

milk compared to a declining level of absorption with whole cow milk.

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## Food Additives Derived from Lactose: Lactitol and Lactitol Palmitate

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Lactitol is prepared by hydrogenation of a lactose solution at about 100 °C with a Raney nickel catalyst. After purification a lactitol syrup is obtained, which is crystallized. A crystalline lactitol monohydrate and a noncrystallizing lactitol syrup are the end products. The properties and applications as well as biological data and toxicity studies of lactitol are described. Lactitol palmitate is prepared by direct esterification with fatty acids of edible fats in such a way that formation of anhydropolyols is minimized. The reaction is carried out at a relatively low temperature of about 160 °C with soaps of the fatty acids as catalyst. The lactitol esters can be used as emulsifiers in foods or as detergents.

The title of this paper is, strictly speaking, somewhat optimistic; lactitol and lactitol palmitate are not yet officially approved as Food Additives by the Food and Drug Administration or the FAO/WHO.

In this study it will be shown, however, that these lactose-based derivatives have interesting potentialities as food additives. Pure lactose is abundantly available as edible or pharmaceutical grade; a considerable amount of lactose is produced in the Netherlands with an annual production of 65 000 tons in 1975. The world production of lactose is about three times as big, whereas the lactose potentially available from all the whey in the world, there is a potential availability of 3-4 million tons of lactose per annum.

#### LACTITOL

**Preparation.** The preparation of lactitol by hydrogenation of lactose with a nickel catalyst is already known for a long time (Karrer and Büchi, 1937; Wolfram et al., 1938). Lactitol can be prepared by reduction of lactose with sodium borohydride (Scholnick et al., 1975), but technically lactitol is prepared by hydrogenation of a lactose solution at about 100 °C with a Raney nickel catalyst. As lactitol is produced essentially in the same way from lactose as sorbitol is produced from glucose, only

a short summary of the reaction conditions is mentioned here. The reaction is carried out in an autoclave under a pressure of 40 atm or more. Due to the lower solubility of lactose, compared to glucose, the lactose concentration at the beginning of the reaction is only 30-40 wt %. When the hydrogenation reaction is completed, the catalyst is sedimented and filtrated and the lactitol solution is purified by ion-exchange resins and activated carbon. The purified lactitol solution is then concentrated by evaporating the water, the obtained syrup is crystallized, and the crystals are separated with a centrifuge and finally dried. The lactitol mother liquor is concentrated again, giving more crops of the lactitol monohydrate. The final mother liquor can be used as a 64% solution or in mixtures with sorbitol to prepare a noncrystallizing lactitol syrup at a concentration of 70%.

**Chemical and Physical Properties.** Lactitol is a sugar alcohol, derived from lactose by reduction of the glucose part of this disaccharide. Synonyms for lactitol are lactit, lactositol, and lactobiositol. Lactitol (C<sub>12</sub>H<sub>24</sub>O<sub>11</sub>, mol wt = 344) has the structural formula illustrated in Figure 1. The systematic name is 4-β-D-galactopyranosyl-D-sorbitol. On hydrolysis the molecule is split into D-galactose and D-sorbitol, both occurring widely in nature. Due to the absence of a carbonyl group, lactitol is chemically more stable than the related disaccharides like lactose. The stability of lactitol in the presence of alkali is markedly higher than that of lactose. Heating for 1 h at 100 °C of

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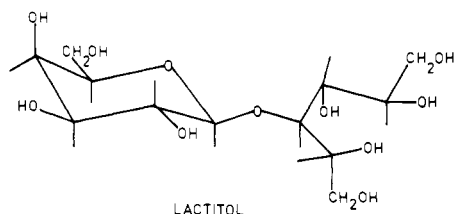


Figure 1. Structural formula of lactitol.

Table I. Heat Resistance of Lactitol: Color Values after Heating for 2 h at Different Temperatures

heating temp, °C	color value (Gardner scale)
150	colorless
160	1
170	3
180	5
190	6
200	7

Table II. Solubility Data of Lactitol Monohydrate

solvent	temp, °C	solubility, g/100 g of solvent
water	25 °C	206
	50 °C	512
	75 °C	917
ethanol	25 °C	0.75
	50 °C	0.88
ether	25 °C	0.40
Me <sub>2</sub> SO	25 °C	233
DMF	25 °C	39

a 10% lactitol solution at pH 13 (adjusted with NaOH) does not produce any discoloration, whereas a lactose solution, heated under the same conditions, shows strong discoloration (color no. 18 on the Gardner scale). The stability of lactitol in the presence of acids is comparable to that of lactose (Wolfrom et al., 1938). When heated at 100 °C for 4 h at pH 1 and 2 (adjusted with HCl) the rates of hydrolysis of 10% lactitol solutions are 5.6 and 1.4%, respectively. The solutions remain colorless under these conditions. The comparable hydrolysis of lactose are 5.4 and 1.3%, respectively. Like other sugar alcohols lactitol shows only a very slight discoloration, as a result of caramelization reactions, when heated at temperatures of 150–200 °C. This is illustrated in Table I. A further consequence of the absence of a carbonyl group is the impossibility for lactitol to react with amino compounds, like amino acids and proteins, causing undesired brownings of food products.

