

APPENDIX A.

**SAMPLING METHODOLOGY**



**ASSESSMENT OF ENVIRONMENTAL IMPACT  
AND INDUSTRIAL SAFETY IN  
DEVELOPMENT WELLS  
PALAGUA – CAIPAL FIELD  
PUERTO BOYACA (BOYACA, COLOMBIA)**



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# SAMPLING METHODOLOGY

## 1. SAMPLING METHODOLOGY FOR AIR

The field work was carried out according to the requirements of the environmental competent authority, Decrees 948/95 and 02/82 and Resolution Number 601 of April 4, 2006.

The team set base camp operations in the municipality of Puerto Boyacá, Boyacá. Appropriate sampling locations were selected. This allowed them to coordinate all the logistics and properly prepare for sampling. High Vol, PM10 and Gas Pumps were calibrated. During the sampling, the initial and final weight of the filters was recorded, as well as the initial and final flow of gas for the collection of NO<sub>x</sub>, SO<sub>x</sub> and O<sub>3</sub>.

### 1.1 LOCATION OF THE EQUIPMENT

To achieve a representative measurement of pollutant levels (Particulate matter, Sulphur oxides, Nitrogen oxides, and Carbon monoxides), the following location criteria were considered:

- Far from high vehicular traffic routes and / or pathways that are not paved.
- To a distance no less than 20 meters of vegetation or trees in the sampling area.
- A radius of 270 ° unrestricted flow around the sampling equipment.

The sampling was held for ten continuous days in each of the three stations: gas, TSP, and PM10. Each site consisted of gas equipment for the collection of nitrogen oxides and sulfur dioxide. The sample collection was performed at intervals of 24 hours in all cases.

The locations were monitored in the production area of this field; alternate paths are uncovered and have no vehicle traffic. A digital infrared non-dispersive scattering instrument was used for carbon monoxide measurements.

### 1.2 SUSPENDED PARTICULATE MATTER METHODOLOGY

The method involves using a sample of air which sucked into an engine through a quartz filter which has been previously dried and weighed. Air passes for 24 hours at a flow rate between 40 and 60 ft<sup>3</sup>/min. After the sampling time, the filter is dried at 85 ° C for one hour in a CaCO<sub>3</sub> oven, and weighed again. The weight difference is the net mass (in micrograms) divided by the volume of air that passed within 24 hours of sample (m<sup>3</sup>), determines the concentration of suspended particles (TSP Total Suspended Particles) in µg/m<sup>3</sup>.

The geometry of the High Volume sampler allows particles to be classified by an Aerodynamic separator and then collected on a quartz filter for quantification and analysis before reaching the horizontal filter, depositing particles there which have not settled via gravity. The sampling time (typically 24 hours) and the air flow passing through the filter are set by a timer during equipment calibration. The instruments control the flow sampled by weight or by volume.

### 1.3 COARSE PARTICLE SAMPLING PM10 AND HIGH VOLUME INSTRUMENTS

Three sampling instruments are 2000H High Vol Grasseby, consisting of a metal structure with an anodized aluminum hood were used (see Figure 1). It had a filter holder fitted to a metal or plastic casing which contained a motor at 1800 rpm, 110-115 volts, 745 watts and 6.25 amps, with a flow rate between 20 and 60 cubic feet per minute. The engine hooked to a timer and a flow meter or rotameter graduated from 0 to 70 ft<sup>3</sup>/min.

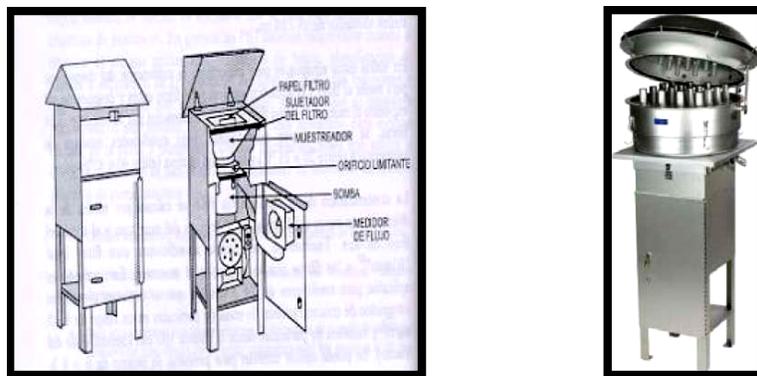


Figure 1 High Vol

#### SPANISH TO ENGLISH TRANSLATION KEY

PAPEL FILTRO	FILTER PAPER
SUJETADOR DEL FILTRO	FILTER HOLDER
MUESTREADOR	SAMPLER
ORIFICIO LIMITANTE	RESTRICTION ORIFICE
BOMBA	PUMP
MEDIDOR DE FLUJO	FLOW METER

