

# PHYSICS AND MATHEMATICS

## DETERMINATION OF PROPIONIC ACID AND ITS SALTS IN BAKERY PRODUCTS

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### Abstract

The conditions and parameters of chromatography were selected for the quantitative determination of propionic acid and its salts in bakery products using high-performance liquid chromatography. Validation studies of the developed measurement technique confirmed its suitability for determining the content of propionic acid and its salts in bakery products in the range from 200 to 3000 mg/kg. It was shown that in 25% of the samples of small-piece bakery products, lamb products and donuts we examined, an excess of the maximum permissible level of propionic acid and its salts was found.

**Keywords:** bakery products, propionic acid, salts of propionic acid, high-performance liquid chromatography, validation of measurement method.

**Introduction.** A flexible and at the same time stable technological process for the production of high-quality bakery products is impossible without the targeted use of microingredients - food additives, baking improvers and other components [3]. These ingredients include: ascorbic acid E300, calcium peroxide E930, azodicarbonamide E927a, calcium sulfate E516, calcium lactate E327, sorbitol E420, calcium propionate E282, etc. [4]. Our analysis of scientific and technical literature indicated that a serious problem is microbiological spoilage of bakery products, to combat which propionic acid and its salts are used [5].

Propionic acid E280 is a monobasic saturated carboxylic acid ( $\text{CH}_3\text{CH}_2\text{COOH}$ ) – a colorless, caustic liquid with a pungent odor. A natural source of propionic acid is the waste products of bacteria of the genus *Propionibacterium*. Industrial production of E280 is carried out using chemical synthesis. It has been established that propionic acid does not have toxic, mutagenic or carcinogenic properties, and it does not have a cumulative effect. However, the possibilities of using propionic acid in the food industry are limited due to changes in the taste of the finished product under its influence. Therefore, propionates are usually used in food production - E281, E282 and E283. Salts of propionic acid (sodium propionate E281 ( $\text{CH}_3\text{CH}_2\text{COONa}$ ), calcium propionate E282 ( $\text{Ca}(\text{C}_2\text{H}_5\text{COO})_2$ ) and potassium propionate E283 ( $\text{CH}_3\text{CH}_2\text{COOK}$ )) are crystalline white powders with a pungent odor, highly soluble in water. Of all the salts of propionic acid, the preservative

E282 is most often used in the food industry to prevent the growth of unwanted microflora. The preservative is used in the production of bakery and flour confectionery products, cheeses and cheese products, and is also included in cosmetics and medicines.

Propionic acid and its salts in food products can be identified using the IR spectrum, thin-layer or paper chromatography, and quantified using high-performance liquid chromatography. Currently, our country uses two standard methods for determining propionic acid in food products using high-performance liquid chromatography. In dairy products - according to GOST 31504 [2], in complex food additives - according to GOST R 70224 [1]. The main difference between these methods is the use of different chemical compositions and quantities of mobile phases. Considering the above, it was of interest to determine the possibility of adapting these methods for the determination of propionic acid and its salts in bakery products.

**Aim.** Determining the possibility of using the high-performance liquid chromatography method for the quantitative determination of propionic acid and its salts in bakery products.

**Materials and methods.** The objects of the study were samples of bakery products (4 samples of buns, 2 samples of tortillas and 1 sample each of bagels and donuts), the recipe of which contains propionic acid and its salts (Table 1).

Table 1

Characteristics of research objects		
Item No.	Research object	Compound
1	2	3
1	Wheat tortilla «Mutlukal» with spinach	Wheat flour, vegetable oil, salt, sugar, spinach powder and spice mixture E422, raising agents E500, E450, emulsifier E471, preservatives E202, E282, acidity regulators E296, E330
2	Bakery products «Rullets» with poppy seeds	Wheat flour, drinking water, Vienna poppy filling, sunflower oil, water, mono- and diglycerides of fatty acids, citric acid, beta-carotene, sugar, pressed baker's yeast, complex food additive Orindsted pro 45, iodized table salt, baking mixture for increasing volume «XXL», flavoring E1422
3	Breakfast buns	Wheat flour, drinking water, white sugar, refined deodorized sunflower oil, fiber, pressed baker's yeast, K-SOFT baking mixture, complex food additive Grinstead pro 45, iodized table salt, E1422 flavoring
4	Buns «For hot dog»	Wheat flour, drinking water, white sugar, solid margarine, pressed baker's yeast, wheat fiber, dry milk product, iodized table rock salt, complex food additive bakery improver «Universal Super», complex food additive Grindsted Pro 45
5	Donuts «Berlin-skie» with «Boiled condensed milk»	Wheat flour, milk containing a product with a milk fat substitute «Premium», melange, special-purpose cooking fat «SolPro», cooking fat «Frying», antioxidants (E320, E321), hard margarine brand MT 82%, mixture for the production of bakery products «Hefikonk», white sugar, pressed baker's yeast, iodized table salt, complex food additive «Flora Super», baking mixture for increasing volume «XXL»
6	Bagels «Barskie» «Vanilla»	Wheat flour, drinking water, sugar, margarine, compressed baker's yeast, dry milk product, baking improver «Universal Super», iodized table rock salt, calcium propionate (preservative E282), food flavoring «Vanillin»
7	Hamburger bun	Wheat flour, drinking water, white sugar, solid margarine, pressed baker's yeast, peeled sesame seeds (99.90%), wheat fiber, dry milk product, iodized table rock salt, baking improver «Universal Super», complex food additive GRINDSTED PRO 45
8	Wheat tortilla «Mutlukal» with tomato	Wheat flour, vegetable oil, salt, sugar, tomato powder and spice mixture 1.8% (onion powder, garlic powder, lemon zest powder, basil powder, dill powder, red sweet pepper, cumin, coriander, thyme, turmeric), E422, raising agents E500, E450, emulsifier E471, preservatives E202, E282, acidity regulators E296, E330

