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Application note 003

Detecting the Addition of Cane Sugar to Authentic Palm Sugar Using $\delta^{13}C‰$ Analysis of an Internal Isotopic Standard

Palm sugar was originally made from the sugary sap of the Palmyra palm or the date palm. Now it is also made from the sap of the sago and coconut palms and may be sold as "coconut sugar." When the palms are 15 to 20 years old they start flowering and it is only then that they yield the sap from which palm sugar is made.

The sap flows when the flower stem is tapped. Palm sugar is analogous to maple syrup in that it is a premium product derived from tree sap and has a high sugar content.

Consequently, it is potentially subject to adulteration with cheaper sources of refined sugar such as cane sucrose. It is common practice to add small amounts of cane sugar to palm sugar during its production. The cane sugar might be used to 'seed' the crystallisation of the palm sugar crystals, but there is a temptation to add excessive cane sugar.

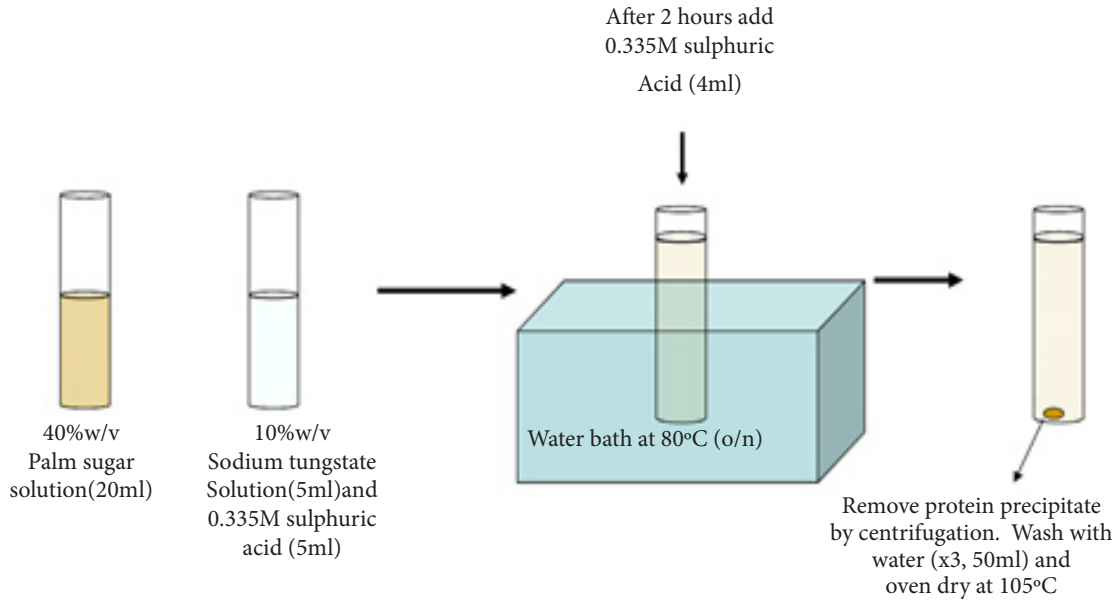
KEYWORDS: Carbon isotope, Palm sugar, cane sugar adulteration, internal Isotopic Reference, IIR, $\delta^{13}C$

Preparation of protein fraction from palm sugar (Figure 1)

10g of palm sugar is added to 20ml of de-ionised water and mixed well. The 50% w/v palm sugar solution is then filtered through a 0.22um syringe filter into a tube. Add 5 ml 10%w/v Sodium Tungstate Dihydrate ($Na_2 WO_4 2H2O$) solution and 5 ml 0.335M sulphuric acid together in a 15ml tube. Mix and immediately add to the filtered palm sugar solution, mixing well. Place the 50ml tube in water bath at about 80°C until a visible floc forms with clear a supernatant (= 2 hours).

If no visible floc forms (= 1 hour), or if the supernatant remains cloudy, add 0.335M sulphuric acid in 4 ml increments, repeating heating between additions. When a stable floc has formed centrifuge the 50 ml tube for 20 minutes at 2000 rpm. The protein floc should form a pellet at the bottom of the tube. The pellet of protein must be washed with several portions of ultrapure water to remove any residual sugars.

Figure 1: Preparation of the protein fraction from palm sugar for use as an internal ^{13}C isotopic standard



Internal isotopic referencing (IRR)

According to the literature, palm sugar should possess a characteristic C3 carbon isotope signature typical for carbohydrate derived from plants that utilise the Calvin Photosynthetic pathway to fix carbon dioxide. The average $\delta^{13}C‰$ value for C3 derived sugars is around 25‰ and cane or corn sugars or syrups (including high fructose corn syrup) average around -10‰.

The isotopically 'heavier' (less negative) carbon isotope signatures observed for corn and cane result from the Hatch & Slack photosynthetic (C4) pathway, which does not fractionate atmospheric carbon dioxide to the same extent as the Calvin C3 pathway.

Figure 2: Interpretation of palm sugar

Repeatability of protein extraction and carbon stable isotope analysis (CSIA)

The repeatability of the protein extraction and carbon stable isotope ratio measurement were assessed. Five separate 50% w/v solutions of a retail palm sugar were prepared and processed and measured according to the protocol described above.

The results of these analyses demonstrated that the procedure was repeatable. The mean $\delta^{13}C‰$ value determined from 3 triplicate determinations on each of the five separate preparations of protein was -24.19‰ with a sample standard deviation [sd ($\sigma n-1$)] of 0.16‰.

Table 1: Carbon isotope analysis of the separated protein fraction from retail palm sugars

SAMPLE I.D.	protein $^{13}C‰$	bulk $^{13}C‰$	estimate of cane sugar content
Palm sugar sample 1	-23.32	-14.04	82
Palm sugar sample 2	-20.63	-12.99	89
Palm sugar sample 3	-21.39	-13.77	81
Palm sugar sample 4	-21.21	-14.86	69

Results and discussion

The Association of Official Analytical Chemists method for the determination of C_4 sugars in honey [AOAC method 991.41] has been successfully adapted for palm sugar analysis. Assessment of the carbon stable isotope composition of bulk palm sugar and the extracted protein fraction has shown that significant quantities of exogenous cane or corn derived sugars are being used to adulterate and extend retail products on sale in the market place (Table 1 above).

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Equipment
CF 20-20 IRMS
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References
AOAC method 991.41. C-4 Plant sugars in Honey. Internal standard stable carbon isotope ratio method. First action 1998.

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