ICUMSA 45 – There's more to white sugar than colour

Introduction

How many times have you heard "I'm looking for ICUMSA¹ 45 sugar"? Depending upon your experience and work within the sugar industry, you may have heard this many times a week or never until now. Regardless of when you have heard this, or similar statements, did it ever result in you thinking "why just colour" and "why this terminology"? ICUMSA 45 is commonly used to describe white sugar in documents relating to trading of sugar or sugar specifications, but it doesn't really define white sugar quality.

I have had several occasions when I received calls (randomly), wherein I was asked if I could provide ICUMSA 45 sugar. As I have never been employed in a commercial role, it was surprising to be contacted by sugar traders or people associated with them. I believe that the reason I was contacted was because of my status as ICUMSA GS2 Referee. Although that position is a voluntary role, it entails responsibility for ICUMSA's global white sugar methods so anyone searching for information about sugar and ICUMSA would readily find contact details for ICUMSA Referees and Officers.

My experience in the sugar industry and as GS2 Referee meant that I couldn't politely just say "no" when I received requests for ICUMSA 45 sugar. Often, I would engage with the caller to ask why they had made the request and discuss why such a statement is technically invalid. If the caller was interested, I would elaborate further about the technical standard for sucrose and the potential for quality concerns if colour was the only parameter of interest. In general, the people I spoke to were interested because they didn't know what was meant by ICUMSA 45.

First things first, ICUMSA 45 is a convenience, in that it has been used in trade descriptions but is distanced from its source. The true parameter of interest that has resulted in the ICUMSA 45 label is solution colour. Sometimes there is a clear reference to how one is supposed to determine ICUMSA 45, but often such information is buried in the contract and/or specification document, or completely missing. Without an obvious link to ICUMSA test methods, ICUMSA 45 stated in a specification introduces ambiguity.

ICUMSA methods for solution colour determination

There are three ICUMSA methods suitable for the determination of the solution colour of white sugar [1]:

- 1) Method GS9/1/2/3-8 (2011) The Determination of Sugar Solution Colour at pH 7.0 by the MOPS Buffer Method Official (Reference) Method
- 2) Method GS2_{/3}-10 (2011) The Determination of White Sugar Solution Colour Official
- 3) Method GS2_{/3}-9 (2005) The Determination of Sugar Solution Colour at pH 7.0 Accepted

The field of application for method 1) above states that the method is applicable to raw, white, speciality and plantation white sugars [2], while method 2) is applicable only to white sugars [3] and method 3) is applicable to all crystalline, powdered white sugars and very pure syrups [4]. Methods 1) and 2) are preferred, as both have been validated via full collaborative testing [1]. The field of application for method 1) indicates it is suitable for a wide range of solution colour values, whereas method 2) is limited to solution colour results of $\leq 50 \text{ IU}^2$. Another significant difference between the methods 1) and 2) is that the first requires adjustment of pH to 7.0 (with the result expressed as IU_{7.0})

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¹ International Commission for the Uniform Methods of Sugar Analysis. ICUMSA® Ltd is a company limited by guarantee and in effect a "not for profit organisation", see www.icumsa.org for further details.

² IU = ICUMSA units, the calculated value determined for solution colour

while no pH adjustment is required for the second. There is significant history behind the development of these two methods and the requirement for pH adjustment [5]. The simplest interpretation of why is to do with performance of the methods across a range of colour values. Adjustment of pH requires additional reagents and disposal thereof but provides a defined and equivalent matrix in which to measure solution colour for a wide variety of sugars. The composition of white sugar is such that pH adjustment doesn't significantly affect the solution colour value (for values ≤ 50 IU) and the sample preparation is simpler.

Although the ICUMSA methods listed here may appear complicated with respect to which to use, the work done by ICUMSA over many years has resulted in standardisation of the basic principles and methodology that has been globally adopted. Previously [6], measurement of solution colour was dependent upon the instrument (such the Duboscq and Stammer colorimeters) in use and the bespoke standards associated with it; there was no consistency or correlation between results obtained by the various combinations of instrumentation and methodology. Thus, ICUMSA 45 should describe the solution colour in IU, but it doesn't fully describe white sugar quality.

