Dear Reader

Since the sugar production industry began in the 19th century, sugar has become commonplace. It is consumed around the world every day in foods and drinks and is used in other areas, such as in pharmaceutical products.

As sugar is used for so many purposes it needs to comply with many requirements. Regardless whether sugar is produced from sugar cane or sugar beet, analyses establish purity, content, processability and shelf-life.

This application brochure presents an overview of how to measure the required quality parameters with the state-of-the-art analytical instruments and balances from METTLER TOLEDO.

These include:
- Analytical balances-with the practical ErgoClick and grid weighing pan.
- Precision balances-with robust and reliable top-loaders.
- Moisture analyzers-fast and reproducible thanks to halogen technology.
- Refractometers-with built-in solid state thermostats.
- Karl Fischer Titrators-with solvent manager.
- Potentiometric Titrators-with color touch screen operation.

We are also happy to present automation solutions which simultaneously increase efficiency and safe costs:
- One Click™ operation.
- LiQC multi-parameter analysis system.
- Auto samplers.

We wish you every success whilst analyzing various sugar samples with our products.

Georg Reutemann
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LAB Division Marketing

Hans-Joachim Muhr
Manager Market Support Group
SBU AnaChem

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Segment Marketing Specialist
LAB Division Marketing

Disclaimer
We carefully developed the methods with METTLER TOLEDO HR83 Halogen Moisture Analyzer, Refractometer, Titrator and Karl-Fischer titrator. However, you should still test the information provided for its suitability to your intended purpose. As the use and transfer of an application are beyond our control, we cannot accept responsibility for this. The general safety rules and precautions of the manufacturer (e.g. for chemicals or solvents) must be observed.
Laboratory Solutions for the Sugar Industry

METTLER TOLEDO offers a comprehensive range of analytical instruments and balances for efficient and reliable quality control in the sugar industry. Samples from all intermediate and end products are analyzed in the laboratory for in-process control and quality control purposes. A selected choice of instruments frequently used at different process stages is presented here. All instruments are designed for easy operation, robustness and reliability.

Sugar Production Process
Sugar or sucrose is produced from either sugar beet or sugar cane.

A Sugar Beet Production Process
Sugar beet is refined into sugar in one single stage, whereas sugar-cane refining has traditionally been carried out in two stages.

B1 Raw Sugar Production Process
Raw sugar is produced locally in sugarcane-producing regions.

B2 Sugar Cane Refinery Process
Subsequent sugar refining is usually carried out in the country where the sugar is consumed.
Moisture in Sugar –
the Key to Product Quality

Fast and accurate moisture information is crucial for optimal control in sugar production plants and for the safe storage and transportation of the final product. Adjusting the moisture content to optimal levels during the drying process and keeping it within tight tolerances, optimizes the production in order to achieve the best quality. The optimal moisture content of white sugar is typically between 0.02 and 0.05% and between 0.25 and 1.10% in raw sugar. The standard method for moisture determinations after ICUMSA* GS2/1/3/9-15 requires oven drying but this is a very time consuming task. The METTLER TOLEDO halogen moisture analyzer HB83 delivers far faster, yet equally as accurate, moisture content determination during production.

* International Comission of Uniform Methods of Sugar Analysis

Instrument
HR83 Halogen Moisture Analyzer

Sample
White sugar

1. Press “Method” button and select method for sugar (both raw and white)
   • Resolution: High
   • Standby temperature: 100 °C
   • Switch-off criteria F (1 mg/ 180 sec)
   • Drying Program: Standard
   • Drying Temperature: 105 °C
   This method is applicable for white sugar and raw sugar

2. Weigh approx. 20 g of sugar into preheated and then tared sample pan

3. Press “Start” button to begin measurement

4. The result shows the moisture content of the sample

Results
The optimal moisture content of white sugar typically lies between 0.02 and 0.05%. International guidelines, such as the EU sugar policy, require a maximum moisture content of 0.06%.
The moisture content of white sugar is measured with the HR83. The result is 0.030% MC and therefore falls into the optimal range for storage and transportation.
Correlation HR83
Versus Drying Oven

A comparison study is performed with raw and white sugar in order to prove the precision of the HR83’s results which are then compared to the drying oven method’s results (ICUMSA GS2/1/3/9-15).

The study shows that the HR83 achieves highly repeatable results fully corresponding to the drying oven in minutes rather than hours.

