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S&P Global
Commodity Insights

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Caking of white sugar and how to prevent (and predict?) it: a technical perspective

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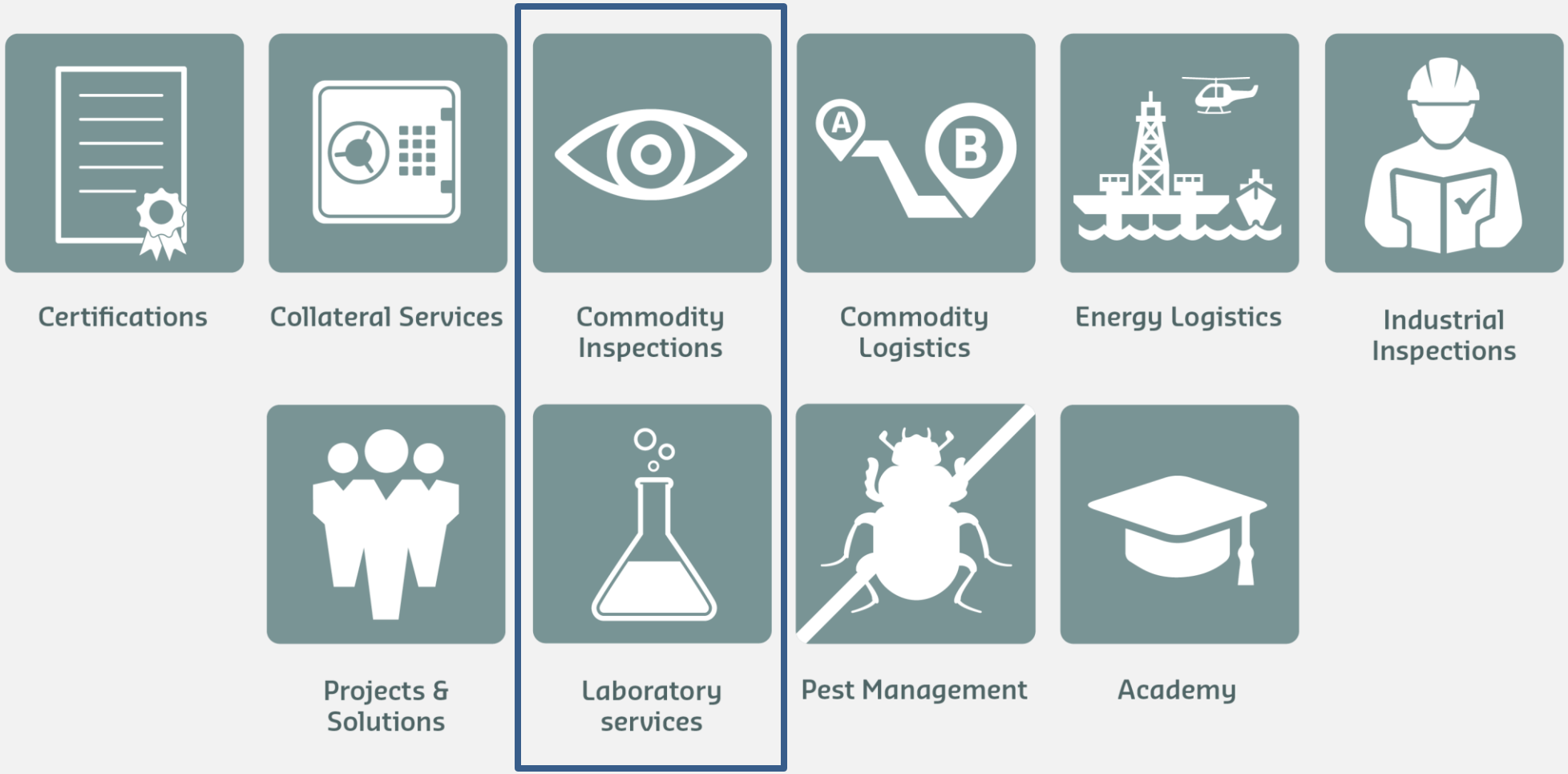

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Grains, Oil seeds & By-product

Molasses & Vegetable oil

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Fertilizers

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Bio Tim

- Tim Thys (Antwerp, °1972) graduated as a **biochemist** and holds a **Master's in environmental sciences**;

- **Global Laboratory Manager** with **Control Union**;

- **Referee with ICUMSA** for the subject **Raw Sugar** and associate referee for **White Sugar and Sampling**
  https://www.icumsa.org/subject/gs1-raw-sugar/

- **advised** sugar trading clients and law firms on **quality issues** for both white and raw sugar;

- **Recurring speaker** at the "Newcomers" **seminars** of the Refined Sugar Association in London.

ICUMSA®
International Commission for Uniform Methods of Sugar Analysis

Caking of white sugar and how to prevent (and predict?) it: a technical perspective

- cargo of white sugar all key **quality parameters** **within specs** ✓

- yet **finding** **lumps or caking** of the same sugar upon discharge **at destination** ✗

- **What** is it and what **causes** it?
- **How** to prevent it;
- Can one **predict** caking?