The subject of the study was propionic acid and its salts. To determine propionic acid and its salts, a method was used that is based on the extraction of propionic acid and its salts from the analyzed sample, their quantitative determination using HPLC using a reverse-phase column and a spectrophotometric detector. The essence of the method we adapted was as follows. (10.000±0.001) g of the analyzed product was transferred into a volumetric flask with a capacity of 100 cm<sup>3</sup>. 100 cm<sup>3</sup> of extraction solution was added to the flask, kept in an ultrasonic bath for 10 minutes, then 5 cm<sup>3</sup> of Carrese I and Carrese II solutions were added to precipitate interfering substances, mixed thoroughly and 5 cm<sup>3</sup> of acetonitrile was added. The contents of the flask were brought to the mark with double-distilled water, mixed thoroughly and filtered through a folded paper filter. 1 cm<sup>3</sup> of the filtrate was transferred to a 10 cm<sup>3</sup> volumetric flask, the volume of the contents in the

flask was adjusted to the mark with the mobile phase for HPLC. The resulting solution, after filtering through a membrane filter, was used for chromatographic analysis.

Using the Chem Station software, the signals of propionic acid and its salts were identified in the resulting chromatograms based on the detection wavelength and the coincidence of retention times in the calibration solutions. Using the signal areas, the mass concentration of the identified propionic acid and its salts was calculated. To ensure that the methodology we adapted is accurate, linear, reproducible and correct, we validated it using a generally accepted method.

**Results and discussion.** The results of the validation of the methodology for measuring the content of propionic acid and its salts in bakery products are presented in Table 2.

Table 2  
Results of validation of the methodology for measuring the content of propionic acid and its salts in bakery products

Parameter and units of measurement	Numerical value
Repeatability limit r, %	14
Relative error limits ±δ, %, P=0,95	23
Reproducibility limit R, %	14
Measuring range, mg/kg	200–3000

As a result of the validation of the measurement technique, the following analytical characteristics were determined and confirmed by calculations: linearity, repeatability, intermediate precision, uncertainty, which met the standards.

The results of studies of the content of propionic acid in the study objects are presented in Table 3.

Table 3

Results of determining the content of propionic acid and its salts in the research objects

Item No.	Sample name	Propionic acid content, mg/kg
1	Breakfast buns	1814,09
2	Buns «For hot dog»	2119,76
3	Hamburger bun	1252,76
4	Bakery products «Rulets» with poppy seeds	1567,41
5	Wheat tortilla «Mutlukal» with tomato	248,78
6	Wheat tortilla «Mutlukal» with spinach	2482,09
7	Bagels «Barskie» «Vanilla»	618,64
8	Donuts «Berlinskie» with «Boiled condensed milk»	250,91
Bread with reduced energy value, butter pastries and flour confectionery products, pita, packaged (TR CU 029/2012) [6]		No more than 2 g/kg

As can be seen from the data presented, the content of propionic acid in the study objects varied widely: from 250 to 2500 mg/kg. Also, the research results indicate that two samples of bakery products (wheat tortilla “Mutlukal” with spinach and a bun «For a hot dog») do not comply with national legislation in terms of the indicator «Mass fraction of preservatives» (Table 2). It should be noted that samples of buns, bagels and donuts produced by Belarusian enterprises had a shelf life of no more than 20 days, samples of tortillas were foreign-made with a shelf life of 9 months. Thus, it is confirmed that the effectiveness of preservatives depends on the microbiological quality of the original recipe components and the degree of compliance with technological regimes and sanitary and hygienic production conditions.

**Conclusions.** Based on the results of the research, the following conclusions can be drawn. Propionic acid and its salts are most often used as a preservative in baked goods made from wheat flour. Validation studies of the method for measuring the content of propionic acid and its salts in bakery products confirmed its suitability in the range from 200 to 3000 mg/kg with a relative error of  $\pm 23\%$ . In 25% of the samples of small-piece bakeries, lamb products and donuts we examined, we found that the maximum permissible level of propionic acid and its salts was exceeded by 6.0% and 24.1%. The high-performance liquid chromatography method we adapted for determining the content of propionic acid and its salts can be used to control these preservatives in baked goods, as well as in low-moisture products (bagels) and donuts.

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