#### 1.3.1 Equipment calibration

For the High Vol samplers calibration a GMW 25 calibration unit was used which is made up of a hole pattern with its respective calibration curve, a five-disc set with 5, 7, 10, 13, and 18 holes, respectively, to simulate different stages of clogging of a standard cellulose filter. It also has a water manometer and an adjustable base to adapt the hole and the respective discs. The calibration procedure relates the flow in ft<sup>3</sup>/min, with the differential pressure in inches of H<sub>2</sub>O for each of the standard disks. The following is a summary of the procedures:



- Place a clean quartz fiber filter on the filter holder and start the computer for 5 minutes. Stop the engine and assemble the calibration unit with the No. 5 disc. Turn on the computer for 5 minutes. Record the reading of the rota meter and the pressure drop in the gauge. Again, stop the engine. Repeat the steps above for disks 7, 10, 13 and 18. Once you know the flow at standard conditions for 29.6 inches of H<sub>2</sub>O and 68 ° F, adjust work flows to local conditions.
- It is important to note that the procedure for calibration of equipment was conducted in accordance with EPA regulations, which are, with the calibration approach of a maximum error of 5% between the values driven by the flow meter equipment and the calculated from the calibration certificate, considering the standard flow (Q<sub>std</sub>) and the flow rate (Q<sub>real</sub>). The equipment was calibrated with a gas bubble meter; obtained flows are thus presented in the result tables, ranging from 0.180 L / min and 0.220 L / min., according to the criteria stated in U.S. EPA Air Quality Criteria.

#### **1.4 NITROGEN OXIDES (NOX).**

##### **1.4.1 Triethanolamine Method.**

Nitrogen dioxide NO<sub>2</sub> is absorbed from the air sample by an aqueous solution of triethanolamine and n-butanol. The collected NO<sub>2</sub> is derived to an azo-compound with sulfanilamide and NEDA and quantified by spectro-photometric measurement at 540 nanometers.

The absorbing solution has absorption efficiency greater than 95% in NO<sub>2</sub> concentrations in parts per billion (ppb) units. The method can be used for short or long monitoring and the sample can be stored for up to four weeks without significant loss of NO<sub>2</sub>.

The range of concentration in the air for which this method can be used confidently is between 10 and 1000 mg/m<sup>3</sup> (0.005 to 0.500 ppm), based on a sampling period of 24 hours. The behavior of the method for NO<sub>2</sub> levels above 1000 mg/m<sup>3</sup> has not been established.

##### **➤ Sensitivity**

The sensitivity of the method depends on the Griess-Saltzman reagent. For cell of optical path of 1.0 cm and 0.1 absorbance units, it is equivalent to 0.14 mg / mL of nitrogen dioxide in the absorption solution.

##### **➤ Precision and accuracy**

The precision of the method in the field, expressed as the standard deviation is ± 12 mg/m<sup>3</sup> (± 0.006 ppm) in a 24-hour sampling, for an average of 81 mg/m<sup>3</sup> (0.043 ppm) compared with a continuous color analyzer.

#### 1.4.2 Gas Equipment

The gas equipment collects an air sample for the analysis of NO<sub>x</sub>. Equipment: The Andersen Gas sampler used in this project consists of a metal box with a lid and two squeezable compartments. The first chamber has a vacuum pump whose specifications meet those of the USEPA.

In the second compartment lies the sampling train, which is connected to the vacuum pump and consists of a distribution pipe connected in series with two collectors of borosilicate glass (impingers) containing the absorbing solution for NO<sub>2</sub>, and the other one is empty and serves as a trap (impinger trap). The air flow passing through the system is controlled by critical orifices and must be calibrated before and after the collection of the sample (every 24 hours). The system is protected by an 8 microns membrane filter placed between the entrance of the sample and the first impinger, and a moisture trap placed between the impinger trap and vacuum pump. Additionally, the collector of NO<sub>2</sub> is embedded in a mini-fridge chilled with dry ice or ice and brine, to maintain the system at low temperature.

