# ANEXOS

# 7. ANEXOS

## Anexo 1: Especificación de equipos utilizados.

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Estufa**<br> | **Modelo:** Q314M243<br>**Marca:** Quimis<br>**Rango de temperatura:** 10-300 °C<br>**Potencia:** 2000 W<br>**Voltaje:** 220 V<br>**Frecuencia:** 50/60 Hz |

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Balanza digital**<br> | **Modelo:**<br>**Marca:** RADWAG<br>**Rango de temperatura**: 10-40 °C<br>**Peso máximo:** 310 g<br>**Peso mínimo:** 10 mg<br>**Error de precisión:** 0,01 g<br>**Voltaje:** 240 V<br>**Frecuencia:** 50/60 Hz |

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Secador de infrarrojos**<br> | **Modelo:** Eurotherm<br>**Marca:** Sartorious<br>**Voltaje:** 220 V<br>**Frecuencia:** 50 Hz |

| **EQUIPO** | **ESPECIFICACIONES** |
|---|---|
| **Molino de discos**<br> | **Modelo:** 4-E avakercity<br>**Marca:** Westinghouse<br>**Potencia:** 1,4 kW<br>**Voltaje:** 220 V<br>**Amper:** 6,3 A<br>**RPM:** 89 |

| **EQUIPO** | **ESPECIFICACIONES** |
|---|---|
| **Tamiz**<br> | **Marca:** Orto Alresa<br>**Potencia:** 80 W<br>**Voltaje:** 230 V<br>**Frecuencia:** 50 Hz<br>**R.P.M:** 2500 |

| **EQUIPO** | **ESPECIFICACIONES** |
|---|---|
| **Mufla**<br> | **Modelo:** FB1410M<br>**Marca:** Thomas Scientific<br>**Rango de temperatura:** 100° a 1100°C<br>**Potencia:** 1,52 kW<br>**Voltaje:** 240 V<br>**Frecuencia:** 50/60 Hz |

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Bomba de vacío**<br> | **Marca:** Sartorius stedim<br>**Potencia:** 0,13 kW<br>**Voltaje:** 230 V<br>**Amper:** 2,0 A<br>**Frecuencia:** 50/60 Hz |

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Calentador**<br> | **Marca:** Leybolo-Heraeus<br>**Potencia:** 640 W<br>**Voltaje:** 230 V<br>**Amper:** 2,7 A<br>**Frecuencia:** 50/60 Hz |

| EQUIPO | ESPECIFICACIONES |
|---|---|
| **Espectrofotómetro**<br> | **Modelo:** VR-2000<br>**Marca:** J.P. Selecta s.a.<br>**Rango de onda:** 320-1000<br>**Potencia:** 25 W<br>**Voltaje:** 230/240 V<br>**Amper:** 112 mA<br>**Frecuencia:** 50/60 Hz |

| **Equipo** | **ESPECIFICACIÓN** |
|---|---|
| **pHmetro**<br> | **Modelo:** HI 4221<br>**Marca:** Hanna<br>**Rango:** -20.0 a 120 °C<br>**Precisión:** ± 0.2ºC (Excluyendo error de sonda)<br>**Temperatura de operación:** 0°C - 50°C |

| **EQUIPO** | **ESPECIFICACIONES** |
|---|---|
| **Agitador Magnético**<br> | **Modelo:** IKA 3581200<br>**Marca:** IKAMAG<br>**Velocidad:** 100-1500 rpm<br>**Rango de temperatura :** 50-500 °C<br>**Potencia:** 1000 W |

# Anexo 2: Metodo de prueba estandar para la determinacion de índice de yodo del carbón activado (ASTM D 4607-14)

**Designation: D4607 – 14**

## Standard Test Method for Determination of Iodine Number of Activated Carbon[1]

This standard is issued under the fixed designation D4607; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the relative activation level of unused or reactivated carbons by adsorption of iodine from aqueous solution. The amount of iodine absorbed (in milligrams) by 1 g of carbon using test conditions listed herein is called the iodine number.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

### 2. Referenced Documents

2.1 *ASTM Standards:*[2]

C819 Test Method for Specific Surface Area of Carbon or Graphite (Withdrawn 1987)[3]
D1193 Specification for Reagent Water
D2652 Terminology Relating to Activated Carbon
D2867 Test Methods for Moisture in Activated Carbon
D3860 Practice for Determination of Adsorptive Capacity of Activated Carbon by Aqueous Phase Isotherm Technique
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E287 Specification for Laboratory Glass Graduated Burets
E288 Specification for Laboratory Glass Volumetric Flasks
E300 Practice for Sampling Industrial Chemicals

2.2 *NIST Publication:*

Circular 602—Testing of Glass Volumetric Apparatus[4]

### 3. Summary of Test Method

3.1 This test method is based upon a three-point adsorption isotherm (see Practices D3860). A standard iodine solution is treated with three different weights of activated carbon under specified conditions. The carbon treated solutions are filtered to separate the carbon from the treated iodine solution (filtrate). Iodine remaining in the filtrate is measured by titration. The amount of iodine removed per gram of carbon is determined for each carbon dosage and the resulting data used to plot an adsorption isotherm. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02 $N$ is reported as the iodine number.

3.2 Iodine concentration in the standard solution affects the capacity of an activated carbon for iodine adsorption. Therefore, the normality of the standard iodine solution must be maintained at a constant value ($0.100 \pm 0.001\ N$) for all iodine number measurements.

3.3 The apparatus required consists of various laboratory glassware used to prepare solutions and contact carbon with the standard iodine solution. Filtration and titration equipment are also required.

### 4. Significance and Use

4.1 The iodine number is a relative indicator of porosity in an activated carbon. It does not necessarily provide a measure of the carbon's ability to absorb other species. Iodine number may be used as an approximation of surface area for some types of activated carbons (see Test Method C819). However, it must be realized that any relationship between surface area and iodine number cannot be generalized. It varies with changes in carbon raw material, processing conditions, and pore volume distribution (see Definitions D2652).

4.2 The presence of adsorbed volatiles, sulfur; and water extractables may affect the measured iodine number of an activated carbon.

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[1] This test method is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.02 on Liquid Phase Evaluation.

Current edition approved March 1, 2014. Published May 2014. Originally approved in 1986. Last previous edition approved in 2006 as D4607 – 94 (Reapproved 2011). DOI: 10.1520/D4607-14.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

## 5. Apparatus

NOTE 1—All volumetric measuring equipment should meet or exceed the requirements of NIST Circular 602. Volumetric glassware meeting these specifications is generally designated as "Class A". See also Specifications E287 and E288.

5.1 *Analytical Balance,* accuracy ±0.0001 g.

5.2 *Buret,* 10-mL capacity or 5-mL precision buret.

5.3 *Flasks,* Erlenmeyer 250-mL capacity with ground glass stoppers.

5.4 *Flask,* Erlenmeyer wide-mouthed, 250-mL capacity.

5.5 *Beakers,* assorted sizes.

5.6 *Bottles,* amber, for storage of iodine and thiosulfate solutions.

5.7 *Funnels,* 100-mm top inside diameter.

5.8 *Filter Paper,* 18.5-cm prefolded paper, Whatman No. 2V or equivalent.

5.9 *Pipets,* volumetric type, 5.0, 10.0, 25.0, 50.0, and 100.0-mL capacity.

5.10 *Volumetric Flasks,* 1 L.

5.11 *Graduated Cylinders,* 100 mL and 500 mL.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—References to water shall be understood to mean reagent water conforming to Specification D1193 for Type II reagent water.

6.3 *Hydrochloric Acid,* concentrated.

6.4 *Sodium Thiosulfate,* ($Na_2S_2O_3 \cdot 5H_2O$).

6.5 *Iodine,* United States Pharmacopeia, resublimed crystals.

6.6 *Potassium Iodide.*

6.7 *Potassium Iodate,* primary standard.

6.8 *Starch,* soluble potato or arrowroot.

6.9 *Sodium Carbonate.*

## 7. Hazards

7.1 Several potential hazards are associated with conducting this test procedure. It is not the purpose of this standard to address all potential health and safety hazards encountered with its use. The user is responsible for establishing appropriate health and safety practices before use of this test procedure. Determine the applicability of federal and state regulations before attempting to use this test method.

