

7.4 Préparation des solutions de calibration

On donne ci-dessous un tableau récapitulatif des différentes concentrations préparées dans des viales pour tracer la courbe de calibration.

Concentration de solutions standards (ppm)	Volume prélève de solutions standards (ml)	Volume ajouté de solution standard d'injection (A) (µl)	Volume ajouté de solution standard interne (Mixture) (B) (µl)
0	1 (a)	10	20
0.5	1 (b)	10	20
1	1(c)	10	20
1.5	1(d)	10	20
2	1(e)	10	20
2.5	1(f)	10	20
3	1(g)	10	20

Note - Ces solutions sont préparées pour chaque série d'échantillon analysé. Elles ne peuvent être réutilisées.

On prépare une autre série de standard pour vérifier le taux de récupération du standards interne on donne ci-dessous un tableau récapitulatif des différentes concentrations préparées dans des viales.

Volume prélevé de l'hexane (ml)	Volume prélevé de standard interne (B) (µl)	Concentration de standard interne (ppm)	Volume prélevé de standards d'injection(A) (µl)	Concentration de standard d'injection (ppm)
1	5	0.15	10	1
1	10	0.30	10	1

1	15	0.45	10	1
1	20	0.60	10	1
1	25	0.75	10	1

8. Procédures de prétraitement

8.1 Prétraitement de l'échantillon (Extraction des HAP par l'Hexane)

Marquer le niveau de l'eau sur le flacon en verre de l'échantillon

↓

Ajouter 200µl de standards interne(C) à l'échantillon
Fermer le flacon et mélanger vigoureusement

↓

Transférer l'échantillon dans une ampoule à decanter de 2000 ml

↓

Ajouter 30g de NaCl anhydre
Agiter pour homogénéiser

↓

Rincer soigneusement la bouteille avec le solvant d'extraction afin d'extraire
les HAP adsorbés et le transférer dans l'ampoule à décanter
(L'extraction se fait avec 100 ml d'Hexane)

↓

Agiter pendant 15mn en évacuant à chaque fois l'air

↓

Laisser décanter puis Transférer l'échantillon (la phase aqueuse) dans le flacon en verre
Récupérer la phase organique dans un ballon à travers un entonnoir
Contenant un peu de coton imbibé d'hexane avec10g de Na_2SO4anhydre

↓

Répéter l'extraction avec 100 ml d'hexane et transférer à nouveau l'échantillon dans l'ampoule
Agiter pendant 15 min en évacuant à chaque fois l'air

↓

Laisser décanter puis récupérer la phase organique dans le même ballon
Contenant un peu de coton imbibé d'hexane avec10g de Na_2SO4anhydre

↓

Concentrer l'extrait récupérer avec l'évaporateur rotatif à 10 ml

↓

Transférer l'extrait dans un tube et le concentré à 1ml sous un jet d'azote

Ajouter 10µl de standard d'injection à l'extrait dans le tube

↓

Transférer l'extrait dans une viale Effectuer l'analyse par GCMS

8.2 Taux de récupération

On prend 1000 ml d'échantillon d'eau pure dans un flacon, on ajoute 1ml de la mixture du standard de HAP (D) et on procède au prétraitement de l'échantillon comme s'est indiqué ci-dessus puis l'analysé avec le GCMS.

8.3 Test du blanc

Un échantillon d'eau pure est utilisé pour le test du blanc, il est traité de la même façon qu'un échantillon réel et doit accompagner chaque série d'analyse dans le but d'assurer une performance de la procédure.
La valeur détectée doit être moins de 50% de la plus basse concentration

8.4 Purification de l'extrait après extraction

La purification est nécessaire seulement si l'extrait est coloré

8.4.1 Préparation du gel de silice

On pèse 95g de gel de silice, on le met dans l'étuve pendant 15 h à 130°C.
On ajoute à 95g de gel de silice, 5ml de H2O (5%), on mélange pendant 30 mn jusqu'à homogénéisation et on laisse refroidir dans un dessiccateur pendant 15 heures
Ce gel est utilisé pendant 7 jours.

8.4.2 Préparation de la colonne de purification

Placer un petit bout de coton imbibé d'hexane à l'intérieur de la colonne ; transférer 5 g du gel de silice dilué avec une quantité d'hexane, laisser décanter puis ajouter 2 cm de Na_2SO_4. Jeter la quantité en excès de l'hexane juste au dessus du Na_2SO_4 pour éviter la formation de bulle d'air.

8.4.3 Élution de l'extrait

Laver la colonne avec 10 ml d'hexane

↓

Transférer l'extrait avec une pipette pasteur
Jeter l'hexane sans dépasser le niveau de Na_2SO_4 dans la colonne à un débit de (1ml/min)

↓

Laver les parois du tube et de la colonne qui contient l'extrait avec 2 ml d'hexane et les transférer dans la colonne

Jeter l'hexane sans dépasser le niveau de Na_2SO_4 dans la colonne à un débit de (1ml/min)

↓

Ajouter 8ml d'hexane dans la colonne
Jeter l'hexane sans dépasser le niveau de Na_2SO_4 dans la colonne à un débit de (1ml/min)

↓

Récupérer l'extrait avec les 100 ml du mélange de 1% acétone / hexane (1ml acétone dans 99ml d'hexane, avec précision)
Goutte à goutte à 1ml/min dans un ballon juste au niveau de Na_2SO_4

↓

Concentré l'extrait avec l'évaporateur rotatif à 10 ml, le transférer dan un tube à essai gradué

↓

Concentrer les 10ml sous un jet d'azote à1ml
Ajouter 10µl de standard d'injection **(A)** à l'extrait dans le tube

↓

Transférer l'extrait dans une viale et effectuer l'analyse par GCMS

9. Evaluation et Mesure

9.1 Mesure

1- Vérifier les conditions d'opération des différents systèmes (GC, MS, échantillonneur)
2- Introduire les viales qui contiennent les solutions de calibration ainsi que la viale qui contient l'extrait (l'échantillon) dans l'échantillonneur automatique
3- effectuer l'analyse.
4- Le benzo(b) fluoranthene et le benzo (k) fluoranthene ne peuvent pas être sépare complètement donc ils ne peuvent pas être déterminé comme le benzofluroranthene
5- Les résultats sont exprimés en µg/L

a. Les paramètres de l'échantillonneur AOC-20i

#of Rinses withpresolvent	2
#of Rinses withsolvent(post)	3
#of Rinses with sample	2
Plunguer speed (suction)	High
Viscosity com.time	0.2
Plunger speed (injection)	High
Syringe insertion speed	High
Injection Mode	Normal
Pumping Times	3
Inj. Port dwell Time	0.3sec
Terminal Air Grap	No

PlunguerWashing Speed	High
Washing Volume	8µl
Syringe suction position	0.0mm
Syringe Injection position	0.0mm
Use 3 solvent vial	1vial

b. Les paramètres chromatographiques

Colum oven température	45°C
Injection température	250°C
Injection mode	Splitless
Sampling Time	1.00 min
Flow control mode	Linear Velocity
Pressure	64.9 KPa
Total Flow	50 mL/min
Column flow	1.20 mL/min
Linear Velocity	40.0 cm/sec
Purge flow	4.0 mL/min
Split Ratio	- 1.0
High Pressure Injection	On
High Press. Inj .pressure	250.0 kpa
High Press. Inj .time	1.5 min
Carrier Gas Saver	OFF
Splitter Hold	OFF