##### ➤ Precision and accuracy

The accuracy of the method used in the field is expressed as the standard deviation of  $\pm 12$  mg/m<sup>3</sup> ( $\pm 0.006$  ppm) in a 24-hour sampling. The reagents for the measurement of NO<sub>2</sub> are, Absorbent Solution, Hydrogen Peroxide Solution, Sulphanilamide, Dihydrochloride N-(1-Naphthyl)-Ethylenediamine and Nitrite stock solution

#### 1.4.3 Sulfur Oxides (SO<sub>x</sub>)

##### ➤ Para-Rosaniline Method

The SO<sub>x</sub> in ambient air is collected by a vacuum pump, they are absorbed in a potassium tetrachloromercurate solution, TCM, which forms the dichlorosulphitomercurate complex, which reacts with methylsulfamic acid, formaldehyde and para-roaniline to form the acid para-roaniline methylsulfone, which is bright red, and whose absorbance is measured by a spectrophotometer at 548 nanometers. The detection method limit is 5 mg/m<sup>3</sup> but can be reduced by using larger sample aliquots.

##### ➤ Gas equipment

The gas equipment collects air samples for analysis of sulfur oxides and nitrogen oxides separately but simultaneously by collecting both gases, with the difference that other solutions are used as absorbents. The method is the same as that described above for nitrogen oxides.



➤ **Methodology for measuring CO**

The Measurement was performed once daily in each of the sampling locations, the equipment used was a MiniPak QUEST TECHNOLOGIES in situ meter for determination of CO, which uses a sensor with an integral-derivative memory module that contains and acknowledges the information of the CO sensor.

These equipments are designed according to the ANSI/ISA-S12.1 standards: Definition and Information Pertaining to Electrical Instruments in Hazardous (Classified EPA) Gases, ANSI-S12.5: Performance Requirements for Gas Detection Instruments, and ANSI Standard Levels With respects to Human Exposure. The data is reported with intrinsic corrections for temperature and local barometric pressure.

## 2. METHODOLOGY USED FOR SOIL SAMPLING

### 2.1 SAMPLING AND PRESERVATION OF THE SAMPLES

The samples were collected by the technical personnel of ICP. The specifications established in the method AWWA 1060/05 “Collection and Preservation” of the Standard Methods, Ed. 21 of 2005 and USEPA SW 846 “METHODS FOR EVALUATION SOLID WASTE PHYSICAL/CHEMICAL METHODS” were addressed. For sampling, the following aspects were considered:

**Containers:** The preparation of the containers used for the recollection of the samples was done with the procedures established in the Water and Soil Laboratories according with the specifications established in the Standard Methods Ed. 21th (2005).

Marking of the containers was done before sampling, having each marking at least the following information: place and date of sampling, hour, parameters to be evaluated, and preservation.

**Preservation:** The preservation of the samples is done with analytic grade additives. To verify the quality of the reactive used for the preservation of the samples it is taken duplicated samples and blank samples. The samples that do not need preserving additives are refrigerated immediately. Table 1 is a summary of the containers used and the preservation of the samples.

**Table 1 Containers used and preservation of the samples**

SOIL				
Parameters	Amount (gr)	Container	Preservation	Storage time
<b>Laboratory Analysis</b>				
pH, conductivity, greases and oils, humidity, cationic interchange capability, interchangeable cations, RAS and metals	2000	Aluminum foil/hermetic polyethylene bags	Refrigerate at 4°C	28 days

Source: Colombian Institute of Petroleum (2009)

### 2.1.1 TYPE OF SAMPLING

The type of sampling done in the soil of Palagua Field was punctual. The monitoring protocol is summarized below:

- Punctual samples for the pH analysis, conductivity, greases and oils, humidity, cationic interchange capability, interchangeable cations, SAR and metals (cadmium, zinc, lead, barium, silver, selenium, arsenic and mercury)
- Refrigeration of the samples
- Delivery to the ICP for analysis.

### 2.2 LABORATORY ANALYSIS

The physicochemical analysis was done following the specifications of the technical norms described in Table 2.