<table>
<thead>
<tr>
<th></th>
<th>HR83 Mean [%MC]</th>
<th>HR83 SD</th>
<th>HR83 Time [min]</th>
<th>Drying Oven Mean [%MC]</th>
<th>Drying Oven SD</th>
<th>Drying Oven Time [min]</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Sugar</td>
<td>0.027</td>
<td>0.002</td>
<td>4</td>
<td>0.026</td>
<td>0.003</td>
<td>180</td>
</tr>
<tr>
<td>Raw Sugar</td>
<td>0.377</td>
<td>0.016</td>
<td>7</td>
<td>0.424</td>
<td>0.008</td>
<td>180</td>
</tr>
</tbody>
</table>

Conclusion
The moisture content of sugar is determined fast and accurately with the easy-to-use HR83 Halogen Moisture Analyzer. The results correspond fully with the reference method. Quick and precise moisture content results can significantly contribute to the operational efficiency of sugar refineries and sugar processing companies.
Efficient Sugar Content Determination
of Molasses and Syrups

The sucrose content of solutions containing mainly sucrose (e.g. molasses or syrups) is measured regularly for quality control purposes. There are two main ICUMSA methods used: i) method GS4/3-13 (2007) which describes the determination of the refractometric dry substance (RDS %) using an Abbe type refractometer and ii) method GS4-15 (1994) describing the determination of apparent dry substance (°Brix) of molasses using a hydrometer. METTLER TOLEDO’s bench top Refracto- and Density meters are suitable for both these methods.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Instruments</strong></td>
<td>Refractometer, Light source, Plastic rod, Therometer, Beaker, Water bath and pump, Magnetic stirrer</td>
<td>Precision balance, Brix hydrometers, Hydrometer cylinder, Thermometers, Beaker</td>
</tr>
<tr>
<td><strong>Analysis</strong></td>
<td>Refractometric Dry Substance (RDS%)</td>
<td>Apparent Dry Substance (°Brix) using a Hydrometer</td>
</tr>
<tr>
<td><strong>Samples</strong></td>
<td>Molasses, Syrups</td>
<td>Molasses</td>
</tr>
<tr>
<td><strong>Description</strong></td>
<td>An Abbe type refractometer with connected water bath is required for the determination of refractometric dry substance (RDS %, also called Brix) according to Method GS4/3-13 (2007). The sample can generally be measured at room temperature but, occasionally, if sugar crystals are in the suspended matter, the sample needs to be heated up to dissolve the crystals completely. Thereafter, a small amount of molasses is transferred to the prism, brought into the desired temperature range (18-28 °C) and subsequently measured.</td>
<td>Method GS4-15 (1994) describes the use of a hydrometer to determine the °Brix. This method requires a hydrometer with a range from 30-50 °C Brix and a balance for weighing the sample with a resolution of 0.1g. Firstly, the sample is diluted 1:1 using distilled water. The mass of the molasses and water and the final weight are recorded. The water and molasses mixture is then stirred well and poured into a hydrometer cylinder and left to stand for 20 minutes. The clean hydrometer is then inserted slowly into the molasses until it comes to rest. The reading is taken once the hydrometer flows freely in the cylinder.</td>
</tr>
<tr>
<td><strong>Remarks</strong></td>
<td>If the measurement temperature deviates from 20 °C, a scale correction must be applied.</td>
<td>Problems can arise if the solution is very dark. In this case a scale correction has to be applied.</td>
</tr>
</tbody>
</table>

ICUMSA Methods Discussion
Both methods require an advanced level of operational skills and, in addition, pose several sources of error due to the proposed instruments’ complexity. Some of the major pitfalls in method GS4/3-13 (2007) are:

i) thermostating the sample in a water bath,

ii) the issue of dark samples causing reduced measurement repeatability and

iii) the light source of a non-defined wavelength.

Likewise, method GS4-15 (1994), apart from being highly time consuming, causes problems regarding the thermostating of the sample and its darkness.

METTLER TOLEDO’s Solution
These problems can easily be overcome by using a METTLER TOLEDO digital refractometer. Samples are thermostated to exactly 20 °C using an integrated peltier element. As measurements are carried out in total reflection, the darkness of the sample has no influence on the measured result and, finally, the meter uses a light source with a defined wave length (D-line of sodium).

We propose a procedure that takes full advantage of measurement automation and uses the Combined Meter for the simultaneous measurement of (RDS %) and °Brix in one run. This greatly increases the ease and speed of the measurements and, as operator dependent errors are excluded, it also increases the repeatability of the measured results.