Lactitol monohydrate has a melting range of 94–97 °C; the water of crystallization is evaporated in the range of 145–185 °C. By heating lactitol (solution) at reduced pressure dehydration takes place at lower temperatures (e.g., 95 °C, 25 mmHg). When heated at temperatures of 170–240 °C lactitol is partly converted into anhydro derivatives (lactitan), sorbitol, and lower polyols. Lactitan formation is accelerated by *p*-toluenesulfonic acid. The specific rotation is  $[\alpha]^{22}_D = +12.3^\circ$ . The heat of solution of lactitol monohydrate at 25 °C is  $-12.7 \text{ cal/g}$  (53.2 kJ/kg). Lactitol monohydrate is a white, odorless, nonhygroscopic crystalline solid with a bland sweet taste. In contrast to the monohydrate, the anhydric form is strongly hygroscopic. Lactitol is soluble in water, Me<sub>2</sub>SO, and DMF and slightly soluble in ethanol and ether (Table II). Lactitol is nonvolatile and is completely miscible with other polyols like sorbitol solution and glycerol. Lactitol monohydrate is less hygroscopic than sorbitol and xylitol, but is (only at high relative humidities) more hygroscopic than mannitol. This is demonstrated in Table III which shows the moisture pick up of crystalline lactitol, sorbitol, xylitol,

Table III. Moisture Absorption of Crystalline Polyols at Different Relative Humidities

% rel. humidity	storage time, days	% moisture absorption at 25 °C			
		lactitol	sorbitol	xylitol	mannitol
55	1	0.1	0.8	0.1	0.1
	2	0.1	0.8	0.1	0.1
	3	0.1	0.8	0.1	0.1
65	1	0.1	2.2	0.1	0.2
	2	0.1	2.2	0.1	0.2
	3	0.1	2.2	0.1	0.2
75	1	0.3	5.5	0.3	0.7
	2	0.3	7.7	0.3	0.7
	3	0.3	8.8	0.3	0.7
85	1	1.7	12.0	4.3	1.0
	2	2.1	22.4	9.6	1.0
	3	2.3	32.3	15.1	1.0
95	1	5.4	17.3	11.5	1.9
	2	10.3	31.7	23.1	1.9
	3	16.1	42.8	34.5	2.0

Table IV. Typical Analysis of Lactitol

description	lactitol monohydrate	noncrystallizing syrup
	white crystalline powder	colorless solution
taste	sweet	sweet
water	5.5%	29.5–30.5%
lactitol	90%	48.5–50.5%
mannitol	3%	3%
sorbitol	0.5%	16.5–17.5%
dulcitol	0.5%	0.4%
lower polyols	0.5%	1.0%
reducing sugars	0.1%	0.1%
ash	max 0.1%	max 0.1%
heavy metals	max 10 ppm	max 10 ppm
chloride	max 70 ppm	max 70 ppm
sulfate	max 100 ppm	max 100 ppm

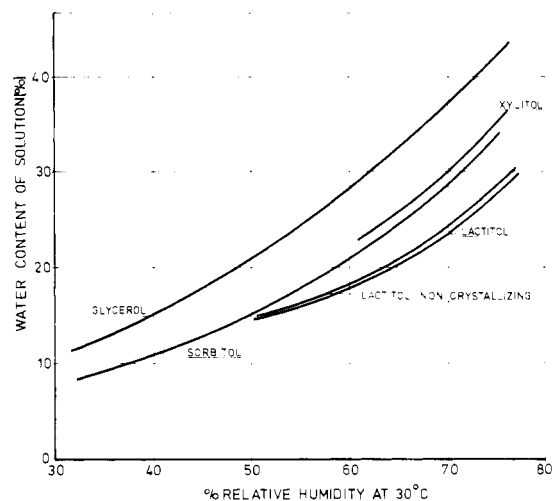


Figure 2. Equilibrium relative humidities of polyol solutions at 30 °C.

and mannitol at different relative humidities. This table shows that only at very high relative humidities moisture is picked up by lactitol, but even then the rate of moisture uptake is much lower than it is for sorbitol and xylitol. The hygroscopic behavior of lactitol solutions is demonstrated in Figure 2, showing the equilibrium relative humidities of lactitol, sorbitol, glycerol, and xylitol solutions. Of these four polyol solutions, lactitol possesses by far the weakest hygroscopicity.

