A SYNOPSIS OF ANALYTICAL PROCEDURES
WHO ARE WE?

Imerys Graphite & Carbon has a strong tradition and history in carbon manufacturing. Its first manufacturing operation was founded in 1908. Today, Imerys Graphite & Carbon facilities produce and market a large variety of synthetic and natural graphite powders, conductive carbon blacks and water-based dispersions of consistent high quality.

Adhering to a philosophy of Total Quality Management and continuous process improvement, all Imerys Graphite & Carbon manufacturing plants comply with ISO 9001:2008. Imerys Graphite & Carbon is committed to produce highly specialized graphite and carbon materials for today’s and tomorrow’s customers needs.

Imerys Graphite & Carbon belongs to Imerys, the world leader in mineral-based specialties for industry.

WHERE ARE WE LOCATED?

With headquarters located in Switzerland, Imerys Graphite & Carbon has an international presence with production facilities and commercial offices located in key markets around the globe. The Company’s industrial and commercial activities are managed by an experienced multinational team of more than 430 employees from many countries on three continents.

For the updated list of commercial offices and distributors please visit www.imerys-graphite-and-carbon.com

WHAT IS OUR MISSION?

To promote our economic, social and cultural advancement with enthusiasm, efficiency and dynamism by offering value, reliability and quality to ensure the lasting success of our customers.

WHAT IS OUR VISION?

To be the worldwide leader and to be recognized as the reference for innovative capability in the field of carbon powder-based solutions.
Analytical procedures for TIMREX® Graphite and Coke and C-NERGY® Graphite

FOREWORD

Where possible analytical SOPs comply with International (ISO) or national (E.g. ASTM, DIN, …) norms, where it’s not possible internal standards, analytical SOPs are derived from similar or compatible norms.

Similarly for calibration and verification existing international primary or secondary standards are used, where it’s not possible internal standard are in place.

References

- ASTM MNL1: Significance of Tests for Petroleum Products (chapter Properties of Petroleum Coke, Pitch, and Manufactured Carbon and Graphite)

ASH CONTENT

Ash is assessed by gravimetric loss. A low-walled ceramic crucible is ignited at 800°C in a muffle furnace and cooled to room temperature in a desiccator. A sample of 10 g of dry powder (accuracy 0.1 mg) is weighed in a low-walled ceramic crucible. The powder is combusted at a temperature of 815°C (at least 8 h) until a constant weight. The residue corresponds to the ash content and is reported as a percentage of the initial weight of the sample.

References

- ISO 1171 Solid mineral fuels – Determination of ash
- ISO 6245 Petroleum products – Determination of ash
- ISO 8005 Carbonaceous materials used in the production of aluminium – Green and calcined coke – Determination of ash content
- DIN 51903 Prüfung von Kohlenstoffmaterialien – Bestimmung des Aschewertes – Feststoffe
- ASTM C561 Standard Test Method for Ash in a Graphite Sample

MOISTURE PER K.F.

Water content is assessed by Coulometric Karl Fischer Titration. A sample of 1 g of powder (accuracy 0.1 mg) is placed in a vial. The vial is closed and heated up, during the heating process the evaporated water is collected in an iodine-sulphur dioxide solution. The water content is determined by a coulometric titration of this solution and is reported as a percentage of the initial weight of the sample.

References

- ISO 760
- ISO 12937
- DIN 51777-1 Prüfung von Mineralöl-Kohlenwasserstoffen und Lösungsmitteln; Bestimmung des Wassergehaltes nach Karl Fischer; Direktes Verfahren
- DIN 51777-2 Prüfung von Mineralöl-Kohlenwasserstoffen und Lösungsmitteln; Bestimmung des Wassergehaltes, nach Karl Fischer, Indirektes Verfahren
- ASTM E203

TRACE ELEMENTS (SDAR – OES)

Trace elements are assessed by Spark Discharge in Argon and Optical Emission Spectrometry. A graphite powder sample is ground to a maximum particle size of 75 μm by means of a vibrated mill, then is pressed into a pill. The sample-pill is placed onto the excitation stand of the spectrometer. Under argon atmosphere spark discharges generate an emission of light, which is collected by the spectrometer. From the collected emission spectrum the concentration of the different elements is calculated and reported then in ppm.

