Bring colour to your workplace: Excellence Plus XP precision balances – for higher productivity, full compliance and amazing solutions

A sparkling solution – making champagne last

METTLE TOLEDO helps to solve a monster mystery
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A sparkling solution
Refractometer and density meter maintain the lasting quality of Champagne

The Big Monster Dig
METTLER TOLEDO helps solve a monster mystery

Under control
Analysis of thermoplastic materials ensures product quality

A trio of titration solutions
Ensure accurate oil analysis for leading Spanish company

Precise and reliable — Titrators save time and improve quality for household detergent manufacturer

Excellent addition to precision balance range

MonoBloc takes weighing out of this world

A sparkling solution!
A matter of time —
Accelerating product and process development

The story of Avantium reads like a businessman’s dream. It all starts by spotting just the niche that an industry needs. Next, influential parties from a range of disciplines are convinced to invest in the idea. The plans are put into action and, in just three years, it is now a company with more than 100 employees and a worldwide reputation.

Avantium is an advanced R&D company that has become internationally renowned in the fields of catalysis, high throughput experimentation, nanotechnology and the study of chemical processes. The company delivers these services in response to a definite market requirement. In order to remain ahead of the game, companies in the chemical and pharmaceutical industries have to speed up the development of new products, optimise their production processes and shorten the time needed to get these products to the market. But to constantly develop the technologies and competencies required for accomplishing all this, each individual company would have to invest substantial amounts of extra money in R&D. So the idea behind Avantium was to create a research centre to provide everything required for accelerated product and process development. This centre could then support industry on a commercial basis by means of contract research.

Rapid research
Avantium has three market-oriented business units of approximately the same size: Chemicals, Pharma and Crystallics.

Crystallics focuses on quickly finding the best possible crystalline form of a substance for a certain application. In the case of many medicines, for example, the effectiveness of an active reagent depends heavily on the form in which it occurs. Crystallics is able to conduct a thorough investigation into the development of a substance’s various crystalline forms within six weeks. To complete this work, fewer than five grams of material is needed to conduct tests under more than a thousand different experimental conditions.

A similar system of rapid research is used in the other two business units. For example, finding just the right catalyst to accelerate a chemical process, to make it more specific, or to increase the yield of a reaction, is often a very prolonged and costly process. Also, the question is not limited to finding the most suitable catalyst, but also to finding the conditions under which it will function most effectively. Avantium can also provide answers to these questions within six weeks. This time it makes use of its Quick Catalyst Screening platform. This is a system that carries out the many experiments automatically and with a minimum of material. Avantium stocks more than five thousand catalysts for use during these screening procedures. Finding combinations of process parameters for optimising specific steps in product synthesis can be done even faster. The Quick Process Screening service developed by Avantium can provide this information within an incredible three weeks.

Investments in people and software
To make such high-speed research projects possible, Avantium had to invest heavily in people, software and hardware. Tom van Aken, Vice President of Business Development for the company’s Life Sciences Division is clearly proud of what he calls “the gigantic amount of brainpower” within Avantium Technologies. “The vast majority of our people”, he explains, “are university-educated mathematicians, chemists, pharmacologists, engineers and IT graduates specialised in software from all over the world.” The fact that the company has already invested heavily in software can also be seen in the wide range of special packages that Avantium uses for its projects. For its research in catalysts, it has accumulated an extensive library of information about heterogeneous, homogeneous and biocatalysts from many different manufacturers which is used for quick access to existing information. Avantium also develops its own software. A good example is its DAP package, a piece of software that helps the chemist deal more efficiently with the vast amounts of data generated by High Throughput Experimentation. In addition, statistical packages help in analysing this data and in planning experiments — both useful for attaining a high degree of efficiency.

Obtaining results and conclusions is made faster by utilising software for modelling and simulating processes and reactions. For this, Avantium does not only utilise commercially available software but also VirtualLab™, a package developed in cooperation with METTLER TOLEDO. This software solution offers the chemist an intuitive platform to design, conduct and analyse experiments. These experiments are then fully compliant with the traceability requirements stated in 21 CFR part 11.

Investments in hardware
Avantium has invested in the FlexiWeigh instrument developed by METTLER TOLEDO. It weighs solid substances from a dispensing vial and then dispenses them into destination tubes. The amounts being weighed are mere milligrams of material. The FlexiWeigh instrument is part of the workflow of Avantium’s High Throughput Experimentation platform. The other part, developed by Avantium itself consists of a block in which 96 reactions can be conducted simultaneously, and under pressure if desired. In fact, finding optimum commercially feasible reaction conditions by means of this parallel processing technique is the first experimental step toward scaling up these processes.
However, scientists are not limited just to finding these optimum conditions — they can also conduct a study into the robustness of a process. The more robust a process is, the less its yield will vary as fluctuations occur in the reaction conditions. This is particularly valuable information for a manufacturing plant.