**Table 2 Technical norms used**

SOIL		
Parameter	Norm	Detection level-quantification
pH	AWWA 4500/05	0-14
Conductivity (MCC)	AWWA 2510/05	0.89 μmhos/cm
Greases and oils (MCC)	EPA 9071 a Re 94	0.010 % w/w
Humidity @ 105°C	METHOD IGAC	0% w/w
Cationic interchange capability (CIC)	EPA 9080 11260-94	-
Interchangeable cations (Ca, Mg, Na, K) with determination by ICP-OES	Method I.G.A.C ASTMD 4593-4698.92	Ca: 0.017 mg/L Mg: 0.001 mg/L Na: 0.008 mg/L K: 0.006 mg/L
Metals by OES: Ba, V, Cd, Zn, Pb, Ag, Se, As (MCD)	AWWA 3125/05	Ba: 0.0632 μg/Kg V: 0.0715 μg/Kg Cd: 0.0636 μg/Kg Zn: 0.0450 μg/Kg Pb: 0.3925 μg/Kg Ag: 0.0181 μg/Kg Se: 0.3985 μg/Kg As: 0.3140 μg/Kg
Metals by Atomic absorption Generator of Hydrocarbons		Hg: 0.123 μg/Kg

MCD: Minimum concentration detectable

MCD: minimum concentration

quantifiable

Source: Colombian Institute of Petroleum (2009)

### 2.3 GENERAL DESCRIPTION OF THE METHODOLOGIES USED

- pH: determination by the use of a pH-meter Orion calibrated with 3 buffer solutions of pH (4.0, 7.0 and 10).
- Conductivity: The determination is done from the saturation extract obtained of the soil sample to establish the quantity of existing salts, through cells of an appropriate constant.
- Humidity: gravimetric determination that corresponds to the relation between the water mass present in a sample of soil and the weight of the dry sample at 105°C ±5°C.
- Greases and oils: gravimetric method with extraction of greases and oils using as solvent n-hexane.
- Sodium Absorption Ratio (SAR): is calculated based on the concentrations of sodium, calcium and magnesium. The concentrations of all the metals are in meq/L.

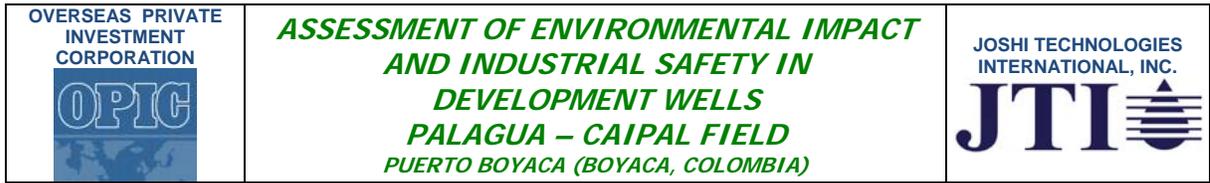
$$\bullet \quad SAR = \frac{Na^+}{\sqrt{(Ca^{++} + Mg^{++})/2}}$$

- Cationic Interchange Capability (CIC): determined by the ammonium acetate method, that implies the saturation of the sample of soil with a solution of such acetate replacing totally the cations present in the colloidal complex by the ammonium ion, with the subsequent rinse with alcohol to eliminate the excess of the acetate. This will reduce to a minimum the hydrolysis and the determination of the ammonium content by distillation.
- Interchangeable cations (calcium, magnesium, sodium, potassium): determination of the calcium, sodium, magnesium and potassium available through the acid treatment of the filtrate obtained from the cationic interchange capacity and afterwards reading by ICP-OES.
- The percentage of interchangeable sodium (PSI): it is calculated the relation between the interchangeable sodium and the cationic interchange capacity. The concentrations are in meq/100g.

$$\bullet \quad \%ESP = \frac{Na \text{ interchangeable (meq/100g)}}{CIC(\text{meq/100g})} * 100$$

- Metals (Ba, V, Cd, Zn, Pb, Ag, Se, As): determination by ICP-OES. The sample was previously acid digested.
- Metals (Hg): determination by atomic absorption by generation of hydrocarbons of the sample previously acid digested.

Before the samples analysis, it was analyzes reference solutions of known concentrations under same conditions. This was done to build the respective calibration curve of the methods of metals. For each method it is analyze a blank of water type 1 to



determine if the elements to be analyzed or other interferences are present in the reagents, materials, equipment or the laboratory environment.