References

- DIN 51008-1 Optische Emissionsspektrometrie (OES) – Teil 1: Begriffe für Systeme mit Funken und Niederdruckentladungen
The crystalline structure is analysed by means of an XRD. A graphite powder sample is ground to a maximum particle size of 75 μm by means of a vibrated mill, then is mixed with a silicon standard. The mix is then placed in the sample holder in thin layer of approx. 150 μm. The sample is irradiated using a CuKα X-ray beam at different angles, the diffracted beam is collected by a detector. The angular position of the peak maximum of the (002) and (004) reflection profiles are determined and the interlayer spacing (c/2) is calculated by applying the Bragg equation. The crystallite size is determined by means of the Iwashita’s algorithm, which is developed for carbon materials, and then by applying the Scherrer’s equation. The calculated values are reported in nm.

References
• ISO 20203 Carbonaceous materials used in the production of aluminium – Calcined coke – Determination of crystallite size of calcined petroleum coke by X-ray diffraction
• ASTM D5187 Standard Test Method for Determination of Crystallite Size – Lc of Calcined Petroleum Coke by X-Ray Diffraction
• P. Scherrer, Göttinger-Nachrichten 2 (1918) S.98.

The density is obtained by xylene displacement. Approx. 2.5 g (accuracy 0.1 mg) of powder is weighed in a pycnometer. Xylene is added under vacuum. After a few hours dwell time under normal pressure, the pycnometer is filled up, conditioned and weighed. The density represents the ratio between mass of graphite and the displaced volume of xylene. The value is reported in g/cm³.

References
• ISO 8004 Carbonaceous materials for the production of aluminium – Calcined coke and calcined carbon products – Determination of – the density in xylene – Pyknometric method
• ISO 9088 Carbonaceous materials used for the production of aluminium – Cathode blocks and prebaked anodes – Determination of the density in xylene by a pyknometric method
• DIN 51901 Prüfung von Kohlenstoffmaterialien – Bestimmung der Dichte nach dem Xylolverfahren – Feststoffe
• ASTM D5004 Standard Test Method for Real Density of Calcined Petroleum Coke by Xylene Displacement

The measure is carried out by measuring the adsorption-desorption isotherm of nitrogen on the surface of the material by means of the Brunauer-Emmet-Teller algorithm. A definite amount (accuracy 0.01 mg) of powder is weighed in a sample tube. Then the sample undergoes a series of heating and cooling runs, from the evolution of the pressure in the sample tube the amount of adsorbed nitrogen during the different step is calculated; from these data the specific surface is calculated and reported in m²/g.

References
• ISO 9277 Determination of the specific surface area of solids by gas adsorption – BET method
• ISO 15901-3 Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption – Part 3: Analysis of micropores by gas adsorption
• ASTM C1069 Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption

The spring-back is measured by compacting the graphite powders. About 5 g (accuracy 0.1 mg) of graphite powder is poured into a die, then the punch is inserted and the air evacuated. The sample undergoes a compression cycle by which the height of the pill is measured under full pressure and after pressure released. The spring-back is the ratio between height difference and minimal height and is reported in %.

References
• ISO 4492 Metallic powders, excluding powders for hardmetals – Determination of dimensional changes associated with compacting and sintering
• ASTM B331 Standard Test Method for Compressibility of Metal Powders in Uniaxial Compaction
APPARENT DENSITY BY SCOTT

The measurement is performed using a Scott Volumeter. The sample is free flowing through a Scott volumeter and is collected in a one cubic inch vessel and weighed to 0.1 mg accuracy. The measurement is performed three times and the average value is used. The ratio of average weight to volume (16.39 cm³) is the Scott density and is reported in g/cm³.

References
- ISO 3923-2 Metallic powders – Determination of apparent density – Part 2: Scott volumeter method

LOOSE AND TAPPED DENSITY

The measurement is performed using a graduated cylinder and a tapping unit. 100 g (accuracy 1 mg) of dry graphite powder is carefully poured into a graduated cylinder. The loose volume is recorded, then the cylinder is fixed on the tapping machine and the tapping cycle is started, at the end the tapped volume is recorded. Both densities are calculated and reported in g/cm³.

