ANNEXES

ANNEX (1)

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MINISTRY OF HOUSING, USE OF LAND AND ENVIRONMENT
ORIENTAL REPUBLIC OF URUGUAY

JAPAN INTERNATIONAL COOPERATION AGENCY (JICA)

"PROJECT ON CAPACITY DEVELOPMENT FOR WATER QUALITY MANAGEMENT IN MONTEVIDEO AND METROPOLITAN AREA"

JOINT WORK AGREEMENT
ON
WATER QUALITY MONITORING
BETWEEN
THE MINISTRY OF HOUSING, USE OF LAND AND ENVIRONMENT (MVOTMA)
AND
THE MUNICIPALITIES OF MONTEVIDEO, CANELONES, SAN JOSÉ, FLORIDA AND LAVALLEJA

SEPTEMBER 11, 2006

In the city of Montevideo, on September 11th, 2006, the **Ministry of Housing Use of Land and Environment** (hereinafter referred to as **MVOTMA**), represented in this legal act by the Minister Mariano Arana, ***party of the first part***; the **Municipality of Montevideo** (hereinafter referred to as **IMM**), represented in this legal act by the Mayor of the Municipality, Dr. Ricardo Ehrlich; the **Municipality of Canelones** (hereinafter referred to as **IMC**), represented in this legal act by the Mayor of the Municipality, Dr. Marcos Carámbula; the **Municipality of San José** (hereinafter referred to as **IMSJ**), represented in this legal act by the Mayor of the Municipality, Mr. Juan Chiruchi; the **Municipality of Florida** (hereinafter referred to as **IMF**), represented in this legal act by the Mayor of the Municipality, Prof. Juan Giachetto; and the **Municipality of Lavalleja** (hereinafter referred to as **IML**), represented in this legal act by the Interim Mayor of the Municipality, Dr. Oscar Ximenez, **all parties of the second part,** agree to execute this Joint Work Agreement, subject to the terms and conditions stated below:

1. BACKGROUND

In Montevideo, on December 5th, 2002, Mr. Saúl Irureta, the Minister of MVOTMA at that time, and Mr. Masahiro Ohta, a representative of the Japan International Cooperation Agency (hereinafter referred to as JICA), signed an agreement for the Study of the Capacity Development for Water Quality Management in Montevideo and the Metropolitan Area.

Within the framework of said agreement, from October 2003, The National Directorate of Environment (hereinafter referred to as DINAMA) and JICA Project team, with the collaboration of the relevant institutions, have been developing the agreed surveys and activities, in order to elaborate by the end of 2006 an Integrated Master Plan on the Capacity Development for Water Quality Management.

This Plan has defined some essential results, including the establishment of a water quality monitoring system for the basins of the Santa Lucía river, Carrasco river and Pando river; the set-up of a collaborative system for the implementation of sampling, analysis and evaluation of water quality; and the establishment of an integrated system on water quality information. To attain such results, DINAMA, IMM, IMC, IMSJ, IMF and IML have been working together regarding water quality monitoring (hereinafter referred to as Joint Work), with the purpose of preserving and improve the quality of the water bodies belonging to the Santa Lucía river, Carrasco river and Pando river basins.

2. OBJECTIVES OF JOINT WORK

Water quality monitoring is necessary to know the quality status of the water bodies and to manage them in a sustainable way. Considering that the management of the Santa Lucia River, Carrasco Stream and Pando Stream Basins involves several local governments as well as DINAMA, and the capacity and need differences regarding water quality monitoring in the institutions involved, the Joint Work aims at cooperating in order to assure a sustainable monitoring of the quality of water bodies, promoting the improvement of the participant institutions' capacity

3. COOPERATION SCOPE

DINAMA and IMM agree to coordinate the water quality monitoring plans, in order to make compatible the dates and sampling frequencies and to facilitate data integration regarding the basins under study.

DINAMA, IMC, IMSJ, IMF and IML agree to carry out the monitoring and the analysis of water and sediments, including sampling, transport, laboratory analysis, etc. in close cooperation, according to the water quality monitoring executive plan, attached to this file.

DINAMA, IMM, IMC, IMSJ, IMF and IML agree to reciprocally exchange the measurements and analysis results obtained by the Joint Work.

4. DEMARCATION OF THE WORK

DINAMA, IMM, IMC, IMSJ, IMF and IML agree to carry out the Joint Work according to the following frame:

(1) IMC, IMSJ, IMF and IML shall be in charge of the water sampling, the sample transport and the measurement/analysis at the laboratory of some of the water parameters, according to the Water Quality Monitoring Executive Plan attached to the present file,

(2) IMM shall be in charge of the water sampling, the sample transport and the measurement/analysis of the water parameters in the Environmental Quality Laboratory Service, according to their monitoring plan for Montevideo and in congruity with the Water Quality Monitoring Executive Plan, attached to the present file,

(3) DINAMA shall be in charge of the measurement/analysis of the water parameters in the laboratory, in congruity with the Water Quality Monitoring Executive Plan, attached to the present file,

(4) DINAMA shall carry out the technology transfer to IMM, IMC, IMSJ, IMF and IML, related to water sampling techniques, laboratory measurement/analysis, whenever needed, and the informatics tools for the water quality and laboratory information system (SISICA and SISILAB),

(5) Any necessary expenses for the materials, reagents and work hours that may arise from the works shall be covered by each party as corresponding, and

(6) The MVOTMA, IMM, IMC, IMSJ, IMF and IML may realize mutual consultation, on any matter that could arise from, or related to, the Joint Work.

5. UNITS IN CHARGE

The MVOTMA, IMM, IMC, IMSJ, IMF and IML, shall cooperate in the Joint Work execution through the responsible units, which have been timely established by each

institution. Nevertheless, each institution would be able to re define such responsibility, if it is deemed necessary.

6. SUPPORTING UNIT

In the frame of this agreement, the Agenda Metropolitan Program, shall develop the following functions: a) Promoting administrative, logistic and facilitation support; b) Promoting the spreading of the information generated within the framework of this agreement, in the involved Departments; c) Monitoring the needs of the information users community; and d) Facilitating the coordination among the departmental governments of the basin.

7. AGREEMENT VALIDITY

This agreement shall be in force for two years, being renewed automatically by equal periods if none of the parties communicates to the other one the intention to not continue with the agreement, at least, sixty days prior to the expiration date.

In case a notification occurs in such sense, only the partial resolution of this agreement shall be deemed operated. The other parties shall analyse the continuation of this agreement without the participation of the rescinding party, within 60 days following the reception date of the last notification, in case they deem necessary to make changes on it.

8. ATTACHMENTS

The Water Quality Monitoring Executive Plan is attached, containing the following annexes included, which are a part of this agreement.

Annex 1: Selection Criteria for the Water Quality Monitoring Points and Measurement Parameters.

Annex 2: Justification for the Selection of Monitoring Points

Annex 3: Geographical Information of the Monitoring Points

Annex 4: Location Map of the Monitoring Points

Annex 5: Analysed Parameters in each Monitoring Point.

IN WITNESS THEREOF, the parties sign this agreement in Montevideo, on September 11th, 2006.

Mariano Arana, Arch.
Minister of Housing, Use of Land and Environment

Dr. Ricardo Ehrlich
Mayor of the Municipality Montevideo

Dr. Marcos Carámbula
Mayor of the Municipality of Canelones

Mr. Juan Chiruchi
Mayor of the Municipality of San José

Prof. Juan F. Giachetto
Mayor of the Municipality of Florida

Dr. Oscar Ximenez
Interim Mayor of the Municipality of Lavalleja

WITNESSES of HONOUR

Mr. Shinichi Kuyama
Ambassador of Japan

Mr. Keiji Sasabe
JICA Project Team Leader

EXECUTIVE PLAN OF WATER QUALITY MONITORING

Santa Lucia River, Carrasco Stream and Pando Stream Basins

1. INTRODUCTION

In order to formalize the joint water quality monitoring between the different institutions and to make this sustainable in the future, the present "Water Quality Monitoring Final Plan" for the Santa Lucia River Basin has been elaborated. The abovementioned Plan represents the technical support for the signature of a Cooperation Agreement between the institutions. This Plan has been elaborated considering not only the information obtained during the Monitoring Pilot Plan, but also the current capacity of DINAMA and the Municipalities involved.

The Executive Plan design and implementation are a product of a Technical Cooperation Project among the Japan International Cooperation Agency (JICA), the National Directorate of Environment (DINAMA) and the Municipalities of the Santa Lucia River Basin, which aim is the Capacity Development for Water Quality Management in Montevideo and Metropolitan Area.

The Monitoring Executive Plan of the Santa Lucia River Basin shall be implemented as a joint work between DINAMA and the following Municipalities: Canelones (IMC), San Jose (IMSJ), Florida (IMF) Lavalleja (IML) and Montevideo (IMM).

The objective of the Executive Plan is to implement a joint Monitoring System between DINAMA and Municipalities. The System consists of a monitoring network that shall allow to obtain information in a continuous and sustainable way about the quality evolution of the Santa Lucia River. The goal is to maintain this program at a long term, managed by the Municipalities, so that in the future, DINAMA and the Municipalities can get systematic information about the basin.

The Monitoring Pilot Plan, which lasted one year, allowed to establish the monitoring network in a stronger way, with technical reliability, and the lessons learned enabled the improvement of the original monitoring plan. In order to establish this system, the topics and specific details of the joint work were discussed among DINAMA and the Municipalities involved, regarding the organization implementation, the work demarcation, etc. The mutually agreed Executive Plan is described further on.

2. SELECTION OF SAMPLING POINTS

Monitoring points have been selected according to the following criteria:

- DINAMA's expertise considering previous monitoring.
- Municipalities' expertise, of those who are now carrying out or carried out the monitoring in the past

- Current maximum capacity of DINAMA and Municipalities regarding sampling and measurement,
- Accessibility to the sampling points.

More detailed criteria applied for the selection of monitoring points are shown in **Annex 1**, while **Annex 2** presents the reasons for the selected points.

As a result of a series of field surveys and coordination among the different actors, **Table 2.1** shows the number of points selected for each water basin:

Table 2.1. Number of Selected Monitoring Points

	Water basin						Total Number of Water Monitoring Points	Total Number of Sediment Monitoring Points
Municipality	Sta. Lucia River		Pando Stream		Carrasco Stream			
	W	S	W	S	W	S		
IMM	5	-			5	-	10	
IMC	8	5	4	3			12	8
IMSJ	9	7					9	7
IMF	5	3					5	3
IML	6	2					6	2
Total	33	17	4	3			42	20

W: Number of Water Monitoring Points
S: Number of Sediment Monitoring Points

Moreover, the data of their environmental monitoring are added to the database by IMM, which covers several sites that are not included in the basin itself, but which provide regional environmental information. There is a coordination for the monitoring schedule carried out by IMM and the one carried out by DINAMA and IMC, in order to obtain complementary data and to include them as a unique seasonal sampling.

In the environmental monitoring program of IMM, a routine, periodical monitoring is carried out as part of their working programs, including water and sediment sampling (streams and bays), measurement, evaluation and annual report.

The coordinates and the geographical features of the monitoring points are shown in **Annex 3**, while their location is presented in the map of **Annex 4**.

3. SAMPLING WORKS AND THEIR FREQUENCY

The monitoring schemes for IMC, IMSJ, IMF and IML, are described below. The monitoring system for IMM is well designed and it has already been executed for several years, so, it has not been altered for the Santa Lucia River network.

3.1 Water Sampling

Water samples are taken from the defined monitoring point. These water samples must be taken by the Municipalities.

Each Laboratory involved in the analytical determinations shall provide the appropriate sampling containers, for the parameters that shall be analyzed in such laboratory.

Although Municipalities are expected to take water samples within one or two days, the exact date and time are decided according to a rotation program, with the frequencies described below in this document.

The transportation of samples to be analyzed by DINAMA´s laboratory is managed by the Municipalities, using their own vehicles or public transport.

The locations downstream Santa Lucia River, as well as in Pando Stream, require water sampling onboard, using a boat, due to the lack of access by land. Since neither IMC nor IMSJ have a boat, DINAMA shall provide its own boat for this water sampling, in 4 locations along the downstream sector of the Santa Lucia River and 1 location in Pando Stream, until the Municipalities involved are provided with an available craft.

3.2 Sediment Sampling

Sediment sampling is currently carried out by DINAMA, because the involved Municipalities do not have the appropriate instruments. The set of instruments is scheduled to be provided at the beginning of 2007, in the frame of a strengthening program of the Water Quality Department of DINAMA to the Municipalities, so that Municipalities are enabled to include this activity from then on.

The training for the handling and conditioning of samples was already carried out during the pilot sampling. However, DINAMA could provide additional training if necessary.

3.3 Frequency

The sampling frequency has been established quarterly, so one water and sediment sampling shall be carried out each season.

The sampling schedule to be executed by Municipalities is the following:

- IMC: on the fourth Wednesdays and Thursdays of July, October, January and April.
- IMSJ, IMF and IML: on the third Wednesdays and Thursdays of July, October, January and April.

This schedule was established with the agreement of the institutions considering the current capacity and the measurement works, especially of DINAMA´s laboratory, since it is the one who has the largest analytical workload of the monitoring plan. The sampling shall be carried out on the third and fourth week of each month, being able to modify it considering the unforeseen events that may arise in the initially agreed dates.

4. ANALYTICAL DETERMINATIONS

4. 1 Analytical determinations to be developed at a medium-term

Analyses for the water quality evaluation in the Santa Lucia River Basin are carried out in coordination between the Laboratory of DINAMA and the different Municipal Laboratories.

However, due to the restrictions that Municipalities present (except from IMM) when developing analytical methodologies in the environmental field, most of water parameters and all sediment parameters are measured by the Laboratory of DINAMA.

BOD_5 is an important parameter for the water quality evaluation. All the Municipalities are expected to be able to measure BOD_5 at a long term, when they are provided with the necessary equipment for the development of this analytical methodology and with the required and properly qualified human resources.

4.2 Analytical Determinations to be executed within the framework of this agreement.

The distribution of the parameters to be measured by the different Municipalities are detailed in **Table 4.1**.

Table 4.1 Parameters to be measured by Municipalities

Municipality	Temp.	pH	EC	DO	BOD_5	COD	Total Solids	Faecal Coliforms
IMC	○	○	○	○	○	○	-	○
IMSJ	○	○	○	○	-	○	-	○
IMF	○	○	○	○	-	○	-	○
IML	○	○	○	○	-	○	○	○

○ Measured parameters

\- Parameters to be scheduled that could be measured in the future.

Annex 5 presents in general the analytical determinations to be carried out by each institution according to the present agreement.

5. QUALITY ASSURANCE

Aiming at generating reliable data which shall be used afterwards in the decision-making process on environmental management, each Laboratory is responsible for the reliability of the information generated by it.

Besides, each Laboratory has the training and material offered by the Laboratory of DINAMA regarding the analytical quality assurance, as well as the expertise generated in the several inter-calibration exercises organized by this institution.

The requirement that each laboratory carries out (in case it is still not carried out) internal quality controls, control charts for each parameter, deviation estimations, among other quality parameters, is stated.

Joint Works aiming at assuring data quality shall be carried out within the frame of the Network of Uruguayan Environmental Laboratories (RLAU) coordinated by DINAMA, expecting the participation of the Municipalities.

6. TRAINING ON SAMPLING AND FIELD ANALYSIS

The sampling and field analysis for water quality measurement shall be carried out by the Municipalities involved. This work must be executed by relevantly qualified members, according to the specific technical standards, in order to assure the accuracy of water quality data. For this reason, DINAMA has provided (and shall provide if necessary) technical training for the Municipalities' staff related to the sampling and field analysis. The Water Quality Monitoring Manual, a product of the DINAMA/JICA Project, shall be used as a text book for this technical training.

7. WATER QUALITY INFORMATION SYSTEM

Water quality data must be properly stored for its effective use. In the framework of the DINAMA/ JICA cooperation project, the following systems have been developed: SISICA (Water Quality Information System) and SISILAB (Laboratory Management Information System).

The Water Quality Information System (SISICA) was developed as a tool for the maintenance and use of environmental water quality data. It has been designed for the effective use of water quality data by the relevant institutions.

DINAMA analyses and evaluates water quality data of SISICA and uses the results as feed-back for the strategic part of water quality management and for pollution source management.

DINAMA has trained Municipalities regarding the use of SISICA software and shall provide training regarding the analysis and evaluation of water quality data.

In the framework of this agreement, Municipalities shall enter the analytical results generated by the samples taken during the monitoring campaigns into SISICA, as it has been stated in item 3, according to the analytical parameters defined for each Municipality, as it is established in item 4.

Besides, Municipalities shall benefit from the access to the analytical results generated by the Laboratory of DINAMA, which shall enter the information into SISICA according to what is established on items 3 and 4.

Computer tools for water quality information management (SISICA) and laboratory data management (SISILAB) are available for its transference and use by any relevant institution related to water quality management. In case of using them, the benefiting institution undertakes to link its system to the SISICA information system, in order to allow the access to the information that the institutions undertake to share under this agreement.

ANNEX 1

SELECTION CRITERIA FOR THE WATER QUALITY MONITORING POINTS AND MEASUREMENT PARAMETERS

1. SELECTION CRITERIA FOR THE WATER QUALITY MONITORING STATIONS

The locations for the 42 monitoring stations have been selected using the following basic criteria:

(1) It was decided that the total number of monitoring stations should be around a maximum of 30, considering that the maximum capacity of DINAMA laboratory is from 35 to 40 samples per week.

(2) Locations that involve water quality from the upper, middle and lower sections of Santa Lucia River were selected.

(3) Easily accessible locations where is possible to arrive by vehicle or where the samples can be taken from bridges were selected.

(4) Sampling points that were used by DINAMA in the past, were selected again due to their accessibility.

(5) In each Municipality, river mouths which due to their surroundings could be affected by pollution sources, such as industries, were selected.

(6) As many existing OSE sampling points as possible were selected. As a result, a total of 7 locations (5 in IMC, 1 in IMSJ and 1 in IMF) were selected.

OSE's purpose is to monitor water quality as a resource for making it drinkable. On the other hand, DINAMA´s purpose is to monitor the environmental water quality. Therefore, it is pretty obvious that the parameters measured by both of them shall differ. Some of the parameters (BOD; DO, pH, etc.) are measured by the two of them, so they can be used as cross-checking to assure accuracy control. The current proposed monitoring scheme should be extremely benefiting for OSE, since it is a beneficiary of the measurement results of additional parameters, apart from the ones that are already monitored.

Four locations in IMSJ (No. 6 to 9, downstream Santa Lucia River), which require the use of a boat, were selected as points used in the past by DINAMA.

2. JUSTIFICATION FOR THE SELECTED MEASUREMENT PARAMETERS.

As a result of the agreement reached at the meeting of the technical group from DINAMA/JICA Project, the measurement parameters were selected based on the following requirements:

(1) The parameters are based on Decree 253/79.

(2) The parameters proposed in this monitoring scheme were included so that the results of these surveys can be compared to those from IMM.

(3) When selecting the parameters, the capacity restrictions of the water laboratory of DINAMA were taken into account.

ANNEX 2

JUSTIFICATION FOR THE SELECTION OF MONITORING STATIONS (1/2)

Canelones	C1	Apart from drinking water quality, this site covers the bathing areas existing downstream this point.
	C2	It summarizes the information of the Canelon Grande and Canelon Chico Rivers, before its confluence with Santa Lucia River.
	C3	Site with a significant number of people since it is a bathing area.
	C4	Located downstream the Canelon Grande dam, monitoring water reserve for purification and a location with a significant number of people (camping site and bathing area)
	C5	It provides information on the Canelon Chico river quality, before its confluence with Canelon Grande river.
	C6	It monitors the environmental impacts on the Colorado river, in the town of El Dorado.
	C7	It provides information on Santa Lucia river quality and on the bathing area.
	C8	It provides information on the potential environmental impact on Pando river, after receiving the contribution of Cochengo river.
	C9	It provides information on the Colorado river, by Route 48.
	C10	It provides information on potential environmental impact deriving from industrial activities and sewerage, on Piedritas river.
	C11	In provides information on potential environmental impact on Pando river.
	C12	It provides information on the quality of Pando river, before its outflow into the De la Plata river.

Note: Points C8, C10, C11 and C12 belong to the Pando river basin

JUSTIFICATION FOR THE SELECTION OF MONITORING STATIONS (2/2)

San Jose	S1	This point evaluates the quality of San Jose river before reaching the city of San Jose, and the water quality for the intake of OSE.
	S2	Suitability for bathing.
	S3	It evaluates water quality downstream the city of San Jose.
	S4	It evaluates the potential environmental impact of industrial undertakings.
	S5	This monitoring point was kept in order to know the final quality of San Jose river.
	S6	Station downstream Colorado river.
	S7	It evaluates water quality upstream the mouth of San Jose river on Santa Lucia river.
	S8	It evaluates water quality in the mouth of Santa Lucia river, due to potential environmental impact of industrial undertakings.
	S9	It evaluates water quality in the mouth of Sarandi river on Santa Lucia river, due to potential environmental impact of industrial undertakings.
Lavalleja	L1	It describes the initial quantity of Campanero river (upstream the open air garbage dump of the city of Minas) generally used as a recreational site.
	L2	It evaluates the water quality of the Santa Lucía river origin.
	L3	It basically evaluates the potential impact of the open air dump of the city of Minas on the San Francisco river.
	L4	It evaluates water quality downstream the city of Minas and the potential impact of the city effluents.
	L5	It evaluates the water quality of San Francisco river, before its arrival to the city of Minas.
	L6	It evaluates the water quality of Santa Lucia river after receiving the contributions from the city of Minas and its industrial area.
Florida	F1	On the Calleros river, it enables to evaluate quality downstream the dairy industry.
	F2	This point enables to evaluate the quality of the Santa Lucia river at the entrance to the city of Florida.
	F3	This site is located at the exit of the city of Florida, near the water intake of OSE, upstream the tannery.
	F4	Located in the town of 25 de Agosto, in order to obtain joint information from the tannery and the wool washery, before Paso Severino dam.
	F5	Site located at the Paso Severino dam outlet. It allows to obtain an initial balance of the dam.

ANNEX 3

GEOGRAPHICAL INFORMATION OF MONITORING POINTS (1/2 (1/2)

Department	No	Longitud (O)	Latitud (S)	X	Y	River/Stream	Route	DINAMA	OSE	Note
Canelones	1*	56° 24'08.0"	34° 26'57.9"	0444690	6188568	Rio Santa Lucia	11	S7.7	O	
	2*	56° 20'27.6"	34° 29'05.3"	0450341	6184667	Arroyo Canelon Grande	11	S2.1	O	
	3	56° 14'56.6"	34° 21'51.6"	0458725	6198075	Rio Santa Lucia	5	S7.8	O	
	4	56° 16'12.5"	34° 27'33.9"	0456835	6187518	Arroyo Canelon Grande	5	S2.5	O	
	5*	56° 16'55.0"	34° 29'56.1"	0455769	6183132	Arroyo Canelon Chico	5			
	6*	55° 14'18.7"	34° 41'32.5"	0459850	6161691	Arroyo Colorado	5			
	7	55° 57'19.5"	34° 16'52.6"	0485720	6207370	Rio Santa Lucia	6	S7.9		San Ramon
	8	55° 55'41.1"	34° 32'11.7"	0488276	6179052	Arroyo Pando	11	L8.3		Paso Perdigon
	9*	56° 10'04.7"	34° 41'14.4"	0466313	6162274	Arroyo Colorado	36			
	10*	55° 57'55.5"	34° 43'37.8"	0484881	6157904	Arroyo Piedritas	8	L10.1		
	11*	55° 56'51.6"	34° 44'18.1"	0486509	6156664	Arroyo Pando				Agua abajo de Cartonera
	12*	55° 53'29.3"	34° 47'11.8"	0491661	6151319	Arroyo Pando	Interbalnearia	L8.7		
San Jose	1	56° 42'51.5"	34° 18'58.2"	0415877	6203129	Rio San Jose			O	Toma de Agua (OSE)
	2	56° 42'30.6"	34° 19'07.1"	0416414	6202863	Rio San Jose	3	S10.3		Playa Picada Varela
	3*	56° 41'37.0"	34° 19'34.9"	0417792	6202017	Rio San Jose	11			Aguas abajo del Puente
	4*	56° 33'45.1"	34° 23'10.5"	0429906	6195471	Arroyo Cagancha	11			Rodrigues
	5*	56° 34'38.9"	34° 32'12.7"	0428660	6178754	Rio San Jose	45	S10.4		
	6*	56° 21'14.0"	34° 43'35.0"	0449300	6157863	Rio Santa Lucia				Abajo desemboca Ayo. Colorado
	7*	56° 29'16.1"	34° 38'29.3"	0436970	6167208	Rio Santa Lucia				Abajo desemboca del Rio San Jose
	8*	56° 23'02.4"	34° 46'34.3"	0446575	6152323	Rio Santa Lucia				Punto banado frente a EFICE
	9*	56° 27'16.8"	34° 41'46.1"	0440048	6161164	Rio Santa Lucia				Desembocadura del Ayo Sarandi
Lavalleja	1	55° 09'34.8"	34° 21'38.9"	0558938	6198372	Arroyo Campanero	8			
	2*	55° 14'02.5"	34° 16'40.7"	0552149	6207602	Rio Santa Lucia				Camino las Tropas
	3*	55° 15'21.2"	34° 19'39.2"	0550106	6202113	Arroyo Campanero				Abajo relleno sanitario de Minas
	4*	55° 15'25.5"	34° 20'35.7"	0549987	6200372	Arroyo San Francisco				Abajo de la planta de tratamiento OSE
	5	55° 12'24.1"	34° 24'52.7"	0554577	6192427	Arroyo San Francisco				Parque de UTE
	6	55° 27'17.9"	34° 16'00.7"	0531807	6208927	Rio Santa Lucia	108	S7.6		
Florida	1*	56° 11'42.4"	34° 04'14.0"	0463559	6230685	Arroyo Calleros				Carretera hacia Cerro de la Macana
	2	56° 09'45.7"	34° 01'45.2"	0466535	6235279	Rio Santa Lucia Chico		S11.3		2do P° de la Arena
	3	56° 12'12.8"	34° 05'29.3"	0462790	6228362	Rio Santa Lucia Chico				Calzada
	4*					Rio Santa Lucia Chico				Arriba de Paso Severino
	5	56° 18'28.4"	34° 15'57.6"	0453256	6208958	Rio Santa Lucia Chico				Aguas abajos Paso Severino (Represa)

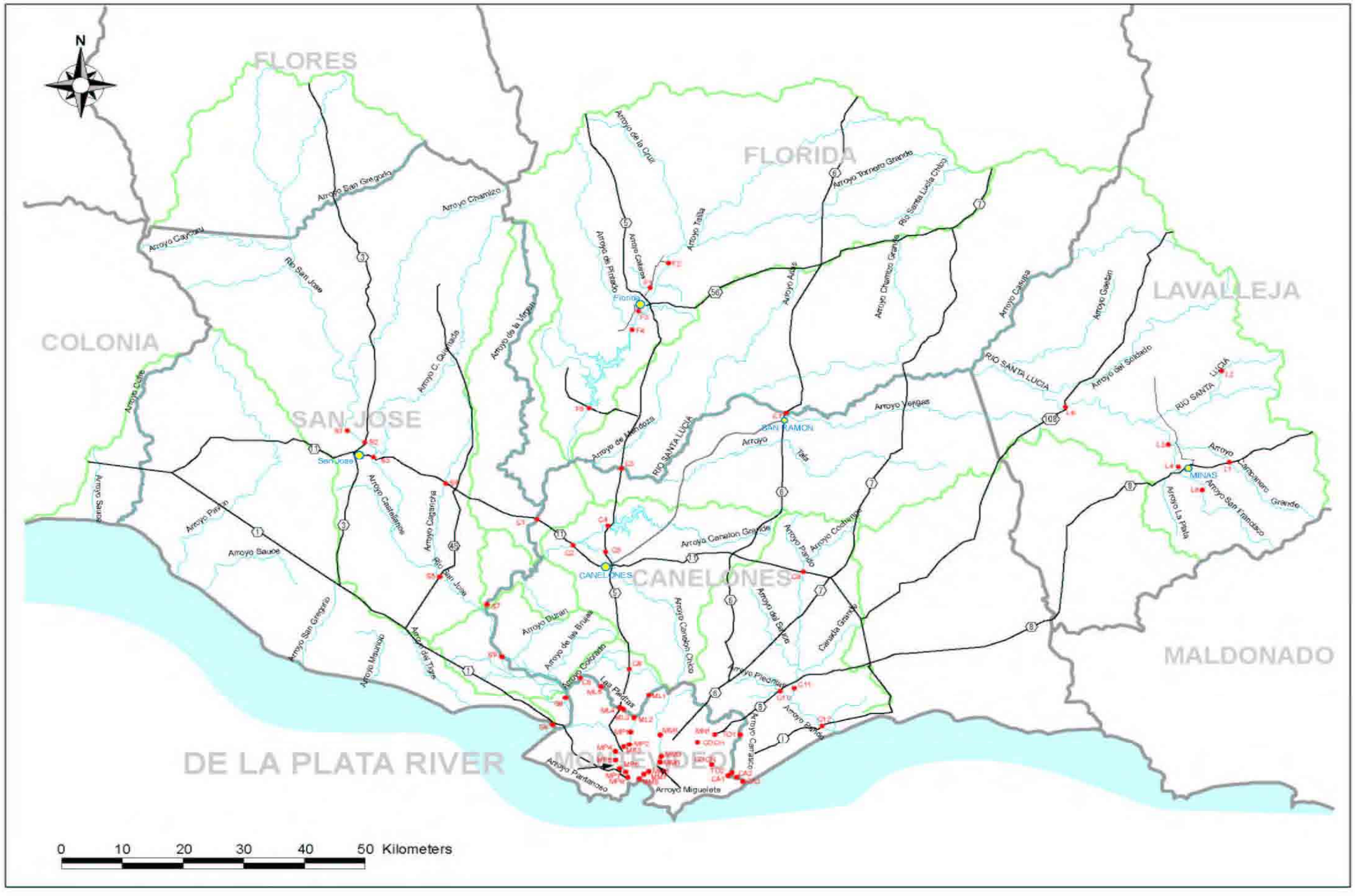
Note: Station numbers with mark "*" are for both water and sediment, and those without "*" are only for water.

ANNEX 3

GEOGRAPHICAL INFORMATION OF MONITORING POINTS (2/2)

Department	No	Longitud (O)	Latitud (S)	X	Y	River/Stream	Route	DINAMA	OSE	NOTA
Pantanoso	MP1	56° 14'13.2"	34° 47'31.2"	0460038	6150637	Arroyo Pantanoso				Cno. Colman
	MP3	56° 15'07.2"	34° 49'01.2"	0458678	6147858	Arroyo Pantanoso				Cno. Melilla
	MP4	56° 15'43.2"	34° 49'26.4"	0457767	6147077	Arroyo Pantanoso				Cno. Dela Granja
	MP5	56° 15'54.0"	34° 50'06.0"	0457498	6145856	Arroyo Pantanoso				Luis Batlle Berres
	MP6	56° 15'18.0"	34° 51'18.0"	0458423	6143641	Arroyo Pantanoso	5			
	MP7	56° 14'31.2"	34° 51'36.0"	0459614	6143092	Arroyo Pantanoso				Aporte-Pluvial Alaska
	MP8	56° 14'38.4"	34° 52'04.8"	0459435	6142203	Arroyo Pantanoso				Accesos
Miguelete	MM1	56° 11'20.4"	34° 47'34.8"	0464432	6150545	Arroyo Miguelete				Cno. O. Rodriguez
	MM2	56° 11'02.4"	34° 48'39.6"	0464897	6148549	Arroyo Miguelete				Cno. Carlos A. Lopez
	MM3	56° 10'51.6"	34° 50'13.2"	0465182	6145666	Arroyo Miguelete				A. Saravia
	MM4	56° 10'37.2"	34° 50'31.2"	0465550	6145113	Arroyo Miguelete				Aporte-Pluvial Casavalle
	MM5	56° 11'09.6"	34° 50'31.2"	0464727	6145110	Arroyo Miguelete				Jose Ma. Silva
	MM6	56° 12'07.2"	34° 51'25.2"	0463270	6143418	Arroyo Miguelete				Luis A. De Herrera
	MM7	56° 13'04.8"	34° 52'04.8"	0461812	6142214	Arroyo Miguelete				Juan Ma. Gutierrez
	MM8	56° 13'15.6"	34° 52'08.4"	0461538	6142101	Arroyo Miguelete				Accesos
Las Piedras	ML1	56° 12'54.0"	34° 44'56.4"	0462032	6155416	Arroyo Las Piedras				Cno. Julio Sosa
	ML2	56° 13'40.8"	34° 46'04.8"	0460851	6153304	Arroyo Las Piedras				Ruta Cesar Mayo Gutierrez
	ML3	56° 14'27.6"	34° 45'43.2"	0459658	6153964	Arroyo Las Piedras				Cno. El Cuarteador
	ML4	56° 15'25.2"	34° 45'14.4"	0458189	6154845	Arroyo Las Piedras	5			
	ML5	55° 17'27.6"	34° 43'08.4"	0546656	6158705	Arroyo Las Piedras	36			
Carrasco	MN1	56° 05'16.8"	34° 48'18.0"	0473679	6149245	Arroyo Carrasco	8			
	TO1	56° 02'49.2"	34° 48'25.2"	0477431	6149033	Arroyo Carrasco	102			
	CDCH	56° 05'24.0"	34° 49'58.8"	0473505	6146138	Arroyo Carrasco				Av. Punta de Rieles
	CDCN	56° 04'55.2"	34° 51'39.6"	0474245	6143034	Arroyo Carrasco				Cno. Colastine-Estacion de Bombeo
	CA2	56° 02'56.4"	34° 52'04.8"	0477265	6142265	Arroyo Carrasco				French
	CA3	56° 02'09.6"	34° 52'33.6"	0478456	6141380	Arroyo Carrasco				Av. Italia
	MN2	56° 03'25.2"	34° 51'36.0"	0476531	6143151	Arroyo Carrasco				Puente OSE
	TO2	56° 03'25.2"	34° 51'36.0"	0476531	6143151	Arroyo Carrasco				Puente OSE
	CA1	56° 03'36.0"	34° 52'01.2"	0476259	6142373	Arroyo Carrasco				Cno. Carrasco
Bahia de Montevideo	MB1	56° 13'30.0"	34° 52'37.2"	0461176	6141212	Bahia Montevideo				Sesembocadura A. Miguelete
	MB2	56° 12'18.0"	34° 53'09.6"	0463009	6140222	Bahia Montevideo				Descarga A. Seco
	MB3	56° 12'21.6"	34° 53'45.6"	0462922	6139112	Bahia Montevideo				Recinto Portuario
	MB4	56° 14'16.8"	34° 53'20.4"	0459994	6139876	Bahia Montevideo				Oeste de la Isla Libertad

ANNEX 4
LOCATION MAP OF MONITORING POINTS



ANNEX 5

Monitoring Points Parameters (1/2)

Local Government	IM Canelones												IM Florida				
Parameters \ Station No.	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12	F1	F2	F3	F4	F5
Turbidity (NTU)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
PH	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
DO (mg/l)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	○	○	○	○	○
BOD_5 (mg/l)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	○	○	○	○	○
Fats and oils (mg/l)		○			○	○	○		○		○		○		○	○	○
Ammonia (mg/l) as N	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Nitrate (mg/l) as N	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Total Phosphorous (mg/l) as P	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
SS (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Fecal coliformes (UFC/100ml)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Cadmium (mg/l)	●	●			●	●			●	●	●	●			●	●	
Total Chromium (mg/l)	●	●			●	●			○●	○●	○●	○●			●	○●	
Mercury (mg/l)	●	●			●	●			●	●	●	●			●	○●	
Lead (mg/l)	●	●			●	●			●	●	●	●			●	●	
Pesticides (µg/l)	○●	○●	○	○	○●	○●	○	○	○●	○●	○●	○●	○●	○	○	○●	○
Nitrite (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Hexavalent chromium (mg/l)					○						○	○	○				
Totals coliformes (UFC/100ml)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
COD (mg/l)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Conductivity (m S/m)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Temperature (℃)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Total Solid (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Sulfide (mg/l)					○	○			○	○	○	○	○			○	

Note: ○ : Analysis of water by DINAMA, ◎: Analysis of water by Local Government, ● : Analysis of sediment by DINAMA

The local government shall be in charge of the water sampling and the transportation of water samples, except for stations Nº S6, S7, S8, S9 y C11. DINAMA shall be in charge of sediment sampling and the transportation of sediment samples. It should be pointed out that monitoring stations for pesticides are listed tentatively. 10 stations shall be selected for water and sediments as an optimal number to be handled by the Laboratory of DINAMA.

ANNEX 5

Monitoring Points Parameters (2/2)

Local Government	IM San José									IM Lavalleja					
Parameters \ Station Nº	S1	S2	S3	S4	S5	S6	S7	S8	S9	L1	L2	L3	L4	L5	L6
Turbidity (NTU)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
PH	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
DO (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
BOD_5 (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Fats and oils (mg/l)							○	○	○						
Ammonia (mg/l) as N	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Nitrate (mg/l) as N	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Total Phosphorous (mg/l) as P	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
SS (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Fecal coliformes (UFC/100ml)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Cadmium (mg/l)			●			●	●	●	●			●	●		
Total chromium (mg/l)			●			○●	●	○●	●			○●	●		
Mercury (mg/l)			○●			●	●	●	●			●	●		
Lead (mg/l)			●			●	●	●	●			●	●		
Pesticides (µg/l)	○	○	○●	○●	○●	○●	○●	○●	○	○	○	○●	○●	○	○
Nitrite (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Hexavalent chromium (mg/l)															
Total Coliformes (UFC/100ml)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
COD (mg/l)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Conductivity (m S/m)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Temperature (℃)	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
Total Solids (mg/l)	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
Sulfide (mg/l)			○			○									

Note: ○: Analysis of water by DINAMA, ◎: Analysis of water by Local Government, ●: Analysis of sediment by DINAMA

The local government shall be in charge of the water sampling and the transportation of water samples, except for stations Nº S6, S7, S8, S9 y C11. DINAMA shall be in charge of sediment sampling and the transportation of sediment samples. It should be pointed out that monitoring stations for pesticides are listed tentatively. 10 stations shall be selected for water and sediments as an optimal number to be handled by the Laboratory of DINAMA.

