AD

TECHNICAL REPORT
FD-12

FLAVORING MATERIALS FOR HIGH CALORIC FOOD BARS

by
E. J. HEWITT

JULY 1965

EVANS RESEARCH AND DEVELOPMENT CORPORATION
New York 17, New York

Contract No. DA19-129-AMC-2113(X) (017012)

U. S. ARMY NATICK LABORATORIES
Natick Massachusetts

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U. S. Army Materiel Command
U. S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01746

FOREWORD

In the development of operational feeding systems to meet specialized military requirements, increasing emphasis is directed toward minimizing weight, volume and preparation for consumption. In theory, these features can be realized by the compression of dehydrated foods into compact bars suitable for direct consumption. Experience has demonstrated, however, that bars of compressed dehydrated food frequently have marginal acceptability. To assure consumption by military personnel, bars must have suitable physical and organoleptic properties, including flavor, after periods of prolonged storage. It is well known that the natural flavor of many dehydrated foods is attenuated, lost, or adversely changed during storage, especially at elevated temperature. Enhancement of flavor of compressed food bars through addition of flavoring materials is expected to result in improved acceptability. This investigation is concerned with the behavior of representative types of flavoring materials in dehydrated food bars under various conditions of storage. Particular attention is directed toward measures for preventing deterioration of these flavors.

The investigation covered by this report was performed by the Evans Research and Development Corporation, 250 East 43rd Street, New York 17, New York, under contract number DA19-129-AMC-2113. The Official Investigation was Dr. E. J. Hewitt. His collaborators were Mr. T. A. Smith, Mr. R. W. Groncki, Mr. P. L. Roller, Miss M. E. Donworth, Mr. P. Mech, Mr. T. Malone, Dr. F. del Valle, Mr. J. Zolotar and Mr. J. Hilovsky.

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ABSTRACT

The object of the project was to establish methods to stabilize flavors for flavor retention in high caloric food bars under varying storage conditions. In a high moisture food bar (18-20% moisture) all methods tested were ineffective. In food bars, 5-8% moisture, good stabilization was achieved by the combination of incorporating BHA in the base bar and encapsulating the added flavor in polyoxyethylene oxide 6,000. These results are based on sensory panel tests of bland food bars containing 15 different type flavors stabilized by various methods and stored under a wide range of conditions in different package types. Adjuncts such as gum arabic and non-fat dry milk solids are effective in retaining flavor as measured by gas-liquid chromatography. Techniques were developed for both solid sampling and vapor sampling for extraction of flavor from food bars for gas-liquid chromatographic analysis.

FLAVORING MATERIALS FOR HIGH CALORIC FOOD BARS

INTRODUCTION

On September 17, 1962 Evans Research and Development Corporation was authorized by the Quartermaster Food and Container Institute of the Armed Forces* to conduct studies on methods to improve the stability of flavors in high caloric food bars.

In the Statement of Work, the primary objective of the project was stated as follows: "To establish the adequacy of several methods for assuring the retention of added flavors in high caloric food bars after a nominal storage period".

Summarized briefly below are the specifications of the project:

1. Flavoring materials and auxiliary components were to conform to requirements of the Food and Drug Administration.

2. Flavoring materials were to be stabilized in any manner deemed advantageous.

3. The flavoring materials were to be natural or artificial. The number to be investigated was to be 12 to 18 types with the following restrictions:

*Currently, Food Division, U. S. Army Natick Laboratories.

a) No more than three common flavors, of which one might be citrus,

b) No more than two spice flavors,

c) One or two systems for flavor development by action of flavor release enzyme*,

d) **Flavors to be studied should include chili, sun dried fish, and soya hydrolysate.

4. Flavorings were to be incorporated into a bland or near-flavorless compressed food bar conforming to the general composition, as given below:

	Percent (dry basis)
Fat	15-25
Protein	15-25
Carbohydrate	50-60
Ash (incl. NaCl)	2- 4

5. Storage tests were to be run for six months and were to include the following variables:

Temperature - 100°F, 70°F, 40°F

2 cycles per week, max. 40°F, min. 0°F

Initial Moisture - (a) Maximun moisture 2% (b) 5-8%

(c) 16-20%***

Package - (a) Metalized polyethylene polyester pouch

(b) Metal can, 0_2 headspace gas 135-145 mm Hg.