Oven Temperature Program

Rate	Température (°C)	Hold Time (min)
-	45.0	1.00
45.00	130.0	0.00
12.00	180.0	0.00
7.00	240.00	0.00
12.00	320.00	4.00

c. Les paramètres du spectromètre de masse

Ion Source Température	200.00 °C
Interface temperature	250°C
Solvent Cut Time	4.50 min
Detector Gain Mode	Relative
Detector Gain	0 kv
Threshold	0

Start Time	4 .8min
End Time	25.00 min
ACQ Mode	Scan
Event Time	0.50 sec
Scan Speed	833
Start m/z	45.00
End m/z	450.00

d. Target masse, référence masse and R.T,

Nom	Target masse (m/z)	Masse de référence (m/z)	Temps de rétention (min)
Naphtalène	128	102	5,02
1-Methylnaphthalene	142	115	5,83
2-Methylnaphthalene	142	115	5,98
Acenaphthylene	152	150	7,14
Acenaphthene	154	153	7,44
Fluorene	166	165	8,38
Phénanthrène	178	152	10,50
Anthracène	178	152	10,62
Fluoranthene	202	200	13,82
Pyrène	202	101	14,48
Benz[a]anthracène	228	226	18,05
Chrysene	228	226	18,15
Benzo[b]fluoranthene	252	250	20,58
Benzo[k]fluoranthene	252	250	20,63
Benzo[a]Pyrène	252	250	21,21
3-Methylcholanthrene	268	269	21,96
Indeno (1, 2,3-cd) Pyrène	276	138	23,26
Dibenzo [a,h]anthracène	278	139	23,30
Benzo[ghi]perylene	276	138	23,72
Phenanthrene-D10	188	80	10,45
Fluoranthene-D10	212	106	13,77
Benzo(a) pyrene-D12	264	-	21,18
P-Terphenyl-D14	244	243	15,20

9.2 Evaluation

Le calcul des standards de calibration et des échantillons se fait par la méthode PAH « new »GCMS solution.

Les valeurs analysés sont automatiquement corrigés parle taux de récupération du standard interne. Des standards internes sont utilisés pour calculer le taux de récupération dans cette méthode on utilise trois déterres qui correspondent a un groupe de composes qui ont des temps de rétention proche voir tableau ci-dessous.

Composés	Standard internes
Acénaphtène	Phenanthrene-d10
Fluorene	Phenanthrene-d10
Phénanthrène	Phenanthrene-d10
Anthracène	Fluoanthene-d10
Fluoranthene	Fluoanthene-d10
Pyrene	Fluoanthene-d10
Benzo(a) anthracene	Benzo(a)pyrene-d10
crysene	Benzo(a)pyrene-d10
Benzo[b]fluoranthene	Benzo(a)pyrene-d10
Benzo[k]fluoranthene	Benzo(a)pyrene-d10
Benzo[a]Pyrène	Benzo(a)pyrene-d10
3-Methylcholanthrene	Benzo(a)pyrene-d10
Indeno (1, 2,3-cd) Pyrène	Benzo(a)pyrene-d10
Dibenzo [a,h]anthracène	Benzo(a)pyrene-d10
Benzo[ghi]perylene	Benzo(a)pyrene-d10

Vérifier le taux de récupération du standard interne par la deuxième courbe de calibration(7.4)
On copie le dossier pour chaque échantillon et batch processing pour la deuxième courbe de calibration puis copier datafile pour chaque échantillon en utilisant GCMS solution méthode « new PAHD » puis le calcule de taux du récupération de pour chaque déterré en utilisant l'équation ci-dessous.
La fiabilité des résultats est vérifiée par le taux de récupération des standards internes misent dans l'échantillon selon la formule suivante :

$$\frac{\textbf{Concentration trouvée}}{\textbf{Concentration théorique}} \textbf{*100}$$

Si le taux est compris ente 70% 130% le résultat est fiable.

Expression des résultats :

La concentration des échantillons est calculée par l'équation suivante :

$$C = \frac{m_{ex}}{V_S}$$

C : concentration de chaque composé de HAP trouvé dans la phase aqueuse en µg/L
$\mathbf{m_{ex}}$: la quantité du composé dans l'extrait en µg
$\mathbf{V_S}$: volume de l'échantillon en L

10. limites de Détection

	limite de detection* (µg/L)	limite de quantification* (µg/L)
Acenaphthene	0.6	2
Fluorene	0.2	0.4
Phenanthrene	0.02	1
Anthracene	0.04	0.2
Fluoranthene	0.02	0.05
Pyrene	0.02	0.1
Benz[a]anthracene	0.02	0.1
Chrysene	0.02	0.05
Benzofluoranthene	0.2	0.5
Benzo[a]pyrene	0.2	0.5
3-Methylcholanthrene	0.2	0.5
Indeno[1,2,3-cd]pyrene	0.2	0.6
Dibenz[a,h]anthracene	0.2	0.6
Benzo[ghi]perylene	0.3	0.9

*Quand 1ℓ D'echantillon est pris

11. Référence

DRAFT INTERNATIONAL STANDARD ISO/ DIS 28540

12. Enregistrement et Révisions des Modes Opératoires Normalisés

Octobre, 2010: première Révision faite par LRC

Fevrier 2012: deuxieme Révision faite par LRC

Titre : Dosage des Hydrocarbures Aromatique polycyclique dans le sol par GCMS

Norme internationale : ISO 18287

Les objectifs : **Analyse de sol**	**Fait par :** **M^me^ NECHAOUNI Leila** **M^me^ KIMRI Leila** **M^me^ OMRI Linda**
Date de préparation **Février 2011**	**Responsable**
Date d'approbation	

1. Elément analysé

Les hydrocarbures aromatiques polycycliques (HAP)

Cette méthode permet l'identification et la quantification des HAP présents dans le sol

2. Mesure de sécurité

Les standards utilisés sont très nocifs pour la santé et l'environnement des gants et des lunettes de protection sont conseillés pendant les pratiques.
Les déchets de standards et de solvant sont stockés.

3. Appareillage et matériel

-Tubes en verre brun de 100 ml.

-Ampoules à décanter de 300 ml

-Support d'ampoules

-Ballon SPC29- de 300 ml

-Entonnoirs

-Colonne chromatographique (pour la purification d'extrait)

-Dessiccateur

-Tubes à essai (20ml ,10 ml).

-fioles jaugées (10ml, 100ml), micro seringues (25µl, 50 µl), pipettes (1ml), et des pipetes pasteur.

-Viales de 1ml

-Ajustable de bouchon de viales

-Evaporateur rotatif LABOROTA 4000, Heidolph.

-Chromatographe en phase gazeuse de marque SHIMADZU, modèle GCMS 2010

-Un échantillonneur automatique AOC-20i

-Un détecteur de spectromètre de masse de marque SHIMADZU, modèle GCMS 2010

-Logiciel permettant l'acquisition et le traitement des données provenant de l'instrument.