ANNEX (2)

Annex (2)

Proyecto SISICA

Especificación de interfaces gráficas de usuario

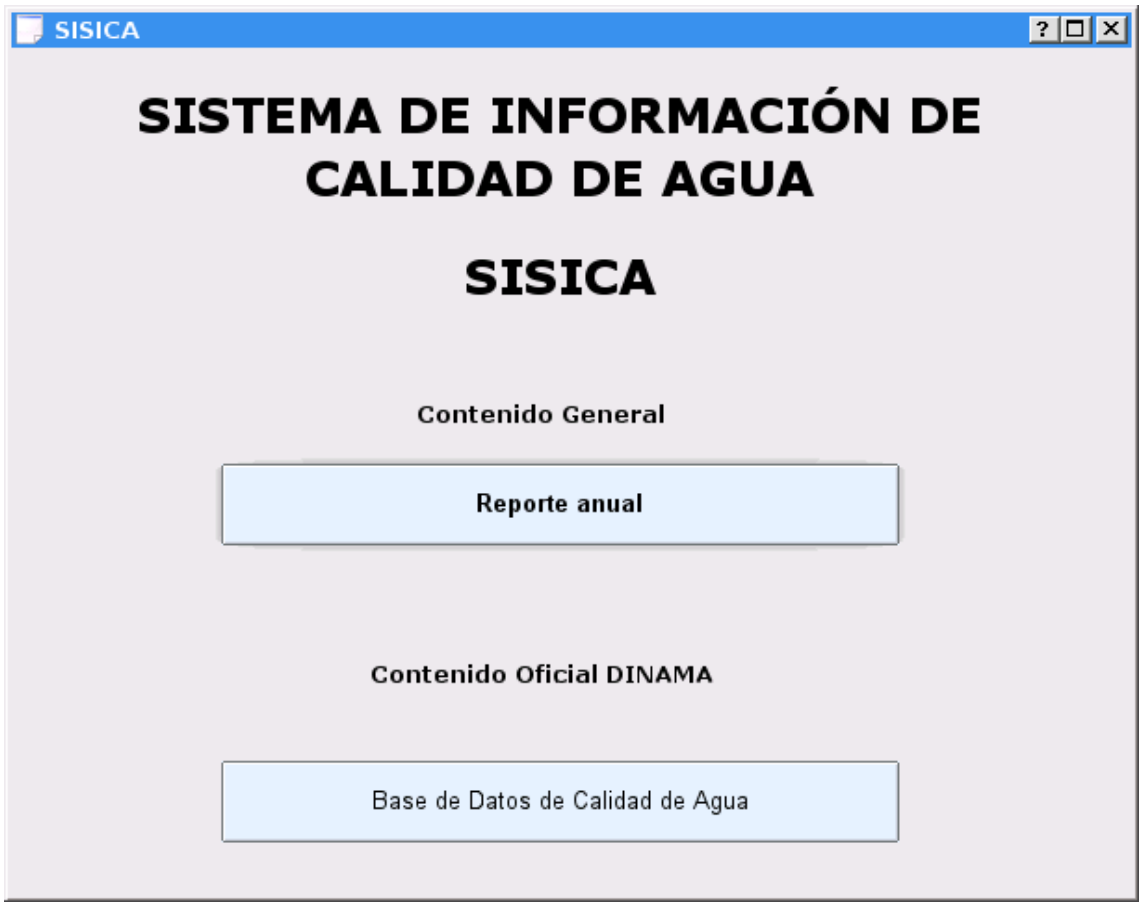
Versión 2.0

Historia de revisiones

Fecha	Versión	Descripción	Autor
22/07/2004	1	Primera especificación de interfaces.	Aníbal Pacheco Claudia Rostagnol
23/07/2004	1.1	Ajustes en la interfaces, se agrega alta de usuario.	Aníbal Pacheco Claudia Rostagnol
29/07/2004	1.2	Ajustes en la interfaces	Aníbal Pacheco Claudia Rostagnol
27/08/2004	2.0	Ajustes en la interfaces, se incluye la totalidad de las interfaces relevantes para la implementación.	Aníbal Pacheco Claudia Rostagnol

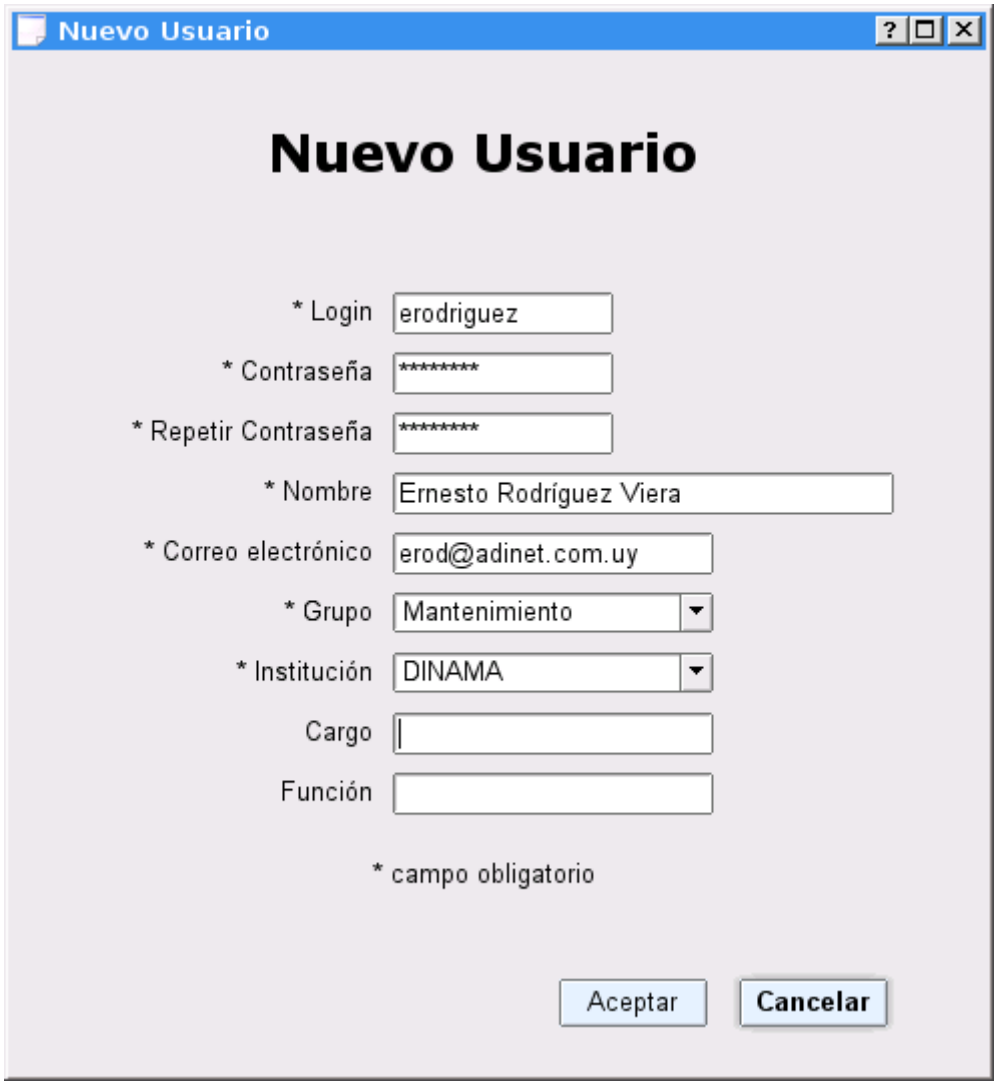
Tabla de Contenido

1 Home Page


SISICA
SISTEMA DE INFORMACIÓN DE CALIDAD DE AGUA
SISICA
Contenido General
Reporte anual
Contenido Oficial DINAMA
Base de Datos de Calidad de Agua

2 Gestión de Usuarios

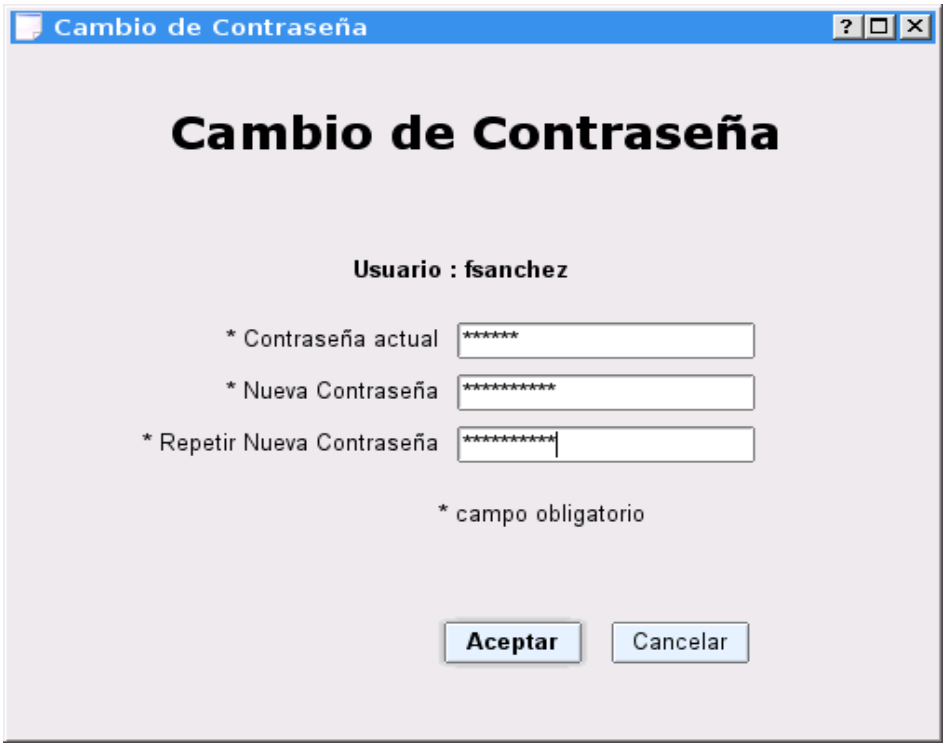
2.1 Alta de Usuario



2.2 Modificación de datos y baja de usuario

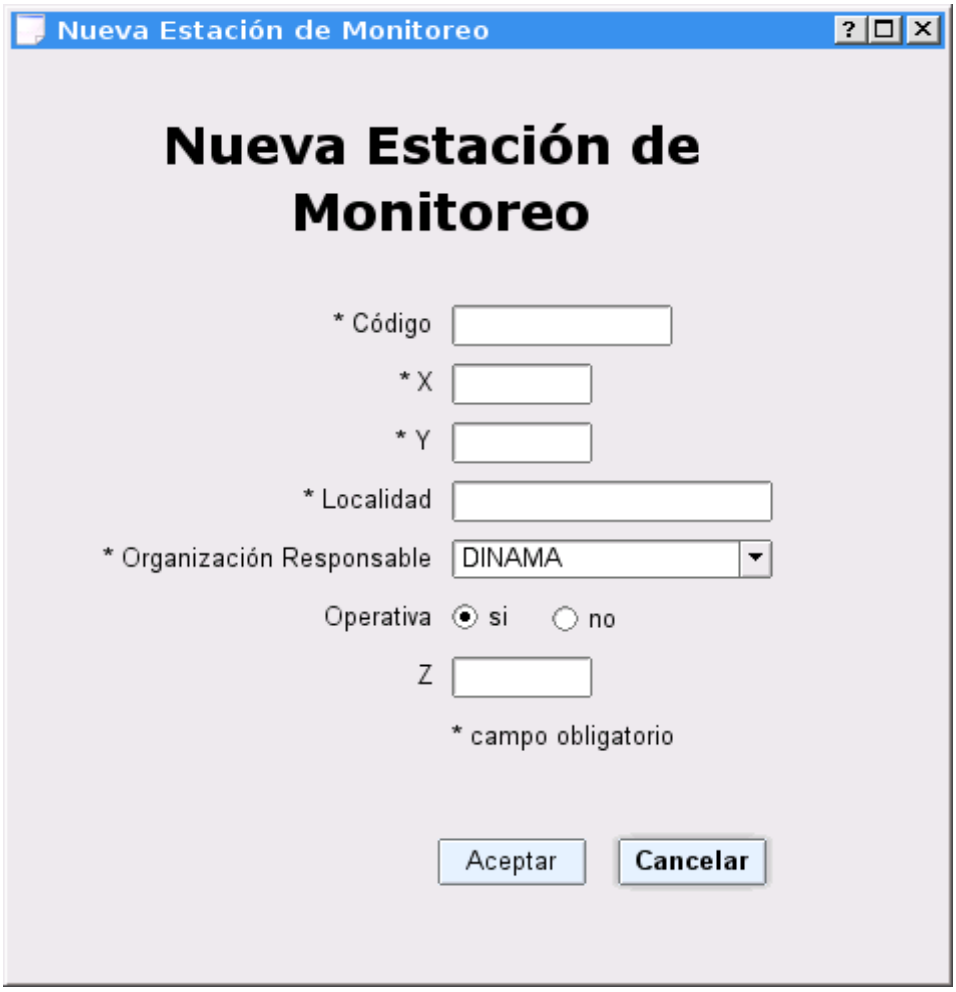
Al seleccionar Modificar datos se obtiene la siguiente interface:

2.3 Cambio de contraseña:



3 Gestión de Estaciones

3.1 Alta de estación

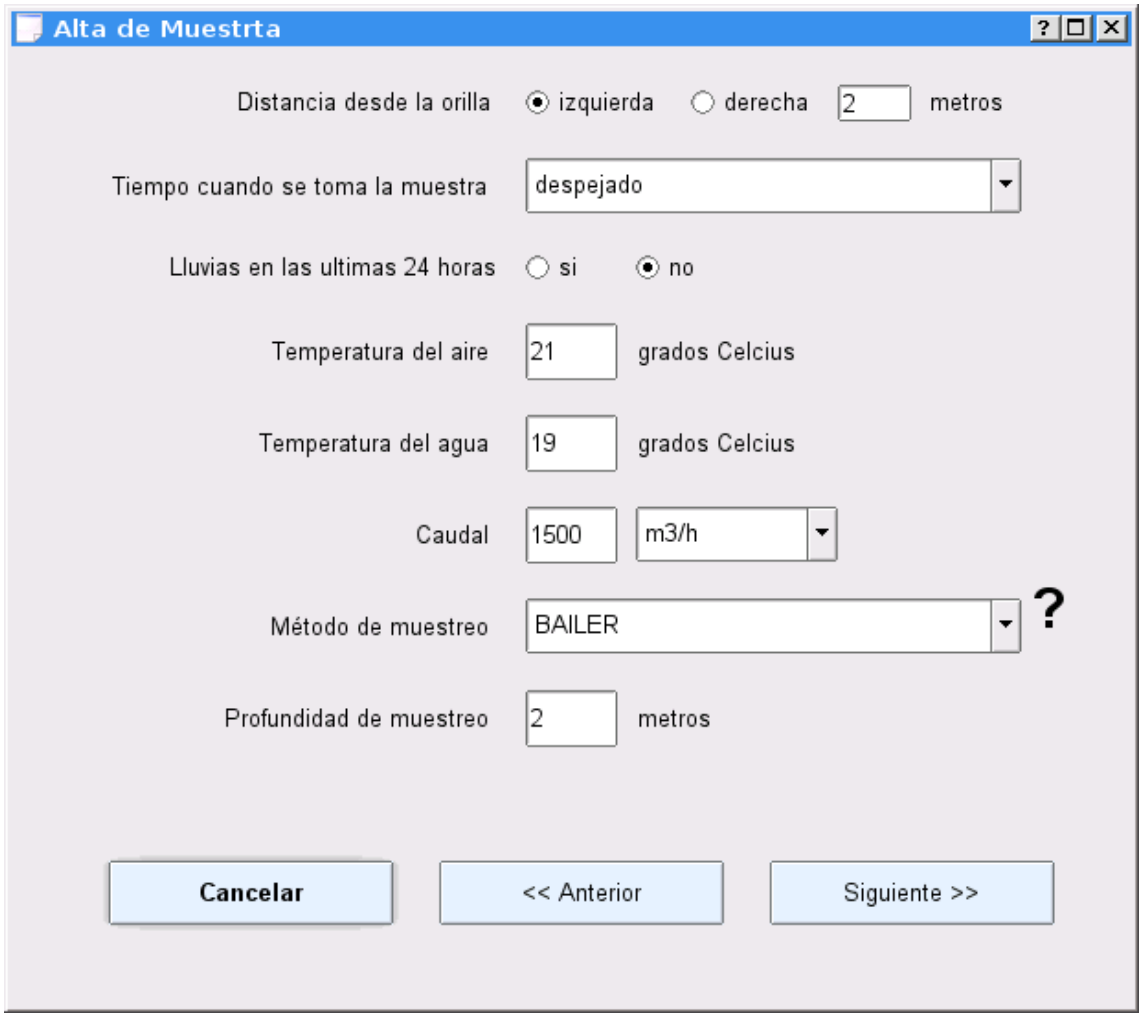


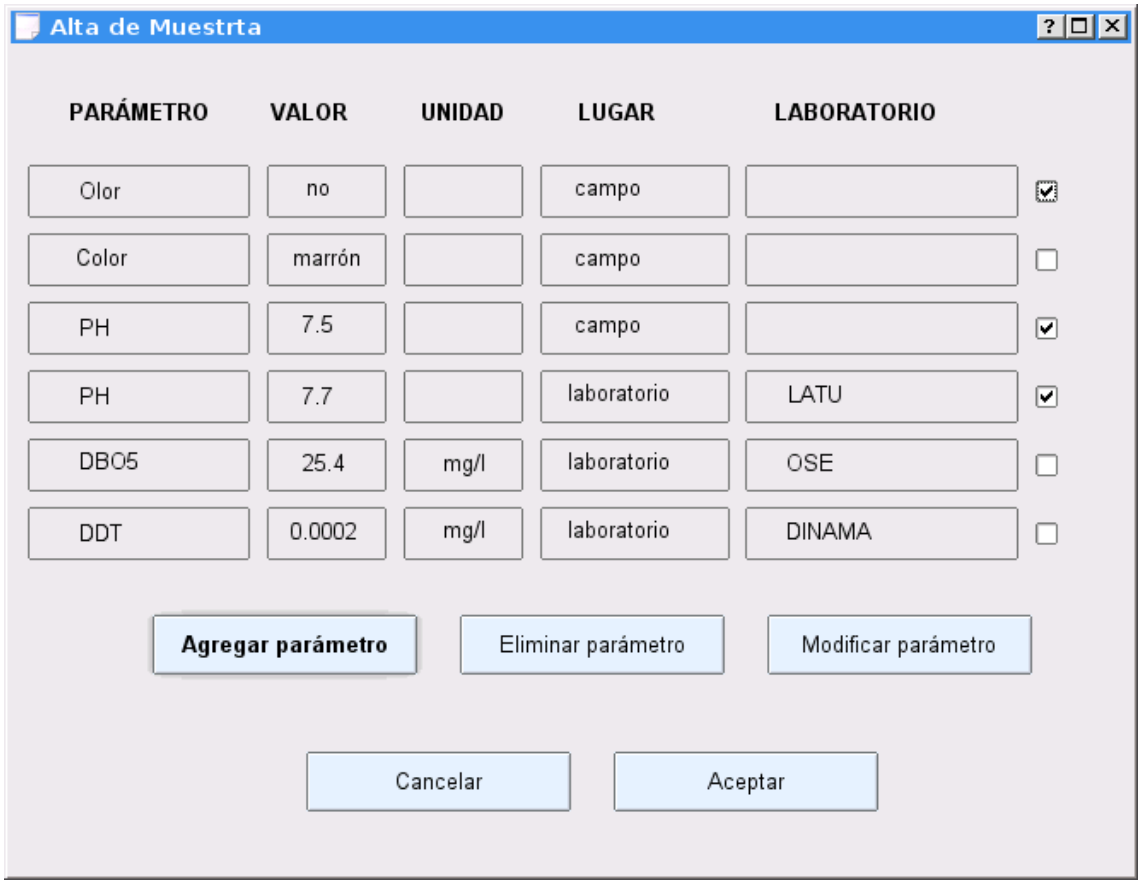
3.2 Modificación de datos y baja de estación

Al seleccionar modificar datos se obtiene la siguiente interface:

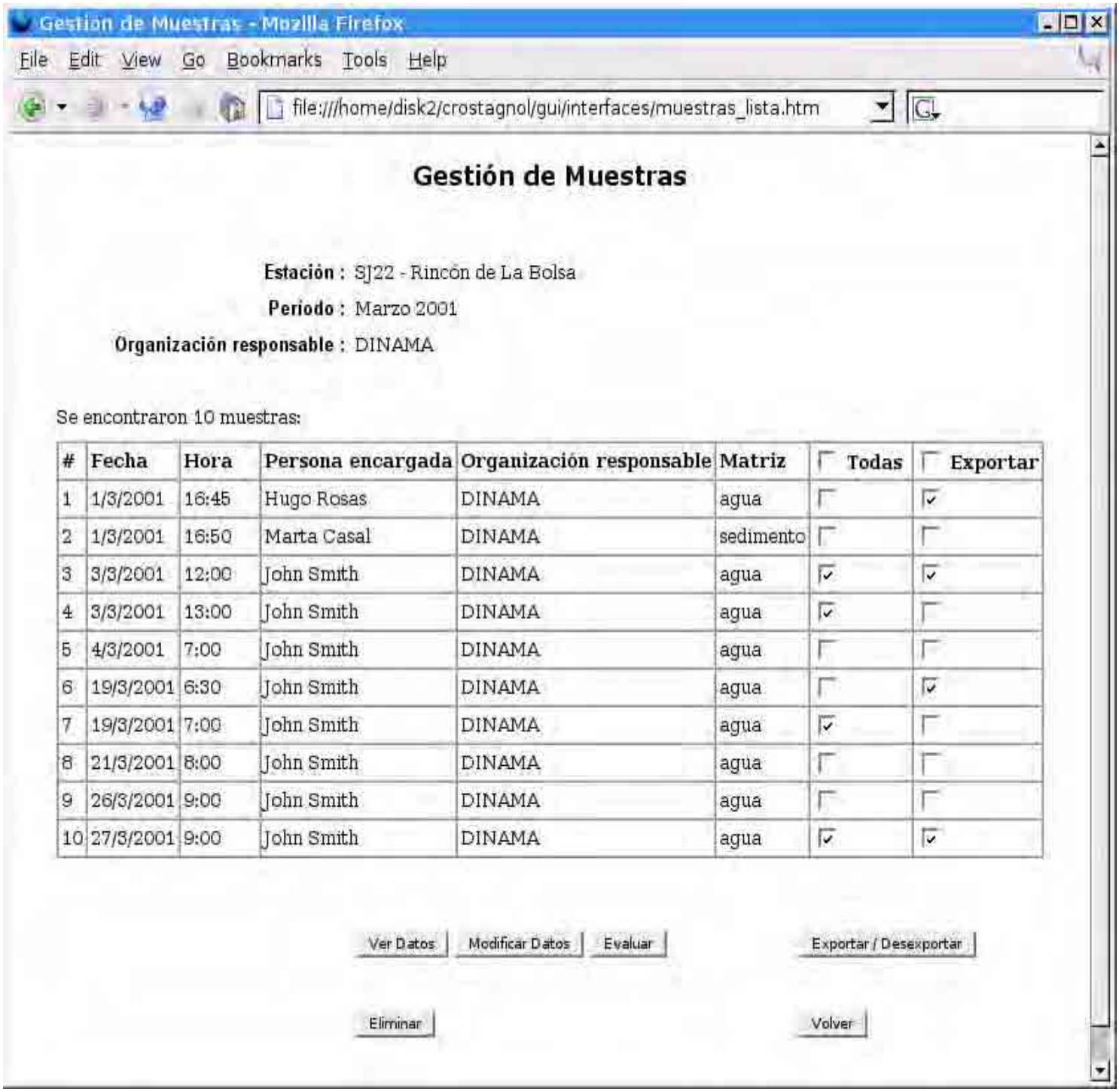
4 Gestión de información de calidad de agua

4.1 Alta de muestra


Alta de Muestrta
Distancia desde la orilla
izquierda
derecha
2
metros
Tiempo cuando se toma la muestra
despejado
Lluvias en las ultimas 24 horas
si
no
Temperatura del aire
21
grados Celcius
Temperatura del agua
19
grados Celcius
Caudal
1500
m3/h
Método de muestreo
BAILER
?
Profundidad de muestreo
2
metros
Cancelar
<< Anterior
Siguiente >>


Alta de Muestrta
PARÁMETRO
VALOR
UNIDAD
LUGAR
LABORATORIO
Olor
no
campo
Color
marrón
campo
PH
7.5
campo
PH
7.7
laboratorio
LATU
DBO5
25.4
mg/l
laboratorio
OSE
DDT
0.0002
mg/l
laboratorio
DINAMA
Agregar parámetro
Eliminar parámetro
Modificar parámetro
Cancelar
Aceptar

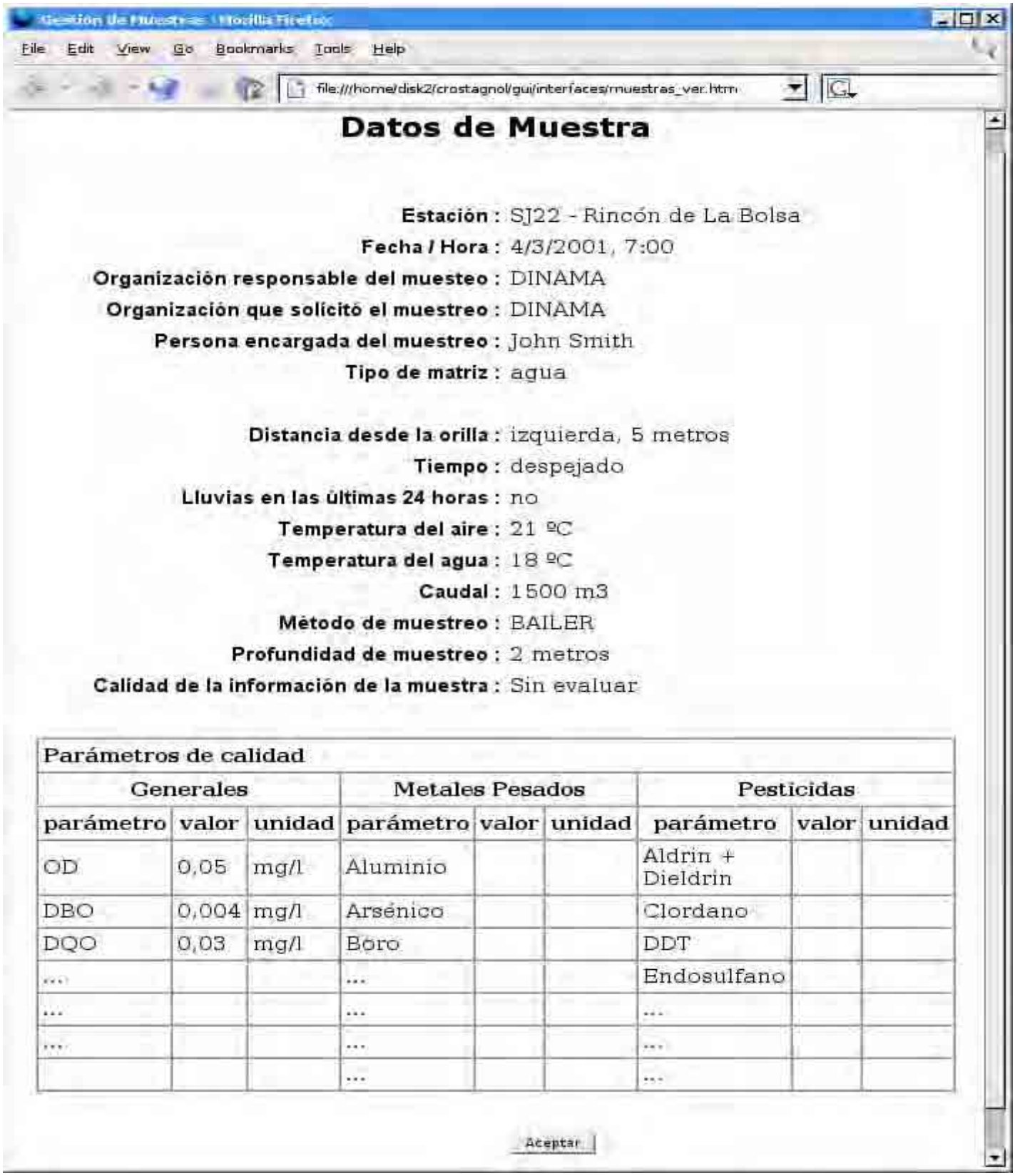
4.2 Visualización de muestras



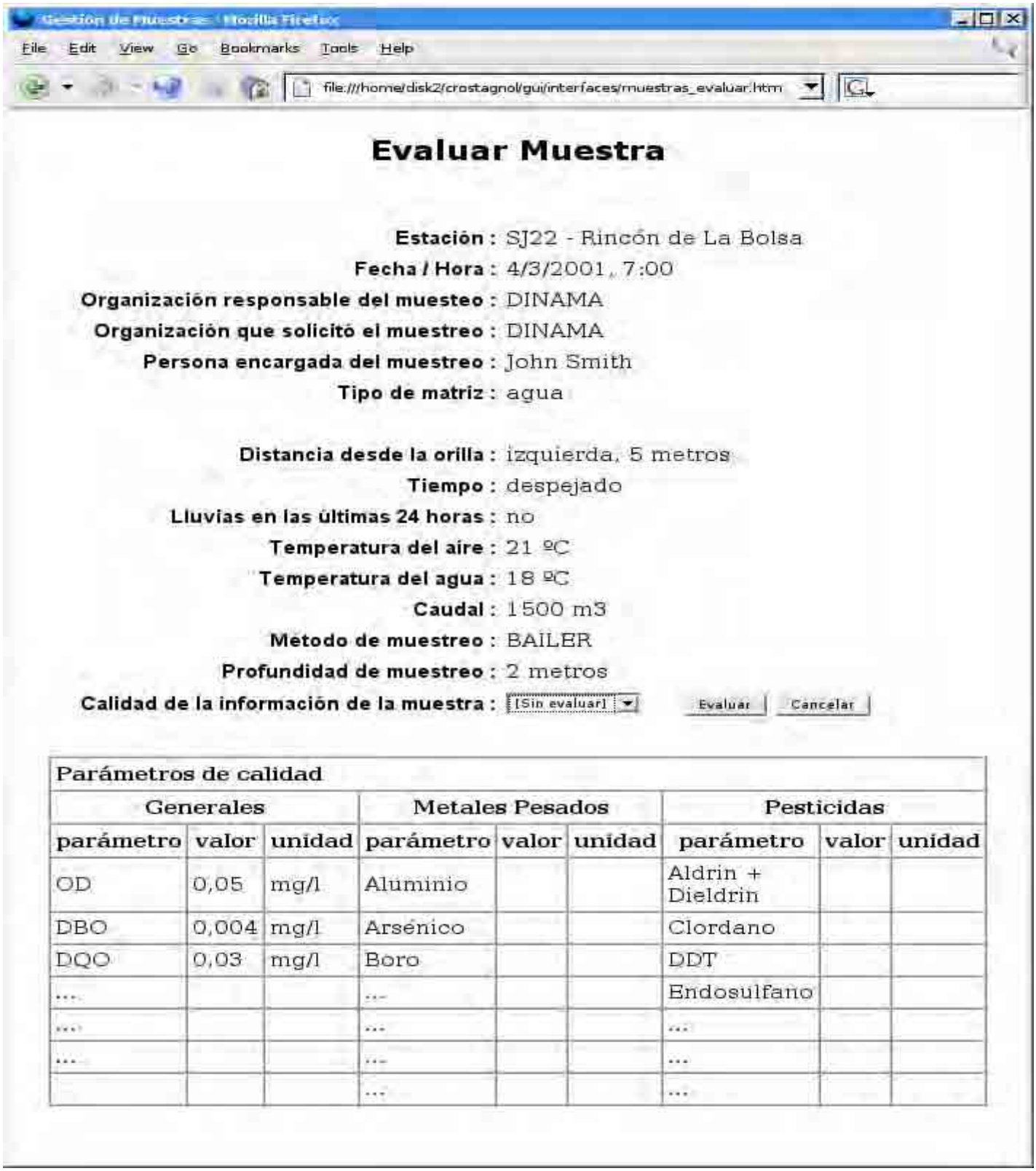
4.3 Modificación de datos de una muestra

La modificación de datos conduce a una interfaz análoga a la de alta de muestra.

4.4 Visualización de datos de muestras



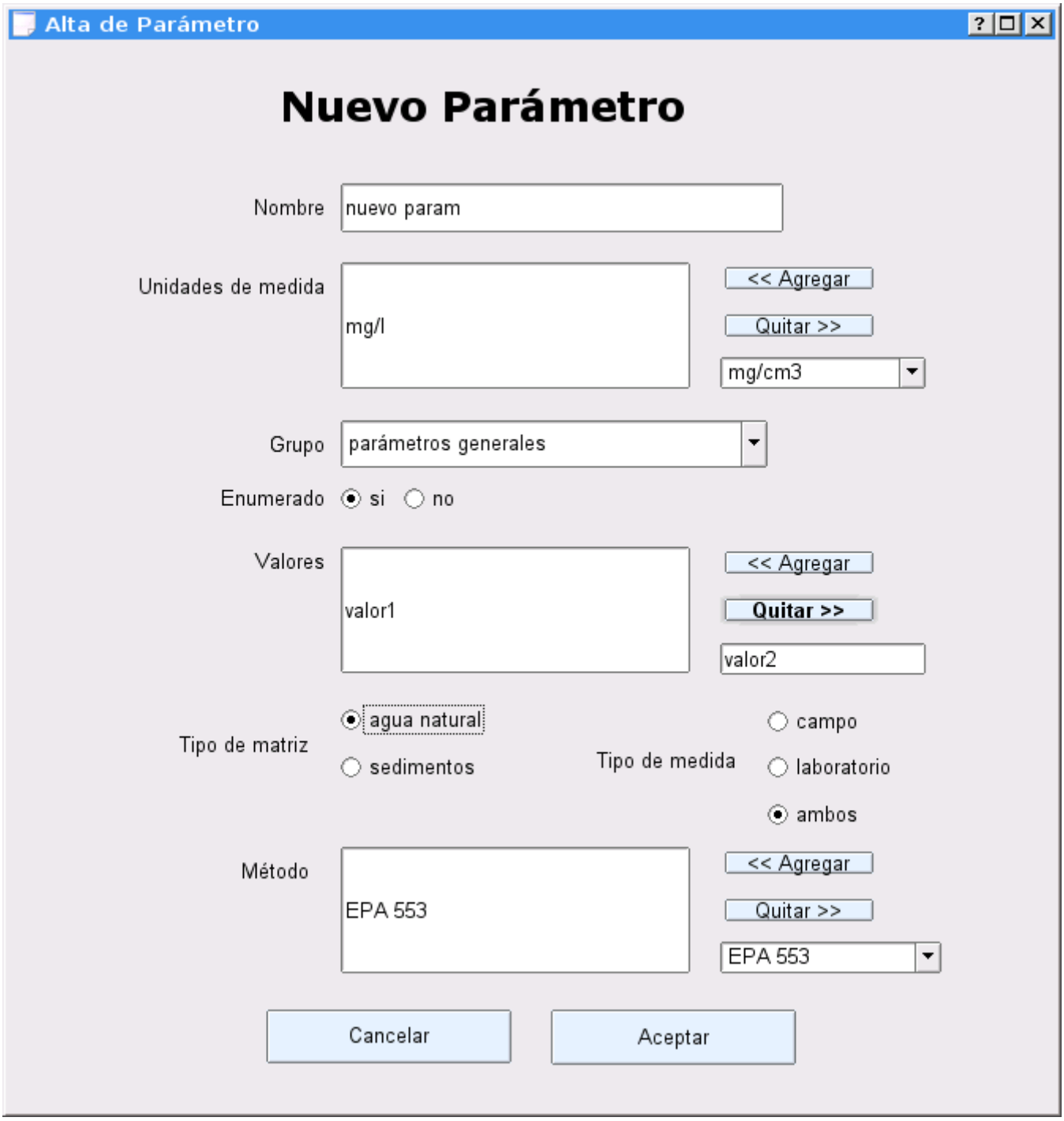
4.5 Evaluación de una muestra



Parámetros de calidad								
Generales			Metales Pesados			Pesticidas		
parámetro	valor	unidad	parámetro	valor	unidad	parámetro	valor	unidad
OD	0,05	mg/l	Aluminio			Aldrin + Dieldrin		
DBO	0,004	mg/l	Arsénico			Clordano		
DQO	0,03	mg/l	Boro			DDT		
...			...			Endosulfano		
...			...			...		
...			...			...		
			...			...		