White sugar specification

To have a better understanding of what other parameters should be considered, one needs an appropriate frame of reference. The Codex Alimentarius³ sets international food standards, guidelines and codes of practice that are considered to contribute to the safety quality and fairness of international food trade. Codex Alimentarius specifications should therefore provide a guideline for safety and quality of food products. The specification for white sugar [7] states:

"White sugar - Purified and crystallised sucrose (saccharose) with a polarisation not less than 99.7 °Z. Specifications: Polarimetric sucrose content \geq 99.7 °Z

Sulphur dioxide \leq 15 mg/kg Conductivity ash \leq 0.04 g/100 g Invert sugar content \leq 0.04 g/100 g Loss on drying \leq 0.1 g/100 g Colour in solution \leq 60 IU."

Additionally, one should also consider how legislative bodies may interpret Codex specification. An easily accessible example of this is the EU specification that relates to sugars [8]:

"Sugar or white sugar - Purified sucrose of sound and fair marketable quality with the following characteristics: Polarisation \geq 99.7 °Z

Invert sugar content ≤ 0.04 % by weight Loss on drying ≤ 0.06 % by weight

Type of colour not more than nine points determined in accordance with point (a) of

Part B"

The above description requires more explanation, as it doesn't list the approved methods to use. The methods relevant to the EU regulation were originally defined in 1969 [9]. Within this document there are two different methods that refer to colour: "type of colour" and "colour in solution". The first is referred to the Brunswick Institute Method; the current ICUMSA method [10] that is closest to this is Method GS2-11 (2007), The Determination of the Visual Appearance of White Sugar using Braunschweig Colour-Types – Official. However, the alternative [11] Method GS2-13 (2017), The Instrumental Determination of the Reflectance Grade of White Sugar – Official should be equivalent. The second is ICUMSA Method 4, which is the basis of GS2/3-10 (method 2), as described earlier).

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³ https://www.fao.org/fao-who-codexalimentarius/en/

However, once one has performed either of these methods, there is a need to calculate the "points", to ensure the sugar as tested meets the specification:

Type of colour = Brunswick Institute Method (reflectance grade), 1 colour type unit = 0.5 points Colour in solution = ICUMSA Method 4, 7.5 ICUMSA units = 1 point

It is meaningless to state solution colour values as anything other than a whole number, which suggests that the assignment of "points" has been done to provide a dimensionless unit of comparison rather than a defined scientific principle. This is further complicated because one also must calculate conductivity ash in terms of "points" too. One might think there is a tentative link between the EU regulation and the Codex specification because 60 IU is equivalent to 8 points. Unfortunately, there is no direct correlation between reflectance grade and solution colour for any given sample.

A more recent EU regulation [12], which provides details of agricultural products with respect to CAP⁴, provides significant information with respect to trade of all sugar products as well as an update for the standard quality for white sugar. The revised specification is for "standard quality" white sugar and in addition to the parameters stated in the original regulation [8], there is a requirement to determine the number of "points" for ash content (\leq 15), colour type (\leq 9) and solution colour (\leq 6), with the total "points" value to be \leq 22. Although relevant ICUMSA Methods should have been included in the newer regulation [12], there are still no references to specific, numbered methods. Ash content is stated as "determined using the ICUMSA method at 28° Brix" while colour type is "the Brunswick method" and solution colour is simply "the ICUMSA method". The ICUMSA Methods for colour type and solution colour have been described earlier. The relevant ICUMSA Method for ash content is GS2/3/9-17 (2011) The Determination of Conductivity Ash in Refined Sugar Products and in Plantation White Sugar – Official [13]. The calculation of "points" for colour type and solution colour remains the same as stated above. The calculation stated for ash content [12] is:

0.0018% of ash content = 1 point

There isn't a reference method listed in Codex [14] for determination of colour in solution for white sugar. It is not clear why no method is listed, as methods are listed for other sugar types. It may be a clerical error or possibly because there is more than one method listed for solution colour of white sugar. The lack of a reference method in a global standard that is referenced in trade documentation is concerning.