The further scaling up of reactions can be done in three successive steps. Here again, the company uses METTLER TOLEDO equipment. The second of these steps involves the use of the MultiMax multiple reactor system. This technology can utilise up to four reactor blocks, each containing either 16, four, or two reactors, with a volume range of 10 ml to 250 ml. Step three involves the use of the LabMax, an automatic single vessel system that can contain up to 1000 ml. An RC1 is also used, with a volume in this case up to six litres.

Avantium has also invested in in-situ technologies to further study what goes on in the vessel during a reaction. Using the Lasentec FBRM system allows scientists to see if and when particles form (crystallise) or dissolve in the reaction vessel. The Raman and the complementary Mid-IR technology make it possible to study how the concentration of chemical substances change during the reaction.

Next to the RC1, we see more thermal analysis equipment. This time, it’s a DSC unit and a TGA unit. The reason for this high amount of METTLER TOLEDO equipment: METTLER TOLEDO had been in contact with Avantium’s founders since 1998 and that the two companies shared many similar ideas in the field of parallel process development. This led to a close cooperation that resulted recently in Avantium’s writing a supplement to the software package for the MultiMax.

**Future**

Avantium Technologies’ customer base has grown to more than 30 customers and its number of partnerships has increased as well. In addition to its initial investors and METTLER TOLEDO, Avantium now works with such organisations as Degussa and Engelhard in the field of catalysis, with Amcis, a subsidiary of Solutia PSD, in the field of scaling up pharmaceutical processes and modelling production, and with the University of Malaysia in the field of developing high-grade products from palm oil. Avantium’s customers are very satisfied with the results the company has achieved in such a short space of time. And it looks very much as if they will continue to do so in the future as well.
As the world’s leading manufacturer of laboratory balances, METTLER TOLEDO pursues one goal: to make weighing easier with innovative solutions. With the Excellence Plus XP, the latest generation of precision balances at the Professional Level, this success story continues. Created for a wide spectrum of applications in highly diverse industries, the Excellence Plus XP offers maximum security in regulated environments, utmost productivity, and unique flexibility when tailored solutions are called for.

**Full regulatory compliance**
The new Excellence Plus XP precision balance actively supports you by ensuring regulatory compliance. A key part in this is played by SmartScreen, the unique color display with touchscreen operation. High-resolution graphics and brilliant colors provide groundbreakingly clear user guidance, while innovative QM tools support you with efficiently enforcing your quality guidelines in everyday routine. For instance, individual access rights for up to eight users can be defined by the administrator and protected with passwords. All changes to protected settings and adjustments are registered in the HistoryFile, and all measurement results can be documented to conform to G x P — for seamless traceability. In addition to proFACT, the automatic internal adjustment mechanism, BalanceCheck reliably enforces validation of the measurement certainty, for example with an external test weight specified by you, at the times you define. In short: the Excellence Plus XP opens up a new, colorful world of weighing in which you decide how much certainty you need.

**Maximum productivity**
Thanks to innovative weighing technology, the results of the Excellence Plus XP stabilize rapidly, even in harsh environments. Color graphics and full-text instructions guide you safely through applications and help avoid errors. If the repeatability of your processes is crucial, the correctness of small weighings is highly important. If desired, visual signals warn if the defined minimum weight is not achieved. Thanks to the MinWeigh function, even the smallest samples are always weighed in with highest accuracy. The Excellence Plus XP actively contributes to your process security and thereby helps increase your productivity.

**Pure ergonomics**
You don’t always have a hand free to operate the balance. So sometimes hands-free operation of the balance would be ideal, for example when you weigh toxic substances in a laminar flow. Thanks to SmartSens and ErgoSens, the intelligent infrared sensors of the Excellence Plus XP, this is possible without difficulty. At the wave of a hand you tare the balance or send your results to the printer — depending on which of the freely selectable functions you have assigned to the sensors. With both hands always free for weighing, you can complete your work quickly and safely.
Data exchange – fluent in all languages

The new Excellence Plus XP was developed to fit into a networked world with total confidence. Whether with state-of-the-art wireless connections based on the Bluetooth standard, USB, PS/2, or network solutions via Ethernet: the flexible interface concept guarantees connection and compatibility, forwards as well as backwards. For clear communication in the information age.

No matter where it is used: the new Excellence Plus XP precision balance sets new standards for weighing in the laboratory.

For more information visit:
www.mt.com/XP
Founded in 1880 in the Bourgogne region of France, Fournier Pharma is one of the leading manufacturers of lipid reducing agents (for treating cholesterol and triglycerides) and hormonal treatment patches. The 4th largest independent laboratory in France, with exports accounting for 70% of its turnover, it invests heavily in R&D in the fields of pathologies of the metabolism and nuclear receptors.

We met Mrs Céline Peter, the Physical/Chemical Laboratory manager, on the dry form (capsules and tablets) production site in Fontaine-lès-Dijon in France, following the installation of a MX5 microbalance equipped with MinWeigh.