The viscosities of lactitol solutions as functions of concentration and temperature as shown in Figure 3. The viscosity of a 70% noncrystallizing syrup at 20 °C is about

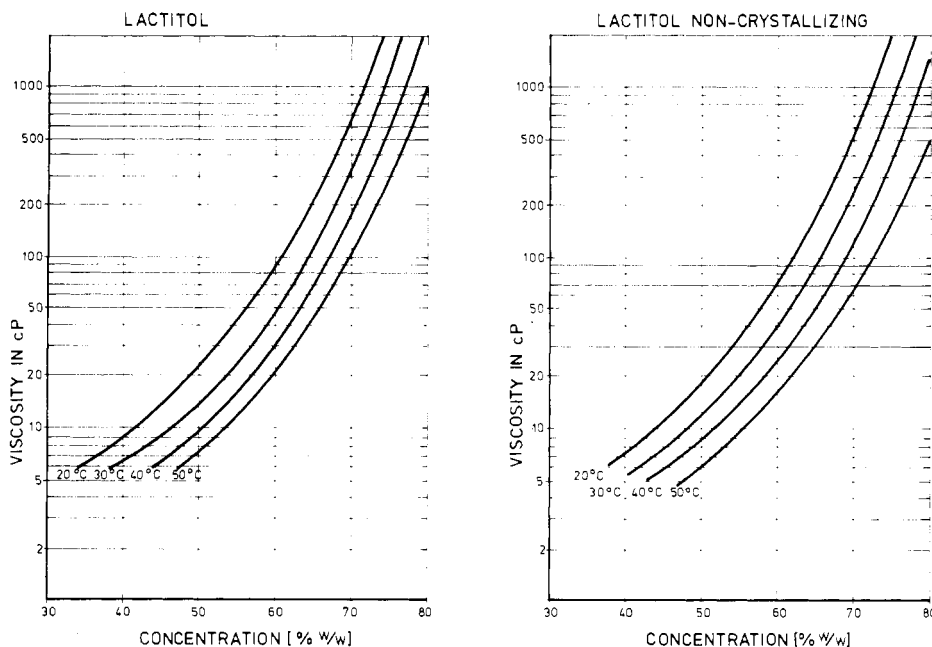


Figure 3. Viscosities of lactitol solutions.

Table V. Relative Sweetness of Polyols

concn (% w/w) of sucrose ref solns	concn of polyol solns of equal sweetness (% w/w)			rel sweetness of polyols		
	lacti- tol	sorbi- tol	xyli- tol	lacti- tol	sorbi- tol	xyli- tol
2	6.7	4.3	2.1	30	47	95
4	11.4	7.7	4.2	35	52	95
6	16.2	10.3	6.2	37	58	97
8	20.5	12.5	8.2	39	64	97

550 cP; a 64% lactitol solution has a viscosity of about 200 cP and a 70% sorbitol solution has a viscosity of about 150 cP.

Specifications for the crystalline lactitol monohydrate and the noncrystallizing syrup are given in Table IV. The specification for the lactitol 64% solution, on dry substance basis, is similar to that for the crystalline material.

Relative sweetness of lactitol, sorbitol, and xylitol, determined in triangle tests by a taste panel at four different sucrose concentrations are given in Table V. The relative sweetness of lactitol, depending of the concentration, is about 35% compared with sucrose, whereas sorbitol is 1.5 times more sweet than lactitol.

**Biological Data and Toxicology of Lactitol.** *Enzymatic Hydrolysis and Absorption of Lactitol.* Karrer and Büchi (1937) have studied the action of  $\beta$ -galactosidase-containing enzyme preparations on the splitting of lactitol into galactose and sorbitol. They found that lactitol is only hydrolyzed very slowly by these enzyme preparations. Later studies reported in a German patent (Maizena, 1974) have confirmed that lactitol is only slowly split by enzymes with about a tenth of the speed at which lactose is split. Based on both these in vitro studies a reduced caloric value of lactitol can be expected. In patents (Hayashibara, 1971, 1976) it is claimed that lactitol has no caloric value because it is not digested or absorbed by digestive organs of a higher animal. This was demonstrated by experiments with living rabbits. The intestines of test rabbits not fed for 24 h beforehand were closed at both ends and were injected with a 20% aqueous solution of lactitol or with an equimolar amount of a sucrose solution. After a lapse of several hours the lactitol or sucrose left in the intestines was determined. It was

then found that, while 85% of the sucrose intake had been lost due to absorption and digestion, all of the lactitol was still present.