Caking of white sugar: what & why?

***Caking* (also called 'setting' or 'hardening')** is the phenomenon in which refined sugar:

- ceases to be free-flowing due to the **formation of lumps of agglomerated sugar crystals**;

- extent to which a sugar is caked may vary:

  from **soft, friable lumps** up to **surface crusting** and even **rock-hard** setting of large amounts of a sugar pile or bagged sugar cargo (Chen and Chou (1993).

Caking of white sugar: what & why?

The main driver for caking is **moisture:**

1. either originating from the **surrounding air,** also called ***deliquescent caking***

2. or from the **uncontrolled release of so-called bound moisture** that still is present inside the crystals of **insufficiently conditioned or non-conditioned sugar,** also called ***efflorescent caking*** (Chen and Chou, 1993).

Caking of white sugar: 1. moisture

White sugar = must be **very low** in moisture (0,015 – 0,030%):

Yet, **moisture content** when leaving centrifuges and after drying and cooling: up to 0,1% = too high.

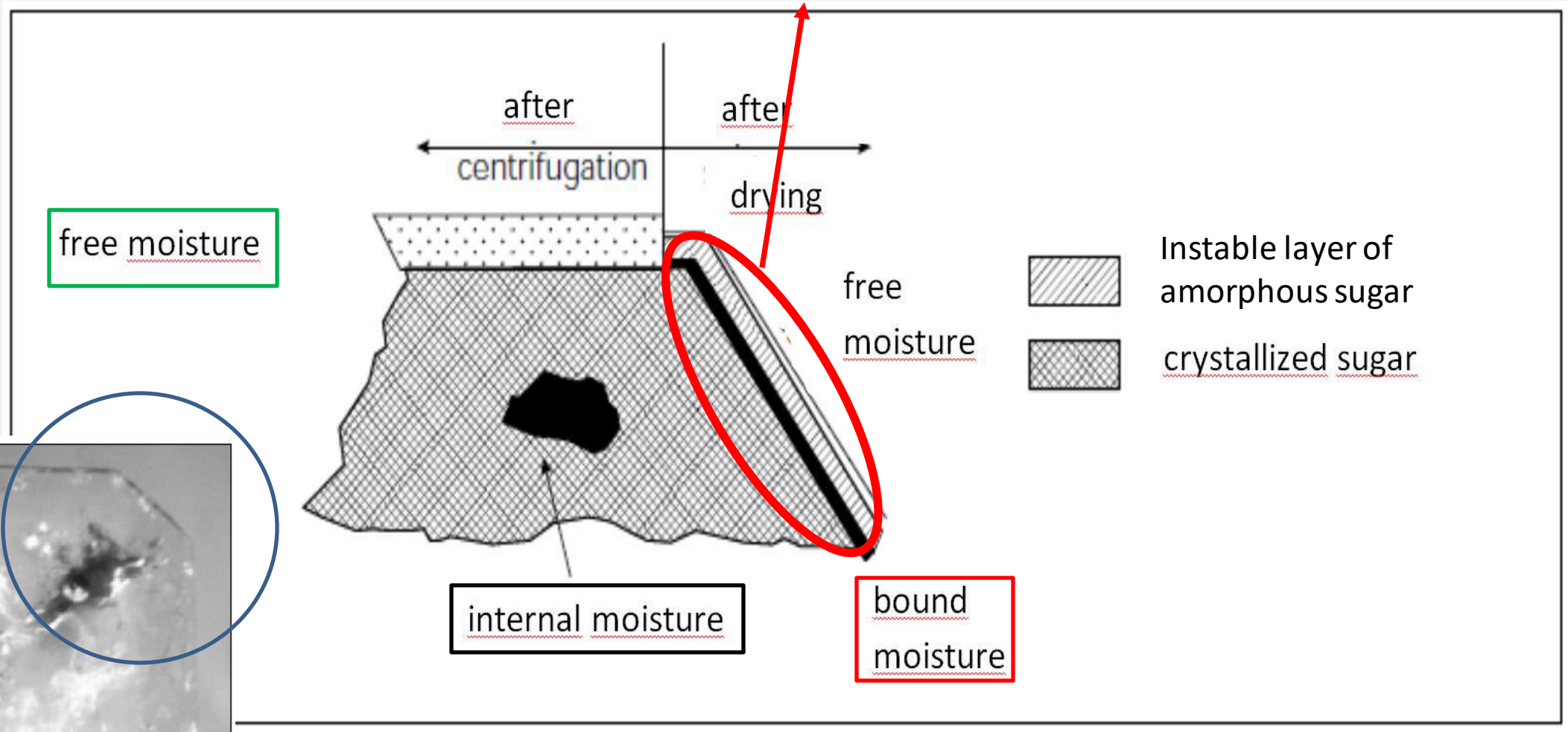
Location	Total moisture (%)
centrifuges	0,9
dryer	0,1
cooler	0,1