-Colonne chromatographique capillaire d'une longueur de 30 m x 0.25 mm Di, d'épaisseur 0.25µm et de type HP5ms

4. Réactifs et étalons

4.1 Réactifs

Noms de réactif	Numéro de CAS	Pureté	Formule chimique	Marque
Sulfate de sodium	77-82-6		Na_2SO_4	Kanto chemical
Chlorure de sodium	7647-14-5	99.5%	NaCl	PROLABO
Hexane	110-54-3	PESTINORM ≥95%	C_6H_{14}	PROLABO
Acétone	016-00346	99.5%	C_3H_6O	WAKO
Ethanol	64-17-5	99.5%	C_2H_6O	Panreac
Gel de silice 40	63231-67-4	*****	*****	Sigma Aldrich

-Sulfate de sodium, Na_2SO_4, anhydre, purifié préalablement par un chauffage à 300 °C pendant 8 h

-Chlorure de sodium, NaCl, anhydre, purifié préalablement par un chauffage à 300 °C pendant 8 h

-Gaz d'Hélium (He) pour la chromatographie en phase gazeuse

-Gaz d'Azote (N_2) pour évaporer les extraits.

4.2 étalons

-Solution standard mère de 2000 ppm RESTEK (mixture de 19HAP)
-Solution standard interne Phénanthrène-D10
-Solution standard interne Fluoranthene-D10
-Solution standard interne Benzo (a) pyrène-D12
-Solution standard d'injection p-Terphenyl-D14

Noms	Numéro de CAS	Pureté	Formule chimique	Marque
Naphtalène	91-20-3	99%	$C_{10}H_8$	RESTEK
1-Methylnaphthalene	90-12-0	99%	$C_{11}H_{10}$	RESTEK
2-Methylnaphthalene	91-57-6	97%	$C_{11}H_{10}$	RESTEK
Acenaphthylene	208-96-8	99%	$C_{12}H_8$	RESTEK
Acenaphthene	83-32-9	99%	$C_{12}H_{10}$	RESTEK
Fluorene	86-73-7	99%	$C_{13}H_{10}$	RESTEK
Phénanthrène	85-01-8	99%	$C_{14}H_{10}$	RESTEK
Anthracène	120-12-7	99%	$C_{14}H_{10}$	RESTEK
Fluoranthene	206-44-0	98%	$C_{16}H_{10}$	RESTEK
Pyrène	129-00-0	98%	$C_{16}H_{10}$	RESTEK
Benz[a]anthracène	56-55-3	99%	$C_{18}H_{12}$	RESTEK
Chrysene	218-01-9	99%	$C_{18}H_{12}$	RESTEK
Benzo[b]fluoranthene	205-99-2	99%	$C_{20}H_{12}$	RESTEK
Benzo[k]fluoranthene	207-08-9	99%	$C_{20}H_{12}$	RESTEK
Benzo[a]Pyrène	50-32-8	99%	$C_{20}H_{12}$	RESTEK
3-Methylcholanthrene	56-49-5	99%	$C_{21}H_{16}$	RESTEK
Indeno (1, 2,3-cd) Pyrène	193-39-5	99%	$C_{22}H_{12}$	RESTEK
Dibenzo [a,h]anthracène	53-70-3	99%	$C_{22}H_{14}$	RESTEK
Benzo [ghi]perylene	191-24-2	99%	$C_{22}H_{12}$	RESTEK
Phénanthrène-D10	1517-22-2		$C_{14}D_{10}$	SUPELCO
Fluoranthene-D10	93951-69-0		$C_{16}D_{10}$	SUPELCO
Benzo(a) pyrène-D12	.63466-71-7		$C_{20}D_{12}$	SUPELCO
P-Terphenyl-D14	1718-51-0		$C_{18}D_{14}$	SUPELCO

5. Prélèvement et conservation

Prélever les échantillons dans des flacons en verre ayants un volume de****ml et fermés avec des bouchons contenant des joints en téflon et les conservés à 4°C. Le délai de conservation entre le prélèvement et l'extraction ne doit pas excéder 7 jours afin d'éviter les pertes.

6. principe

La détermination des hydrocarbures polycycliques aromatiques dans un échantillon de sédiment se fait par extraction ultrasonique et après purification si nécessaire et concentration avec le rotavapeur puis un jet d'azote l'extrait est analysé par chromatographe en phase gazeuse couplé à un spectre de masse.

7. Solutions standards

7.1 Préparation des solutions standards

Le domaine d'application utilisé pour le dosage par chromatographie en phase gazeuse couplé à un spectromètre de masse se situe entre 0.5 et 3 mg/l de HAP

On prépare une série de dilutions avec l'hexane dans des fioles de 10 ml à partir de la solution mère de 2000 ppm selon les proportions suivantes :

	Concentration de solution étalon (ppm)	Volume prélevé de solution mère (µl)	Volume final (ml)
(a)	0	0	10
(b)	0.5	2.5	10
(c)	1	5	10
(d)	1.5	7.5	10
(e)	2	10	10
(f)	2.5	12.5	10
(g)	3	15	10
(h)	10	50	10

Note - Ces solutions sont conservées à -25°C et utilisées pendant un mois

7.2 Préparation de solution standard d'injection

A partir de la masse pesée (0.01g) du standard d'injection p-Terphenyl-D14, on prépare une dilution avec l'hexane dans une fiole de 100 ml. **(A)**

Note - Ces solutions sont conservées à -25°C.

7.3 Préparation de solution standard interne

-A partir de la masse pesée (0.01g) du standard interne Phénanthrène-D10, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

-A partir de la masse pesée (0.01g) du standard interne Benzo (a) pyrène-D12, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

-A partir de la masse pesée (0.01g) du standard interne Fluoranthene-D10, on prépare une dilution avec l'hexane dans une fiole de 100 ml (100 ppm).

On prépare une mixture de 30 ppm de standard interne en prélevant 30 ml des trois solutions préparées dans une fiole de 100 ml et on complète avec 10 ml d'hexane. **(B)**

Note - Ces solutions sont conservées à -25°C

Préparation de la solution du standard interne ajouté aux échantillons

-A partir de la mixture de 30ppm du standard interne **(B)**on prend 10 ml et on prépare une dilution avec l'acétone dans une fiole de 100 ml (3ppm)**(C)**

Note - Cette solution est ajoutée aux échantillons d'eau

Préparation de la solution standards pour le calcule du taux de récupération

A partir de la solution (h) de 10 ppm, on prend 1ml et on prépare une dilution avec l'acétone dans une fiole de 10ml (1ppm) **(D)**

7.4 Préparation des solutions de calibration

On donne ci-dessous un tableau récapitulatif des différentes concentrations préparées dans des viales pour tracer la courbe de calibration.

Concentration de solutions standards (ppm)	Volume prélève de solutions standards (ml)	Volume ajouté de solution standard d'injection (A) (µl)	Volume ajouté de solution standard interne (Mixture) (B) (µl)
0	1 (a)	10	20
0.5	1 (b)	10	20
1	1(c)	10	20
1.5	1(d)	10	20
2	1(e)	10	20
2.5	1(f)	10	20
3	1(g)	10	20

Note - Ces solutions sont préparées pour chaque série d'échantillon analysé. Elles ne peuvent être réutilisées.

On prépare une autre série de standard pour vérifier le taux de récupération du standards interne on donne ci-dessous un tableau récapitulatif des différentes concentrations préparées dans des viales.

