

## STATE CONTROL OVER POLLUTANTS MEASUREMENTS IN BRAZIL

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**Abstract:** National Environmental Policy established quality standards. In order to verify compliance with this standards, controlling and monitoring are necessary, which depend on the measurement of environmental pollutants. Accordingly, Inmetro's performance by means of metrology has contributed for increasing the confidence in measurements results. International Organization of Legal Metrology (OIML) has developed recommendations applicable to environmental monitoring, aiming at filling society's needs. This paper presents and discusses the control applied to measuring instruments used in the environmental field, along with a presentation of new perspectives related to the topic.

**Key-words:** metrological control, environmental pollution, environmental monitoring.

### 1. INTRODUCTION

It is remarkable the incompatibility between sustainable development and current production and consumption standards. Therefore, properly identification and quantification of the impact human activities have on the environment become especially important when reviewing such standards.

In general, the main negative environmental impacts result from the exploitation of natural resources in a higher rate than its natural capacity of regeneration, or from waste disposal beyond the environmental assimilation capacity [1].

Due to it, one way of efficiently controlling the environment, according to legislation, is through the definition of desirable quality standards for atmosphere, water and soil in a certain environment [2], and through the establishment of a monitoring plan, which involves measurements.

In this manner, a sound national measurement system is an essential factor for the protection of the environment. In Brazil, the National Institute of Metrology, Standardization and Industrial Quality (Inmetro) has contributed for ensuring the credibility of measurements results in metrology<sup>1</sup>, both in scientific<sup>2</sup> and industrial<sup>3</sup>, and legal<sup>4</sup> branches.

<sup>1</sup> Metrology is the science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology.

<sup>2</sup> Scientific metrology concerns the establishment of quantity systems, unit systems, units of measurement, the development of new measurement methods, realization of measurement standards and the transfer of traceability from these standards to users in society.

<sup>3</sup> Industrial metrology concerns the application of measurement science to manufacturing and other processes and their use in society, ensuring the suitability of measuring instruments, their calibration and quality control of measurements.

An example of the importance of the science of measurement in environmental field is the control of vehicles emissions, which involves two types of instruments: opacimeters and exhaust gas meters. These instruments must meet all metrological and technical requirements specified by Inmetro, which provides reliability to measurements results. In other words, by means of reliable measurements, authorities can prohibit the use of vehicles that emit more pollutants than allowed by regulation. Other examples are water meters and electricity meters, which can save water and energy when functioning properly.

### 2. OBJECTIVE

State control over pollutants measurements in Brazil will be analyzed. The main regulations in environmental field are identified as well as measuring instruments used for monitoring natural resources, along with the type of metrological control adopted nowadays. Therefore, the goal of this paper is to show Inmetro's role in controlling pollutants measurements.

### 3. ENVIRONMENTAL CONTROL

Considering Brazilian federal legislation, environmental quality regulation began with Decree-law 1.413/1975, which provides for the environmental pollution caused by industrial activities [3, 4].

Some years later, Brazilian National Environmental Policy was established by means of Law 6.938/1981 [5], considered a response to United Nations Conference on the Human Environment, held in Stockholm in 1972.

Through this law, pollution was defined as environmental quality deterioration resulting from activities that directly or indirectly: a) are harmful to health, safety and well-being of the population; b) create adverse conditions to social and economic activities; c) affect unfavorably the biota; d) affect aesthetic or sanitary environmental conditions; e) release matter or energy in disagreement with established environmental standards (article 3, item III).

In article 8, the law conferred the Environmental National Council (Conama), among others, competence to:

- establish, exclusively, norms and national standards for pollution caused by road vehicles, aircrafts and boats;

<sup>4</sup> Legal metrology is the part of metrology relating to activities which result from statutory requirements and concern measurement, units of measurement, measuring instruments and methods of measurement and which are performed by competent bodies.

- establish norms, criteria and standards related to environmental quality control and maintenance for the rational use of natural resources, mainly the water ones.

Therefore, Conama has been building, through resolutions, the normative base for controlling pollutants emissions, enabling the establishment of environmental quality standards, one of the tools of Brazilian Environmental Policy.