References
- ISO 567 Coke – Determination of bulk density in a small container
- ISO 787-11 General methods of test for pigments and extenders – Part 11: Determination of tamped volume and apparent density after tamping
- ISO 3953 Metallic powders – Determination of tap density
- ISO 10236 Carbonaceous materials for the production of aluminium – Green coke and calcined coke for electrodes – Determination of bulk density (tapped)
- DIN 51705 Prüfung fester Brennstoffe – Bestimmung der Schüttdichte
- DIN 51916 Prüfung von Kohlenstoffmaterialien – Bestimmung der Stampfdichte – Feststoffe
- DIN 51918 Prüfung von Kohlenstoffmaterialien – Bestimmung der Rohdichte und der offenen Porosität
- ASTM D7481 Standard Test Methods for Determining Loose and Tapped Bulk Densities of Powders using a Graduated Cylinder

PRESSED DENSITY

The measurement is performed by pressing a powder sample in a die and measuring mass and volume of the resulting specimen. About 40 g (accuracy 0.01 g) of graphite powder is poured into a die. Then a punch inserted and a compression force is applied. After the compression cycle, the specimen is weighed (accuracy 0.1 mg) and the volume calculated by the external masses (accuracy 1 μm³). The pressed density is the ratio of the mass to the volume and is reported in g/cm³.

References
- ISO 2738 Sintered metal materials, excluding hardmetals – Permeable sintered metal materials – Determination of density, oil content and open porosity
- ISO 3369 Impermeable sintered metal materials and hardmetals – Determination of density
- ISO 14427 Carbonaceous materials used in the production of aluminium – Cold and tepid ramming pastes – Preparation of unbaked test specimens and determination of apparent density after compaction
- DIN 51918 Prüfung von Kohlenstoffmaterialien – Bestimmung der Rohdichte und der offenen Porosität
- DIN 51925 Prüfung von Kohlenstoffmaterialien – Herstellung von Probekörpern aus kohlenstoffhaltigen Stampfmasse und Bestimmung ihrer Rohdichte – Binde- und Imprägniermittel
A LASER beam lights up a cell which contains the sample of graphite suspended in water, the generated diffraction pattern is collected by the system and analysed using the light scattering theory developed by Mie. The particle size distribution is calculated and reported in μm for the three quantiles: 10%, 50% and 90%.

**References**
- ISO 13320 Particle size analysis – Laser diffraction methods
- ISO 14887 Sample preparation – Dispersing procedures for powders in liquids
- ASTM D4464 Standard Test Method for Particle Size Distribution of Catalytic Material by Laser Light Scattering
- ASTM E2651 Standard Guide for Powder Particle Size Analysis

The measurement is performed using a pile of sieves with descending mesh size on a sieving machine. 100 g (accuracy 0.01 g) of graphite sample is poured onto the top screen, then a vibration cycle is started. The residue on the screens and in the bottom pan is weighed and the particle size distribution is calculated and reported in %.

**References**
- ISO 1953 Hard coal – Size analysis by sieving
- ISO 4497 Metallic powders – Determination of particle size by dry sieving
- DIN 51938 Prüfung von Kohlenstoffmaterialien; Bestimmung der Korngrößenverteilung durch Sieben; Feststoffe
- DIN 66165-2 Partikelgrößenanalyse; Siebanalyse; Durchführung
- ASTM D4749 Standard Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size
- ASTM E2651 Standard Guide for Powder Particle Size Analysis
- ASTM MNL32 Manual on Test Sieving Methods
- ASTM STP447A Manual on Test Sieving Methods

The measurement is performed using a single sieve, through which an air flow keeps the powder in constant movement and transports the fine particles through the screen. 20 g (accuracy 0.01 g) of graphite sample a sieve of desired mesh size and the sieving cycle is then started. Then the residue on the screen is weighed and the fraction is calculated and reported in %.