Volume prélevé de l'hexane (ml)	Volume prélevé de standard interne (B) (µl)	Concentration de standard interne (ppm)	Volume prélevé de standards d'injection(A) (µl)	Concentration de standard d'injection (ppm)
1	5	0.15	10	1
1	10	0.30	10	1
1	15	0.45	10	1
1	20	0.60	10	1
1	25	0.75	10	1

8. Procédure de prétraitement

8.1 Prétraitement de l'échantillon (Extraction des HAP)

Prendre un tube en verre brun de 100 ml et mettre 20g de sédiment non séché

↓

Ajouter 200µl du standard interne (C)(dilué dans l'acétone)
Agiter pour homogénéiser

↓

Ajouter 50 ml d'acétone
Agiter le tube verticalement pendant 10 min
Placer le tube dans l'ultrason pour extraction pendant 10min
Laisser reposer

↓

Récupérer la phase organique (acétone) dans une ampoule de 300 ml
Ajouter 50ml d'acétone dans la phase solide
Agiter le tube verticalement pendant 10 min
Placer le tube dans l'ultrason pour extraction pendant 10min
Laisser reposer

↓

Récupérer la phase organique dans la même ampoule
Ajouter 20 ml du mélange Ethanol/Hexane (1 :1) dans la phase solide
Récupérer la phase organique dans la même ampoule
Laver la phase solide avec 50ml d'hexane

↓

Récupérer la phase organique dans l'ampoule et jeter la phase solide
Ajouter 50ml de la solution NaCl de 5% à la phase organique
Agiter l'ampoule pendant 10min

↓

Récupérer la phase organique dans une autre ampoule de 300ml
Ajouter 50ml d'hexane dans la phase aqueuse
Mélanger pendant 10 min

↓

Récupérer la phase organique dans la même ampoule 300ml
Jeter la phase aqueuse

↓

Laver la phase organique avec 50ml d'eau ultrapure
Agiter doucement
Jeter la phase aqueuse après séparation

↓

Laver la phase organique avec 25ml d'eau ultrapure
Agiter doucement
Jeter la phase aqueuse après séparation

↓

Récupérer la phase organique dans un ballon muni d'un entonnoir contenant du coton imbibé d'hexane avec 10g du Na_2SO_4 anhydre

↓

Concentrer avec l'évaporateur rotatif à 10 ml
Concentrer avec un jet d'azote à 1 ml puis ajouter 20µl du standard d'injection (A) au moment

de l'analyse par GC/MS

8.2 Préparation du taux de récupération

On prend un tube brun de 100 ml vide et on met 10ml de l'eau pure ;on ajoute 1ml de la mixture des19HAP (D) et on procède aux mêmes étapes du prétraitement de l'échantillon comme c'est indiqué ci-dessus puis l'analyse avec le GCMS.
Le taux de récupération doit être compris entre 70% à 130% pour les standards certifier.
(Cette préparation se fait une fois par moi).

8.3 Test du blanc

On prend un tube brun de 100 ml vide et on met 200µl de standard interne et on procède aux mêmes étapes du prétraitement de l'échantillon comme c'est indiqué ci-dessus puis l'analyse avec le GCMS

8.4 Purification de l'extrait après extraction

La purification est nécessaire seulement si l'extrait est coloré

8.4.1 Préparation du gel de silice

On pèse 95g de gel de silice, on le met dans l'étuve pendant 15 h à 130°C.
On ajoute à 95g de gel de silice, 5ml de H2O (5%), on mélange pendant 30 mn jusqu'à homogénéisation et on laisse refroidir dans un dessiccateur pendant 15 hours
Ce gel est utilisé pendant 7 jours.

8.4.2 Préparation de la colonne de purification

Placer un petit bout de Cotton imbibé d'hexane à l'intérieur de la colonne
Transférer 5 g du gel de silice dilué avec une quantité d'hexane, laisser décanter puis ajouter 2 cm de Na_2SO_4
Jeter la quantité en excès de l'hexane juste au dessus du Na_2SO_4 pour éviter la formation de bulle d'air

8.4.3 Élution de l'extrait

Laver la colonne avec 10 ml d'hexane

↓

Transférer l'extrait avec une pipette pasteur
Jeter l'hexane sans dépasser le niveau de Na_2SO_4 dans la colonne à un débit de (1ml/mn)
Laver les parois du tube et de la colonne qui contient l'extrait avec 2 ml d'hexane et les transférer dans la colonne

↓

Ajouter 8ml d'hexane dans la colonne
Jeter l'hexane sans dépasser le niveau de Na_2SO_4 dans la colonne à un débit de (1ml/min)

↓

Récupérer l'extrait avec les 100 ml du mélange de 1% acétone / hexane (1ml acétone dans 99ml d'hexane, avec précision)
Goutte à goutte à 1ml/min dans un ballon juste au niveau de Na_2SO_4

↓

Concentré l'extrait avec l'évaporateur rotatif à 10 ml, le transférer dan un tube à essai gradué

↓

Concentrer les 10ml sous un jet d'azote à1ml

Ajouter 10µl de standard d'injection (A) à l'extrait dans le tube

↓

Transférer l'extrait dans une viale et effectuer l'analyse par GCMS

9. Evaluation et Mesure

9.1 Mesure

1- Vérifier les conditions d'opération des différents systèmes (GC, MS, échantillonneur)

2- Introduire les viales qui contiennent les solutions de calibration ainsi que la viale qui contient l'extrait (l'échantillon) dans l'échantillonneur automatique
3- effectuer l'analyse
4- Le benzo(b) fluoranthene et le benzo (k fluoranthene ne peuvent pas être sépare complètement donc ils ne peuvent pas être déterminé comme le benzofluroranthene
5- Les résultats d'analyse sont obtenus à l'aide d'un système informatisé de traitement de données.
6- Les résultats sont exprimés en mg/Kg

a. Les paramètres de l'échantillonneur AOC-20i

#of Rinses withpresolvent	2
#of Rinses withsolvent(post)	3
#of Rinses with sample	2
Plunguer speed (suction)	High
Viscosity com.time	0.2
Plunger speed (injection)	High
Syringe insertion speed	High
Injection Mode	Normal
Pumping Times	3
Inj. Port dwell Time	0.3sec
Terminal Air Grap	No
PlunguerWashing Speed	High
Washing Volume	8µl
Syringe suction position	0.0mm
Syringe Injection position	0.0mm
Use 3 olvent vial	1vial

b. Les paramètres chromatographiques

Colum oven empérature	45°C
Injection température	250°C
Injection mode	Splitless
Sampling Time	1.00 min
Flow control mode	Linear Velocity
Pressure	64.9 KPa
Total Flow	50 mL/min
Column flow	1.20 mL/min
Linear Velocity	40.0 cm/sec
Purge flow	4.0 mL/min
Split Ratio	- 1.0
High Pressure Injection	On
High Press. Inj .pressure	250.0 kpa
High Press. Inj .time	1.5 min
Carrier Gas Saver	OFF
Splitter Hold	OFF

Oven Temperature Program

Rate	Température (°C)	Hold Time (min)
-	45.0	1.00
45.00	130.0	0.00
12.00	180.0	0.00