In turn, 1988 Constitution, in its article 225, considers that everyone has the right to have an ecologically balanced environment, imposing to public authorities and to all citizens the duty of protecting and preserving it for present and future generations. Due to it, those who damage the environment are subject to penalties, independently of the obligation to fix the damages caused [6].

In this regard, Law 9.605/1998 consolidated all infringements and penalties provided in federal environmental legislation and established as crime “to cause pollution of any kind in levels that result or can result in damage to human health, or that provoke the killing of animals or significant flora destruction” [7].

#### 4. MEASUREMENTS CONTROL

A system of continuous and systematic monitoring that verifies the compliance with quality standards established in law is necessary to promote an effective environmental control. Therefore, an adequate confidence level must be ensured to measurements in order to provide credibility to results. One way of ensuring such credibility is by means of legal metrological control.

In Brazil, Inmetro is the institution responsible for the legal metrological control, which involves a set of procedures, technical resources and operations needed for ensuring measurements results credibility. This control occurs when there are statutory requirements, enacted by means of a legal metrological regulation, implemented by the government in order to ensure quality and credibility to measurements related to official control, trade, health, safety and environment [8].

The control of measuring instruments is ensured through the following legal operations:

- Type approval;
- Verification<sup>5</sup>;
- Inspection;

When specified in technical metrological regulation, instruments must have their type approved by Inmetro, which examines and tests them to evaluate if they are adequate to the purpose. Before being sold, each instrument must be submitted to an initial<sup>6</sup> verification to ascertain its conformity with the approved type. Any further verification is called subsequent, including mandatory periodic verification and verification after repair [9]. Besides, inspection is the examination of a measuring instrument to ascertain all or some of the following:

- verification mark and/or certificate is valid;

<sup>5</sup> Verification of a measuring instrument is a procedure which includes the examination and marking and/or issuing of a verification certificate, that ascertains and confirms that the measuring instrument complies with the statutory requirements.

<sup>6</sup> Verification of a measuring instrument which has not been verified previously.

- no sealing marks are damaged;

- after verification the instrument suffered no obvious modification;

- its errors do not exceed the maximum permissible in-service errors.

Internationally, in 1955 it was established the International Organization of Legal Metrology (OIML) in order to promote the global harmonization of legal metrology procedures. Since that time, OIML has developed a worldwide technical structure that provides its Members with metrological guidelines for the elaboration of national and regional requirements concerning the manufacture and use of measuring instruments for legal metrology applications<sup>7</sup> [10].

OIML develops model regulations, known as international recommendations, for various categories of measuring instruments. The OIML technical committee (TC) responsible for instruments for measuring pollutants is TC16<sup>8</sup>, which has 4 Subcommittees: Air Pollution; Water Pollution; Pesticides and other pollutant toxic substances; Field measurements of hazardous (toxic) pollutants.

Regarding environmental pollutants measuring instruments, OIML presents nine recommendations [11]:

- R 82 (1989) - *Gas chromatographic systems for measuring the pollution from pesticides and other toxic substances*;

- R 83 (1990) - *Gas chromatograph/mass spectrometer systems for the analysis of organic pollutants in water*;

- R 99 (2008) - *Instruments for measuring vehicle exhaust emissions*;

- R 100 (1991) - *Atomic absorption spectrometers for measuring metal pollutants in water*;

- R 112 (1994) - *High performance liquid chromatographs for measurement of pesticides and other toxic substances*;

- R 113 (1994) - *Portable gas chromatographs for field measurements of hazardous chemical pollutants*;

- R 116 (1995) - *Inductively coupled plasma atomic emission spectrometers for the measurement of metal pollutants in water*;

- R 123 (1997) - *Portable and transportable X-ray fluorescence spectrometers for field measurement of hazardous elemental pollutants*;

- R 143 (2009) - *Instruments for the continuous measurement of SO<sub>2</sub> in stationary source emissions*.

OIML International Document<sup>9</sup> 12, published in 1986, states that measuring instruments should be verified when used in trade, that is to say in connection with commercial activities, whenever it is clear that measurement errors could have a significant adverse economic impact on the buyer or seller. It might be argued that, to completely protect the public, all instruments used in trade should be subject to legal controls and verified. However, because effective

<sup>7</sup> OIML recommendations are meant only to be the basis for the establishment of national legislation in each country, and are not mandatory.