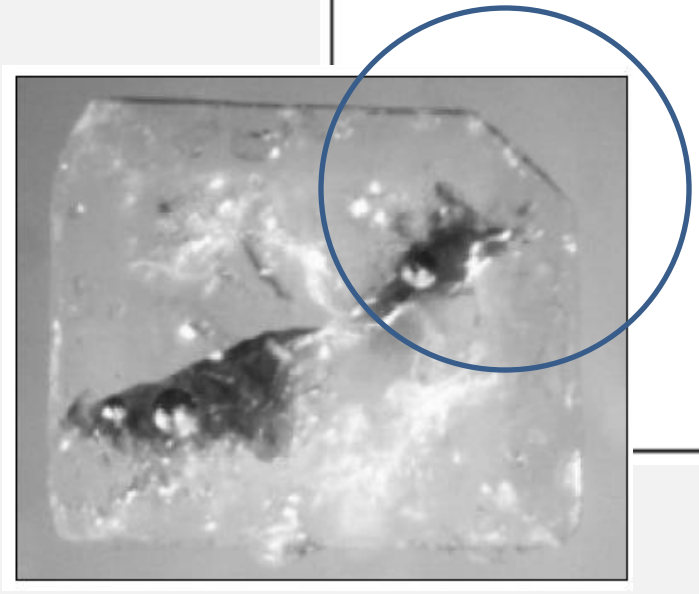
Picture: sugar dryer

Therefore: **sugar needs further "conditioning", also called "maturing" or "ripening.**

Caking of white sugar: 1. moisture

- **Instable** layer → will **crystalize**;
- **Bound** be **converted** to "**free**" moisture → leaves crystals
- In "**conditioning**" silo = "**controlled**" environment.





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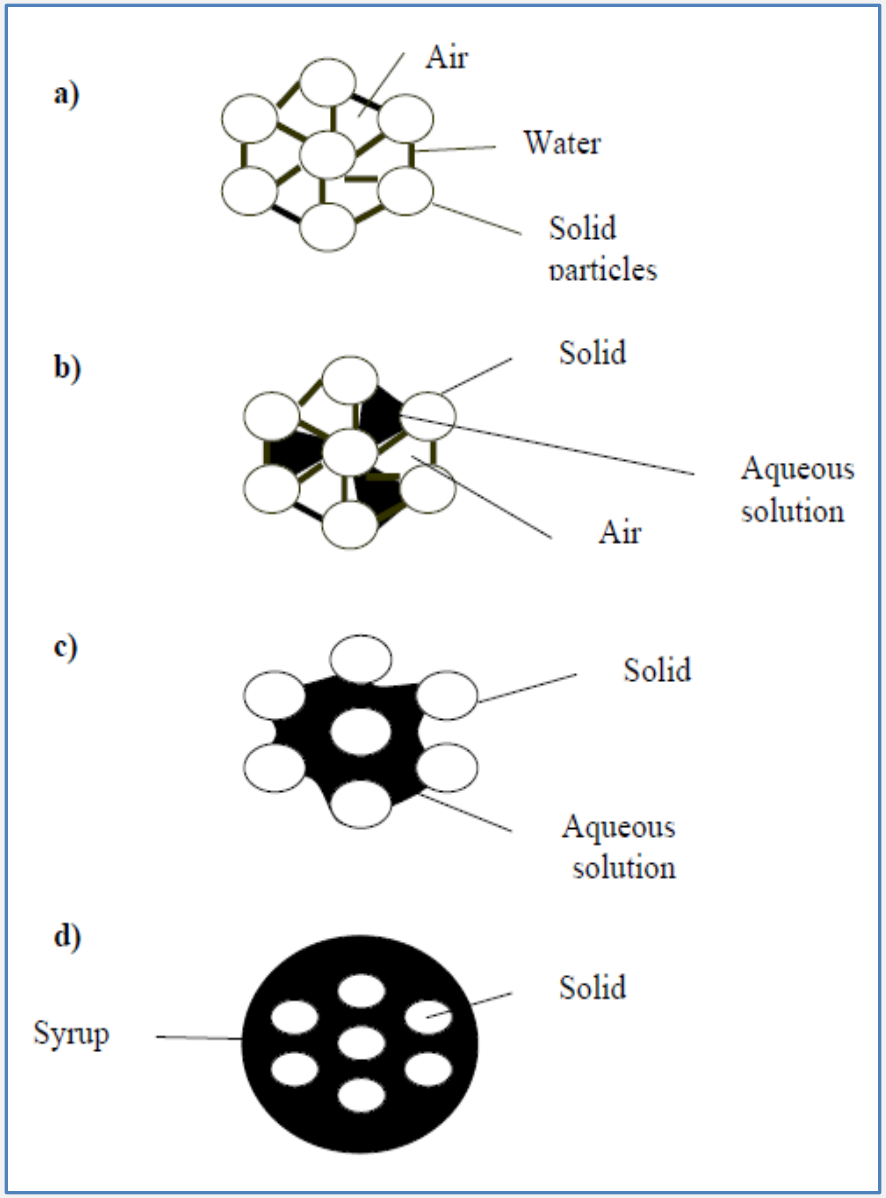
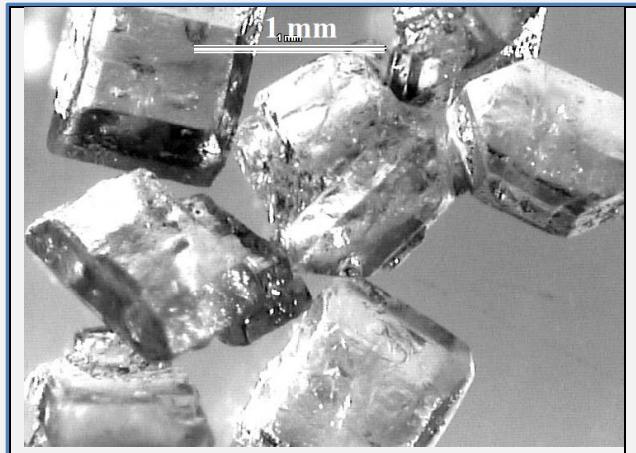
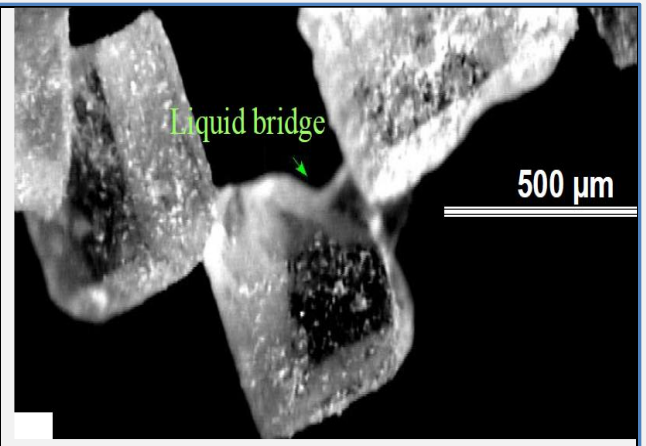
Caking of white sugar: 1. moisture