7.00	240.00	0.00
12.00	320.00	4.00

c. Les paramètres du spectromètre de masse

Ion Source Température	200.00 °C
Interface temperature	250°C
Solvent Cut Time	4.50 min
Detector Gain Mode	Relative
Detector Gain	0 kv
Threshold	0
Start Time	4 .8min
End Time	25.00 min
ACQ Mode	Scan
Event Time	0.50 sec
Scan Speed	833
Start m/z	45.00
End m/z	450.00

d. Target masse, référence masse and R.T,

Nom	Target masse (m/z)	Temps de rétention (mn)	Masse de référence (m/z)
Naphtalène	128	5,0242	102
1-Methylnaphthalene	142	5,833	115
2-Methylnaphthalene	142	5,975	115
Acenaphthylene	152	7,142	150
Acenaphthene	154	7,442	153
Fluorene	166	8,383	165
Phénanthrène	178	10,508	152
Anthracène	178	10,617	152
Fluoranthene	202	13,817	200
Pyrène	202	14,483	101
Benz[a]anthracène	228	18,050	226
Chrysene	228	18,150	226
Benzo[b]fluoranthene	252	20,583	250
Benzo[k]fluoranthene	252	20,633	250
Benzo[a]Pyrène	252	21,208	250
3-Methylcholanthrene	268	21,958	269
Indeno (1, 2,3-cd) Pyrène	276	23,258	138
Dibenzo [a,h]anthracène	278	23,300	139

Benzo[ghi]perylene	276	23,717	138
Phenanthrene-D10	188	10,450	80
Fluoranthene-D10	212	13,767	106
Benzo(a) pyrene-D12	264	21,175	-
P-Terphenyl-D14	244	15,200	243

9.2 Evaluation

Le calcul des standards de calibration et des échantillons se fait par la méthode PAH « new »GCMS solution.

Les valeurs analysés sont automatiquement corrigés parle taux de récupération du standard interne. Des standards internes sont utilisés pour calculer le taux de récupération dans cette méthode on utilise trois déterres qui correspondent a un groupe de composes qui ont des temps de rétention proche voir tableau ci-dessous.

Composés	Standard internes
Acénaphtène	Phenanthrene-d10
Fluorene	Phenanthrene-d10
Phénanthrène	Phenanthrene-d10
Anthracène	Fluoanthene-d10
Fluoranthene	Fluoanthene-d10
Pyrene	Fluoanthene-d10
Benzo(a) anthracene	Benzo(a)pyrene-d10
crysene	Benzo(a)pyrene-d10
Benzo[b]fluoranthene	Benzo(a)pyrene-d10
Benzo[k]fluoranthene	Benzo(a)pyrene-d10
Benzo[a]Pyrène	Benzo(a)pyrene-d10
3-Methylcholanthrene	Benzo(a)pyrene-d10
Indeno (1, 2,3-cd) Pyrène	Benzo(a)pyrene-d10
Dibenzo [a,h]anthracène	Benzo(a)pyrene-d10
Benzo[ghi]perylene	Benzo(a)pyrene-d10

Vérifier le taux de récupération du standard interne par la deuxième courbe de calibration (7.4)
On copie le dossier pour chaque échantillon et batch processing pour la deuxième courbe de calibration puis copier datafile pour chaque échantillon en utilisant GCMS solution méthode « new PAHD » puis le calcule de taux de la récupération de pour chaque déterré en utilisant l'équation ci-dessous.

La fiabilité des résultats est vérifiée par le taux de récupération des standards internes misent dans l'échantillon selon la formule suivante :

$$\frac{\textbf{Concentration trouvée}}{\textbf{Concentration théorique}} \textbf{*100}$$

Si le taux est compris ente 70% 130% le résultat est fiable.

Expression des résultats :

$$Wn = \frac{m_{ex}}{m * \frac{Pw_s}{100}}$$

Wn : la concentration de chaque composé de HAP trouvé dans le sédiment séché en µg/g (mg/Kg)
$\mathbf{m_{ex}}$: la quantité de chaque composé de HAP dans l'extrait en µg.
m : la masse du sédiment utilisé pour l'extraction en g.
$\mathbf{Pw_s}$: le pourcentage massique d'humidité présente dans le sédiment séché (ce facteur n'est pas utilisé si le sédiment est déjà séché).

$$Pw_s = \frac{W_d}{W} * 100$$

W : la masse du sédiment avant séchage.
$\mathbf{W_d}$ **:** la masse du sédiment après séchage.

$$Pw = \frac{W - W_d}{W} * 100$$

Pw : le pourcentage en eau

10. limites de Détection

noms	Limites de détection µg/Kg	Limites de quantification µg/Kg
Acénaphtènes	30	70
Fluorene	6	20
Phénanthrène	1	4
Anthracène	2	6
Fluoranthene	0.6	2
Pyrène	1	4
Benz[a]anthracène	1	4
Chrysene	1	3
Benzo[b]fluoranthene	7	20
Benzo[a]Pyrène	7	30
3-Methylcholanthrene	7	30
Indeno (1, 2,3-cd) Pyrène	8	30
Dibenzo [a, h] anthracène	10	30
Benzo [ghi] perylene	20	50

11. Référence

ISO 18287

12. Enregistrement et Révisions des Modes Opératoires Normalisés

Février 2010: première Révision faite par LRC

Février 2012: deuxieme Révision faite par LRC

Titre : maintenance du septum

Les objectifs :	**Fait par :** **M^me NECHAOUNI Leila** **M^me KIMRI Leila** **M^me OMRI Linda**
Date de préparation **Octobre 2011**	**Responsable**
Date d'approbation	

1. Appareillage et matériel

Chromatographie en phase gazeuse de marque SHIMADZU .modéleGCMS 2010plus

2. procédure : Changement de septum

1		Arrêter l'appareil
2		Desserrer l'écrou du septum et le retirer

2-1		image illustrant l'état de la machine après avoir retirer l'écrou
3		Retirer l'aiguille guide
4		Retirer le septum utilisé et le remplacer par un nouveau

5		Joindre l'aiguille guide
6		Serrer l'écrou du septum le plus fort possible en le tournant à 180°
7		Démarrer l'appareil

Titre : changement de linéaire

Les objectifs :	**Fait par :** **M^me NECHAOUNI Leila** **M^me KIMRI Leila** **M^me OMRI Linda**
Date de préparation **Octobre 2011**	**Responsable**
Date d'approbation	

Maintenance : remplacement de linear (glass insert)

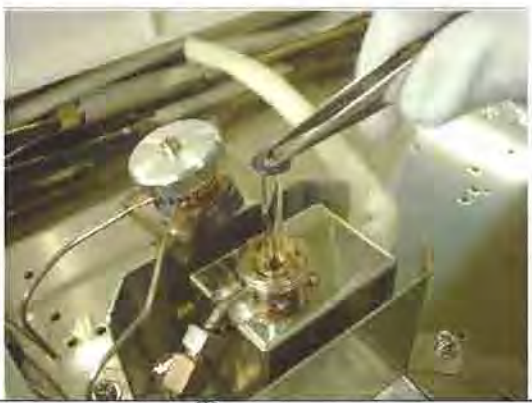
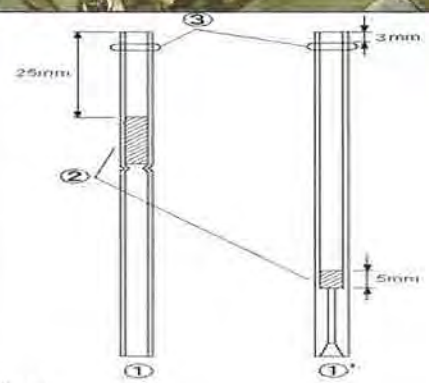
1-Appareillage et matériel:

Chromatographe en phase gazeuse de marque shimadzu.modelGCMS-2010 PLUS

2-Procédure :

Remplacement de linear (glass insert)