**References**

The measurement is performed using a single sieve, through which a water flow transports the fine particles through the screen. About 1 g of graphite powder (accuracy 0.1 mg) is dispersed in 500 ml water. In order to achieve complete de-agglomeration a wetting agent is added. The dispersion is poured into the wet sieving unit holding a pre-weighted sieve. Then equipment run is rinsed for 10 min with running tap water. The sieve is removed, dried and weighted. The amount of particles on the screen is determined and the fraction is calculated and reported in %.

**References**
- ISO 787-18 General methods of test for pigments and extenders – Part 18: Determination of residue on sieve – Mechanical flushing procedure
- ISO 23202:2006 Aluminium oxide used for the production of aluminium – Determination of particles passing a 20 micrometre aperture sieve
- ISO 24369 Fine ceramics (advanced ceramics, advanced technical ceramics) – Determination of content of coarse particles in ceramic powders by wet sieving method
- DIN 66165-2 Partikelgrößenanalyse; Siebanalyse; Durchführung
- ASTM C925 Standard Guide for Precision Electroformed Wet Sieve Analysis of Nonplastic Ceramic Powders
Where possible analytical SOPs comply with international (ISO) or national (e.g., ASTM, DIN, …) norms, where no dedicated norms exist, analytical SOPs are derived from similar or compatible norms. Similarly for calibration and verification existing international primary or secondary standards are used, where it’s not possible internal standard are in place.

**SOLID CONTENT**

The solid content is measured using a Halogen Moisture Determination thermobalance. About 2 g of the dispersion is placed on the sample holder and dried at 120 °C until a constant weight is achieved. The remaining weight is recorded and expressed as a percentage.

**References**
- ASTM D5368-13 Standard Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples

**MOISTURE**

The water content is measured by weight difference between before and after a drying cycle at 120°C. About 50 g (accuracy 0.1 mg) of powder is weighed in a glass beaker and dried at 120° C until constant weight is reached (at least 8 h). The weight loss corresponds to the water content and it is expressed as %.

**References**
- DIN 51718 Prüfung fester Brennstoffe – Bestimmung des Wassergehaltes und der Analysenfeuchtigkeit
- DIN 51904 Prüfung von Kohlenstoffmaterialien – Bestimmung des Wassergehaltes – Feststoffe
- ASTM C562 Standard Test Method for Moisture in a Graphite Sample
- ASTM D4959 Standard Test Method for Determination of Water (Moisture) Content of Soil By Direct Heating
Analytical procedures for TIMREX® Dispersions

**VISCOSITY**

Viscosity is measured in the steady state as a function of the shear rate as well as in oscillating mode as a function of the amplitude with a constant frequency. Thixotropic behaviour is measured by observing the viscosity in an oscillating test as a function of the time. A rotational rheometer with a coneplate setup is used and the measurements are performed at room temperature. About 2 g of liquid sample are put into the gap between a stationary plane and a rotating cone. A laminar flow of layers is created. The viscosity is obtained from measuring the internal resistance and a flow curve is obtained.

**References**

- ISO 2555 Plastics – Resins in the liquid state or as emulsions or dispersions – Determination of apparent viscosity by the Brookfield Test method
- ISO 3219 Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate
- ISO 6388 Surface active agents – Determination of flow properties using a rotational viscometer
- DIN 51810-1 Prüfung von Schmierstoffen – Bestimmung der Scherviskosität von Schmierfetten mit dem Rotationsviskosimeter – Teil 1: Messsystem Kegel/Platte
- DIN 53019-1 Viskosimetrie – Messung von Viskositäten und Fließkurven mit Rotationsviskosimetern – Teil 1: Grundlagen und Messgeometrie
- DIN 53019-2 Viskosimetrie – Messung von Viskositäten und Fließkurven mit Rotationsviskosimetern – Teil 2: Viskosimeterkalibrierung und Ermittlung der Messunsicherheit
- DIN 53019-3 Viskosimetrie – Messung von Viskositäten und Fließkurven mit Rotationsviskosimetern – Teil 3: Messabweichungen und Korrekturen
- ASTM D2196 Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer
- ASTM D2983-09 Standard Test Method for Low-Temperature Viscosity of Lubricants Measured by Brookfield Viscometer1, 2

**pH**

The pH is measured using a Digital pH Meter with a calibrated pH electrode. The clean electrode is immersed into the liquid. After stabilization of the reading the pH is recorded.