(c) Metal can, 0_2 headspace gas below 1 mm Hq.

6. A description of the effect, if any, of the presence of acceptable concentrations of the following adjuncts on representative types of flavors: Sugar (sucrose), sodium chloride at maximum concentrations, food acid at acceptable levels of tartness, protein hydrolysate, flavor enhancers (such as inosinic acid derivatives).

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*U.S.Patent 2,924,521, assigned to Evans Research and Development Corporation.

**This was added in Phase II, the second 12-month period of the 24-month program; therefore, the number of flavoring materials to be studied should not be less than 15.

***In Phase II, the tests with moisture 16-20% was to be eliminated.

7. The phenomenon of flavor masking was to be described in quantitative terms, and recommendations were to be given on how it can be avoided or substantially reduced. Observations were to include relationship of fat, protein, sugar and polymeric carbohydrates to flavor masking.

8. Bars were to be evaluated for changes in intensity and quality of flavor after the prescribed storage time by any objective method to correlate with an experienced flavor panel.

This Final Report includes a consolidation of all experimental findings and results, notes, data, and conclusions.

SUMMARY

A method was developed to stabilize flavors for flavor retention in high caloric food bars. The method consists of two steps: (1) the addition of an approved antioxidant (BHA) to the base formula of the food bar, and (2) the encapsulation of the added flavor in poly oxyethylene oxide. This combination was found effective in a wide range of storage conditions in food bars containing 5-8 percent moisture. The system, however, was not effective for a high-moisture-level bar (18-20 percent), and no success was achieved by any other method for stabilization of a high-moisture-level food bar.

No problems can be foreseen in large-scale use and exploitation of the method given in this report for stabilization of flavorings in food bars of average moisture (2-8 percent).

In the informational part of the project, certain flavor adjuncts were studied for their effect in retaining flavor. It was found that gum arabic and non-fat dried milk solids were much more effective for flavor retention than sodium chloride. These results were obtained by a sensory panel and gas-liquid chromatography.

Techniques were developed for extraction of flavor from food bars and analysis by gas-liquid chromatography. The extraction and sampling can be accomplished by either a solid-sampling or vapor-sampling method. Further refinements have to be developed and tested before the gas-chromatographic method (consisting of sample preparation, extraction and instrumental analysis) can be considered reliable for quality control or for systematic studies of changes of flavors in storage.

EXPERIMENTAL DISCUSSION

I. BLAND COMPRESSION FOOD BAR

A. Screening of Materials

The first step in the flavor stabilization program was directed toward the development of a relatively bland compressed food bar. Specifications called for a general composition of:

Fat	15-25%
Protein	15-25%
Carbohydrate metabolizable	50-60%
Ash (incl. NaCl)	2- 4%

Numerous possible constituents were screened individually for their flavor by informal panel procedures, and only those materials with relatively bland flavors were given further consideration.

As the initial trial formulation, a mixture of oatmeal, soy flour (LSP-15) and hydrogenated fat were arbitrarily selected. The ingredients were combined in the quantities calculated to meet the analytical specifications. When compressed into a food tablet, this combination had an undesirable soy flavor.

Attempts were made to mask the soy flavor by adding confectionery sugar and non-fat dry milk solids. These ingredients were also selected for their bland flavor and binding properties which would improve the over-all bar quality. Improvement in physical quality was sufficient to warrant their inclusion in subsequent formulations, although the predominant soy flavor was still apparent. Since the results indicated that an alternate protein source was needed to replace the soy flour, a variety of protein sources was investigated.

The materials screened to replace soy flour were principally in the casein and toasted soy fraction categories. Of all the materials screened, four were selected as being the most promising: calcium caseinate, high nitrogen casein, toasted soy protein and Promine-D.