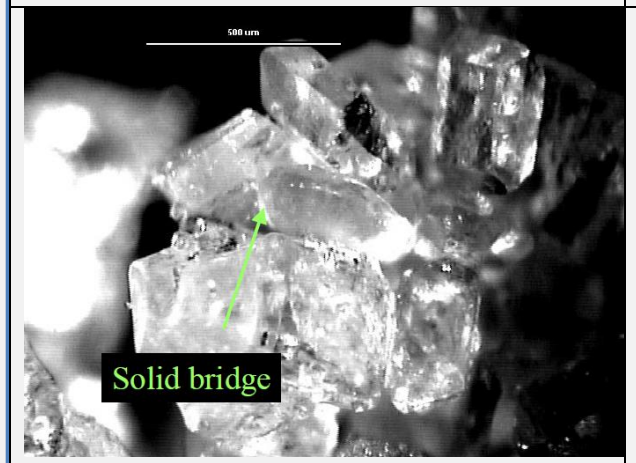
Four stages in caking (Mathlouthi and Rogé, 2004)



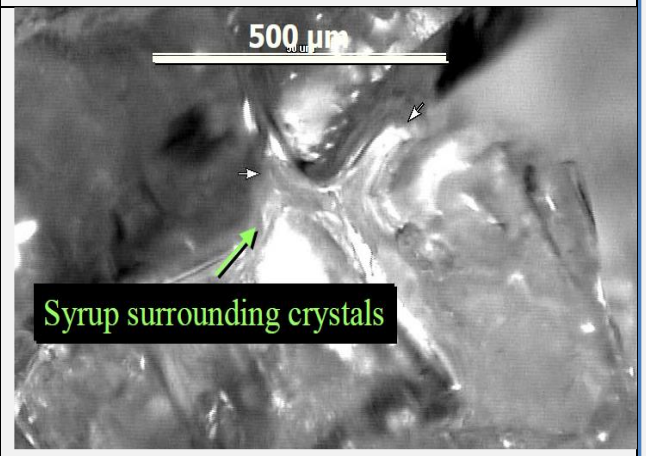
a) Pendular stage



b) Funicular stage



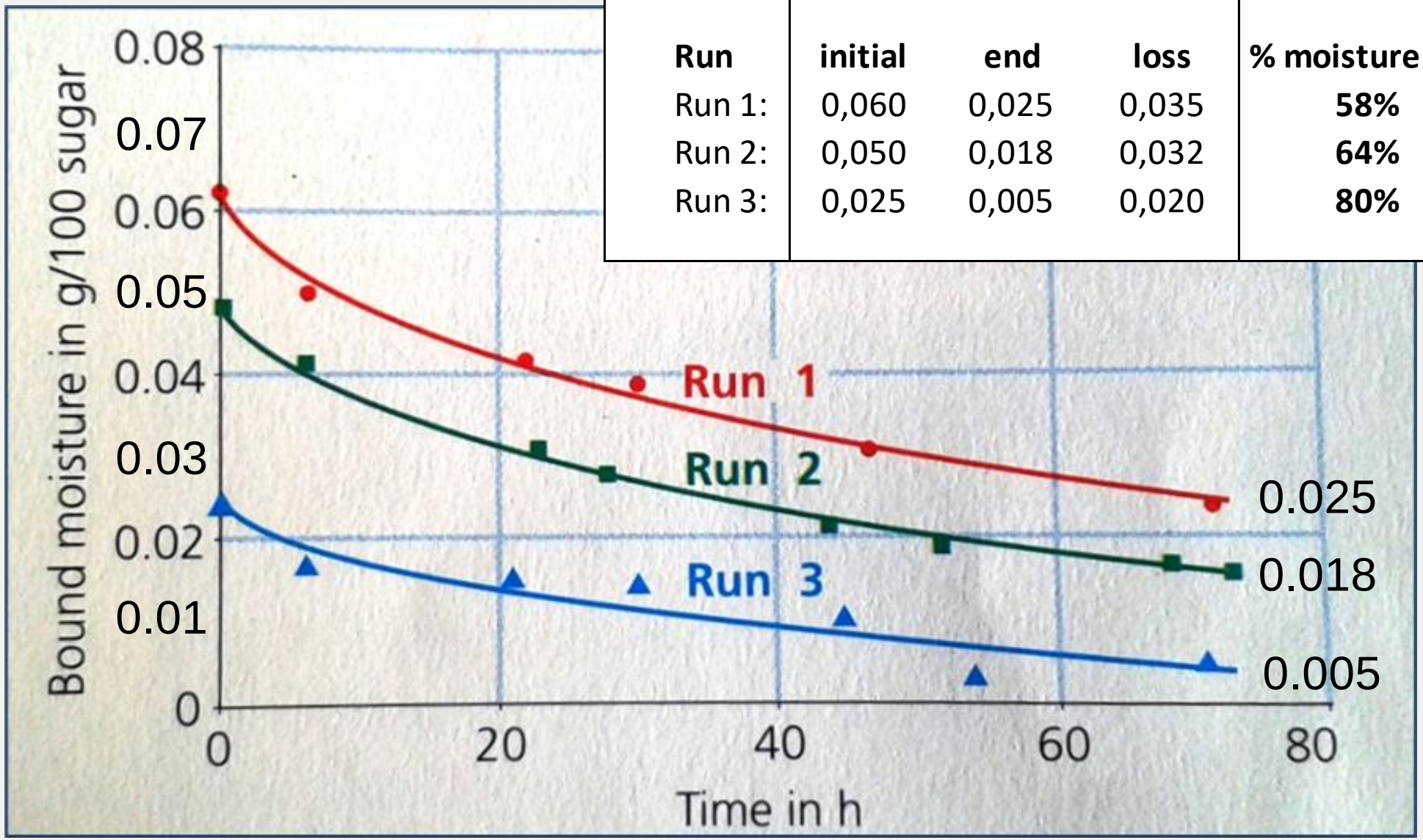
c) Capillary stage



d) Drop stage

Caking of white sugar: 1. moisture

Run	initial	end	loss	% moisture lost
Run 1:	0,060	0,025	0,035	**58%**
Run 2:	0,050	0,018	0,032	**64%**
Run 3:	0,025	0,005	0,020	**80%**