Studies at the University of Groningen on the absorption of lactitol by the intestine of human beings, primarily to obtain evidence on the caloric utilization of lactitol, have not yet given a complete picture of the metabolism of lactitol in men. In these studies (van Velthuisen, 1977; Doorenbos, 1977), it has been demonstrated that after consumption of 24 g of lactitol, it is not detected in the blood and that less than 0.5% of the consumed lactitol is recovered in the urine, while in the feces only negligible amounts were recovered. Apparently lactitol is predominantly not absorbed or hydrolyzed in the small intestine; in the large intestine lactitol is used by the microflora. It was not possible to conclude to what extent the microbial metabolites from lactitol have caloric value. In the feces no increased contents for lactic acid or galactose was found.

*Effect of Lactitol on Sugar Absorption.* In a patent application (Hayashibara, 1974) it was demonstrated with rats that lactitol inhibits the absorption of sucrose and also the formation of cholesterol. The increase of the blood glucose content after consumption of a 1:1 mixture of sucrose and lactitol was about half of the increase after consumption of sucrose only (the amount of sucrose intake was the same), whereas the formation of liver glycogen with the 1:1 mixture was only one-fifth of that with only sucrose. Diets with added cholesterol showed that the inclusion of lactitol in the diets of rats resulted in a reduction to about 50% of the contents of liver cholesterol and of the total serum cholesterol.

*Tooth Protective Properties.* At the School of Dentistry of the University of Utrecht (Havenaar, 1976; Havenaar et al., 1978) a microbial study was executed on the influence of lactitol on the dental plaque formation by *Streptococcus mutans*. It was demonstrated that *Streptococcus mutans* and other bacteria isolated from the dental plaque form acid from lactitol, but the acid formation is rather slow, comparable to that of sorbitol. It was proved that no polysaccharides were formed by nonadapted strains. After adaptation of *Streptococcus mutans* to sorbitol and lactitol by subculturing 20 times some intracellular polysaccharide formation occurred

Table VI. Sensory Evaluation of Strawberry Jams with Different Polyols after 0, 3, 6 and 12 Months, of Storage

polyol	0 months			3 months			6 months			12 months		
	rank sums	no. of panelists	block limits $P < 0.05$	rank sums	no. of panelists	block limits $P < 0.05$	rank sums	no. of panelists	block limits $P < 0.05$	rank sums	no. of panelists	block limits $P < 0.05$
lactitol	31.5		27	47		43	39		38	51		41
			till		25	till		22	till			till
sorbitol	28	16	37	48		57	46		50	46		55
xylitol	36.5			55			47			47		

(Havenaar et al., 1978), but this capacity disappeared after one subsequent cultivation of the adapted strain in glucose.

At the University of Zürich (Schneider and Mühlemann, 1976), a special method has been developed for the in vivo determination of the plaque pH in human. After consumption of chocolate or confectionery in which the sucrose is replaced by lactitol, changes in plaque acidification are detected by the electrodes and transmitted electronically to a graph recorder.

The term "zahnschonend" (tooth protective) is used in Switzerland when the pH of dental plaque does not increase below 5.7 during a 30-min period. It is demonstrated (Mühlemann, 1977) in chocolates, that lactitol was "tooth protective". That means that it does not increase the incidence of dental caries.

In vitro tests in our laboratory with bacteria whose habitat is human dental plaque or intestine, have shown that *Escherichia coli* do not produce acids or gas from lactitol. Several *Lactobacillus sp.* produce as much total acids from lactitol as from lactose; however, from lactitol the acid formed is predominantly L-(+)-lactic acid. *Bifidobacteria* produce much less acids from lactitol than from lactose.

**Lactitol and Diabetes.** In a study of the Medical College of the University of Groningen (Doorenbos, 1977) on the effect of lactitol on blood glucose and insulin content in human blood, with diabetics and "normal" people, it has been proved that lactitol is potentially a useful sweetener for diabetics. Consumption of lactitol does not influence the blood glucose content nor the insulin of either group of persons.

**Laxative Properties.** In order to compare the relative laxative properties of lactitol, xylitol, sorbitol, and lactose, the T.N.O. Institute has carried out a study with rats (de Groot, 1976). When fed at a level of 10% in the diet, lactitol, xylitol, and sorbitol were distinctly more purgative than lactose, while xylitol was slightly more active than lactitol and sorbitol. When fed at the 5% level, lactitol was less purgative than xylitol and sorbitol, but comparable to 10% lactose. Our experience with human beings is that unpleasant physiological side effects of lactitol consumption (osmotic diarrhea, intestinal gas formation) soon disappear at regular consumption of this polyol. When lactitol is consumed in confectionery products, single portions of about 20 g do not cause any unpleasant effect in adapted persons; when smaller portions are taken spread out over a day, far higher daily doses can be consumed without noticeable effect.

