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Advances in Applied Science Research, 2013, 4(1):337-344

ISSN: 0976-8610
CODEN (USA): AASRFC

The application of target purity formula to quantify molasses exhaustion at Dangote sugar refinery, Apapa, Nigeria

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ABSTRACT

Over the last fourteen years of Dangote Sugar Refinery's existence, sucrose in the final molasses continues to be a source of major financial loss to the company. This study aims at rectifying this anomaly. Samples from different stages of the refining process were analyzed. A benchmark 'Target Purity Formula' which is a function of Reducing Sugar/Ash ratio (RS/A) was considered as most appropriate for assessing the degree of molasses exhaustion achieved. Reducing sugar and Sucrose were analyzed using the ICUMSA recognized Lane and Eynon method. The results indicate that RS/A in the final molasses were too high compared to those determined in the melt, clarified, decolourized and fine liquors. RS/A values for the final molasses were substituted in the various target purity formulae proposed by different authors. ASI 1993 gave the best fit at the refinery's conditions, suggesting that it is the most appropriate to quantify molasses exhaustion at the refinery.

Key words: Exhaustion, Molasses, Purity, Sucrose

INTRODUCTION

The purity of molasses is a very important process variable in table sugar refining. It is a measure of factory sucrose loss. This loss has a direct and significant impact on the profitability of the factory given the monetary value of table sugar. The presence of ash and reducing sugars account for major impurities in molasses [1]. Reducing sugars (Fructose and Glucose) are formed as a result of low pH and high temperatures involved in the refining processes. Ash content of molasses is increased due to Maillard reaction in which reducing sugars react with amino nitrogen to give brown colour in a non enzymatic reaction (2, 3, 4, 5, 6). The four conditions necessary for Maillard reaction to take place are low purity and high brix of the massecuite, presence of nitrogen compound(amino acid) and high operating temperature [7, 8]. Reducing sugars decrease the solubility of sucrose in molasses, while most inorganic components, which constitute the ash, tend to increase the solubility [9, 10].

Target purity (or true purity) is the lowest molasses purity realistically achievable in a factory. Target purity equations reflect the dependence of limiting or optimum molasses exhaustion on the ratio of two factors – the ratio of reducing sugars content to ash content (RS/A). RS/A is used as the criterion for molasses exhaustion because it is the only parameter that affects final molasses purity that cannot be controlled [11]. A number of **target purity equations** have been proposed in the past [12,13, 14, 15, 16. 17]. The aim of this study is to determine appropriate Target purity formula that can be used to quantify molasses exhaustion at the Dangote Sugar Refinery. The apparent purity concept,used at present, is considered insufficient to quantify the degree of molasses exhaustion.

MATERIALS AND METHODS

The Dangote Sugar Refinery *(DSR)* is the foremost Sugar Refinery in Nigeria. At the plant, the sucrose available in the liquor is crystallized in several stages, conducted at descending purities. This is because the liquor becomes more and more exhausted of sugar and crystallization becomes more difficult, since the non- sugars in the liquor inhibit sugar crystallization. The sugar in the raw sugar typically makes up 98% to 99% of the dissolved solid. The final molasses in the plant has sucrose content of between 40% and 45%, which always translate to between 15% and 20% of the entering raw sugar. The true purity (sucrose/dissolved solids) of the final molasses in the refinery is also between 45% and 60% as against the 35% and 45% range that are obtained in places like South Africa and Louisiana.

Sampling

Sampling points for the study are indicated in Figure 1 below. Three sets of raw melt and the liquors samples (clarified, decolourized and fine) were taken at two hour interval and composited. Final molasses samples were collected from the sampling point of the continuous centrifugal machine. Three sets of final molasses samples were collected at two hour intervals composited and analyzed.

Raw Melt and Liquors Brix

Dilution 1:1 by weight was carried out with water and the Brix measured directly on the refractometer.

$$Brix = Refractometer\ reading * Dilution\ factor \quad (1)$$

Exactly 160 g of liquor or melt was weighted into a 200ml Kohlrausch flask and made up to mark with distilled water.