**References**

- ISO 10390:2005 Soil quality – Determination of pH
### ASH CONTENT

A platinum crucible is ignited for 1 h at 550°C ± 25°C in a muffle furnace. An adequate amount of carbon black is dried at 125°C for 1 h. A sample of ± 10 g of the dried carbon black is weighed into the crucible and placed in the muffle furnace for 16 h at 550°C. The residue corresponds to the ash content. It is expressed as a percentage of the initial weight of the dried sample.

**References**
- ASTM D1506

### METAL IMPURITIES

The residue obtained after the determination of the ash content is dissolved in an acid mixture and transferred to a volumetric flask and diluted to the 100 ml mark. Metal impurities are analyzed by atomizing the liquid into the Argon plasma of an ICP analyzer and determining the different components by using a spectrophotometric technique.

**References**
- IMERYS method

### HEATING LOSS (MOISTURE)

A sample of 2 g of carbon black (accuracy 0.1 mg) is placed in a dried weighing bottle. The bottle is heated for 1 h at 125°C together with a stopper. The heating loss is expressed as a percentage of the initial weight of the sample.

**References**
- ASTM D1509 (method A)

### VOLATILE MATTER

A suitable amount of pre-dried (105°C) sample is weighed in a proper vessel and placed inside a nitrogen flushed tubular furnace. The furnace is heated up to 950°C for 30 minutes. After cooling in a nitrogen atmosphere, the loss of weight of the sample is determined and expressed in percent. Afterwards, a blank determination is carried out in the same way on the treated sample, and the result subtracted.

**References**
- IMERYS method

### SULPHUR CONTENT

Carbon black is burned in a tube furnace at a minimum operating temperature of 1350°C in a stream of oxygen to oxidize the sulphur. The gas stream is passed through a cell in which sulphur dioxide is measured by an infrared (IR) absorption detector.

**References**
- ASTM D1619

### SOLVENT EXTRACTABLE

This analysis is based on solvent (toluene) extraction of an adequate amount of dried (at 125°C for 1 h) carbon black. Extraction time is 48 h (or other time if specified). After extraction the solvent is evaporated from the residue until constant weight. A blank determination on the solvent is also performed.

