

A World First -**Verification of Petroleum Analysers** According to ISO-Standards

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The petroleum industry nowadays relies entirely on the proficiency of QC laboratories to ensure the manufactured products meet the imposed quality standards, that they are safe for usage and that the products meet the environmental requirements. Fuels and oil products are subjected to countless automated tests to assure the highest level of safety. A combination of correct standard test methods, the right equipment and performance evaluation of the automated testing methods is key. European Lab Services (ELS) has over 25 years of experience in this complicated matter.



The quality of every existing oil product is checked constantly, the safety of the end user stands or falls with correct or incorrect quality supervision. This quality supervision became more important over the years and this is why high-tech instruments to determine flash point, vapour pressure. viscosity or other physical properties are now the current standard in petroleum testing laboratories. There is no doubt that these analysers are smart and it is a natural reflex to rely on the results of the equipment, but even the smallest mistake can have disastrous consequences for the end user's safety. This is where the verification of method and equipment becomes important.

#### **Calibration or Verification?**



Yes, there is a significant difference despite the fact that these two words are used interchangeably. Both are of crucial importance to maintain the exactitude of an instrument, but accreditation organisations such as BELAC

European Lab Services has a calibration and test accreditation, granted by BELAC

(The Belgian Accreditation institution) make a clear difference here. Calibration is the determination of deviation of a measuring instrument. The device under test is placed in the same environmental conditions as a reference with a known deviation. The two instrument measurements are compared and the deviation of the device under test can be determined on the basis of the reference values. A well-known example is the calibration of

temperature probes in a liquid bath at a certain temperature. Verification or testing is the performance evaluation of an analysis instrument.

It is the determination whether an instrument meets the requirements of the working method used for a certain analysis (ASTM, EN, ISO, GPA, DIN, GOST, etc.). Every step described in the standard test method has to be performed with care in order to verify the instrument properly.

Therefore, calibration is often part of a verification procedure. In many standard procedures the calibration of temperature probes is an indispensable step. In order to perform verification, an accredited scope of certain parameters is required.

#### Verification Requirements

ELS was the first and is still the only company worldwide accredited for the verification of petroleum analysers on-site according international standards. Through the years, ELS has developed a unique set of checks to establish whether the device complies with the requirements of the test method used. This corresponds directly to the requirements of ISO/IEC 17025 regarding traceability of the working method.

Temperature, differential pressure, pressure, rotation, time, volume, flow, steam flow and length are the most important parameters required for the verification of most petroleum analysers. All these parameters are described in an accreditation scope, a test-certificate can be granted for each of these parameters.



A performance evaluation on an atmospheric distillation apparatus according to ASTM D86 is performed

an accreditation for on-site verification is a must have for any company performing the performance evaluation. Without this kind of accreditation, the verification certificate is not compliant to international standards.

Various verification procedures for petroleum analysers are written for a variety of industries and applications. For many of used standards, ELS has written an own standard procedure for the verification in accordance with ISO/IEC 17025.

The performance evaluation can be carried out in laboratories for crude, fuels and bio diesel, lubricating oil and grease, asphalt, waxes and bitumen. The most common verifications are performed on analysers for Flash Point (ASTM D93, ASTM D92, ASTM D56 and IP170), Atmospheric Distillation (ASTM D86), Vacuum Distillation (ASTM D1160), Cold Filter Plugging Point (EN116), Cloud and Pour Point (ASTM D2500 and ASTM D97), Jet Fuel Thermal Oxidation (ASTM D3241), Viscosity (ASTM D445), Evaporation Loss (ASTM D5800), Softening Point (ASTM D36), Micro Carbon Residue (ASTM D4530), Vapour Pressure (ASTM D5191), Freezing Point (IP468), Density (ASTM D4052), Gum Content (ASTM D381) and various other custom made procedures.

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Because various testing methods require a calibration of temperature probes, ELS is also accredited for temperature calibration going from -60°C up to 660°C according to ISO/IEC 17025. This is necessary to perform a flawless verification.

Accordance to ISO/IEC standards is proof of international recognition and traceability with minimal risk: therefore it is a great asset for petrochemical analysis laboratories and their customers. Costs will be reduced and trust will be built up, accordance to ISO/IEC 17025 will result in credibility.