Replacement of oatmeal, rice and barley cereal as carbohydrate sources was tested by preparing and evaluating other formulations containing oat flour, potato granules and white wheat flour. The results of the tests indicated that all of these carbohydrate sources are interchangeable with respect to

blandness. However, all three of these items also produced tablets with a slightly pasty texture.

B. Experimental Formulas

Based upon screening work described above, Formulas 15 and 17 were devised (see Table I).

As the panel found the pasty consistency of Formula 15 objectionable, Formula 17 was developed. Formula 17 is a somewhat different product, utilizing corn flakes and dry milk solids because Formula 15 could not be modified directly to yield the desired properties. Other ingredients were also tested, such as Edi-Pro or spun soya made by the Ralston Purina Company, but none offered enough advantages to warrant incorporation in the formulation. In Formula 17, the following ingredients were combined and produced a satisfactory bland food bar: non-fat dried milk solids, hydrogenated vegetable shortening, lactose, Promine-D and ground corn flakes.

Formula 17 was found acceptable in a sensory panel evaluation using hedonic scale ratings. It was rated satisfactory for blandness, mouth feel and texture. It was studied, as discussed later, under accelerated shelf-life tests.

Due to the instability of Formula 17 in storage, it was necessary to find a substitute for the hydrogenated vegetable shortening and, in its place, a solid cotton seed stearin was used. See Formula 18 in Table I. Formula 18 was used extensively in shelf-life studies which will be discussed later.

C. Preparation of Compressed Food Bars

The dry ingredients of Formula 17 (non-fat dried milk solids, Promine-D, hydrogenated vegetable shortening, lactose, and ground corn flakes) were weighed out and thoroughly mixed in a Hobart mixer.

The mixture was then granulated using the wet granulation process. This process consisted of the following: (1) addition of water to form a plastic mass, (2) extrusion of the plastic mass into thin sheets, (3) drying of the sheets to the desired moisture level (5-8% or 2-3%), and (4) grinding of the sheets to the desired mesh size. The granulation was made into a tablet or food bar by a Stokes DS-3 machine. The pressure and dwell time in the press varied according to the nature of the flavor additive. The average pressure used was 5000 lbs. with a dwell time of two tenths of a second. Tablets were formed 3/16 inch in thickness and 5/8 inch in diameter, at a rate of

1 to 1-1/2 pounds of tablets per minute. A vibrator feeder was used to insure proper filling of the die and a uniform tablet.

Bars from Formula 18 were prepared in the same manner except that 7,000 psi were used for compression.

Formulas 17 and 18 at this point appeared to be suitable as a bland base for the flavor stability studies. These formulas produced satisfactory bars in which the flavors could be incorporated and at the same time complied with contract specifications on carbohydrate, protein, fat and ash. Analyses are found in Table I.

II. METHODS FOR FLAVOR STABILIZATION

The primary concern of this project was the stabilization of flavor in compressed food bars. Several aspects of commercial stabilization were examined in an exploratory way to give some insight into the methods which would be most effective. The techniques used in these preliminary studies and the results are given in the following sections.

A. Encapsulation of Flavors

1. Spray Drying with Gum Arabic

In order to evaluate spray-dried flavors in compressed food bars, several samples of spray-dried citrus oils (20% oil and 80% gum) and vanilla flavor from several of the larger flavor houses were tested.

Approximately 5% of the spray-dried citrus oil composition was added to base mix Formula No. 17. The same was done with pure spray-dried vanilla flavor. The mixes were then compressed in a Stokes DS-3 machine.

The resultant tablets were put into aluminum foil polyethylene polyester pouches and heated for one week at 120°F.

At the low moisture level (2 to 3%) and intermediate moisture level (5 to 8%), the spray-dried flavors were judged informally to have held up well. However, when enough moisture was incorporated into the food bar to bring the moisture up to the 20% level, the flavors (and the bars) had deteriorated considerably and lost much strength after several days.