**Example of a MinWeigh certificate issued by the Technical Department when the balance is installed.**

**Mrs Céline Peter: “Unlike balances accurate to 1/100th of a milligram, the MX5 fully meets our requirements.”**

**How low can you go – MX5 and MinWeigh for precise and reproducible results**

**A group with a strong presence on the American market**

Fournier Pharma has several establishments in France: production, chemical synthesis (manufacture of active ingredients), R&D and galenic and analytical development. However, its second largest market, after France, is the United States. The company therefore has to operate in accordance with American standards (each process modification requiring an audit) whereby everything can be traced and everything can be validated, notably in accordance with Food and Drug Administration standards — its three production sites in Chenôve and Fontaine-lès-Dijon in France and Cork in Ireland are all FDA approved.

**We have to weigh out 50 mg maximum**

“Why did we decide to upgrade to this type of balance? When preparing chromatography standards, we weigh out between 5 and 50 milligrams in micro-cups. We needed more sensitivity and precision for our standards, which are used for the different dosages: the MX5 gives us a display precision to three decimal points, i.e. to one µg, and the MinWeigh provides us with the security we required as regards minimum weighing operations,” explains Mrs Peter.

The MinWeigh is a facility that was installed by the METTLER TOLEDO technician and is memorised in the balance for carrying out minimum weight operations.
Galenic formulation is named after a 3rd Century AD Greek physician, Claudius Galen, who recorded important discoveries on anatomy and paved the way in the Western world to research into how a body absorbs different medicines. His name is associated with principles of preparing and compounding medicines that still have their counterparts in today’s laboratories.

The minimum weight of a balance is affected by the balance’s repeatability, the tare weight, the relative error given in % and the expansion factor (usually 2 or 3). To calculate the permissible minimum weight of a balance according to USP the following formula can be used:

Minimum weight = \frac{\text{Expansion factor} \times \text{Repeatability (s)}}{\text{Uncertainty (U)}}

USP requires an expansion factor of 3 and a relative uncertainty of 0.1%. For an MX5 balance with a repeatability of 1µg the minimum weight results in:

Minimum weight = \frac{3 \times 0.001\, \text{mg} \times 100}{0.1} = 3\, \text{mg}

Céline Peter has decided to dedicate the MX5 balance uniquely to these small weighing operations and told us that her American customer was very impressed by this METTLER TOLEDO technology.

It integrates the tare so that the operator can work on the net weight, in other words the actual quantity of sample. In other words, the lower the tare weight, the better the repeatability of the balance and the more one can reduce the minimum weight.

As Article 41 of the US Pharmacopeia stipulates, “the measurement uncertainty is met if three times the standard deviation of at least 10 successive weighings, divided by the quantity to be weighed, does not exceed 0.001 (0.1%)” : if the minimum value has not been validated, it cannot be exported to the United States. Thanks to the MX5 equipped with the MinWeigh, Fournier Pharma can now perform minimum weight operations of 5 mg with a repeatability of 1 µg! If the operator does not reach the defined limit memorised in the balance, a message appears in the form of a contrast on the display unit and the weighing cannot be validated.

For more information also visit: www.mt.com/MinWeigh

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Balance meets ‘UKAS’ criteria
As well as needing to upgrade existing balances, which were growing tired, the Torquay-based company was seeking UKAS accreditation and new equipment was essential to reflect this prestigious certification. In order to meet the UKAS criteria, Pipette Doctor® set its own requirements for the new balances. It specified 0.01 mg readability, a reliable and robust design and instruments that were easily transportable. The company also wanted to buy a well-known and respected brand.

When searching for suitable equipment, Pipette Doctor®, approached all the leading manufacturers before finally deciding on the SAG105 weighing module. One of the decisive factors was the efficient manner in which the METTLER TOLEDO team treated them, as Ian Hemmings, Managing Director, explains. “Other companies we approached failed to return our calls, did not turn up to demonstrate the product or were unable to show us the specific product we had requested to see. As a purchaser all these factors influence the buying process and METTLER TOLEDO provided us with the confidence we needed.”

The flexible choice for fast weighing
The SAG105 is a flexible solution for unusual weighing tasks and is ideal for use in time-critical processes where a quick balance makes a major contribution to higher productivity. Under optimal environmental conditions, the SAG105 shows the weighing result accurate to 0.01 mg and is four times faster than conventional semi-micro balances. The speed and accuracy of this model is enhanced by the use of separate weighing and control modules. The flexible draught shield provided with the SAG105 helps achieve the rapid stabilisation that is a feature of the entire SAG range, which in this model is one second.

For pipette calibration software installation on a PC is essential. The volume of liquid is calculated from its weight and density and compared with the nominal volume of the pipette. The evaporation trap prevents liquid being lost and the balance transfers the weight values to the PC for automatic calculation by the software.

Ian Hemmings continues “the SAG105 met all the technical criteria we had stated. The carry cases make them easily transportable and secure and they have proved to be very reliable and robust over the past 17 months. Even when we had a fault on one of the balances, due to it being dropped, the Service Department completed a quick and efficient repair.”