7.2 Personnel conducting the iodine number procedure should be aware of potential safety and health hazards associated with the chemicals used in this procedure. The "Material Safety Data Sheet" (MSDS) for each reagent listed in Section 6 should be read and understood. Special precautions to be taken during use of each reagent are included on the "Material Safety Data Sheet" (MSDS). First aid procedures for contact with a chemical are also listed on its "MSDS." A "Material Safety Data Sheet" for each reagent may be obtained from the manufacturer. Other safety and health hazard information on reagents used in this procedure is available.[6,7,8]

7.3 Careful handling and good laboratory technique should always be used when working with chemicals. Avoid contact with hydrochloric acid or acid vapor. Care should also be taken to prevent burns during heating of various solutions during this test procedure.

7.4 The user of this test method should comply with federal, state, and local regulations for safe disposal of all samples and reagents used.

## 8. Preparation of Solutions

8.1 *Hydrochloric Acid Solution* (5 % by weight)—Add 70 mL of concentrated hydrochloric acid to 550 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

8.2 *Sodium Thiosulfate* (0.100 *N*)—Dissolve 24.820 g of sodium thiosulfate in approximately 75 ± 25 mL of freshly boiled distilled water. Add 0.10 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Quantitatively transfer the mixture to a 1-L volumetric flask and dilute to the mark. Allow the solution to stand at least 4 days before standardizing. The solution should be stored in an amber bottle.

8.3 *Standard Iodine Solution* (0.100 ± 0.001 *N*)—Weigh 12.700 g of iodine and 19.100 g of potassium iodide (KI) into a beaker. Mix the dry iodine and potassium iodide. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increments of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the solution to stand a minimum of 4 h to ensure that all crystals are thoroughly dissolved. Occasional stirring during this 4-h period will aid in the dissolution. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. It is important that the standard iodine solution has an iodide-to-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

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[5] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

[6] The "Chemical Safety Data Sheet" for the subject chemical is available from the Manufacturing Chemists Association, Washington, DC.

[7] Sax, N. I., *Dangerous Properties of Industrial Materials* , 4th edition, 1975, Van Nostrand Reinhold Company, New York, NY.

[8] *NIOSH/OSHA Pocket Guide to Chemical Hazards*, 1978, U.S. Department of Labor, Occupational Safety and Health Administration, Washington, DC. Available from U.S. Government Printing Office, Washington, DC.

8.4 *Potassium Iodate Solution* (0.1000 *N*)—Dry 4 or more grams of primary standard grade potassium iodate ($KIO_3$) at 110 ± 5°C for 2 h and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 mL of distilled water. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

8.5 *Starch Solution*—Mix 1.0 ± 0.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 ± 5 mL of water while stirring to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water and boil for 4 to 5 min. This solution should be made fresh daily.

## 9. Standardization of Solutions

9.1 *Standardization of 0.100 N Sodium Thiosulfate* —Pipet 25.0 mL of potassium iodate ($KIO_3$) solution from 8.4 into a 250-mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and shake the flask to dissolve the potassium iodide crystals. Pipet 5.0 mL of concentrated hydrochloric acid into the flask. Titrate the free iodine with sodium thiosulfate solution until a light yellow color is observed in the flask. Add a few drops of starch indicator (8.5) and continue the titration dropwise until one drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_1 = (P \cdot R)/S \tag{1}$$

where:

$N_1$ = sodium thiosulfate, *N*,
$P$ = potassium iodate, mL,
$R$ = potassium iodate, *N*, and
$S$ = sodium thiosulfate, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*.

9.2 *Standardization of 0.100 ± 0.001 N Iodine Solution*—Pipet 25.0 mL of iodine solution (8.3) into a 250-mL wide-mouthed Erlenmeyer flask. Titrate with standardized sodium thiosulfate (9.1) until the iodine solution is a light yellow color. Add a few drops of starch indicator and continue titration dropwise until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_2 = (S \cdot N_1)/I \tag{2}$$

where:

$N_2$ = iodine, *N*,
$S$ = sodium thiosulfate, mL,
$N_1$ = sodium thiosulfate, *N*, and
$I$ = iodine, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*. The iodine solution concentration must be 0.100 ± 0.001 *N*. If this requirement is not met, repeat 8.3 and 9.2.

## 10. Procedure

10.1 The procedure applies to either powdered or granular activated carbon. When granular carbon is to be tested, grind a representative sample (see Practice E300) of carbon until 60 wt % (or more will pass through a 325-mesh screen) and 95 wt % or more will pass through a 100-mesh screen (U.S. sieve series, see Specification E11). Carbon received in the powdered form may need additional grinding to meet the particle size requirement given above.

10.2 Dry the ground carbon from 10.1 in accordance with Test Method D2867. Cool the dry carbon to room temperature in a desiccator.

10.3 Determination of iodine number requires an estimation of three carbon dosages. Section 11.4 describes how to estimate the carbon dosages to be used. After estimating carbon dosages, weigh three appropriate amounts of dry carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, dry 250-mL Erlenmeyer flask equipped with a ground glass stopper.

10.4 Pipet 10.0 mL of 5 wt % hydrochloric acid solution into each flask containing carbon. Stopper each flask and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30 ± 2 s to remove any sulfur which may interfere with the test results. Remove the flasks from the hot plate and cool to room temperature.

10.5 Pipet 100.0 mL of 0.100 *N* iodine solution into each flask. Standardize the iodine solution just prior to use. Stagger the addition of iodine to the three flasks so that no delays are encountered in handling. Immediately stopper the flasks, and shake the contents vigorously for 30 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper (Whatman No. 2V or equivalent) into a beaker. Filtration equipment must be prepared in advance so no delay is encountered in filtering the samples.

10.6 For each filtrate, use the first 20 to 30 mL to rinse a pipet. Discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker and pipet 50.0 mL of each filtrate into a clean 250-mL Erlenmeyer flask. Titrate each filtrate with standardized 0.100 *N* sodium thiosulfate solution until the solution is a pale yellow. Add 2 mL of the starch indicator solution and continue the titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

## 11. Calculation

11.1 The capacity of a carbon for any adsorbate is dependent upon the concentration of the adsorbate in solution. The concentrations of the standard iodine solution and filtrates must be specified or known. This is necessary to determine an appropriate carbon weight to produce final concentrations agreeing with the definition of iodine number. The amount of carbon sample to be used in the determination is governed by the activity of the carbon. If filtrate normalities (*C*) are not within the range of 0.008 *N* to 0.040 *N*, repeat the procedure using different carbon weights.

11.2 Two calculations are required for each carbon dosage, as *X/M* and *C*.

11.2.1 To calculate the value of *X/M* , first derive the following values:

$$A = (N_2)(12693.0)$$

where:

$N_2$ = iodine, *N* (from 9.2).

$$B = (N_1)(126.93) \tag{4}$$

where:

$N_1$ = sodium thiosulfate, *N* (from 9.1).

$$DF = (I + H)/F \tag{5}$$

where:

*DF* = dilution factor,
*I* = iodine, mL (from 10.5),
*H* = 5 % hydrochloric acid used, mL, and
*F* = filtrate, mL.

For example, if 10 mL of HCl and 50 mL of filtrate are used: *DF* = (100 + 10)/50 = 2.2.

11.2.1.1 Calculate the value of *X/M* as follows:

$$X/M = [A - (DF)(B)(S)]/M$$

where:

*X/M* = iodine absorbed per gram of carbon, mg/g,
*S* = sodium thiosulfate, mL, and
*M* = carbon used, g.

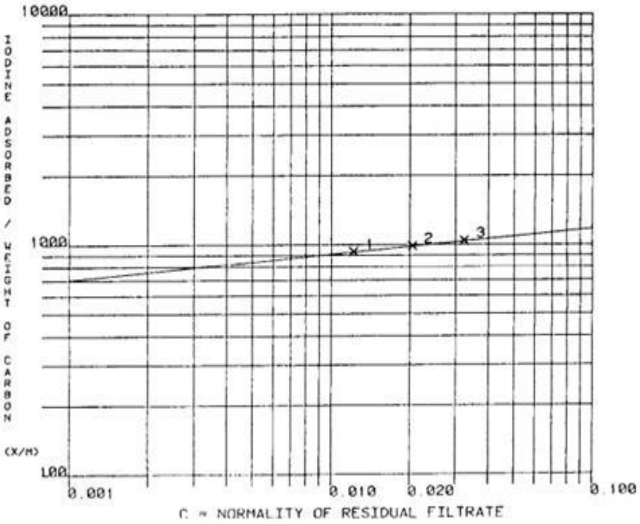
11.2.2 Calculate the value of *C* as follows:

$$C = (N_1 \cdot S)/F \tag{7}$$

where:

*C* = residual filtrate, *N*,
$N_1$ = sodium thiosulfate, *N*, and
*F* = filtrate, mL.