Instruments
- DR40/DR45 Combined Meter
- Automation unit (SC1 or SC30)
- PPU drying pump
- Balance (PB1502)

Samples
- Molasses

Method
- Temp.: R.I. 20.00 °C
- Stability: 2
- Wait time: 0 s
- Limit time: 300 s
- Sequence: On
- Sampling Seq.: Auto
- Samp. Limit: 0 s
- Drain Seq.: Auto
- Drain Rate: 100%
- Rinse-1 Seq.: Set
- Rinse-1 time: 15 s
- Rinse-2 Seq.: Set
- Rinse-2 Time: 25 s
- Purge seq.: Set
- Purge Time: 30 s
- Cell Test: Off
- Calib.: Air & water

Parameters
- Stability: 2
- Wait time: 0 s
- Limit time: 300 s
- Sequence: On
- Sampling Seq.: Auto
- Samp. Limit: 0 s
- Drain Seq.: Auto
- Drain Rate: 100%
- Rinse-1 Seq.: Set
- Rinse-1 time: 15 s
- Rinse-2 Seq.: Set
- Rinse-2 time: 25 s
- Purge Seq.: Set
- Purge Time: 30 s
- Cell Test: Off
- Calib.: Air & water

Conclusion
This proposed method clearly demonstrates that fully automated molasses measurements are possible. As the samples are highly viscous and sometimes solid at room temperature, dilution of the molasses is recommended.

Sample Preparation
1. Dilute sample to approx. 1:1. Tare the beaker and add 50 ± 0.5 g of molasses. Record the mass of molasses. Add 50 ± 1 ml of distilled water. Record the total mass of molasses and water. Mix the molasses and water thoroughly to ensure that a uniform solution of molasses has been obtained.

2. Pour the solution into clean 20 mL sample vials, place them into the automation unit and perform the measurement as described in the instrument manual. It is recommended to activate the repeat measurement function in order to prevent erroneous readings due to air bubbles or solid particles in the sample.

Results (n = 5)
- Brix Refractive Index: 37.27% BrixnD, SD: < 0.01% BrixnD
- Brix Density: 37.93% Brixd; SD: 0.01% Brixd
- me (mass of molasses + water) = 100.5
- md (mass of molasses) = 50.1

Calculations
- BrixnD (Molasses) = (37.27 x me)/ md = 74.74% = RDS %
- Brixd (Molasses) = (37.93 x me)/ md = 76.06% = apparent dry substance

Remarks
To exclude possible measurement errors due to non homogeneous samples (e.g. air bubbles), an automatic threefold repeat measurement is carried out for each sample. The samples are moved by 25% between measurements.

The proposed setup allows the easy integration of a colorimeter for ICUMSA color measurements according to ICUMSA methods GS1-7(2002) and GS2/3-9 (2005). If required, either pH or conductivity measurements can also be included into the measurement loop.

The suggested parameters can also be used for measurements of Brix using an RE40/RE50 (plus SC1/SC30 and flow-through cell) according to method GS4/3-13 (2007) or by using a DE40 (plus SC1/SC30) according to method GS4-15 (1994).

Note:
1) Brix readings based on the refractive index and density differ slightly from each other due to other substances in the molasses. These additional substances have a different effect on the refractive index than on the density.
2) The sample amount was adapted from the original methods as METTLER TOLEDO’s digital density measurement principle requires less sample than the hydrometer method.
Purification Process Monitoring by Titrimetric Analyses

The sugar beet purification processing technique is known as carbonatation. Depending on region and technical status quo, sugar cane producers apply different technologies for juice purifications, such as the carbonatation process or phosphatation process.

Titrimetric analyses, such as alkalinity, total lime and total hardness, offer important data to the purification control procedure in both sugar cane and sugar beet production processes. METTLER TOLEDO’s Excellence line titrators add security, speed and efficiency to this process control during sugar production.

### Analysis 1  Hardness Determination

**Instruments**  
T90 Excellence titrator, Rondo sample changer, DGi115-SC pH sensor, DX240-SC (calcium ion selective) sensor with a DX200 reference and barcode reader

**Samples**  
- Power water
- Press lime filtrate
- Thin juice
- Thick juice

**Description**  
The determination of the total hardness of water is based on a complexometric titration of calcium and magnesium with an aqueous solution of the di-sodium salt of EDTA at pH 10.

**Tips**  
- Calcium ionic selective electrode sensor is the best choice to determine total hardness for samples which are very turbid, such as power water, press lime filtrate and thin and thick juices.
- Thin and thick juice samples vary strongly in color depending on the beet root (e.g. from light to dark brown). Therefore, the colorimetric indication using a Phototrode® DP5 is not recommended for this application. The changing color of the samples gives different color darkness by the addition of Erio T as indicator, which results in a decreasing signal.