<sup>8</sup> Although noise is also a type of pollution, this issue does not belong to TC16, but to TC 13 – Measuring instruments for acoustics and vibration – and will not be discussed in this paper.

<sup>9</sup> OIML Documents are informative in nature and intend to harmonize and improve work in the field of legal metrology.

controls are often expensive, judgment must be exercised in determining which measurements to control. Instruments considered to have the highest priority may be selected [12].

Considering that legal metrological control is not applicable to every measuring instrument used in areas of public interest, another way of ensuring measurements results credibility is by performing the analyses in accredited laboratories.

The General Coordination for Accreditation (Cgcre) is the accreditation body of conformity assessment bodies recognized by the Brazilian Government. The accreditation carried out by Cgcre is voluntary and represents the formal recognition of the competence of a laboratory or body to perform conformity assessment tasks according to established requirements<sup>10</sup>.

One step to be accomplished consists in measurement audits, when the laboratories must prove they have means and objective criteria to perform the tests and to ensure reliable and adequate results to the required quality. In such audits it is verified, for example, if the results obtained by the laboratory, including uncertainty in measurement, are compatible with the true value assigned to the itinerant standard or if uncertainties are compatible with the best measurement capacity of the laboratory [13].

In view of these considerations, the current status and perspectives concerning the control established over pollutants measurements in Brazil will be analyzed.

## 5. POLLUTANTS MEASUREMENTS

### 5.1. AIR POLLUTION

The setting of parameters for gaseous pollutants and particulate matter started to be done through Conama Resolution 05/1989, which provides for Air Pollution National Control Program (Pronar). In this way, quality standards were established according to the zoning of certain areas. Conama Resolutions 03/1990 and 08/1990 complement Pronar, setting limits for the concentration of specific air pollutants [14, 15, 16].

In Conama Resolution 03/1990, national standards for air quality were set, aiming at preventing damages to health, safety and environment. Criteria for total suspended particles, smoke, inhalable particles, sulfur dioxide, carbon monoxide, ozone and nitrogen dioxide were defined.

For each of these pollutants the resolution defines one sampling and analysis method, but the use of similar alternative methods is allowed. Methods approved by Inmetro were established as reference ones and, in the lack of them, those recommended by Ibama should be used.

Many studies highlight the contribution of mobile sources, such as means of transportation, for degrading the air quality of urban centers. This type of pollution has been the subject of many studies and researches in various countries, including Brazil [17]. Due to the gravity of the problem, Federal Government created the Automotive

Vehicles Air Pollution Control Program (Proconve), through Conama Resolution 18/1986. According to it, all the new models of national and imported vehicles must be submitted to homologation concerning ceiling limits of pollutants emission [18].

For in-use vehicles, Proconve demanded to all Brazilian states the implementation of an Inspection and Maintenance Program, aiming at controlling emissions levels by using exhaust gas meters (Otto cycle engines) and opacimeters (Diesel engines) [19]. Measurements are performed in situ, when vehicles are inspected.

On account of this demand, exhaust gas meters are under legal metrological control. Inmetro Order 155/2005, which approves the Technical Metrological Regulation on Gas Meters, establishes that such instruments must be submitted to type approval, initial verification and subsequent verifications [20], aligned to OIML **R 99 (2008)**.

For opacimeters, legal metrological control is based on type approval, initial verification and subsequent verifications, according to Inmetro Order 060/2008, which approves the Technical Metrological Regulation on Opacimeters [21]. This instrument was regulated to meet Conama's demand, even though there is no specific OIML recommendation.

With regard to stationary sources of air pollution, Conama Resolution 08/1990 establishes the ceiling limits for emission of atmospheric pollutants in processes of external combustion that use fuel oil or coal. As other types of fuels were incorporated in the Brazilian energy matrix, as well as other types of stationary sources besides those listed by Resolution 08/1990, the need of a new legal requirement was observed.

Consequently, Conama Resolution 382/2006 was developed, which sets the ceiling emission limits for each pollutant, according to the type of pollution source: oil boilers, gas boilers, sugarcane bagasse boilers, wood-burning boilers, gas turbines, oil refineries, pulp mills, lead smelting industries, aluminum smelting industries, glass melting industries, cement kilns, fertilizers industries and steelworks [22].