The solution was properly mixed and titrated against Fehling's solution

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$$RS = \frac{Fehling's\ Factor * 100}{Conc.(g/ml) * Titre} \quad (2)$$

Where concentration = 0.8g/ml and Fehling's factor for the Fehling's solution used is equal to 0.053.

Raw Melt and Liquors Ash Content

A quantity of raw melt or liquor equivalent to 500/Brix grams was weighed into a 100ml Kohlrausch flask and made up to mark with distilled water.

The conductivity (micro siemens/cm) of the solution was measured against distilled water.

$$\%Ash = \{Sample\ reading - Water\ reading\} * 0.9 * 0.0018 \quad (3)$$

Raw Melt and Liquors pH Measurement

The pH of the composite melt and the liquor samples was measured directly using a pH meter.

Final Molasses pH

Final molasses pH was determined using a suitably calibrated pH meter.

Final Molasses Brix

Composited final molasses samples were diluted with distilled water in the ratio 1:1 by weight and thoroughly mixed. The resulting solution brix was read directly on the refractometer.

$$Brix = Refractometer\ reading * Dilution\ factor \quad (4)$$

Final Molasses Pol

Twenty-six grams of the 1:1 diluted sample is weighed in a 200ml Kohlrausch flask and diluted to volume with distilled water. The solution is then transferred to a glass jar and two teaspoons of Octapol are added. The sample is shaken well filtered and read on the Saccharimeter using a 200mm tube.

$$Pol = polarimeter\ reading * 4.4 \quad (5)$$

Final Molasses Apparent Purity
Final molasses purity is given by

$$\frac{Pol * 100}{Brix} \quad (6)$$

Final Molasses Reducing Sugar
Reducing sugar determination in the final molasses is based on the reduction of copper under standard condition (Lane and Eynon constant volume method).

$$RS = \frac{500}{Titre} \quad (7)$$

Final Molasses Sucrose
Total sugar was determined as invert after the inversion of sucrose with acid (and heat treatment). The difference between this value and RS (determined on another portion of sample) gave the percentage sucrose.

$$Total\ sugar\ (as\ invert) = \frac{2000 \quad * 0.95}{Titre} \quad (8)$$

$$Sucrose = Total\ sugar - RS \quad (9)$$

Final Molasses Ash Content
Determined directly using conductivity meter

Calculation
$$\%\ Ash = \{(Sample\ Reading - Water\ reading) * 0.185 * 0.084\} + 0.8 \quad (10)$$

Final Molasses True Purity
True purity is estimated as

$$True\ Purity = \frac{Sucrose \ *100}{Dry\ Solid} \quad (11)$$

Where Dry solid is estimated from the respective brix and pol results of the final molasses using Hoekstra correlation given as

$$DS = brix * (1 - 0.00066 * (brix - pol\ )\ ) \quad (12)$$

Final Molasses Target Purity
Reducing sugar to ash content ratio results gotten from the analysis of the reducing sugars and the determined ash will be substituted into the target formulae equations proposed by Rein & Smith 1981; Smith 1995 and ASI 1993.

Rein & Smith 1981	$33.9 - 13.4\ \log(RS/A)$	13
Smith 1995	$43.1 - 17.5[1 - exp\ (-0.74RS/A)]$	14
ASI 1993	$42.4 - 12.3 log\ (RS/A)$	15

The target purity formula that gives the reasonable target purities within the reasonable range of between the range of 35 and 40 will be adopted.