With respect to the other specification parameters listed in Codex the relevant, currently published ICUMSA methods [15] for white sugar are:

- Polarimetric sucrose content
 - Method GS2/3-1 (2011) The Braunschweig Method for the Polarisation of White Sugar by Polarimetry – Official (Reference) Method
 - o Method GS1/_{2/3/9}-1 (2011) The Determination of the Polarisation of Raw Sugar by Polarimetry Official
- Sulphur dioxide
 - Method GS2/1/7/9-33 (2011) The Determination of Sulphite by the Rosaniline Colorimetric Method in White Sugar – Official (Reference) Method; in VVHP Raw Sugar – Tentative; in Cane Juices and Syrups – Accepted; and in Plantation White Sugar – Accepted
 - Method GS2/3-35 (2011) The Determination of Sulphite in Refined Sugar Products Excepting Brown Sugars by an Enzymatic Method – Official; The Determination of Sulphite in Brown Sugars - Tentative
- Conductivity ash

⁴ Common Agricultural Policy

- o Method GS2/3/9-17 (described above)
- Invert sugar content
 - Method GS2_{/3/9}-5 (2011) The Determination of Reducing Sugars in Purified Sugar by the Knight and Allen EDTA Method – Official (Reference) Method
 - o Method GS2/9-6 (2011) The Determination of Reducing Sugars in White Sugar and Plantation White Sugar by the Modified Ofner Titrimetric Method Official
- Loss on drying
 - Method GS2/1/3/9-15 (2007) The Determination of Sugar Moisture by Loss on Drying Official

Codex lists only one ICUMSA method for each of the above parameters. Only for sulphur dioxide does it not choose the Official (Reference) Method; ICUMSA recommends the use of Official (Reference) Methods if there is any dispute about test results, but choice of method to use routinely is not prescribed. The EU regulation lists methods that are essentially precursors to the ICUMSA methods listed above.

In the discussion so far, ICUMSA 45 is potentially a more ambiguous specification than it seems, even if one were to assume it was to be interpreted as solution colour. The units would depend on the method and ICUMSA 45.

Trade or sale descriptions

My original interest in ICUMSA 45 was based on interactions I've had with traders or brokers. From those interactions, I was left with the impression that the general opinion is that ICUMSA 45 is commercially defined. I have no evidence that there is a single definition for this, and the discussion about measurement and white sugar specifications within this report clearly indicate that there is considerable ambiguity in such a term.

When searching for information about white sugar for trade, ICUMSA 45 is most often the top of the results list. There may be detailed description of the product on offer and/or a list of specifications on any given webpage, but most often polarisation is at the top of the specification list, not solution colour. Polarisation is a slightly ambiguous term and should instead be stated as polarimetric sucrose content, to ensure it is relevant to white sugar. Polarimetric sucrose content is a relative measure of purity as the measurement is directly related to pure, dry sucrose.

In comparing multiple sources of global sugar for trade, if there is a list of specifications, it frequently mirrors the Codex specification. There is also often at least a suggestion that ICUMSA methods should be used. However, specific test requirements are often missing, which further devalues the ICUMSA 45 description. Traded commodities should have a clear definition that includes testing requirements and ICUMSA 45 doesn't fulfil this, based on how it is applied. The name ICUMSA 45 also implies that ICUMSA has somehow certified the product as described by this moniker. This is false. The work of ICUMSA is solely focussed on analytical methods for the sugar industry.

In general, the more detail there is available in the specification, the more likely the information has come from a producer, rather than a trader. Sugar producers will have various specifications for different commercial products, but will also have to adhere to local, national and global standards for sugar. Additionally, the particle size and/or particle size distribution of sugar for trade of interest, but often details of this are not prominent in the stated specification. Certainly ICUMSA 45 doesn't indicate anything about particle size.

Sampling concerns

What is being tested to meet ICUMSA 45? This may seem obvious, based on description of testing methods, but even before performing any test, the sample for testing needs to be defined. How a

sample for testing has been obtained, stored and sub-sampled to provide the necessary amount for an individual test, will all affect the result.

Whatever method is used for testing of any parameter, there should be a stated range for the value, to account for variation in samples as tested. That is, there will be errors associated with both collection and preparation of samples requiring testing and the testing of those samples, so any value quoted is not absolute but will be within a stated range. Often there is little information about errors associated with sampling, and many assume that the bulk material is essentially homogeneous, when it isn't. Fully validated methods of analysis should state the acceptable range, repeatability and reproducibility for the measured parameter (as a minimum).

There are various guidelines available for management of sampling that may be interpreted for use globally down to locally in a lab. Contractual obligations for sampling can have a significant effect on trade, so agreed procedures must be well defined and appropriate. This can lead to complex procedures that can be difficult to implement, due to local conditions where the sampling needs to occur. A revised generic guide for sampling of foodstuffs is available from Codex [16], but interpretation is required so that sampling is appropriate to specific commodities. This is especially important because producers and handlers of any given commodity will have the best knowledge of problems associated with obtaining samples for testing.