According to this resolution, compliance with emission limits must be verified in conformity with sampling and analyses methods specified in technical standards scientifically recognized and accepted by the environmental licensing body. In the specific case of particulate matter, resolution determines the use of the measuring method of particulate emission from point source, as described in technical standards ABNT NBR 12019 and ABNT NBR 12827, or other equivalent method, as far as it is accepted by the environmental licensing body.

Along with these, the standards ABNT NBR 10700, ABNT NBR 10701, ABNT NBR 11966, ABNT NBR 12020, ABNT NBR 12021, ABNT NBR 12022, among others, define methods for analyzing other pollutants, such as total particulate (particulate matter), sulfur dioxide, volatile organic compounds, total reduced sulfur and nitrogen monoxide.

In this case of pollution from stationary sources, there is an OIML specific recommendation for sulfur dioxide measuring instruments. It is **R 143 (2009)**, which applies to measuring instruments (hereafter referred to as "gas analytical systems") that serve to determine the volume

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<sup>10</sup> In the case of laboratory accreditation, Inmetro verifies compliance with the requirements established in ABNT NBR ISO/IEC 17025. There are criteria to be met that are specifically related to measurements and to the use of measuring instruments, notably in item 5, Technical requirements. For example, the standard defines requirements about: 5.3) accommodation and environmental conditions; 5.4) testing and calibration methods and methods validation; 5.5) equipment; 5.6) measurement traceability; and, mainly, 5.9) quality assurance of test and calibration results.

fraction of SO<sub>2</sub> and establishes the conditions and requirements with which such systems shall comply in order to meet the requirements for measurement results of the pollutant content in stationary source emissions.

This recommendation applies to gas analytical systems intended for analyzing emissions of industrial enterprises and to gas analytical systems for controlling the technological processes whose principle of operation is based on UV absorption, fluorescence or nondispersive IR methods. The recommended metrological control is basically type approval, initial verification and subsequent verification.

However, legal metrological control for such instrument was not demanded by Conama and, as consequence, it is not regulated by Inmetro.

## 5.2. WATER POLLUTION

Conama provides, through Resolution 357/2005, among others, about water bodies classification and the guidelines for their categorization, as well as about effluent releasing conditions and standards [23].

Regarding water quality conditions and standards, this resolution sets ceiling limits for each toxic substance or pollutant that may be present in water bodies in Brazilian territories, which must be periodically monitored by public authorities. The limits established also determine conditions and standards that must be obeyed in cases of effluent releasing.

The resolution determines that water quality parameters analysis and evaluation must be done by public authorities, by means of its own laboratory or by contracted laboratories. These laboratories must adopt the procedures of analytical quality control needed to fulfill the necessary conditions.

Presently, Conama has been discussing a proposal of amendment to resolution 357/2005, in which is proposed, for the adequate effluent releasing and recipient water bodies management, that tests be performed by laboratories accredited by Inmetro or in laboratories accepted by the competent environmental authority [24].

The partnership with Inmetro in this area began through the National Laboratories Accreditation Program in Water Quality Analysis (Prolab), created by the National Water Agency (ANA). This program aims to implement a network of accredited laboratories that provides support to actions of regulation on usage and monitoring of water quality in national territory. ANA's intention is to provide proof of laboratories technical capacity through accreditation [25].

It's important to notice that Conama established a demand concerning the laboratories in which water analyses are performed, not specifying the measuring instrument involved. About this topic, OIML proposes three recommendations.

**R 83 (2006)** provides performance requirements and testing procedures for a gas chromatograph/mass spectrometer (GC/MS) system when used for measurements in determining water pollutants in pollution control programs and in assessing the quality of water as may be mandated by national laws and regulations. The mass spectrometers addressed and coupled with appropriate gas chromatographs may be used successfully to analyze a variety of water samples, such as ground waters, surface

waters, aqueous municipal and industrial effluents, and saline waters.

In Brazil, there are laboratories accredited by Inmetro that use GC/MS in the states of São Paulo, Bahia, Minas Gerais and Espírito Santo.

**R 100 (1991)** provides requirements for atomic absorption spectrometers (AAS systems) when used for legal measurements of metal pollutants in water. An AAS system can be applied simply and rapidly for the determination of a large variety of metals in drinking, surface and saline waters as well as domestic and industrial waste waters.