11.3 Using logarithmic paper, plot *X/M* (as the ordinate) versus *C* (as the abscissa) for each of the three carbon dosages (see Fig. 1). Calculate the least squares fit for the three points and plot. The iodine number is the *X/M* value at a residual



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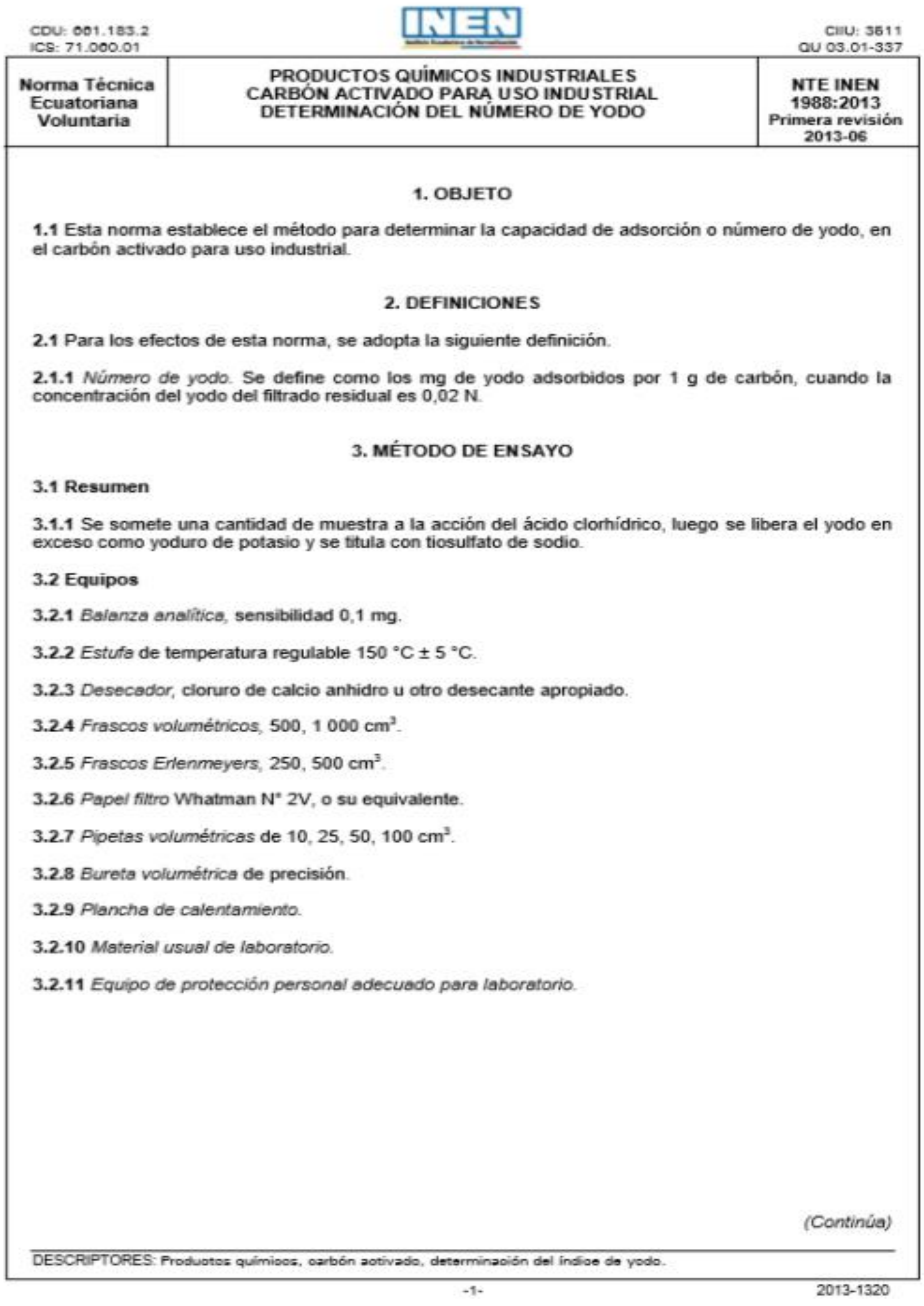
TECH. PRP

POINT 1
X/M – 932
C – 0.012
POINT 2
X/M – 984
C – 0.020
POINT 3
X/M – 1040
C – 0.032
IODINE NO. – 964
SLOPE – 0.111
CORR. COEF. – 0.999

**FIG. 1 Activated Carbon Iodine Adsorption Isotherm**

**Anexo 3: (NORMA NTE INEN 1988, 2013), productos químicos industriales. Carbón activado para uso industrial. Determinación del número de yodo**



CDU: 661.183.2
ICS: 71.060.01

CIIU: 3511
QU 03.01-337

| Norma Técnica Ecuatoriana Voluntaria | PRODUCTOS QUÍMICOS INDUSTRIALES<br>CARBÓN ACTIVADO PARA USO INDUSTRIAL<br>DETERMINACIÓN DEL NÚMERO DE YODO | NTE INEN 1988:2013<br>Primera revisión<br>2013-06 |
|---|---|---|

## 1. OBJETO

**1.1** Esta norma establece el método para determinar la capacidad de adsorción o número de yodo, en el carbón activado para uso industrial.

## 2. DEFINICIONES

**2.1** Para los efectos de esta norma, se adopta la siguiente definición.

**2.1.1** *Número de yodo.* Se define como los mg de yodo adsorbidos por 1 g de carbón, cuando la concentración del yodo del filtrado residual es 0,02 N.

## 3. MÉTODO DE ENSAYO

**3.1 Resumen**

**3.1.1** Se somete una cantidad de muestra a la acción del ácido clorhídrico, luego se libera el yodo en exceso como yoduro de potasio y se titula con tiosulfato de sodio.

**3.2 Equipos**

**3.2.1** *Balanza analítica,* sensibilidad 0,1 mg.

**3.2.2** *Estufa* de temperatura regulable 150 °C ± 5 °C.

**3.2.3** *Desecador,* cloruro de calcio anhidro u otro desecante apropiado.

**3.2.4** *Frascos volumétricos,* 500, 1 000 $cm^3$.

**3.2.5** *Frascos Erlenmeyers,* 250, 500 $cm^3$.

**3.2.6** *Papel filtro* Whatman N° 2V, o su equivalente.

**3.2.7** *Pipetas volumétricas* de 10, 25, 50, 100 $cm^3$.

**3.2.8** *Bureta volumétrica* de precisión.

**3.2.9** *Plancha de calentamiento.*

**3.2.10** *Material usual de laboratorio.*

**3.2.11** *Equipo de protección personal adecuado para laboratorio.*

*(Continúa)*

DESCRIPTORES: Productos químicos, carbón activado, determinación del índice de yodo.

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### 3.3 Reactivos

**3.3.1** *Acido clorhídrico al 5% (m/v).* A 550 $cm^3$ de agua destilada, añadir 70 $cm^3$ de ácido clorhídrico concentrado, d =1,19 (ver nota 1).

**3.3.2** *Solución estándar de Tiosulfato de sodio estándar 0,1 N.* Disolver 24,82 g de tiosulfato de sodio, para análisis, $Na_2S_2O_3 \cdot 5H_2O$, en 1 litro de agua destilada fresca y hervida. Añadir unas pocas gotas de cloroformo para minimizar la descomposición bacteriana de la solución de tiosulfato (ver anexo A).

**3.3.3** *Solución estándar de yodo 0,1 N.* Disolver 12,7 g de yodo, ($I_2$) y 19,1 g de yoduro de potasio en aproximadamente 25 $cm^3$ de agua destilada. Dejar la solución en reposo durante 4 h, agitando ocasionalmente. Diluir a 1 litro en un frasco aforado con agua destilada. Colocar en una botella de vidrio de color oscuro con tapa; almacenar en un lugar oscuro (ver anexo B).