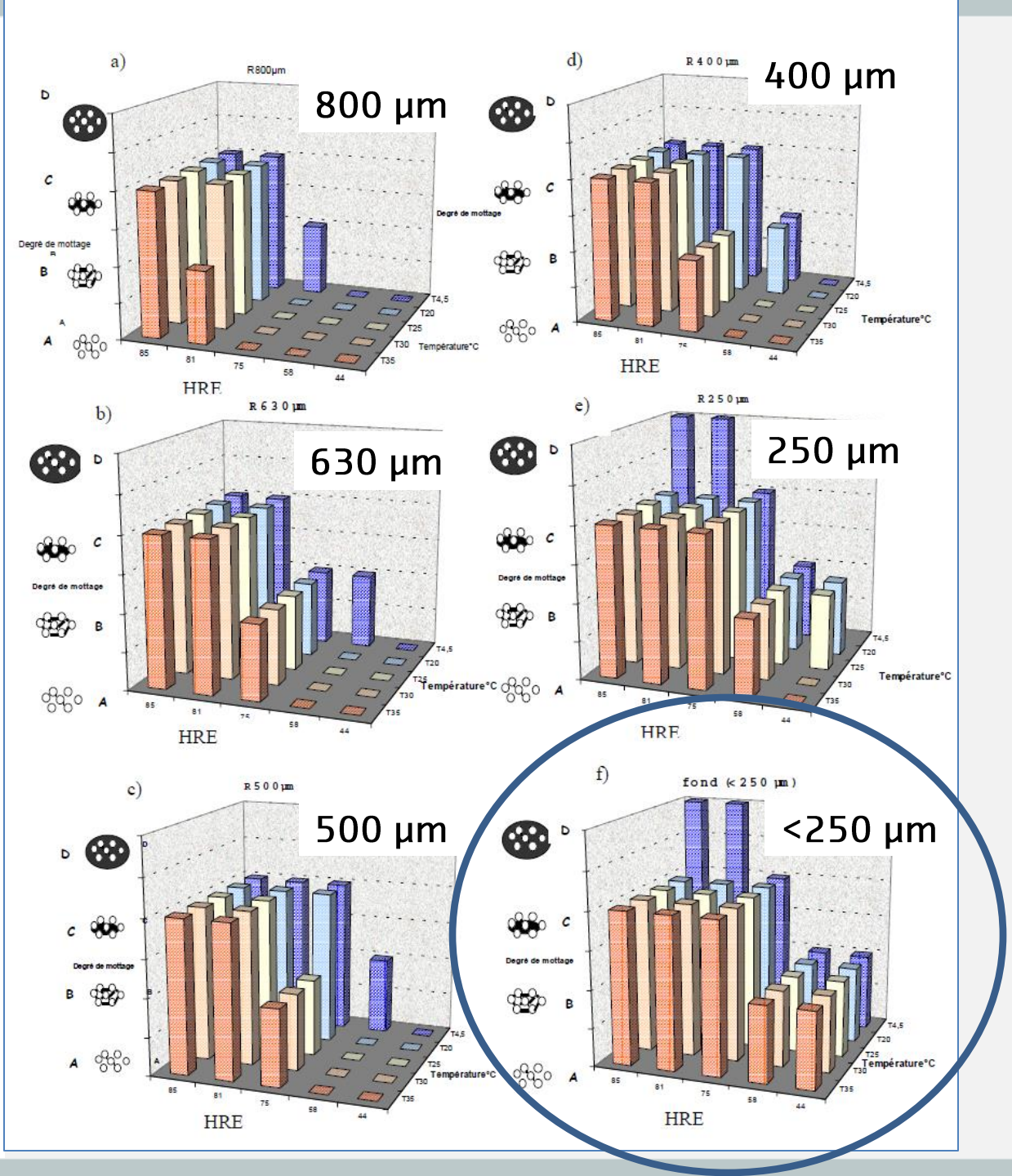


Typical conditioning curve in three runs (Rein,P. 2007)