### Analysis 2  Alkalinity Determination

**Samples**  
- Pre-liming
- Main liming
- 1st carbonatation
- 2nd carbonatation

**Description**  
The samples are titrated with HCl 0.3571 mol/L to an endpoint of pH 8.2

### Analysis 3  Total Lime Determination

**Samples**  
- Mud recirculates
- Milk of lime
- 1st carbonatation

**Description**  
With the addition of hydrochloride acid, the sample is titrated to pH 1. The CaCO3 is then decomposed to CaO and CO2. The remaining CaO is titrated with sodium hydroxide to endpoint pH 5.1

### Automated Titration System

The complete analytical sequence can be performed automatically. The refinery worker inserts the sample onto the Rondo rack and starts the analytical process with one click of the touch screen. Depending on the sample, the system measures pH, alkalinity and total hardness with the corresponding method.

### Advantages

The system will run 24 hours a day during the harvest campaign and measures samples hourly. To be confident that no process steps are out of limits during production, the range of results is defined within the method. If any one sample is out of the defined limit, the operator can see this immediately and clearly on the display and is able to optimize the production process without losing time and materials. All methods and results are stored in the LabX® pro titration software and can be transferred to the LIMS System.
Titrimetric Quality Control
in Sugar Beet Production

Quality controls of the sugar production process in sugar refineries are fundamental. During the production season, the alkalinity, total hardness, pH value and total lime are important control parameters which are analyzed and measured hourly in different production steps.

<table>
<thead>
<tr>
<th>Process Step</th>
<th>Process Description</th>
<th>Analytic</th>
<th>Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weighing beet roots</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Washing and beet</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>slicing to cossettes</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Extraction by</td>
<td>Raw juice with 12-18% of sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>diffusion at 70 °C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Milk of lime</td>
<td>Milk of lime is added (Ca(OH)₂) for clarification on non-sugar compounds which will</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pre-liming Samples</td>
<td>flocculate as calcium, e.g. oxalates, phosphates, sulphates, invert sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>of milk of lime and</td>
<td>Ca(OH)₂ + (C₂O₄)²⁻ → Ca(C₂O₄) + 2OH⁻</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pre-liming</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Juice purification by</td>
<td>Milk of lime is added (Ca(OH)₂) for clarification on non-sugar compounds which will</td>
<td></td>
<td></td>
</tr>
<tr>
<td>maintaining (increas-</td>
<td>flocculate as calcium salts, e.g. oxalates, phosphates, sulphates, invert sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ing the temperature)</td>
<td>Ca(OH)₂ + (C₂O₄)²⁻ → Ca(C₂O₄) + 2OH⁻</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1st carbonation</td>
<td>Introducing of lime kiln gas (approx. 44% CO₂) so that the juice becomes filterable</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mud recirculate</td>
<td>and therefore reducing the calcium content</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2nd carbonation</td>
<td>Introducing of lime kiln gas (approx. 44% CO₂) so that the juice becomes filterable</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mud recirculate</td>
<td>and therefore reducing the calcium content</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Filiation</td>
<td>Thin juice with 14 - 16% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Evaporation</td>
<td>Thick juice with 60% sugar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Crystallisation</td>
<td>Mixture of sugar crystals</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Centrifugation</td>
<td>Molasses</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(DM = dry matter)
One Click® Water Determination of Raw Sugar

Water content is an important sugar quality control parameter. Volumetric Karl Fischer titration enables the specific and selective determination of either the total water content or only the surface water content of sugar. Since water contributes to the weight of sugar, it is important to know its exact amount in order to obtain a meaningful product quality parameter. The surface water content of granulated sugar is a critical parameter for its transformation into cubes as well as for silo storage. The volumetric Karl Fischer titrators of the compact V20, V30 and Excellence line T70 and T90 offer everything necessary for fast and accurate water determination in One Click®.

Analysis 1  Total water content determination

<table>
<thead>
<tr>
<th>Instruments</th>
<th>Compact volumetric Karl Fischer titrator V30 Kinematica Polytron 1200CL operated by an optional T-Box DR42 for 220 V power supply.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Samples</td>
<td>Raw sugar</td>
</tr>
<tr>
<td>Sample size</td>
<td>~ 1 g</td>
</tr>
<tr>
<td>Titrant</td>
<td>KF one-component reagent 2 mg/g or KF two-component reagent 2 mg/g</td>
</tr>
<tr>
<td>Solvent</td>
<td>150 mL Solvent for two-component reagent</td>
</tr>
<tr>
<td>Method parameters</td>
<td>Minimum titration time = 1200 s Mix time for homogenizer = 90 s</td>
</tr>
</tbody>
</table>

Sample Preparation and Titration

1. 150 mL solvent for the two-component reagent dissolves a maximum of 2 g of raw sugar at ambient temperature. The maximum amount can be increased to 2.5 g at 45 °C.
2. Complete dissolution of the sugar sample is achieved in a 90 s mixing time with the application of a high speed homogenizer.
3. The sugar dissolution is a time-consuming, gradual process. Therefore, a minimum titration time of 1200 s is applied. This avoids a premature titration end.