Engineer’s efficiency improved
The SAG105’s have been issued to seven field engineers as an essential part of their kit enabling them to increase efficiency. They are quick to stabilise when set up at a new location and work well in a variety of conditions, which is critical for the engineers as they are not always presented with the ideal balance environment. The specialist pipette calibration glassware are expertly designed allowing all-round access, which is key for calibrating multi-channel pipettes.

Expanding operation
Pipette Doctor® offers a pan-European service and has advised companies in Europe and South America on how to establish a “Pipette Doctor” operation. Ian Hemmings explains, “We have now established a full Pipette Doctor franchise in Ireland and Northern Ireland which is running well. We have not completed the set-up for South America although they are still looking to do something with regard to service. In all cases, the Mettler SAG 105 was the balance supplied and the only balance we recommend for field calibrations.”

Following a successful audit in April 2003, Pipette Doctor® secured UKAS accreditation. Ian Hemmings adds, “I am pleased to announce that we became UKAS accredited in 2003. We have also expanded our Torquay Calibration Lab and are adding another 6 place balance – needless to say a Mettler AX26 comparator!”
Speed it up –
Ultra microbalance dramatically increase sample throughput at environmental laboratory

The introduction of a new analytical balance has improved the speed of analysis, stability and accuracy of weighing results for a leading environmental consultancy.

Established in 1970, RPS Laboratories is now part of the Health, Safety and Environment Division of the RPS Group PLC – the largest independent environmental consultancy in Europe.

The site in Salford Quays, Manchester is one of the UK’s leading environmental and occupational health laboratories. It provides private and public sector clients with UKAS accredited analytical services for workplace and ambient atmospheric pollutants, stack emissions and biological monitoring. The expert staff – chemists, biologists and environmental scientists – provide analytical solutions to a wide range of industries from around the world. Wendy Martindale, Operational and Sales Manager, explains “People are exposed to hazardous products and substances throughout their working day – it is imperative that these are monitored to prevent ill health and disease and comply with strict Health and Safety legislation. RPS Laboratories specialise in helping our customers to identify and measure these hazardous substances accurately and with confidence.”

Stability and accuracy are vital
In order to provide the service its customers’ demand, RPS Laboratories needed to upgrade one of its existing balances. The replacement balance had to be sufficiently accurate to comply with the exacting standards of UKAS accreditation. Typically, the equipment is used to weigh filters and membranes used to assess nuisance dust levels. As the samples are very small, the new instruments had to have excellent stability as well as provide accurate and reliable results. Following a product demonstration and consultation, the company decided that the MX5 microbalance with a Haug anti-static 20cm de-ionising bar was ideal for its requirements.

Automatic operation ideal for delicate samples
The balance features SmartSens which allows hands-free operation of the draught shield. The system comprises two sensors, at each side of the user display, which activate the automatic door function. SmartSens ensures fast and accurate weighing by eliminating environmental effects. This was one of the main reasons the MX5 was chosen as Joanne Dewhurst, Laboratory Manager, explains “the automatic doors enable both left and right handed users to effectively use the balance and with the delicate samples we analyse, it is essential that we have the best equipment.” As well as the MX5 balance, a Haug anti-static unit was required as the filters can be prone to static, which results in unstable readings. When powdery samples are electrostatically charged, a draught is often enough to blow the substance into the air. With minute changes in the overall weight of a sample being critical, incidences such as this can be problematic. The Haug ioniser discharges even the smallest objects. Its probe, with a diameter of approximately 25mm, can also be installed into the inside of containers and the flexible cable allows it to be used in any position simply and easily.

With the installation of the new equipment, stability has increased – readings now only take seconds to stabilise and not minutes. “Our ability to process urgent samples on a daily basis has increased by 50%” concludes Joanne Dewhurst.
Prismo Products Services has mastered moisture control with help from METTLER TOLEDO. Thanks to the introduction of a halogen dryer, analysis time has been cut from one and a half hours to just one and a half minutes.

**Road safety matters – Fast and easy moisture content determination**

The Chorley-based company specialises in the design, development and manufacture of road markings, surface dressings and bituminous products – including traffic safety products, thermoplastic anti-skids and coloured imprints. It supplies a wide range of innovative product technology to customers worldwide.

The site has two laboratories – Quality Control and Research and Development – but it was the Quality Control laboratory that was experiencing problems, including extended analysis times, when testing and checking deliveries of raw materials. The company uses a pneumatic transfer system to move raw materials from the delivery vehicle to its silos and mixers. If moisture is present in any of these areas the system becomes clogged and production halts – costing time and money.

**Fast, accurate results needed**

Gail Stephenson, QA Manager, explains: “The system we use is very sensitive to moisture and it is essential that the raw materials delivered do not contain excess moisture. We must obtain fast and accurate results but our existing moisture analyser took one and a half hours to dry the sample and the delivery driver was not happy to wait for that length of time!” Following a consultation and product demonstration by the local METTLER TOLEDO Account Manager, Prismo decided that the HB43 halogen moisture balance was ideal for its requirements.