5 Gestión de parámetros de muestra

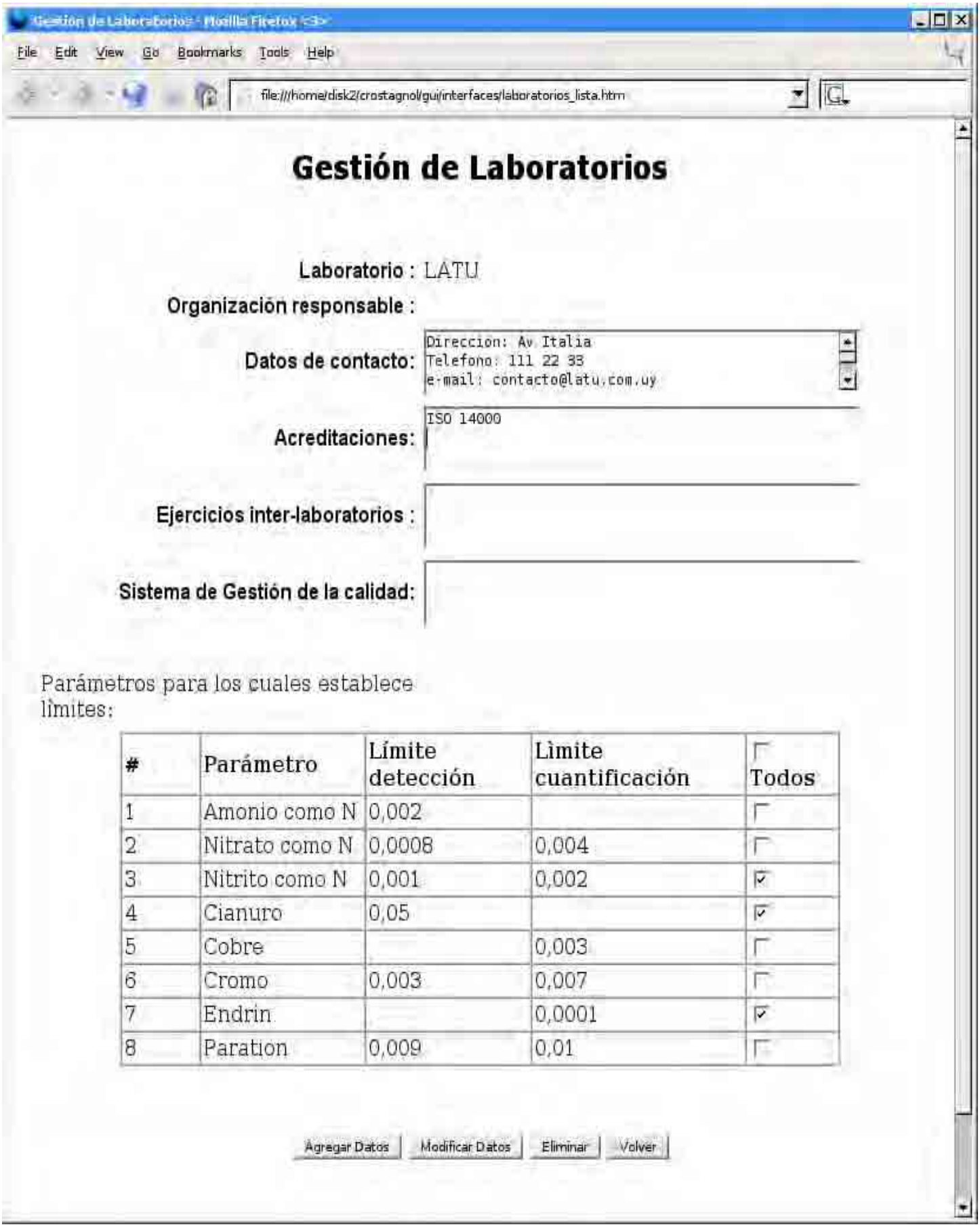
5.1 Alta de parámetro



5.2 Baja de parámetro

6 Gestión de laboratorios

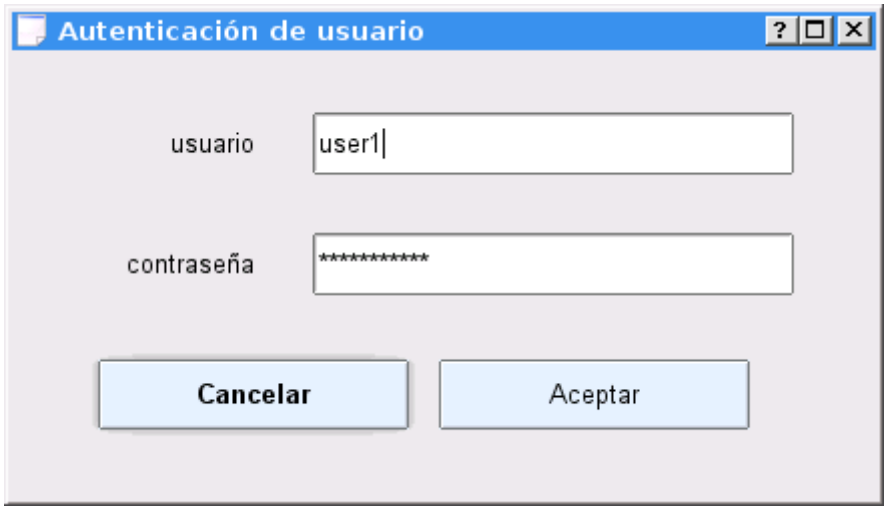
6.1 Visualización / Modificación / Alta de laboratorios


Gestión de Laboratorios - Mozilla Firefox
File Edit View Go Bookmarks Tools Help
file:///home/disk2/crostagnol/gui/interfaces/laboratorios_lista.htm
Gestión de Laboratorios
Laboratorio : LATU
Organización responsable :
Datos de contacto:
Direccion: Av. Italia
Telefono: 111 22 33
e-mail: contacto@latu.com.uy
Acreditaciones:
ISO 14000
Ejercicios inter-laboratorios :
Sistema de Gestión de la calidad:
Parámetros para los cuales establece límites:
Parámetro Límite detección Límite cuantificación Todos
1 Amonio como N 0,002
2 Nitrato como N 0,0008 0,004
3 Nitrito como N 0,001 0,002
4 Cianuro 0,05
5 Cobre 0,003
6 Cromo 0,003 0,007
7 Endrin 0,0001
8 Paration 0,009 0,01
Agregar Datos
Modificar Datos
Eliminar
Volver

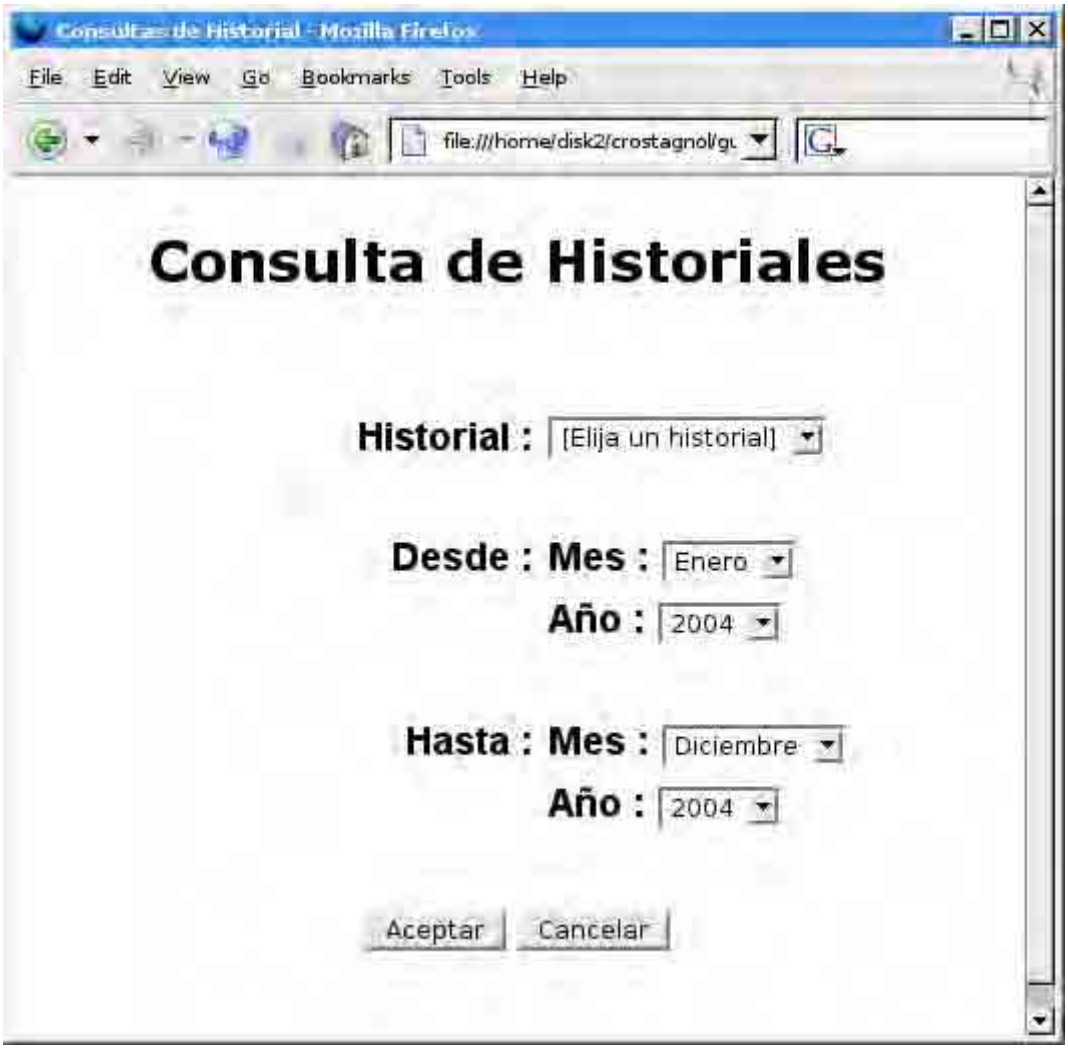

Gestión de Laboratorios - Mozilla Firefox
File Edit View Go Bookmarks Tools Help
file:///home/disk2/crostagnol/gui/interfaces/laboratorios_nuevo.htm
Nuevo Laboratorio
Nombre :
Organización responsable :
(Seleccione una Organizacion)
Datos de contacto:
Acreditaciones:
Ejercicios inter-laboratorios :
Sistema de Gestión de la calidad:
Parámetros para los cuales establece límites:
#
Parámetro
Límite detección
Límite cuantificación
Todos
Aceptar
Agregar Datos
Modificar Datos
Eliminar
Volver

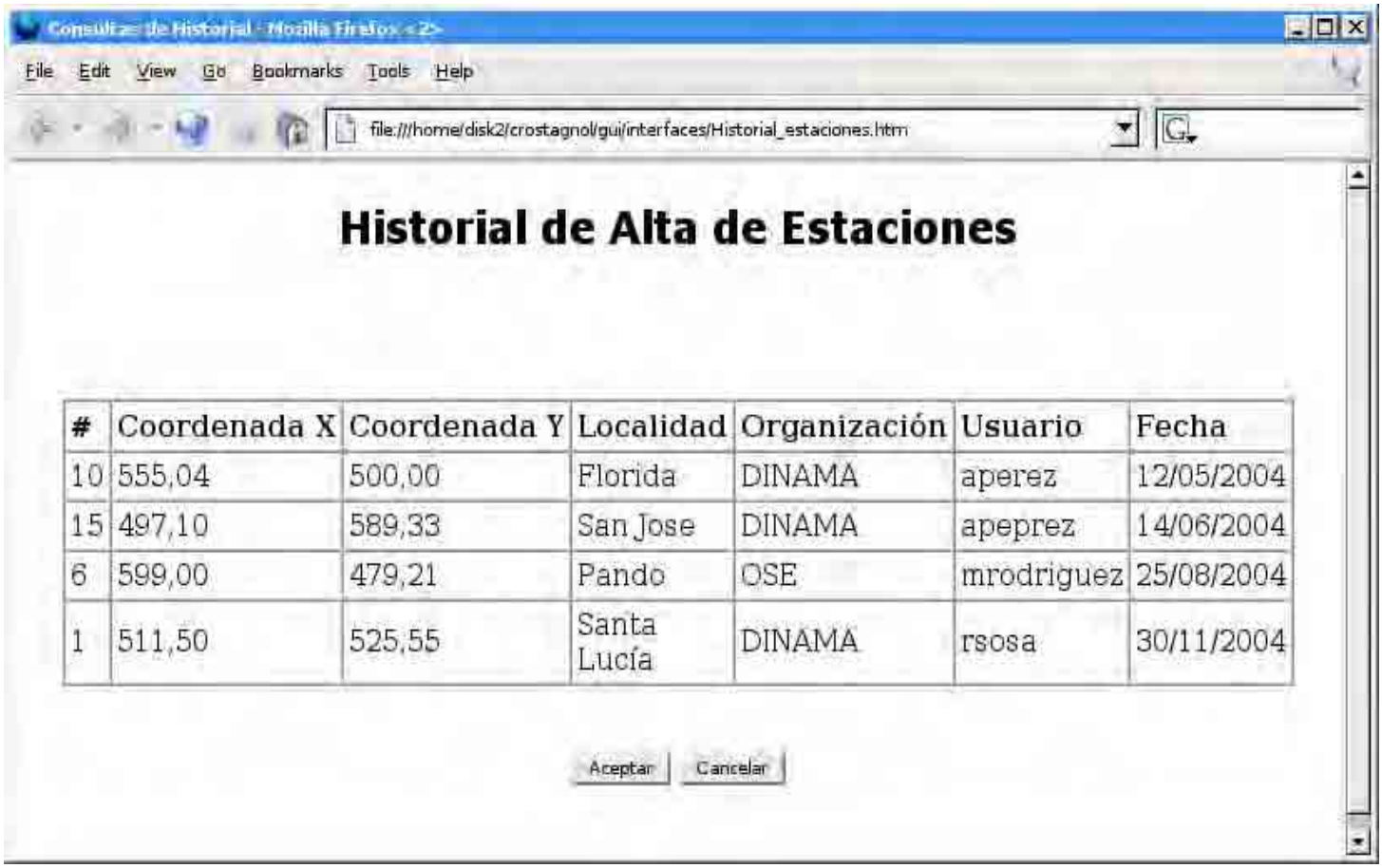
7 Uso del sistema

7.1 Login



7.2 Consultas al historial

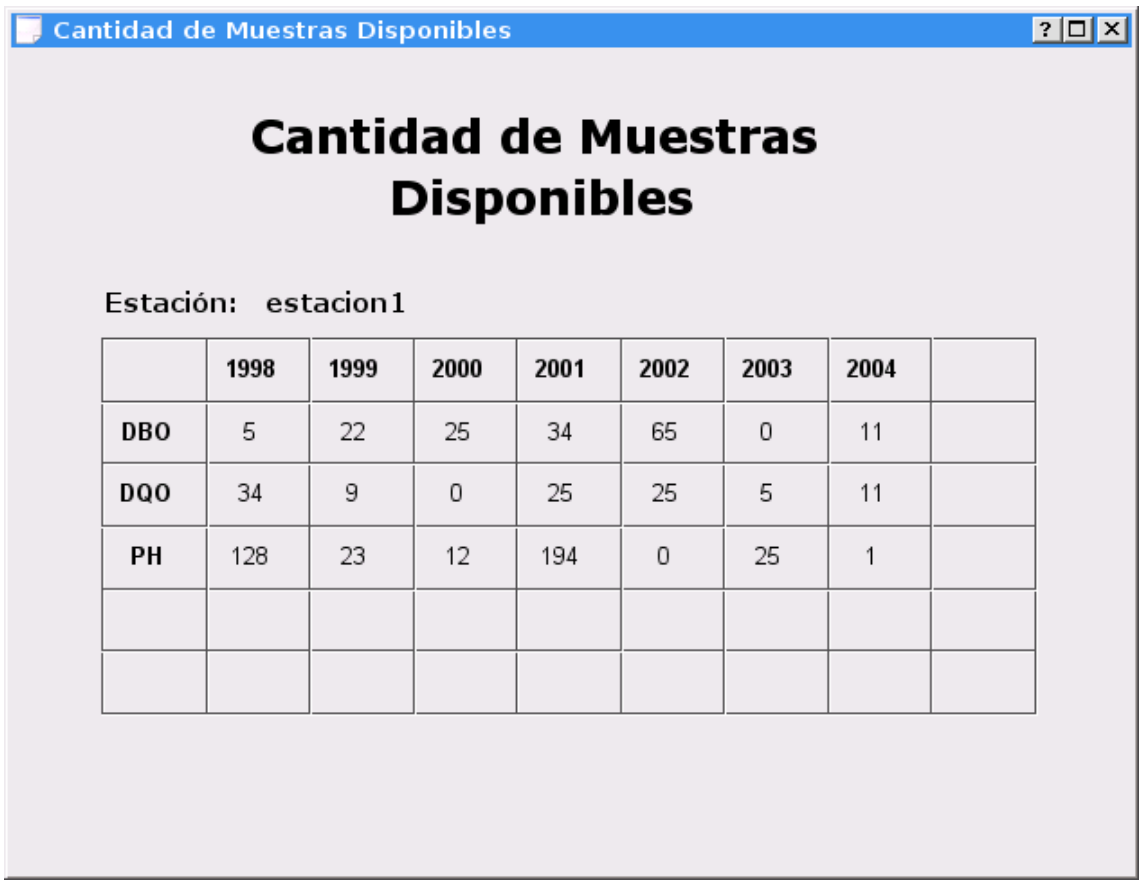

Consultas de Historial - Mozilla Firefox
File Edit View Go Bookmarks Tools Help
file:///home/disk2/crostagnol/gu
Consulta de Historiales
Historial : [Elija un historial]
Desde : Mes : Enero
Año : 2004
Hasta : Mes : Diciembre
Año : 2004
Aceptar
Cancelar


Consulta de Historial - Mozilla Firefox <2>
File Edit View Go Bookmarks Tools Help
file:///home/disk2/crostagnol/gui/interfaces/Historial_estaciones.htm
Historial de Alta de Estaciones
Coordenada X Coordenada Y Localidad Organización Usuario Fecha
10 555,04 500,00 Florida DINAMA aperez 12/05/2004
15 497,10 589,33 San Jose DINAMA apeprez 14/06/2004
6 599,00 479,21 Pando OSE mrodriguez 25/08/2004
1 511,50 525,55 Santa Lucía DINAMA rsosa 30/11/2004
Aceptar
Cancelar

8 Consultas

Aquí se presentan ejemplos de los dos tipos de consultas que tendrá SISICA, consultas donde se muestra información tabular y consultas donde se muestra información geográfica. Hemos elegido dos de las variadas consultas que tendrá SISICA para representar estas funcionalidades. Las interfaces para las consultas restantes no se detallan debido a su gran similitud.

8.1 Cantidad de muestras disponibles



	1998	1999	2000	2001	2002	2003	2004	
DBO	5	22	25	34	65	0	11	
DQO	34	9	0	25	25	5	11	
PH	128	23	12	194	0	25	1	

8.2 Distribución espacial de un parámetro

ANNEX (3)

TABLE OF CONTENTS

Policy of Quality

The Laboratory of the National Directorate of Environment undertakes to:

- Guarantee the **reliability** and **confidentiality** of the executed analyses.
- To elaborate the Standard Operating Procedures according to the quality objectives established. To document, revise and periodically update depending on the internationally endorsed procedures and according to the experience gained from the analyses.
- To carry out the analyses according to the Standard Operating Procedures.
- To establish and keep the registration of the analysis data generated so they can be reproduced whenever wanted.
- To look for constant improvement with technical staff having the appropriate profile and being trained for the activities carried out and familiarized with the corresponding quality documentation.
- To meet the customer's requirements established in the signed agreement and contracts.
- To meet the legal and regulatory requirements regarding the service provided.

Obligation of the National Directorate of Environment:

To observe this policy of quality, the National Directorate of Environment undertakes to:

1. To provide the necessary human resources, according to the profiles defined by the Quality Management System of the Laboratory.
2. To guarantee the allocation of financial resources for the purchase of analysis supplies, including reagents, materials, equipment, maintenance services of the equipment and participation in performance analytical tests.
3. To approve and implement the external training plan identified by the Head of the Laboratory, required for the Quality Management System.

Date: Apr/2005

Version 6

1. PURPOSE AND SCOPE OF THE MANUAL

1.1 Purpose

This document reflects the Quality System of the Technical Normalization Department (Laboratory) of the National Directorate of Environment (DINAMA).

The main objectives of the Quality Manual are:

- To spread and support the Quality System of the Laboratory.
- To establish the analytical and administrative procedures that guarantee the quality of analyses and of the service provided by the Laboratory to its customers.

1.2 Scope of the System

The scope of the quality system covers from the reception of the sample in the Laboratory to the issue of the report with the results to the Customer, including the analytical and administrative process and the service subsequent to the issue of the results. Any analysis carried out outside our facilities and that should be engaged with third parties shall be excluded from the scope.

The Laboratory is not responsible for the sampling, in spite of which it provides the flasks required by the customer for each kind of analysis, the minimum volume required for the sample, and the conditioning and preservation of the sample until its arrival to the Laboratory. It does not specify the way in which the sampling should be carried out.

The customers of the Laboratory are mainly from the Departments and Divisions of DINAMA; we have occasionally participated in external programmes such as binational agreements or carried out analyses for third-parties.

1.3 Regulatory references

This Manual has been implemented to meet the General Requirements regarding the competence of Calibration and Assay Laboratories included in the UNIT ISO/IEC 17025:2000 Guide, and in the Standard UNIT-ISO 9001-2000 for Quality Management Systems.

The regulatory references of the Laboratory are mainly:

- Standard ISO/IEC 17025 ***General requirements for the competence of assay and calibration Laboratories.***
- Standard UNIT-ISO 9001:2000 ***Quality Management Systems – Requirements.***
- Decree 253/79 and its amendments. “Standards to avoid environmental pollution through water pollution control”.

1.4 Management requirements

1.4.1 Organization

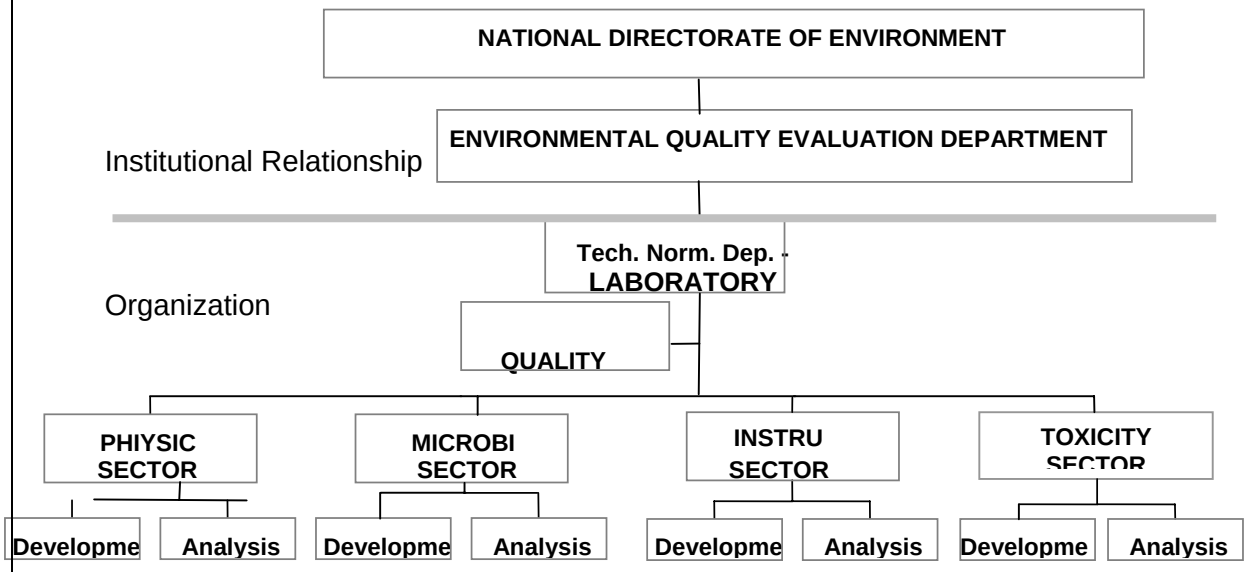
The Laboratory's main assignments are:

- The responsibility of determining the referential analytical methods for the establishment of the natural water quality and discharge control, such as it is stated in Decree 253/79 and its subsequent amendments.
- It is the Laboratory of Reference regarding the analysis of natural water, liquid and solid discharges, both at a national level and regarding international relations.
- It provides analytical service to all the divisions of DINAMA which thus require it through its participation in different programmes. In case external analytical services are required, it is responsible for the selection of the Laboratory, the coordination and the reception of results.

As the Laboratory of Reference it develops the following Specific Functions:

- It elaborates manuals, guides and technical notes to diffuse and exchange information.
- It provides technical consultancy to Municipal Laboratories in issues regarding analytical methodologies.
- It organizes training courses for human resources.
- It provides technical support to different investigation programmes and projects, by means of agreements with the University or other Institutions.

Functional Organization Chart of the Laboratory



The Laboratory has permanent staff (employees) and interns. Interns enter DINAMA to gain practice in environmental analysis Laboratories, to elaborate their graduate or postgraduate thesis in the Laboratory or through a technical agreement for special programmes. This

Laboratory function is authorized by the Head of the Laboratory and supervised by each immediate person in charge, depending on the sector the intern is appointed to.

Interns may be students or academics from the both the analytical and administrative areas.

The intern cooperates with the Laboratory in the following ways:

- Participating in a specific project. The person responsible for the project has to train and advise the intern so that he/she can afterwards develop specific aspects of it.
- Cooperating with the routine tasks. In this case, the intern gives support for the execution of analyses under the supervision of the responsible technicians.
- Adjustment of new assays. The person responsible for the sector should train and advice the intern so that the validation of the technique intending to be implemented can be carried out.

The organizational structure indicating the staff and its responsibilities are included in the folder labelled ***Human Resources,*** where the profiles of the position for each function and the résumés of the Laboratory staff and interns are stated. Interns should fulfil the position profile for the appointed function.

1.4.2 Description of the processes implemented in the Technical Normalization Department of DINAMA

The basic process carried out within the organization and the support processes are presented in the Process Map in Annex I. This Annex also describes the main processes in the flow chart.

1.4.3 Description of the Laboratory Sectors

Annex II shows a schematic plant of the Laboratory. DINAMA's laboratory is organized in the following analytical sectors:

Physicochemical Sector
This sector includes activities of analyses and assays by classical physical and chemical analytical techniques. Annex III includes the analytical techniques implemented in the sector.

Microbiology Sector
This sector covers microbiological analyses activities. Annex III indicates the analytical techniques implemented in the sector.

Instrumental Sector
This sector includes activities of instrumental analytical techniques assays. Annex III indicates the analytical techniques implemented in the sector.

Ecotoxicity Sector
This sector includes activities of toxicity routine analyses as it is stated in Annex III.

Quality Area

There is a person responsible for this area. The Quality Management System covers all the activities carried out in the Department. All the officers and interns are involved in the System and are familiar with its procedures.

Support Activities

Administrative Activities
Supervised by the Head of the Laboratory and executed by all the Laboratory's staff. They cover all the non-technical tasks resulting from the Laboratory functions.

External Service of Equipment Maintenance
The equipment maintenance is subcontracted. The selected company is hired to carry out the preventive and corrective maintenance of equipments, to keep the electrical circuits in good conditions, as well as to supervise and support the installation of equipments.

The performance of the selected company is supervised by the Head of the Laboratory. The selection of the company after fulfilling what is stated in the contract is carried out through procedure PR03 Purchases.

Preparation of Materials
This activity covers cleaning and conditioning of all the sampling containers and analysis materials of the Laboratory. There is one person responsible for this activity, the "Person preparing the material".

2. STRUCTURE OF THE DOCUMENTATION

2.1 Quality Manual Management

- Structure of the Manual.

The manual is divided in 5 chapters covering all the requirements of standards ISO 17025, all the requirements of standard ISO 9001-2000 are considered in these chapters. If the structure of the technical standard guiding the quality management system is modified, the manual's structure shall be revised.

- Codification

The whole manual has the name as heading, and in the foot of the page the version number and issuing date is stated.

- Review and Approval

The quality Manual is approved by the Head of the Laboratory through his/her signature and date in the corresponding page. It is systematically reviewed every two years and reissued when corresponding, despite the fact that it can be reviewed on a shorter period if the Head of the Laboratory or the Person responsible for Quality deem it necessary. This review may or may not produce changes in the document, if it is not modified, the reissuing of the document shall not be necessary. In which case, the Head of the Laboratory shall sign the approval page indicating the date of the new revision, being the manual in force for two more years.

In case a reissuing is required, all the copies of the substituted manual shall be withdrawn and the original shall be filed in the folder labelled ***"Obsolete documents of QMS (Quality Management System)"***, with the following caption in the cover "SUBSTITUTED ON XX/XX/XX". The person responsible for quality shall be the one in charge of destroying all the other controlled copies in circulation.

- Distribution

The distribution of the quality manual is carried out according to the following scheme:

3 controlled copies – one for the Head of the Laboratory and two for its library, free access for the staff.

2 uncontrolled copies – one for the Director of DINAMA and one for the Director of the Environmental Quality Evaluation Division.

2.2 Document control

All the documents of the quality management system of the Laboratory are controlled according to procedure ***02 Document control***. This procedure guarantees that no obsolete documents are circulating in the Laboratory and that all the members of the organization are handling duly revised and approved documents.

The obsolete documents are taken out of circulation and destroyed in order to avoid inconsistencies in the system.

The document changes are approved and revised by the same function that originally issued them, enabling manuscript provisional changes provided that the approval date of the change is duly identified in all the controlled copies.

All the documents and formats incorporated into the quality management system are duly identified with a title and a code that appears described in procedure ***01 Elaboration of Documents*** is allocated to them.

As of 30-08-04 the word ***version*** shall be substituted with: revision, issue, edition, in the sense of printing a new document. The term ***revision*** is still valid regarding the new reading of a document when updated.

2.3 Control of Records

All the records of the Laboratory's quality management system are controlled according to procedure ***05 Control of Records***. This procedure guarantees that the records generated in the Laboratory are protected from loss or deterioration during the period they are considered useful. The maintenance of records of data, reports and information files shall enable the sample follow-up and the recovery of information whenever required.

The records are withdrawn from circulation and stored for the period stated in the abovementioned procedure and subsequently prepared for their destruction.

Changes of the record formats are carried out according to the needs stated by the technician or the Person responsible for the sector, without saving a copy of the former record format, these formats are identified with a code, title and date of coming into operation; enabling manuscript provisional modifications provided that they are duly identified.

When the filing period expires, they are destroyed with the authorization of the Person responsible for Quality.

2.4 Structure of the documentation.

<table>
<tr>
<td rowspan="4">Quality Manual</td>
<td rowspan="2">Analytical Quality Control Manual</td>
<td rowspan="4">Standard Operating Procedures</td>
<td rowspan="2">Equipment Maintenance and Control Folder</td>
<td>Analysis records</td>
<td rowspan="4">Report on Issued Results</td>
<td rowspan="4">General Quality Documents</td>
</tr>
<tr>
<td>Quality records</td>
</tr>
<tr>
<td rowspan="2">Quality Management Manual</td>
<td rowspan="2">Equipment Use Instructions</td>
<td>Equipment Maintenance and Control records</td>
</tr>
<tr>
<td>Management and Administrative Procedures records</td>
</tr>
</table>

- Analytical Quality Control Manual: it deals with specific procedures for controlling the quality of analyses.
- Management Manual: it describes the general management procedures identified in the Quality Manual.
- Standard Operating Procedures: corresponding to analysis procedures.
- Equipment Maintenance and Control Folder: it covers the Equipment Maintenance and Control Programme, as well as records derived from the programme's execution..
- Equipment Use Instructions: corresponding to operation instructions of the equipment used for the analyses.
- Records: corresponding to all those generated by the Quality Management System.
- General Quality Documents: all those documents and records of the Quality Management System that do not have a unique defined format, nor codification, such as agreements with customers, quality planning, audit verification lists, quality meeting minutes, etc.

3. STRUCTURE OF THE MANAGEMENT SYSTEM

3.1 Review of Requests, agreements and contracts.

Depending on the technical capacity of the Laboratory and on the customer request, the Head of the Laboratory shall elaborate an "Agreement" according to procedure ***PR10 Agreement Review.***

Before the signature of the agreement, all legal and regulatory requirements regarding the provided service shall be analysed, as well as any other additional requirement requested by

the customer. Requirements that arise subsequently shall be analysed at that time and the agreements shall be reviewed, if appropriate.

This agreement is accepted by the parties by means of the signature and it is filed in the binder "Sample Entry Filing Records", if any of the parties is interested in changing it, it shall be revised following the same procedure.

In the case of external customers, the Head of the Laboratory evaluates the Laboratory's capacity to meet the customer's requirements and notifies by telephone regarding availability and cost.

Samples should be entered together with the entry filing record which shall serve as a **contract** with the customer. The filing record is previously provided by the Laboratory for each kind of sample. In case it is not entered with the corresponding filing record, the customer together with the person receiving samples should fill in the generic filing record at the time of delivery. These filing records should state all the relevant data of the sample and the parameters requested. The person in charge of the samples assigns an analysis number to each sample, this number is unique and correlative. The entry records are filed in the folder labelled ***Entry Records of Samples***, located in the Laboratory's library, according to procedure ***PR14 Sample Entering and Handling***, this procedure states how to carry out the contract's review.

Analyses corresponding to special agreements or programmes, apart from the routine programmes in support of other sectors of DINAMA, shall be studied for each specific case, saving a copy of the agreements or programmes in the folder labelled ***Special Programmes*** in the Laboratory and it will contain the agreements.

In order to access the analysis services, we shall proceed depending on the customer:

Service to DINAMA and Agreements with institutions

The analysis request is coordinated via telephone or fax, according to procedure ***PR13 Sampling coordination and sample preservation***, requesting sampling flasks and sample entry filing records and stating the date when the flasks shall be collected and the date in which the sample shall enter the Laboratory.

The customer (DINAMA or another one) shall hand over the samples with the Entry Filing Record where the requested parameters for each sample are stated.

After the analysis is carried out, the results are reported to the interested party.

Service of Analyses to Third Parties

According to Decree 253/79, where the analyses list and their costs are stated, analyses to third parties shall be paid to MVOTMA's account in the BROU (Bank of the Oriental Republic of Uruguay).

The people in charge or the Head of the Laboratory make the budget and send it to the interested party. The day when the samples shall be entered is coordinated following the previous procedure. The result report is issued to the customer when a copy of the corresponding deposit at the BROU is handed over.

3.2 Subcontracting of assays and calibrations

Subcontracting is only carried out for internal analyses of DINAMA and for those parameters that the Laboratory does not analyse, for which the following procedure is applied:

a) A national Laboratory with guaranteed results is selected based on: whether the Laboratory is authorized, whether it participates in inter-calibration exercises with acknowledged agencies, the Laboratory's credibility and the certainty that it carries out the analysis of interest.

b) The subcontracting of the analysis is coordinated in writing with the selected Laboratory.

c) A report is elaborated for the customer, attaching a copy of the subcontracted Laboratory's report.

d) The original result report is filed attached to the report issued to the customer.

3.3 Purchase of Services and Supplies

3.3.1 Purchase of materials

Purchases of materials, reagents and equipment are carried out according to procedure ***PR03 Purchases***. It is applied for purchases whose total amount does not exceed the direct purchase amount stated by the Nation's General Accounting Office. The requesting technician is responsible for verifying the maximum amount by MVOTMA's Accounting Office before proceeding.

In the case of purchases whose amount exceeds the direct purchase amount, a tendering process is started, where the laboratory is in charge of making the list of materials with their quality specifications and MVOTMA's Administration is in charge of organizing the tender and the award by an advisory committee appointed by MVOTMA and including at least one technician from the Laboratory. This committee compares the offers, analysing all the specifications according to the document. The receipt of materials and follow-up of the delivery is carried out in the same way as for a direct purchase, according to procedure ***PR03 Purchases***.

3.3.2 Hiring of Services outside the Laboratory

In the case of hiring of any external service, such as the preventive and corrective maintenance of equipment, conditioning of the Laboratory infrastructure, training courses, etc, any of the two purchase procedures described in the previous section is followed, depending on the total amount of the service. This procedure is described in ***PR03 Purchases***.

3.3.3 Evaluation of Suppliers

Every year we proceed to evaluate suppliers according to procedure ***PR04 Evaluation of Suppliers.***

3.3.4 Purchase Planning

According to the analyses and new programmes that shall be implemented, the purchase of equipment and reagents necessary for the operation, as well as the renewal of infrastructure is planned. The People responsible for the analyses in each sector are in charge of arranging the purchase of inputs in order to have them when needed. The Head of the Laboratory is responsible for implementing this process to send to the Division Direction the funding request in order to make the purchase effective, or the tendering request in case of exceeding the amount stated by the Nation's General Accounting Office.

3.3.5 Verification of purchased products

The verification of purchased products is the responsibility of the person in charge of purchases, the process is detailed on procedure ***PR03 Purchases.***

3.3.6 Claims regarding made purchases

The claims regarding unsatisfactory acquired products are made by the person responsible for purchases, following procedure ***PR03 Purchases.***

3.4 Customer Services

The Laboratory provides customers with the sampling containers conditioned according to the requested analysis and carries out the analyses.

When the analysis is coordinated, the People responsible for each sector where the assay should be carried out shall examine the possibility of carrying it out in due time and form, and if due to any particular reason such sector cannot process the requested samples, the person in charge reports this situation to the Head of the Laboratory so he/she can contact the customer in order to notify him/her that it will be impossible to process the requested samples. This procedure is described in ***PR 10 Review of the agreement.***

The result report issued by our Laboratory belongs to the customer, the original analysis report is handed over to the customer and a copy is filed in the Laboratory, in the folder ***Issued Results***, restricting its use only to the Laboratory technicians. It is not available for the public and the dissemination of the reported information is not allowed. Permanent workers and interns undertake to maintain secrecy by means of the signature of a confidentiality agreement, which is kept in the folder of Issued Results.

Only the customer can request information on the analysis status, used methodology and issuing of results. The Head of the Laboratory or the People responsible for the involved sectors are authorized to provide the requested information.

3.5 Feedback from Customers

The customer voice is taken into account in the claims and a customer satisfaction survey. These channels are instrumented and their evaluation is an entrance element for the review by the Direction aiming at satisfying the customer's requirements.

The survey stated in ***RGC12 Customer Satisfaction Survey*** is issued at least once a year to all the customers that interact with the Laboratory. Afterwards, their annual evaluation is carried out.

Claims are made according to procedure ***PR11 Customer Complaints***. The technician receiving the customer claim request is the one in charge of registering it in the book of **"Claims and Non-conformities",** and notifies it to the Head

The Head of the Laboratory puts the claim in course, follows it up, answers the customer and registers the claim result. If it is necessary to take corrective actions, procedure ***PR07 Generation and Implementation of Corrective and Preventive Actions*** is followed.

3.6 Non-Conformities Control

The treatment of Non-Conformities is carried out according to ***Procedure PR06 Non-conformity Control***, which guarantees that all necessary actions are taken in order to correct them, that the responsible people are identified, as well as the study of what caused them. The records are filed in the book of ***"Claims and Non-Conformities".***

Non-conformities on assays might be detected by the customer through a claim or within the Laboratory. In any of both cases, if possible, the analysis is carried out once again in the same sample.

3.6.1 Control of non-conformity analysis results detected in the Laboratory:

a) If the result report has not been handed over to the customer yet: the analysis is carried out again, if due to sample preservation reasons is not possible to analyse it again, the Person Responsible for Quality is notified and the unreported datum is registered on the Analysis Route, stating the causes, and the omission reasons are clarified in the result issue in the result report handed over to the Customer.

b) If the report has been already handed over to the customer, it shall be registered as non-conformity and the same procedure as when the non-conformity is detected by the customer is followed, according to section 3.6.2 a).

3.6.2 Control of non-conformities detected by the customer:

It is registered as a claim from the customer according to ***PR11 Customer Claims***.

a) If the non-conformity is confirmed:

- The previous result on the previously issued report is annulled.
- The customer is notified by issuing a new report of analysis results deleting the previously issued result.
- This new report is copied and attached to the original from the customer, in case it is not possible to have the original, it is attached to the old copy of the Laboratory, annulling the previous result. It is filed in the folder of issued results, with the caption "ANNULLED".

b) If eventually the complaint is not a non-conformity:

- The customer is notified and it is registered in the above mentioned book considering the claim finalised.

3.7 Corrective Actions

When a Corrective Action is needed as a result of a non-conformity, procedure ***PR07 Corrective and Preventive Actions*** is followed, which identifies who is responsible for it, guarantees that the appropriate corrective actions are carried out in order to correct the non-conformity and to avoid its repetition by analysing its causes, and establishes an implementation and verification term regarding the effectiveness of the actions taken.

The corrective action/s to be taken are discussed among the involved technicians and Quality.

Corrective actions are recorded in the book ***"Claims and Non-conformities"***, and the Person Responsible for Quality is the one in charge of disseminating them among the involved staff.