Final Molasses Target Purity Difference (TPD)
$$Target\ purity\ difference = True\ purity - Target\ purity \quad (16)$$

RESULTS AND DISCUSSION

RS/A

Reducing sugars to ash content ratios for clarified decolourized and the fine liquors were very close but a sharp increase was noticed in the final molasses as shown in Figure 2. The high final molasses RS/Ash is indicative of poor molasses exhaustion, of poor sucrose recovery. For optimum operation and good molasses exhaustion, constant RS/Ash content ratio should be maintained throughout the refining process. The results of the study conducted by [11,15] are in conformity with the present confirmation that purity of exhausted molasses is strongly dependent on the Reducing Sugar to Ash ratio.

pH

Final molasses pHs were observed to be on the lower (acidic) side. Figure 4 illustrates the pH profile of the plant. Lower pHs cause sucrose inversion and this would explain the high RS values determined in the final molasses samples [9]. pH measurement will enable decision to be made regarding the next stage of production, help to pin point areas of problem and assist the sugar technologists to effect necessary corrections.

Target Purity and Target Purity Difference

Foster (1960); Bruijn *et al.* (1972); Matthesius and Mellet (1976); Rein and Smith (1981); Audubon Sugar Institute 1993; Smith 1995; and Miller *et al*,1998 modeled target purity to obtain the equations presented in Table 1. These are some of the target purity equations that have been proposed.