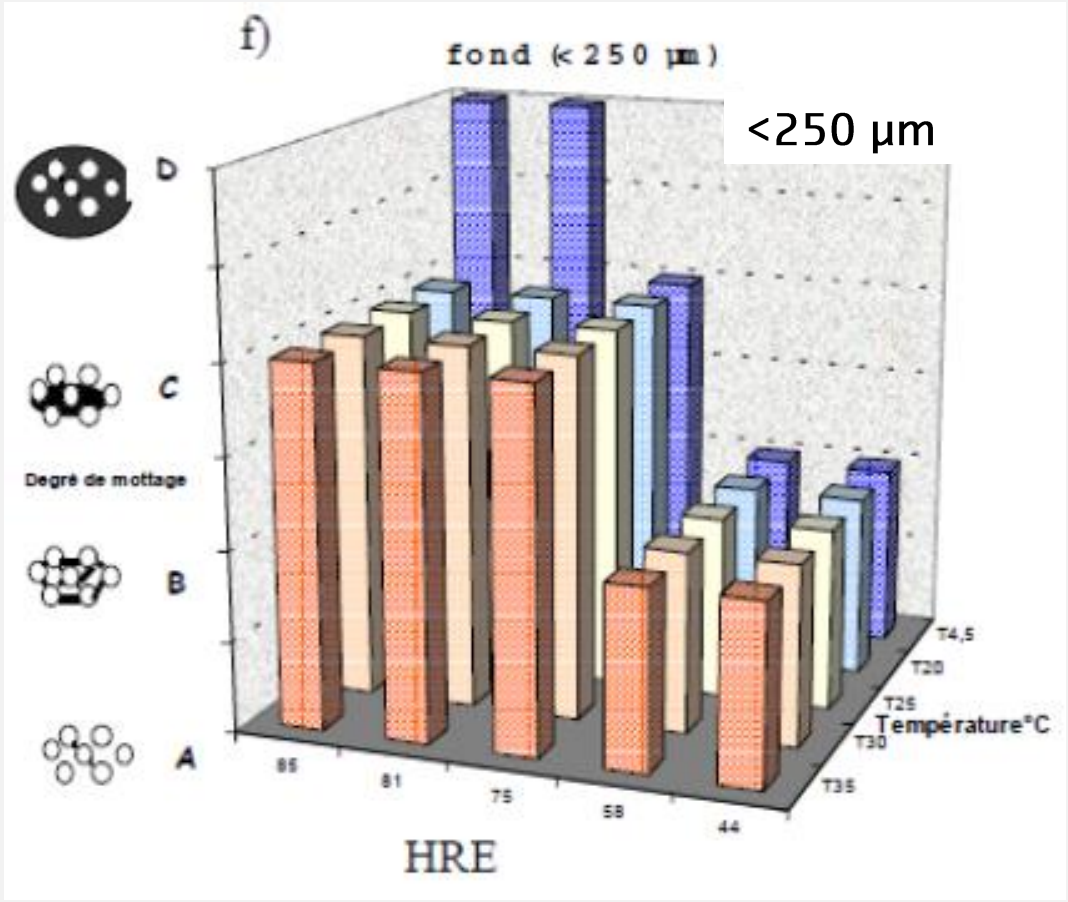
Caking of white sugar: 2. temperature

Conditioning plant guidelines (Rein, 2007)	
Conditioning temperature:	40-50 °C
Minimum conditioning time:	24 hrs
Recommanded conditioning time:	48 hrs
Relative humidity air:	10-20 %
Feed sugar moisture (from dryers):	0,10% max
Fine crystals (less than 300µm):	10% max
Post-conditioning target in °C:	35 °C max

Inspection: max temperature limit for loading ops



Caking stages (A, B, C and D) for sugar samples with varying crystal sizes 800, 630, 500, 400, 250 and below 250 µm (a, b, c, d, e and f) exposed to varying ERH and temperatures (Mathlouthi and Rogé, 2004)