3.8 Preventive Actions

When a potential problem or an improvement opportunity is detected, based on the documentation study, improvement meetings, reviews by the direction or internal audits, procedure ***PR07 Corrective and Preventive Actions*** is carried out, which guarantees that the causes are examined and the appropriate preventive action is taken, it establishes the implementation and verification term of the effectiveness of the preventive action implemented.

In the case of a potential problem of non-conformity, the same procedure as for a corrective action is implemented.

3.9 Internal Audits

Internal audits are carried out annually with the purpose of guaranteeing the operation of the quality management system, evaluating the achievement of the quality goals and determining improvement opportunities in the quality management system.

The Person responsible for quality is in charge of coordination, the selection of auditors and implementation of auditing and follow-up programme of the proposed corrective actions. Internal audits are carried out according to procedure ***PR 09 Internal Audits.***

3.10 Reviews by the Direction

The review of the Quality Management System by the Direction is the responsibility of the Head of the Laboratory, and it is annually carried out with the coordination of the Person responsible for Quality and the participation of Technicians.

This process is described in procedure ***PR08 Review by the Direction***. The existing documentation, the internal and external audit reports, the minutes of quality meetings, non-conformity records, corrective and preventive actions, periodical goals and indicators of each process shall be taken into account for the review.

The documentation is examined and its reissuing or the modification of processes is proposed whenever necessary. The potential changes to the Quality Policy and the management system are evaluated at these meetings, and new goals are established for that year.

From the analysis of the entrance elements, a report with the conclusions is elaborated and the actions to be taken are stated in order to improve the Quality Management System.

3.11 Responsibility of the Direction

3.11.1 Commitment by the Direction

The Head of the Laboratory states his/her responsibility regarding the maintenance and improvement of the Quality Management System.

3.11.2 Focus on the customer

The Head of the Laboratory guarantees the fulfilment of the customer's requirements through the review of customer contracts and the establishment of the appropriate communication channels with each one of the internal or external customers.

In the case of any exceptional situation, the customer is notified orally or in writing and it is solved with the customer's approval.

The complaints service is carried out according to procedure ***PR11 Complaints*** and a record is kept for future evaluation.

3.11.3 Quality Policy

It is established and approved by the Head of the Laboratory. The policy agrees with the Laboratory's purpose and provides a reference framework to establish and examine the quality objectives.

The Person responsible for Quality is in charge of guaranteeing the dissemination of the quality policy, which shall be executed in training activities and it shall be placed on an approachable place for the whole staff. A copy is send to customers together with the contract.

3.11.4 Planning

The Person in charge of Quality plans the development and control of processes belonging to the Quality Management System yearly, sending a report to the Head of the Laboratory for his/her approval.

3.11.5 Quality Objectives

They are yearly established by the Head of the Laboratory in the Quality Management System Review of the Direction. They are presented in a logical chart: indicating the objective, term, person in charge of execution, indicator and its verification. The objective follow-up is carried out through quality meetings between at least the Person responsible for Quality and the Head of the Laboratory.

The Person responsible for Quality is in charge of notifying the objectives.

3.11.6 Quality System Planning

The head of the Laboratory is in charge of guaranteeing that the Quality Management System planning is carried out by planning the System processes and their interactions.

When any project, programme, etc. emerges, that alters or changes the operation according to the established planning, the Head of the Laboratory, together with the Person responsible for Quality and for the Sector that may be affected, shall decide the measures to be taken, stating how the new situation shall be developed, the controls to be implemented, if needed, and when this implementation shall be started. Eventually, the Head of the Department shall be in charge of approving the alterations to the initial plan.

3.11.7 Responsibility, authority and communication.

Responsibilities and authorities are defined in the function description of section 5.2 of this manual.

3.11.8 Representative of the direction

The Person responsible for quality is the direction representative and he/she is responsible for implementing and maintaining the Quality Management System..

3.11.9 Internal communication

Internal communication is carried out orally and in writing, with a whiteboard used as a communication means.

3.12 Process follow-up and measurement

The follow-up and measurement of processes is carried out according to the table with the same name. This table states the involved processes, the person in charge of carrying it out, when and how it shall be done and the measurement indicators, when appropriate. This table is examined annually, together with the Review by the Direction and the follow-up is carried out in the quality meetings, filing its record and estimated indicators in the binder where records of ***Quality Meeting RGC08*** are kept.

The table is filed in the Binder of Quality System Documents.

The follow-up of analytical procedures is carried out as it is stated in each Standard Operating Procedure by using charts of accuracy and precision control, as appropriate.

3.13 Data Analysis

The analysis of data and process measurement indicators of the Quality Management system provides information to detect system weaknesses and to highlight improvement opportunities.

It is carried out annually and the Head of the Laboratory and the Person responsible for Quality are in charge. It is recorded in the folder ***Quality Meetings RGC08.*** This analysis is used as an entry for the Review by the Direction.

Data analysis involves:

- a trend study of control charts of the parameters usually requested by customers and/or defined as keypoints when carrying out analyses,
- an evaluation of the results of the inter-laboratory exercises (% of agreed parameters/total of carried out parameters),
- customer satisfaction survey,
- non-conformity analysis,
- supplier evaluation analysis,
- verification of the agreement compliance, and
- customer complaints.

4. TECHNICAL REQUIREMENTS

4.1 General Aspects

4.2 Planning of the product execution

According to the customer's request or to the strategic planning of the Direction of the Environmental Evaluation Division, the Head of the Laboratory decides the inclusion of new assays in the Laboratory as well as the volume of samples that shall be monthly processed in each area. This is carried out in order to avoid the excess of assays that may cause a failure to comply with the stipulated terms or that may damage the quality of the issued results.

4.3 Processes related to customers

During the planning we contact the requesting or potential customers in order to agree on the basic requirements so we are able to adjust or select the most appropriate analytical methodologies or techniques.

4.4 Design and development

When a new analysis is carried out at the customer's request or due to the internal interest of the Department, the Laboratory carries previously out the development of this parameter determination, in the matrix defined to use a given standard, following procedure ***PR17 Analytical Development.***

The Laboratory does not design methodologies of its own, the executed analyses are based on standardised techniques of the Environmental Protection Agency (EPA), or Standard Methods for Water and Wastewater (APHA). These techniques are validated within the development process according to the technique validation procedure of the Analytical Quality Control Manual.

The stages of development verification and validation are carried out concurrently.

4.5 Production and provision of services

The Person responsible for each sector appoints an analyst responsible for each technique, said analyst must know the Standard Operating Procedure, the equipment involved in the assay, as well as its operational controls, and a follow-up of the performance during his/her training by the Person responsible for the sector is advised. Once it is finished, the analyst is responsible for executing all the stated controls and for issuing the results, as well as for the decision to repeat the assay in case of non-conformity.

The process that covers from the entry of the sample to the Laboratory, to the issuing of the result report is valid by the determination of the parameters of the results delivery terms and the number of analysis carried out compared to the requested analyses. Whenever the report

of the results is issued, the delivery terms and the executed analyses are compared to the analyses requested by the customer, in order to check the compliance of the agreement and contract, stating it in the entry filing record. The revalidation of the process is presented in the Activity annual report and it is an entrance element for the Review of the Direction.

4.6 Production control

Controls for each assays are stated in the corresponding standard operating procedures and are implemented by the analyst under the supervision of the Person responsible for quality.

Acceptance or rejection limits of the production quality are stated according to the quality control procedures presented in the Analytical Quality Control Manual.

5. RESOURCE MANAGEMENT

5.1 Resource Provision

The Head of the Laboratory sends to the Direction of the Division a report stating the provision of financial and/or human resources required for the operation of the Laboratory. The direction of the Division evaluates said report and carries out the corresponding proceedings in the Administration of DINAMA for the allocation of resources.

5.2 Human Resources

5.2.1 Functions of the Organizational Structure

It should be noted that these are not descriptions of positions but of functions, thus, there is no necessarily one person appointed for each function.

a) Functions of the Head of the Laboratory

This person is under the supervision of DINAMA's Evaluation Division Director and shall be in responsible for:

- Being responsible for the observance of Laboratory policies and regulations.
- Planning and supervising the Laboratory activities.
- Coordinating the Laboratory activities with the different sectors of DINAMA.
- Authorizing documents issued by the Laboratory staff.
- Reviewing and controlling the reports and comments issued by the Laboratory staff.
- Supervising the adequate use of the Laboratory installations.
- Authorizing the results and reports of results reported by the Laboratory.
- Implementing plans regarding the analytical capacity development of the Laboratory.
- Implementing plans regarding the continuous training of staff.
- Proposing the contracting and reclassification of functions.

- Supervising the use of resources and identification of improvable areas.
- Evaluating and promoting the incorporation of new methodologies and analysis techniques, according to the customers' needs and the Laboratory's technical and financial means.
- Informing the Direction of the Division on the staff performance and the use of resources.
- Coordinating and appointing supervisors for the interns working in the Laboratory.
- Coordinating work meetings with the Laboratory technicians.
- Establishing relations with other Heads of Laboratory in the region, as well as with Research Units of Universities, institutes and other institutions related to the environment.

b) Functions of the Person Responsible for Quality

Under the supervision of the Head of the Laboratory.

This person is responsible for the Quality Management programme, which includes:

- Guaranteeing the quality of the information reported.
- Supervising and assessing validation data of analytical techniques that should be included in the routine work of the Laboratory.
- Evaluating the suitability of the analytical methodology used for the analysis of samples.
- Coordinating the meetings for the improvement of the quality management system.
- Coordinating internal audits of the quality management system.
- Internally diffusing the policy of quality of the Laboratory.
- Planning and implementing plans related to the Quality management system.
- Elaborating quality plans for the processes that thus require it.
- Proposing to the Head of the Laboratory training plans in the quality area.
- Supervising the development of the equipment maintenance and control process.
- Formulating, implementing, supervising and evaluating the quality control programmes applied to the different areas of the Laboratory.

c) Functions of the Person Responsible for Development

Under the supervision of the Head of the Laboratory.

This person is responsible for the adaptation of analytical methodologies and techniques, as well as for the improvements in those regarding routine use of the Laboratory, which includes:

- Planning the incorporation of normalized analytical methodologies and techniques applicable to his/her sector.
- Evaluating and proposing the techniques and methodologies to be incorporated according to the Head of the Laboratory's instructions, considering the customers' needs and the technical and financial means of the Laboratory.
- Executing and supervising the adjustment of the selected analytical techniques and evaluating the results before their incorporation into the system.

- Evaluating the incorporation requirements of equipment for the improvement of the currently used techniques or the incorporation of new analytical techniques.
- Elaborating a final report after the evaluation of each revised technique.
- Elaborating reports, articles, technical notes or any other required publication related to his/her area.
- Spreading within the organization through internal or external technical and informative meetings the progress status of the new techniques or methodologies used once they have been validated by him/her.
- Providing training to other Laboratory staff regarding the evaluated and incorporated techniques.

d) Functions of the Person Responsible for Analysis

Under the supervision of the Head of the Laboratory.

This person is responsible for coordinating any necessary activity for the execution of the routine analysis of the samples delivered to the Laboratory, from their entry to the presentation of results.

- To verify the quality of the information reported in his/her sector.
- To execute the indications of the analytical quality control programme regarding Techniques of his/her sector.
- To cooperate with the person responsible for Quality to implement the programme for quality assurance in his/her sector.
- To cooperate with the Head of the Laboratory regarding technical aspects related to his/her sector.
- To elaborate reports, documents an manuals regarding the sector of his/her responsibility.
- To present to the Head of the Laboratory reports on the activities developed in his/her sector.
- To participate in internal or external technical and informative meetings.
- To advise and train technicians belonging to the organization.
- To elaborate internship plans and supervise them when they are appointed by the Head of the Laboratory for his/her sector.
- To notify any equipment failure detected in his/her sector for its repair.
- To propose the training plans for the staff on his/her charge when deemed convenient.
- To plan and execute the purchase of reagents and supplies for the necessary analysis in his sector.
- To evaluate the convenience of incorporating or reconfiguring the equipment when required.

e) Functions of the analyst

Under the supervision of the Person Responsible for analysis or development, depending on the assigned task.

This person is responsible for:

- Executing the established routine analyses, as well as the necessary activities for their execution.
- Carrying out the analytical quality controls of the used technique, according to the indications of the Quality Control programme.
- Carrying out the control of the used equipments according to their control schedule.
- Carrying out the assigned activities of the development programmes.
- Participating in meetings regarding technique adjustment and improvements.
- Participating in the elaboration of the documents related to the technique under his/her responsibility.

f) Person preparing the material

Being under the supervision of the Person responsible for development or analyses, this person is responsible for:

- The preparation of the materials used for the analyses: common and special cleansing and rinsing.
- Preparing the sampling flasks previously requested by the customer, provided that the analysis request has been approved by the Head of the Laboratory.
- The sterilization of the microbiology materials and their waste.
- Conditioning of the working table.
- The operation of the distiller and of the automatic washing machine.
- The final disposal of the waste generated in the analyses.

5.2.2 Position Profile

The Head of the Laboratory establishes the profiles for each position. These profiles, together with the merit relation of all the members of the organization, are filed under the ***Human Resources*** folder. They are yearly reviewed by the Head of the Laboratory, on the basis of which the training programmes are established.

The training identified as necessary is planned, implemented and its efficiency is evaluated according to the procedure ***PR 12 Training***. The annual training programme is filed in the same folder.

Appendix IV describes the permanent staff of the Laboratory and their corresponding position.

5.3 Environmental installations and conditions

5.3.1 Installations

Access to the Laboratory is restricted to the staff. Any unrelated person shall be previously authorized by the Head of the Laboratory or by the People Responsible for each sector.

Appendix II of this manual presents a scheme describing DINAMA's Laboratory.

Its facilities are the following:

- Light and energy source from UTE (the State Electric Company)

- Voltage regulators.
- Emergency current for equipment that must be operating 24hrs., such as incubators, refrigerators and freezers.
- Air conditioning equipment with temperature control.
- Distilled water automatically generated by a glass distiller.
- Distilled and deionised water in line.
- Safety – There is a Safety Programme.

DINAMA's Laboratory can be divided in two areas, depending on the activity developed in them:

a) Laboratories:

- Sampling treatment for the determination of Metals.
- Sampling treatment for instrumental analysis.
- Ecotoxicity.
- Physicochemical I.
- Physicochemical II.
- Instrumental.
- Microbiology.

We tried to divide the Laboratories where the sampling processing and the analysis itself is carried out, in order to minimize the potential pollution sources or any other incompatibility among the different analytical procedures.

b) General Sectors

- Meeting Room and offices
- Dining Room

In the Meeting room and offices the staff carries out the administrative activities of the Laboratory.

5.3.2 Working Environment

The Head of the Laboratory is responsible for ensuring the adequate environmental conditions required by specifications, procedures and methods, provided that they have an impact on the results. People Responsible for each sector are in charge of establishing and carrying out the verification of the environmental conditions required for the technical activities.

When the environmental conditions question the result of the assay, the Person Responsible for the sector should inform and stop the corresponding assay, and include a note in the issued results that may be affected.

5.4 Assay methods and methods validations

The Laboratory validates the normalized methods currently used, as well as their expansions or modifications, to confirm that the methods are appropriate for the intended use, following the procedure ***PGC 01 Technique Validation.***

The Laboratory registers the obtained results and the procedures used for the validation.

5.5 Equipments

All the equipment of DINAMA's Laboratory is stated in the corresponding inventory located in the file ***Equipment Maintenance and Control***.

To guarantee the appropriate operation of the equipment, there are user guides of the equipment for those that thus require it, depending on the difficulty of the operation. The guides are located next to the equipment. The existing guides are stated in the "List of Valid Documents".

We have an Equipment Maintenance and Control programme that guarantees the status of operation, inspection and preventive maintenance of all equipment related to analytical determinations, which is included in the aforementioned folder. The programme is annually revised and it states the frequency of preventive maintenance and of operation controls, provides guidance on how to carry them out and identifies the people responsible for its execution.

The Person responsible for Quality, together with the one responsible for maintenance, establishes the schedule depending on the use frequency, the stability of the equipment and the accuracy of the operation variables required for the analytical determinations. This programme is approved by the Head of the Laboratory.

The equipment used for the routine analyses are listed in the ***Equipment Maintenance and control*** folder, where there is a record stating:

- The equipment specifications.
- The instructions of the preventive maintenance to be carried out.
- The controls of the operation variables or their calibration.

Together with the record there are the preventive and corrective maintenance records, as well as the records of the operation controls and calibrations, if appropriate.

The Laboratory has hired a company that provides permanent service of preventive and corrective maintenance for most of the equipments, except for those that have an exclusive representative firm in the country which are derived to the supplier's technical service.

If the control of the operational variables do not match the specifications stated for each equipment, it is notified to the person responsible for maintenance.

It is the responsibility of Quality to carry out the Control and Maintenance programme of the equipment.

5.6 Measurement traceability

The equipment used for assays is calibrated before being used, according to the Programme for the Maintenance and Calibration of Equipments.

The reference patterns are calibrated by an organism that ensures traceability and are only used for calibration.

<u>Equipment calibration:</u>

The Laboratory carries out calibrations and verifications of the equipment using primary patterns or reference materials that guarantee their comparison with certified patterns or reference material.

The folder **Documents of the Quality Management System**, includes a List of primary patterns and Reference material used in the Laboratory, the certificate of the patterns is filed in the section of Quality Certificates.

Analysis results:

Each parameter determined in the Laboratory corresponds to a folder including the recent and currently in use analysis routes.

The assay results of each sample are registered in the corresponding analysis routes, where they are identified by the Laboratory analysis number.

The analysis number is given to each sample by the person in charge for its entry and it is recorded in the Sample Record book, in the Entry Record given by the customer and in each of the sample containers. Only one analysis number is given to all the containers having a sample from a specific sampling point, which were extracted at the same time.

Each analyst is responsible for maintaining the individuality of samples.

Analysis record:

The analyses are registered in the analysis route forms of each kind of parameter. The analysis routes are numbered correlatively.

Within the forms or analysis routes the following is recorded:

- The date.
- The used solutions.
- The dilutions of the standards prepared for the calibration curve.
- The collected measures.
- The analyst responsible for the assay.
- The analytical controls carried out.

The analysis routes are records of the Quality System and as such they are ruled by procedure ***PR 05 Record Control***.

Preparation of the Standard Solutions:

The preparation of the stock standard solutions and reagents is recorded in the “Preparation of Solutions Form” of each technique, this form is in the folder corresponding to each parameter.

The solution flasks are labelled with a label containing the name of the technician who prepared it, the name of the solution, the concentration, the storage conditions and the disposal date. They may be identified with an initial letter (S, V or R, indicating whether it is a primary pattern, a standard that has to be valued or a reagent, respectively) followed by a 6-digit number indicating the preparation date, if thus the person responsible for the Sector deems convenient.

5.7 Sampling

The sampling is not carried out by the Laboratory, it is on the Customer’s charge.

Procedure ***PR 13 Sampling Coordination and Sample Preservation*** defines the stages for the coordination of the sampling, the preservation conditions during the transfer and in the Laboratory, the required minimum sample amount, the flask type, the presence or absence of air chamber and the specifications for the identification of the sample and the fill-in of the analysis request form.

The Laboratory is responsible for:

- Providing different kinds of containers where the samples are taken, depending on the analyses requested by the customer and the entry form of the samples.
- Conditioning the sample containers: cleaning, special rinses and sterilization, as appropriate.
- Indicating the required minimum amount of the sample, the sample preservation and the presence or absence of an air chamber in the container.
- In case that the sample needs to be preserved with any chemical agent: to provide the sampler with the preserver or to add it in the corresponding container at the Laboratory, before the collection or immediately after receiving the sample.
- At the reception, controlling the appropriate filling-in of the entry form, verifying the agreement, checking whether the samples are in the appropriate conditions for the analysis.

5.8 Sample Handling.

Procedure ***PR14*** for the ***Sample Entering and Handling*** specifies the handling of the sample from its entrance to the Laboratory to its final disposal.

From the reception, during the analysis and until the issue of the report, samples are stored at 4ºC in the corresponding sector refrigerators.

After delivering the final report to the customer, their final disposition is carried out whenever possible, due to the amount of sample delivered or the analytical process, a reference sample is saved. The reference sample is stored at 4ºC at least for 10 days after issuing the report to the customer.

The final disposal of the samples is carried out according to the ***PR15 Final Disposal of the generated waste***.

5.9 Assurance of the Assay Results Quality

The assurance of the quality of the issued results is carried out through the procedures stated in the **Analytical Quality Control Manual**.

5.9.1 Responsible for the Analysis:

The analyst responsible for each technique should make sure that the analyses requested by the customer are carried out in due time and form, he/she is also responsible for calculating the corresponding results, each analytical procedure states what kind of control should be implemented and the actions to take, provided that the control conformity is not verified.

Results are calculated by the analysts and are recorded in the analysis route, the Person Responsible for the analysis sector shall verify the issued results before issuing the report.

Routine analyses are carried out according to the ***Standard Operating Procedures*** in force and updated by the Laboratory. These are located in the place of work of the analysts.

5.9.2 Internal Quality Control Programme:

The analytical work methodology to guarantee the quality of the results is defined in the ***Analytical Quality Control Manual***, which states the specific Quality Control Procedures for each Technique and the General Quality Control Procedures.

5.9.3 External Quality Control

Participation in Inter-Laboratory Exercises

The participation in Inter-Laboratory exercises involves the planning, the recording of the participation and the evaluation of results, a process that is carried out according to what is stated in Appendix II of the Analytical Quality Control Manual.

The folder named ***Inter-Laboratory Exercises*** defines the participation plan corresponding to each year, indicating parameters, organizing entity, approximate date and matrix.

The organizing entity should provide test samples codified with the indications of the parameters to analyse.

It should receive the indications to handle the samples and indications to issue the results.

The Inter-Laboratory results and the evaluation sent by the organizing institution are filed in the folder named ***Inter-Laboratory Exercises***. Once the result has been received, if there is any result outside the acceptance limits, it shall be registered as non-conformity and the cause is investigated in order to take the corresponding corrective action, according to ***PR 06 Non-conformity control***.

5.10 Report on the results

The report is issued as a hard copy, being this one the only valid format.

The report on the results does not have a defined format and shall include at least the following information:

- The date of the report.
- A reference to the sample's origin: of the industry or the location of the sampling point, as appropriate.
- The sampling point.
- The sampling date (if it differs in more than one day from the date of entrance to the Laboratory this date is also included).
- The person responsible for the sampling.
- The applicant: corresponding to the customer.
- The requested analysis with the results.
- Observations if needed.
- The analysts responsible for the requested analyses.
- Authorized signature.

The analytical methodology to be used is indicated beforehand to the customer through the **Agreement with the customer**, so it is not included in the report.

Before the signature, the Head of the Laboratory verifies the consistency of the data of one sample and verifies by the signature the fulfilment of the agreement in the Entry Record.
A copy of the original is made; in case the Head of the Laboratory is not available the reports are issued with the signature of the Head's substitute. The copy is filed in the folder ***Issued Results***, by the analysis number and the kind of matrix. The original is handed over to the customer at the signature of the delivery receipt, which is filed in the same folder. The date of the report issuing is registered in the book ***Sample Entrance***.

The Head of the Laboratory is responsible for the delivery of the report to the customer. In case some amendments are needed in the Report after it has been handed over to the customer, we should proceed as indicated in the section ***Non-Conformity Control*** of this Manual.

6. APPROVAL AND REGISTRATION OF REVIEWS

Version	Date	Amendments
1	October 2000	First issue
2	August 2002	Second issue
3	May 2003	Third issue
4	August 2005	Fourth issue

REVISED	**DATE**
APPROVED	**DATE**

ANEXOS

ANEXO I: PROCESOS DESARROLLADOS EN EL LABORATORIO DE DINAMA.

Diagrama de flujo general procesos del Departamento

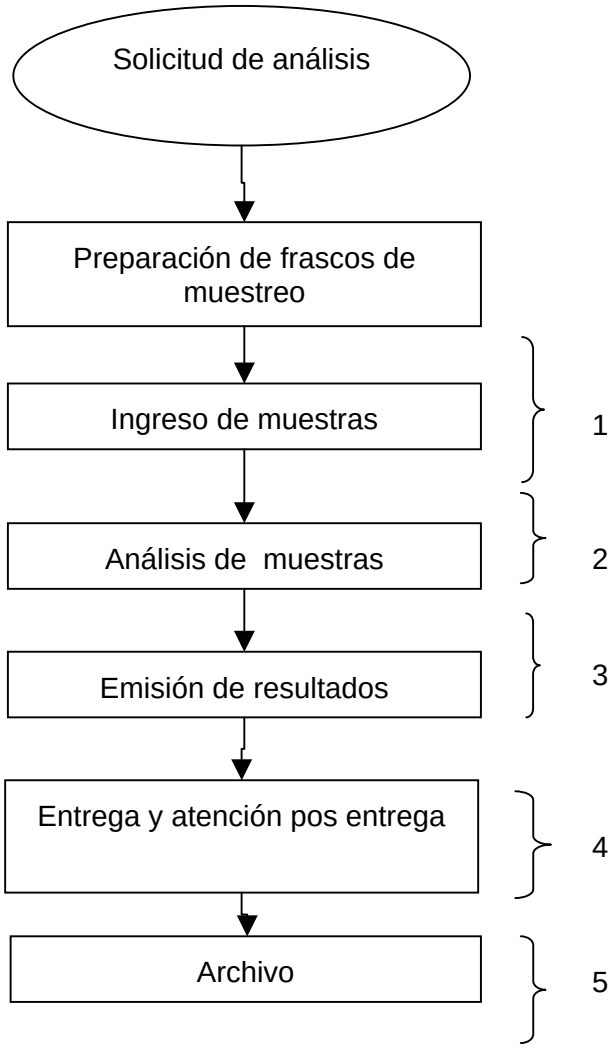


Diagrama de flujo ingreso de muestras (1)

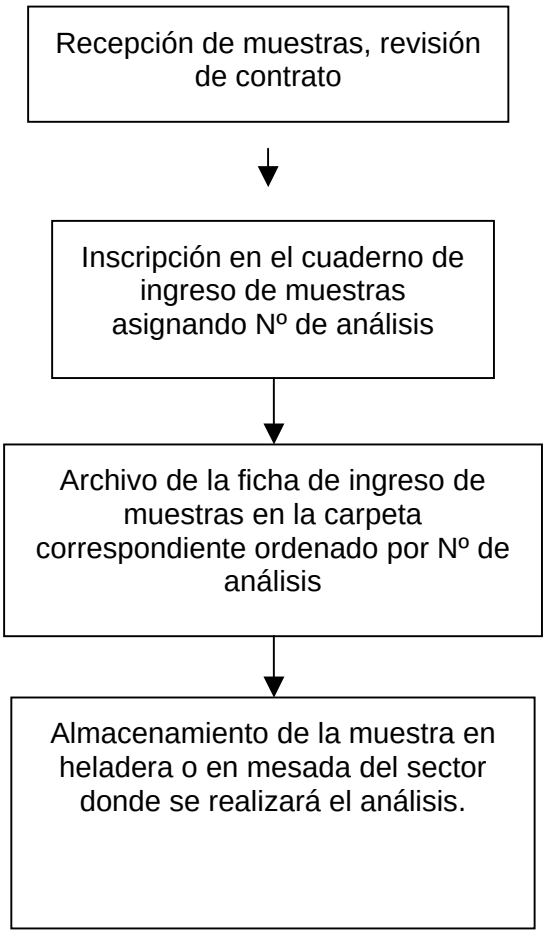


Diagrama de flujo ingreso de análisis de rutina (2)

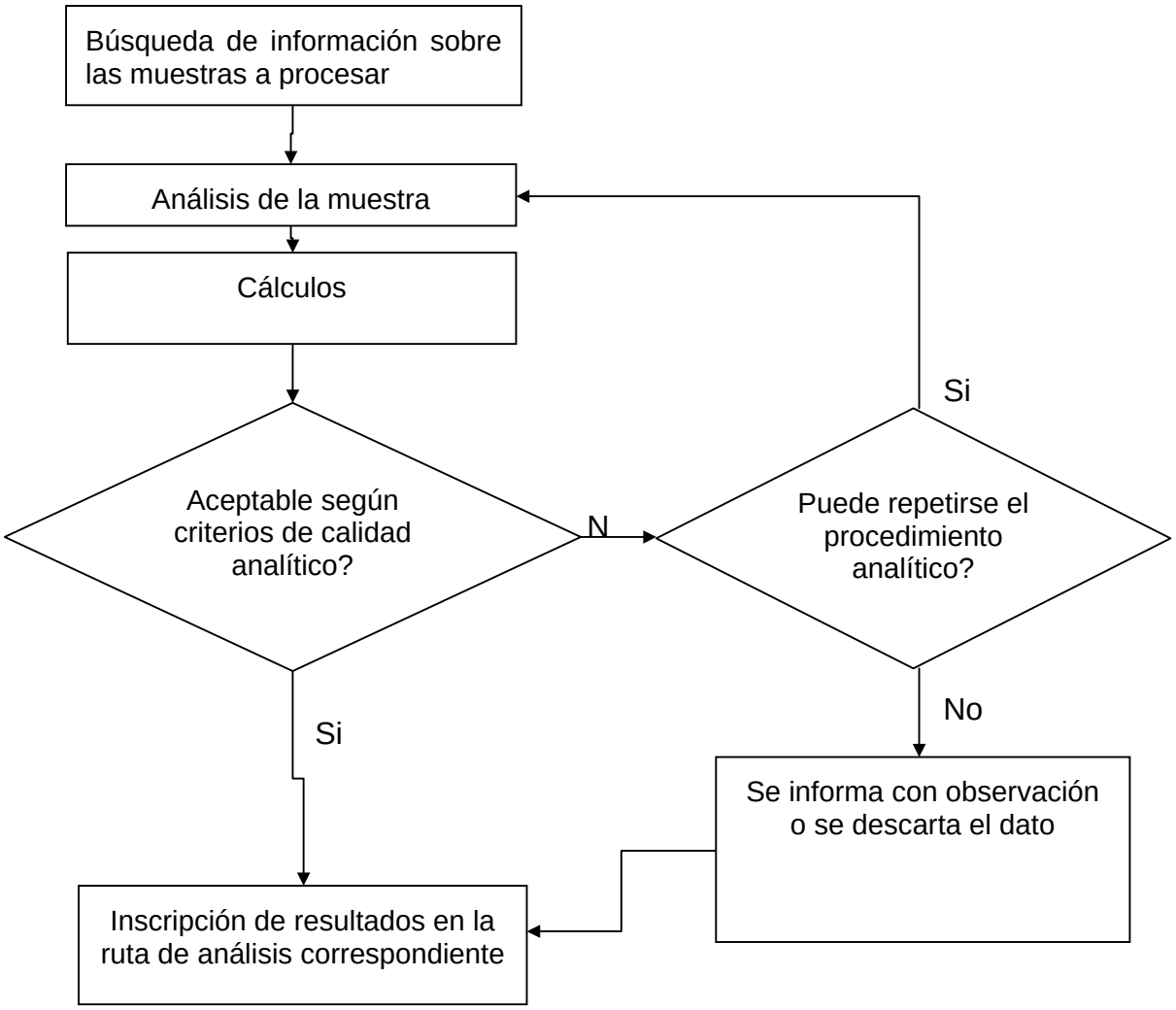


Diagrama de flujo emisión de resultados (3)

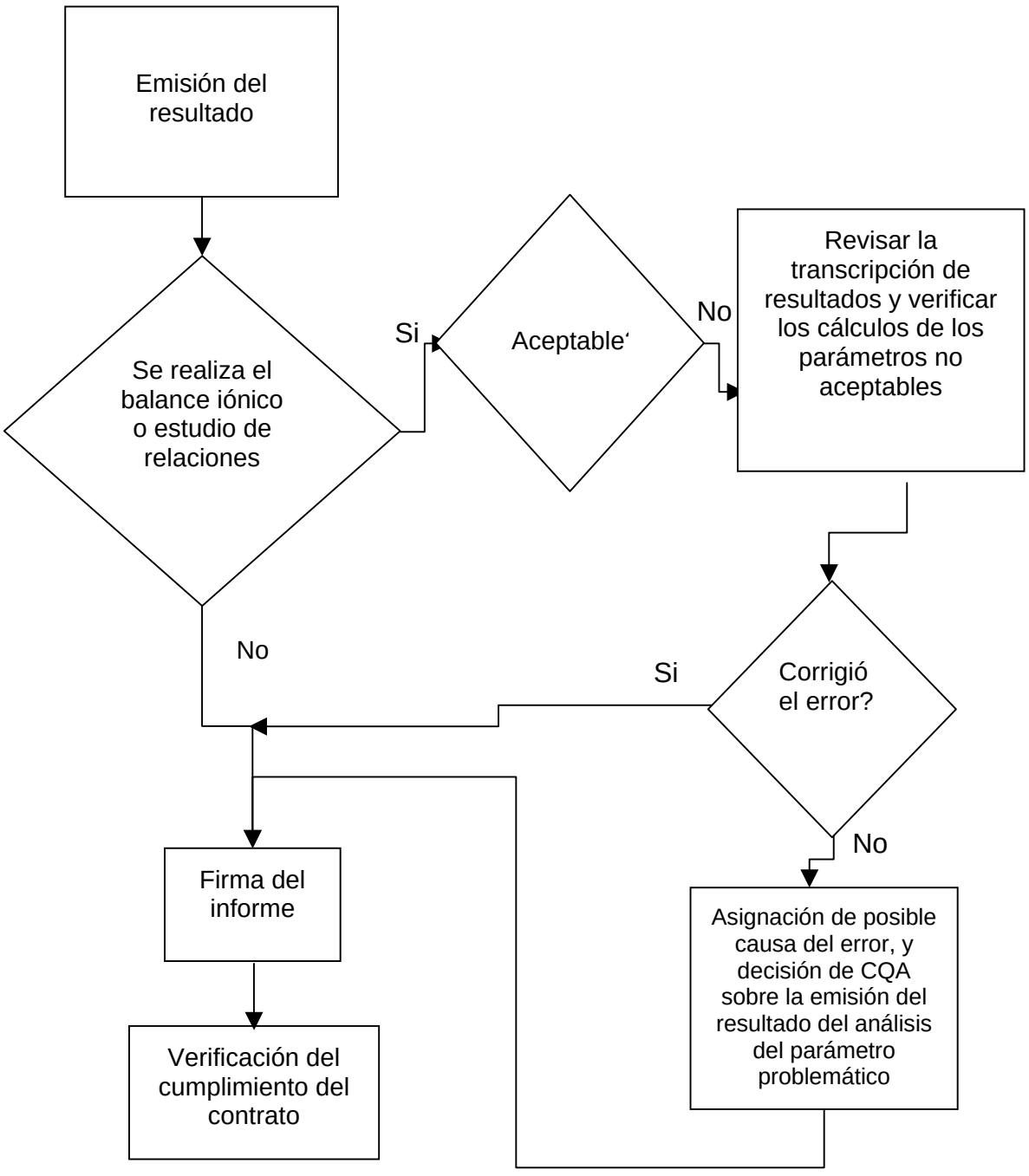


Diagrama de flujo entrega de resultados y atención pos entrega (4)