Although the flavors at the low and intermediate moisture range appeared to be stable in the food bars, it was found

that a 5% level of spray-dried flavor could hardly be noticed when the food bar was tasted. The combination of the base mix and the spray-dried oils appears to mask the flavors. Specifically, orange flavor had to be increased from 5% to 15% on a weight basis until an informal panel was able to discern the actual flavor. At this higher level of flavor, the panel members could identify it immediately as orange but, after the bar completely dissolved in the mouth, the flavor level became overwhelming and distinctly unpleasant. It appears that:

1. Flavors at low levels could be tasted only as an aftertaste and were not perceptible during chewing.

2. When a flavor was increased to a level where it could be immediately recognized, the flavor became objectionable in character.

3. Panel members complained, particularly with citrus products, that the aroma was pleasant, but an overwhelming peel oil character appeared at the high level during chewing.

A probable explanation for the masking of flavors by the spray-drying of the materials is the presence of the encapsulating polymer in large quantity in the dried flavor. This masking effect is accentuated by the introduction of the spray-dried flavor into a product which itself has masking properties.

In addition, when high levels of spray-dried flavors were incorporated into the base mix, the general composition as specified in the contract for carbohydrate, protein, fat, ash would be unbalanced.

In view of these findings, a means for encapsulation other than spray-drying was sought where a smaller amount of polymer would be required.

2. Dispersion in Carbowax 6000

One product for encapsulation of flavors which appeared promising was polyethylene glycol (molecular weight 6000-7500), when used in the proportion of 40% polymer to 60% oil.

The polyethylene glycol selected was Carbowax 6000 which is a solid at room temperature. It melts at about 155°F, is highly soluble in water and has no taste or aroma to speak of. In addition, it is nontoxic and is permitted for use in compressed food products. Its main advantages are that it does not noticeably mask flavors and rapidly releases them when dissolved in

water. In some cases (orange oils) it has a tendency to round out or reduce the "chemical" sharpness of flavors. The flavor was dispersed in melted polyethylene glycol, cooled, and then ground.

In order to test the glycol-encapsulated flavors against a spray-dried vanilla (80% gum arabic, 20% flavor), a pure bourbon vanilla was dispersed in the polyethylene glycol at the 20% by weight level and ground. The two encapsulated flavors were placed into food bars and the bars tasted (5% flavor was added to the bars).

It was the opinion of an informal panel that the polyethylene glycol-encapsulated sample released more flavor than the spray-dried product and, therefore, bars containing flavors encapsulated in polyethylene glycol should be placed under six month stability tests.

B. Granulation

One of the methods which can be utilized to stabilize flavors in a compressed food bar is the physical isolation of flavors from the greater portion of mix. This can be accomplished by taking approximately 10% of the base mix, adding the flavor to this fraction and making a separate flavor granulation or flavor premix. The flavored premix is then combined with the remainder of the unflavored base mix and mechanically blended. After the blending operation, compressed bars can be made where within each bar there is a homogeneous distribution of flavor particles.

In order to determine whether this method of stabilization should be investigated fully, an initial test was made with lemon flavored food bars.

Lemon flavor was incorporated into bars of 5-8% moisture content by two different methods. In the first set of bars, the flavored premix described above, was added to the base mix. In the second set, the flavor was added to the bars by spraying the flavor itself directly onto the total granulation. Both sets of flavored base mixes were then blended, compressed into bars and stored for one week at 120°F, after which they were examined by an informal sensory panel.

A noticeable loss in flavor was reported in the bars containing the sprayed-on flavor, while the bars containing the lemon, produced by the granulation method, were judged to be considerably better than the unstabilized control bar in both flavor and aroma. It was then decided to conduct a preliminary six-month storage test on this lemon flavored bar.

C. Chemical Stabilization

Chemical stabilization has proven to be the most effective means of insuring the quality of flavor during accelerated shelf-life tests and six-month tests at 100°F. These tests were conducted on the flavors themselves. In the earlier experiments, the chemical antioxidants, butylated hydroxyanisole (BHA) and 2, 4,5,-trihydroxybutyrophenone (THBP) were screened for use as stabilizing agents with citrus oils (lemon, orange and lime) and spice oils (parsley and cinnamon).