Caking of white sugar: 3. crystal size

Caking of white sugar: how to prevent it?

1. Avoid packing / bagging of unconditioned sugar.....but how do you know????

2. Avoid bagging / loading sugar that is warmer than 35°C (unconditioned);

3. Sugar that is low in fines (<250 µm), with a higher Mean Aperture (MA) and low Coefficient of Variation (CV).

Crystal size distribution of two samples of sugar

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Sample 1

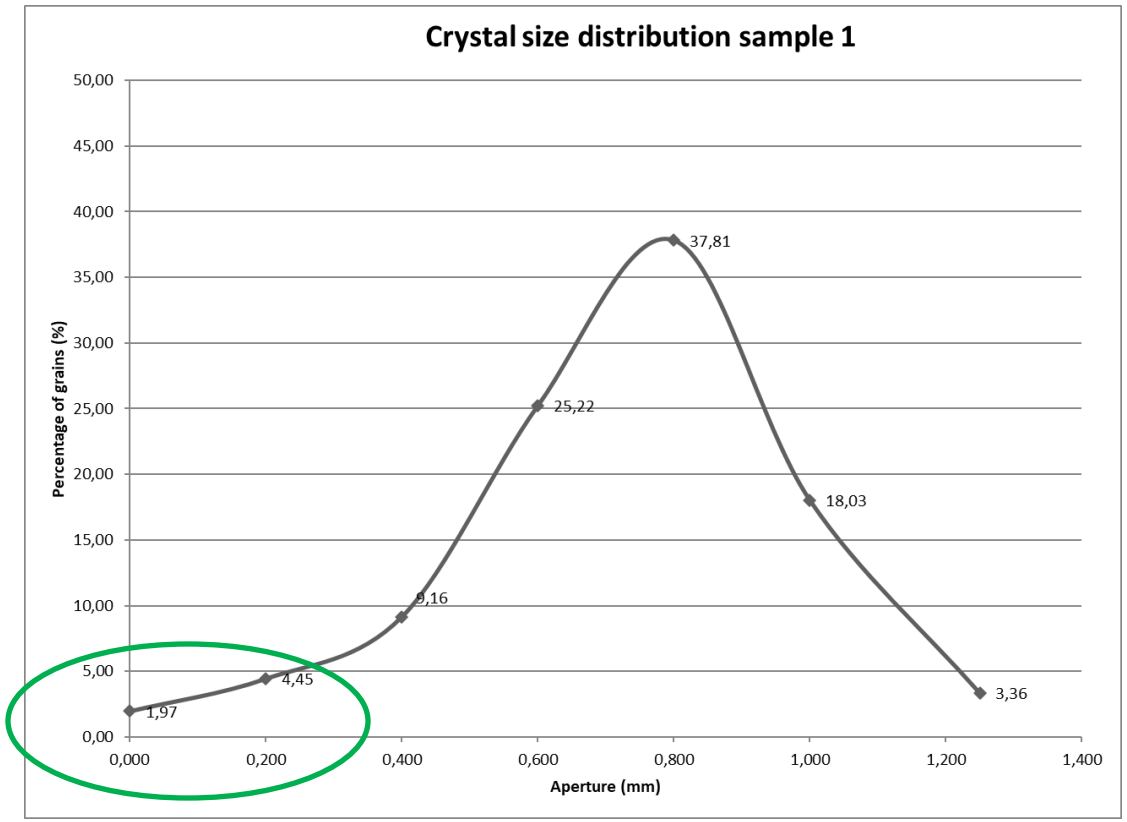
size (mm)	% crystals
1,250	3,4
1,000	18,0
0,800	37,8
0,600	25,2
0,400	9,2
0,200	**4,4**
0,000	**2,0**