#### **2.4 EQUIPMENT USED**

The equipment used in the analytic determination by the different laboratories is described below:

- pH-meter Orion 920 A
- Digital scale Sartorius MC 235p
- Conduct-meter Horiba DS-12
- Absorption Spectro-photometer ICP-OES Optima 2100DV
- Atomic Absorption Spectro-photometer Perkin Elmer A Analyst 400

### 3. METHODOLOGY USED FOR UNDERGROUND WATER

The samples were collected by the technical personnel of the Colombian Petroleum In situ, following the specifications of the method AWWA 1060/05 “collection and preservation” of Standard Methods, Ed. 21 of 2005. For the sampling the following considerations were taken:

#### 3.1 PRESERVATION

The preparation of the containers used for the recollection of the samples was done according with the procedures established in the Water and Soil Laboratory following the specifications of the standard mentioned previously. In the following table the process for each container is described.

Container	Description
Plastic container	- Rinse with sufficient tap water - Rinse with distilled water
Container with blue lid	- Rinse with tap water and “extran” soap - Add sulpho-cromic mix - Clean with hexane
Ambar container	- Rinse with sufficient tap water - Rinse with distilled water
Container for metals	- Rinse with sufficient tap water - Purge with HNO <sub>3</sub> 1:1 solution - Rinse with sufficient distilled water

SOURCE, ICP (2009)

Labeling of the containers is done before each sampling, labeling with the following minimal information: Sampling site, hour, parameter to be evaluated and preservation.

The preservation of the samples is done with analytic grade reagents, in order to verify the quality of the reagents used for the preservation of the samples duplicated and blank samples are taken. The samples that do not require preservation are refrigerated immediately. The following table details the containers and preservation of the samples.

**Table 3 Containers and preservation of the samples**

Parameter	VLN ml	Container	Preservation	Storage type
<b>In situ analysis</b>				
pH –Temperature	25	Plastic/ Glass	Not required	In Situ
Dissolved oxygen	25	Glass	Not required	In Situ
Turbidity	25	Glass	Not required	In Situ
<b>Laboratory Analysis</b>				
pH –Temperature	25	Plastic/ Glass	Not required	In Situ
Conductivity, alkalinity, carbonates, bicarbonates, chloride, sulphates, nitrates, nitrites, and dissolved solids.	2000	Plastic/ Glass	Refrigerate at 4°C	8 days
Greases, oils and total hydrocarbons	500	Wide mouth Glass	HCl pH <2.0, Refrigerate at 4°C	28 days
Ammoniac nitrogen	500	Plastic/ Glass	H <sub>2</sub> SO <sub>4</sub> a pH < 2.0, Refrigerate at 4°C	28 days
Phenols (photometric)	500	Plastic/ Glass	H <sub>2</sub> SO <sub>4</sub> a pH < 2.0- Refrigerate at 4°C	28 days
Metals	500	Plastic/ Glass	HNO <sub>3</sub> a pH <2.0 Refrigerate at 4 °C	6 months
Polyaromatic hydrocarbons (PAH's)	20	Glass	Refrigerate at 4°C	7 days
BTEX's	20	Glass	Refrigerate at 4°C	7 days
Extractable hydrocarbons (HC's)	20	Glass	Refrigerate at 4°C	7 days

SOURCE, ICP (2009)

### 3.2 SAMPLING

The type of sampling done in Palagua Field was punctual. The monitoring protocol can be described as:

- Measurement at the piezometer level with probe
- Purge of the monitoring well by extraction of a volume of water that guarantees the sampled water is fresh (approximately a volume of 15 bailers used in the sampling – 0.75L)
- Measurement of dissolved oxygen in-situ by the Winkler methods
- Measurement of pH and temperature in-situ
- Measurement of turbidity in situ
- Sampling for the analysis of pH/Temperature, alkalinity, carbonates, bicarbonates, conductivity, dissolved solids, anions (chloride, sulphate, nitrate,

nitrite), ammoniac nitrogen, phenols, greases and oils, oils and total hydrocarbons, metals, BTEX's, Poly-aromatic hydrocarbons (PAHs) and Extractable Hydrocarbons (HCs)

- Vacuum filtration by a member of 0.45µm of the metals samples
- Preservation (acidification) of the samples that required it and cooling
- Delivery of samples to the lab for analysis.

### 3.3 LABORATORY ANALYSIS

The physicochemical analysis was done following the specifications of the technical norms described in the Table 3.

