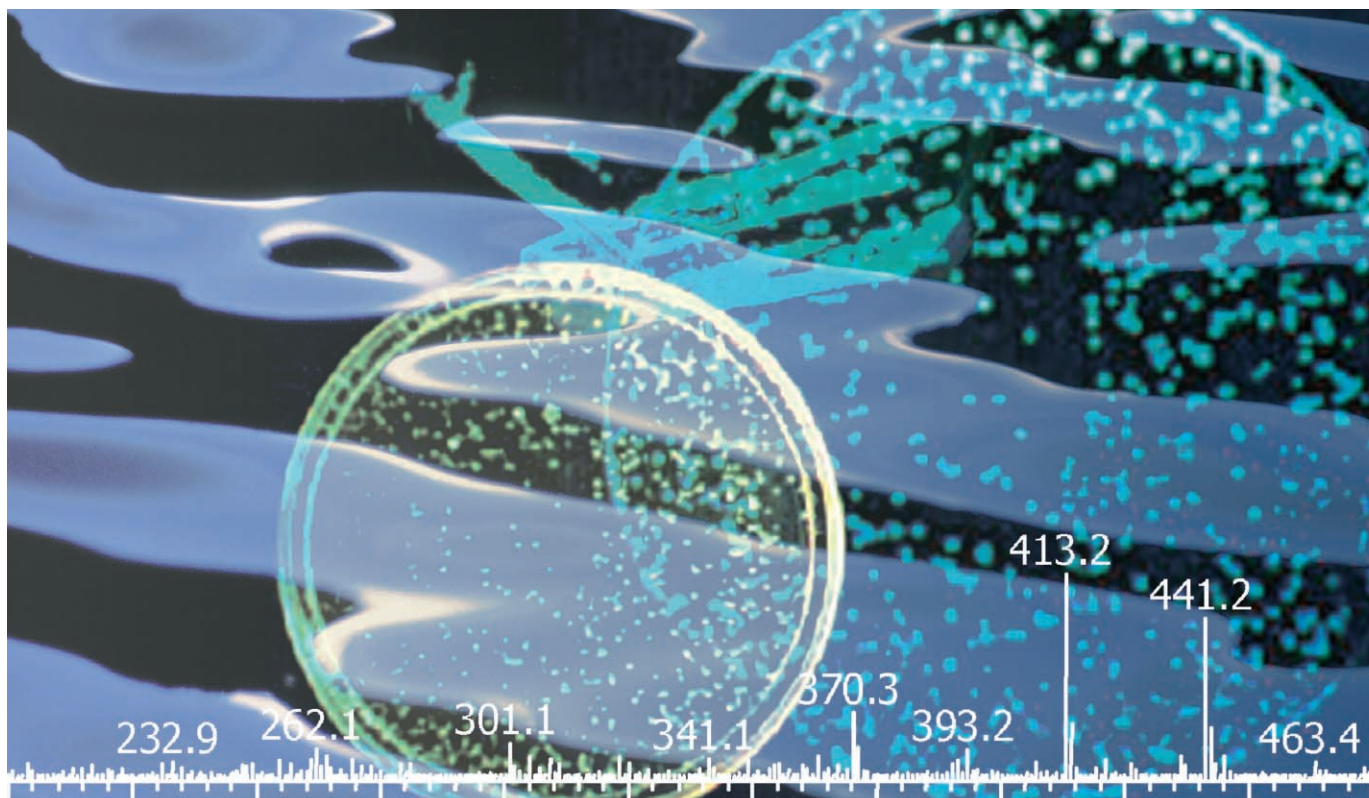


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**Specific modes of detection –
merits of planar chromatography**

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Amino phases for derivatization of sucralose in milk-based confection



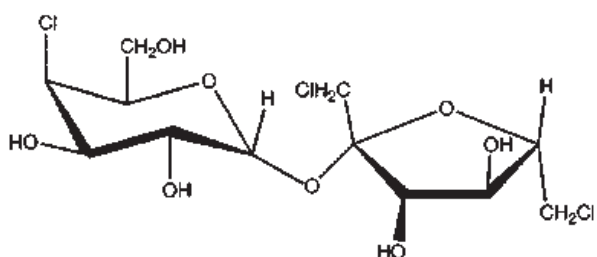
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▲ HPTLC team: Shashi Prabha, Dr. Gerda Morlock

In the last issue of CBS we outlined the working group of Prof. Schwack at University of Hohenheim, Stuttgart. In this study planar chromatography was preferred for analysis of sucralose due to the advantages offered by the derivatization step.

Introduction

Sucralose is a relatively new sweetener with a sweetening intensity of 600–650 as compared to sucrose, having no calories and a taste profile very similar to sugar without any aftertaste. Because of its exceptional heat stability, excellent solubility characteristics, and high compatibility with commonly used food ingredients, it is employed especially in USA for many low-calorie products. Since 1998 it has been approved by more than 40 countries and it is approved as an additive (E955) for use in European Countries since 2005.



▲ Structure of sucralose (4,1',6'-trichlorogalactosucrose): 1,6-dichloro-1,6-dideoxy- β -D-fructofuranosyl-(2-1)-4-chloro-4-deoxy- α -D-galactopyranoside

Regarding minor UV absorbance (< 200 nm) detection of sucralose is performed by derivatization, refractive index, pulsed amperometric detection or mass selective detector. There is evidence that planar chromatography is advantageous in this case. The amino phase itself reacts as derivatization so that no separate transfer step is needed. By just heating the plate after chromatography sucralose reacts with the amino groups of the layer to fluorescent zones (1). The derivatization step to fluorescent zones can rapidly and simultaneously be performed for all substance zones.