**3.3.4** *Solución de almidón.* Tomar 2,5 g de almidón, añadir una pequeña cantidad de agua destilada y moler en un mortero para formar una pasta delgada. Verter en una botella de 1 litro que contenga agua destilada hirviendo, enrasar. Preservar con 1,25 g de ácido salicílico por 1 litro de solución de almidón.

### 3.4 Procedimiento

**3.4.1** Moler una muestra representativa hasta que el 95 % del carbón, pase el tamiz INEN de 45 µm. Se puede determinar la finura del carbón en base de un análisis por tamizado en húmedo.

**3.4.2** Secar una porción de muestra del carbón pulverizado, a 140 °C por 2 h o tambien110 °C por 3 h.

**3.4.3** Dependiendo del carbón usado, pesar de 1,0 g a 1,6 g de carbón pulverizado seco y colocar en un matraz Erlenmeyer de 250 $cm^3$.

**3.4.4** Añadir, 10 $cm^3$ de HCl al 5% (m/m) y dar vueltas el frasco Erlenmeyer, hasta que todo el carbón se haya humedecido.

**3.4.5** Colocar el frasco Erlenmeyer sobre una plancha de calentamiento, llevar el contenido a ebullición y mantener así durante 30 s exactamente.

**3.4.6** Dejar enfriar el Erlenmeyer y su contenido a temperatura ambiente y añadir mediante pipeta volumétrica, 100 $cm^3$ de solución estándar de yodo 0,1 N.

**3.4.7** Tapar inmediatamente el frasco Erlenmeyer y agitar vigorosamente el contenido durante 30 s, filtrar a través del papel filtro Whatman N° 2V o su equivalente.

**3.4.8** Descartar los primeros 20 $cm^3$ a 30 $cm^3$ de filtrado, y recoger el filtrado en un frasco Erlenmeyer limpio.

**3.4.9** Mezclar el filtrado en el frasco Erlenmeyer con una varilla de vidrio y pipetear 50 $cm^3$ del filtrado en un Erlenmeyer de 250 $cm^3$.

**3.4.10** Titular los 50 $cm^3$ de muestra, con una solución estándar de tiosulfato de sodio 0,1 N, hasta que el color amarillo haya desaparecido. Añadir aproximadamente 1 $cm^3$ de solución de almidón y continuar la titulación hasta que el color azul del indicador desaparezca.

**3.4.11** Anotar el volumen usado de la solución estándar de tiosulfato de sodio 0,1 N (ver nota 2).

**3.4.12** Efectuar la determinación por duplicado.

### 3.5 Cálculos

**3.5.1** El número de yodo del carbón se calcula aplicando la ecuación siguiente:

$$Número\ \ de\ \ yodo = \frac{V}{m} \times f$$

En donde

$$\frac{V}{m} = \frac{A - (2{,}2 \times B \times V_t)}{m_0}$$

$$C = \frac{N_2 \times V_t}{50}$$

$$A = N_1 \times 12693$$

$$B = N_2 \times 126{,}93$$

*V/m* = mg de yodo absorbidos por g de carbón activado
*C* = normalidad del filtrado residual (para calcular *f*)
*f* = factor de corrección (ver Anexo C)
$V_t$ = volumen utilizado de tiosulfato de sodio 0,1 N, en $cm^3$
$N_1$ = normalidad de la solución estándar de yodo 0,1 N
$N_2$ = normalidad de la solución estándar de tiosulfato de sodio 0,1 N

### 3.6 Errores de Método

**3.6.1** La diferencia entre los resultados de una determinación efectuada por duplicado no debe exceder del 5% del valor promedio, en caso contrario, debe repetirse la determinación.

### 3.7 Informe de resultados

**3.7.1** Como resultado final, debe informarse la media aritmética de los resultados de la determinación.

**3.7.2** En el informe de resultados debe indicarse el método usado y el resultado obtenido; debe mencionarse, además, cualquier condición no especificada en esta norma o considerada como opcional, así como cualquier circunstancia que pueda haber influido sobre el resultado.

**3.7.3** Debe incluirse todos los detalles para la completa identificación de la muestra.

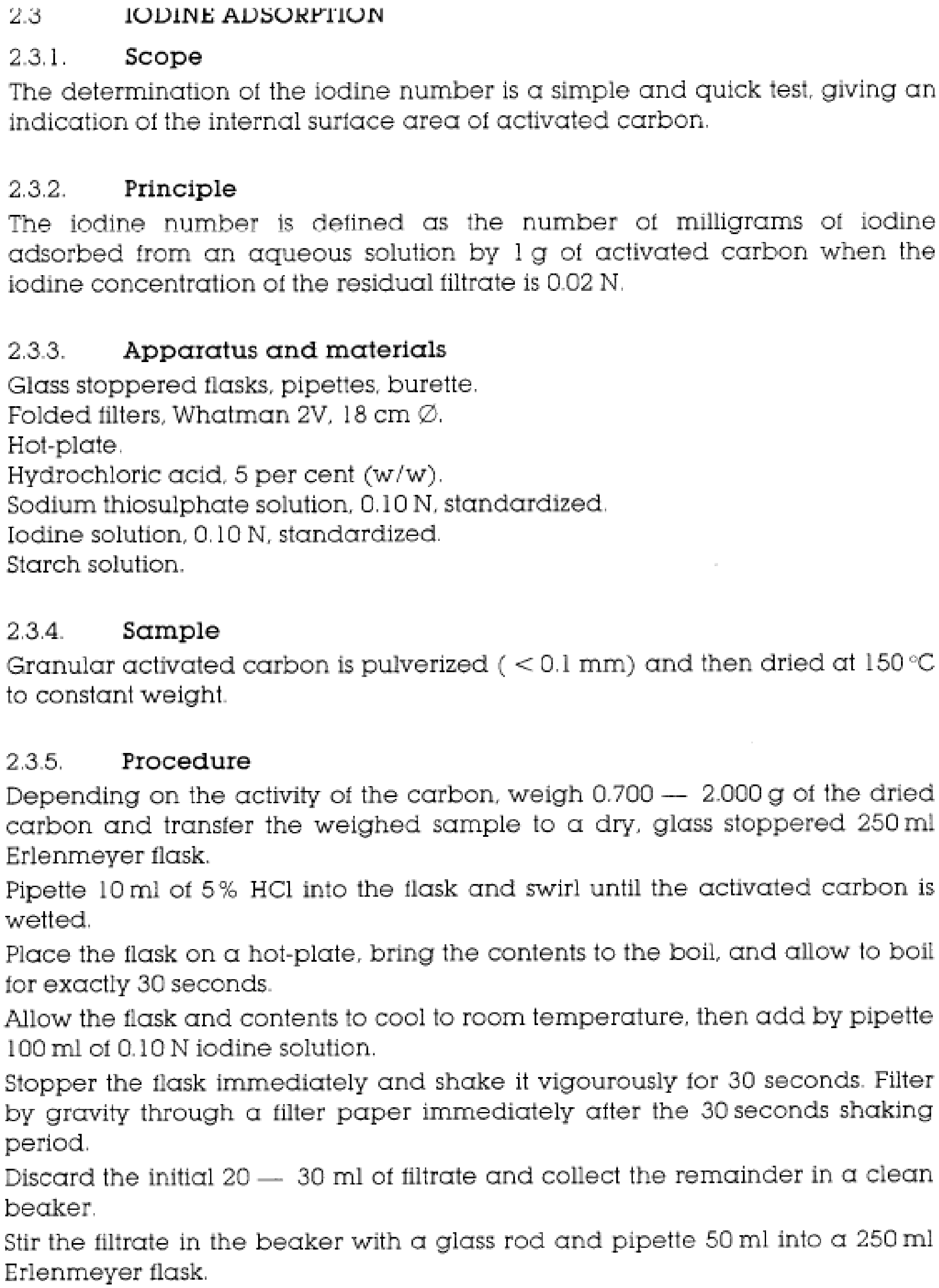
---

NOTA 2. La capacidad de un carbón para cualquier adsorción depende de la concentración de la adsorción en el medio que contiene el carbón. De esta manera la concentración del filtrado residual debe ser especificada para que los factores adecuados puedan ser aplicados para corregir la concentración para que concuerde con la definición.

La cantidad de muestra que va usarse en la determinación está regulada por la actividad del carbón. Si la normalidad "C" del filtrado residual, no está dentro del rango 0,0080 N - 0,0334 N indicado en el Anexo C, repetir el procedimiento utilizando una cantidad de muestra diferente.