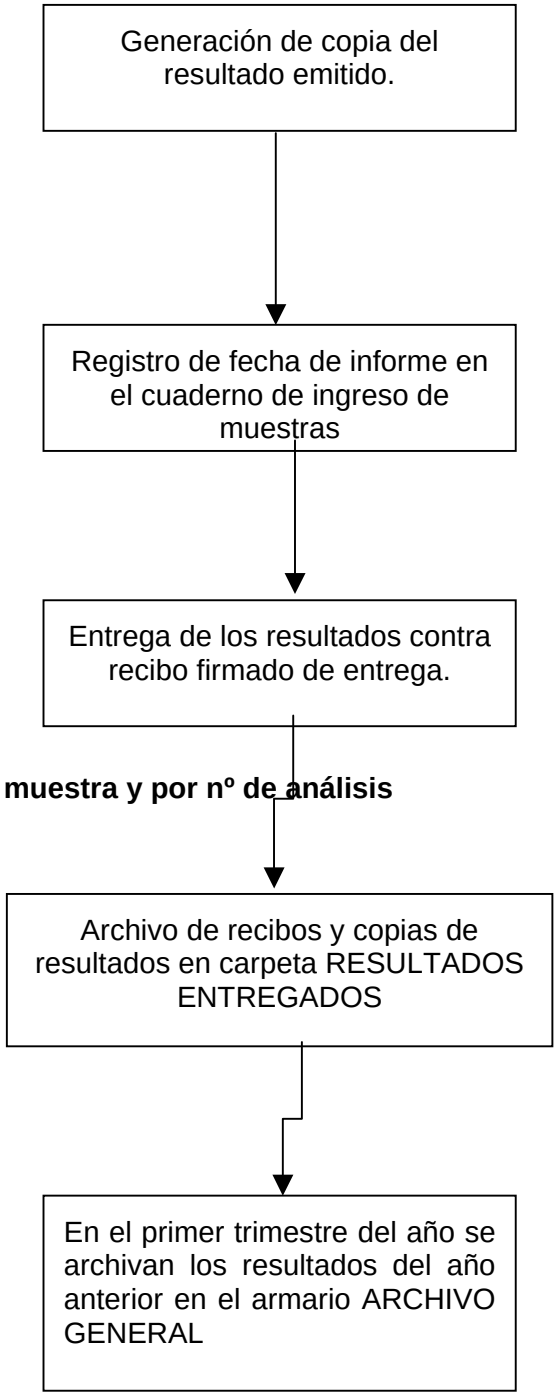


Diagrama de flujo de Reclamos de clientes

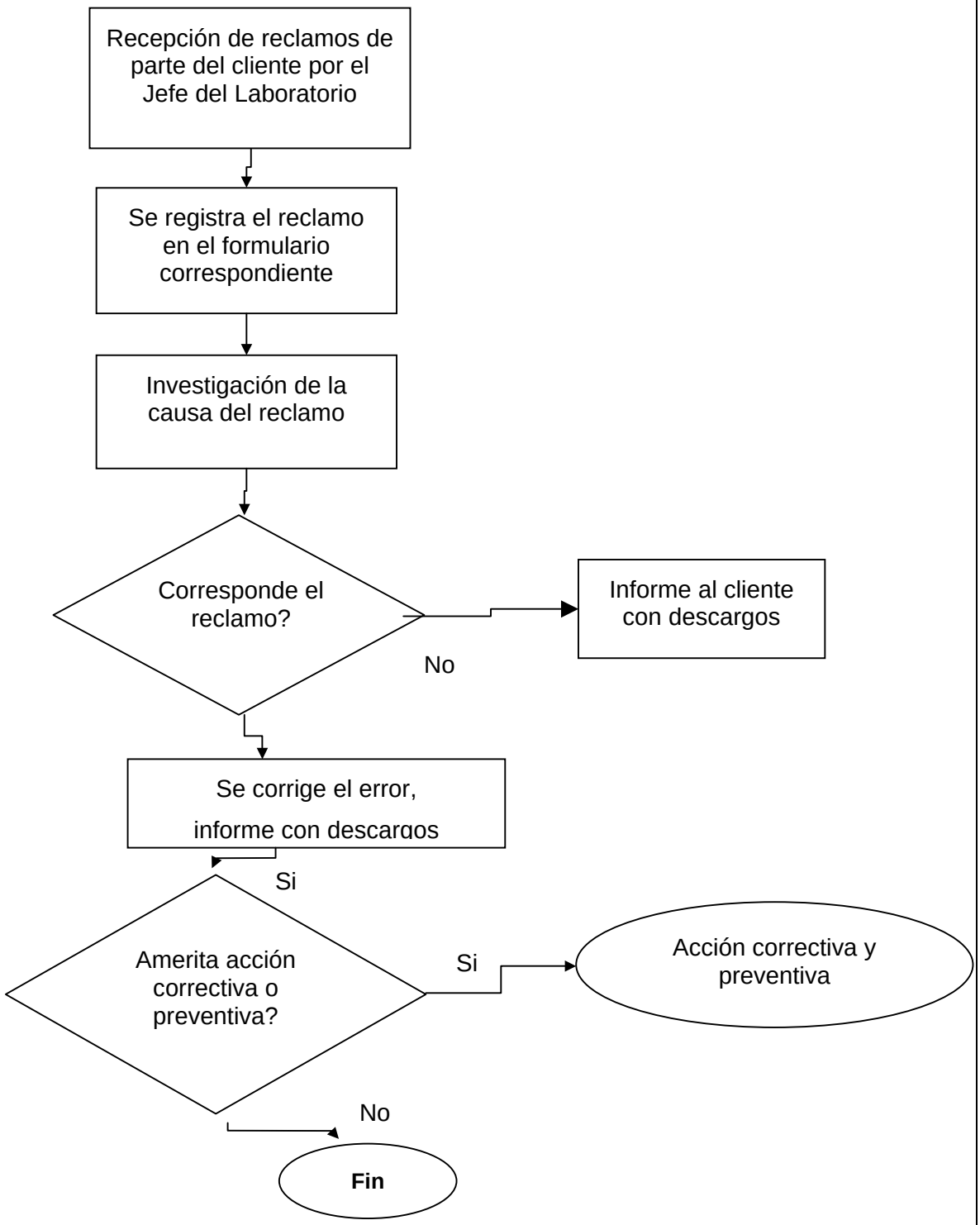
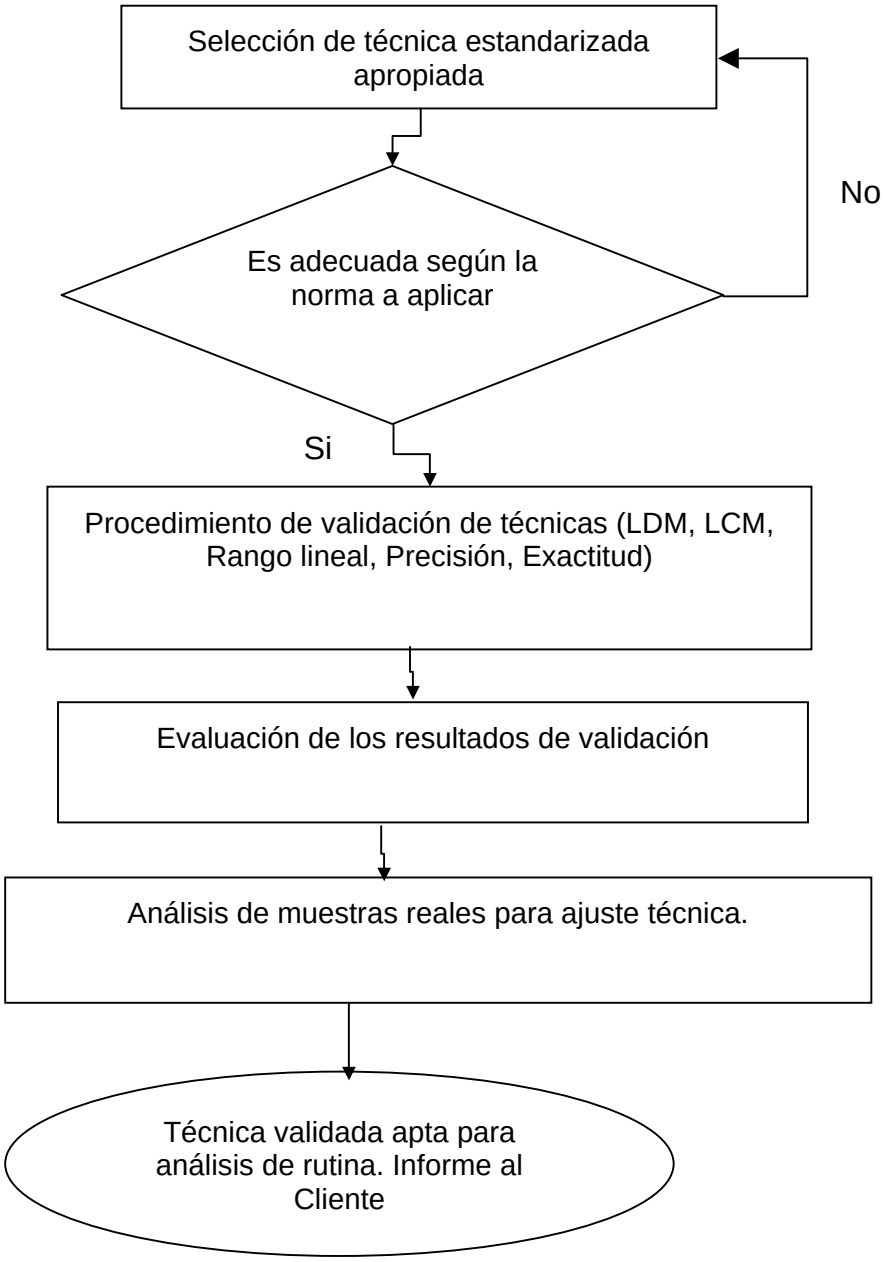


Diagrama de flujo de desarrollo analítico



ANEXO II: DESCRIPCIÓN DEL LABORATORIO

Laboratorio de Instrumental:

Donde se realizan las medidas en equipos instrumentales:

Área de Preparación de muestras para análisis de Metales:

Donde se realiza el tratamiento de las muestras para determinación de metales y se acondiciona el material para ese fin.

Área de Preparación de Muestras para análisis de Aniones y Plaguicidas:

Donde se realiza el tratamiento de las muestras para determinación de aniones inorgánicos por metodología instrumenta y las muestras para determinación de contaminantes orgánicos.

Laboratorio de Toxicidad:

Donde se realizan los análisis de toxicidad por Microtox.

Laboratorio de Microbiología:

Donde se realizan los análisis microbiológicos.

Laboratorios de Fisicoquímico I :

Donde se realizan los análisis del sector Fisicoquímico .

Laboratorios de Fisicoquímico II :

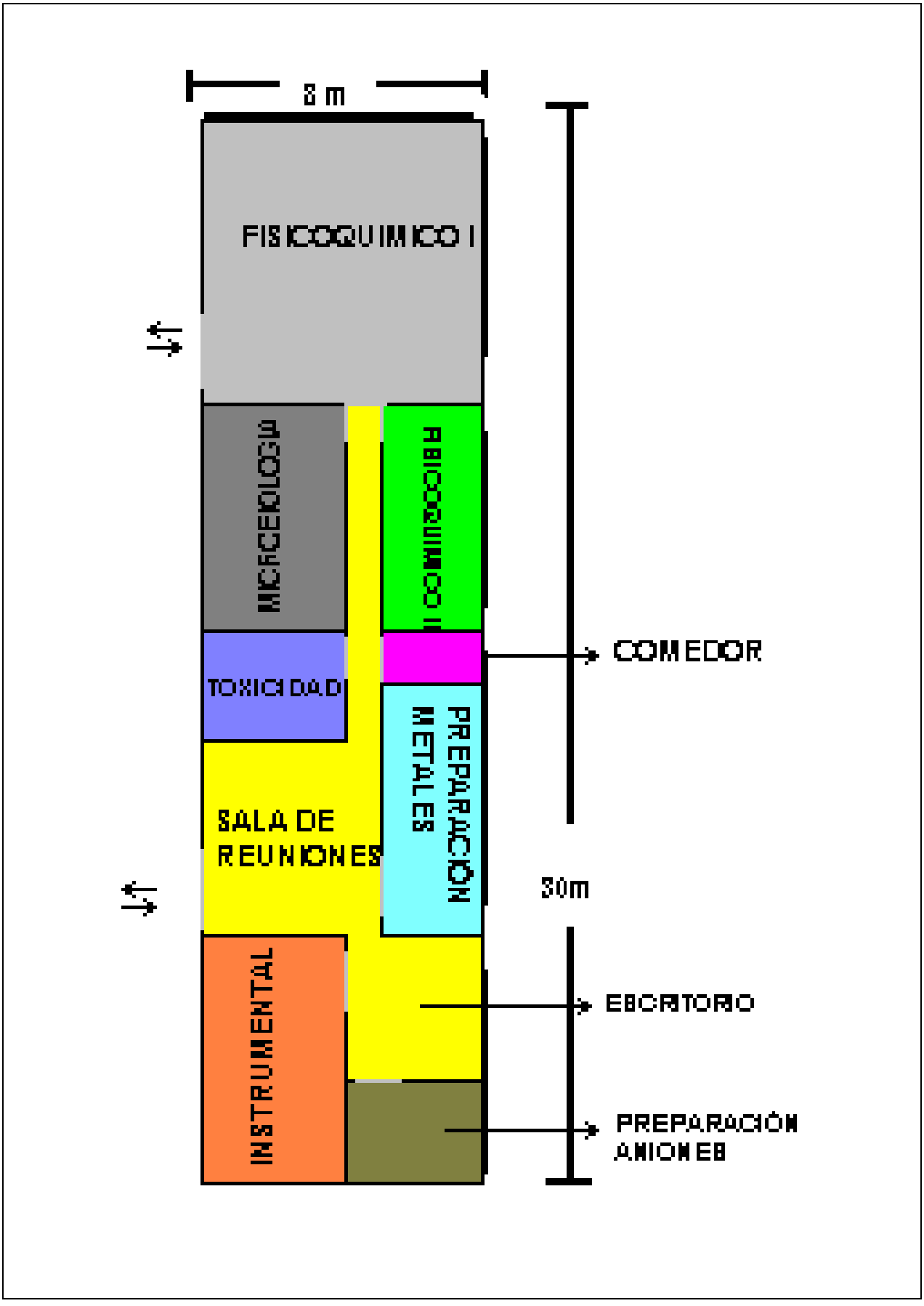
Donde se realizan los pesadas y secado de muestras del sector Fisicoquímico .

Escritorios

Sala de reuniones

Comedor

En la siguiente página se muestra el plano de corte de la planta física del Laboratorio.


8 m
FISICOQUIMICO I
MICROBIOLOGIA
BIOQUIMICO II
TOXICIDAD
PREPARACION METALES
SALA DE REUNIONES
INSTRUMENTAL
COMEDOR
30m
LABORATORIO
PREPARACIÓN ANIONES

ANEXO III: ENUMERACION DE ENSAYOS Y METODOLOGIAS DE ANÁLISIS.

Se indican a continuación los análisis que se realizan y en que tipo de muestras así como la metodología aplicada.

Código de tipo de muestra:
- ASUP : agua superficial
- ASUB : agua subterránea
- ALLUV : agua de lluvia
- EI : efluente líquido industrial
- RSI : residuo sólido industrial (contenido total y en el lixiviado)
- SUE : suelos
- SED : sedimentos
- AIR : aire y material particulado en aire
- LUB : aceites lubricantes
- ARE : arena

Parámetros	Tipo de muestras	Métodos de determinación
Sector Fisicoquímico		
pH	ASUP - ASUB - ALLUV - EI	Método electrométrico
Conductividad	ASUP - ASUB - ALLUV - EI	Método conductimétrico
Dureza	ASUP - ASUB	Volumetrico con EDTA
Turbidez	ASUP - ASUB	Nefelométrico
Color	ASUP - ASUB	Comparación visual
Sólidos totales, fijos y volátiles	ASUP - ASUB - EI	Gravimétrico
Sólidos Suspendidos totales, fijos y volátiles	ASUP - ASUB - EI	Gravimétrico
Sólidos sedimentables	EI	Gravimétrico
Alcalinidad	ASUP - ASUB - ALLUV	Potenciométrico
Silicato	ASUP - ASUB	Colorimétrico
Nitrito	ASUP - ASUB - ALLUV	Colorimétrico
Fósforo total	ASUP - ASUB - EI	Digestíon ácida y determinación colorimétrica con ác. ascórbico
Ortofosfato	ASUP - ASUB - ALLUV	Determinación colorimétrica con ác. Ascórbico / HPLC Cromatografía Iónica
Aceites y Grasas	ASUP - EI	Extracción Soxhlet con hexano
Demanda Bioquímica de Oxígeno	ASUP - EI	Técnica de dilución, medida electrométrica.
Demanda Química de Oxígeno	ASUP - EI	Colorimétrico, reflujo cerrado

Clorofila	ASUP	Extracción con acetona y medida espectrofotométrico
Detergentes, MBAS	EI	Colorimétrico
Material Particulado (PTS y PM10)	AIR	Gravimétrico
Indice de Corrosividad	AIR	Gravimétrico
Sector Microbiología		
Coliformes totales y termotolerantes	ASUP - ASUB - EI	Técnica de filtración por membrana o Sustrato Definido, Colilert
Estreptococos fecales	ASUP - ASUB - EI	Técnica de filtración por membrana
Heterótrofos	ASUP - EI	Técnica de filtración por membrana
Escherichia coli	ASUP - ASUB - EI	Sustrato Definido - Colilert.
Enterococos	ASUP - EI	Técnica de filtración por membrana
Coliformes totales	ARE	Recuento por inclusión
Coliformes termotolerantes	ARE	Recuento por inclusión
Estreptococos fecales	ARE	Recuento por inclusión
Sector Ecotoxicidad		
Toxicidad por Microtox	ASUP -EI -SUE -SED -RSI	Con *Vibrio fischeri*
Sector Instrumental		
Cloruro	ASUP - ASUB - ALLUV	HPLC/ Cromatografía iónica
Sulfato	ASUP - ASUB - ALLUV	HPLC / Cromatografía iónica
Cianuro (libre y total)	ASUP - ASUB – EI	Colorimétrico
Sulfuro	ASUP - EI	Electrométrico
Amonio	ASUP - ASUB - ALLUV - EI	Electrométrico
Nitrato	ASUP - ASUB - ALLUV - EI	HPLC/Cromatografía iónica y/o Electrométrico
Calcio	ASUP - ASUB - ALLUV	FLAAS (Espectrofotometría de absorción atómica por llama)
Magnesio	ASUP - ASUB - ALLUV	FLAAS
Potasio	ASUP - ASUB - ALLUV	FLAAS
Sodio	ASUP - ASUB - ALLUV	FLAAS
Aluminio	ASUP - EI - SUE - SED -RSI	FLAAS

Arsénico	ASUP - EI - SUE - SED -RSI	FLAAS ó ETAAS (Espectrofotometría de absorción atómica con atomización electrotérmica)
Cadmio	ASUP - EI - SUE - SED -RSI	FLAAS
Cinc	ASUP - EI - SUE - SED -RSI	FLAAS
Cobre	ASUP - EI - SUE - SED -RSI	FLAAS
Cromo total	ASUP - EI - SUE - SED -RSI	FLAAS
Cromo VI	SUE - SED -RSI	Colorimétrico
Hierro	ASUP – ASUB - RSI	FLAAS
Manganeso	ASUP – ASUB - RSI	FLAAS
Mercurio	ASUP - EI - SUE - SED -RSI	Espectrofotometría de absorción atómica por generación de vapor frío
Níquel	ASUP - EI - SUE - SED -RSI	FLAAS
Plomo	ASUP-EI-SUE-SED- RSI -AIR	FLAAS
Plata	RSI	FLAAS
Selenio	RSI	FLAAS ó ETAAS
Bario	RSI	FLAAS
Anhídrido sulfuroso	AIR	HPLC/Cromatografía Iónica
PCB´s	LUB	Screening colorimétrico
Plomo	SUE	Espectroscopía de rayos X
Acetato	ASUP-ASUB-ALLUV	HPLC/Cromatografía Iónica
Formiato	ASUP-ASUB-ALLUV	HPLC/Cromatografía Iónica
Fluoruro	ASUP-ASUB-ALLUV	HPLC/Cromatografía Iónica

Tipo de muestra	**Pretratamiento de la muestra para la determinación de metales según el tipo de matriz**
Suelo y sedimentos	Tamizado por 2 mm y digestión en medio ácido nítrico en caliente
Residuos sólidos	Lixiviación en medio ácido acético a pH 5, 18 hs.
Efluentes líquidos y aguas contaminadas	Digestión en medio ácido nítrico en caliente
Material particulado de aire	Digestión en medio ácido nítrico –clorhídrico en caliente

ANEXO IV: PERSONAL DEL LAORATORIO Y CARGO

Nombre y Profesión	Sector / Funciones
Sandra Castro Licenciada en Biología	*Jefe de Laboratorio*
	Sector Microbiología y Sector Ecotoxicidad Responsable de Análisis y Desarrollo
Patricia Simone Química Farmacéutica	*Responsable de Calidad*
	Sector Fisicoquímico Responsable de Análisis y Desarrollo
Mariana Menéndez Técnico Laboratorista	*Sector Fisicoquímico* Analista
Gabriela Medina Química Farmacéutica	*Sector Instrumental* Responsable de Análisis y Desarrollo
Natalia Barboza Química Farmacéutica	Analista
Alejandro Mangarelli Bachiller en Química	Analista
Gabriela Pistone Licenciada en Biología	Analista
Luis Borda	Preparador de material

Nota: los Responsables son además analistas en los sectores que desempeñan el cargo
Los técnicos habilitados a realizar los análisis de cada parámetro se indica en la carpeta de ***Recursos Humanos***

ANEXO V: Fe de erratas.

ELABORACION DE DOCUMENTOS

1. OBJETIVO

Establecer una metodología para asegurar que la elaboración de documentos se realiza en forma consistente.

2. ALCANCE

Este procedimiento es aplicable a la elaboración de los procedimientos generales del sistema de la calidad.

3. INVOLUCRADOS

Todo el personal del departamento

4. DESARROLLO

4.1 Manuales

El Departamento cuenta con los manuales que se detallan a continuación: Manual de Calidad, Manual de Gestión de Calidad, Manual de Control de Calidad Analítico.

El **Manual de Calidad** es elaborado según la norma UNIT – ISO 17025:2000, conteniendo asimismo los puntos requeridos por la norma UNIT – ISO 9001:2000. La aprobación la realiza el Jefe del Departamento. En el encabezado de todas las páginas figura el nombre del documento “Manual de Calidad” y en el pie de página consta el nombre del archivo del mismo, el número de versión y paginado. El número de versión, fecha de aprobación y firma del Jefe del Departamento se ubica en la última hoja del documento previo a los anexos. La última hoja corresponde a la Fe de Erratas

El **Manual del Sistema de Gestión de Calidad** se compone de una introducción, un índice y los procedimientos de gestión del departamento. Los procedimientos tienen como encabezado su código y nombre y como pie de página el nombre del archivo magnético, la versión, la fecha de la misma y el paginado. La constancia de aprobación del manual figura en la segunda hoja, al final de la introducción. En la misma aparece la fecha y firma de la persona que revisa (Responsable de Calidad) y fecha y firma del que aprueba (Jefe del Departamento). La última hoja del mismo corresponde a la Fe de Erratas.

El **Manual de Control de Calidad Analítico** contiene los procedimientos de control de análisis. Su encabezado indica el nombre del mismo, y en el pie de página se ubica la revisión, la fecha de la misma y el paginado. Este documento posee en su última hoja

el registro de revisión por el Responsable de Calidad y la fecha de la misma, así como también la fecha y firma de aprobación por parte del Jefe del Departamento.

Las modificaciones realizadas a estos tres Manuales pueden ser también aprobados por el Responsable de Calidad.

4.2 Carpeta de mantenimiento y control de equipos

Se elabora conjuntamente entre el Responsable de Calidad y el Encargado de Mantenimiento de los equipos, generando una ficha de registro de equipo y formatos para registro de control y de mantenimiento de los mismos. Los formatos para cada tipo de registro se encuentra en esta carpeta. Los mismos son aprobados por el Responsable de Calidad.

4.3 Documentos Generales del Sistema de Gestión de Calidad

Corresponde a toda otra documentación que no posee codificación de registro tales como: Planificación de Sistema de Gestión de Calidad, Acuerdo con clientes, Listas de verificación de auditorías, Perfiles de Cargo, Actas de reunión de Calidad, etc. La fecha de elaboración y/o revisión del documento queda registrado en el documento. Son elaborados y aprobados por el Responsable de Calidad y/o Jefe de Departamento. Este tipo de documentación no posee formato definido para su elaboración.

4.4 Formatos para registros

Para darle número a un nuevo registro se realiza en forma consecutiva al último existente en el Listado de documentación.

Tipo de Registro	**Codificación**	**Numeración**	**Aprobación**
Ruta de análisis	RXX (XX indica sector)	Correlativa	R. S. y/o J. D.
Control de calidad analítico	RQA	Correlativa	R. C. y/o J. D.
Administrativo	RAD	Correlativa	R. C. y/o J. D.
Gestión de Calidad	RGC	Correlativa	R. C. y/o J. D.
Ficha de registro de equipo	FRE	Correlativa	R. C. o a quien designe
Ficha de mantenimiento de equipo	RME	Correlativa	R. C. o a quien designe
Tipo de	**Codificación**	**Numeración**	**Aprobación**

Registro			
Ficha de control de equipo	RCE	Correlativa	R. C. o a quien designe
Ingreso de muestras	RIG	Correlativa	R. C. y/o J. D.
Preparación de reactivos y std.	RPR	Correlativa	R. C. y/o J. D.

R. S.: Responsable del Sector
R.C.: Responsable de Calidad
J.D.: Jefe del Departamento

En el pie de página de los formatos para registros se indica: Aprobado, nombre, firma del responsable y fecha.

4.5 Resultados emitidos

Son aprobados por el Jefe del departamento, codificado por el número de análisis, conteniendo fecha de emisión. Debe contener la siguiente información:

- la fecha del informe
- referencia del origen de la muestra: de la industria o de la localización del punto de muestreo según corresponda.
- punto de muestreo
- fecha de muestreo (si difiere en más de un día con la fecha de ingreso al laboratorio también se incluye esta fecha)
- responsable del muestreo
- solicitante: que corresponde al cliente
- análisis solicitado con los resultados
- observaciones en caso de ser necesario,
- analistas responsables de los análisis solicitados
- firma autorizada

4.6 Procedimientos

Se utilizará una hoja tamaño A4

Procedimientos de gestión: en la ultima página mediante una tabla, se indicará quien lo revisó, quien lo aprobó y las respectivas fechas.

Procedimientos normalizados de operación (SOP): en la carátula del documento figura una tabla donde se incluyen los nombres y las firmas de los responsables de la elaboración, revisión y aprobación así como las fechas respectivas.

Procedimientos de Control de Calidad Analítico: se aprueba el manual en su totalidad (ver Manual de Control de Calidad Analítico).

Todos poseerán como pie de página a la izquierda nombre del archivo magnético, número de revisión y fecha; y a la derecha el paginado.

El encabezado de página consistirá del código y nombre del documento en cuestión, como mínimo en la primera hoja del documento.

4.6.1 Codificación

Los procedimientos de gestión se codifican según: PR xx

Los procedimientos normalizados de operación se codifican según: SOP xx.

Los procedimientos Generales de Control de Calidad se codifican según: PGC xx.

4.6.2 Estructura

Procedimientos de Gestión (PR)

1. Objetivo: Establece el propósito por el cual existe el procedimiento
2. Alcance: Establece el tipo de actividad y los lugares en los cuales se aplica el procedimiento.
3. Involucrados: Establece aquellos cargos que están involucrados en las acciones descritas en el procedimiento.
4. Desarrollo: Establece las acciones que deben ser ejecutadas para cumplir con el procedimiento.
5. Referencias: Menciona aquellos documentos que son necesarios para llevar a cabo el procedimiento.
6. Registro de revisiones: establece mediante una tabla las modificaciones que se le han efectuado al procedimiento desde la edición anterior.

Procedimientos Normalizados de Operación (SOP)

La primera página especifica el título, y la firma de quien lo elabora, revisa y aprueba.

1. Aplicación: Indica para que se utiliza la normativa técnica.
2. Referencias
3. Resumen del Método
4. Precauciones de seguridad
5. Interferencias
6. Muestreo y preservación de la muestra.
7. Instrumental y materiales.
8. Reactivos
9. Precauciones para la operación
10. Calibración del Método.
11. Análisis de la Muestra.

12. Análisis de datos.
13. Control de Calidad Analítico
14. Bibliografía

Procedimientos Generales de Control de Calidad (PGC)

1. Objetivo
2. Definiciones (opcional)
3. Alcance
4. Referencia (opcional)
5. Resumen (opcional)
6. Procedimiento
7. Cálculos (opcional)
8. Bibliografía

4.7 Instructivos

Poseen como título el nombre y la codificación. En el pie de página se lee la revisión y la fecha de la misma.

Son aprobados por el Responsable del Sector mediante firma y fecha al pie de la página, habilitando la colocación de una etiqueta con este fin.

Los instructivos se codifican: INC/E/A/G xx, siendo:

C: Calidad
E: Equipos
A: Administrativo
G: General
xx es un número de identificación correlativo.

5. REFERENCIAS

PR 02 Control de Documentos

RGC 02 Listado de Documentos Vigentes

Norma UNIT-ISO 17025

Norma UNIT-ISO 9001

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Marzo 2002	Primera emisión
1	Mayo 2003	Segunda emisión
2	Junio 2003	Tercera emisión
3	Agosto 2005	Cuarta impresión

Revisado	Fecha
Aprobado	Fecha

CONTROL DE DOCUMENTOS DEL SISTEMA DE LA CALIDAD

1. OBJETIVO

Establecer el procedimiento para la identificación, vigencia y distribución de documentos del Sistema de la Calidad.

2. ALCANCE

Este procedimiento es aplicable a toda la documentación del Sistema de Gestión de Calidad la que se encuentra indicada en el Listado de Documentos Vigentes ubicado en el Bibliorato de Documentos del Sistema de Gestión de Calidad.

3. INVOLUCRADOS

Todos los integrantes del Departamento.

4. DESARROLLO

4.1 A partir de la generación de un documento nuevo, el Responsable de Calidad revisa el formato y la presentación, asignando:

- Tipo y código del documento (correlativo al número anterior asignado al documento del mismo tipo)
- Revisión
- Fecha de revisión

4.3 El Jefe del Laboratorio aprueba el documento mediante la firma y fecha de la aprobación.

4.4 El Responsable de Calidad ingresará al Listado de Documentos Vigentes, que está ubicado en el "Bibliorato de Documentos de Sistema de Gestión de Calidad" indicando su fecha de aprobación y estado de revisión.

4.5 El Responsable de Calidad distribuye copias controladas. Cada Responsable de Sector que recibe copias controladas de documentos firma el Registro de Control de Documentos (RGC 01) en el espacio correspondiente a tal fin.

4.6 Cada copia controlada llevará la firma de los involucrados en la realización del documento, según consta en el Procedimiento de Elaboración de Documentos.

4.7 El Responsable de Calidad retirará de circulación el documento obsoleto, destruyendo las copias de los mismos y dará al mismo de baja en el Registro de Control de Documentos (RGC 01). Si el documento no es sustituído por otro con versión posterior, el número de código del documento en cuestión no será utilizado para ningún otro documento, quedará anulado.

4.8 Una sola de las versiones (la original) es archivada en el archivador "Documentos obsoletos" por el Responsable de Calidad como antecedente indicándola con la inscripción "documento obsoleto" o “documento fuera de uso”, adicionándole fecha y firma. El resto serán destruidas y recolectadas por el servicio de recolección de residuos domiciliarios. En esta versión es donde se mantiene la información que luego fue modificada en versión posterior.

4.9 Cuando un área necesite copias adicionales controladas de un documento, deberá solicitarlas al Responsable de Calidad.

4.10 Cuando se requieran copias no controladas de un documento, el sector que las requiera deberá solicitarlas al Responsable de Calidad, quien las emitirá sin registro, y con la inscripción manuscrita de "Copia no controlada".

4.11 El Responsable de Calidad asegurará que los documentos en sus versiones actuales se encuentren disponibles en las bibliotecas correspondientes a cada sector, y que los mismos sean legibles.

4.12 Los documentos son revisados a los efectos de asegurar continuamente su conformidad y cumplimiento con los requisitos aplicables. Los responsables de la revisión y posterior aprobación de los mismos son los que se definen en el Procedimiento PR 01 Elaboración de Documentos, de acuerdo al tipo de documento de que se trate, o en su defecto por la misma función que realizó la revisión original.

4.13 Los instructivos de uso de equipos que se encuentran en cada equipo o en la carpeta que contiene el Procedimiento Normalizado de Operación (SOP) para el cual se utiliza el mismo, serán revisados y aprobados por el Responsable del Sector que lo emplea . La aprobación se registra mediante la fecha y firma del Responsable del Sector anteriormente mencionado, en el Instructivo mismo o adicionando una etiqueta sobre el mismo con el citado registro. Esta revisión se realiza cuando el equipo sufre alguna modificación.

4.14 Para llevar a cabo el control de la documentación externa, la misma es dada de alta e indexada en el listado ubicado en la carpeta denominada “Inventario de Libros, Artículos, CD y otros”. La misma se encuentra en la biblioteca del Departamento. El responsable de mantener vigente el listado se encuentra indicado en la carátula de la carpeta.

5. REFERENCIAS

RGC 01: Control de Documentos
RGC 02: Listado de documentos vigentes
PR 01: Procedimiento de Elaboración de Documentos
Inventario de Libros, Artículos, CD y otros.

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	13/03/02	Primera emisión.
1	29/05/03	Segunda emisión
2	25/06/03	Tercera emisión
3	Agosto 2005	Cuarta impresión

Revisado	Fecha
Aprobado	Fecha

COMPRAS

1. **OBJETIVO**

El objetivo de este procedimiento es establecer la metodología para realizar las compras.

2. **ALCANCE**

Este procedimiento se aplica a las compras directas y licitaciones a ejecutar por el Departamento de Normalización Técnica – Laboratorio de DI.NA.MA.

Compras directas se pueden realizar cuando el monto total es inferior al monto indicado por la Contaduría General de la Nación y montos superiores a este solamente para aquellos artículos únicos que tengan un proveedor con la representación exclusiva en el país. Estas compras directas deben justificarse presentando un certificado de representación exclusiva del proveedor.

3. **INVOLUCRADOS**

Todas aquellas personas pertenecientes al departamento y el responsable de Gestión y seguimiento de las compras.

4. **DESARROLLO**

4.1 Se realiza una nota de la solicitud indicando el/los productos de interés con sus especificaciones, las que se indican en el Procedimiento Normalizado de Operación (SOP) para el cual se utilizará el insumo (las especificaciones de equipos también se indican en el Inventario de Equipos). Para el caso de licitaciones se pasa al punto 4.6.

4.2 Para las compras directas el técnico interesado, responsable de la compra, debe realizar la solicitud de cotización por lo menos a tres proveedores, tomando en cuenta la lista de proveedores y el registro de la evaluación de proveedores que se encuentran en el bibliorato de Compras Pendientes. Excepto para materiales específicos que existen un solo proveedor con la representación exclusiva en el país y para compras urgentes que salen por caja chica de DINAMA.

4.3 La solicitud de cotización se envía por fax. Se debe imprimir la confirmación del fax.

4.5 Luego cuando se reciben las cotizaciones de las distintas empresas se deben adjuntar junto con las confirmaciones de fax de aquellas que no cotizaron y notas de pedido de cotización, en la carpeta de compras pendientes en la sección **Cotizaciones** hasta que salga la solicitud del Laboratorio.

4.6 Iniciar la solicitud de compras o licitación a través de una nota al Jefe del Laboratorio justificando el pedido, para el caso de compras directas indicando la empresa

recomendada, el material con su especificación así como el precio. Una vez aprobada la solicitud por el jefe, el responsable de Gestión y seguimiento de las compras (quien se indica en la tapa del carpetín de seguimiento de expedientes) realiza el trámite correspondiente indicado por la División Administración de DINAMA. El Jefe del laboratorio puede iniciar directamente una compra.

4.7 Se deja copia de la solicitud de compra y de las cotizaciones del/ o los proveedores en la carpeta "Compras pendientes" en la sección del proveedor adjudicado , en caso de no contar con sección para el proveedor, en la sección “Otros” Para licitaciones se guarda copia de la solicitud en la sección licitaciones.

Cuando en una compra se adjudican múltiples proveedores, la copia del expediente competo queda archivada en el lugar correspondiente al primer proveedor adjudicado que figura en el mismo; dejando una copia de la primera hoja del expediente en el lugar correspondiente a cada uno de los demás proveedores adjudicados.

Se archivan las cotizaciones no incluidas en el expediente y registro de especificaciones en la carpeta creada a tales efectos llamada “Cotizaciones y especificaciones”

4.8 Se inicia el registro en "RAD 01 Seguimiento de expedientes" ubicado en la misma carpeta.

4.9 Una vez recibido el material solicitado, la solicitud de compra con la copia de la factura, se coloca en el bibliorato "Compras Entregadas" donde se encuentra la documentación perteneciente a las compras del año anterior y las del año en curso. Luego se guardan en el archivo general.

4.10 Recepción de material

4.10.1 El funcionario que recibe el pedido en el Laboratorio debe corroborar el material entregado con la factura, conformar la factura y sacar copia de esta y entregarla al responsable de la compra o licitación. En caso de desconocerse el responsable se indica en el pizarrón la recepción del material para que el responsable continúe con el trámite. La factura original se entrega al proveedor, quien es el responsable de hacerlo llegar a la sección correspondiente para que se concrete el pago.

4.10.2 El responsable de la compra o licitación adjunta la copia de la factura a la copia de la solicitud de la compra. Si la entrega del pedido es parcial debe hacer el seguimiento y recuento de lo que se va entregando. Cuando la entrega se completa, debe archivar la copia de la solicitud en el bibliorato "Compras entregadas ". En el caso de que el producto sea no conforme con lo solicitado, el personal del Laboratorio que solicitó la compra debe comunicarse con el proveedor a los efectos de que el mismo cambie el producto de acuerdo a lo solicitado.

5. REFERENCIAS

RAD 01 Seguimiento de expedientes, Bibliorato "Compras Pendientes", Bibliorato de "Compras entregadas ", PR 04 Evaluación y calificación de proveedores

6. **REGISTRO DE REVISIONES**

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión
1	Mayo 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

EVALUACION Y CALIFICACION DE PROVEEDORES

1. OBJETIVO

Establecer la metodología para la evaluación y calificación de Proveedores.

2. ALCANCE

Este procedimiento se aplica a proveedores de los reactivos, materiales, equipos y a las empresas que realizan el mantenimiento de equipos.

3. INVOLUCRADOS

Responsables de Sectores y Responsable de Calidad.

4. DESARROLLO

4.1 El responsable de realizar la evaluación de proveedores y de mantener al día la lista de proveedores autorizados es el Responsable de Calidad, o quien él designe. El encargado de calidad consulta a los responsables de sectores cuando se le presente dudas. Para realizar la evaluación se chequea las copias de las solicitudes de compras realizadas donde figura la respuesta de cotización, los tiempos de entrega y los productos entregados.

4.2 La evaluación se realiza anualmente y los criterios de evaluación se analizan en esa oportunidad.

4.3 Los proveedores autorizados a los que se pueden solicitar cotización son los que superan el 50% del puntaje máximo.

Aquellos proveedores que no alcancen el 50% pero que sean únicos en plaza ó que por motivos estratégicos sea conveniente incorporarlos en alguna compra serán tratados como proveedores especiales, siendo el responsable de la compra el encargado del seguimiento personalizado de la misma.

4.4 Los proveedores se clasifican en dos categorías:

Proveedores habituales: los que ya han suministrado insumos a DI.NA.MA

Proveedores potenciales: los que no han suministrado ningún tipo de materia prima. Estos no se evalúan hasta que se ejecute una compra.

Los proveedores habituales se encuentran listados en la lista de proveedores, donde se indica: nombre comercial o de referencia, teléfono, fax, contacto y productos

ofrecidos (RGC 03). Esta lista está ubicada en la carpeta "Compras pendientes". Este listado se actualiza anualmente cuando se realiza la evaluación de proveedores.

4.5 Criterios de evaluación para proveedores habituales:

Se evalúa según los siguientes 4 criterios

- Respuesta a la solicitud de cotización
- Atención al cliente
- Calidad del producto (correspondencia de lo entregado con lo cotizado) o servicio de mantenimiento según corresponda.
- Tiempo de entrega del producto o de realización del servicio de mantenimiento

La descripción de los criterios y los puntajes asignados a cada criterio se indican en el registro según corresponda:

- RGC 04 Evaluación de proveedores, para proveedores de insumos, ó
- RGC14 Evaluación de Proveedores de Servicios de Mantenimientos de Equipos

4.8 Los registros de la evaluación se archivan en el Bibliorato de Compras Pendientes.

5. REFERENCIAS

RGC 03 Registro de Lista de Proveedores

RGC 04 Evaluación de proveedores para proveedores de insumos

RGC 14 Evaluación de Proveedores de Servicios de Mantenimientos de Equipos

PR 03 Compras

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión.
1	Mayo 2003	Segunda emisión.
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

CONTROL DE REGISTROS

1. OBJETIVO

Establecer la metodología para el control de los registros del sistema de calidad.

2. ALCANCE

Este procedimiento es aplicable para la identificación, recolección, indexación, acceso, archivo, almacenamiento, mantenimiento y disposición de los registros técnicos.

3. INVOLUCRADOS

Todos los integrantes del Laboratorio.

4. DESARROLLO

4.1 En la carpeta denominada "Planillas de Registros", se encuentran identificados todos los formatos para registros del Sistema de Gestión de Calidad. La misma posee un índice y la persona encargada del su actualización es el Responsable de Calidad. Los formatos poseen:

- Código
- Nombre
- Ubicación

4.2 En el anexo 1 se listarán los tipos de formatos para registros existentes en el laboratorio, dónde se encuentran, cómo y durante cuánto tiempo se archivan, y cómo se destruyen. La confidencialidad es responsabilidad de todos los integrantes de nuestro laboratorio. El responsable de este proceso es el Responsable de Calidad.

4.3 Cada Responsable de Sector cuidará que los registros sean completados en forma legible por los que tienen responsabilidad de registrar datos en los mismos.