The untreated oil and oils treated with either BHA (0.1% by weight) or THBP (0.05% by weight) were heated to 50°C and air sparged. While being air sparged the samples were frequently examined for odor change by an informal panel. When changes were detected the test was stopped. Changes in the odor of the untreated citrus oils were found after 15 hours.

The BHA and THBP treated samples remained stable for 60 hours, indicating an extension of the shelf-life of the citrus oils of 400%. The BHA and THBP treated spice oils were stable more than twice as long as the untreated control samples. However, no conclusions were drawn about the comparative superiority in stabilization effectiveness of the two antioxidants. It was decided to incorporate BHA in the base mix as an over-all chemical stabilizer which would be effective for both the flavor and ingredients in the bland food bar.

D. Dispersion of Flavors in Fat (Cottonseed Stearines)

One method for addition of flavor to bars is the incorporation of flavors in the shortening in the base mix. The advantages of this technique are that the flavor is isolated from the high moisture portion of the product and a uniform distribution of the flavor is attained.

In the initial accelerated tests to determine the validity of this method, a lemon flavor was incorporated into the melted shortening. The mixture was then cooled, ground, and added to base mix Formula No. 17. Food bars based on this mixture were stored for one week at 120°F.

Results indicated that a food bar based on base mix Formula No. 17 and flavored by dispersing the flavor in the fat portion was preferred over a bar with the flavor directly mixed or sprayed on the granulation. Therefore, it appeared promising to carry out six month stability tests on bars in which the flavor is dispersed in the fat.

The three methods of stabilization selected for the flavor-stability storage tests were encapsulation with Carbowax 6000, granulation, and dispersion in cottonseed stearine.

III. SELECTION OF FLAVORS FOR STORAGE TESTS

The selection of flavors was based on preferences given in the booklet on Food Preferences of Men in the United States Armed Forces*. The flavors were derived from natural and synthetic sources. In general the natural flavors were used at higher levels than the synthetics in order to obtain a satisfactory flavored food bar. Therefore, in some cases it was found necessary to modify the composition of the unflavored bar in order to incorporate the necessary amounts of natural flavor. During the course of the work a number of other observations were made which influenced the selection of the flavors. In some samples, it was noted that the compressed unflavored bar had the tendency to enmesh or entrap much of the added flavor. In other instances, when the flavor was increased to a satisfactory level, unpleasant off tastes were noted by the panel. It was therefore necessary to discard many of the flavors which were developed earlier in the program.

Finally, as a result of sensory tests, fifteen flavors were indicated as satisfactory for the extensive stabilization and storage studies required by the program. The flavors were: cinnamon-apple, curry, chili, chocolate, coffee, vanilla, spaghetti-spice, rice spice, beef-tomato, tomato spice, chicken spice, bacon and tomato, banana, lemon, and cherry. Their composition and flavor-to-base ratio are given in Table II and their hedonic scale ratings when incorporated in the food bar are given in Table III. In order to have replacements for the flavors which might not stand up under the stability tests, the reformulation of flavors was continued throughout the project. Some of these reformulations are given in Table IV, and their hedonic scale ratings when used in the food bar are given in Table V. Sources of materials used in formulations are given in Table VI.

IV. STORAGE STABILITY TESTS

The contract specifications required that storage tests

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*Peryam, D.R., Polemis, B.W., Kamen, J.M , Eindhoven, J. and Pilgrim, F.J., QM. Research and Development Command, Quartermaster Food and Container Institute for the Armed Forces, January, 1960.

be run for 6 months to include the following variables:

Temperature - 100°F, 70°F, 40°F

Initial Moisture - (a) Maximum moisture 2%

(b) 5-8%

(c) 16-20%

Package - (a) Metalized polyethylene polyester pouch

(b) Metal can, O_2 headspace gas 135-145 mm Hg

(c) Metal can, O_2 headspace gas below 1 mm Hg.