Lo anterior es importante para el análisis, ya que la relación de peso de yoduro de potasio a yodo es 1,5: 1 de la solución estándar de yodo.

## Anexo 4: Determinación del índice de yodo



### 2.3 IODINE ADSORPTION

### 2.3.1. Scope

The determination of the iodine number is a simple and quick test, giving an indication of the internal surface area of activated carbon.

### 2.3.2. Principle

The iodine number is defined as the number of milligrams of iodine adsorbed from an aqueous solution by 1 g of activated carbon when the iodine concentration of the residual filtrate is 0.02 N.

### 2.3.3. Apparatus and materials

Glass stoppered flasks, pipettes, burette.
Folded filters, Whatman 2V, 18 cm ∅.
Hot-plate.
Hydrochloric acid, 5 per cent (w/w).
Sodium thiosulphate solution, 0.10 N, standardized.
Iodine solution, 0.10 N, standardized.
Starch solution.

### 2.3.4. Sample

Granular activated carbon is pulverized ( < 0.1 mm) and then dried at 150 °C to constant weight.

### 2.3.5. Procedure

Depending on the activity of the carbon, weigh 0.700 — 2.000 g of the dried carbon and transfer the weighed sample to a dry, glass stoppered 250 ml Erlenmeyer flask.

Pipette 10 ml of 5% HCl into the flask and swirl until the activated carbon is wetted.

Place the flask on a hot-plate, bring the contents to the boil, and allow to boil for exactly 30 seconds.

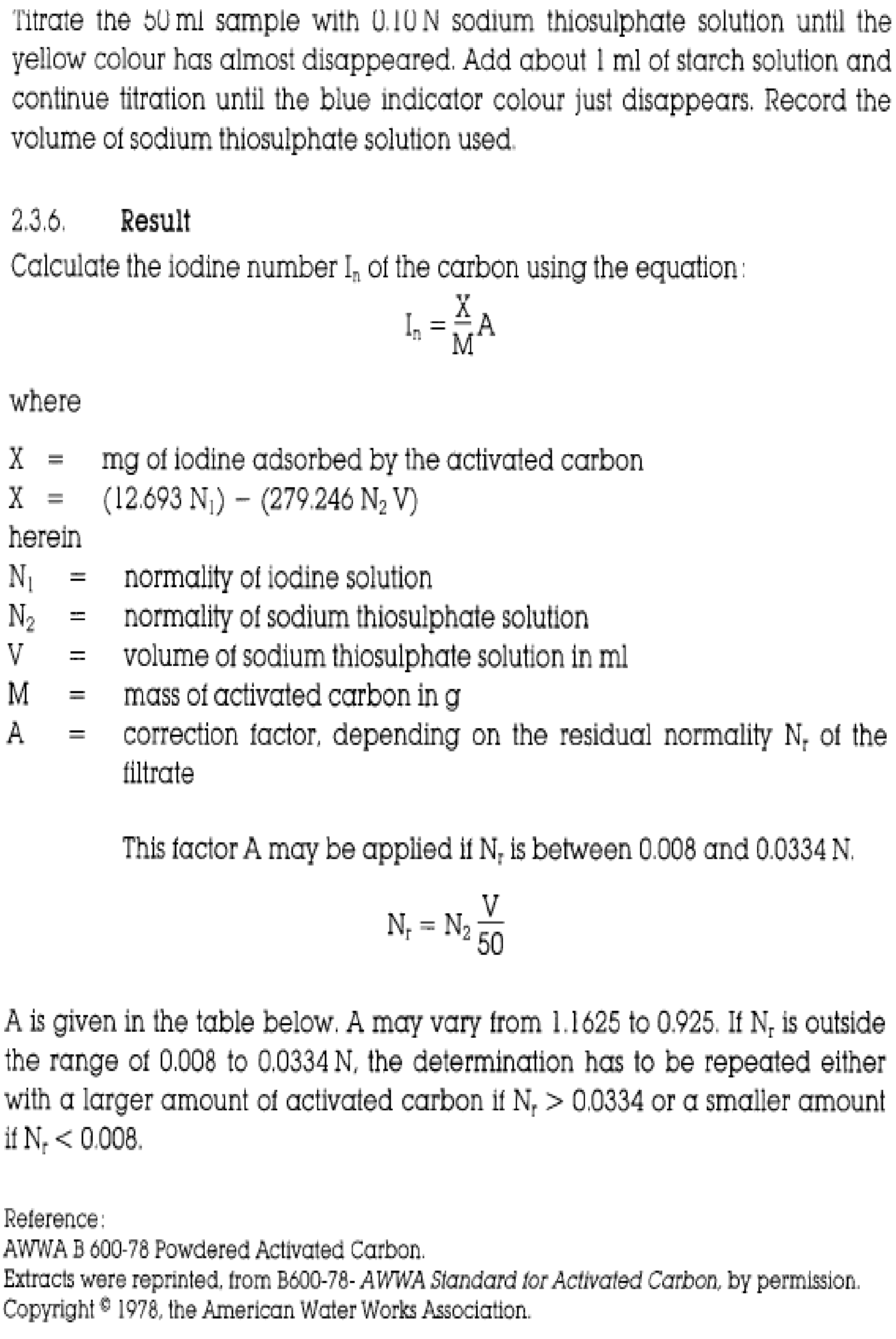
Allow the flask and contents to cool to room temperature, then add by pipette 100 ml of 0.10 N iodine solution.

Stopper the flask immediately and shake it vigourously for 30 seconds. Filter by gravity through a filter paper immediately after the 30 seconds shaking period.

Discard the initial 20 — 30 ml of filtrate and collect the remainder in a clean beaker.

Stir the filtrate in the beaker with a glass rod and pipette 50 ml into a 250 ml Erlenmeyer flask.

Fuente: Test methodos for actived carbon, 1986.



Titrate the 50 ml sample with 0.10 N sodium thiosulphate solution until the yellow colour has almost disappeared. Add about 1 ml of starch solution and continue titration until the blue indicator colour just disappears. Record the volume of sodium thiosulphate solution used.

### 2.3.6. **Result**

Calculate the iodine number $I_n$ of the carbon using the equation:

$$I_n = \frac{X}{M} A$$

where

$X$ = mg of iodine adsorbed by the activated carbon
$X$ = $(12.693\ N_1) - (279.246\ N_2 V)$
herein
$N_1$ = normality of iodine solution
$N_2$ = normality of sodium thiosulphate solution
$V$ = volume of sodium thiosulphate solution in ml
$M$ = mass of activated carbon in g
$A$ = correction factor, depending on the residual normality $N_r$ of the filtrate

This factor A may be applied if $N_r$ is between 0.008 and 0.0334 N.