#### TOXICITY STUDIES

The Central Institute for Nutrition and Food Research T.N.O. (Zeist, Holland) has executed a subchronic (90 days) toxicity study with lactitol in rats (Sinkeldam et al., 1976). The test substance was fed at dietary levels of 5, 10, and 20% at the expense of equal amounts of wheat starch. A diet containing 25% lactose was included for comparison. Slight diarrhea was noticed in the 20%

lactitol and 25% lactose groups. Growth was retarded in a dose-related way in all groups receiving lactitol. The relative weights of the kidneys and liver were increased in the groups fed 20% lactitol. The cecum weights were increased by feeding both lactose and lactitol. Microscopical liver changes were dose related in the 10 and 20% lactitol group, but absent in the 5% lactitol group. It was concluded that the nontoxic effect level of lactitol was 5% in the diet, corresponding to a noneffect level of 2.5 g (kg body weight)<sup>-1</sup> day<sup>-1</sup>. The higher dose levels do not give real toxic effects, but as long as it is not clear what the meaning is of certain metabolic effects, the 5% dose level will be used for the determination of the A.D.I.

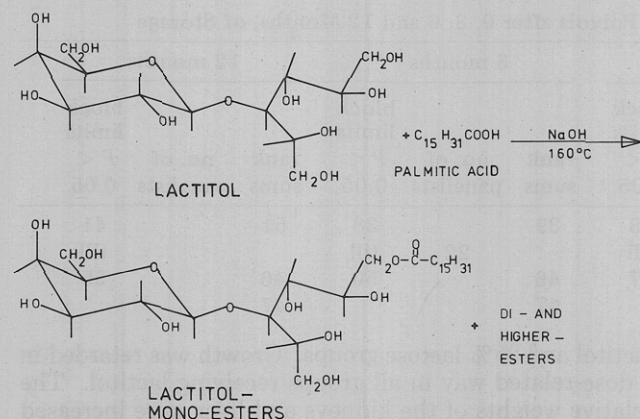
The problem that needs to be solved is to establish the safety factor for man. The normally used safety factor of 100× can not be used for substances like lactitol, sorbitol, xylitol, and sugars like lactose. The metabolic effects implied are related to the laxative properties of sugars and sugar alcohols, to which rats are very sensitive. A safety factor of 2 or 3, therefore, seems to be more realistic than a factor of 100.

**Applications.** Lactitol offers many interesting applications for the food industry, especially for the fields of dietetic and low-caloric foods. In many products lactitol can replace sucrose without drastic changes in formulations or manufacturing processes. Depending on the specific application, lactitol can be used as a solution (e.g., for jams, beverages, hard candies) or as the crystalline, nonhygroscopic monohydrate (e.g., for chocolate, chewing gum, bakery products). Some of the food products in which lactitol can be used are given below.

**Jams and Marmalades.** In these products sucrose can be replaced completely by lactitol, requiring only minor adaptations in formulations. Manufacturing processes do not need to be changed. In a sensory evaluation, using the ranking tests of Kahan et al. (1973) of strawberry jams in which sucrose was replaced in a 1:1 ratio by lactitol, sorbitol, or xylitol, a rank order for total appreciation of pleasantness of taste is given in Table VI. Significant differences among these polyols were not observed. Although there was no preference with regard to total pleasantness, there were differences in sweetness. The jam with lactitol was less sweet, but some panelist prefer the less sweet jam. If desired, the weaker sweetness of the lactitol jam can be increased by adding synthetic sweeteners.

**Chocolate.** In chocolate products sucrose can be replaced by lactitol in a 1:1 ratio. Lactitol enhances the chocolate flavor and improves the keeping qualities of the products. The use of lactitol does not require modifications in the manufacturing process.

**Hard Candy.** In the production of hard candies sucrose and glucose can be entirely replaced by lactitol. Lactitol candies can be manufactured using conventional equipment; after cooling down the cooked products and addition of acid and flavor, the mass can be drawn into a rope and cut or stamped into candies. If necessary the sweetness



**Figure 4.** Reaction scheme for the conversion of lactitol into lactitol palmitate.

of the lactitol candies can be increased by adding synthetic sweeteners. The concentration of added sweetener should be as low as possible in order to avoid a bitter aftertaste of the candies.