In the storage work the above variables were followed with the following exceptions:

1. It was not possible to prepare food bars with moisture content 2% or lower. The 2% food bar did not readily undergo the compression in a Stokes DS-3 tabletting machine. The 2% moisture base mix under a pressure of 5,000 psi gave tablets which capped, crumbled readily when handled and were unusually dry to the taste.

2. In preliminary tests it was found also that food bars formulated at 20% moisture deteriorated so quickly (major discoloration, off-flavor development and increase in hardness) that the recommendation was made to drop this variable which was accepted by the Quartermaster Corps.

3. In packaging the specifications require O_2 headspace of less than 1 mm Hg. This could not be obtained by flushing with air; therefore, N_2 was used.

A. Preparation of Samples for Storage Tests

Using Formulas 17 and 18, the flavored food bars for storage tests were prepared according to the method outlined on page 5. See Table II for formulations of tablets including flavor. The flavors were stabilized by three methods, namely granulation, dispersion in fat, and encapsulation in Carbowax 6000. The dried flavor was blended in with the granulated ingredients for the bland food bar and compressed according to the method given. As discussed earlier all the samples were compounded at 5-8% moisture level.

The sample tablets were placed in storage in the following containers according to specifications:

1. Aluminum-foil polyethylene polyester pouch.
2. Sealed metal can, air-packed, each can containing two perforated glassine pouches to determine flavor transfer on storage (one pouch contained flavored tablets and the second contained unflavored tablets).
3. Same as No. 2 except nitrogen-flushed to remove oxygen.

Table VII gives the numerical breakdown of the number of samples and tablets placed under storage.

B. Results of Six-Month Storage Tests

1. Bars Prepared from Formula No. 17

As explained in the previous sections, tablets formulated with bland food bar Formula 17 and containing flavor were placed in storage. Although the control bar made from base mix Formula No. 17 appeared stable in the earlier tests at 120°F for 3 months, the same bars were not sufficiently stable to withstand the storage at 100°F for six months. The deterioration of the bars was due to the instability of the hydrogenated vegetable shortening. On this basis a modification in the bland food bar base was made.

The composition of the bland food bar was left unchanged with the exception of the fat. In place of hydrogenated vegetable shortening, solid cottonseed stearine was substituted in the formula and this fat was stabilized with BHA. The new base mix, the composition of which is given in Table I, was designated as Formula 18.

2. Bars Prepared from Formula 18

The entire line of flavored bars, repeating those which had already been under shelf life studies in bland food bar Formula 17, were remade using base mix No. 18 and placed under study as discussed below.

C. Sensory Panel Evaluation of Flavored Food Bars

Six trained members of the Evans Research Sensory Panel evaluated the bars organoleptically, giving an hedonic scale rating (hedonic scale = 0 to 9) to the food bars. It should be restated that the score given to the basic, bland bar was 4.1. The results are presented in tabular form in Tables VIII-XXI.

The following discussion of results of the six-month evaluation of food bars is limited to the 100°F tests; each of the fourteen types of flavored food bars is discussed individually.

1. Banana Bar

After six months of storage, the banana food bars which were stabilized by the granulation method were rated acceptable under all three methods of storage, i.e., nitrogen-packed in tin cans, air-packed in tin cans, and pouch-packed in air. For this natural material, all packaging systems appear to be acceptable. Little or no flavor transfer was organoleptically noted. Hedonic scale ratings are presented in Table VIII.

2. Tomato Spice Bar

Of all the 100°F samples of tomato spice food bars tested, no particular method of preservation of flavor or packaging proved to be better than the other. Organoleptically, no flavor transfer was found. The results are recorded in Table IX.

3. Chili Bar

For the chili-flavored food bars, the nitrogen-packed samples were slightly preferred over the regularly can-packed bars. No noticeable flavor transfer was found in the canned samples. Among the three flavor preservation systems used, no difference was found in the canned items except one failure which was found in the pouch-packed bars stabilized by the cottonseed stearine method. A slight preference, however, exists for the nitrogen-packed bars. The results are presented in Table X.