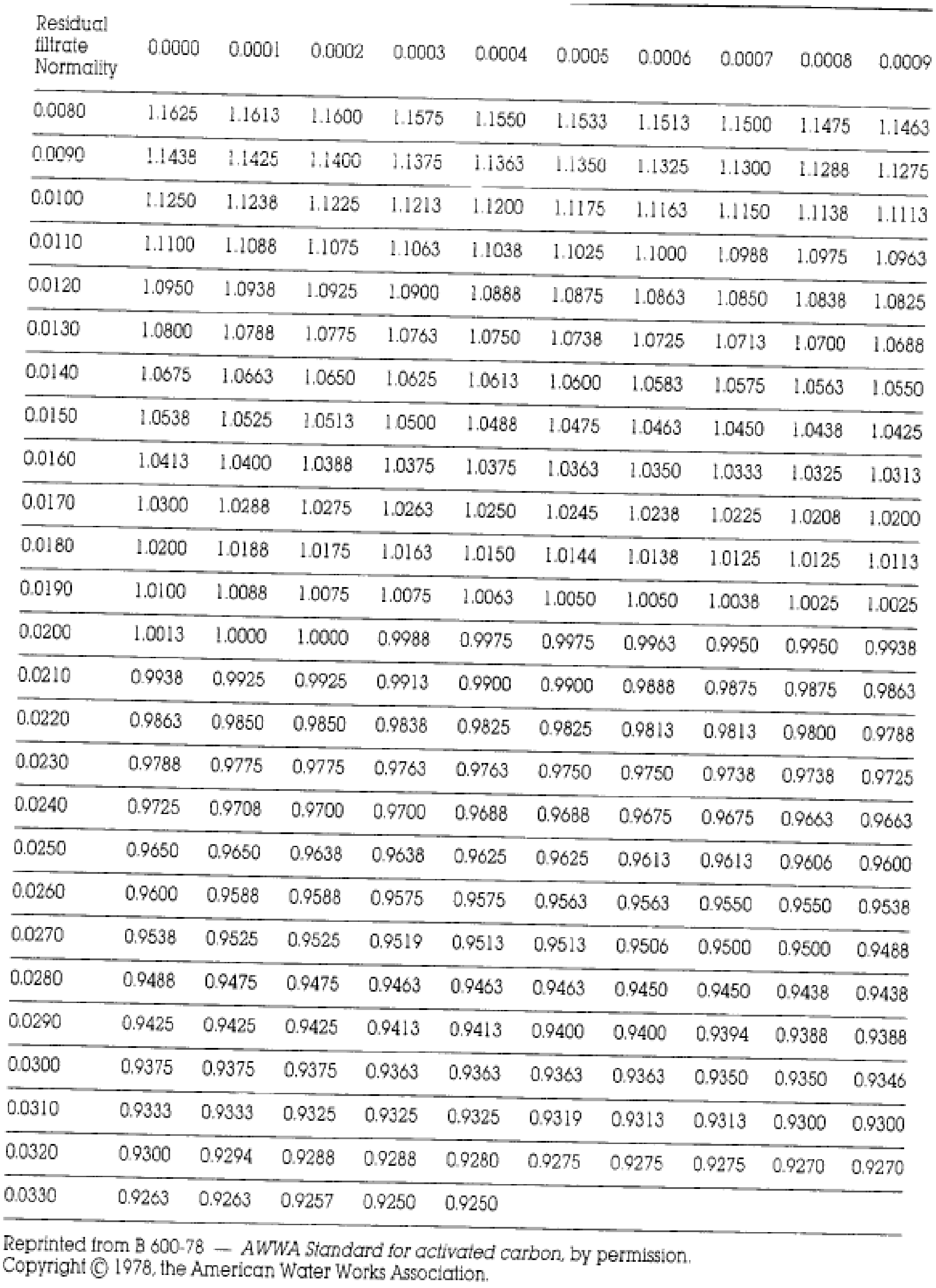
$$N_r = N_2 \frac{V}{50}$$

A is given in the table below. A may vary from 1.1625 to 0.925. If $N_r$ is outside the range of 0.008 to 0.0334 N, the determination has to be repeated either with a larger amount of activated carbon if $N_r > 0.0334$ or a smaller amount if $N_r < 0.008$.

Reference:
AWWA B 600-78 Powdered Activated Carbon.
Extracts were reprinted, from B600-78- *AWWA Standard for Activated Carbon*, by permission.

Fuente: Test methodos for actived carbon, 1986.