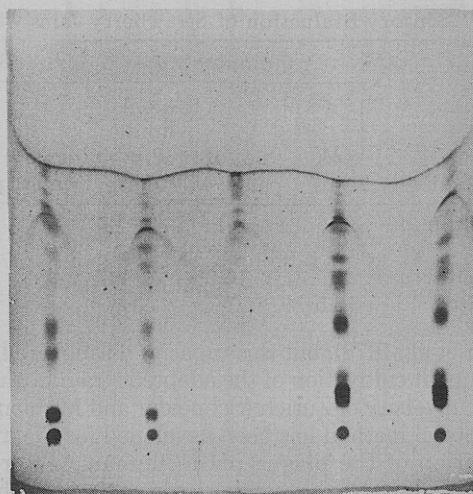
**Bakery Products.** Due to its weak hygroscopicity lactitol can be used in various diabetic fine bakery wares, especially in crisp products. It has been successfully used in light biscuit-like products for diabetics. These products, having a specific volume of 4–5 cm<sup>3</sup>/g had a total carbohydrate content below 10% and a sugar content below 4%. Texture and eating properties were fully comparable to conventional, sucrose-based products.

Being nonhygroscopic, lactitol can be used as a coating for moisture sensitive sweets, e.g., jellies. Coating of the surface of these products with crystalline lactitol will protect these products against water absorption. Other confectionery products in which lactitol can be used are chewing gum, jellies, fondant, etc. Finally, its possible applications in the soft drink field should be mentioned.

#### LACTITOL PALMITATE

**Preparation.** Esters of polyols (polyalcohols) and higher fatty acids are frequently used as nonionic emulsifying agents. Well known are the sorbitan esters and the sucrose esters of fatty acids.

We have found that polyalcohol esters can be obtained, containing a minimal percentage of anhydro compounds, if the fatty acid is esterified in the presence of soap at a temperature between 100 °C and 190 °C with simultaneous elimination of the water formed during the reaction. Lactitol palmitate is prepared (Figure 4) at 160 °C in the presence of fatty acid soap, in which case only a very small part of the lactitol is converted to anhydro compounds (2.5%). Soap is a catalyst and a miscibility promoting agent. The fatty acid soap may be added as an alkali metal salt of the fatty acids or prepared "in situ" by adding, for instance, alkali hydroxides. The quantity of soap for the esterification of lactitol is 20–30% by weight of the quantity of lactitol. By the choice of the reaction temperature and quantity of soap, the reaction velocity can be influenced. At higher temperatures a darker color is obtained, the usage of more soap results in shorter reaction time. A characteristic difference with the preparation of sucrose esters is due to the higher stability of lactitol so that it can withstand the relatively long reaction times needed for the direct esterification with fatty acids in a heterogenous reaction mixture without a solvent. Sucrose has eight hydroxyl groups which can be esterified, whereas lactitol contains nine hydroxyl groups. The hydrophilic-lipophilic balance of the lactitol esters is varying by modification of the percentage of monoester content. By



**Figure 5.** Thin-layer chromatography of lactitol palmitate, mole ratio respectively 0.7:1, 0.4:1, 0.2:1, sucrose stearate HLB 7 and sucrose stearate HLB 11 (from left to the right). After spraying with aniline-diphenylamine-phosphoric acid reagent.

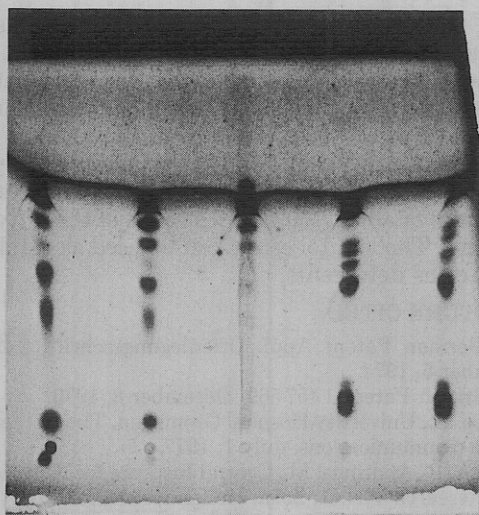
regulating the molar ratio lactitol/fatty acid, the content of mono- and diesters and higher esters can be varied. For preparation of the fatty ester with a high monoester content, an excess of lactitol has to be used; however, the more lactitol used in the reaction mixture, the higher the viscosity of the reaction mixture. The molar ratio lactitol/fatty acid is practically limited to 1.5:1, as a result of very high viscosity of the product which can not be stirred sufficiently. A molar ratio of 0.7:1 results in a mono-ester, a moderate viscous reaction product.

For preparation of the higher esters of lactitol, like lactitol penta- and hexapalmitate, it is preferably executed in a stepwise manner: first an ester mixture with a molar ratio of 0.4:1.0 is prepared and after reaction to a homogenous melt an additional amount of fatty acid is added to convert the primary product to higher esters (the amount of anhydro compounds increases to 8%).