Results
3 samples, Mean water content = 816 ppm, Relative standard deviation = 2.5%

Remarks
It is also possible to use methanol/formamide 1:1 as the solvent together with the homogenizer. 150 mL of this solvent mixture can dissolve a maximum amount of 2 g raw sugar at ambient temperature. At 45 °C, the maximum amount can be increased to 6 g. At 45 °C it is possible to work without a homogenizer. Since the dissolution of the sample in this solvent mixture is reasonably fast, a minimum titration time of 600 s is required.

Analysis 2  Surface water content determination

| Samples                     | Raw sugar                                                                                                                     |
| Sample size                 | ~ 4.5 g                                                                                                                       |
| Titrant                     | KF one-component reagent 2 mg/g                                                                                               |
| Solvent                     | Methanol/Chloroform 1:4                                                                                                       |
| Method parameters           | Cautious titration start                                                                                                     |
| parameters                  | Minimum titration time = 80 s                                                                                                 |

Sample Preparation and Titration

1. A solvent mixture of methanol/chloroform 1:4 is used to avoid sugar dissolution and to selectively determine the surface water of sugar.
2. The titration is accomplished within 1.5 – 2.5 min by applying a short delay time of 3 s as termination parameter.
3. A premature end to the titration is avoided by using a minimum titration time of 80 s.

Results
3 samples, Mean water content = 125 ppm, Relative standard deviation = 7.5%

Conclusions
It is possible to determine the total water content of sugar without using the poisonous (teratogenic) formamide as an auxiliary reagent if the homogenizer and the solvent for two-component reagent are used. The homogenizer action is control-led by the titrator’s method. The surface water of sugar can be selectively determined with the appropriate solvent and a finely tuned titration control.
Amazing Weighing Solutions for Different Applications

Most Accurate Differential Weighing
Quality control analysis, such as sulphated ash content determination, requires the weighing-in and back-weighing procedures of small samples. METTLER TOLEDO’s XP Analytical Balances offer numerous innovations in order to provide intuitive user handling, unprecedented measurement performance and full data security.

Additional ErgoClips secure positioning and weighing-in directly into the tare container becomes safer and much faster compared to weighing paper methods.

Requirements
• Small sample weighing. ICUMSA requires 0.1mg readability
• Fast and accurate weighing
• Easy operation
• Reduce training time

Benefits
• Smart Grid for safe weighing-in of small samples
• Numerous ErgoClips for safe positioning of tare containers
• Very fast weighing times
• Highly accurate results
• Efficient operator training
• FACT, the fully automatic internal adjustment function, ensures precise results at all times

Robust and Easy to Clean Precision Balances
Sugar samples and liquid samples such as juices and syrups are weighed-in regularly to prepare solutions for further analysis. METTLER TOLEDO Precision Balances offer unparalleled weighing performance even in the harshest environments. Thanks to world-leading technology, our precision balances are exceptionally accurate, fast and very easy to use.

Requirements
• Weighing of crude samples
• Reliable results
• Easy operation to reduce training time
• Robustness for high throughput environment
• Easy to clean

Benefits
• Fast, precise and accurate results
• Higher productivity
• High reliability
• Stainless steel cover reduces cleaning time
• FACT, the fully automatic internal adjustment function, ensures precise results at all times
Application Brochure 41
An Overview of Sugar Analyses

Several of METTLER TOLEDO's analytical instruments and balances provide ideal solutions for the sugar quality control lab. Robustness, ease of use and the provision for automation guarantee reliable results and increased efficiency during the entire sugar production campaign.

This application brochure presents several techniques and methods for sugar refinery sample testing from incoming raw material and process monitoring to final product inspection.

Product Information
For detailed product information go to:
www.mt.com/sugarMA

Service
Our ServiceXXL approach provides cost effective service solutions to ensure optimized equipment uptime, traceability of results and regulatory compliance.

- Preventive maintenance – maintains factory specifications and expands the life cycle of equipment.
- Extended warranty – for added peace of mind and budgetary control.
- Calibration – cost-effective solution that ensures accuracy and reliability of results.
- Professional documentation – stay in compliance with your industry-specific norms and regulations.

www.mt.com
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