4.4 Los formatos originales en uso se encuentran en la carpeta Planillas de Registros. Cuando un formato particular necesite ser modificado, el mismo es sustituido en la carpeta por el Responsable de Calidad por la nueva versión. Si el mismo debe ser anulado, quien lo da de baja en el Listado de Documentos Vigentes es el Responsable de Calidad.

4.5 Registros: Cuando ocurre un error en los registros, se tacha el error con lapicera, sin borrarlo, ni eliminarlo, de forma tal que permanezca legible, y el valor correcto es colocado al lado, junto con las iniciales del responsable. La numeración de las hojas de registro dentro de un mismo tipo de formato es correlativo.

4.6 Codificación de los registros: El mismo se establece en el Procedimiento PR 01 Elaboración de Documentos. La numeración se establece en orden correlativo a su anterior dentro del tipo de formato para registro correspondiente.

5. REFERENCIAS

Planillas de Registros
Listado de Documentos Vigentes
PR 01 Elaboración de Documentos

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión.
1	Mayo 2003	Segunda emisión
2	Junio 2003	Tercera emisión
3	Agosto 2005	Cuarta impresión

Revisado	Fecha
Aprobado	Fecha

ANEXO 1

Ref.	Tipo	Ubicación	Archivo	Tiempo mínimo	Destrucción
RRE	Registro de resultados	Bibliorato de resultados entregados	En Armario de Archivo	10 años	Recolección de residuos
RFQ	Registro Ruta de Análisis del Sector Fisicoquímico	Carpetas de análisis de cada parámetro y luego en Bibliorato de Planillas de Análisis	Armario de archivo, con rótulo del año correspondiente	3 años	Recolección de residuos
RIN	Registro Ruta de Análisis del Sector Instrumental	Carpetas de rutas de análisis y luego Bibliorato de Instrumental, Año 2003	Armario de archivo, con rótulo del año correspondiente	3 años	Recolección de residuos
RMB	Registro Ruta de Análisis del Sector Microbiología	Bibliorato de Micro. Año 2003.	Armario de archivo, con rótulo del año correspondiente	3 años	Recolección de residuos
RQA	Registro de Control de Calidad Analitico	Carpeta: Control de Calidad Analítico	En la carpeta de Antecedentes de Calidad	3 años	Recolección de residuos
RIG	Registro Ingreso de Muestras	Bibliorato Fichas de Ingreso de Muestras	Armario de archivo, con rótulo de año correspondiente	3 años	Recolección de residuos
RET	Registro Ruta de Análisis del Sector Ecotoxicidad	Carpetas: Toxicidad ficha aguas, Toxicidad ficha industrias, Resultados residuos sólidos.	Armario de archivo, con rótulo del año correspondiente	3 años	Recolección de residuos

Ref.	Tipo	Ubicación	Archivo	Tiempo mínimo	Destrucción
RAD	Registros Administrativos	Ver procedimiento correspondiente	En armario de archivos	3 años	Recolección de residuos
RCE	Registro de control de Equipos	Junto al equipo o en Bibliorato de Mantenimiento y Control de Equipos	Armario de archivos	3 años	Recolección de residuos
RME	Registro de Mantenimiento de Equipos	Bibliorato de Mantenimiento y Control de Equipos	Armario de archivo	10 años	Recolección de residuos
FRE	Ficha de registro de Equipos	Bibliorato de Mantenimiento y Control de Equipos	Bibliorato de Mantenimiento y Control de Equipos	Permanente	Recolección de residuos
RPR	Registro de Preparación de reactivos y estándares	Carpetas de análisis de cada parámetro	Se archiva junto con las rutas de análisis	3 años	Recolección de residuos
RGC	Registro de Calidad	Ver procedimiento correspondiente	Armario de archivo	3 años	Recolección de residuos

CONTROL DE NO CONFORMIDADES

1. OBJETIVO

Establecer la metodología para el control y registro de las No Conformidades de forma de posibilitar su estudio y evitar su reiteración.

2. ALCANCE

Este procedimiento es aplicable a todas las No Conformidades detectadas en el departamento de Normalización Técnica – Laboratorio de DI.NA.MA.

3. INVOLUCRADOS

Todos los integrantes del Departamento.

4. DESARROLLO

4.1 Todo integrante del Laboratorio que detecte una No Conformidad deberá registrarla en el registro de No confomidad e Implementación de Acciones Correctivas /Preventivas (RGC06) en el cuaderno rotulado “Reclamos y No Conformidades”, completando los puntos indicados. Quien detecte la no conformidad, si puede, define las acciones a tomar para corregirla.

4.2 Notifica al responsable de Calidad de la no conformidad y/o de la acción tomada. En caso de quién la detectó no pudo corregir la no conformidad el responsable de Calidad o el Jefe del Laboratorio, determinarán las acciones a tomar para corregirla. Se definirá además la necesidad de implementar una acción correctiva o preventiva, según el procedimiento PR 07.

4.3 El Responsable de Calidad y/o el Jefe firmará la culminación cuando haya sido completado todo el proceso y se hayan efectuado las acciones a tomar.

4.4 El Responsable de Calidad informará a aquellas personas que estén directamente relacionadas con la No Conformidad y a la persona que la detectó.

5. REFERENCIAS

RGC06: Registro No Conformidad e Implementación de Acciones Correctivas /Preventivas
PR 07: Generación e implementación de Acciones Correctivas y Preventivas.

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión.
1	Mayo 2003	Segunda emisión.
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

GENERACION E IMPLEMENTACION DE ACCIONES CORRECTIVAS Y PREVENTIVAS

1. OBJETIVO

Establecer el procedimiento para asegurar que las desviaciones, reales o potenciales, de la política y de los procedimientos del sistema de la calidad o de las operaciones técnicas, sean analizadas para identificar su causa y que se apliquen las acciones correctivas o preventivas apropiadas para evitar su recurrencia u ocurrencia.

2. ALCANCE

Este procedimiento es aplicable a todas las actividades del Sistema de Gestión de Calidad del Departamento.

3. INVOLUCRADOS

Jefe del Laboratorio, Responsable de Calidad y Responsables de Sectores.

4. DESARROLLO

4.1 El Jefe del Laboratorio o el Responsable de Calidad o de Sector podrá identificar una necesidad de generación e implementación de una acción correctiva o preventiva en base al análisis de cualquier documentación, información o registros referentes a calidad donde se pueden identificar desvíos de lo establecido. Se registra en el formulario de No conformidad e Implementación de Acciones Correctivas/Preventivas RGC 06, los cuales se encuentran en el **Cuaderno de No Conformidades y Reclamos**. En caso de que la identificación de la generación no sea realizada por el Responsable de Calidad, se debe notificar al mismo.

4.2 El Responsable de Calidad coordina con el o los Responsables de Sector y/o el Jefe las acciones correctivas o preventivas a tomar, su plazo de implementación y una fecha para verificar la eficacia de la acción implementada.

4.3 El Responsable del Sector efectiviza que se implemente la acción correctiva o preventiva.

4.4 El Jefe del Laboratorio y/o el Responsable de Calidad verifican la implementación de la acción correctiva o preventiva propuesta (eficacia y plazo de ejecución de la misma), dejando constancia de ello en el registro.

4.5 El responsable de Calidad cada tres meses, revisa el **Registro de No Conformidades e Implementación de Acciones Correctivas y Preventivas RGC 06**, para realizar la verificación de la eficacia en aquellas acciones que correspondan, quedando a criterio del Responsable de Calidad, la verificación en ese momento o en una revisión posterior de acuerdo al tipo de acción tomada y el plazo estipulado para la misma. El registro de tal verificación, lo realiza el Responsable de Calidad en el

mismo registro de la acción en cuestión. En el caso de que la acción tomada no sea eficaz de acuerdo a lo verificado por el mismo, esto genera una No Conformidad, lo cual amerita actuar de acuerdo al **PR 06 Control de No Conformidades.**

5. REFERENCIAS

RGC06 Registro No Conformidad e Implementación de Acciones Correctivas /Preventivas

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión.
1	Junio 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

REVISION POR LA DIRECCION

1. OBJETIVO

Establecer la metodología para la realización de revisiones del sistema de gestión de calidad por la Dirección.

2. ALCANCE

Este procedimiento se aplica a todas las áreas del Laboratorio, comprendidas dentro del sistema de gestión de la calidad.

3. INVOLUCRADOS

Jefe del Laboratorio
Responsable de calidad
Responsable del Sector a revisar

4. DESARROLLO

4.1 La Dirección hará revisiones anuales del sistema de la calidad. Para realizar dichas revisiones se basará en los siguientes parámetros:

- La adecuación de las políticas y procedimientos
- Los informes del personal directivo y de supervisión
- El desempeño de los procesos y conformidad del producto (análisis de datos)
- Los resultados de auditorías internas recientes
- Las acciones correctivas y preventivas
- Las evaluaciones por organismos externos
- Los resultados de las comparaciones interlaboratorios o ensayos de aptitud
- Los cambios en el volumen y tipo de trabajo
- La retroalimentación del cliente
- Los reclamos
- Las acciones de seguimiento de revisiones por la dirección previas
- Las recomendaciones para la mejora de la gestión

Otros factores pertinentes, tales como medios de control de la calidad, recursos y entrenamiento de los empleados.

4.2 Para el registro de las revisiones se utilizará el formulario RGC 08, donde se indicará: los participantes, los temas abordados y resultados de las evaluaciones.

5. REFERENCIAS

RGC 08 **Revisión por la Dirección**

6. REGISTRO DE VERSIONES

Versión	Fecha	Modificaciones
Rev. 0	Abril 2002	----------
1	Oct. 2004	Se amplía el 4.1
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

AUDITORIAS INTERNAS

1. OBJETIVO

Evaluar si el sistema de gestión de calidad es conforme con los requisitos de la norma ISO 9001:2000 ó ISO/IEC 17025:2000 y con los requisitos establecidos por la organización y evaluar si se ha implementado y se mantiene de manera eficaz.

2. ALCANCE

Este procedimiento se aplica a todo el sistema de gestión de la calidad del Laboratorio incluyendo la competencia técnica.

3. INVOLUCRADOS

El responsable de que se lleven a cabo las auditorías es el Jefe del Laboratorio.
Todos los integrantes del Laboratorio están involucrados en el desarrollo de la misma.

4. DESARROLLO

4.1 Programa Anual

4.1.1 El programa anual de auditorías establece el orden en que las actividades del laboratorio son auditadas.

4.1.2 Para la elaboración del programa anual de auditorías se consideran: la relevancia de la actividad, los resultados de auditorías internas y externas anteriores y las modificaciones realizadas en las actividades.

Nota: Cada actividad del Sistema de Gestión de la Calidad se audita como mínimo 1 vez al año.

4.1.3 El programa anual lo elabora el Responsable de Calidad y lo aprueba el Jefe del Laboratorio.

4.2 Equipo Auditor

4.2.1 El Equipo Auditor es designado por la Responsable de Calidad pudiendo estar conformado por personal interno del Laboratorio o externo. El personal interno no puede auditar su propio trabajo. Por lo menos uno de los auditores debe haber realizado cursos de capacitación en auditorias del Sistema de Gestión de Calidad en base a las normas UNIT-ISO 9000:2000 o ISO 17025, o contar con experiencia demostrada habiendo realizado al menos una auditoria relacionada con las normas mencionadas anteriormente.

4.3 Preparación

El Equipo Auditor prepara el Plan de Auditoría, el cual incluye:

- Alcance de la auditoría (identifica la actividad a ser auditada)

- Documentos de referencia: identifica los documentos aplicables al área a ser auditada
- Identificación de la persona responsable del área a auditar
- Integración del equipo auditor
- Fecha, hora de comienzo de la auditoría

El Equipo Auditor prepara además la Lista de verificación a utilizar en la auditoría.

El Responsable de Calidad coordina la fecha definitiva de la auditoría y la comunica al Responsable del Sector y al equipo auditor con un mínimo de 7 días de anticipación.

El plan de auditoría se entrega al Responsable del Sector a ser auditado 2 días de anticipación.

El responsable de la actividad auditada es quien debe informar al personal involucrado el alcance de la auditoría y los detalles de la misma.

4.3 Desarrollo de la auditoría

Durante la auditoría, los auditores determinan si:

- se cumplen los requisitos de la norma ISO 9001:2002 ó ISO/IEC 17025 aplicables a la actividad
- los procedimientos son cumplidos y están implantados y registran los resultados en Lista de Verificación utilizada.

Se procede de acuerdo a los puntos de la lista de verificación, buscando evidencias objetivas de cumplimiento, mediante entrevistas, examen de documentos, verificación de las actividades realizadas y las condiciones de trabajo.

El Equipo Auditor acuerda la fecha de realización de la reunión final de auditoría con el Responsable de Calidad y con los responsables de los sectores auditados, la cual debe llevarse a cabo en un plazo menor a cinco días desde la fecha de la auditoría.

4.4 Reunión final

Durante la reunión final el Equipo Auditor informa a las partes involucradas las distintas no conformidades y observaciones detectadas y entrega el Informe final de auditoría. En el mismo se incluye:

- Alcance
- Responsable de la actividad auditada
- Identificación de los documentos de referencia
- Integración del equipo auditor
- Fecha de realización de la auditoría
- Descripción de las no conformidades y referencia a los puntos de la norma
- Descripción de las fortalezas encontradas
- Descripción de las observaciones y oportunidades de mejora

El informe es entregado al Responsable de Calidad quien lo distribuye a los sectores involucrados. El Responsable de Calidad archiva una copia del informe final de la auditoría y la Lista de verificación entregada por el Equipo Auditor, en la carpeta de Auditorías Internas.

4.5 No Conformidades y Acciones Correctivas

El Responsable de Sector registra cada no conformidad en el formulario ***RGC 06*** y define la acción correctiva y/o preventiva, los plazos para su implementación. Además indica en el formulario la causa de la no conformidad y verificación de la eficacia, según el procedimiento de Control de No Conformidades.

El Responsable de Sector y un integrante del Equipo Auditor deben firmar el Informe de no conformidad en auditoría estableciendo de esta forma su acuerdo con lo allí registrado.

4.6 Seguimiento

El Responsable de Calidad es responsable del seguimiento de las acciones correctivas y/o preventivas propuestas verificando “in situ” la implementación de las acciones tomadas una vez cumplido el plazo establecido para la misma.

Debe registrar los resultados de esta verificación en el sector correspondiente del Informe de no conformidades en auditorías y en caso de que la acción correctiva propuesta no se haya implementado en el plazo acordado, debe registrar la causa de dicho incumplimiento y asignar un nuevo plazo para su implementación, en caso que corresponda.

Una vez verificada la implementación y la eficacia, el Responsable de Calidad y el Responsable de sector firman en el Informe de no conformidad en auditoría.

5. REFERENCIAS

PR 06 Control de No Conformidades

Lista de verificación utilizada

RGC 06 Registro de No conformidades e Implementación de Acciones Correctivas/Preventivas

Informe final de auditoría

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión
1	Junio 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

REVISION DE ACUERDO CON LOS CLIENTES

1. OBJETIVO

El objetivo de este procedimiento es describir la forma de establecer los acuerdos con los clientes y sus modificaciones.

2. ALCANCE

El Laboratorio, realiza acuerdos con los diversos clientes para la realización de análisis. Este procedimiento se aplica a cada nuevo programa a llevar adelante por el Laboratorio de DI.NA.MA. y para la modificación de los acuerdos ya existentes.

3. INVOLUCRADOS

Jefe del Laboratorio y Responsable de Calidad

4. DESARROLLO

4.1 Los clientes realizan la solicitud de análisis telefónicamente o por escrito al departamento indicando claramente tipos de muestras y parámetros. El Jefe del mismo o quien designe, evalúa la capacidad del laboratorio y los acuerdos ya establecidos con otros clientes para aceptar la solicitud, consultando a los responsables de sectores.

4.2 Se deja constancia de la solicitud aceptada, por medio de la aprobación de un **Acuerdo** entre el Laboratorio y el Cliente. Este acuerdo firmado por el jefe, contiene los siguientes puntos:

- Cliente
- Cupo máximo de muestras semanales si corresponde
- Días de ingreso según los parámetros
- Metodología analítica
- Tiempo máximo de entrega de resultados
- Condiciones de muestreo y preservación
- Forma de coordinación de ingreso de las muestras

Este acuerdo se guarda en el Bibliorato de **Fichas de Ingreso de muestras**.

Las solicitudes de análisis, correspondientes a denuncias o auditorías ambientales, que llegan desde la dirección como resolución del Director serán considerados como el acuerdo.

4.3 Anualmente o en cualquier momento por interés de alguna de las partes se realiza revisión del acuerdo, pactando nuevamente con el cliente. Si existen modificaciones se debe hacer un nuevo acuerdo, de lo contrario sigue en vigencia el anterior.

4.4 Si fuese necesario gestionar con el cliente la modificación de algunos términos del contrato, la persona responsable será la Jefa del laboratorio.

4.5 Es responsabilidad del Jefe y/o Responsable de Calidad divulgar entre los responsables de análisis los nuevos acuerdos realizados con el cliente.

4.6 Cuando el Laboratorio no pudiera cumplir con algún término del acuerdo, el Jefe del Laboratorio deberá completar un informe de No Conformidad. El jefe es responsable de hacerle llegar por escrito al Cliente las razones del incumplimiento del acuerdo, en la sección observaciones del Informe de Resultados o con una nota adjunta.

El incumplimiento de un acuerdo puede deberse a:

- Falta de disponibilidad de los técnicos
- Capacidad de volumen de trabajo
- Problemas específicos de la metodología analítica.
- Problemas de infraestructura
- Otros

5. REFERENCIAS

Informe de No conformidad, el cual puede ser una frase dentro del informe de resultados o ser un descargo en hoja separada.

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión
1	Mayo 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

RECLAMOS DEL CLIENTE

1. OBJETIVO

Establecer una metodología para asegurar que todo reclamo del cliente es recibido, identificado, analizado y resuelto.

2. ALCANCE

Este procedimiento se aplica a cualquier reclamo recibido del cliente.

3. INVOLUCRADOS

El Jefe y todos los técnicos del Laboratorio.

4. DESARROLLO

4.1 El técnico del laboratorio que recibe el reclamo, lo registra en el formulario de Registro de Reclamos del Cliente (RGC 13), que se encuentran en el cuaderno de **Reclamos y No Conformidades**, asignándole un número de reclamo. Luego lo comunica al Jefe del Laboratorio. (Cualquier técnico del Laboratorio puede recibir un reclamo).

5 El Jefe del Laboratorio es responsable de la atención del reclamo del cliente. Estudia el reclamo y evalúa si éste corresponde o no. En caso de estar de acuerdo con el reclamo resuelve la acción a tomar y evalúa si es necesario iniciar una acción correctiva o preventiva, en cuyo caso se sigue el procedimiento ***PR 07***.

Si existe una discrepancia con el reclamo, se realizan los descargos correspondientes.

Si el reclamo corresponde a una no conformidad en el resultado de análisis se re-emite el informe de resultado, se envía el nuevo original solicitando al cliente el anterior. El original viejo se lo rotula como Anulado y se lo adjunta a la copia del nuevo informe y se archivan en Resultados Entregados.

4.4 El jefe del laboratorio comunica al cliente y a los involucrados, las acciones a tomar del reclamo. Deja constancia y fecha de la comunicación al cliente en el registro del reclamo.

5. REFERENCIAS

PR 07 Generación e implementación de acciones correctivas y preventivas.

RGC 06 Registro de No Conformidades e implementación de Acciones Correctivas/Preventivas

RGC 13 Registro de Reclamos del Cliente

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Mayo 2002	Primera emisión.
1	Mayo 2003	Segunda emisión
2	Agosto 205	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

CAPACITACION

1. OBJETIVO

Asegurar la competencia técnica del personal del Laboratorio.

2. ALCANCE

Este procedimiento corresponde a todo el personal del Departamento de Normalización Técnica de DINAMA y a los pasantes. Aplica a capacitación externa e interna.

3. INVOLUCRADOS

Los Responsables de Sectores y el Jefe del Laboratorio son los responsables de identificar las necesidades de capacitación y son los encargados de elaborar el programa de capacitación del Laboratorio junto al Responsable de Calidad.

Los Responsables de Sectores son los encargados de evaluar la capacitación impartida.

4. DESARROLLO

4.1 Identificación de necesidades de capacitación para personal permanente

Anualmente los Responsables de Sectores, el Jefe y el Responsable de Calidad identifican las necesidades de capacitación del personal, por medio de una reunión de calidad.

El Responsable de Calidad elabora el Programa anual de capacitación y lo presenta al Jefe del Dpto. para su aprobación. Este plan anual se archiva en la carpeta de **Recursos Humanos**.

Cuando surgen ofertas a cursos en cualquier fecha del año, se evalúan si es de interés y si están dentro de los objetivos del Laboratorio se incluye en el plan anual de capacitación.

4.2 Identificación de necesidades de capacitación para pasantes:

Cuando ingresa un pasante al Laboratorio el Responsable de Sector adonde desempeñará las funciones el pasante, realiza un programa de capacitación para ese pasante y lo incluye en la carpeta **Pasantías**. Si la capacitación se realiza con el objetivo de que desempeñe tareas, en el Laboratorio, como un analista habilitado se evalúa la capacitación recibida igual que la de un funcionario permanente.

4.2 Desarrollo de la capacitación

Los cursos de capacitación pueden ser dictados tanto dentro como fuera del Departamento.

El nivel jerárquico inmediato superior al personal que recibió la capacitación, es responsable de evaluar el aprovechamiento de la capacitación recibida en el desarrollo de las actividades. Esta evaluación se realiza dentro de los tres meses después de recibida la capacitación según los parámetros presentados en el formato del ***RGC 10 Evaluación de Capacitación***. Se registra la evaluación en éste formulario.

Los resultados de las evaluaciones se utilizan como insumos para el seguimiento y evaluación de los programas de capacitación.

La Encargada de calidad mantiene los registros de capacitación en la carpeta "Recursos Humanos"

5. REFERENCIAS

RGC 10 Evaluación de Capacitación

Programa de capacitación

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Junio 2003	Primera emisión
1	Agosto 2005	Segunda impresión

Revisado	Fecha
Aprobado	Fecha

COORDINACION DEL MUESTREO Y PRESERVACION DE MUESTRAS

1. OBJETIVO

El objetivo de este procedimiento es optimizar las condiciones de conservación de la muestra desde el lugar de muestreo hasta la ejecución del análisis.

2. ALCANCE

Todas las muestras que ingresan al laboratorio deben seguir este procedimiento, es observado por los técnicos responsables del muestreo y por el personal del Laboratorio.

3. INVOLUCRADOS

Todos los técnicos del Laboratorio.

4. DESARROLLO

En la tabla adjunta se indica las condiciones físicas y químicas de preservación del parámetro, plazo máximo de análisis, tipo de recipiente para la muestra, volumen mínimo necesario, requerimientos especiales y días posibles de recepción de la muestra según el parámetro solicitado.

4.1 Coordinación del muestreo:

3.1.1 El Cliente coordina con el Laboratorio telefónicamente o por escrito el número de muestras, los análisis solicitados, fecha de retiro de frascos y fecha de ingreso de muestras. El cliente es el responsable de retirar los recipientes de muestreo en el Laboratorio

3.1.2 El personal del laboratorio:

- determina tipo y número de frascos según los análisis solicitado (ver tabla adjunta)
- prepara los frascos necesarios etiquetados con las etiquetas según corresponda
- adjunta las fichas de ingreso de muestras que deben llevar los técnicos al muestreo, cuyos formatos se encuentran en la carpeta de Planilla de Registros en la sección correspondiente.

Los parámetros que figuran con igual código de frasco se pueden muestrean en el mismo frasco.

4.2 Muestreo:

Las muestras una vez recolectadas deben ser **rotuladas** con la identificación y fecha de muestreo y según corresponda **refrigeradas en conservadora con hielo** suficiente para conservar la muestra a menos de 4ºC hasta el ingreso de las mismas al Laboratorio.

El responsable del muestreo debe completar la información requerida en la ficha de ingreso de muestra: solicitante, identificación de muestras, análisis solicitados, fecha de muestreo, técnico responsable y firmar la ficha de ingreso.

4.3 Recepción de muestras:

El responsable del muestreo debe entregar las muestras a un técnico del Laboratorio con sus correspondientes **fichas de ingreso de muestra** completas. En caso de no contar con ficha de ingreso debe solicitarla al ingreso y completarla.

El técnico del laboratorio que las recibe debe ingresarlas según el ***PR14 Procedimiento de Manipulación e Ingreso de la Muestra***.

Si las muestras llegan fuera del horario coordinado y no hay nadie en el Laboratorio para recibirlas el cliente es el responsable de mantener las muestras en condiciones de preservación adecuadas y entregarlas a la brevedad al Laboratorio.

5. REFERENCIAS

PR14 Procedimiento de Manipulación e Ingreso de la Muestra.

Carpeta de Planilla de Registros

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2000	Primera emisión
1	Mayo 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

TABLA DE CONDICIONES DE MUESTREO Y PRESERVACIÓN PARA LOS DIFERENTES PARÁMETROS

Los parámetros que figuran con igual código de frasco se pueden muestrear en el mismo frasco.
Las filas se encuentran ordenadas primero por los códigos de recipiente y luego alfabéticamente por parámetro.

SECTOR MICROBIOLOGIA

MUESTRAS LIQUIDAS					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Coli. termotolerantes y totales, estreptococos fecales, heterótrofos, enterococos, e. coli	MI	Vidrio boca ancha estéril ó polipropileno autoclavable	600 mL	4 ºC	8 h
Dejar cámara de aire. Mantener el frasco tapado hasta el momento de su uso, no apoyar la tapa en ningún lugar que se pueda contaminar. Efluentes de aguas de desechos clorados los frascos deben contener 0.5 mL de Na_2S2O_3, que será suministrado por el Laboratorio. La muestra no puede ser usada para otros análisis. Ingresar las muestras de lunes a jueves, excepto estreptococos de lunes a miércoles. Para muestras de aguas dulces y potables analizar antes de 24 hs					

MUESTRAS SOLIDAS - ARENA					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Coliformes totales, termotolerantes y Estreptococos fecales	Y	Bolsa de polipropileno impermeable estéril	500 gr.	4 ºC	8 h
.Ingresar las muestras de lunes a jueves, excepto estreptococos de lunes a miércoles.					

SECTOR ECOTOXICIDAD

MUESTRAS LIQUIDAS					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Bioensayo de Toxicidad por Microtox	T	Plástico	60 mL	4 ºC	48 h
MUESTRAS SOLIDAS Residuos sólidos industriales, suelos y sedimentos					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínima necesaria**	**Preservación**	**Plazo de análisis**
Bioensayo de toxicidad por Microtox	Z	Bolsa con cierre hermético, impermeable	500 g	4 ºC	48 h

SECTOR FISICO-QUÍMICO

MUESTRAS LIQUIDAS					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Alcalinidad	A	Plástico Vidrio	250 mL	4 ºC	24 h
Llenar el frasco sin cámara de aire. No abrir el frasco hasta antes de analizar					
Amonio	A	Plástico Vidrio	250 mL	4 ºC	48 h
Conductividad	A	Plástico Vidrio	250 mL	4ºC	7 días
DBO_5	A	Plástico Vidrio	500 mL	4 ºC	24 h
Llenar el frasco evitando la aireación de la muestra, no dejar cámara de aire. Ingreso al lab. martes, miércoles, jueves y viernes hasta 12h.					

MUESTRAS LIQUIDAS					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
DQO	A	Plástico Vidrio	100 mL	4 ºC	24 h
Si la muestra no se analiza inmediatamente preservar a pH< 2 con $H_2SO_{4\ conc.}$ hasta 7 días					
Nitrito	A	Plástico Vidrio	100 mL	4 ºC	48 h
pH	A	Plástico Vidrio	250 mL	4 ºC	2 h
Silicato	A	Plástico	50 mL	4 ºC	28 días
Sólidos sedimentables	A	Plástico Vidrio	1000 mL	4 ºC	24 h
Sólidos suspendidos	A	Plástico Vidrio	1000 mL	4 ºC	48 h
Sólidos totales	A	Plástico Vidrio	500 mL	4 ºC	7 días
Turbidez	A	Plástico Vidrio	250 mL	4 ºC en oscuridad	24 h
Detergentes	A	Plástico Vidrio	250 mL	4 ºC	7 días
Clorofila	A	Plástico Vidrio	1000 mL	4 ºC en oscuridad	24 h
Aceites y grasas	G	Vidrio boca ancha	500 mL	4 ºC	28 días
La muestra no puede ser usada para otros análisis. Si se estiman concentraciones altas de grasas, muestrear un volumen menor. En laboratorio llevar la muestra a pH < 2 con HCl cc					
Fósforo total	P	Vidrio	500 mL	4 ºC	48 h
Ortofosfato	P	Vidrio	250 mL	4 ºC	48 h

MUESTRAS DE AIRE					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
PTS y PM10	F	Filtro codificado y pesado en sobre de nylon	1 filtro por punto de muestreo	Filtro con muestra doblado hacia adentro, a la mitad, en sobre de nylon. Temp. ambiente	-------

MUESTRAS DE AIRE					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Indice de corrosividad	D	Disco pesado envuelto en papel de aluminio	2 discos por punto de muestreo	Disco envuelto en papel de aluminio.Temp. ambiente	-------

SECTOR INSTRUMENTAL

MUESTRAS LIQUIDAS					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínimo necesaria**	**Preservación**	**Plazo de análisis**
Cloruro	A	Plástico Vidrio	50 mL	4 ºC	28 días
Metales nutrientes (Ca, Mg, Na, K)	A	Plástico ó bolsas descartables polietileno	200 mL	en laboratorio: llevar a pH < 2 con HNO_3 cc (se pueden analizar en el frasco de metales tóxicos, M)	6 meses
Nitrato	A	Plástico	250 mL	4 ºC	7 días
Sulfatos	A	Plástico Vidrio	50 mL	4 ºC	28 días
Cianuro	B	Plástico Vidrio	1000 mL	4 ºC llevar a pH >12 con NaOH 10 M mantener en la oscuridad	14 días
Metales tóxicos (As-Cr-Cd-Cu-Hg-Ni-Fe-Mn-Pb-Zn)	M	Plástico ó bolsas polietileno	500 mL	en laboratorio: llevar a pH < 2 con HNO_3 cc	6 meses excepto Hg 28 días
Frasco lavado con HNO_3 suministrado por el Laboratorio o bolsas descartables especiales para muestras líquidas. Dejar cámara de aire en el frasco ó bolsa de 20 mL mínimo. Para muestras para análisis de Cr VI una alícuota se refrigera sin acidificación.					
Sulfuro	S	Plástico	250 mL	4ºC + 125 mL buffer agregado previamente al frasco, mantener en la oscuridad.	7 días

MUESTRAS SOLIDAS Residuos sólidos industriales, suelos y sedimentos					
Parámetro	**Código**	**Tipo de recipiente**	**Cantidad mínima necesaria**	**Preservación**	**Plazo de análisis**
Metales tóxicos	Z	Bolsa con cierre hermético, impermeable	500 g	Suelos: $T_{ambiente}$ Sed. - residuos: 4 ºC	6 meses, Excepto Hg 28 días

MANIPULACION E INGRESO DE LA MUESTRA

1. OBJETIVO

El objetivo de este procedimiento es establecer las condiciones de recepción de las muestras, su manipulación y trazabilidad dentro del laboratorio hasta su disposición final.

2. ALCANCE

Todas las muestras que ingresan al Departamento de Normalización Técnica – Laboratorio. Este procedimiento incluye la revisión del contrato.

3. INVOLUCRADOS

Todos los funcionarios del Laboratorio.

4. DESARROLLO

4.1 Recepción de muestras:

El personal técnico del Laboratorio recibe las muestras con sus correspondientes **Fichas de ingreso de muestra** (la que oficiará como ***Contrato)*** y es responsable de

- Verificar el correcto llenado de las ficha (solicitante, identificación de la muestra, parámetros solicitados, fecha de muestreo y técnico responsable de muestreo).
- Chequear que los recipientes de muestreo estén debidamente rotulados, las condiciones de conservación de acuerdo a lo estipulado en el **PR 13 Coordinación de muestreo y preservación**, en caso de no observarse alguna de los requisitos se registra en el item observaciones de la planilla de ingreso.
- Firmar la planilla e indicar fecha y hora de ingreso de las muestras.
- El cliente debe firmar la Ficha de Ingreso y si lo requiere se le da una copia.

4.2 Revisión del contrato:

Se puede modificar la Ficha de Ingreso con el cliente en los siguientes casos:

- Si no coincide lo coordinado previamente con el cliente (**PR 13 Coordinación de muestreo y preservación**) y lo indicado en la Ficha de ingreso.
- Si no coincide alguna de los puntos del **Acuerdo** con el cliente y lo indicado en la Ficha de muestreo
- Si no corresponden los parámetros solicitados en la Ficha de Ingreso con los frascos presentados.

La Ficha de Ingreso también puede ser modificada luego de ingresada la muestra con la aprobación del jefe. Debe dejarse constancia en la misma ficha que se le comunicó al cliente la modificación. Si un análisis no se puede realizar se debe comunicar al cliente lo antes posible, dejando notificación del mismo en el contrato.

4.3 Ingreso de la muestra y del análisis:

Las muestras se almacenan según los requerimientos indicados en la tabla del PR 13.

Las muestras que requieren refrigeración se guardan inmediatamente en la heladera.

El técnico que recibió la muestra es responsable de:

- Ingresar las muestras, con número de análisis correlativo al cuaderno de "Ingreso de Muestras".
- Derivar las muestras a los sectores correspondientes y almacenarlas en las heladeras de cada sector o mesada de análisis según corresponda.
- Archivar la Ficha de Ingreso en el bibliorato correspondiente **"Fichas de Ingreso de Muestras" .**

4.4 Manipulación de la muestra:

El técnico responsable del análisis solicitado es el responsable de:

- tomar conocimiento del ingreso de la muestra fijándose en el bibliorato de **"Fichas de Ingreso de Muestras"**.
- de mantener la individualidad de la muestra y asegurar la trazabilidad de la misma y del análisis.
- de realizar los análisis tomando en cuenta el tiempo máximo de conservación del parámetro**.**

Las muestras desde la recepción, durante el análisis y hasta la salida del informe se almacenan a 4ºC en las heladeras del sector correspondiente.

Luego de entregado el informe final al cliente se procede a la disposición final de las mismas según ***PR 15 Disposición Final De Residuos*** .

La contra muestra se almacena a T ≤ 4°C por lo menos 10 días luego de entregado el informe al cliente, excepto las muestras destinadas a análisis microbiológico que se descartan inmediatamente de realizado el análisis. Las muestras sólidas secas pueden almacenarse a T ambiente.

5. REFERENCIA

PR 13 Coordinación de muestreo y preservación
Fichas de Ingreso de muestras
PR 15 Disposición Final De Residuos

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión
1	Mayo 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

DISPOSICIÓN FINAL DE LOS RESIDUOS GENERADOS

1. OBJETIVO

Asegurar la disposición correcta de los residuos generados durante el proceso analítico.

2. ALCANCE

Todos los residuos generados en el Laboratorio.

3. INVOLUCRADOS

Todos los analistas del laboratorio y el preparador de material.

4. DESARROLLO

Los excedentes de muestras se desechan luego de entregado el informe al cliente, guardando una contramuestra como mínimo 10 días luego de entregado el informe, excepto las muestras para análisis microbiológicos las cuales se descartan inmediatamente.

4.1 Disposición de residuos del Sector Fisicoquímico e Instrumental

El responsable del cumplimiento de estas disposiciones es el responsable del sector correspondiente

4.1.1 Los residuos sólidos se disponen en envases para ser retirados por la IMM.

4.1.2 Las muestras que no fueron preservadas con agentes químicos se vierten directamente por la pileta de desagüe con el grifo abierto.

4.1.3 Residuos ácidos y básicos: las muestras preservadas con agentes químicos que se encuentren a pH < 4 y pH > 10, así como los residuos ácidos y básicos generados del análisis se disponen en el tanque de polipropileno para residuos a neutralizar, identificado como "**Residuos Líquidos**", ubicado en el la campana del Laboratorio I de Fisicoquímico.

Estos se componen de:

- muestras para análisis de sulfuro (pH > 12) cuya concentración sea < 1 ppm
- muestras para análisis de grasas y metales preservadas con ácido (pH < 2)
- muestras para análisis de cianuro (pH > 12) con concentración menor a 1 ppm
- residuos ácidos de análisis de metales, DQO, fósforo total, destilación de cianuro
- residuos básicos de análisis de sulfuro, de amonio y de cianuro con concentraciones menor a 1 ppm.
- Otros residuos ácidos o básicos generados

El preparador de material es el responsable de la neutralización previo a ser descartado por la pileta de desagüe con el grifo abierto. Para neutralizar agregar

bicarbonato o carbonato de sodio comercial, mezclar, dejar reposar unos minutos volver a agregar y repetir la secuencia hasta que no se desprenda más CO_2 o hasta que el pH esté en 5 indicado con papel pH.

4.1.4. Los residuos del análisis de DQO, , previo a ser dispensados en el tanque de "**Residuos Líquidos" ,** si contienen Cr_{VI} remanente (presencia de color amarillo) se reducen con el agregado de azúcar comestible hasta color verde. Este proceso lo realiza el preparador de material.

4.1.5 Los residuos de hexano generados en el análisis de aceites y grasas se recuperan o disponen según se indica en la campana donde se realiza el análisis. Este proceso es responsabilidad del analista.

4.2 Disposición de residuos del Sector Microbiología:

El responsable del cumplimiento de estas disposiciones es el responsable del sector.

Los desechos generados en microbiología corresponden a la muestra de análisis, los medios de cultivo sólidos y líquidos inoculados, ansas descartables y tirillas de pruebas bioquímicas. Luego de descontaminados, los residuos sólidos son dispuestos para la recolección de residuos sólidos urbanos y los líquidos por el desagüe que va a colector.

4.2.1 Esterilización de residuos por autoclavado: se esteriliza en auctoclave de material contaminado a 121ºC durante 30 minutos y corresponde a los siguientes residuos:

- medios cultivo sólidos con y sin filtros, sembrados y con crecimiento colocados en bolsa de polipropileno autoclavable
- bandejas de análisis de sustrato definido
- medios de cultivo líquidos inoculados

4.2.2 Inactivación con hipoclorito: el analista dispone los residuos en recipiente con hipoclorito conc. comercial como mínimo 2 hs, inmediatamente despúes de su uso, luego el preparador de material lo enjuaga y descarta. Corresponde a los siguientes residuos:

- Ansas descartables

Tirillas de pruebas enzimáticas

5. REFERENCIAS

PR 14 Procedimiento para el ingreso de la muestra

Method 1100C Standard Methods for the examination of water and wastewater, 20th Ed.

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Abril 2002	Primera emisión.
1	Mayo 2003	Segunda emisión
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LIMPIEZA DE MATERIALES

1. OBJETIVO

El objetivo de este procedimiento es minimizar las posibles fuentes de contaminación y optimizar la preparación (limpieza y acondicionamiento) del material para los análisis.