For more experimental details of the preparation of these polyol esters, we can refer to the patents in several countries: C.C.A., 1974, 1976; Heesen et al., 1976. According to this process a variety of products with different properties can be prepared, which can be used in analogy with the known emulsifiers on the basis of glycerol, sorbitan, and sucrose.

**Analysis.** The composition of the lactitol esters is determined by means of quantitative thin-layer chromatography. The esters are separated on silica gel thin-layer plates. For the determination of the content of the mono- and diesters and the sum of tri- and higher esters, the elution is carried out with chloroform-acetic acid-methanol-water (80:10:8:2, v/v). The separated hydrophilic lactitol esters are made visible by spraying with an aniline-diphenylamine-phosphoric acid reagent (0.75 g of aniline, 0.75 g of diphenylamine in 50 mL of ethanol with 5 mL of 85% phosphoric acid) and heated for 30 min at 110 °C. In order to make also the nonhydrophilic components visible, the plate is sprayed with 10% sulfuric acid and charred at 200 °C, during 30 min.

Concentrations are measured on the plate by means of a Vitatron TLD100 "flying spot" densitometer, referring them to standard mixtures of known concentrations. The retention of the various components of the lactitol fatty acid esters correspond with those of sucrose fatty acid esters. This is demonstrated in Figures 5 and 6, pictures of thin-layer plates, showing the separated components of the fatty acids of lactitol and sucrose, after spraying with



**Figure 6.** Thin-layer chromatography of lactitol palmitate, mole ratio respectively 0.7:1, 0.4:1, 0.2:1, sucrose stearate HLB 7 and sucrose stearate HLB 11 (from left to the right). After spraying with 10% sulfuric acid and charring.

**Table VII.** Emulsion Stability Data

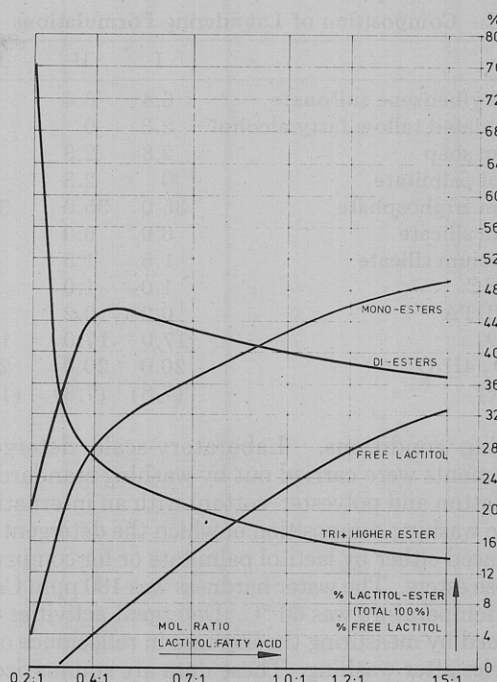
emulsifier	% upper phase after 24 h
lactitol mono-dipalmitate	1
sorbitan monolaurate	8
sorbitan monopalmitate	37
glycerol monostearate	6

the two mentioned methods.

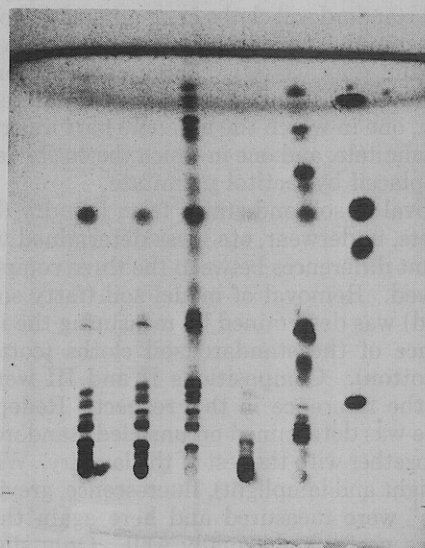
Figure 7 is an illustration of the influence of the molar ratio lactitol/fatty acid on the composition of the reaction product. Figure 8 is a picture of a thin-layer plate, showing the components of several emulsifiers and a highly esterified lactitol palmitate, after elution with a mixture of benzene, ether, and methanol (75:20:5, v/v) and charring with sulfuric acid.

**Properties and Applications.** For several applications a certain analogy was found between the properties of lactitol esters and sucrose esters.