4. Coffee Bar

For the coffee-flavored bars, only one unacceptable sample was found, that of the pouch-packed sample, stabilized by the granulation method, which developed a strong, bitter taste. A slight preference for the air-packed samples in cans was noted, and a slight flavor transfer was noted in the canned-packed bars. The results of the panel evaluations are presented in Table XI.

5. Chicken Spice Bar

The chicken spice bars were rated acceptable under all conditions. The hedonic scale ratings showed the nitrogen-packed bars to be preferred slightly over the air-packed canned bars and the pouch-packed bars. No significant organoleptic difference between the flavor-preservation systems could be found. Little or no flavor transfer was found in the canned samples. The results are recorded in Table XII.

6. Cherry Bar

All methods of flavor preservation were rated unacceptable with the pouch-packed cherry samples. In contrast, all three methods with the can-packed bars were found acceptable, with a preference for the nitrogen-packed bars; flavor transfer was noted as very slight in the can-packed samples. The hedonic ratings are presented in Table XIII.

7. Bacon and Tomato Spice Bar

The bars which were air-packed in cans were preferred over the nitrogen- and pouch-packed samples. The cottonseed-stearine-stabilized samples in pouch packs definitely failed in achieving acceptable ratings. No flavor transfer was found in the can-packed bars. The results are recorded in Table XIV.

8. Imitation Vanilla Bar

All samples were rated above the 4.1 score of unflavored food bar. The lowest of the scores was found in the pouch-packed samples. Nitrogen-packed bars were slightly preferred over the air-packed canned samples. Only a slight flavor transfer was found in the unflavored samples packed in pouches with the flavored can-packed bars. Results are recorded in Table XV.

9. Spaghetti Spice Bar

No Spaghetti Spice bars were rated below the 4.1 basic food bar score. The nitrogen-packed bars rated highest in acceptability; the air-packed bars placed second; and the pouch-packed bars third. No flavor transfer was noted in the can-packed samples. In Table XVI are presented the hedonic scale ratings.

10. Chocolate Bar

The nitrogen-packed bars were preferred over the air-packed and pouch-packed bars. No samples scored below the 4.1 basic blend bar score although the pouch-packed samples received the lowest ratings. A slight flavor transfer was noted in the can-packed samples. The ratings are presented in Table XVII.

11. Beef-Tomato Spice Bar

The nitrogen-packed bars were preferred. No difference was noted between the flavor stabilization systems, and generally only a slight flavor transfer was noted with the can-packed samples. The hedonic scale ratings are given in Table XVIII.

12. Lemon Bar

No significant preference was given to the bars of any of the three methods of stabilization or of any of the storage methods. All samples were acceptable with a slight flavor transfer noted in the can-packed bars. The ratings are presented in Table XIX.

13. Curry Bar

The nitrogen-packed bars were preferred. No sample was rated below 4.1, but the lowest scores were given to pouch-packed bars. No flavor transfer was noted in the food bars packed in cans. The results are recorded in Table XX.

14. Rice Spice Bar

For the Rice Spice bars, the nitrogen-packed samples were preferred over the air-packed. No food bar was rated below 4.1, but the lowest scores were given to the pouch-packed bars. Little or no flavor transfer was found between flavored and unflavored samples packed together in cans. The results are presented in Table XXI.

D. Discussion of Results of Six-Month Shelf Life Tests

1. Packaging Conditions

The six-month storage tests indicated that the most protective storage system was that of food bars nitrogen-packed in tin cans. Food bars air-packed in cans were rated second while those packed in pouches were rated third.

Twenty-five samples were rated below 5 on the hedonic scale; of these twenty-five, twenty-two samples had been pouch-packed while only three had been packed in cans. Two of the twenty-five had been packed in nitrogen, and the remaining twenty-three under regular atmospheric conditions in air.

Based upon the above test results, the preferred method of packaging would be nitrogen-packed in tin cans. It must be pointed out, however, that the majority of pouch-packed samples are acceptable, but that their organoleptic ratings are not as high.