Sucralose was determined in Burfi, a popular Indian ethnic milk delicacy, which is produced in all kinds of modes with various food ingredients. Low-calorie burfi is produced by heat desiccation of milk accompanied by continuous stirring and addition of sucralose, maltodextrin and sorbitol instead of sucrose. Stability has been established for various storage conditions in milk-based confection.



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▲ Burfi containing pistachios, coco pulp or butter

Sample preparation

5g Burfi were suspended in water by shaking; after precipitation of proteins and extraction in a ultrasonic bath, the extract was centrifuged and filtered.

Standard solution

Sucralose dissolved in methanol (0,15 mg/100 mL)

Layer

HPTLC plates NH_2 F₂₅₄ (Merck), 20×10 cm

Sample application

Bandwise with Automatic TLC Sampler 4, 22 tracks, application volume 4 μL of sample and 2–10 μL of standard solution, band length 5 mm, track distance 7.8 mm, distance from lower edge 8 mm, distance from the side 15 mm

Chromatography

In horizontal developing chamber with acetonitrile – water 4:1, migration distance 70 mm from lower edge, migration time about 15 min, after chromatography the plate must be dried for 5 min in a stream of warm air

Derivatization

Heating of the plate for 20 min at 190 °C with TLC plate heater III

Densitometry

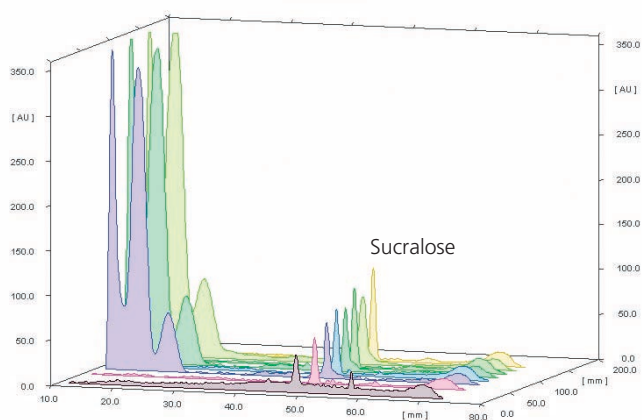
TLC Scanner 3 with winCATS software, fluorescence measurement at UV 366/>400 nm, linear calibration via peak height

Documentation

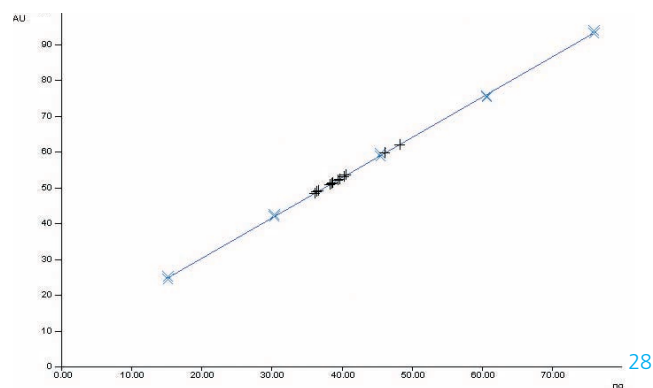
With DigiStore documentation system under UV 366/>400 nm

Results and discussion

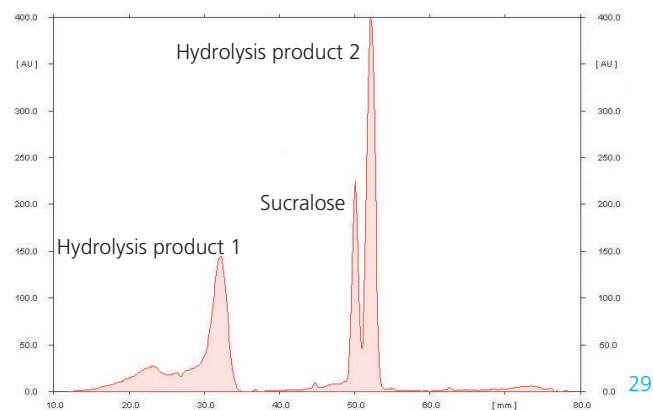
The limit of quantitation of sucralose is found to be in the lower ng range. Calibration is linear (sdv = 0.97 %, $r = 0.99977$) with results falling well within the acceptable working range. Two hydrolysis products are well separated from sucralose, thereby efficiently monitoring the degradation of sucralose in burfi samples stored under different conditions.



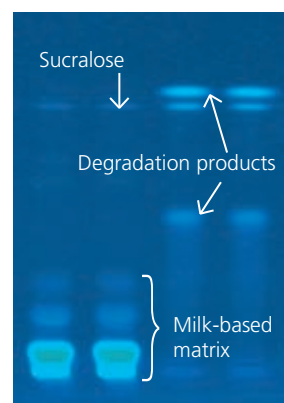
▲ 3D-graphic of standard and sample tracks



▲ Linear calibration of sucralose ($y = 1,124x + 7,817$, $sdv = 0.97\%$, $r = 0.99977$)



▲ Track display of a hydrolyzed solution of sucralose showing sucralose and degradation products



▲ Documentation under UV 366/>400 nm; track 1 and 2: sample, track 3 and 4: hydrolyzed solution of sucralose

Further information is available from the authors on request.

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