2. ALCANCE

Es aplicable a todo el material tanto de muestreo como el empleado para las determinaciones analíticas del Departamento.

3. INVOLUCRADOS

Todos los integrantes del Departamento. Es utilizado por el preparador de material del Laboratorio y los técnicos analistas del Laboratorio, para el material de análisis fisicoquímico e instrumental.

4. DESARROLLO

Relevar el material a ser preparado, enjuagar con agua previo al lavado que se detalla en los distintos apartados.

Sector Físicoquímico

Procedimiento operativo	Material	Programa/Lavado	Enjuague	Secado
DBO5	Botellas de incubación	B – con jabón	Agua desionizada	Propio equipo
DQO	Tubos	D – sin jabón	Agua desionizada	Propio equipo
ACEITES Y GRASAS	Frascos de muestreo	Hexano 24 hs y luego B – con jabón	Agua desionizada	Propio equipo
	Balones	hexano 24 hs y luego B – con jabón	Agua desionizada	Propio equipo
	Perlas de ebullición	hexano 24 y luego agua caliente con jabón	Agua destilada	Estufa a 105ºC
	Tapas de frascos de muestreo	limpieza manual con agua caliente y jabón	Agua destilada	Secado en estufa a 105ºC

Procedimiento operativo	Material	Programa/Lavado	Enjuague	Secado
SOLIDOS TOTALES	Cápsulas	limpieza con esponja y jabón	Agua destilada	A temperatura ambiente. Muflar a 550 ºC por 2 hs. sin superponer
FOSFORO	Frascos de muestreo, frascos de digestión, tubos desarrollo de color, erlenmeyers.	D – sin jabón	Acido Clorhídrico 1+1 en caliente Enjuague posterior con agua destilada	En secador de materiales
DETERGENTES	Bolas de decantación y material accesorio.	D – sin jabón	Enjuague con agua desionizada	Secado en el propio equipo

Material	Lavado	Enjuague	Secado
Material de vidrio en general	D – con jabón	Agua desionizada	Propio equipo
Botellas de plástico de muestreo	D - con jabón	Agua desionizada	Propio equipo

Programa B: 93 ºC

Programa D: 60 ºC

Sector Instrumental

Utilizar los programas de lavado C o D a las temperaturas de 60ºC tanto de lavado como de enjuague. Estas temperaturas indicadas como T1 y T2 en la máquina no alteraran la calibración del material aforado.

A) Material de vidrio y plástico:
Se utiliza el programa de lavado D incluyendo el paso de secado.

B) Metales
El material de metales para lavar se encuentra en un carro en el laboratorio de metales.

Se utiliza el programa de lavado C sin el paso de secado.

Se sumerge totalmente en el tanque de plástico de enjuague con HNO_3(10%) calidad ppa, durante 24 hs como mínimo. Luego se enjuaga 3 veces con agua desionizada.

Se seca a menos de 60ºC en estufa.

Se guarda el material seco en el estante correspondiente a material de metales.

C) Aniones por HPLC:
El material de aniones para lavar se ubica en la mesada del laboratorio de aniones.

Se utiliza el programa de lavado D incluyendo el paso de secado.

Se guarda el material en el estante correspondiente a material de aniones.

D) Sulfuro y Cianuro:
Se procede igual que en el punto C)

Sector Microbiología

1. Placas de Petri de Plástico No Autoclavables

DESINFECCION DE PLACAS DE PETRI DE PLASTICO

Esta técnica se utiliza como un método de desinfección de placas de Petri de plástico.

Los materiales que se emplean a tales efectos son los siguientes:

- Agua destilada
- Estufa de secado de hasta 100ºC
- Pinza
- Bolsa autoclavable
- Solución detergente al 3%
- Solución de hipoclorito de sodio
- Etanol 70%

El procedimiento consiste en retirar el filtro y el medio de cultivo de la placa de Petri con una pinza. Acondicionar el residuo en una bolsa autoclavable para su esterilización.

Sumergir las placas en solución de detergente al 3% por 12-24 hs.

Con el uso de una esponja lavar bien las placas tratando de quitar los restos de medio que pudieran quedar adheridos a las placas. Enjuagar con abundante agua corriente y destilada con el fin de eliminar el resto de detergente.

Posteriormente sumergir las placas en hipoclorito de sodio por 2 hs.Volver a enjuagar con abundante agua corriente y destilada.

Colocarlas en una solución de etanol al 70% de 1 a 2 hs.

Secar las placas en la estufa de secado a una temperatura de no más de 50ºC.

ESTERILIZACION POR RADIACION ELECTROMAGNETICA UV

Esta técnica es utilizada como un método de esterilización de placas de Petri de plástico que no pueden ser autoclavables.

La luz ultravioleta es una radiación electromagnética cuya longitud de onda está comprendida entre 150-4000 Aº.

Su efecto sobre las células es a nivel del ADN ocasionando graves daños (mutaciones) en dicha molécula, lo que lleva a la muerte celular. El mayor efectos sobre los microorganismos se da a una longitud de onda de 2600 Aº.

Las lámparas germicidas emiten luz ultravioleta de aproximadamente 2573 Aº, esto hace que tengan un 85% de la capacidad germicida de la radiación de 2600Aº.

Los materiales que se emplean son los siguientes:

- Estufa de secado de hasta 100ºC
- Lámpara germicida de UV de longitud de onda de 2573 Aº.
- Agua Destilada
- Solución detergente al 3%

El Procedimiento de esterilización consta de varias partes:

Preparación de las placas de Petri: para el lavado del material luego de retirado el medio de cultivo de las placas sumergir estas en solución de detergente al 3%, lavar bien con una esponja tratando de quitar los restos de medios que pudieran quedar adheridos a las paredes de las placas.

Enjuagar con abundante agua corriente y luego con agua destilada con el fin de eliminar el resto de detergente.

Secar las placas en estufa de secado a una temperatura de no más de 50ºC.

Preparación a la Esterilización: exponer las paredes internas del fondo y de la tapa las cuales deben estar secas a la acción directa de las radiaciones UV por un tiempo mínimo de 3 hs. Luego de expuestas cerrarlas rápidamente evitando tocar su interior

Guardar las placas envueltas y en la oscuridad para evitar la fotoreparación por no más de 2 semanas después de esterilizadas.

Periódicamente limpiar la lámpara germicida ya que la acumulación de por ej. del polvo del ambiente puede interferir con la emisión de la luz UV reduciendo su poder germicida.

2. Descarte y Esterilización del Material Contaminado de Análisis Microbiológicos.

ESTERILIZACIÓN DEL MATERIAL DE MUESTREO CONTAMINADO

Es una actividad obligatoria a ser realizada antes del lavaje de todo el material de vidrio u otros materiales (excluir los recipientes de plástico) que fueran utilizados en análisis microbiológicos.

Todo material debe ser autoclavado antes de ser desechado con el fin de evitar posibles riesgos para la salud.

DESARROLLO ANALITICO

1. OBJETIVO

El objetivo de este documento es establecer el procedimiento para habilitar un nuevo parámetro y/o matriz a brindar como servicio analítico, asegurando que el análisis satisface los requerimientos de aplicación previsto.

2. ALCANCE

El Laboratorio no diseña metodologías analíticas, valida los métodos normalizados utilizados, así como las ampliaciones o modificaciones de éstos para confirmar que los métodos son adecuados al uso pretendido.

Este procedimiento se aplica a un nuevo parámetro o a un nuevo tipo de muestra a analizar que es interés de un cliente interno de DINAMA o es de interés del laboratorio realizarlo. También es aplicable a análisis que ya realiza el Laboratorio pero se cambia la metodología de determinación.

3. INVOLUCRADOS

El Jefe del Laboratorio, los responsables de Desarrollo de los Sectores, y todos los analistas.

4. DESARROLLO

4.1 Identificación de los elementos de entrada :

Se establecen los elementos de entrada necesarios, en caso de desarrollar un nuevo análisis los elementos de entrada a considerar son:

- el parámetro
- la matriz (tipo de muestra)
- tiempo pactado de realización del desarrollo (en caso que corresponda)
- norma de referencia (si existe norma nacional ó si está establecida por el cliente. El valor límite de la norma corresponde al límite de detección requerido)
- Exactitud y precisión máxima a admitir comparando con técnicas similares o valores de referencia en la bibiliografía.

El Responsable de Desarrollo del Sector involucrado es el responsable de revisar de que estén todos los elementos de entrada identificados. Además de los indicados puede ser necesario identificar otros elementos de entrada de acuerdo al desarrollo a realizar.

4.2 Planificación

Se planifica el desarrollo estableciendo las actividades que se van a realizan para cada etapa, el responsable y el plazo estipulado para las mismas.

Se establece cuando se realizará la revisión y la verificación del desarrollo y los responsables de las mismas.

4.2.1 Etapa de selección de método (si corresponde): se identifica la normativa que se va a aplicar para el parámetro y/o tipo de muestra, con el objetivo de determinar cual es el límite al cual se debe llegar. Si existe norma nacional, se utiliza esa norma, en caso contrario se busca normas o guías internacionales de referencia. Si el valor que fija la norma está determinado para un tipo de metodología, esa será la metodología seleccionada.

Según el parámetro, el tipo de muestra a analizar y el límite al que hay que llegar se realiza la selección de la metodología a utilizar en base a la bibliografía de referencia como el Standard Methods of Examination of Water and Wastewater, Environmental Protection Agency, u otra bibliografía de referencia internacional acreditada, teniendo en cuenta:

- límite de detección
- equipos y materiales necesarios
- sencillez en la ejecución del método

Se establece el equipamiento, materiales y reactivos necesarios, en caso de no tener en el laboratorio se realiza la adquisición de los mismos.

4.2.2 Etapa de Pruebas analíticas:

El responsable de Desarrollo del Sector involucrado, planifica las pruebas a realizar, tipo de muestras y cantidad, para la validación del análisis. Se realiza la validación según el ***PGC 01 Procedimiento de Validación de Técnicas***, para establecer por análisis estadístico, siempre y cuando se aplique, los datos de Precisión, Exactitud, % de recuperación, Límite de Detección y Cuantificación, etc. Éste y demás registros asociados a este desarrollo se archivan junto en la carpeta de **Validación de Técnicas** del Sector Correspondientes.

4.3 Revisión

4.3.1 El responsable de Desarrollo revisa que se esté llevando a cabo el mismo de acuerdo a lo planificado. Además evalúa si la planificación es adecuada y si están bien establecidos los elementos de entrada. Se deja registro de esta revisión y de los cambios realizados, así como la comunicación de los cambios a los involucrados en el desarrollo.

4.4 Verificación y validación

4.4.1 El responsable de Desarrollo verifica de acuerdo con lo planificado que el desarrollo cumple con la aplicación prevista, contrastando que los resultados cumplan con los elementos de entrada establecidos de límite de detección, exactitud y precisión, y/o

todos los demás elementos de entrada identificados para el propio desarrollo. Se deja registro de esta verificación y de las conclusiones de la misma. La validación y la verificación se realizan simultáneamente ya que al verificar se corrobora la aplicabilidad del análisis de acuerdo a los requisitos del solicitante.

4.4.2 El Jefe aprueba el nuevo análisis teniendo en cuenta esta verificación y se incorpora a la lista de análisis que realiza el Laboratorio.

4.5 Control de cambios

Cualquier modificación a la técnica seleccionada realizado durante el desarrollo, se registran, se validan de igual manera que el punto 4.2.2.

4.6 Elementos de salida

Se elabora o modifica el Procedimiento Normalizado de Operación según el ***PR 01 Elaboración de Documentos***, el cual es uno de los elementos de salida del desarrollo.

Se determinan los datos de validación de la técnica como LD, Exactitud, precisión , etc., si corresponde.

4.7 Informe al Cliente:

Si el desarrollo fue solicitado por el cliente, se le notifica la conclusión del desarrollo.

5. REFERENCIAS

PGC 01 Procedimiento de Validación de Técnicas

PR 01 Procedimiento de Elaboración de Documentos

6. REGISTRO DE REVISIONES

Versión	Fecha	Modificaciones
0	Junio 2003	Primera emisión
1	Agosto 2003	Segunda emisión
2	Agosto 2005	Tercera impresión

Revisado	Fecha
Aprobado	Fecha

ANNEX (4)

INDEX

ANALYTICAL QUALITY CONTROL MANUAL

INTRODUCTION

Aim:

The Analytical Quality Control Manual is aimed at documenting the analytical control procedures applied in the analysis of the samples processed by DINAMA Laboratory in order to ensure the quality of the results to be published.

The procedures employed for the analytical control are based upon those published in the "Standard Methods for the Examination of Water and Wastewater", and others which have been evaluated, selected and adapted with the experience and the knowledge of the technicians in our Laboratory.

Scope:

The analytical quality control procedures documented are of general application, one or more may be used by analysis technique, as indicated in the Standard Operating Procedures (SOP).

Annex I includes a table with the controls required for each analytical determination. The Quality Control point in the Standard Operational Procedure of the parameter, indicates the particular procedure to be used.

The Manual includes:

- Index
- Glossary of terms, definitions and basic concepts
- ACCURACY control procedures.
  - Control of solutions with a known concentration
  - Recovery percentage control
  - Procedure for the preparation of control solutions
- PRECISION control procedures.
  - Determination of the precision and control of the Standardised Range
  - Precision criterion for microbiological analysis with the filtering membrane technique
  - Precision control for BOD_5 analysis
- Analysis of white control
- Validation of analysis techniques
- Determination of the detection and quantification limit
- Quality control procedure in the determination of microbiological parameters by the filtering membrane technique

- Quality control procedure for the determination of acute toxicity with the Microtox System®
- Verification of the consistency of the analytical results
- Annexes:

  Annex I: analytical quality control for each analytical parameter and frequency of the treatment and assessment of data.

  Annex II: planning, evaluation and register of the participation in inter-laboratory tests

People responsible:

The routine checks of the analyses, as well as the corresponding control graphs, are carried out by the analysis technician each time the analysis is run. The person responsible for the analyses in each section makes sure that they are carried out.

The technician responsible for the analysis makes the evaluation and, depending on the result, they proceed as indicated in each Standardised Operation Procedure.

The person responsible for quality is in charge of preparing the control solutions and purchases the certified reference materials in coordination with the people responsible of the section. They must also check that the statistical treatment (control graphs) and the quality control assessment are made.

BASIC CONCEPTS – GLOSSARY OF TERMS AND DEFINITIONS

Standard deviation:

If the same sample is analysed an infinite number of times, the frequency of the results will be distributed according to the Normal curve or the Gauss bell. This curve is defined by the average between the results and the standard deviation (σ). If there are no systematic errors, the average is the real value and the standard deviation represents the dispersion of the results, that is, the random error.

The following table shows the proportions of the results which fall between the multiples of the standard deviation:

Multiples of the σ	% of results implied
$\pm 1\ \sigma$	68.3 %
$\pm 2\ \sigma$	95.45 %
$\pm 3\ \sigma$	99.7 %

I.e.: 99.7 % of the results fall within the average ± 3 standard deviations.

Relative dispersion:

It is a measure of the method's precision. It corresponds to the method's relative standard deviation.

Error:

The error in an analytical result is the difference between the result and the real value. It can be classified as random or systematic:

a) Random error: repeated analyses of a homogeneous sample, the results of which are not identical. The results show a larger or a smaller dispersion, this dispersion is called random error, because this particular result varies randomly and it cannot be predicted.

Random results are caused by the variation of different factors such as the variation in the time of the chemical reactions, contamination, fluctuations of the instrument, change of analysts, etc.

Random errors are always present and it is necessary to have statistic techniques in order to be able to determine them.

b) Systematic error: (or non-random error) it is indicated by the tendency of the result to be higher or lower than the real value. The term 'accuracy' is used as a synonym of error.

Systematic errors may be eliminated through corrections, while random errors are always present; they may be reduced but not eliminated.

Accuracy:

It is the degree of agreement of the value of a measure with the real value. Accuracy is a measure of the systematic error.

The accuracy in a determination is expressed as an error percentage:

$$\text{Accuracy (\% E)} = (R - V) / V * 100$$

R being the average of the replicas of the reference material and V the real value of the reference.

Accuracy is affected by two components, one due to the method itself and the other one due to the use of the method in the Laboratory. The accuracy of a given method may be satisfactorily assessed in a comparative study between laboratories, in which the difference between the average value of the laboratories and the real known value is the accuracy of the method.

The accuracy for each Laboratory is the difference between the value reported by the Laboratory and the real value. However, this is a combination of both accuracies. In order to establish the Laboratory's specific accuracy, it is necessary to carry out recovery measures, to calculate the average of the recovered concentration and to subtract the value of the method's accuracy obtained from an inter-laboratory study. This difference is interpreted as the accuracy due to the practice in the Laboratory.

Another way to determine the accuracy is by means of a certified reference material or by means of the recovery percentage of a known concentration.

Strengthening:

It is the concentration of a standard of the analyte of interest, to which a problem sample is added.

Control graphs:

Control charts are graphic presentations of the magnitude of a parameter in relation to the time with statistically defined limits, drawn along the X axis. The concentration measures are expected to be found within these limits. These graphs are used to know whether the methodology used is in statistical control.

At least 20 data are necessary to prepare a control graph or a control chart. The average of the data, the standard deviation(s) of the same and the limits are calculated.

The control limits show that there is a 99.7 % probability that a measure will fall in the average ± 3, or, in other words, 99.7 % of the values are between the average and the higher control limits and the threshold.

The alarm limits establish that there is a 95.45 % probability that a measurement might be within these limits.

Control limits are determined based on:

Higher control limit = Mean + 3s

Control threshold = Mean - 3s

Higher alarm limit = Mean + 2s

Alarm threshold = Mean - 2s

In order to build the graph, parallel lines to the X axis are drawn, which indicate the value of the mean and the higher control and alarm limits and threshold on both sides of the median line. The individual values of the measure are then introduced and the result is interpreted.

Interpretation of the result:

- If the result is within the control limits, the determination process is under statistical control.
- If a datum exceeds the control limits, the control analysis must be repeated as well as the set of samples, to confirm that the next one is within control, otherwise discontinue the analysis and correct the problem.
- If two out of three data exceed the alarm limits on one side of the median line, repeat the analysis and continue the analysis only if it falls below the control limits; otherwise discontinue the analysis and correct the problem.

Control graphs are essential tools to control the quality of the samples:

Four kinds of graphs can be used:

- Reference material control or control solutions: accuracy graphs

- Strengthened samples control: recovery percentage graphs

- Duplicate control: precision graphs

- White control: white graphs

Range control graphs:

For duplicate sample analysis, the graph shall appear different due to the variation in the concentration. The range may be expressed as a function of the relative standard deviation, for which the range is normalised dividing it by the duplicate's concentration average. This quotient, expressed as a percentage, is called relative dispersion. The relative dispersion average is determined for the duplicates analysed and the standard deviation of the relative dispersions. Once this information has been obtained, proceed as with any control graph.

Control graphs have another important function: to ensure the improvement in the method's precision. If the measures, both in means graphs and in the range charts never or rarely exceed the alarm limits (Mean ± 2s), then the alarm and control limits must be recalculated using at least the 20 most recent data.

Certified reference material:

Certified reference materials are those for which one or two parameter values have been certified by a technically validated procedure and which have a certificate proving the concentration of their analytes. In the reference materials there appear synthetic references to the matrix of interest in natural waters, rainwater, effluents, etc.

The certified reference materials, as well as the control solutions are used to know and control the accuracy of the method. Following the control graph of their concentration, it is possible to spot the systematic errors or non-random errors during the analysis procedure.

Detection and quantification limit:

In any analytical method requiring small concentrations, the analytical responses should be compared for the sample and the white. These two responses may differ due to the random errors, even when the sample does not contain the analyte under analysis. As the difference between both responses increases, its presence in the sample becomes more evident.

Detection limit of the instrument: (DLI) it is the analyte's lowest detectable concentration and it is statistically different from the response obtained from the instrument's background noise. The DLI is established by several direct measures in the instrument of a solution of the analyte at a concentration of the analyte of five times the estimated DLI, then the standard deviation of the replicas is analysed.

Detection limit of the method (MDL) it is the lowest detectable concentration of an analyte by a given method and it is statistically different from the response obtained by a white by the complete method, including chemical extraction and pre-treatment of the samples.

Quantification limit: (QL) it is the concentration of an analyte which produces a signal significantly higher than the white; this signal may be detected with specific limits during the routine analyses.

Precision:

Precision is the measure of the degree in which multiple analyses of a given sample agree with any of the latter. In statistics, the concept is linked to dispersion. It is the measure of the random variability of the analytical result of the method due only to non-assignable causes, from the preparation of the sample to the measurement of the same, including the variation of the analysts.

Precision is generally reported as the standard deviation (s) or the relative standard deviation (RSD).

When only a few replicas per sample are used, the result range, R, is as efficient as the standard deviation, due to the fact that both measures differ in a constant ($R = 1.128s$ for analyses in duplicate).

Range:

Range is the maximum width between the replicas in absolute value, in the case of duplicate analyses, it is the difference between both results.

Normalised range:

It is the quotient between the range and the average of the duplicates expressed as a percentage.

Recovery:

Recovery is the capacity of the method to determine the amount of analyte of interest contained in the sample. In the absence of certified reference materials, it is possible to estimate the accuracy through the determination of the recovery of the strength of a given parameter. In order to determine the recovery percentage, a known mass of the analyte of interest is injected into the sample and calculated as follows:

$$\text{Recovery \%} = \frac{C_{\text{sample + strengthening}} - C_{\text{sample}}}{C_{\text{strengthening}}} \times 100$$

$C_{\text{sample + strengthening}}$ is the measure of the concentration of the strengthened sample

C_{sample} is the concentration of the non-strengthened sample

$C_{\text{strengthening}}$ known concentration of the strength added to the sample.

Prepared control solutions:

It is a solution of one or more analytes of interest in known concentrations. These solutions are prepared in the Laboratory with primary high-quality patterns, by the person responsible for quality. These solutions as well as the certified reference materials are used to know and control the accuracy of the method. Following the control graph of its concentration, it is possible to detect the systematic errors or non-random errors during the analysis procedure.

PROCEDURE FOR THE VALIDATION OF THE TECHNIQUE (PGC 01)

1. Aim

This method is used to confirm, through the examination and the supply of objective data, that the performance characteristics of a given technique fulfil the requirements for the application of the desired analysis.

2. Scope

The analytical techniques used in the laboratory are generally standardised methods as referred to in *Standard Methods for the Examination of Water and Wastewater*. Most of the techniques have been altered at some stage, for them to be applicable in our laboratory.

For the standardised techniques, altered or not, at least the following parameters of the technique's performance must be determined:

- Detection and quantification limits
- Linearity and work range
- Accuracy
- Precision
- Uncertainty of the method for the determined matrix

This validation procedure must be applied completely for the non-standardised techniques. (see point 6)

3. References

Procedure for the determination of the detection and quantification limit

Procedure for the determination of the calculation of uncertainties

4. Summary of the method

A technique must be validated when it is necessary to verify that its performance parameters are adequate for the resolution of a given analytical problem.

The objective parameters which allow the validation of a technique are the following:

Selectivity: It is the ability of a method to determine the analyte of interest in the presence of other components present in the sample's matrix both, accurately and specifically.

Detection limit: Lowest concentration of the analyte that can be detected but not necessarily quantified.

Quantification limit: Lowest concentration of the analyte that can be quantified with adequate precision and accuracy.

Linearity: It is the ability of the method to obtain results which are directly proportional to the concentration of the analyte, within a given work range.

Work range: Series of values of measures for which the instrument's measurement error is expected to fall within the specified limits.

Accuracy: Degree of agreement between the result of a measurement and the accepted reference value. It includes reliability and precision.

Precision: Degree of agreement between the independent test results obtained in the established conditions. The precision only depends on the distribution of the random errors.

Sensitivity: The change in the response of a measurement instrument, divided by the corresponding change in the stimulus.

Recovery percentage: It is mathematically defined as:

%R=[(CF-CU)/CA]*100

Where CF is the concentration of the analyte measured in the strengthened sample; CU is the concentration of the analyte measured in the non-strengthened sample; CA is the concentration of the analyte added to the strengthened sample.

5. Validation tools

Reagent white: The reagents used in analytical processes (including solvents) are analysed, in order to determine if any of them contributes to the signal. Thus, the measured signal can be corrected.

Strengthened solutions: They are materials or solutions which have been strengthened with the analyte of interest. They are usually prepared by adding a solution with a known concentration. These materials or solutions may already contain the analyte of interest, thus we must be cautious that the strengthening does not take it to levels above the method's application range. Strengthening the solution with a known analyte, allows for the improvement of the response. Please note that in the strengthening techniques, the added analyte is not highly introduced in the matrix, as the sample is naturally found, thus even if the interferences with the matrix are taken into consideration, they are not completely eliminated.

Standard solutions: They are substances used for calibration or identification. In practise, they may be any substance in which a particular parameter or property has been fully quantified, for them to be used as reference or for calibration purposes.

Reference materials: Materials or substances, one or more of the properties of which have been homogeneous enough and properly established, so as to be used for the calibration of a piece of equipment, the confirmation of a measurement method, or to assign values to materials.

Certified reference materials: Reference materials accompanied by a certificate, which states that one or more of its properties are certified by a procedure, which establishes its traceability to an exact realisation of the unit, in which the values of the property are expressed and for which each certified value entails an uncertainty at a set trust level.

6. PROCEDURE

For the routine techniques in the Laboratory, the validation of which has not been previously run, collect the necessary data to determine the performance parameters, the analysis routes and the inter-laboratory tests.

The analytical registers which correspond to the validation, the calculation and the results of the performance parameters are saved in each section's "Validation File".

6.1 Selectivity confirmation:

One of the following procedures may be used:

a) Analyse the samples and/or reference materials using the chosen method and other independent methods. The ability of the method to identify and measure the analyte free from interferences is verified.

b) Participate in inter-laboratory tests of the parameter of interest.

6.2 Determination of the Detection limit:

Follow the “Procedure for the determination of the detection and quantification limit”

6.3 Linearity and work range:

Analyse a reagents’ white plus standard solutions of at least 6 different concentrations. Show the response against the concentration of the analyte in a graph and verify that the work range of each parameter is within the linear range. Determine the work range and its higher limit and threshold.

6.4 Accuracy:

The reagent white and the certified reference material is analysed or an inter-laboratory sample at least 10 times with the selected method. The average of the white is subtracted from the average of the concentration and it is compared to the real or accepted value for the reference material. This provides a measure of the method’s bias.

6.5 Precision:

Standard solutions, reference materials or strengthened whites are analysed at different concentrations along the work range, making 10 independent repetitions for the different cases. It is also possible to analyse at least 20 real samples in duplicate and to determine the precision with the Standardised Range (see “Precision determination procedure“).

This procedure should be carried out by the analysts analysing this parameter in the laboratory over a long period of time. Determine the standard deviation at each concentration (or for each range of concentrations in case the samples analysed are duplicates of real samples). In this way, the precision is determined according to the range of concentrations, under intra-laboratory reproducibility conditions.

6.6 Recovery percentage:

The blank matrixes or the samples strengthened or not strengthened with the analyte of interest are analysed 6 times in a work range. From the data, the recovery of the analyte at various concentrations in relation to the expected concentration value is obtained.

The recovery of the strengthened samples or white matrixes is usually better than in real samples in which the analyte is more bound.

7. Bibliography:

EURACHEM WORKING GROUP. The Fitness for Purpose of Analytical Methods. A Laboratory Guide to Method Validation and Related Topics. Edition 1.0, 1998. pp 2-33 y

Annex A Definitions.

CONTROL PROCEDURE WITH KNOWN CONCENTRATION SOLUTIONS

(PGC 02)

1. Aim

To know and to control the analytical accuracy of the method used; to detect systematic errors during the analysis.

2. Scope

This is a routine procedure to determine:

- **Natural waters**:	Total solids; BOD5; COD hardness; calcium; magnesium; sodium; potassium; silicate; Acute toxicity (Microtox ®) chloride; sulphur; nutrients
- **Rainwater**	Fluoride; chloride; sulphur and nitrate
- **Industrial effluents**	Total solids; Acute toxicity (Microtox ®) BOD_5 ; COD; Ammonium and toxic metals
- **Sediments and soils**	Toxic metals

The following may be used as control solution:

control solutions prepared by the person responsible for quality,

certified reference materials

inter-laboratory samples of a known value and within the allowed storage time, (ex: metals) or which are stable over time within an acceptance range between 80 and 120%.

3. Reference:

See the following definitions in the glossary of basic terms and concepts:

- Standard deviation
- Systematic error
- Accuracy
- Control graphs
- Control limits
- Certified reference materials
- Prepared control solutions

4. Summary of the method:

The chosen control solution is analysed simultaneously with the samples. The same procedure is applied to the solution and the samples the concentration of the analyte of interest is determined and its value is controlled with a control graph.

If the concentration of the control solution is beyond the control limits, we are probably facing a possible systematic error at one of the stages of the analytical procedure. The analyst shall then consult the person responsible for quality and, analysing jointly the remaining controls applied to the analytical technique, they shall conclude whether the analyses of all the samples in the run should be repeated, researching the cause of the deviation from the expected behaviour.

5. Procedure

5.1 Preparation of the prepared control solution:

Determine the reagent to be used as control standard. The solution must be stable at least during the time of the analysis.

The control solutions are prepared in each case following the indications in the **Procedure for the preparation of control solutions**. The registers of the preparation are kept in the format "Preparation of control solutions to check techniques ". (RQA formats)

In the case of Certified reference materials, the preparation steps are overlooked.

The control solutions may be preserved samples from inter-laboratory tests which have been preserved, as is the case of samples for metals.

5.2 Control of the concentration of the control solution:

1. Analyse the control solution and the run of the samples. After obtaining at least 20 values of the concentration, calculate the average of the values (M) and the standard deviation (s).
2. Determine the central line (CL) and the higher limits (HIL) and the thresholds (TL) for the control graph, according to the following:

   CL = M

   HIL = M + 3s

   TL = M - 3s
3. Build the control graph of the concentrations. Verify that all the points are within the control limits, otherwise eliminate the point or points which are out of control, adding as many values as necessary until the 20 values mentioned are obtained and re-calculate the average and the control limits.
4. Keep the control graph according to the schedule indicated in Annex I. For each new analyses run, verify if the concentration of the control solution is within the limits.

5.3 Determination of the accuracy of the method:

The accuracy of the method may be determined with a certified reference material.

Accuracy is expressed as error percentage:

$$\text{Accuracy (\% E)} = \frac{\text{ABS (R – V)} * 100}{\text{V}}$$

R = the measure of the replicas of a reference material

V = the real value of the reference material

ABS = the absolute value of the difference

Accuracy has two components: one due to the method and the other one due to the use of the method in the Laboratory. A method's accuracy may be assessed by means of reference material or in a comparative study between laboratories, in which the difference between the average value informed by the laboratories and the real or known value is the accuracy of the method.

The accuracy of the method may also be calculated by the calculation of the recovery percentage of the concentration of the strengthened sample (see corresponding procedure).

6. Bibliography

1- AMERICANN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

PROCEDURE TO CONTROL THE RECOVERY PERCENTAGE (PGC 03)

1. Aim

To know and to control the accuracy of the method; as well as to detect systematic errors during the analysis.

2. Scope:

This is a routine procedure for the determination of: COD, chromium, lead, sulphur and ammonium in industrial effluents.

3. Reference:

See the following definitions in the glossary of basic terms and concepts:

- Standard deviation
- Systematic error
- Accuracy
- Control graphs
- Control limits
- Recovery
- Control solutions
- Strengthening

4. Summary of the method:

Simultaneously with the analysis of the set of samples a sample is chosen from the matrix of interest and analysed by triplicate. A known concentration of the analyte is added to one of the replicas, so that the concentration estimated in the sample, plus the one in the added analyte does not go beyond the reading range. The recovery of the concentration of the added and the recovery percentage are controlled with a control graph.

If the recovery percentage exceeds the control limits or the acceptance limits established for the parameter, we assume that we are facing a systematic error at some of the stages of the analytical procedure or we are facing a problem of matrix interference.

5. Procedure:

Choose the strengthening solution, which may be a prepared solution with a known concentration, a certified reference material or an inter-laboratory sample, the real value of which and the concentration of which is stable within the control period, as in the case of metal samples.

5.1 Control of the recovery percentage of the strengthening:

1. Select the sample to be strengthened (select at least one kind of industry per analyses run, alternately).
2. Strengthen the triplicate of the sample with an aliquot of the corresponding control solution, as indicated in the parameter's Standardised Operation Procedure.
3. Analyse the strengthened sample together with the run of the samples. Calculate the recovery percentage according to the following formula:

$$\text{recovery \%} = \frac{(C_{\text{sample + strengthening}} - C_{\text{sample}}) \times 100}{C_{\text{strengthening}}}$$

$C_{\text{sample + strengthening}}$ is the measure of the concentration of the strengthened sample

C_{sample} is the concentration mean of the duplicates of the non-strengthened sample

$C_{\text{strengthening}}$ known concentration of the strengthening added to the sample.

4. After obtaining at least 20 values of the recovery percentage of the strengthened concentration, calculate the average of the recovery percentages ($\%R_{med}$) and the standard deviation (s).
5. Determine the central line (CL) and the higher limits (HIL) and thresholds (TL) for the control graph, according to:

$LC = \%R_{med}$ $HIL = \%R_{med} + 3s$ $TL = \%R_{med} - 3s$

6. Build the control graph of the recovery % concentrations. Verify that all the points are within the control limits, otherwise eliminate the point or points which are out of control and re-calculate the average and the control limits.
7. Keep the control graph according to the schedule indicated in Annex I. For each new analysis run, verify if the recovery percentage is within the limits or the acceptable limits established for the parameter.

5.2 Determination of the accuracy of the method:

The accuracy of the method may be determined with the recovery percentage of the concentration with which the sample was strengthened.

Accuracy is expressed as percentage error:

$$\text{Accuracy (\% E)} = \text{ABS}\,(100 - \%R_{med})$$

Where $\%R_{med}$ is the average of recovery percentage of several replicas of strengthened samples and ABS is the absolute value of the difference.

Accuracy has two components: one due to the method and the other one due to the use of the method in the Laboratory.

6. Bibliography

1- AMERICANN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

PROCEDURE FOR THE PREPARATION OF CONTROL SOLUTIONS (PGC 04)

1. Aim

This procedure describes the preparation of the control solutions used in the accuracy controls.

2. Scope

The preparation of the control solutions is the responsibility of the person responsible for quality. This procedure contains the preparation of the following solutions:

- Ammonium
- Nitrate
- Total solids
- Total Phosphorus and orthophosphate
- COD
- Total hardness and calcium
- Sodium and Potassium
- Silicate
- BOD5

3. Procedure:

a) Prepare and store the solution as indicated in the annexed table. Take the volume of the weighed material or take the weight, taking the density as 1,0 g/mL. Register the takes and volumes in the “Preparation of Control solutions” form, which is together with the procedure of each technique.

b) Calculate the exact concentration of the analyte as is indicated in the table attached, in the desired units.

c) Label the bottles with the identification of the parameter to be controlled, the preparation and expiry dates, exact concentration, name of the person responsible for the preparation and way of storage.

See tables further on.

4. Bibliography

1- AMERICAN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1999. Pages corresponding to the reference analysis methodologies.

TABLE FOR THE PREPARATION OF THE CONTROL SOLUTIONS

AMMONIUM CONTROL SOLUTION

a) Dry ammonium chloride (NH4Cl) at 105ºC during 2 hours.

b) Weigh 0.3819g of NH4Cl and dissolve it in 500,00 mL of distilled water.

Register the weight, the volume of the water and the exact concentration of the solution expressed as mgN/L in the “Preparation of Control Solutions” form.

c) Calculate the concentration of the control solution expressed as mgN/L, according to:

$$mgN/L = \frac{\text{Take NH4Cl (mg)}}{0.500} \times \frac{14}{53.5}$$

FW (NH4Cl)=53.5 FW (N) = 14

d) Store the solution in a labelled plastic bottle, stating the name of the person responsible for the preparation, the date of the same and the expiry date, as well as the exact concentration. Store in the fridge for 6 months.

NITRATE CONTROL SOLUTION

a) Dry potassium nitrate (KNO_3) at 105ºC during 2 hours.

b) Weigh 0.7218g of KNO3 and dissolve in 500,00 mL of distilled water.

Register the weight, the volume of the water and the exact concentration of the solution expressed as mgN/L in the “Preparation of Control Solutions” form.

c) Calculate the concentration of the control solution expressed as mgN/mL, according to:

$$mgN/L = \frac{\text{Take KNO3 (mg)}}{0.500} \times \frac{14}{101}$$

FW (KNO3) =101 FW (N) = 14

d) Store the solution in a labelled plastic bottle, stating the name of the person responsible for the preparation, the date of the same and the expiry date, as well as the exact concentration. Store in the fridge for 6 months.

TOTAL SOLIDS CONTROL SOLUTION

a) Dry potassium hydrogen phthalate (KHP) and sodium chloride (NaCl) at 105ºC during 2 hours.

b) Weigh 0.300g of KHP and 0.500g of NaCl and dissolve it in 1000,00 mL of distilled water.

Register the weight, the volume of water and the exact concentration of the total, fixed and volatile solids, expressed as mg/L, in the "Preparation of Control Solutions" form.

c) Calculate the concentration of the control solution expressed in mg/L, according to:

Total solids (mg/L) = Take NaCl (mg) + Take KHP (mg)

Fixed solids (mg/L) = Take NaCl (mg) + 0.23 * Take KHP (mg)

Volatile solids (mg/L) = 0.769 * Take KHP (mg)

d) Store the solution in a labelled plastic bottle, stating the name of the person responsible for the preparation, the date of the same and the expiry date, as well as the exact concentration. Store in the fridge for 6 months.

COD CONTROL SOLUTION

a) Dry potassium hydrogen phthalate (KHP) at 120ºC during 2 hours.

b) Weigh 0.400g of KHP and dissolve in 1000,00 mL of distilled water.

Register the weight, the volume of water and the exact concentration of the solution expressed as mgO2/L in the "Preparation of Control Solutions" form.

c) Calculate the concentration of the control solution expressed as mgO_2/L, according to:

COD mgO_2/L = Take KHP x F

F = 1186

d) Store the solution in a labelled plastic bottle, stating the name of the person responsible for the preparation, the date of the same and the expiry date, as well as the exact concentration. Store in the fridge for 4 months.

TOTAL PHOSPHORUS AND ORTHOPHOSPHATE CONTROL SOLUTION

For high-concentrations curve (1 to 4 mg P/L)

a) Dry potassium di-hydrogen phosphate (KH_2PO_4) at 105ºC during 1 hour.

b) Weigh 0.1500g of KH_2PO_4 and dissolve it in 1000,00 mL of distilled water. Dilute 2.5 mL of this solution in 500,00 mL of 0.3% sulphuric acid.

