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QUANTITATIVE DETERMINATION OF RAFFINOSE IN COTTONSEED MEAL

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A chromatographic method is proposed for the quantitative determination of raffinose in cottonseed meal. The relative error of a single determination is ±4.0%.

We have previously [1] reported the isolation of raffinose from cottonseed meal. Raffinose pentahydrate has been proposed for use in animal husbandry for the breeding of cattle and in bacteriology for the preparation of media.

We have developed a procedure for the quantitative determination of raffinose in cottonseed meal. It consists in the extraction of the total saccharides from raw material with 80% ethanol, chromatographic separation in a thin layer of silica gel, and the photoelectric determination of the raffinose by means of its color reaction with anthrone [2]. In studying the stage of chromatographic separation of the total saccharides, we have carried out experiments with different solvents and solvent systems. The most suitable proved to be the methyl ethyl ketone—acetic acid—water (5:4:1) system.

To confirm the completeness of desorption of the raffinose from the silica gel, a standard solution of raffinose was chromatographed with the subsequent quantitative determination of the raffinose in the eluate:

Deposited, mg	Found, mg
0,067	97.5
0.075	98,0
0,080	97,8

Using the method developed, we determined the amount of raffinose in the meals from cottonseeds of the 1980-1982 harvests. The metrological characteristics of the results of the analysis of the means are given below:

f	$\overline{X}$	S	P, %	$t(p,f)$	$\Delta\overline{X}$	E,%
5	4.44	±0,1245	95	2,571	±0,154	±3.47
5	3,91	±0.1345	95	2,571	±0,154	±3.94
5	4.17	±0.1020	95	2.571	±0.116	±2,78

The absence of a systematic error of the method was shown by experiments with the addition of a definite amount of a standard solution of raffinose to an extract with a known concentration of raffinose:

Found in extract, g	Added, g	Found, g	Relative error, %
0,0391	0,0112	0,0485	—3,5
0,0391	0,0093	0,0498	+2,7
0,0391	0,0045	0,0422	—3,2

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As can be seen the relative error falls within the limits of accuracy of the method developed.

Determination Procedure. In a Soxhlet apparatus, 1 g of raw material was extracted with 200 ml of 80% ethanol, and the extract was concentrated and transferred quantitatively to a 25-ml measuring flask. The time of extraction was 4 h.

Plates with a fixed layer of silica gel (L 5/40 μ for thin-layer chromatography) divided into two parts were used. On one part was deposited 0.07-0.08 ml of the ethanolic raffinose extract, and on the other 0.07-0.08 ml of a standard solution of raffinose in concentration of 1 mg/ml. Chromatography was carried out with methyl ethyl ketone–acetic acid–water (5:4:1) system. That part of the plate upon which the solution of standard raffinose had been deposited was developed with a mixture of 2% solution of aniline in acetone containing diphenylamine phosphate [3], and after being sprayed the plate was heated to 110°C for 10 min. The time of exposure was 2.5-3 h.

The section of silica gel from the other part of the plate corresponding to the raffinose spot ($R_f \approx 0.35$) was transferred to a Schott No. 3 funnel and was eluted with 5 ml of boiling distilled water. To the eluate were added 4 ml of concentrated sulfuric acid and 6 ml of freshly prepared 0.2% solution of anthrone in 80% sulfuric acid.

The comparison solution was a mixture of 5 ml of distilled water, 4 ml of concentrated sulfuric acid, and 6 ml of the 0.2% solution of anthrone. The two test-tubes containing these mixtures were heated in hot water (80-90°C) for 15 min. The optical densities were measured in a photocolorimeter with a red filter in a cell with a layer thickness of 1 cm.

SUMMARY

A method has been developed for the quantitative determination of raffinose in cottonseed meal. The relative error of a single determination is ±4%.

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LIPOPOLYSACCHARIDES FROM *Mastigocladus laminosus*

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A lipopolysaccharide (LPS) has been isolated by phenol–water extraction from the cells of the blue-green alga *Mastigocladus laminosus*. It has been shown that the LPS contains polysaccharide and lipid components. The polysaccharide component includes a rhamnan fragment constructed of β-1,3- and, possibly, -1,2-bound L-rhamnose residues. The lipid component is constructed of glucosamine, glucose, and fatty acid residues, among which palmitic acid predominates.

Using Westphal's method [1] we have isolated a lipopolysaccharide fraction from air-dry cells of the blue-green alga *Mastigocladus laminosus* collected in thermal springs of Kamchatka. The crude preparation was purified from free phospholipid components by extraction with chloroform–methanol (2:1 by volume), and the accompanying nucleic acids and glucan were eliminated by ultracentrifugation. As a result, a purified lipopolysaccharide (LPS) with a low content of accompanying protein and nucleic acids (not more than 1% and 2%, respectively) was obtained.

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