**Table 4 Technical norms used**

SOIL		
Parameter	Norm	Detection level-quantification
pH	AWWA 4500/05	0-14
Dissolved oxygen (MCD)	AWWA 4500-O/05	0.0 mg/L
Conductivity (MCC)	AWWA 2510/05	0.89 µmhos/cm
Turbidity (MCC)	AWWA 2130/05	0.060 NTU
Dissolved solids (MCC)	AWWA 2540/05	0.10 mg/L
Phenols	AWWA 5530-05	0.029 mg/L
Greases and oils (MCC)	AWWA 5520-C-H/05	1.07 mg/L
Alkalinity (MCC)	AWWA 2320/05	0.32 mgCaCO <sub>3</sub> /L
Nitrites, nitrates, Chlorides, sulfates by ionic chromatography (MCC)	AWWA 410-C/05	NO <sub>3</sub> : 0.059 mg NO <sub>3</sub> /L NO <sub>2</sub> : 0.031 mg NO <sub>2</sub> /L Cl <sub>2</sub> : 0.23mg Cl <sub>2</sub> /L SO <sub>4</sub> <sup>-2</sup> : 0.45mg SO <sub>4</sub> <sup>-2</sup> /L
Ammoniac nitrogen	AWWA 4500-NH3-05	0.59 mgNH <sub>3</sub> /L
Metals by ICP-MS (Mg, Na, K, Fe, Al, Mn, Cr, Cu, Cd, Zn, Pb, Ba, Ni, V, Mo, Se, As) (MCD)	AWWA 3125/05	Mg: 1.614µg/L Na: 2.639 µg/L K: 5.286 µg/L Fe: 10.029 µg/L Al:3.508 µg/L Mn: 0.215 µg/L Cr: 0.115 µg/L Cu: 2.102 µg/L Cd: 0.0636 µg/L Zn: 0.0450 µg/L Pb: 0.3925 µg/L Ba: 0.0632 µg/L Ni: 0.249 µg/L V: 0.0715 µg/L Mo: 0.099 µg/L Se: 0.3985 µg/Kg As: 0.3140 µg/Kg
Metals by AA: Hg (MCD)	AWWA 3125/05	0.123 µg/L
Metals by ICP-EOS : Ca (MCD)	AWWA 3125-05	Ca: 3.076 µg/L
Poly-aromatic hydrocarbons (PAH's)	US EPA SW 846 METHODS 8000C/8100/3500C/3510 C/3580A/3600C/3610B/36 11B/3630C	Naftalene : 1.60E-4 mg/L Acenaftilene: 1.90E-4 mg/L Acenaftene: 1.90E-4 mg/L Fluorene: 2.00 E-4 mg/L Fenantrene: 2.20E-4 mg/L

		<p>Antracene: 2.20E-4 mg/L          Fluorantene: 2.20E-4 mg/L          Pirene: 2.20E-4 mg/L          Benzo(a)antracene: 2.50E-4 mg/L          Crisene: 2.50E-4 mg/L          Benzo(b)fluorantene: 2.60E-4 mg/L          Benzo(k)fluorantene: 2.80E-4 mg/L          Benzo(a)pirene: 2.80E-4 mg/L          Indeno(1,2,3-cd)pirene: 3.60E-4 mg/L          Dibenzo(a,h)antracene: 4.10E-4 mg/L          Benzo(g,h,i)perilene: 4.00E-4 mg/L</p>
Extractable aliphatic hydrocarbons (HC's)	<p>US EPA SW 846          METHODS          8000C/8100/3500C/3510          C/3580A/3600C/3610B/3611B/3630C</p>	<p>Octane (C8): 9.00E-5 mg/L          Nonane (C9): 8.00E-5 mg/L          Decane (C10): 8.00E-5 mg/L          Undecane (C11): 9.00E-5 mg/L          Dodecane (C12): 9.00E-5 mg/L          Tridecane (C13): 9.00E-5 mg/L          Tetradecane (C14): 9.00E-5 mg/L          Pentadecane (C15): 9.00E-5 mg/L          Hexadecane (C16): 9.00E-5 mg/L          Heptadecane (C17): 9.00E-5 mg/L          Octadecane (C18): 9.00E-5 mg/L          Nonadecane (C19): 9.00E-5 mg/L          Pristane (n-C19): 9.00E-5 mg/L          Eicosane (C20): 9.00E-5 mg/L          Fitane (n-C20): 9.00E-5 mg/L          Heneicosane (C21): 8.00E-5 mg/L          Docosane (C22): 9.00E-5 mg/L          Tricosane (C23): 8.00E-5 mg/L          Tetracosane (C24): 8.00E-5 mg/L          Pentacosane (C25): 8.00E-5 mg/L          Hexacosane (C26): 8.00E-5 mg/L          Heptacosane (C27): 8.00E-5 mg/L          Octacosane (C28): 8.00E-5 mg/L          Nonacosane (C29): 8.00E-5 mg/L          Triacontane (C30): 9.00E-5 mg/L          Hentriacontane (C31): 9.00E-5 mg/L          Dotriacontane (C32): 1.00E-4 mg/L          Tritriacontane (C33): 1.00E-4 mg/L          Tetratriacontane (C34): 1.10E-4 mg/L          Pentatriacontane (C35): 1.20E-4 mg/L          Hexatriacontane (C36): 1.30E-4 mg/L          Heptatriacontane (C37): 1.40E-4 mg/L          Octatriacontane (C38): 1.70E-4 mg/L          Tetracontane (C40): .2.50E-4 mg/L</p>
BTEX's (MCC)	<p>US EPA, SW 846,          METHODS          8000C/8021B/8260C/500          0A/5021A/3500C/3580A</p>	<p>Bencene: 2.76 µg/L          Toluene: 2.23 µg/L          Etilbencene: 1.82 µg/L          M,P-Xilene: 1.85 µg/L          O-Xilene: 2.30 µg/L</p>