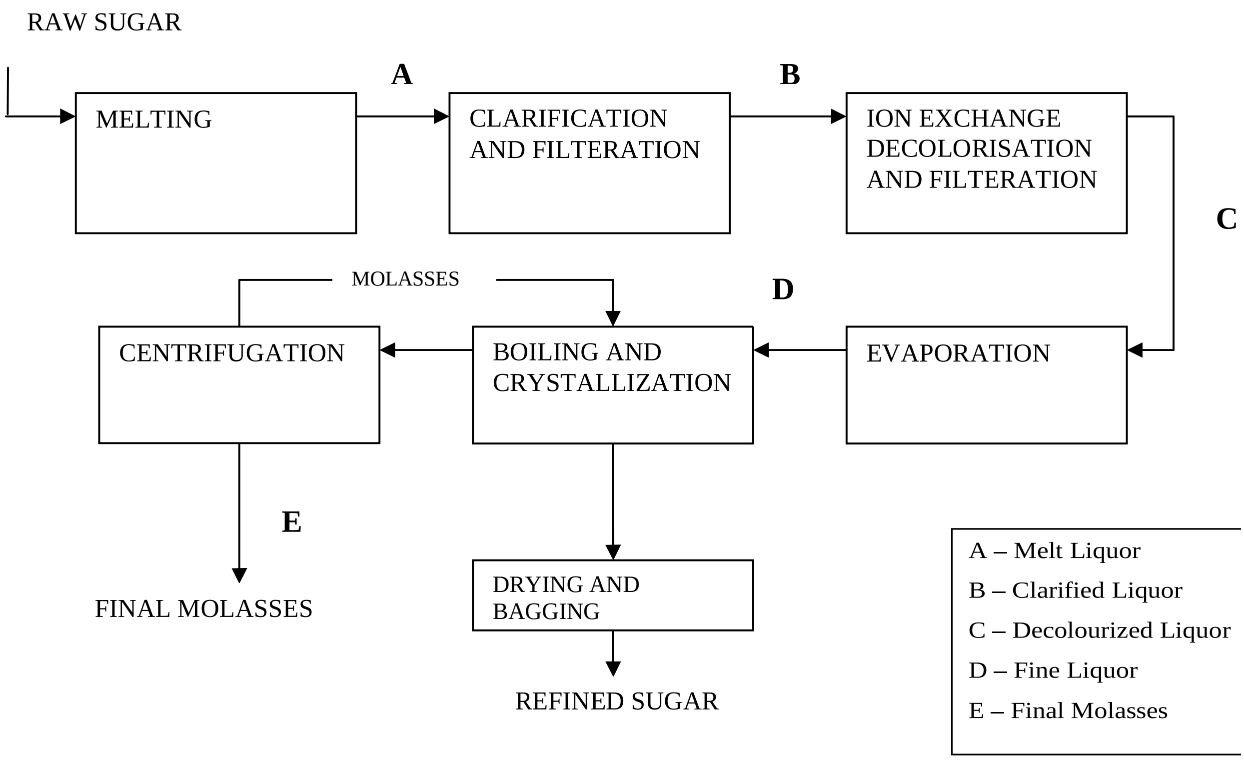

Fig. 1: Process Flow Diagram of the Refinery indicating the Sampling Poi

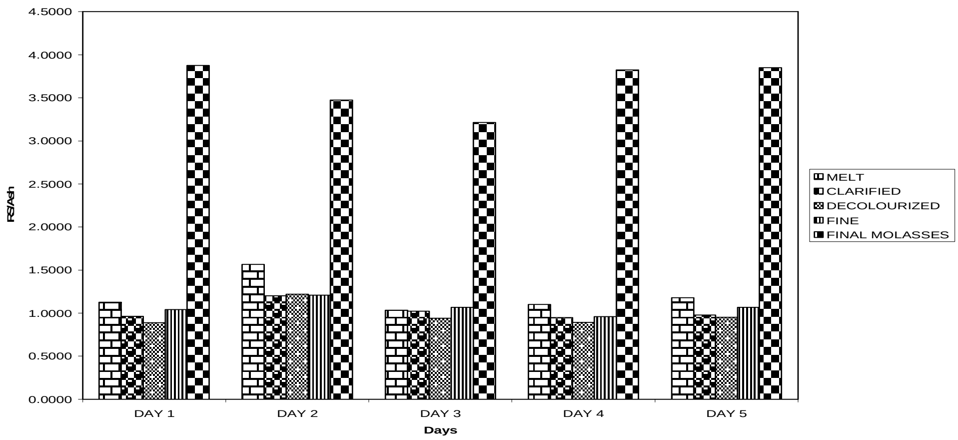

Fig. 2 : Reducing Sugars to Ash Content Variation Chart

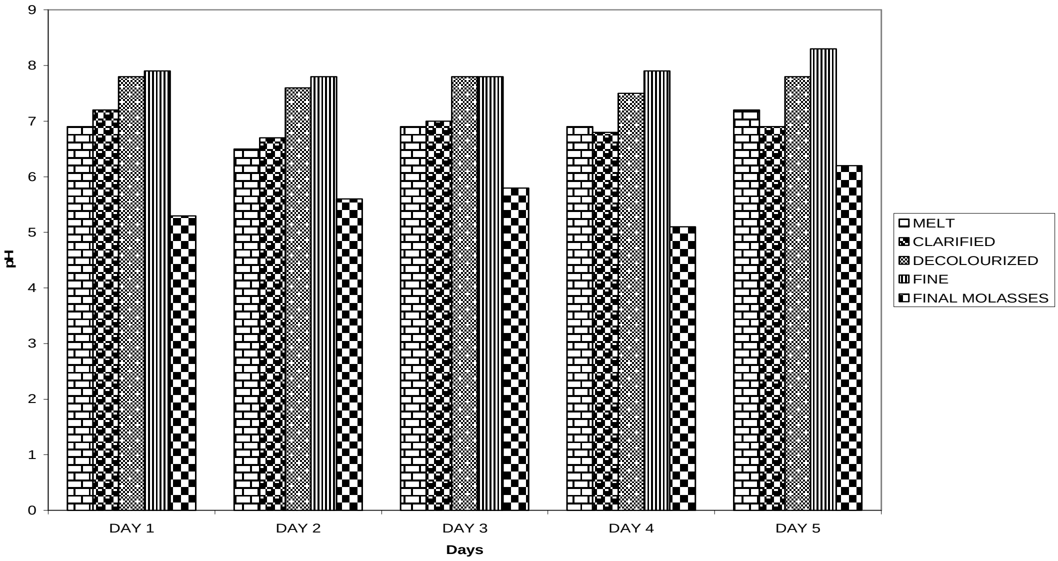

Fig. 3: pH Trends in the Various Refining Stages

Table 1: Target purity equations proposed in the literature

Reference	Equation
Foster 1960	$40.7 - 17.8log(RS/A)$
Miller *et al.*, 1998	$39.4 - 10.6log(RS/A)$
Bruijn *et al.*, 1972	$39.9 - 19.6log(RS/A)$
Rein & Smith 1981(a)	$37.7 - 17.6log(RS/A)$
Rein & Smith 1981(b)	$33.9 - 13.4log(RS/A)$
Smith 1995	$43.1 - 17.5[1 - exp(-0.74RS/A)]$
ASI 1993	$42.4 - 12.3log(RS/A)$

Source: Rein (2002)

RS/Ash value of the final molasses were substituted into the target purity formulae of Rein and Smith 1981; Smith 1995 and ASI 1993. From the available results in Figure 4 and the Appendix , Rein and Smith 1981 target purity formula gave the average target purity for the Final molasses to be 27.83 and the target purity difference (TPD) of between 17.76 and 24.05. Smith 1995 target purity formula gave an average of 27.83 for TPD range of between 18.58 and 25.29. Audubon Sugar Institute Target purity formula (ASI 1993) is thereby considered to be the most appropriate for the Refinery. This is premised on the fact it was expected that the purity of the final molasses of the refinery to be within the range of 35 and 40. A target purity range of between 35 and 37 was observed using the ASI target purity formula and this confirms its applicability to the Dangote Sugar Refinery situation. However, using this formula (ASI, 1993) still gives the TPD range to be within the range 9 and 17. The high TPD values signify poor molasses exhaustibility and high sucrose loss in the final molasses.