## Anexo 5: Tabla de factor de corrección para el número de yodo

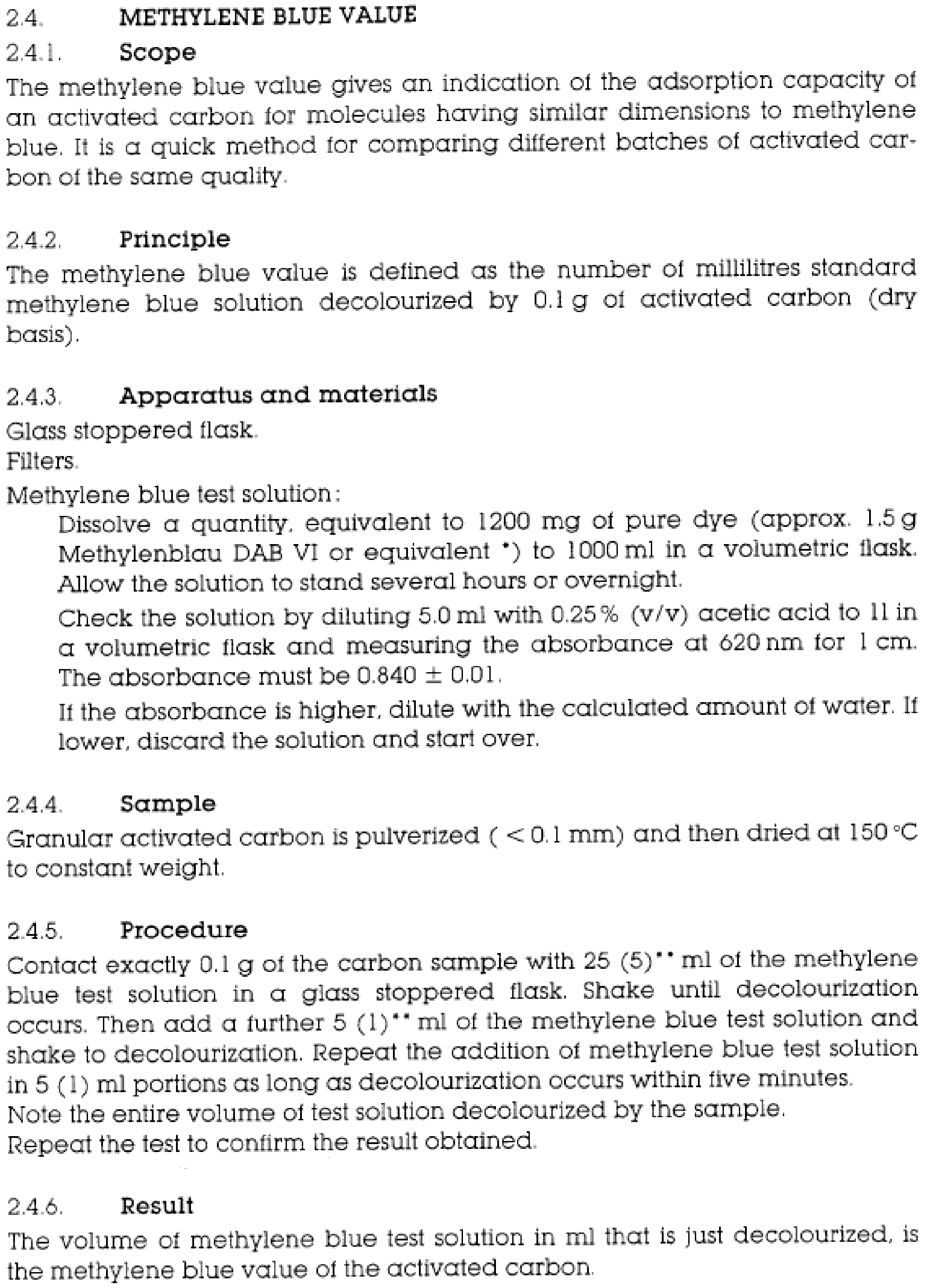


| Residual filtrate Normality | 0.0000 | 0.0001 | 0.0002 | 0.0003 | 0.0004 | 0.0005 | 0.0006 | 0.0007 | 0.0008 | 0.0009 |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.0080 | 1.1625 | 1.1613 | 1.1600 | 1.1575 | 1.1550 | 1.1533 | 1.1513 | 1.1500 | 1.1475 | 1.1463 |
| 0.0090 | 1.1438 | 1.1425 | 1.1400 | 1.1375 | 1.1363 | 1.1350 | 1.1325 | 1.1300 | 1.1288 | 1.1275 |
| 0.0100 | 1.1250 | 1.1238 | 1.1225 | 1.1213 | 1.1200 | 1.1175 | 1.1163 | 1.1150 | 1.1138 | 1.1113 |
| 0.0110 | 1.1100 | 1.1088 | 1.1075 | 1.1063 | 1.1038 | 1.1025 | 1.1000 | 1.0988 | 1.0975 | 1.0963 |
| 0.0120 | 1.0950 | 1.0938 | 1.0925 | 1.0900 | 1.0888 | 1.0875 | 1.0863 | 1.0850 | 1.0838 | 1.0825 |
| 0.0130 | 1.0800 | 1.0788 | 1.0775 | 1.0763 | 1.0750 | 1.0738 | 1.0725 | 1.0713 | 1.0700 | 1.0688 |
| 0.0140 | 1.0675 | 1.0663 | 1.0650 | 1.0625 | 1.0613 | 1.0600 | 1.0583 | 1.0575 | 1.0563 | 1.0550 |
| 0.0150 | 1.0538 | 1.0525 | 1.0513 | 1.0500 | 1.0488 | 1.0475 | 1.0463 | 1.0450 | 1.0438 | 1.0425 |
| 0.0160 | 1.0413 | 1.0400 | 1.0388 | 1.0375 | 1.0375 | 1.0363 | 1.0350 | 1.0333 | 1.0325 | 1.0313 |
| 0.0170 | 1.0300 | 1.0288 | 1.0275 | 1.0263 | 1.0250 | 1.0245 | 1.0238 | 1.0225 | 1.0208 | 1.0200 |
| 0.0180 | 1.0200 | 1.0188 | 1.0175 | 1.0163 | 1.0150 | 1.0144 | 1.0138 | 1.0125 | 1.0125 | 1.0113 |
| 0.0190 | 1.0100 | 1.0088 | 1.0075 | 1.0075 | 1.0063 | 1.0050 | 1.0050 | 1.0038 | 1.0025 | 1.0025 |
| 0.0200 | 1.0013 | 1.0000 | 1.0000 | 0.9988 | 0.9975 | 0.9975 | 0.9963 | 0.9950 | 0.9950 | 0.9938 |
| 0.0210 | 0.9938 | 0.9925 | 0.9925 | 0.9913 | 0.9900 | 0.9900 | 0.9888 | 0.9875 | 0.9875 | 0.9863 |
| 0.0220 | 0.9863 | 0.9850 | 0.9850 | 0.9838 | 0.9825 | 0.9825 | 0.9813 | 0.9813 | 0.9800 | 0.9788 |
| 0.0230 | 0.9788 | 0.9775 | 0.9775 | 0.9763 | 0.9763 | 0.9750 | 0.9750 | 0.9738 | 0.9738 | 0.9725 |
| 0.0240 | 0.9725 | 0.9708 | 0.9700 | 0.9700 | 0.9688 | 0.9688 | 0.9675 | 0.9675 | 0.9663 | 0.9663 |
| 0.0250 | 0.9650 | 0.9650 | 0.9638 | 0.9638 | 0.9625 | 0.9625 | 0.9613 | 0.9613 | 0.9606 | 0.9600 |
| 0.0260 | 0.9600 | 0.9588 | 0.9588 | 0.9575 | 0.9575 | 0.9563 | 0.9563 | 0.9550 | 0.9550 | 0.9538 |
| 0.0270 | 0.9538 | 0.9525 | 0.9525 | 0.9519 | 0.9513 | 0.9513 | 0.9506 | 0.9500 | 0.9500 | 0.9488 |
| 0.0280 | 0.9488 | 0.9475 | 0.9475 | 0.9463 | 0.9463 | 0.9463 | 0.9450 | 0.9450 | 0.9438 | 0.9438 |
| 0.0290 | 0.9425 | 0.9425 | 0.9425 | 0.9413 | 0.9413 | 0.9400 | 0.9400 | 0.9394 | 0.9388 | 0.9388 |
| 0.0300 | 0.9375 | 0.9375 | 0.9375 | 0.9363 | 0.9363 | 0.9363 | 0.9363 | 0.9350 | 0.9350 | 0.9346 |
| 0.0310 | 0.9333 | 0.9333 | 0.9325 | 0.9325 | 0.9325 | 0.9319 | 0.9313 | 0.9313 | 0.9300 | 0.9300 |
| 0.0320 | 0.9300 | 0.9294 | 0.9288 | 0.9288 | 0.9280 | 0.9275 | 0.9275 | 0.9275 | 0.9270 | 0.9270 |
| 0.0330 | 0.9263 | 0.9263 | 0.9257 | 0.9250 | 0.9250 | | | | | |

Reprinted from B 600-78 — *AWWA Standard for activated carbon*, by permission.

Fuente: Test methodos for actived carbon, 1986.

## Anexo 6: Determinacion de indice de azul de metileno



### 2.4. METHYLENE BLUE VALUE

#### 2.4.1. Scope

The methylene blue value gives an indication of the adsorption capacity of an activated carbon for molecules having similar dimensions to methylene blue. It is a quick method for comparing different batches of activated carbon of the same quality.

#### 2.4.2. Principle

The methylene blue value is defined as the number of millilitres standard methylene blue solution decolourized by 0.1 g of activated carbon (dry basis).

#### 2.4.3. Apparatus and materials

Glass stoppered flask.
Filters.
Methylene blue test solution:

> Dissolve a quantity, equivalent to 1200 mg of pure dye (approx. 1.5 g Methylenblau DAB VI or equivalent *) to 1000 ml in a volumetric flask. Allow the solution to stand several hours or overnight.
>
> Check the solution by diluting 5.0 ml with 0.25% (v/v) acetic acid to 1 l in a volumetric flask and measuring the absorbance at 620 nm for 1 cm. The absorbance must be 0.840 ± 0.01.
>
> If the absorbance is higher, dilute with the calculated amount of water. If lower, discard the solution and start over.

#### 2.4.4. Sample

Granular activated carbon is pulverized ( < 0.1 mm) and then dried at 150 °C to constant weight.

#### 2.4.5. Procedure

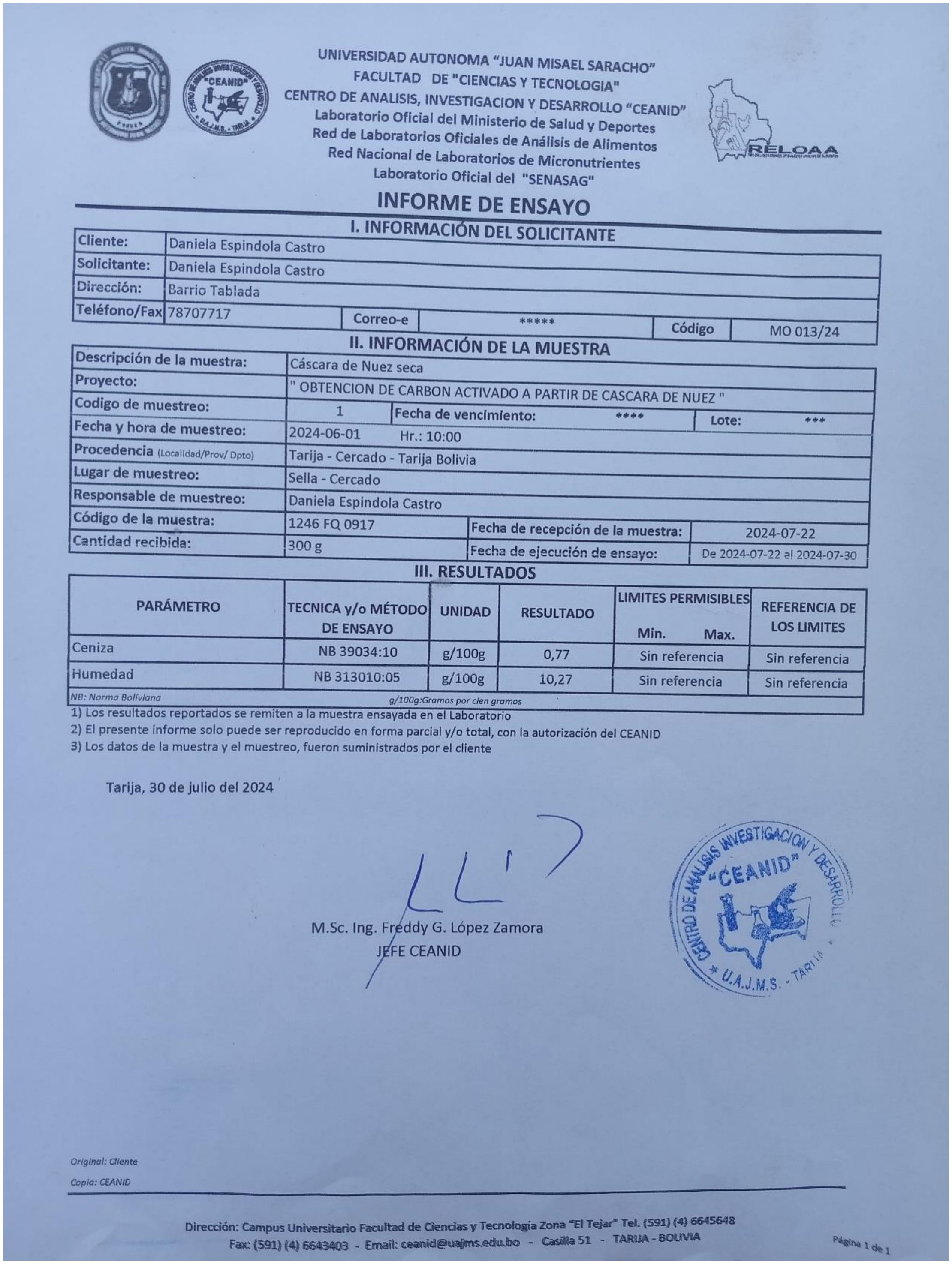
Contact exactly 0.1 g of the carbon sample with 25 (5)** ml of the methylene blue test solution in a glass stoppered flask. Shake until decolourization occurs. Then add a further 5 (1)** ml of the methylene blue test solution and shake to decolourization. Repeat the addition of methylene blue test solution in 5 (1) ml portions as long as decolourization occurs within five minutes.
Note the entire volume of test solution decolourized by the sample.
Repeat the test to confirm the result obtained.