#### Performing a Verification

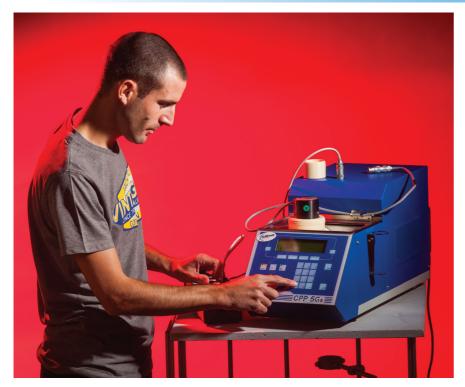
The verification of an analyser has to be performed in the laboratory where it is used. Petroleum analysers are fragile and because physical parameters are determined, the influences of environmental physical conditions can be considerable. Therefore is it important to emphasise that

### **Example Case: Verification of an Atmospheric Distillation System According to ASTM D86**

The ASTM D86 is well-known standard for atmospheric distillation units and therefore a very common verification. Each step of the procedure has to be checked thoroughly to result in a successful and trustworthy performance evaluation. Four parameters are of importance in ASTM D86: temperature, pressure, distillation speed and volume.

The first step is the calibration of the temperature sensor, which is a Pt100 temperature probe with indicator. The calibration is performed in the accredited temperature calibration laboratory for temperatures going from 0 °C up to +400 °C. A temperature calibration certificate is granted (ISO/ IEC 17025)





Technicians who perform verifications on analysers are trained and tested frequently.

Thereafter the temperature indicator is verified on-site. A precision resistance box is used for this verification and is performed independent of the temperature sensor.

The requirements for temperature lag time are strictly followed in the verification by running a test in automatic operation, replacing the electronic thermometer by a 7c or 8c thermometer and rerun the sample. The results are compared afterwards and checked if the difference is not greater than the repeatability of the test method.

Volume verification will be performed by using a calibrated precision volumetric pipette to verify the distillation volume measurement. The samples of the user are maintained and a verification has to be performed on at least 2 points minimum.

Barometric pressure correction is verified using a precision barometer with a correction on two points.

Eventually a final verification will be performed to confirm the overall performance and calibration of the distillation analyser. The temperature is verified again with toluene and hexadecane. A

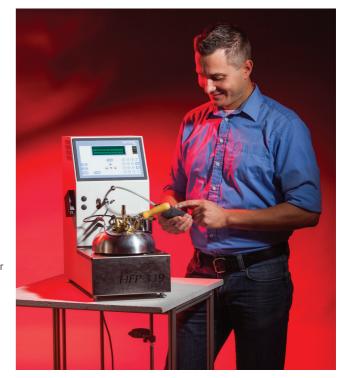
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Certified Reference Material (CRM) is used after the verification to make sure all quality requirements are met, ensure inter-laboratory correlation, generate reliable test data and meet the ISO/IEC 17025 requirements.

To guarantee continuous and reliable performance, these procedure has to be carried out every six months, the verification of the volume measurement even has to be performed every three months in order sustain constant quality.

## Trustworthy Supervision

The people of ELS are qualified to perform verifications of the test results and deviations in the analysers according the ISO/



Performance evaluation of a flash point analyser

IEC 17025 standard. This verification eliminates any possible mistake and secures the end user of the product. ELS obtained this ISO quality accreditation form BELAC, The Belgian accreditation institution.

During PEFTEC, ELS will be present together with two of its most important partners Optimus Instruments, Benelux's principal distributor of petroleum analysers for physical property testing and element analysis, and PAC, manufacturer of various physical property tests. During the exhibition Frank Van Hoorick, Managing Director of ELS and specialist concerning verification, will be giving a seminar about the activities of European Lab Services and the importance of verification in the petrochemical industry nowadays.

"We see ourselves as the concealed guard of every car driver, every passenger of a plane and even every paratrooper using a helicopter. The world of oil refinement is very complex and goes much further than basic distilling processes. The supervision of the products has become one of the main duties for the oil giants and ELS supports these processes of the oil companies."

- Frank Van Hoorick, Managing Director at European Lab Services. -



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