1		arrêter l'appareil
2		retirer l'écrou de linear en le tournant avec une clef à Monnet dans le sens contraire des aiguilles d'une montre avec une seule main
2.1		image qui illustre l'état de la machine après avoir retirer l'écrou de linear

		
3		retire linear avec une pince
4	 Split splitless	Insérer une quantité appropriée de la laine de silica dans un linear propre ou neuf (2g) Placer un joint dans la partie supérieur du linear

5		Le joint est placé approximativement à 4mm de la partie supérieur du linear Insérer linear dans la chambre de vaporisation en utilisant une pince en poussant doucement jusqu'a ce qui' il touche le fond.
6		remettre l'écrou du linear Serrer l'écrou manuellement Sécuriser l'écrou en le tournant de 45° avec la clef a Monnet
7		Démarrer l'appareil

Titre : Connexion de la colonne

	Fait par : **M^me^ NECHAOUNI Leila** **M^me^ KIMRI Leila** **M^me^ OMRI Linda**
Date de préparation **Octobre 2011**	**Responsable**
Date d'approbation	

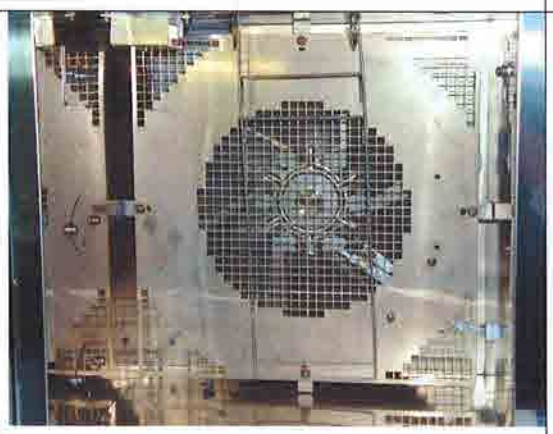
1-Appareilage et materiel:

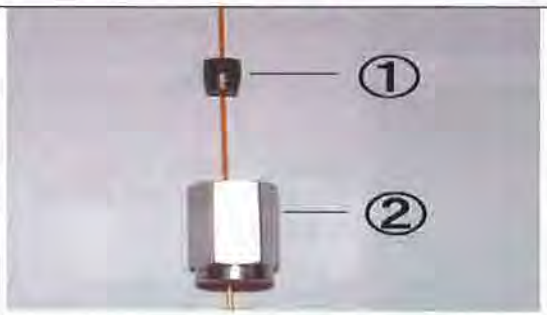
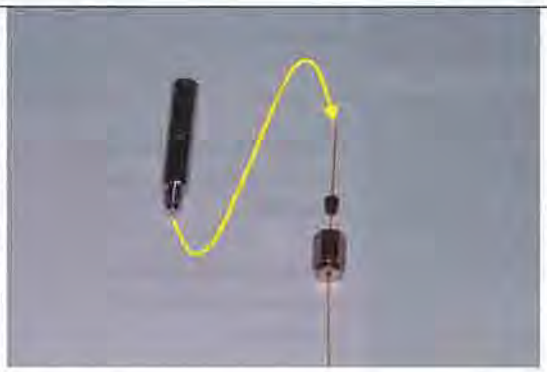
Chromatographe en phase gazeuse de marque shimadzu.modelGCMS-2010 PLUS

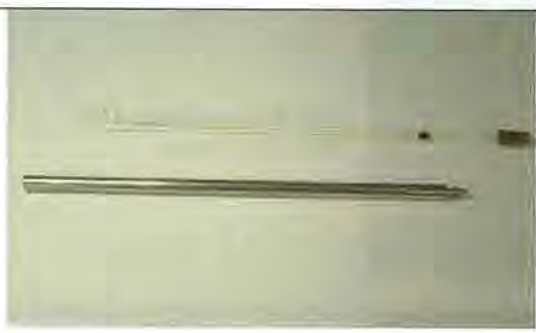
2-Procédure :

Changement de la colonne

1		arrêter l'appareil
2		Tirer le loquet du coté droit de la porte du four vers le bas pour ouvrir

3		Retirer l'écrou du coté injecteur
4		Retirer l'écrou du coté MS
5		Retirer la colonne de son emplacement

6		Glisser l'écrou dans le coté injecteur de la colonne Glisser une férrule vespel Orienter la férrule vespel de sorte qu'elle pénètre bien dans l'écrou
7		1-férules vespel 2-écrou
8		Insérer la **colonne JIG** dans la colonne
9		-Viser la colonne JIG avec au moins 1cm de colonne qui dépasse -Serrer la colonne JIG en doucement utilisant deux clefs à Monnet jusqu'à ce qu'elle ne tourne plus Coté gauche : clef 6mm (6x8mm) Coté droit : clef ¼-inch clef(1/4x5/16inch) -Couper la partie protubérante avec un cutteur de colonne

		-Marquer avec un septum le niveau à la fin de l'écrou
10		Retire la colonne et l'écrou de la colonne JIG Assurer que la férrule et l'écrou ne se détachent pas de la colonne Assurer que le niveau marqué est bien fixé
11		Glisser l'écrou et une férrule dans le coté MS de la colonne Procéder de la même manière que le coté injecteur
12		Insérer la **colonne JIG** dans la colonne pour le coté MS Procéder de la même manière que le coté injecteur
12-1		Avant de joindre la colonne essuyer ses deux bouts avec une gaze imbibée d'acétone S'assurer que le niveau marqué n'est pas déplacé

13		fixer la colonne joindre le coté injecteur de la colonne joindre le coté MS de la colonne viser les deux écrous à la main puis bien serrer avec une clef à Monnet à 20° à 40° si une nouvelle férule est placée visé avec la main puis serré avec une clef en tournant avec un angle de 360°
14		Assurer que les écrous sont bien serrer du coté MS et du coté injecteur Fermer la porte du four de GC Démarrer l'appareil
15		Si une nouvelle férule vespel est utilisée, une fuite d'aire causée par le cycle d'échauffement peut se développer au niveau de cette férule En cas de fuite, réchauffer la colonne du four et la chambre de vaporisation de l'échantillon à la température utilisée lors de l'analyse Laisser la colonne du four refroidir puis serrer les écrous des cotés injecteur et MS à nouveau.

Titre : Nettoyage linéaire

Les objectifs :	**Fait par :** **M^me^ NECHAOUNI Leila** **M^me^ KIMRI Leila** **M^me^ OMRI Linda**
Date de préparation **Octobre 2011**	**Responsable**
Date d'approbation	

Maintenance : nettoyage de linear (glass insert)

Le nettoyage du linear split :
Méthode 1 :

- Enlevé la laine à l'intérieur du linéaire
- Lavé avec de l'acétone
- Laisser trempé dans un bécher avec de l'acétone et le mettre dans l'ultrason

Méthode 2 :

- -Lavé avec un détergent alcalin, laissé tremper toute une nuit dans la solution de détergent
- Rince avec de l'eau puis avec de l'acétone
-
- -si linéaire est trop sale trempe dans du HNO3 1 Mole/ L laisser trempé 8 heures puis rincée avec de l'eau distillée puis acétone

Titre : Procédure de connexion

Les objectifs : **Analyse de l'eau**	**Fait par :** **M^me NECHAOUNI Leila** **M^me KIMRI Leila** **M^me OMRI Linda**
Date de préparation **Octobre 2011**	**Responsable**
Date d'approbation	