#### 2.4.6. Result

The volume of methylene blue test solution in ml that is just decolourized, is the methylene blue value of the activated carbon.

Fuente: Test methodos for actived carbon, 1986.

## Anexo 7: Resultados del CEANID de contenido de cenizas de la cáscara de nuez.



UNIVERSIDAD AUTONOMA "JUAN MISAEL SARACHO"
FACULTAD DE "CIENCIAS Y TECNOLOGIA"
CENTRO DE ANALISIS, INVESTIGACION Y DESARROLLO "CEANID"
Laboratorio Oficial del Ministerio de Salud y Deportes
Red de Laboratorios Oficiales de Análisis de Alimentos
Red Nacional de Laboratorios de Micronutrientes
Laboratorio Oficial del "SENASAG"

**INFORME DE ENSAYO**

**I. INFORMACIÓN DEL SOLICITANTE**

| | | | | | |
|---|---|---|---|---|---|
| **Cliente:** | Daniela Espindola Castro | | | | |
| **Solicitante:** | Daniela Espindola Castro | | | | |
| **Dirección:** | Barrio Tablada | | | | |
| **Teléfono/Fax** | 78707717 | **Correo-e** | ***** | **Código** | MO 013/24 |

**II. INFORMACIÓN DE LA MUESTRA**

| | | | | | |
|---|---|---|---|---|---|
| **Descripción de la muestra:** | Cáscara de Nuez seca | | | | |
| **Proyecto:** | " OBTENCION DE CARBON ACTIVADO A PARTIR DE CASCARA DE NUEZ " | | | | |
| **Codigo de muestreo:** | 1 | **Fecha de vencimiento:** | **** | **Lote:** | *** |
| **Fecha y hora de muestreo:** | 2024-06-01 Hr.: 10:00 | | | | |
| **Procedencia** (Localidad/Prov/ Dpto) | Tarija - Cercado - Tarija Bolivia | | | | |
| **Lugar de muestreo:** | Sella - Cercado | | | | |
| **Responsable de muestreo:** | Daniela Espindola Castro | | | | |
| **Código de la muestra:** | 1246 FQ 0917 | **Fecha de recepción de la muestra:** | 2024-07-22 | | |
| **Cantidad recibida:** | 300 g | **Fecha de ejecución de ensayo:** | De 2024-07-22 al 2024-07-30 | | |

**III. RESULTADOS**

| PARÁMETRO | TECNICA y/o MÉTODO DE ENSAYO | UNIDAD | RESULTADO | LIMITES PERMISIBLES Min. Max. | REFERENCIA DE LOS LIMITES |
|---|---|---|---|---|---|
| Ceniza | NB 39034:10 | g/100g | 0,77 | Sin referencia | Sin referencia |
| Humedad | NB 313010:05 | g/100g | 10,27 | Sin referencia | Sin referencia |

*NB: Norma Boliviana* *g/100g:Gramos por cien gramos*

1) Los resultados reportados se remiten a la muestra ensayada en el Laboratorio
2) El presente informe solo puede ser reproducido en forma parcial y/o total, con la autorización del CEANID
3) Los datos de la muestra y el muestreo, fueron suministrados por el cliente

Tarija, 30 de julio del 2024

M.Sc. Ing. Freddy G. López Zamora
JEFE CEANID

*Original: Cliente*
*Copia: CEANID*

Dirección: Campus Universitario Facultad de Ciencias y Tecnología Zona "El Tejar" Tel. (591) (4) 6645648
Fax: (591) (4) 6643403 - Email: ceanid@uajms.edu.bo - Casilla 51 - TARIJA - BOLIVIA

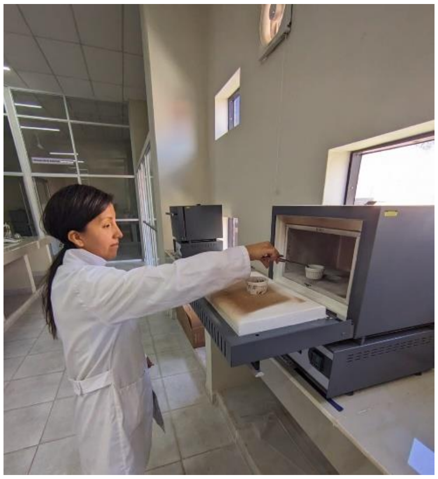
Página 1 de 1

## Anexo 8: Galería de fotos

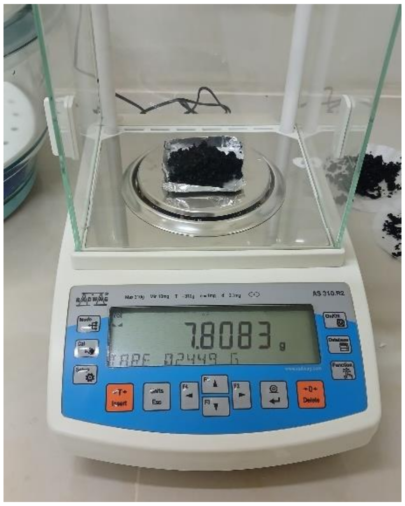
Secado

Molienda y tamizado

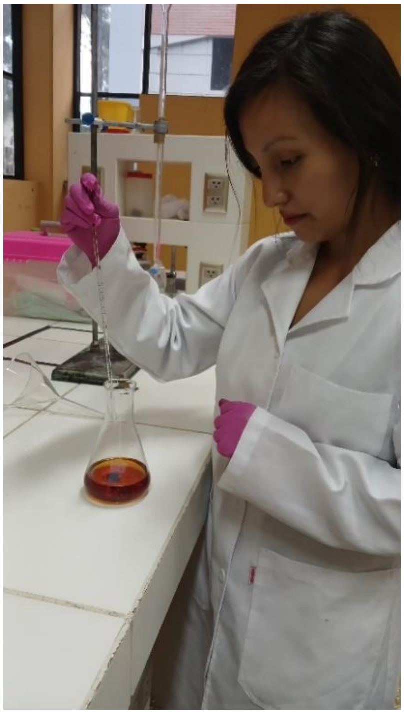
Carbonización y Activación



Secado del carbón activado

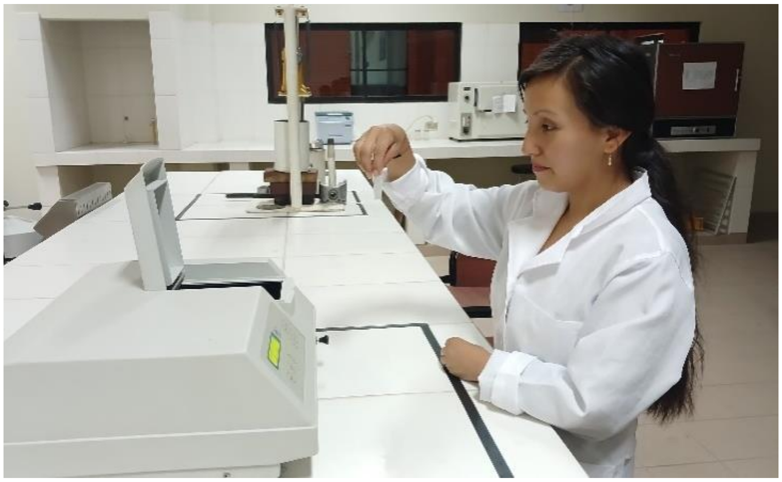


Determinacion del indice de yodo



Determinación del azul de metileno

Lectura de absorbancias



Producto final