Register the weight, the volume of the water, the dilution and the exact concentration of the solution expressed as mgP/L in the "Preparation of Control Solutions" form.

c) Calculate the concentration of the control solution expressed as mgP/L, according to:

$$mgP/L = \frac{\text{Take } KH_2PO_4 \text{ (mg)}}{FD} \times \frac{31}{136}$$

$$FD = \frac{\text{Final volume (mL or g)}}{\text{Take of the solution (mL or g)}}$$

FW (KH_2PO_4)=136 FW (P) = 31

d) Pour the solution into a labelled yellow coloured glass bottle, indicating the name of the person responsible for the preparation, the date and expiry of the same, the exact date, the exact concentration and the limits of acceptance. Store in the fridge for 6 months.

For the low-concentration curve, (between 0 and 500 micrograms/L) dilute 10 times the concentration of the solution above. Make the dilution in the moment.

SILICATE CONTROL SOLUTION

a) In a 500.00mL graded bottle take the weight of 3.5mL of a silicate pattern solution of 1005μg/mL, from the instrumental section (commercial solution).

b) Take to volume.

Register the weight of the take, the final volume, the concentration of the initial pattern solution and the concentration of the control solution expressed as mgSiO_2/L, in the "Preparation of Control Solutions" form.

c) Calculate the concentration of the control solution expressed as mgSiO_2/L L, according to:

$$mgSiO_2/L = \frac{\text{Take standard solution (g)}}{\text{Final volume (g)}} * 1.005 \text{ (mg/mL)} * \frac{60}{28} * 1000$$

FW (Si) = 28 FW (SiO_2) = 60

d) Pour into a labelled plastic bottle, indicating the person responsible for the preparation, the date and expiry of the same and the exact concentration. Store at room temperature for 6 months.

SODIUM AND POTASSIUM CONTROL SOLUTION

Commercial solution used

Store the solution in a labelled bottle, indicating the exact concentration, the date and the expiry of the preparation. Store at room temperature during 12 months.

TOTAL HARDNESS – CALCIUM HARDNESS CONTROL SOLUTION

Solution to be used in titulometry

a) Mother solution: weigh 0.700 g ofde CaCO3 and 0.2487 g MgO dried at 180ºC for one hour. Dilute with the same amount of HNO3 1+1 and take to 1000mL in a graded bottle.

b) STOCK Solution: dilute 100 mL of the mother solution in a de 1000 mL graded bottle adding 10 mL of concentrated HNO3.

c) To control Total hardness and Calcium by titulometry use the STOCK solution.

d) To control Ca and Mg for FLAAS, dilute 1/10 at the time of the analysis of the above solution.

e) Register the weights of each take, the final volume, the concentration of the initial pattern solution and the final concentrations expressed as mgCaCO3/L, in the "Preparation of Control Solutions" form.

f) Calculate the total hardness and calcium hardness concentration in the control solution, according to the Mother solution:

$$\textbf{Ca}\ \text{mg/L} = \frac{\text{CaCO}_3\text{ (g) Weight}}{\text{Final volume (g)}} * \frac{40}{100} * 1000\ \text{(mg/L)}$$

$$\textbf{Mg}\ \text{mg/L} = \frac{\text{MgO (g)) Weight}}{\text{Final volume (g)}} * \frac{24}{100} * 1000\ \text{(mg/L)}$$

g) Concentration of the STOCK solution

$$\textbf{Ca}\ \text{mg/L} = \frac{\textbf{Ca}\text{ Mother Sol. weight mg/L} * \text{conc. Mother sol. (Ca)}}{\text{Final volume (g)}}$$

$$\textbf{Mg}\ \text{mg/L} = \frac{\textbf{Mg}\text{ Mother Sol. weight mg/L} * \text{conc. Mother sol. (Mg)}}{\text{Final volume (g)}}$$

h) Store the solution in a labelled plastic bottle, stating the name of the person responsible for the preparation, the date of the same and the expiry date, as well as the exact concentration. Store in the fridge for 12 months.

Solution to be used in FLAAS

Commercial solution used.

Store the solution in a labelled bottle, indicating the exact concentration, the date and the expiry of the preparation. Store at room temperature for 12 months.

BOD5 CONTROL SOLUTION

a) Weigh 75.0 mg of glucose and 75.0 mg of glutamic acid, previously dried at 105ºC during 2 hours. Place in a 500.00mL graded bottle and dissolve with distilled water.

b) Take to volume.

Register the weight of the take, the final volume, the concentration of the initial pattern solution and the concentration of the control solution expressed as mgSiO2/L, in the “Preparation of Control Solutions” form

c) Pour into a labelled plastic bottle, indicating the person responsible for the preparation, the date and expiry of the same and the exact concentration. Store at 4 ºC for 1 month.

PROCEDURE FOR THE DETERMINACION OF THE PRECISION AND THE CONTROL OF NORMALISED RANGES (PGC 05)

1. Aim

To determine the precision of the analytical procedures and to verify whether the precision of the analysis is under control.

2. Scope

This is a routine procedure to determine:

Natural waters and rainwater	Total solids; pH; conductivity, COD Suspended solids; alkalinity; silicate; nitrite; Hardness; calcium; magnesium; sodium; potassium; chloride; sulphur; nitrate; ammonium; sodium; potassium; orthophosphate and total phosphorus.
Industrial effluents	Total solids, pH; conductivity; Suspended solids; COD; Chromium; lead; copper; zinc; nickel; mercury; arsenic; cadmium, sulphur and ammonium.
Soils and sediments	Chromium, lead, zinc, copper, cadmium, nickel, mercury and arsenic.

3. Reference

See the following definitions in the glossary of basic terms and concepts:

- Relative dispersion
- Random error
- Control graphs
- Control limits
- Precision
- Range and Standardised Range

4. Summary of the method

The precision of the method is determined by the relative standard deviation of the analyses. The relative standard deviation is calculated based upon the normalised ranges. The samples are analysed in duplicate and with the result of at least 20 duplicates, the average of the normalised ranges and the standard deviation of the same are obtained.

The average of the normalised ranges divided by a 1.128 factor is the relative standard deviation of the method.

The control of the precision is made through a control graph of normalised ranges.

5. Procedure

5.1 Control of the precision of the method:

1. Collect at least the 20 duplicate analyses of the parameter of interest made by the same analytical procedure.
2. Determine the range and the Standardised Range for each duplicate:

   $R = ABS (D1 – D2)$ $\qquad R_{norm} = \frac{R * 100 * 2}{(D1+D2)}$

   D1 and D2 are the values of the duplicate, ABS is the absolute value of the difference and R_{norm} is the Standardised Range
3. Calculate the average (M) and the standard deviation (s) of the normalised ranges.
4. Calculate the central line (CL) and the higher limits(HIL) and threshold (TL) for the control graph.

   $CL = M \qquad HIL = M + 3s \qquad TL = M - 3s$
5. Build the control graph for the precision with the values of the normalised ranges. Verify that all the points are within the control limits, otherwise eliminate the point/s which are out of control and recalculate the control limits and the average.
6. Keep the control graph following the schedule indicated in Annex I. Verify that the Standardised Range of the following analyses is within control or the limits of acceptance for the Standardised Range established for this parameter.

   If the duplicates are out of control, check the calculations and unless it is otherwise indicated in the operative standardised procedure, if the error in the calculations is disregarded, repeat the analysis of the sample which is out of control (for the cases in which all of them are analysed in duplicate) or the set of samples (for the case of those in which a duplicate is analysed every several samples).

5.2 Determination of the precision of the method:

The average of at least 20 normalised ranges of duplicates is calculated and the precision is determined as a percentage according to:

$$P , \% = \frac{RSD = Mean_{\text{normalised ranges}}}{1.128}$$

RSD is the relative standard deviation

1.128 is the conversion factor to calculate the standard deviation(s) from the range.

6. Bibliography

1- AMERICANN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

PROCEDURE FOR THE DETERMINATION AND CONTROL OF THE PRECISION FOR BOD_5 ANALYSIS (PGC 06)

1. Aim

To determine the precision of the analytical procedure and to verify whether the precision of the analysis is under control.

2. Scope

This procedure is used to determine the precision of the BOD_5 analysis and to verify whether the precision of the analysis is under control.

3. Reference

See the following definitions in the glossary of basic terms and concepts:

- Relative dispersion
- Random error
- Control graphs
- Control limits
- Precision

4. Summary of the method

The BOD_5 analysis is made by incubating at least four different dilutions of the samples during 5 days. The slope of the dissolved oxygen curve in relation with the dilution of the sample is directly proportional to the BOD_5. The precision of the analysis is estimated as the error of the slope. This parameter is the S_y of the linear regression.

The control of the precision is followed with a control graph of errors in the slope of the BOD_5 determination curve.

5. Procedure:

5.1 Control of the precision of the method:

a) Carry out the linear regression analyses for the BOD_5 determination curve. From this analysis, take the value of the slope and the standard deviation of the slope called error in the Y axis: s_y

b) Calculate the relative standard deviation of BOD_5 as:

$$RSD = s_y \times slope \times 100$$

c) Collect at least the RSD of the last 20 BOD_5 analyses.

d) Calculate the average (M) and the standard deviation (s) of the RSD. Calculate the central line and the higher limits and the threshold for the control graph.

$$CL = M \qquad HIL = M + 3s \qquad TL = M - 3s$$

e) Build the precision control graph with the RSD values. Verify that all the points are within the control limits, otherwise eliminate the point or points which are out of control and re-calculate the average and control limits.

f) Keep the control graph according to the according to the schedule indicated in Annex I. Verify that the RSD of the following analyses are in control. Otherwise:

- If the concentration is lower than the BOD_5 quantification limit, the analysis must not be repeated and it is informed as being under the quantification limit

5.2 Determination of the precision of the method:

The average of at least 20 RSD of BOD_5 analyses is calculated and the precision percentage is calculated according to:

$$P\ ,\ \% = RSD_{Mean}$$

6. Bibliography

1- AMERICAN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

QUALITY CONTROL PROCEDURE FOR BACTERIOLOGICAL ANALYSES WITH FILTERING MEMBRANE (PGC 07)

1. Aim

The aim of this procedure is to minimise and identify the experimental errors, due to assignable causes, and the random errors' analytical process. In this way it is possible to obtain more precise, higher quality and more reliable results.

2. Scope

This procedure is applied to all the bacteriological analyses of environmental samples made with the filtering membrane technique.

3. References

Standardised Operation Procedure for the Determination of Thermotolerant Cs. (SOP 53)

Standardised Operation Procedure for the Determination of Total Coliforms (SOP 54)

Standardised Operation Procedure for the Determination of Faecal Streptococcus (SOP 56)

Standardised Operation Procedure for the Determination of Enterococcus (SOP 58)

Procedure for the Verification of Thermotolerant Coliforms (SOP 72)

Procedure for the Verification of Total Coliforms (SOP 71)

Procedure for the Verification of Faecal Streptococcus (SOP 70)

4. Summary

The measures always entail a certain uncertainty. If independent determinations of a certain experimental quantity are carried out, the values obtained are not identical, even under the same experimental conditions. The error may have three origins: mistakes, systematic errors and random or accidental errors, distributed at random.

On principle, both, the mistakes as well as the systematic errors may be eliminated through careful experimentation, but random errors persist even when the other two have been eliminated.

5. Procedure

The analyst must dedicate 15% of his total working time to quality control procedures.

5.1 Quality of the analytical process

5.1.1 Control of the sterility of the system

Check the effectiveness of the autoclave's sterilisation once a month, using a spore suspension of *Bacillus stearothermophilus* (biological indicator). In order to do so, sterilise normally at 121ºC, for 15 min. Then incubate at 56 ± 2ºC in a FANEM water bath (MB017) for 48 hours, confirming the absence of spores if there is no colour change (the means remains purple). A change in the colour to yellow indicates that the sterilisation procedure has failed. Register the results obtained 12 and 24 hours later. The final negative result is obtained after 48 hours of incubation.

Every time samples are analysed, the sterility of the materials used should be controlled, filtering 100mL of sterile water and incubating the plaque with the filter under the same conditions the samples are incubated, at the beginning of the determination.

Every time samples are analysed, the incubation conditions should be checked (temperature control).

5.1.2 Pure cultures

Control the means of culture, using pure cultures of one of the groups of bacteria detailed below.

	CONTROL CULTURES	
GROUP	**POSITIVE**	**NEGATIVE**
TOTAL COLIFORMS	*Escherichia coli* *Enterobacter aerogenes*	*Pseudomona sp.* *Staphylococcus aureus*
FAECAL COLIFORMS	*Escherichia coli*	*Enterobacter aerogenes* *Streptococcus faecalis*
STREPTOCOCCUS	*Streptococcus faecalis*	*Staphylococcus aureus* *Escherichia coli*

This procedures is carried out under flow chamber. Over a sterile filter placed on a plaque with the corresponding means of culture, sow pure bacterial cultures by points or lines which shall develop typical and non-typical colonies in the means of culture used. These plaques are incubated at the same time and under the same conditions as the processed samples. In case the result should be different than expected, check the feasibility and the purity of the culture, the preparation of the means of culture and the incubation temperature. If all the parameters are correct, the set of analyses is rejected.

Preparation of pure cultures. Preparation of pure work cultures and mother cultures for preservation.

Pure cultures of *E. coli*

There are two possible procedures to obtain pure cultures of *E. coli*:

a) To isolate typical colonies of Aquachek samples, count the *E.coli* on petri plaques with Triptych Soy Agar and incubate them at 35 ± 0.5 ºC for 24 hours.

b) Isolate typical colonies of Total Coliform plaques on petri plaques with a TSA means, incubate at 35 ± 0.5 ºC for 24 hours. From the growth obtained, run the Enterobacteria biochemical tests with the API 20E following the instructions and the procedure established therein to identify the gender and the species. In case the API 20E is not available, run the verification biochemical tests described in the verification procedures (PGC).

Whichever the method used to obtain the *E. Coli* colonies, once they have been identified, make several working replicas of the colony in glass tubes with a screw cap, with an inclined TSA. Incubate at 35 ± 0.5 ºC for 24h. and keep in refrigerator to be used in the routine controls. Label.

Separately, from that same isolated colony, make replicas in test tubes with TSB, incubate in the same conditions and for the same period as above. Check the purity of the culture with the microscope.

Then proceed to place an adequate volume in sterile eppendorff tubes with sterile glycerol, attaining a final concentration of 20%. Label.

Keep in the freezer at – 20 ºC for one year maximum as mother cultures.

In case the laboratory should run out of working culture or should it be contaminated, recur to a mother culture, defrost it to room temperature and sow by lines on plaques with TSA, incubate at 35 ºC for 24 hours. Check its purity and proceed to make working cultures from it.

Pure cultures of *Streptococcus faecalis*

There are two possible procedures to obtain pure cultures of *Streptococcus faecalis*:

a) Isolating typical colonies of Aquachek samples and counting the *Streptococcus faecalis* therein on petri plaques with TSA and incubating them at 35ºC for 4 hours.

b) Isolating typical colonies of *Streptococcus faecalis* on petri plaques with TSA, incubating them 35 ± 0.5 ºC for 24 hours. Making the necessary biochemical tests from the growth obtained, following the Bergey Manual to identify them.

Whichever the method used to obtain the colonies, once they are identified, make several work replicas of the colony in glass tubes with a screw cap with inclined TSA. Incubate at 35 ± 0.5 ºC for 24h. and store in refrigerator for use in routine controls.

Apart from this, make replicas of this same isolated colony in test tubes with TSB, incubate under the same conditions and for the same period of time as above. Check the purity of the culture with the microscope.

Then proceed to pour an adequate volume in sterile eppendorff tubes with sterile glycerol, at a final concentration of 20%.

Store in freezer at – 20 ºC for one year maximum as mother cultures.

In case the laboratory should run out of working culture or should it be contaminated, recur to a mother culture, defrost it to room temperature and sow by lines on plaques with TSA, incubate at 35 ºC for 24 hours. Check its purity and proceed to make working cultures from it.

5.1.3 Verification of isolated colonies

Verify 5% of the samples analysed in the section every month, isolating 10 colonies from each environmental sample. If the sample analysed is water for human consumption, verify the total colonies grown on the plaques.

5.2 Precision criterion

5.2.1. Calculation

In order to establish whether the filtering membrane procedure, as well as the colony count are statistically acceptable, the precision criterion must be calculated.

The precision criterion is established through the statistical analysis of a group of 15 environmental samples in duplicate. The precision value obtained is used to check the analysts' precision every time samples are analysed in the section with the filtering membrane procedure. The precision criterion must be calculated for each environmental matrix (beach water, well water, industries, etc) and for each parameter developed in the section (Total Coliforms, Thermotolerant Coliforms, etc.).

In order to establish the section's precision criterion, it is necessary to have 15 samples made in duplicate (D_1 y D_2). The count for each duplicate must be carried out by the same analyst, but in order to establish the precision criterion, all the analysts in the section must be included.

In order to make the statistical analysis of the data corresponding to that group of 15 samples, first the logarithms (L_1 y L_2) of the counts corresponding to each sample (D_1 y D_2) are made. In case the count is zero, one unit is added to the two values obtained.

After this, the difference between each pair of logarithms (L_1-L_2) is calculated and then the arithmetic measure of those differences (R). The precision criterion shall be the product of the 3,27 coefficient times the value obtained from R.

$\sum R_{log}$ = sum of the differences of the logarithms of the counts.

n = number of samples

R = arithmetic average of the logarithms of the counts, $R = \frac{\sum R_{log}}{n}$

Precision criterion = 3,27 X R

Example of determination of the precision criterion:

Nº Analyses	Duplicates		Log duplicates		Log. range (R_{log})
	D_1	D_2	L_1	L_2	$L_1 - L_2$
1	50	52	1.6989	1.7170	0.019
2	99	90	1.9956	1.9542	0.041
3	45	48	1.6532	1.6812	0.028
4	15	19	1.1761	1.2788	0.103

5	47	49	1.6721	1.6902	0.018
.	.	.	.	.	.
15	8	5	0.9030	0.6990	0.204

$\sum R_{log} = 0.019 + 0.041 + 0.028 + 0.103 + 0.018 + \ldots + 0.204 = 0.413$

$R = \frac{0.413}{15} = 0.028$

Precision criterion = 3.27 x 0.028 = **0.090**

5.2.2. Routine precision check

Once the precision criterion has been established, analyse 10% of the samples which enter the section in duplicate and calculate the logarithm of each couple of data. The difference between the logarithms of each couple of duplicates must be compared to the precision criterion established. If said difference is higher than the precision criterion, la variability of the analysis is excessive; thus the problem must be identified and solved before new determinations are made.

If the difference between the logarithms is higher than the precision criterion, this means that the analyst's precision is good and the procedure and its result are classified as acceptable.

Example:

Analysis Date	Duplicates		Log duplicates		R_{log}	Acceptance of the range
	D_1	D_2	L_1	L_2	$L_1 - L_2$	
29/8	50	52	1.6990	1.7160	0.017	Acceptable
30/8	91	90	1.9590	1.9542	0.005	Acceptable
31/8	48	20	1.6812	1.3010	0.382	Not Acceptable

Precision criterion: 0.090

The precision criterion must be updated every time there is a new set of 15 samples processed in duplicate.

5.3 Reproducibility

Check of the count among the analysts: the analysts must count their plaques reciprocally every month, the highest standard deviation acceptable being 10%.

5.4 Repeatability

Checking the count made by an analyst: every month, each analyst must count the same plaque twice, accepting a maximum Standard deviation of 5%.

6. Bibliography

American Public Health Association. Standard methods for the examination of water and wastewater, 18 ed, APHA, AWWA, WPCF, 1992.

Avaliaçao of theboratories de analises bacteriologicos de water, Sao Paulo, CETESB, 1989, Norma técnica L5.010.

EPA Microbiological Manual. 1978. Part IV. Quality Assurance, Section C, Analytic Quality Control Procedures, pp 231-232.

PROCEDURE FOR THE CONTROL OF ANALYSIS WHITES (PGC 08)

1. Aim

To control the whites of analysis reagents in order to be able to detect systematic errors.

2. Scope

This procedure is made to control the dilution water used for the BOD_5 analyses.

It may be used for whites of any physical-chemical analysis methodology.

3. Reference

See the following definitions in the glossary of basic terms and concepts:

- Standard deviation
- Systematic error
- Control graphs
- Control limits

4. Summary of the method

The value of the concentration of the white or the readings of the white in the measurement equipment for each analysis run, by means of a control graph.

If the reading on the measurement equipment or the concentration of the white exceed the limits, we assume that we have come upon one of the errors referred to in the aim. In this case, the cause must be studied and the analytical responsible of the technique must register the decision made and the justification thereof in writing.

5. Procedure

5.1 Control of the concentration of the white:

1. Calculate the concentration of the white. After obtaining at least 20 values of the concentration of the white, calculate the average of the values (C_{Bco}) and the standard deviation (s).
2. Determine the central line and the higher limits and thresholds for the control graph, according to:

$$CL = C_{Bco} \qquad HIL = C_{Bco} + 3s \qquad TL = C_{Bco} - 3s$$

5. 3. Build the control graph of the concentrations. Verify that all the points are within the control limits, otherwise eliminate the point or points which are out of control and re-calculate the average and the control limits.

4. Keep the control graph with the concentration corresponding to the white every day the analysis is run, and verify if the concentration is within the limits. Otherwise, repeat the

analysis of the white, and if it is out of control again, study the cause to take the necessary corrective action.

5.2 Control of the reading of the white:

1. Calculate the mean of the values (B) and the standard deviation (s) with at least 20 readings of the white.
2. Determine the central line and the higher limits and thresholds for the control graph, according to:

   CL = B HIL = B + 3s TL = B - 3s

3. Build the control graph of the readings. Verify that all the points are within the control limits, otherwise eliminate the point or points which are out of control and re-calculate the average and the control limits.
4. Keep the control graph according to the schedule on annex I with the reading of the white and verify if the concentration is within the limits. Otherwise, repeat the analysis of the white and if it is out of control again, study the cause to take the necessary corrective action.

6. Bibliography

1- AMERICANN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater APHA, 1995. p1-1 a p1-11.

PROCEDURE TO DETERMINE THE DETECTION LIMIT AND THE PRACTICAL QUANTIFICATION OF THE METHOD (PGC 09)

1. Aim

To know the detection and quantification limits of the analytical determinations carried out in the department.

2. Scope

This procedure is used for the determination of the detection and quantification limits of the physical-chemical methods used by the Laboratory.

3. Definitions

The method's detection limit (MDL) is defined as the minimum concentration of analyte that may be identified, measured and reported with a 95% reliability that the concentration of the analyte is higher than zero and determined from the analysis of the sample in a matrix containing analyte.

The quantification limit (QL) is defined as 5 times the method's detection limit (MDL).

4. Summary

A statistic student test is applied to at least 7 replicas of a matrix of the sample containing the analyte in a concentration of 1 to 5 times the estimated detection limit.

The solution then undergoes all the steps of the analytical procedure to which the sample is subjected.

5. Procedure

5.1 Estimate the MDL as one of the following:

a) Concentration value which corresponds to a signal/noise of the instrument relation of 2.5-5.

b) Concentration value which corresponds to 3 times the standard deviation of the measurements of replicas for the analyte in water.

c) Concentration value where the calibration curve has a significant change in sensitivity in low concentrations (break in the slope of the curve).

d) Known limitations of the equipment.

5.2 Prepare reagent water free of analyte and interferences (non-detectable in the method).

5.3 If the MDL is determined in reagent water: prepare a concentration standard 1 to 5 times the estimated MDL.

5.4 If the MDL is determined in a sample matrix, the concentration of the analyte in the sample may be:

a) 1 to 5 times the estimated MDL; in this case, continue in N° 5.

b) < estimated MDL, then the sample is strengthened to 1-5 times the estimated MDL and continues in 5.

c) > estimated MDL and < 10 times the MDL continue in number 5, but bear in mind that the variation increases with the concentration.

d) > 10 estimated MDL: the sample cannot be used.

5.5 Take at least 7 aliquots of the sample to be used to calculate the MDL and apply the analytical procedure to each one. If the measure of the white is required to calculate the concentration of the analyte, they must be obtained for each aliquot. The average of the measure of the whites is subtracted from the measure of each aliquot of the sample.

6. Calculations

6.1 Determine the standard deviation (S) of the measures of the replicas

6.2 Determine the detection limit according to:

$$MDL = t_{(n-1,1\ ,\ldots,\ 99)}\ (S)$$

Where $t_{(n-1,1\ ,\ldots,\ 99)}$ is the value of the student test for a 99% reliability level and (n-1) degrees of freedom. (see table below)

6.3 Determine the practical quantification limit according to:

$$LQ = 5 * MDL$$

Table of Values of the Student Test at a 99 % Reliability Level

Nº of replicas	Degrees of freedom	T
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602

7. Bibliography

1- AMERICANN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

QUALITY CONTROL PROCEDURE FOR THE DETERMINATION OF THE ACUTE TOXICITY THROUGH THE MICROTOX® SYSTEM (PGC 10)

1. Aim

The aim of this procedure is to ensure the validity of the analyses every time acute toxicity of an environmental sample is determined through the Microtox® system. In this aim, reference toxics which provide a general measure of the precision of an analytical measure are used.

2. Scope

This procedure is applicable to all acute toxicity analyses of environmental samples through the Microtox® system.

3. References

3.1. Standardised Operation Procedure for the Determination of Acute toxicity in Liquid Phase using *Vibrio fischeri.* Microtox System®-Basic Test (SOP 59)

3.2. Standardised Operation Procedure for the Determination of Acute toxicity in Liquid Phase using *Vibrio fischeri.* Microtox System®-Whole Effluent Toxicity Test (SOP 69).

4. Summary of the method

In order to ensure the validity of the analyses and to test the sensitivity of the bacteria used, every time a bacterial reagent phial is reconstructed, the Basic Test protocol is carried out, using a 100 mg/l phenol solution as the toxic of reference. The IC_{50}(5 min.) of the same must be between 13 and 26 mg/L. The value obtained is then compared to the previously established phenol control graph, to identify whether said value is within an acceptable variation range. Zinc sulphur may also be used as standard (ZnSO4.7H2O). In such case, the IC_{50}(15 min.) must be between 5 and 12 mg/L.

5. Procedure

5.1 Preparation of the standard phenol:

- Weigh 50 mg of crystalline phenol and place it in a 500 ml. amber glass container. If this kind of container is not available, cover the container used with aluminium foil to protect the phenol standard from photo degradation.
- Add 500 ml of diluent into the container.
- Mix the content by turning the container upside-down several times.
- Label properly and store between 2 and 8°C for 3 to 4 months.

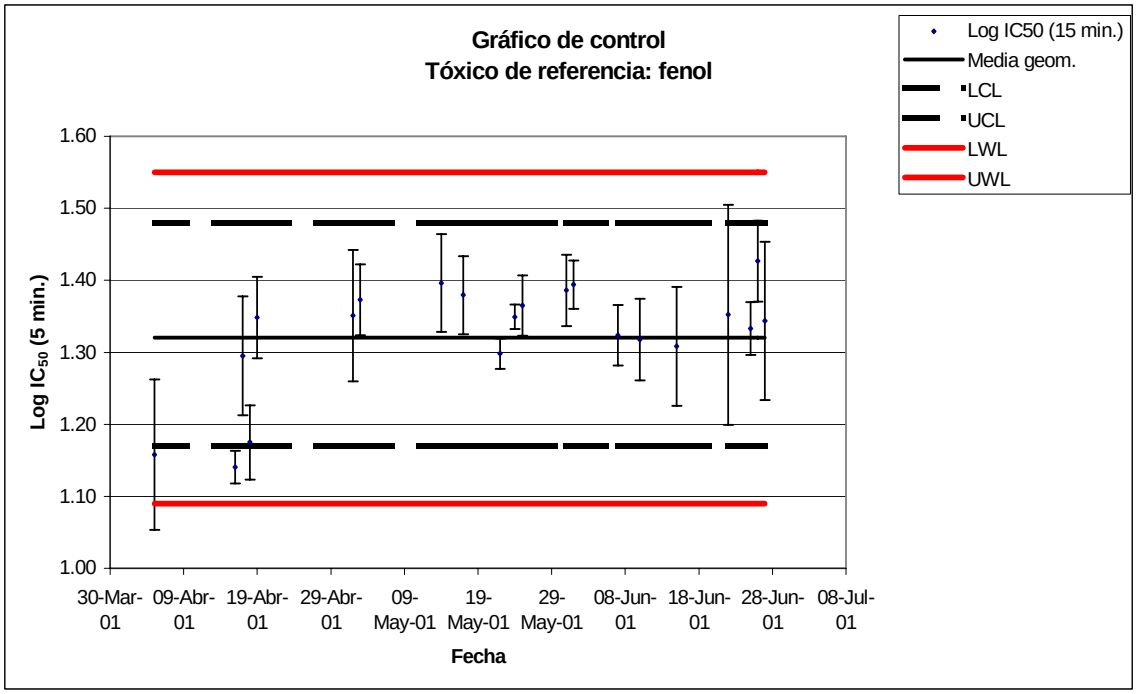
5.2. Preparation of the zinc sulphur standard (ZnSO4.7H2O)

- Weigh 50 mg of ZnSO4.7H2O and place it in a 500 ml. amber glass container. If this kind of container is not available, cover the container used with aluminium foil to protect the phenol standard from photo degradation.
- Add 500 ml of diluent into the container.
- Mix the content by turning the container upside-down several times.
- Label properly and store between 2 and 8°C for 3 to 4 months.

5.3. Building the phenol control graph:

Phenol (or zinc sulphur) control graphs, are built from the results obtained from a successive series of analyses (it is necessary to have 20 IC_{50} pieces of data using the Basic Test protocol). The X-axis represents the date of the analysis, and the Y-axis represents the IC_{50}(5 min) logarithmic values. The geometrical average and the standard deviation of the logarithm decimal of the IC_{50}(5 min.) values obtained with the phenol standard, are used to define the acceptable range for the test's variability. The sum of the geometrical average plus two standard deviations is the higher control limit which represents a 95% reliable interval (UCL). The sum of the average plus three standard deviations is called higher warning limit and it represents a 99% reliable interval. (UWL). Likewise, control Thresholds are taken (LCL) as well as warning thresholds (LWL), subtracting the deviations from the standard from the mean.

The following is an example of a phenol control graph:



Geometrical measure	1.32

Standard deviation (SD)	0.08
2SD	0.16
3SD	0.23
UCL (Mean + 2SD)	1.48
LCL (Mean - 2SD)	1.17
UWL (Mean + 3SD)	1.55
LWL (Mean - 3SD)	1.09
%CV	5.87

Every time 20 new IC_{50}(5min) values of the phenol standard are available, a new phenol control graph must be established.

When establishing the first control graph, it is important to bear in mind that the IC50 data with a wide 95% reliable interval determine great standard deviations in relation to the average, causing the control limits to be too broad. It is thus suggested that the data used in the control graph do not present a variation coefficient higher than 30%. This is determined in the following way:

$$\% = ((IC_{50} / \min.IC_{50}) - 1)\ (2)\ (100)$$

Example:

IC_{50} (mg/l)	Min. IC_{50}	Max. IC_{50}	%	
17.50	16.32	18.72	14.4	Acceptable
3.93	3.46	4.48	27.6	Acceptable
1.64	1.35	2.00	43.8	Unacceptable

6. Routine check of the quality of the data

Every time the acute toxicity of environmental samples is analysed through the Microtox System, a test using the Basic Test protocol with the phenol standard must be launched. The IC_{50}(5min.) value obtained with the phenol standard must be between 13 and 26 mg/L.

More precisely, said value must be checked against the IC_{50}(5min.) value established by SDI in the performance certificate issued for every lot of bacteria produced.

Apart from this, the IC_{50}(5min.) value obtained must be compared to the previously established phenol control graph, in order to identify whether said value is within an acceptable variability range fulfilling the following requirements:

In the 95% reliability interval, 1 out of 20 analyses (5%) is expected to be out of the control limits. Thus, the IC_{50} logarithm of the samples of the phenol standard should not exceed the control range more than 1 time in 20. likewise, the IC_{50} logarithm should not exceed the warning range more than once in 100 times, which is the expected behaviour for the 99% reliability range.

If the IC_{50} (5 min) values of the phenol standard are within the control and warning limits, the laboratory is generating consistent results.

When the IC_{50} (5 min) values of the phenol standard are beyond the warning range, the system must be revised completely. The most common sources of errors are due to operation: adjustment of salinity, dilution of the sample, reconstitution of the bacteria phial, pipeting errors, homogenisation of the solutions. In such cases the test must be repeated.

7. Bibliography

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PROCEDURE TO CHECK THE CONSISTENCY OF THE ANALYTICAL RESULTS (PGC 11)

1. Aim

This procedure is used to detect inconsistencies in the results of the analyses.

2. Scope

It is applied to control the quality of the analytical results in water and industrial effluent samples, after all the analyses requested have been made.

3. Procedure

Prior to issuing a report, the person responsible for quality or the chief must check the following correlations:

Industrial effluents analyses**:**

Checking correlation	Correct relation	Observations
For organic matter:		
COD/BOD_5	> 1	The biodegradable organic matter is a fraction of the total
Volatile solids/COD	< 1	This may not be the case when there is a high concentration of carbonates which are volatilised together with the organic matter.
(Fats and oils) / volatile solids	< 1	This may not be the case when there is a high concentration of carbonates which are volatilised together with the organic matter.
Solids:		
(Total dissolved solids) / Conductivity	0.55 - 0.70	Conductivity expressed in µS/cm
Total solids / Susp. Solids	> 1	This relationship must be the same for total, volatile and fixed solids at the same time.

Natural waters

3.1 Ionic balance:

The water must be electrically neutral, thus the amount of anions and cations must be equivalent. Since the only ones which are determined are the most frequent ions, a percentage of the difference may be accepted, due to their analysis errors and to the ions which are not analysed.

The controlled relationship is the following:

$$\text{Ionic balance} = \frac{(\sum \text{cations} - \sum \text{anions}) * 100}{(\sum \text{cations} + \sum \text{anions})}$$

Where anions and cations must be expressed in meq/L.

Up to 5% is acceptable for natural waters. In case of higher balances, each analysis must be revised in search for possible errors; repeat the dubious parameter(s) and in the event the % should be higher than 15% analyse the sample again. The ionic balance of the waters is registered in the water-result notebook.

Table used to convert the concentrations of the determined ions into meq/L.

Ion	**Units**	**Conversion factor FC** **$meq/L_{ion} = Conc_{ion}/FC$**
Calcium	mg/L	20.04
	mg $CaCO_3$/L	50.00
Magnesium	mg/L	12.15
	mg $CaCO_3$/L	50.00
Sodium	mg/L	22.99
Potassium	mg/L	39.1
Ammonium	mg/L	18.04
	mg N /L	14.00
Chloride	mg/L	34.46
Sulphur	mg/L	48.03
Alkalinity	mg $CaCO_3$/L	50.00
Nitrate	mg/L	64.01
	mg SiO_2 /L	14.01
Silicate	mg/L	30.04

3.2 Other correlations to be considered:

Checking correlation	Correct relation	Remarks
$\frac{TDS_{measured}}{TDS_{calculated}}$	1.0 - 1.2	The TDS calculated are the mass sum of the majoritary inorganic ions analysed as: TDS = Cl + SO_4 + NO_3 + alkal. + Ca + Mg + Na + K + SiO_2
$\frac{\text{Total solids}}{\text{Suspended solids}}$	> 1	This relationship must be the same for total, volatile and fixed solids at the same time.

Note: TDS* - Total dissolved solids

4. Bibliography

1- AMERICAN PUBLIC HEALTH ASSOCIATION. Standard Methods for the Examination of Water and Wastewater, APHA, 1995. p1-1 a p1-11.

ANNEX I: ANALYTICAL QUALITY CONTROL FOR EACH ANALYTICAL PARAMETER

The following table shows the controls made for each analysis and the maintenance frequency for the control graphs.

PARAMETER	Accuracy Control	Precision control	Frequency
PH	Inter-laboratory tests	Normalised ranges	Half-yearly
Conductivity	Inter-laboratory tests	Normalised ranges	Half-yearly
BOD_5	• Known concentration solution • White control • Inter-laboratory tests	Error en the slope of the curve	Quarterly
COD	• Known concentration solution • recovery % • Inter-laboratory tests	Normalised ranges	Quarterly
Total solids	Known concentration solution	Normalised ranges	Quarterly
Suspended solids	Inter-laboratory tests	Normalised ranges	Quarterly
Fats and oils	recovery %	Not controlled	Yearly
Inorganic anions F^- - Cl^- - SO_4 - NO_3	• Known concentration solution • Inter-laboratory tests	Normalised ranges	Half-yearly
Alkalinity	Inter-laboratory tests	Normalised ranges	Yearly
Silicate	• Known concentration solution • Inter-laboratory tests	Normalised ranges	Yearly
Sulphur	recovery %	Standardised Range	Yearly
PARAMETER	**Accuracy Control**	**Precision control**	**Frequency**
Nutrient metals Ca-Mg-Na-K	• Known concentration solution • Inter-laboratory tests	Normalised ranges	Half-yearly
Toxic metals	• Known concentration solution • recovery % • Inter-laboratory tests	Normalised ranges	Half-yearly

Total cyanide	• recovery % • Inter-laboratory tests	Normalised ranges	Yearly
Ammonium - Nitrate	• Known concentration solution • Inter-laboratory tests	Half-yearly	Half-yearly
Phosphorus T. – Orthophosphate	• Known concentration solution • Inter-laboratory tests	Normalised ranges	Yearly
Microbiology by filtering membrane	• Inter-laboratory tests	Precision criterion	

FREQUENCY	SCHEDULE
Quarterly	March - July – October – December
Half-yearly	July – December
Yearly	March

ANNEX II: PLANNING, EVALUATION AND REGISTER OF THE PARTICIPATION IN INTER-LABORATORY TESTS

1. Planning:

The participation in inter-laboratory tests is planned in the first quarter of the year, depending on the offers available. The plan is made and filed in the “Inter-laboratory tests” folder. Once the test is finished, the results are filed in that same folder.

2. Analyses:

The person responsible for quality control and/or the laboratory chief is in charge fo communicating the arrival of the samples to the corresponding analysts, collecting the results and sending them to the organisers of the test.

The analysis routes of the tests are filed together with the analysis routes of the samples, by parameter.

A copy of the results sent is kept in the “Inter-laboratory tests” file, with the name of the analyst in charge.

3. Test Evaluation:

When the test results are in, the responsible analysts are informed thereof by means of a copy.

For those analyses the results of which were beyond the acceptable limits, the cause is researched and the cause and the corrective action to be taken are registered on the result sheet. The person responsible for the analysis is informed of the same and he acknowledges his reception of the same by signing a register.

Version	Date	Amendments
1	2002	First edition
2	Sept. 2004	Second edition

REVISED	DATE
APPROVED	DATE