AAS systems can be used for measuring metal pollutants in water, in assessing water quality, and as a means of monitoring the effectiveness of pollution control programs mandated by national laws or regulations. Such control programs are usually designed to cover both toxic and non-toxic pollutants in potable water, surface water, ground water, and effluents to assess whether metal levels are below regulated limits.

OIML considers that an atomic absorption spectrometer is a complex instrument. Traditional legal metrology controls of initial and subsequent verification may not be practicable for this instrument. In this regard, higher accuracy and repeatability of measurements can be accomplished by careful attention to optimize the performance of each of an instrument's components. Besides it, analyses success may also depend on the knowledge, skill, and experience of the analyst.

In Brazil, at present, there are accredited laboratories that use AAS for determining pollutants in water in the states of São Paulo, Minas Gerais, Rio Grande do Sul, Paraná, Sergipe and Alagoas.

**R 116 (2006)** provides requirements for defining, testing, and verifying the performance of inductively coupled plasma atomic emission spectrometers (ICP systems) when used for measuring metallic and some non-metallic pollutants in water. An ICP system can be applied for rapid multi-element determination of a large number of elements in samples of potable, surface, ground, and saline waters as well as domestic and industrial waste waters.

These instruments provide a means for monitoring and assessing water quality that may be prescribed by pollution control programs mandated by national laws or regulations. Such control programs usually cover both toxic and non-toxic pollutants in potable, surface, ground, and saline water, as well as domestic and industrial-waste waters to assess whether the concentrations of pollutants are within established regulated limits.

There are accredited laboratories that analyze heavy metals using ICP in the states of São Paulo, Rio de Janeiro, Rio Grande do Sul, Minas Gerais and Paraná.

In Brazil, there is no legal metrological control over the instruments covered in this section. Credibility of measurement results is obtained by means of quality control procedures adopted in laboratories that perform the analyses of Conama Resolution 357/2005.

## 5.3. DIFFUSE POLLUTION BY PESTICIDES AND OTHER TOXIC SUBSTANCES

Analysis of agrochemicals environmental perilousness potential arose with the publication of Ibama Order

139/1994, which established the obligation of laboratories registration or recognition by Inmetro, for performing the mandatory ecotoxicological tests [26].

In 1997, Joint Order Ibama/Inmetro 66 was published, which established criteria for registration/recognition, by Inmetro, of laboratories that carry out physical-chemical, toxicological and ecotoxicological studies, for environmental evaluation of chemical, biochemical and biotechnological products, mandated by Ibama, according to Good Laboratory Practice (GLP)<sup>11</sup> [27].

Recently, Conama Resolution 420/2009 established criteria and guiding values for groundwater and soil quality concerning the presence of chemical substances. The document presents a series of parameters that will indicate, for example, quality references (which chemical substances are naturally present in soil), prevention references (which contaminants concentration, if exceeded, indicate a dangerous quality change to soil functions and to groundwater); and investigation references (concentrations that, if exceeded, indicate potential risks to human health) [28]. According to the resolution, analyses for soil and groundwater characterization and monitoring must be performed in laboratories accredited by Inmetro.

In the scope of toxic substances measurements listed by Conama, there are some OIML recommendations that deal with the involved measuring instruments.

**R 82 (2006)** covers gas chromatograph systems used for the measurement of pesticides and other toxic substances in carrying out pollution control programs and in assessing the quality of food products as mandated by national laws and regulations. Such systems are applied for the analysis of a variety of sample types such as ground water, surface water, industrial effluents, soil and sediments, plant and animal tissue, and food.

In Brazil, there are accredited laboratories that perform tests for determining pesticides residues using gas chromatography in the states of São Paulo, Rio Grande do Sul and Goiás.

**R 112 (1994)** provides requirements for performance of high performance liquid chromatographic (HPLC) systems when used for measurement of pesticides and toxic substances in carrying out pollution control programs and in assessing the quality of food products as mandated by national laws and regulations.

According to this recommendation, the responsible national body should consider adopting the control procedures as a means of assuring the continued metrological integrity of an HPLC system. Such control procedures may be established for specific analytical methods and may include a means for assessing laboratories using HPLC systems. Appropriate assessment procedures may include the following: accreditation of the user laboratory, self certification by the user laboratory, and proficiency testing through intercomparisons of measurements among user laboratories.