Maintenance
1-Appareillage et matériel:

Chromatographe en phase gazeuse de marque shimadzu.modelGCMS-2010 PLUS

2-Procédure :

Procédure opératoire pour passer de P& T à l'injection directe

1. Changer la connexion des câbles au niveau du GC

Connexion du P& T

1- rouge
2- noire
3- blanc
4- vert

Connexion directe AOC-20i

1- jaune
2- rouge
3- blanc
4- noire

2. Vérifier le septum et le linear (nombre d'utilisation)
3. Connecter la colonne coté injecteur SPL2 (SPL1pour P& T)
4. Sélectionner AOC-20i et SPL2 dans le système de configuration
5. Ouvrir la bouteille d'hélium
6. Démarrer le vacuum après une heure
7. Vérifier les fuites water / air avec le tuning (15minute à 2jours)
8. Réaliser l'autotuning si les conditions sont bonnes à 150μA (60 μA pour P& T)
9. Vérifier les résultats de l'autotuning à partir du manuel

Annex 2-1-2-1　Record of training for FTIR analysis

Procés verbal de la formation

Enregistrement de Formation sur 1er année

2ème Travail
C/P＝Bensouilah Quahiba, Anane Radhia

Date	Jour	
2/7	Dim	Elaboration et dépôt du SOP, Power Point.
2/8	Lun	Préparation de la pastille KBr, après correction, suivant la procédure en utilisant de l'acide citrique.
2/9	Mar	Mise au point de la technique pratiquée le 8/02/2010 et discussion sur les spectres Acide citrique.
2/10	Mer	Tirage des spectres selon AIST
2/11	Jeu	Comparaison des spectres: analyse TOC
2/14	Dim	Acquisition des documents sur le FTIR
2/15	Lun	Projection sur l'interprétation du FTIR. Actualisation du SOP selon la projection d'une vidéo tirée d'Internet
2/16	Mar	Installation des équipements (FTIR, PC, presse) après nettoyage. Rédaction du rapport partiel de formation en cours.
2/17	Mer	Dépôt du rapport partiel de formation et traduction avec interprétation des documents remis. Analyse d'échantillons d'autres paramètres.
2/18	Jeu	Analyse d'échantillons DBO_5
2/21	Dim	Suite des analyses (phénol)
2/22	Lun	Réunion de travail avec Mr Tireche (Directeur de l'ONEDD) Analyses CN et Phénol
2/23	Mar	Analyse NTK, DBO5 et phénol
2/24	Mer	Analyse NTK, (CN-) et phénol
2/25	Jeu	Séminaire
2/28	Dim	Dépôt de tous les documents traduits, mise au point de la note sur la gestion du FTIR Réunion d'évaluation de la formation avec Mr Tireche
3/1	Lun	Présentation orale d'un échantillon liquide Préparation avec manipulation d'un échantillon solide Réactualisation du SPOP Analyse, DBO_5, Phénol, NTK
3/2	Mar	Préparation avec manipulation d'échantillons huileux Analyses, phénol, NTK, TOC, TN
3/3	Mer	Analyse d'échantillon, modification du SOP de préparation des échantillons
3/4	Jeu	Exemples de quelques spectres inorganiques. Initiation à la méthode ATR

Enregistrement de Formation sur 2ème année

1er Travail

C/P＝Bensouilah Quahiba, Anane Radhia

Date	Jour	
6/3	Jeu	Remise du SOP, versificatrice des pastilles ,comparaisons et interprétation de quelque spectres inorganique
6/6	Dim	Recommandation: 1- établissement d'un tableau de collecte d'information sur les échantillons. 2- consultation du panorama air analyse. 3- établir les grandes lignes de la présentation de février. 4- préparer un manuel de gestion interne. 5- continuer à préparer les Pastilles en fonction du plan de charge

2ème Travail

C/P＝Bensouilah Quahiba, Anane Radhia, Bouadi Fatima Zohra*

Date	Jour	
10/3	Dim	- Inspection du FTIR - Procédure d'analyse sur l'appareil - Mise a jour et analyser des échantillons préparer
10/4	Lun	- Suite de analyse des echs avec une petite interprétation
10/5	Mar	- Prise d'essai de plusieurs ech pour analyse
10/6	Mer	- Refaire l'analyse de 4 pastilles diluées avec une petite interprétation, initialisation sur ATR
10/7	Jeu	- Préparation de qlqe pastilles . - consultation du hand books
10/10	Dim	- Analyse des pastilles. - initialisation et analyse sur l'ATR - préparation et analyse de 3 produits chimique de qualité
10/11	Lun	- Préparation et analyse de quelque pastilles (suite) - préparation d'une fiche technique FTIR
10/12	Mar	- Suite de la saisie (liste d'échantillons) - vérification de qlques pics sur le hand book
10/13	Mer	- Suite de la saisie –correction des noms des spectres
10/14	Jeu	- Finalisation de la liste des échantillons . - Impression des deux BKRD sur CD.
10/17	Dim	- Remise des SOP FTIR, maintenance, stockage ATR KBr . - Correction des SOP.
1018	Lun	- Réunion avec DJ .la mise au point sur les SOP et la validation de ce dernier .
10/19	Mar	- Finalisation du rapport de la formation. les rocomondations de l'expert sur FTIR
10/20	Mer	- Consultation de la recommandation -interprétation de qlq spectres

3ème Travail
C/P＝Bensouilah Quahiba, Anane Radhia

Date	Jour	Matin	Après-midi
2/1	Mar	-Transfer technique avec les données bibliothèquaire	Douane
2/2	Mer	- 1er Essaie de l'utilisation de la clé IR analyse. - Initialisation sur l'interprétation de quelque spectre organique en utilisant la clé (ATR) et KBr). - Interprétation de quelque spectre en utilisant le hand book (inorganique).	Douane Rédaction du templaite de la technique KBr.
2/3	Jeu	- Prétraitement des échantillons - Analyse des spectres en utilisant la bibliothèque - Réutilisation de la clé a fin d'interprété les spectres organique. - Mode opératoire théorique sur l'extraction des hydrocarbures dans le sole. - Mode opératoire théorique sur l'extraction des hydrocarbures dans les eaux naturels. - Conseille pour l'utilisation du dispositif ATR - Interprétation de quelque spectre et utilisation de la clé pour interprétation	
2/6	Dim	Réunion du contrôle de qualité	- Préparation des SOP et des manuels - Templaite
2/7	Lun	- Extraction des composants organiques - Extraction des matières organique non volatile dans 2 échantillons solides.	- Analyse des spectres en utilisant la bibliothèque -Analyse et interprétation des 2 spectres en utilisant la clé. -Mise au point du tableau d'évaluation
2/8	Mar	- mise à jour du tableau (liste des échantillons 2010 et 2011) avec séparation entre mode ATR, KBr - Installation des recommandations et des rapports dans le PC (FTIR). (Absence Ouahiba)	
2/9	Mer	- Révision de toutes les recommandations installées au PC (FTIR). - Remise de 4 documents sous forme électronique contenant les noms des matières organiques non volatiles sur SDBS. - Finaliser le templaite (après midi) (Absence Ouahiba)	
2/10	Jeu	- Révisions sur PC FTIR: SOP: ATR. KBr - Révisions sur PC FTIR: manuel: stockage et maintenance - Révisions sur PC FTIR: templaite KBr - Révisions sur PC FTIR: liste des échantillons partagés (Absence Ouahiba)	
2/13	Dim	Réunion du contrôle de qualité	- Analyse des spectres en utilisant la bibliothèque - Correction manuel maintenance. - Réunion avec DG et JICA - Correction SOP KBr ATR
2/14	Lun	Analyse des spectres en utilisant la bibliothèque	
2/15	Mar	Jour férié en Algérie	
2/16	Mer	Analyse des spectres en utilisant la bibliothèque	
2/17	Jeu	Analyse des spectres en utilisant la bibliothèque	
2/20	Dim	Analyse des spectres en utilisant la bibliothèque	
2/21	Lun	Séminaire GCMS	Séminaire FTIR