MCD: Minimum concentration detectable    MCD: minimum concentration quantifiable  
 Source: Colombian Institute of Petroleum (2009)

### 3.3.1 GENERAL DESCRIPTION OF THE METHODOLOGY APPLIED

- pH: determination by the use of a pH-meter Orion calibrated with 3 buffer solutions of pH (4.0, 7.0 and 10).
- Temperature: determination using a pH-meter Orion
- Turbidity: determination by nefelo-metric method using a turbidimeter
- Dissolved oxygen: Determination by the Winkler method using a kit from Merck
- Conductivity: The determination is done with a Conductimeter Horiba DS-12 calibrated with a KCl solution for a high and low range.
- Alkalinity: Potentiometer method at pH 4.5
- Carbonates and bicarbonates: potentiometer method at pH: 8.3 and pH: 4.5 respectively.
- Dissolved soils: correspond to the fraction of solids present in a sample that pass through a filter of 0.45 um, dry at 180°C until constant weight.
- Greases and oils: gravimetric method with extraction of greases and oils using as solvent n-hexane.
- Total hydrocarbons: gravimetric method by re-dissolution of the grease and oils extract with n-hexane solvent, using a column of silica gel where the polar compounds are retained. Later the solvent with the hydrocarbons is recovered to quantify with a gravimetric method.
- Chloride, sulphates, nitrite, nitrate: determination with ionic chromatography. The concentrations of chlorides and sulphates are calculated by measurements of the conductivity detector while the detector UV at 210 nm calculates the concentration of nitrates and nitrites.
- Ammoniac nitrogen: distillation of the sample and quantification by titration with potentiometer.
- Phenols: Spectrophotometer determination at 500 nm by development of color with 4-amino-antipirine
- BTEX's: determination with gas chromatography with headspace and flame ionization detector (FID). The compounds analyzed are: benzene, toluene, etilbencene, m+p-xilene and o-xilne.
- Poly-aromatic hydrocarbons (PAHS): determination through gas chromatography. The compounds analyzed are named in the previous table.
- Extractable hydrocarbons (HC's): determined by gas chromatography. The analyzed compounds are from N-8 (Octane) to N-40 (Tetracontane). The list is in the previous table.



- Metals (Mg, Na, K, Fe, Al, Mn, Cr, Cu, Cd, Zn, Pb, Ba, Ni, V, Mo, Se, As): determination by mass spectrometry with ICP-MS
- Metals (Ca): determination by optic emission spectrometry (ICP-OES)
- Metals (Hg): determination by atomic absorption by generation of hydrocarbons of the sample previously acid digested.

Before analyzing the samples, the reference samples with different known concentrations were analyzed under the same conditions in order to obtain the calibration curve for the phenols, metals, PAHs, BTEXs, HCs, and anions (chloride, sulphates, nitrates and nitrites). For each method the lab analyzed a blank of water type 1 in order to determine presence of the interest compounds or interference in the reagent, material, equipment or laboratory the environment.