The emulsifier properties of lactitol mono-dipalmitate were determined as follows. Five grams of the emulsifier was dispersed in 450 g of water. Then 50 g of soya oil was added and this mixture was homogenized. The emulsions were poured into 100 mL of calibrated cylinder, and the emulsion stability was determined after 24 h by measuring the percentage of upper layer of the emulsion. The results are shown in Table VII. Results obtained to date indicate that higher lactitol fatty esters (containing at least four fatty acid groups per molecule of lactitol) are hydrolyzed to a very minor degree by the human digestive system and hence are low in caloric value. The higher esters may prove to be useful low caloric fat substitutes for various fat containing food products. An advantage of the lactitol



**Figure 7.** Influence of the molar ratio lactitol/fatty acid on the composition of the reaction product.



**Figure 8.** Thin-layer chromatography of lactitol palmitate, mole ratio respectively 0.7:1, 0.4:1, 0.2:1, sucrose stearate HLB 11, sorbitan stearate, and mono diglyceride (from left to the right). After spraying with 10% sulfuric acid and charring.

esters is that in most applications they can be used as an unpurified product. The unpurified product containing soap and free lactitol can easily be milled to a fine powder.

Lactitol palmitate shows promising detergent activity in laboratory-scale trials as well as under household

**Table VIII.** Detergency Data of Lactitol and Sugar Esters Expressed as Increase of Reflectance after Washing of the Test Fabric

detergent		no surfactant	ethoxylated tallow-fatty alcohol	lactitol mono-dipalmitate (0,7:1)	sugar ester HLB 7	sugar ester HLB 14
stain type	fabric type					
fat + soil stains	cotton	14.6	19.4	19.9	11.7	21.4
	PE cotton	13.4	23.4	23.8	13.1	26.2
food stains	cotton	7.8	4.5	17.3	15.6	15.9
	PE cotton	37.0	36.7	39.2	40.1	40.6
natural colors	cotton	25.2	22.9	22.4	22.3	24.4

Table IX. Composition of Laundering Formulations

	I	II	III
lin. alkylbenzene sulfonate	6.4	6.4	0
ethoxylated tallow fatty alcohol	2.3	0	0
sodium soap	2.8	2.8	0
lactitol palmitate	0	2.3	5.4
sodium triphosphate	35.0	35.0	35.0
sodium silicate	6.0	6.0	6.0
magnesium silicate	1.5	1.5	1.5
Na-CMC	1.0	1.0	1.0
Na <sub>4</sub> -EDTA	0.2	0.2	0.2
Na <sub>2</sub> SO <sub>4</sub>	17.0	17.0	17.0
NaBO <sub>3</sub> ·4H <sub>2</sub> O	20.0	20.0	20.0
(water)	(7.8)	(7.8)	(13.9)

laundering conditions. Laboratory-scale detergency measurements were carried out by washing standard soil cloths (cotton and polyester-cotton) with an international reference washing composition of which the detergent part was replaced either by lactitol palmitate or for comparison by sucrose esters. The water hardness was 180 ppm CaCO<sub>3</sub> and the temperature was 60 °C. Detergent activities were determined by measuring the increase in reflectance of the test fabrics after washing. These data are summarized in Table VIII.

The performance of lactitol palmitate under household laundering conditions was determined by washing normal laundry and standard soil cloths in an automatic household washing machine. The water hardness in these experiments was 162 ppm CaCO<sub>3</sub>. Three compositions were investigated (Table IX): the international reference composition, one in which the nonionic part was replaced by lactitol palmitate, and one in which the entire detergent part was replaced by lactitol palmitate.

The removal of soil and stains from laundry (kitchen towels, sheets, underwear, etc.) was determined visually. No significant differences between the three compositions were observed. Removal of model soil (fatty soil, food stains, blood) was determined by measuring the increase in reflectance of the standard soil cloths (cotton and polyester-cotton). Compositions II and III were comparable to the reference in this respect. Redeposition performance was determined on soiled standard cloths laundered together with the rest of the laundry. Whiteness (under daylight and lamplight), fluorescence, greying, and "yellowness" were measured and here again the three compositions performed equally well. Only slight differences in foaming were observed. Composition I had the strongest, composition III the weakest foaming properties.

#### SUMMARY

Lactitol is prepared by hydrogenation of a lactose solution at about 100 °C with a Raney nickel catalyst. After purification a lactitol syrup is obtained, which is crys-

tallized. A crystalline lactitol monohydrate and a non-crystallizing lactitol syrup are the end products. The properties and applications as well as biological data and toxicity studies of lactitol are described. Lactitol palmitate is prepared by direct esterification with fatty acids of edible fats in such a way that formation of anhydropolyols is minimized. The reaction is carried out at a relatively low temperature of about 160 °C with soaps of the fatty acids as catalyst. The lactitol esters can be used as emulsifiers in foods or as detergents.

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