**References**
- ASTM D4527
Analytical procedures for ENSACO®, SUPER P® and C-NERGY® Conductive Carbon Black

<table>
<thead>
<tr>
<th>pH VALUE</th>
<th>A sample of 1.5 g of carbon black is dispersed in distilled water with the aid of a few drops of acetone and of an alternating stirring and ultrasonic treatment. The electrode of the calibrated pH-meter is placed in the slurry. After a stabilization time of 2 minutes the slurry is stirred and the pH value is recorded to the nearest 0.05 unit.</th>
</tr>
</thead>
<tbody>
<tr>
<td>References</td>
<td>• ASTM D1512 (method B)</td>
</tr>
<tr>
<td>OIL ABSORPTION NUMBER</td>
<td>Paraffin oil is added by means of a constant rate burette to a dried (1 h at 125 °C) carbon black sample in the mixer chamber of the absorptometer. As the sample absorbs the oil, the mixture changes from a free-flowing state to one of a semi-plastic agglomeration, with an accompanying increase in viscosity. This increased viscosity is transmitted to the torque-sensing system. When the viscosity reaches a predetermined torque level, the absorptometer and burette will shut off simultaneously. The volume of the added oil is read from the burette. The volume of oil per unit mass of carbon black is the oil absorption number.</td>
</tr>
<tr>
<td>References</td>
<td>• ASTM D2414</td>
</tr>
<tr>
<td>OIL ABSORPTION NUMBER OF A COMPRESSED CARBON BLACK SAMPLE</td>
<td>A sample of carbon black is compressed 4 times at a pressure of 165 MPa which is then tested in an absorptometer to determine the oil absorption number according to method ASTM D2414. The difference between the initial OAN number and the OAN number of the compressed sample reflects the stability of the structure of that sample.</td>
</tr>
<tr>
<td>References</td>
<td>• ASTM D3493</td>
</tr>
<tr>
<td>ABSORPTION STIFFNESS (AS TEST)</td>
<td>This test determines the amount of liquid (10% of acetone in water) which can be absorbed by 5 grams of carbon black. The carbon black is placed in a 500 ml Erlenmeyer flask. While shaking vigorously with a rotating motion, small quantities of liquid are added to the carbon until finally one ball is formed. At first, this ball is fragile and breaks when shaking but at the end the ball resists to fairly vigorous shaking without disintegrating. The quantity of liquid added during this time is counted and the test is expressed in ml/5g.</td>
</tr>
<tr>
<td>References</td>
<td>• IMERYS method</td>
</tr>
<tr>
<td>HYDROCHLORIC ACID ABSORPTION NUMBER</td>
<td>This test determines the amount of liquid (2N hydrochloric acid) which can be absorbed by 5 g of carbon black. The method used is the Japanese test JISK 1469-1984 whose procedure is rather similar but less practical than the one used in the AS Test method.</td>
</tr>
<tr>
<td>References</td>
<td>• IMERYS method</td>
</tr>
<tr>
<td>GRIT</td>
<td>A sample of carbon black is washed with water through a sieve of 45 μm until only coarse particles remain on the sieve. This residue is dried, weighed and the amount of residue is expressed as mg/kg (ppm) of the original sample.</td>
</tr>
<tr>
<td>References</td>
<td>• ASTM D1514</td>
</tr>
</tbody>
</table>
FINES
Weigh 20 g of carbon black into three 0.125 mm sieves, each with a receiver beneath and assemble the stack in a Ro-Tap siever. Shake for 2 minutes and weigh the carbon in the receiver to the nearest 0.1 g. Express the result in percent.

References
• ASTM D1508

POUR DENSITY OF GRANULATED CARBON BLACK
From a height no more than 50 mm above the rim, carbon black is poured in excess into the center of a tared cylindrical container having a volume of 624 ml. The surface is leveled with a single sweep and the filled container is weighed to the nearest 0.1 g. The calculated result is expressed in g/l.

References
• ASTM D 1513

SPECIFIC SURFACE AREA BY BET
The method is based on the registration of the absorption of Nitrogen at 77 K. Following the model proposed by Brunauer, Emmet and Teller (BET), the monolayer capacity can be determined. On the basis of the cross-sectional area of the Nitrogen molecule, the monolayer capacity and the weight of the sample, the specific surface can then be calculated.

References
• ASTM D3037-89

THE SPECIFIC ELECTRICAL RESISTANCE OF THE CARBON BLACK
A carbon black sample of 0.5 g is placed between two copper electrodes in a non-conductive mould and pressed by means of a pneumatic driven piston at pressures of 50 kg/cm² and 100 kg/cm² respectively. The electrical resistance and the thickness of the carbon black are measured at the two pressures and the electrical resistivity is calculated and expressed in ohm.cm.

References
• IMERYS method

ELECTRICAL RESISTIVITY MEASUREMENTS OF COMPOUNDS
In a Brabender roller mixer of 50 ml the HDPE is melted at 160 °C for 2 minutes at a rotating speed of 60 rpm. Then the carbon is added and the compounding continues for 5 minutes. The compound plate(s) is(are) pressed at 180 °C for 2 minutes applying a force of 200 kN. The measurement uses 10 samples of polymer/carbon black compounds with thickness of about 2 mm, having a minimum length of 4 cm and a width of +/- 2 cm. Each sample is measured by using a 4 point contacts method in a sample holder respecting ASTM D4496.

The electrical resistivity of the compound is the average of the 10 resistivities calculated from the electrical resistances of the samples and their geometrical shapes. If the resistance of the sample or the contact resistances are larger than +/- 1010 ohm the measurement is performed with an electrometer with respect to ASTM D257. In this case the sample should have a minimum diameter of 6.5 cm.

References
• IMERYS method