The HB43, which is suitable for both laboratory and production environments, offers the company ultra-rapid halogen drying as the heat is distributed evenly across the sample in seconds. The drying temperature can be precisely controlled ensuring better reproducibility and reduced measurement times. In Prismo’s case the drying time was reduced to just one and a half minutes.

**Halogen dryer saves time**

The back-lit display is easy to reach and two different drying programmes allow the Quality Control laboratory to test various samples – helping to increase productivity, improve results and save time. The flat surface of the instrument provides easy access to the sample chamber ensuring it is easy to clean. This was one of the reasons the HB43 was selected as Gail outlines: “One of the products we test is aggregate which is quite messy. By having easy access to the drying pan the equipment is kept clean. For the dry powders we test, the re-usable trays are ideal and cost-efficient.” The new instrument has improved processes at the site. “It saves us time as our processes are now quicker,” explains Gail.

Other features including the user-friendly menu and RS232 Interface for recording data on a printer or PC to meet GLP and GMP requirements have also been beneficial according to Gail. “We also have confidence in our results as the printer ticket can be attached to reports confirming the levels of moisture content in the raw materials. It is also simple to use and the robust design is ideal for our laboratory environment.”

**‘Wet’ your appetite with new moisture solutions**

Increase productivity, save time and improve results with our enhanced halogen moisture analysers.

The top-of-the-range HR83 now has 40 methods, three level password and sample identification with bar code reader. In addition it offers AutoMet – automatic method development – which can cut development time by 50%. Our HB43 halogen dryer can now run methods developed on the HR83, while the HG63 has 10 methods and sample ID – ideal when more than one product is tested with different drying criteria.
The mass measurement of small bodies in a space laboratory cannot be made using traditional balances since they give the value of the mass of a body by measuring the gravitational force acting on it. In the absence of gravity or when gravity is exactly counter-balanced by a centrifugal force (as in stable orbital conditions), a traditional balance does not work. In these environments the mass of a substance can only be determined by evaluating its inertial properties.

NASA and the Soviet Space Agency have developed inertial scales for their space laboratories to study the weight changes of astronauts during space missions. These scales were based on the evaluation of the natural frequency of an oscillating system consisting of two elements: a preloaded spring and an astronaut (fastened to one end of the spring). This system allowed mass determination up to 10 kg with an uncertainty of about 0.1%.

Although the oscillating technique is a good way to measure the inertial mass of rigid bodies, it cannot be used for estimating the mass of substances in liquids, powders or gels. For these materials the centrifugal method should be used – an object rotating at constant angular speed around a vertical axis is subjected to a steady force radially directed. The mass of a rotating object is achieved by accurately measuring the centrifugal force, angular speed and distance between the axis and centre of gravity. The most critical measurement is the last one. While it is difficult to precisely locate the centre of gravity that is geometrically regular, it is almost impossible to do so with an irregular body or powder.

A simple artifice may be adopted to overcome this difficulty. Instead of a single measurement, take two of the same object at different radial positions – provided that the radial distance between these two positions is accurately known.

IMGC (Istituto di Metrologia Gustavo Colonnetti), the Italian metrology institute responsible for mass measurement, has developed an inertial balance to accurately measure the mass of small bodies in zero-gravity conditions. Three ground prototypes have been built at IMGC (CNR) in co-operation with Alenia Spazio and the Italian Space Agency (ASI). The ground prototype has to be compatible (weight and dimension) with the shuttle flight.

MonoBloc – the heart of the ‘space balance’
The IMGC inertial centrifugal balance

There are several possible ways to build an inertial scale by application of the centrifugal method. In conventional precision balances, the gravitational force acting on the mass is not measured directly but is counterbalanced by another force. The same approach was adopted — the centrifugal force acting on the mass opposes the centripetal force exerted by a pre-loaded helical spring. Through closed-loop control, the motor angular speed is adjusted so that the centrifugal force exactly counterbalances the elastic force of the spring. A linear position sensor, which also operates as the sensing element of the control loop, indicates whether such a condition is satisfied. Three study prototypes were assembled at IMGC to demonstrate the suitability of this method. One of the key components of the prototype was a MonoBloc weighing cell manufactured by METTLER TOLEDO.

New prototype developed

To cover the lower range required by biological application (50 g full scale) and to improve the performance of the second prototype, a new design was developed. Although based on the same centrifugal method, the new balance (BIC 3) adopts a different functional approach from previous prototypes. Instead of a known constant centripetal force (the spring) to be equalised by a centrifugal force through a controlled angular speed, a known constant angular speed (then a constant centrifugal field) is produced. An integral beam measures the centrifugal force which is directly derived from a METTLER TOLEDO electronic balance. The new prototype is not affected by dynamic unbalancing due to the radial displacement of the platform housing the beam.