In Brazil, there are accredited laboratories for performing pollutants analyses using HPLC only in the states of São Paulo and Rio de Janeiro.

Regarding portable instruments used in field measurements of pollutants, OIML edited two recommendations, as follow.

**R 113 (1994)** provides procedures for testing and verifying the performance of a portable gas chromatograph (GC) for use in measuring potentially hazardous chemical pollutants associated with hazardous waste sites as mandated by national laws and regulations.

Such instruments can provide real-time measurements at hazardous waste sites and may be the basis for prescribing sampling plans, occupational and public health medical surveillance, public access, evacuation zones, and hazardous waste containment and cleanup. Hazardous waste sites are locations containing hazardous chemical wastes that can affect or have the potential to affect a larger surrounding area and include, for example, sites of uncontrolled hazardous waste dumping, sites of licensed hazardous wastes disposal facilities, and sites of accidents, spills, or fires involving hazardous chemicals either in fixed facilities or during transport. Portable GC may be used for monitoring other sites having hazardous chemical pollutants such as the work place, private homes, apartments, and other buildings, and public transports.

The other recommendation, **R 123 (1997)**, provides requirements for defining, testing and verifying the performance of portable and transportable X-ray fluorescence (XRF) spectrometers which may be used for legal measurements of hazardous elemental pollutants found in various media, especially soil and waste water. These instruments may be prescribed in national laws and regulations for field assessment and control of hazardous elemental pollutants. Examples are on-site analysis of pollutants in soils, ground water, waste water and sludge and particulate filter samples of ambient air at waste and industrial sites.

There is no legal metrological control, in Brazil, over the instruments covered in this section. Results credibility is assured by procedures adopted in accredited laboratories that perform the analyses established in Conama Resolution 420/2009.

## 6. DISCUSSION

As shown throughout this paper, control over environmental measurements has been carried out as a response to demands presented by the environmental sector, particularly by Conama resolutions.

This happened in the case of Proconve, whose resolution dates 1986. Subsequent complementary resolutions, still in the 1990s, established ceiling limits for pollutants emissions from in-use automotive vehicles. Due to it, Inmetro is responsible for carrying out the legal metrological control of opacimeters (even though there is no OIML recommendation) and exhaust gas meters (aligned to OIML **R 99 (2008)**), instruments used to measure pollutants emitted by in-use vehicles.

Concerning equipment for the measurement of pollutants in stationary sources, there was no demand for regulation from Brazilian environmental sector.

About water and soil pollution, control on measurements results quality is being demanded by means of laboratory accreditation, as stated in Conama Resolution 420/2009 and in the draft of Conama Resolution 357/2005 revision.

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<sup>11</sup> GLP is a set of principles that assure reliability to reports issued by a certain laboratory and is applicable to studies regarding the safe use of products related to plant, animal, human health, and the environment.

However, it should be noted that Inmetro's accreditation only ensures that a laboratory has potential to provide reliable results, as observed by Jardim & Sodré in recent study [29].

Concern in ensuring reliable measurements results in the environmental field was expressed in OIML Document 12 (1986): verification of measuring instruments in the fields of environmental protection, occupational safety, and accident prevention at work and elsewhere should provide assurance of correct measurement results and improve the accuracy of these instruments.

The verification of measuring instruments in the field of environmental protection can also be of importance in connection with any legal consequences of unacceptable environmental pollution. Measuring instruments used for official purposes should meet particularly severe requirements because measurement results obtained with them may be used, where appropriate, as the basis for decisions which may involve major economic consequences for those causing the pollution [12].

## 7. CONCLUSIONS

Currently, there is a growing recognition of metrology's importance for environmental quality assurance because reliable measurements are essential when monitoring natural resources.

In Brazil, opacimeters and exhaust gas meters are regulated by Inmetro and, consequently, submitted to legal metrological control, in order to comply with Conama's demand for reliable instruments.

However, a tendency of the environmental sector demanding pollutants analyses to be performed in laboratories that are accredited by Inmetro can be noticed. This means that the control of the procedures adopted while performing the tests is chosen instead of the control focused simply in the measuring instrument.

As discussed, accreditation process provides credibility to environmental analyses results, having potential for assuring quality measurements and for complying with Conama's environmental control goals.

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