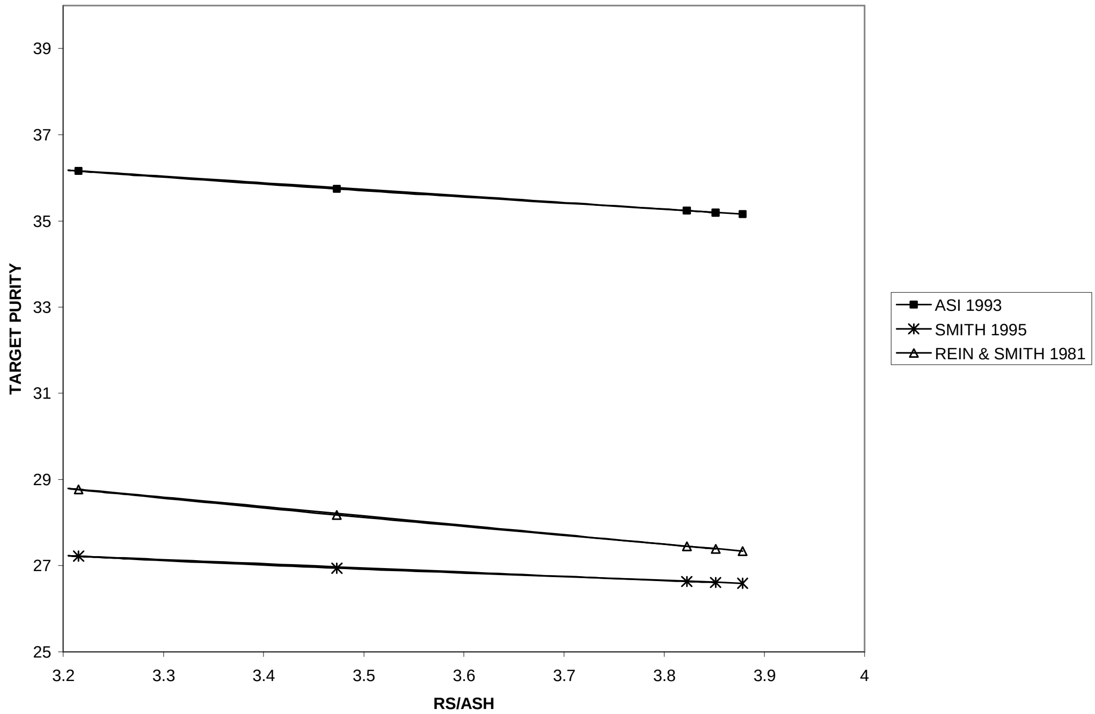

Fig.4 : Comparison of Target Purity Formulae with RS/A Values for Final Molasses

The dry solids were not measured but were inferred from the brix and pol values using mathematical correlation (Hoekstra's correlation). However it is unlikely that this simplification affects the true purity by more than one unit, within the accuracy of the whole determination.

Target Purity equation is considered as a benchmark because

i. the most accurate and reliable measurements - Lane and Eynon method [21] was used to determine reducing sugars.

ii. the concept been used extensively in Southern Africa, Australia, Cuba, Swaziland, Zimbabwe, Louisiana, Reunion Island and other countries for years, and has been reliable under all conditions [1, 18,19,20].

iii. a recent survey of molasses from around the world shows TPD values for well exhausted molasses in the range of 3 to 7 units [20], in the expected range, confirming its general applicability. The use of ASI 1993 at Dangote Sugar Refinery gave TPD of between 9 (which are close to the value for well exhausted molasses) and 17.

iv. at present, ASI 1993 is most appropriate because RS/A obtainable at DSR is between 3 and 4 which will give the desired target purity of between 35 and 40. Other Target purity formulae could be applied in countries where RS/A is less than 3 to achieve a target purity of between 35 and 40.

CONCLUSION

Final molasses of the DSR are not well exhausted with reference to the high purity values and the high target purity difference values obtained for the samples analysed (see appendix). Inversion of sucrose to reducing sugars occurs mostly in the boiling house of the refinery as a result of the operating temperature and the acidic pH. Ash content rise was also noticed in the final molasses. Efforts to reduce molasses purities would be assisted by neutral pH maintenance throughout the refining process. The new target purity formula (ASI 1993) seems to represent the Dangote Sugar Refinery conditions.

Efforts should be made to reduce the Reducing Sugar and Ash content ratio (RS/A) at the final molasses end by monitoring and maintaining low RS/A values throughout the boiling and crystallization stage. Doubling the cooling of the Massecuites will help.

Optimum pH should be maintained throughout the refining process so as to reduce sucrose inversion and reduce the prevalence of Caramelisation reaction especially at the clarification stage. This would be achieved by the installation of tight pH control systems.

The improvement of 'A' Massecuite exhaustion should be pursued as this will have a positive effect on the final molasses purity and the TPD value using the proposed target purity formula (ASI 1993). 'C' Massecuite should be cooled immediately after dropping the strike (before curing) in order to maximize 'C' sugar recovery.

High massecuite brix at the boiling house should be targeted at all times. Lower operating temperatures from melt to sugar or final molasses should be strived for at all times. Molasses back blending may also help.

Acknowledgements
The authors are grateful to the management and staff of DSR for granting the study.

REFERENCES

[1] Sahadeo P, *Proc S Afr. Sug Technol Ass*, **1998**, 72, 285 – 289.
[2] Ajandouz EH, Tchiakpe, LS, Dalle-Ore F, Benajiba, A, Puigserve, A, *Journal of Food Science*, **2001**, Vol. 66, No. 7, 926-931
[3] Sharma PC, Jain S, Yadav G, Sharma A, *Der Pharmacia Sinica,* **2010**, 1 (3): 95-108.
[4] Verma N, Bansal M, Kumar S, *Advances in Applied Science Res*earch, **2012**, 3 (2):809-814.
[5] Martins SIFS, Ph.D. Thesis, Wageningen University, (The Netherlands, **2003**).
[6] Komthong P Katoh T, Igura N, Shimoda M, Hayakawa, I, *J. Fac. Agr., Kyushu Univ.*, **2003**, 48 (1 – 2), 135-142.
[7] Ninela M, Rajoo N, *Proc. S. Afr. Sugar Technol. Ass.*, **2006**, 80, 448 -461.
[8] Newell GM, *Proc. S. Afr. Sugar Technol. Ass.*, **1979**, 53: 62 – 65.