### 3.3.2 EQUIPMENT USED

- pH-meter Orion 920 A
- Conductivimeter Horiba DS-12
- Turbidimeter Micro 100 IR
- Turbidimeter Turbiquant 1100 IR from Merck
- pH-meter Orion 710
- Spectrophotometer UV-VIS NIR Cary Varian 5000
- Digital Scale Sartorius MC 210p
- Digital Scale Sartorius ME 235p
- pH-meter Orion 3 STAR B09781
- Liquid Chromatograph HP 1200-1 Agilent Technologies
- Spectrophotometer ICP-OES 2100 DV
- Mass Spectrophotometer ICP-MS ELAN 6000
- Atomic absorption Spectrophotometer Perkin Elmer A Analyst 400
- Gas chromatograph 6890N with headspace coupled with flame ionization detector (FID).

#### 4. SAMPLING METHODOLOGY FOR NOISE

At the Location point sites described in Chapter 3 section F, noise measurements were taken with a sound level meter, QUEST ELECTRONIC INC. Brand, model 2900, previously calibrated with scaled pondering filter A. The sound level meter features are presented in Table 1.

**Table 5 Characteristics SLM (Sound Level Meter)**

SLM QUEST ELECTRONIC MOD. 2900	
STANDARDS	ANSI S 1.4-1983 TYPE 2 IEC 651-1979 TYPE 2 IEC 804-1985 TYPE 2 RMS, 63 dB
DETECTOR:	20 – 140 dB
RANGE:	A, C, LINEAL
FREQUENCY:	120 dB
UPPER LIMIT:	80 – 90 dB
THRESHOLD:	4 DIGIT LIQUID CRYSTAL
READING:	0.01 – 19999
DOSE:	½ " WITH CONDENSATION
MICROPHONE:	Pre-polarized
TEMPERATURE:	-10 A +50°C
HUMIDITY :	0 – 95%
MAGNETIC FIELD EFFECT:	NOT SIGNIFICANT

SOURCE: ANTEK S.A. (November, 2009)

##### 4.1 PRE-POLARIZED CONDENSER MICROPHONE

Pre-polarized microphones operate according to the same principle as condenser microphones of conventional design, which relies on changes in electrical capacity to develop corresponding changes in voltage. However, the electric field is not set or determined by an external voltage polarization bias, but it is set by charges that are trapped by "permanently" on or attached to a special polymer material, so that the positive charge preponderance resides on one side of the material and the negative charges on the other.

Due to the fact that pre-polarized microphones may easily have the same characteristics as conventional electro- microphones that condenser microphones are suitable for



measuring sound levels meeting the accuracy requirements of Class (0) lab or Class (1) Accuracy.

The main advantage of these microphones is their ability to operate in wet environments: since there are no free electric charges, there is a much greater freedom for the discharge voltage in the hole between the back plate and diaphragm.

## **4.2 CONSIDERED FREQUENCY**

The pondered frequency in a sound-level alters the characteristics of the frequency response according to the specifications of a national or international standard. Thus, the indication of an instrument to measure the sound level for a given level of pressure input noise depends on the frequency of the sound reaching the microphone and the weight of the selected frequency.

### **4.2.1 Considered A**

The national and international standards require that all devices that measure the sound level, frequency weighing incorporate designated by the letter A. Many years of study and practical experience have shown that “A-weighted sound levels” provide adequate correlation with several human responses (from people or groups in a community) for different types of noise sources and consequently, is the most commonly used frequency weighing. Feature is that the weighing takes into account the reduced sensitivity of normal human hearing for low frequencies compared to the response at high frequencies.

## **4.3 MODEL SOUND PRESSURE LEVELS (SPL)**

The modeling of sound pressure levels is a widely used tool to project the impact and / or changes in the acoustic environment, urban development and industrial processes. In the same way it can be used as a planning tool for certain environments where noise control is necessary. Currently the market has multiple computer programs that are being used for programming external noise such as:

### **4.3.1 Sound plan 5.0.**

This plan was used for modeling simulation software for noise control and environmental protection. SOUNDPLAN 5.0 is a program that enables to optimize control measures and visualize the effect of noise propagation in industrial processes. This program has been known worldwide for over a decade for being the most reliable, accurate and rapid tool for the progress of science in Engineering Acoustics and Noise Control, counting among its users over 2000 universities worldwide in over 30 countries, SOUNDPLAN 5.0 is a software application that delivers clear graphic for practical applications.



To feed the software shows the precise locations of the measurements and the values reported by the meter, preferably forming a network day of imaginary values where more information is the most representative results presented in isophonic curves and equivalent to the dispersion of noise in magnitude and direction.