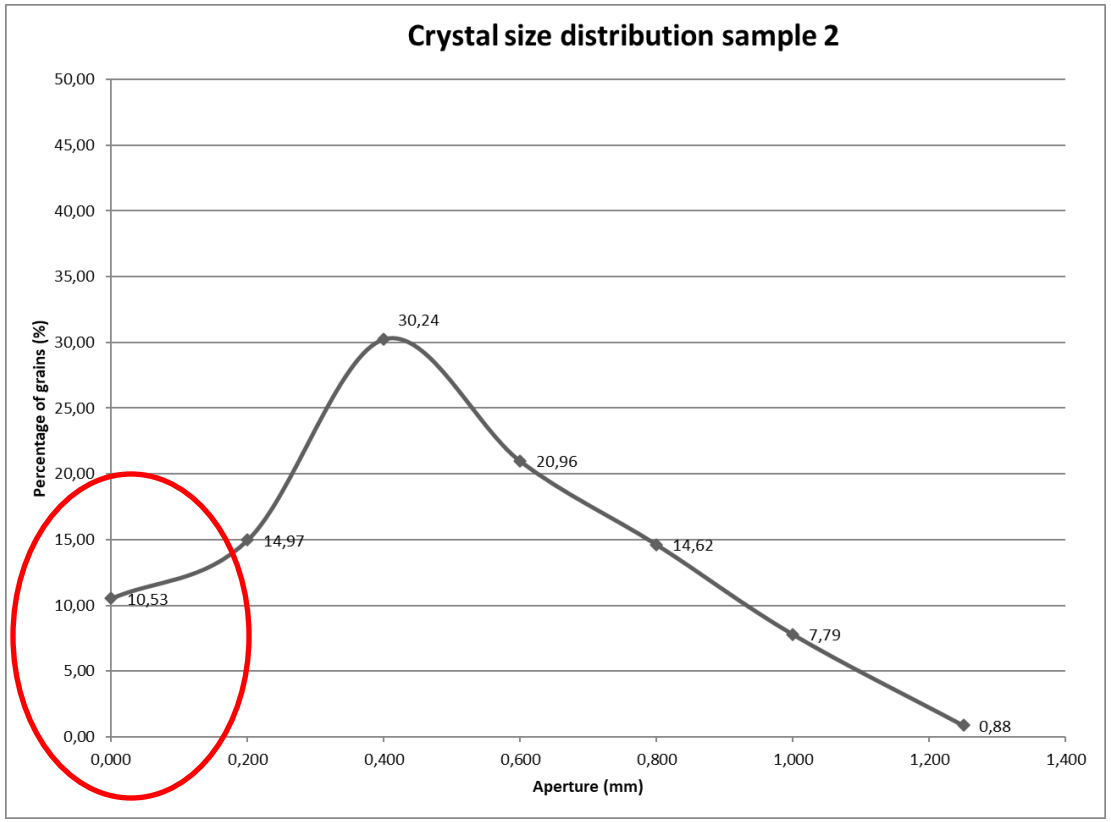
Mean Aperture MA: 0,820 mm
Coefficient of Variation CV: **30,0** %

Sample 2

size (mm)	% crystals
1,250	0,9
1,000	7,8
0,800	14,6
0,600	21,0
0,400	30,2
0,200	**15,0**
0,000	**10,5**

Mean Aperture MA: 0,580 mm
Coefficient of Variation **49,3** %





Caking of white sugar: how to predict it?

1. Most commonly used moisture determination (ICUMSA $GS2_{/1/3/9}$-15 (2007) - **oven method "loss on drying" (LOD)**: only measures free moisture;

2. In unconditioned sugar, the high amount of bound moisture not detected when using "loss on drying" method;

Location	Total moisture (%)	Moisture "loss on drying" method" (%)
centrifuges	0,9	0,9
dryer	0,1	0,015 - 0,025
cooler	0,1	0,015 - 0,025

Location and total moisture versus LOD method (Burroughs and de Bruijn, 2008)

Caking of white sugar: how to predict it?

3. "total moisture determination" (Karl Fisher titration method) can capture **both free and bound moisture:**

bound moisture = total moisture (KF) – free moisture

4. Total moisture content of properly **conditionned** sugar should not exceed 0,05-0,06% (van der poel et al, 1998).

5. ICUMSA GS4/7/3-12 to be upgraded to official method on white sugar.

Caking of white sugar: to conclude.....

1. Caking of white sugar is caused by three factors: moisture, temperature and crystal size

2. Unconditioned sugar can reveal perfectly normal "within specs" analysis results;

3. The loss on drying (oven) method only detects free moisture, not bound (which is high in unconditioned sugar)

4. "total moisture determination" capture both free and bound moisture, thereby predicting caking risks to a certain extent (& ceteris paribus).

5. Total moisture content of properly conditionned sugar should not exceed 0,05 – 0,06%

Thank you for your time

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Chen, J. C.P. and Chou, C. (1993). *Cane Sugar Handbook – a manual for cane sugar manufacturers and their chemists – 12th Edition.* p. 499-523. New York: John Wiley & Sons.

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