Considerable work has been done by ICUMSA to define sampling protocols for sugar products, most recently coordinated by Subject 7 (Sampling, Sample Handling and Sample Preparation). At the most recent ICUMSA Session in March 2021, the Referee for Subject 7 (Alan Mead) presented a report [17] that included new protocols for sampling of free-flowing sugars and another for syrup products. The Recommendations proposed at the Session were accepted by the members, so the new methods will be incorporated into the ICUMSA Methods book. These methods provide clear guidelines for sampling of white sugar, but interpretation and use will depend upon contractual requirements. Use of the protocols should minimise errors in sampling, such that any samples for laboratory testing should be representative of the bulk material and therefore suitable for assessment of meeting the required specification values.

Unless the samples for testing are representative of the bulk material, test results may only be considered indicative of the bulk material. This is something that should be of significant concern to traders. However, often when lab results do not support the specification, the lab is considered to be at fault, rather than that the sample as tested might be questionable. Part of the problem with sample homogeneity for solid materials is that the material is comprised of different sized particles. Thus, sampling from bulk material and further handling of the sample for testing in the laboratory are critical to the performance of the testing. White sugar is crystalline, but unless it has been specifically processed (e.g. sieved) to manage the particle size range, settling of the crystals will happen during transport and storage (larger particles at the top, smaller at the bottom). Such segregation of different-sized particles can have a significant effect on test results, such that test samples fail the stated specifications. Smaller particles are more likely to produce adverse results for any of the specification parameters listed [7,8], as compounds other than sucrose tend to accumulate on the crystal surface (surface area to volume ratio). Therefore, there is a need for protocols for sampling and the sampling methods defined by the ICUMSA S7 Referee are most appropriate for white sugar.

Summary and suggestions for improvement

The focus of this paper has been white sugar, but the principles of specification apply to any commodity, including raw and intermediate materials related to sugar production. Thus, beet, cane, raw sugar and factory juices also need clear specification that is linked to globally recognised testing protocols.

ICUMSA 45 doesn't seem to mean the same thing to everyone. On the surface, it may seem like the consensus definition is 'white sugar with a solution colour of no more than 45 IU', but that isn't clear. If ICUMSA 45 was fully defined just in terms of a set solution colour, is that sufficient to ensure quality?

There is a need to use a term that is standardised, linked to the testing required and globally recognised. The ambiguity in units used in the product specification and testing required to meet the specification needs to be resolved to provide clear guidance to producers and traders. Adherence to appropriate sampling and handling procedures also needs to be addressed and linked to the specification. The term ICUMSA 45 is inappropriate as it doesn't meet these ideals and implies that it is a certified product, which it isn't.

References:

- [1] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, pp 13-18
- [2] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, p 416
- [3] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, p 102
- [4] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, p 98.
- [5] H Puke and K Thielecke, Zuckerindustrie 123 (1998) Nr. 3, pp 177-183
- [6] H Puke, Zuckerindustrie 142 Nr. 1 (2017), pp 45-56
- [7] Codex Alimentarius, Codex Standard for Sugars, CODEX STAN 212-199, Amendment 2001
- [8] Council Directive 2001/111/EC of 20 December 2001 relating to certain sugars intended for human consumption (OJ L 010, 12.1.2002., p53-57)
- [9] Regulation (EEC) No. 1265/69 of The Commission of 1 July 1969 establishing methods for determining the quality of sugar bought-in by intervention agencies (OJ L 163, 4.7, 1969)
- [10] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, pp 104-105
- [11] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, pp 106-108
- [12] Regulation (EU) No. 1308/2013 of The European Parliament and of The Council of 17 December 2013 establishing a common organisation of the markets in agricultural products and repealing Council Regulations (EEC) No. 922/72, (EEC) No. 234/79, (EC) No. 1037 and (EC) No. 1234/2007
- [13] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, pp 112-113
- [14] Codex Alimentarius, Recommended Methods of Analysis and Sampling, CXS 234-1999 (2021)
- [15] ICUMSA Methods Book 2019, ©Verlag Dr Bartens KG, pp 5-6
- [16] Codex Alimentarius Commission, Codex Committee on Methods of Analysis and Sampling, Revision of the General Guidelines on Sampling (CXG 50 – 2004), CX/MAS 21/41/9, March 2021
- [17] S7 Report will be published (in 2022) as part of the ICUMSA Proceedings 2021, 32nd Session, Virtual Session. The final Recommendations from the Session are available on the ICUMSA website.