Better results than expected

The ground model of the inertial balance, from the mechanical point of view, is finished. The testing phase provided better results than those forecast, so a flight prototype could be designed in the future with enhanced performance. The co-operation between METTLER TOLEDO and the Italian Research Council is setting a new milestone in weighing. After the analytical balances installed in the research lab on K2 (5500 m) the next step will hopefully be the International Space Station.
A sparkling solution –

Refractometer and density meter maintain the lasting quality of Champagne

The Comité Interprofessionnel des Vins de Champagne (CIVC) is a semi-public interprofessional organisation which groups together wine growers (grape producers) and traders (Champagne firms) in order to protect the interest of the Champagne “Appellation” (guaranteed vintage). Its Technical Department carries out research work and provides wine growing, oenological and environmental advice to producers.

The rule is simple: there is only one champagne and that is real Champagne

The CIVC acts as a management, regulatory and advisory body for the whole Champagne production chain. It is responsible for registering and licensing all of the professional organisations situated in the Appellation d’Origine Contrôlée region, organising the grape harvest (for example, by setting the date for the start of the grape harvesting season each year), pressing and production. The production of champagne, as we know it today, began in the early 18th Century and now stands at 300 million bottles per year and covers a wine growing area of 34,000 hectares. In 1950, the CIVC set up a Technical Department to carry out research and experimentation to improve wine making and oenological techniques: combating frost or pest damage, selecting yeasts and bacteria in order to guarantee the quality of the alcoholic and malolactic fermentations that determine the effervescent quality of the wine, approving wine presses and press houses (a requirement that is specific to the region). The Technical Department also carries out research on monitoring production quality, from the grape pressing stage up to the final bottling stage.

The Technical Department laboratory has 3 principal missions

As Dominique Tusseau, the laboratory manager, explains: “Our missions are to provide experimental support, to carry out analyses for exports and to help defend the Champagne Appellation both in France and abroad.” Champagne is a very high quality product and it should remain so! The laboratory has ISO 17025 Standard accreditation and is fully geared to adapting or adopting new analysis methods.

METTLER TOLEDO instruments are used to measure parameters that are critical in monitoring experimental fermentations. These include total acidity, sulphur dioxide concentration, pH, density and refractive index of musts and wines and the volatile acidity and the alcoholic strength of wines using the officially approved assaying method.

Measurements carried out with a high degree of reliability and excellent precision

The key words for Dominique Tusseau are therefore exactness (accuracy and precision), equipment reliability and the speed of measurement: “We needed high technology, autonomous equipment, with a print
out of the results so that they could be checked through cross referencing. We have been clients of METTLER TOLEDO for many years for our weighing and titration equipment, and that’s why we turned to you for a densitometer, refractometer and auto-sampler. Equipment reliability is very important to us, particularly at grape harvesting time, when a very large number of musts have to be analysed in a very short period. We use a RE50 refractometer to measure the refractive index of musts and a DE45 electronic densitometer (accurate to five decimals) to measure their density and thus estimate their potential alcoholic strength by volume. With the DE45, we measure the alcohol strength of the wines using the officially approved method. We are only a small team — 7 persons in all — but we carry out 10,000 measurement per year for export and 45,000 for research and development purposes!

Obviously, all of the analyses carried out in the laboratory are backed up by tasting tests in order to control aroma, taste and colour.”
Each of the seven programmes focused on a particular geographical area and explored the life that once roamed it by piecing together clues from a current excavation, previous historical fossil finds and today’s geology. The fossil-hunting series discovered new relics from the age of dinosaurs and every week the crack team of palaeontologists solved a mystery dating from the deepest recesses of our geological past. During the series the team investigated sabre-tooth tigers in Spain, ice-age mammoths in Wiltshire and a pterosaur on the Isle of Wight. Integral to investigations for the Spanish adventure was the analytical instrumentation used to determine scientific facts and METTLER TOLEDO was happy to help.

**The Big Monster Dig – METTLER TOLEDO helps solve monster mystery**

METTLER TOLEDO has helped unearth important clues relating to the extinction of the sabre-tooth tiger. In the autumn UK broadcasting company Channel 4 screened The Big Monster Dig – a factual programme from leading independent production company RDF Media. And a METTLER TOLEDO instrument enjoyed a leading role in one of the episodes.