[9] Panpae K, Jaturonrusmee W, Mingvanish W, Nuntiwattanawong C, Chunwiset S, Santudrob K, Triphanpitak S, *The Malaysian Journal of Analytical Sciences*, **2008**, Vol 12, No 3, 513 – 519.
[10] Rein PW, *The Sugar Bulletin,* **2002**, Vol. 80 No. 12: 17-19.
[11] Saska, M, Goudeau SL, Beyene F, Annual Meeting of American Society of Sugar Cane Technologists, Louisiana Division,Lafayette, **2010**, 253.
[12] Foster DH, *Mackay Techn.Rep*. **1960**, 64.
[13] Bruijn J, Fitzergerald JR, Koenig S, Macgillivary AW Proceedings of The South African Sugar Technologists' Association-June **1972**, 103 – 109
[14] Matthesius GA Mellet, P, Proceedings of The South African Sugar Technologists' Association, South Africa, **1976**, 206-207.
[15] Rein, PW, Smith, IA. *Proc. S. Afr. Sugar Technol. Ass.*, **1981**, 55, 85-91
[16] Smith IA. *Proc S Afr Sugar Technol Ass* **1995** 69, 163 – 165.
[17] Matthesius, G.A. and Mellet P *Proc S Afr Sugar Technol Ass*. **1976** 50, 206 – 207.
[18] Goza -León, CO, Kazemian, CH, Hormaza, CJ, Rojas, LYM, Santana, CR, *The International Journal of Cuban Studies* , **2008**, 2, 96 – 103
[19] Miller KF, Ingram GD, Murry JD, *Proc. Aust. Soc. Sugar Cane Technol.***1998**, 20, 506-513.
[20] Rein PW, Saska M, White BE, Audubon Sugar Institute, **2002**, Louisiana
[21] ICUMSA. ICUMSA methods book. **2007,** Bartens.

Appendix
Final Molasses Comprehensive Analysis Results

Parameters	Day 1	Day 2	Day 3	Day 4	Day 5	Average
Brix	88.4000	86.7200	87.2200	86.0600	88.0000	87.2800
pH	5.3000	5.6000	5.8000	5.1000	6.7000	5.7000
Pol	43.8800	60.0400	46.3200	35.0000	46.8400	46.4160
Apparent Purity	49.6380	69.2343	53.1071	40.6693	53.2273	53.1752
RS	24.2000	26.5000	26.4000	23.7000	28.5000	25.8600
Ash	6.2400	7.6300	8.2100	6.2000	7.4000	7.1360
RS/Ash	3.8782	3.4731	3.2156	3.8226	3.8514	3.6482
Sucrose	39.4000	44.5000	40.1000	37.6000	40.4000	40.4000
Dry Solid	85.8025	85.1930	84.8656	83.1598	85.6094	84.9261
True Purity	45.9194	52.2344	47.2512	45.2141	47.1911	47.5620
Target purity (ASI)	35.1600	35.7400	36.1600	35.2400	35.1900	35.4980
TPD(ASI)	10.7594	16.4944	11.0912	9.9741	12.0011	12.0640
Target Purity (Smith,1995)	26.5900	26.9400	27.2200	26.6300	26.6100	26.7980
TPD(Smith,1995)	19.3294	25.2944	20.0312	18.5841	20.5811	20.7640
Target purity(Rein & Smith,1981)	27.3400	28.1800	28.7700	27.4500	27.3900	27.8260
TPD (Rein&Smith,1981)	18.5794	24.0544	18.4812	17.7641	19.8011	19.7360