Annex 2-1-2-2　Mid-term evaluation for FTIR analysis

Algérie - Projet de développement de la capacité de suivi de l'environnement (Phase 2) ; Tableau d'évaluation de l'avancement du transfert technique (Output 1: FTIR)

Rédigé le 21 Février 2011

■ Evaluation de l'avancement du transfert technique au niveau organisationnel

▼ Début de Projet ▼ A mi-période de Projet (A la fin du transfert technique) ▼ A la fin du Projet

Noms du équipement	Eléments de transfert technique	Contenu d'exécution du transfert technique	Année / Mois / Période de séjour des experts sur place	Perspectives à la fin du Projet
			2009: 10 11 12 | 2010: 1 2 3 4 5 6 7 8 9 10 11 12 | 1 2 3 | 2011: 4 5 6 7 8 9 10 11 12 | 2012: 1 2 3 4 5 6 7 8 9	
FTIR	Manipulation des matériels, etc.	Manipulation des matériels	A cause de dysfonctionnement du matériel, explication donnée sur les points à noter lors de la manipulation avec des documents et des vidéos | Pas réalisée à cause de dysfonctionnement du matériel | Suffisamment maîtrisé	Nécessite de demander une maintenance régulière au fabricant
		Mode d'emploi des matériels, nombre de pages	Instruction donnée pour la méthode de rédaction du manuel de manipulation simplifiée | Manuel de manipulation simplifiée de la métho de des boulettes de Kbr rédigé | Mise à jour du manuel de manipulation simplifiée	
		Manuel de maintenance des matériels, nombre de pages	Instruction donnée pour la méthode de rédaction du manuel de maintenance de matériels | Manuel de maintenance de matériels en cours de rédaction | Manuel de maintenance rédigé	
	Mesure des composés organiques non volatils des é chantillons par la m éthode des boulettes de KBr	SOP de méthode KBr, nombre de pages	Début de rédaction de la SOP (procédure opé rationnelle permanente) | SOP, première édition rédigée | SOP (procédure opérationnelle permanente) révisée	
		Technique de conditionnement et de prétraitement d'échantillons	Instruction donnée pour la méthode de prétraitement des échantillons de dépôt de fond, aquatiques, et huileux, et pour la préparation des boulettes de KBr | Instruction donnée pour la méthode de prétraitement des échantillons de dépôt de fond, aquatiques, et huileux, et pour la préparation des boulettes de KBr | Suffisamment maîtrisé	
		Analyse de matières standard et d'échantillons réels Nombre de données	45 boulettes d'échantillons réels préparées. Mesure pas réalisée à cause de dysfonctionnement du spectre de puissance | Pas réalisée à cause de dysfonctionnement du matériel | 45 échantillons réels 5 échantillons standard	
	Mesure des composés organiques non volatils des é chantillons par la m éthode d'ATR	SOP de la méthode d'ATR, nombre de pages	A cause de dysfonctionnement du matériel, l'explication sur la méthode de monture et sur la manipulation est donnée avec un manuel et une vidé o. | Début de rédaction de la SOP | SOP (procédure opérationnelle permanente) révisée	
		Technique de conditionnement et de prétraitement d'échantillons	Explication donnée sur la méthode de prétraitement des échantillons huileux | Instruction donnée sur la méthode de prétraitement des échantillons huileux et la méthode de nettoyage après l'utilisation | Suffisamment maîtrisé	
		Analyses de matières standard et d'échantillons réels Nombre de données	Pas réalisée à cause de dysfonctionnement du maté riel | Pas réalisée à cause de dysfonctionnement du matériel | 50 échantillons réels 10 échantillons standard	
	Appartenance du spectre des composés organiques non volatils (analyse/é valuation)	Compréhension du changement de nombre d'ondes à absorption par l'indice de liaison de carbone-hydrogè ne	Explication donnée avec des documents et des vidé os | Explication donnée avec des documents et des vidéos | Presque maîtrisé	
		Compréhension de la disposition de la bande d'absorption caractéristique et de la forme concernant les groupes d'atomes tels que silicate, sulfate, carbonate, etc.	Explication donnée avec des documents et des vidé os | Explication donnée avec des documents et des vidéos | Presque maîtrisé	
		Appartenance du spectre en utilisant une bibliothè que spectrale et un recueil de données	Pas réalisé à cause de dysfonctionnement du matériel et de retard de fourniture de matériaux | Pas réalisée à cause de dysfonctionnement du matériel | Presque maîtrisé	
	Conservation des échantillons et de la bibliothèque spectrale	Conservation des échantillons mesurés	Explication donnée sur comment obtenir des informations lors du prélèvement des échantillons et la méthode de classification | Personne en charge capable de réaliser | Personne en charge capable de réaliser	
		Conservation de boulettes de Kbr mesurées	Explication donnée sur la méthode de conservation des boulettes d'échantillon | Personne en charge capable de réaliser | Personne en charge capable de réaliser	
		Gestion de données spectrales	Instruction donnée concernant le moment de prélè vement d'échantillons et la méthode de gestion des résultats d'analyse des données du spectre | Instruction donnée concernant le moment de prélè vement d'échantillons et la méthode de gestion des résultats d'analyse des données du spectre | Personne en charge capable de réaliser	

■ **Evaluation de l'avancement du transfert technique au niveau individuel**	Critères d'évaluation （par les experts japonais）	+ : niveau débutant (quasiment pas de connaissance ni expérience) ++ : niveau intermédiaire (Technique acquise mais nécessite plus d'expérience) +++ : niveau supérieur (ayant la compétence technique suffisante et capable d'instruire les autres)					
Personne en charge	Période d'évaluation (année, mois)	Utilisation des matériels, etc	Mesure des composés organiques non volatils des échantillons par la méthode des boulettes de KBr	Mesure des composés organiques non volatils des échantillons par la méthode d'ATR	Appartenance du spectre des composés organiques non volatils (analyse/évaluation)	Conservation des échantillons et de la bibliothèque spectrale	Perspectives à la fin du Projet
Anane Radhia	Au début du Projet (octobre 2009)	+	+	+	+	+	
	A la fin du transfert technique （mars 2011）	++	++	++	++	++	
	A la fin du Projet （juin 2012）						
Bensouilah Quahiba	Au début du Projet (octobre 2009)	+	+	+	+	+	
	A la fin du transfert technique （mars 2011）	++	++	++	++	++	
	A la fin du Projet （juin 2012）						
Bouadi Fatima Zohra	Au début du Projet (octobre 2009)	+	+	+	+	+	
	A la fin du transfert technique （mars 2011）	absence	absence	absence	absence	absence	
	A la fin du Projet （juin 2012）						