**Sabre-tooth tigers under the spotlight**

A high concentration of predators including the extinct sabre-tooth tiger were found in a group of mysterious, large earth mounds left over from the mine in a limestone quarry near Banyoles, Catalonia, North Eastern Spain. Carnivore fossils normally make up to 10% of the fossil record from any given location - but the level here was much higher. An important part of the investigation into sabre-tooth tigers was to establish why these earth mounds were so rich in fossils of vertebrate creatures but contained none of the associated matter – for example plant material and molluscs. Perhaps the answer to this would lead to the mysterious cause of their death. This hugely significant find suggested that this area may have been some kind of pre-historic death trap which is very exciting in the world of palaeontology. To aid the investigations RDF Media needed a fully functional portable pH meter and approached METTLER TOLEDO who had been recommended as the “best in the business.”
**pH readings provide important data**

The MP125 instrument was used to determine the pH level of the water in a newly formed natural lake near to the fossil site which the scientists believe would have been similar to the million year old lake that formed the earth mounds. The ancient earth mounds were formed by the build up of sediments in pre-historic lakes. The underlying bedrock in this area means that similar lakes are geologically formed in the region today. Professor John Howell analysed the sample from the lake to obtain the pH level. Certain levels of acidity/alkalinity can stop plant matter, shells or shelly organisms from either living or being preserved in the fossil record. The sample was pH 6.2 and at that low acidity things like bones which are made of phosphates will be preserved. But anything else would have little chance of being found in the fossil record. The pH meter is easy to use as Professor Howell explains “the equipment was great for its portability and ease of use and the level of pin-point accuracy it provided in field experimentation was impressive. I actually refer to it in the programme as my «little James Bond case».”

**The ‘pHortable’ choice**

The portable pH meter has been designed with hard-working routine measurement in mind. The RS232 output enables the meter to be directly linked to a printer or computer allowing results to be stored, analysed or simply printed out. An integrated analogue output can link to a chart recorder for the monitoring of process and reactions. The wider measuring range of the meter caters for the most unusual samples while the three point calibration gives the flexibility to measure samples with widely varying pH values accurately. The selectable resolution allows the choice between accuracy and speed in sample measurement depending upon the application. The InLab®413 electrode used by the Big Monster Dig team is a versatile combination pH electrode with integrated temperature sensor — ideal for a wide range of applications. It has a polymeric electrolyte and requires no junction at all. As a result these sensors are particularly insensitive to contamination — making it ideal for the Spanish adventure.
Thermal analysis is playing a key role in the control and research of new thermoplastic products and materials for a leading Italian company. Industrie Plastiche Lombardo S.p.A. (IPL) monitors and studies raw materials and products to guarantee high quality standards and constant innovation.

IPL produces flexible thermoplastic tubes for different applications such as agriculture, industry, gardening and hobbies. The range of products includes spiralled tubes and flat hoses as well as screened and metallic reinforced spiral tubes manufactured from materials such as PVC-P, PVC-U, polymeric alloy, polyurethane TPU, TPV thermoplastic rubber, EVA and polyolefines.

IPL was established in 1975 and, thanks to continuous technology innovation, has been able to develop products and processes to achieve impressive results, regarding both products and commercial performances. Today it is based in Besozzo and employs 110 people with an annual turnover of approximately 20,000,000 Euros.

Application and needs

Raw materials are one of IPL’s most important resources. Adopting the right quality control policy and prudent management of suppliers has helped improve product quality. More than 200 raw materials are managed daily — therefore the Quality Control and R&D Laboratory wanted to monitor and investigate raw materials and finished products more thoroughly.

The usual characterisation previously carried out on materials — density, hardness, traction characteristics, etc — no longer guaranteed consistent tube quality. In addition the company also wanted to analyse the products’ composition as well as verify the qualitative and quantitative nature of the formulations used to reproduce and optimise product performance. As a result, Paolo Negri, Head of the QC/R&D Laboratory, selected a METTLER TOLEDO TGA/SDTA851e instrument for thermal analysis, to complement the other equipment used in the laboratory.

Benefits

Despite being a sophisticated instrument, the TGA/SDTA851e is not difficult to operate. It allows precise qualitative and quantitative analysis on materials and finished products and provides fast and accurate results.
The instrument is principally used to characterise:

- polymeric alloys
- contents of possible plasticisers
- content of inorganic fillers
- efficacy of PVC thermal stabilisers (dehydrochlorination)

The TGA/SDTA851e also produces a DTA signal (comparable to DSC) which is extremely useful for identifying polymers in the compounds – particularly semi-crystalline polymers.

Thermal analysis has proved to be very useful when there have been complaints about raw materials. It allows the identification of unsuitable materials from the point of view of composition as well as the investigation of production problems.

Mr Negri explains: “It is also good to know that we can expand the instruments in the future especially with the possibility of coupling the TGA with an evolved gas analyser (EGA). Regarding the quantitative analysis of volatile compounds such as plasticisers we already obtained good results by using the TGA under vacuum.”

IPL found METTLER TOLEDO to be a qualified and reliable partner. “I knew METTLER TOLEDO thermal analysis instruments from previous working experiences,” Mr Negri explains “and I was sure of its usefulness, reliability, ease of use and of the support from, and availability of, METTLER TOLEDO technicians.”

“The TGA/SDTA851e works every time, meets our expectations and has allowed us to solve some previously unsolved problems,” Mr Negri continues, “our choice has been justified by the results achieved.”
A trio of titration solutions ensure accurate oil analysis for leading Spanish company

AGIP España is a subsidiary of the ENI Group’s Refining & Marketing Division. ENI (Ente Nacionale di Idrocarburi) is one of the largest energy companies in the world, specialising in the oil, natural gas, electricity generation and petrochemicals sectors.

METTLER TOLEDO visited AGIP España’s oil and lubricant production plant in Gavá (Barcelona). Plant Manager Mr Antonio Torres explained the factory’s main activity is producing, marketing and selling oils and lubricants for industry and the automotive sector – for both the Spanish market and export to Europe and overseas. The facility also produces fuel for its own chain of service stations and for sale direct to end clients; in the latter field, it is Spain’s leading operator.

The Gavá plant produces 47,500 tons of oils and lubricants per year and employs 38 people, working in two shifts. In terms of capacity, it is ENI R&M’s second largest lubricant producer in Europe.

Laboratory Head Mr Joan Pallí showed us the plant’s Quality Control Laboratory where monitoring of raw materials for production takes place. The department also analyses finished products and packaging and provides technical assistance for used oils.

Variety of tests for quality purposes
To carry out these controls and thus fulfil quality regulations, the four-strong Quality Control laboratory team undertake a number of analyses. Total Acid Number analysis (TAN) ascertains the durability of the oil, as well as the acidity and alkaline reserve, while Total Base Number analysis (TBN) counteracts any acidity created and examines viscosity, point of ignition, corrosive metals and density.

Mr Pallí commented that to carry out these mandatory analyses, where the priority is to achieve the most accurate results, the company uses METTLER TOLEDO equipment to ensure optimum precision.

Automation speeds up analyses
The Quality Control Laboratory uses a DL53 titrator with a Rondo sample changer to determine the state of the used oils submitted by carrying out TAN and TBN analyses. Based on the findings it subsequently advises clients on the oil’s durability. Now that the laboratory is undertaking more and more technical support services, including examining oils for buses and machinery oils, the automation which the Rondo equipment provides saves time and increases efficiency as staff can carry out analyses faster.

To determine water levels, analysis is made via electrochemical reactions using a Karl Fischer DL39 coulometer and Stromboli sample changer/oven. The equipment is used extensively for analysing base oils as...
well as oils for industry because the oven allows additives in the oils (which can cause interference) to be tested directly. Mr Pallí explained that the titrator is optimised for straightforward routine analyses and is therefore ideal for the type of diagnosis they need. Again, thanks to the automation offered by the sample changer, the laboratory staff, who work in shifts, are able to undertake other tasks. As a result the DL39 and Stromboli has helped improve the testing process for the plant’s Quality Control Laboratory.

In order to determine the water level in certain used oils, where the ignition point is too low due to contamination from external agents, such as petrol, the company uses a Karl Fischer volumetric titrator.

It is through the implementation of these processes that AGIP España manages to maintain and assure its quality standards.
Precise and reliable –
Titrators save time and improve quality for household detergent manufacturer

The Madel S.p.a. company was established 20 years ago by the Della Cuna family. Initially it produced and distributed its range of homecare detergents locally, before moving into personal care products which were sold throughout Italy.

Today Madel is established worldwide, is ISO 9001 certified and is a member of several associations including AISE (International association for soaps, detergents and maintenance products), ASSOCASA (National association for household detergents) and UNIPRO (Italian cosmetics produc. association).

The company is particularly focused on improving products, to ensure that it is safeguarding the environment and saving energy. One of its leading brands is PULIRAPID, a liquid lime scale remover, which enjoys a market share of 10%. This has doubled in the last two years after distribution channels were strengthened.

Need to optimise productivity
The R&D and QC team, which is constantly committed to researching new formulations, plays a fundamental role in the process of keeping pace in this highly competitive market. Dr Cavalli, head of the team, explains how they have been improving processes. “A decision was made to optimise productivity and we needed accurate and reproducible results. So we decided to buy another automatic titrator to be used with the one we bought two years ago.

“Now we are running two DL5X titrators equipped with phototrodes that are used to analyse tensides, in particular cationic tenside employed in softener manufacturing. After quick preparation, samples are tested and the instrument supplies accurate and reliable results rapidly avoiding the use of solvents such as chloroform normally employed in the classic two-phase titration. The whole system is supervised by a PC using LabX software.

“Considering the necessity to fully consider all requirements related to the ISO 9001 certification, METTLER TOLEDO instruments were chosen for their precision and repeatability. We also wanted to reduce operator interpretation and errors. Our customers benefit from us using the latest technology because it ensures them improved and consistent final product quality.

“As METTLER TOLEDO titrators are easy to operate, we started to analyse not only tensides but also other active principles which we previously investigated using other analytical techniques. We have now developed and validated specific methods for these as well. As a result the laboratory operators are relieved from routine tests and are free to carry out other analysis to ensure a higher standard of quality of our products.”

Recently the need to upgrade and automate the analytical process has meant that Madel has purchased a Rondolino sampler changer. “With the introduction of this the productivity of our laboratory has considerably improved, as we can run the instrument also overnight,” adds Dr Cavalli.