2. Stabilization Techniques

All the food bars tested were stabilized by the use of

BHA at 0.1 percent levels of flavor and fat content. Upon examination of the hedonic panel ratings, no significant difference was found in the food bars which were packed in cans using granulation, Carbowax, and cottonseed stearine techniques. This is probably due to the fact that the tin can offers maximum protection to its contents, particularly when nitrogen is used in place of air. Of the twenty-five samples which were rated under 5, only six were Carbowax while nine were granulation and ten were cottonseed stearine. Six of the same twenty-five samples fell below the basic bland food bar rating of 4.1. One was a Carbowax bar, two were granulation bars, and three were cottonseed stearine bars.

Based upon the six-month storage tests, the best method of stabilization of food bars is Carbowax encapsulation in pouches. All three stabilization methods, however, were found to be acceptable with food bars which had been packed in cans.

The most acceptable method of packaging was found to be the nitrogen-pack in tin cans.

V. IMPROVEMENT OF BLAND FOOD BAR

As explained in the previous section, Formula 18 containing corn flakes was found to be acceptable, but with the following limitations after 6-month storage tests:

1. Corn flakes tend to mask and contribute a characteristic flavor of its own.

2. Corn flakes pick up moisture in storage and tend to accelerate the deterioration of the food bar.

3. The texture quality contributed by the corn flakes after storage is not sufficient to substantiate its use in the food bar.

It was decided, therefore, to develop new formulations which are described below.

New Formulations

The basic bland food bar (Formula 18) which was found not to be completely satisfactory was composed of non-fat dry milk solids (35.27%), cottonseed stearine (8.82%), lactose (27.34%), Promine D (10.93%), and ground corn flakes (17.64%).

A new ingredient sold under the trade name "Lolac"* was found, which proved to be a good carbohydrate-protein source and replacement for corn flakes in Formula 18. Several formulations were developed for an improved bland food bar, as described below:

Formula 19 - Corn flakes was removed, the quantity of Promine D was lowered, and Lolac (high lactose dry milk solids) was added in amount to compensate for these changes.

Formula 20 - This was a totally new formula consisting of Lolac, cottonseed stearin, and lactose.

Formula 21 - This was also a new formula containing Lolac with the quantity of Promine D lowered, and cottonseed stearin unchanged.

The new formulations were granulated using water and tabletted using a Carver press at a pressure of 6000 psi with a one-inch die. The food bars made in this manner from the various formulations were hedonically rated as acceptable. The ratings were actually higher than those for Formula 18. Samples of the three formulations were stored at 130-135°F for 10 days at which time their acceptability was again rated. Although it was generally considered as borderline, Formula 20 was judged to be the most bland of the three.

The advantage, therefore, of using bars made from Formula 20 would be its blandness which will permit a more precise evaluation of flavors, particularly for enzyme flavor systems. As most enzyme flavors are not particularly pronounced, a more delicate medium for experimental testing is needed. The section on enzyme flavor systems is in a later part of this report.

The compositions and analyses of Formulas 19, 20, 21 for the Bland Food Bar are given in Table XXII.

No storage tests were run with Formula 20 as food bars containing flavor as with Formula 18 because of insufficient time to repeat the storage series.

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*Lolac is sold by Foremost Dairies, Inc., and is a special carbohydrate-protein dry product derived from milk.

VI. FLAVOR ENZYME STUDIES

The contract specified that one or two systems for flavor development by action of enzymes on a suitable precursor should be studied under U.S. Patent 2,924,521, assigned to Evans Research and Development Corporation. The object of using flavor enzymes in connection with the broad problem of flavor stabilization was to incorporate an enzyme and substrate in a food bar which will develop a flavor when placed in the mouth. Thus the flavor will be in an inactive form until eaten.

A. Preliminary Investigations

These investigations were designed to explore the possibilities and determine the problems in the applications of flavor enzymes as a flavor precursor in food bars. The two essential components of such an enzyme system are the enzyme itself and the substrate.

The preliminary investigations were made with blueberry, horseradish, and watercress.

1. Extraction of Enzymes--The general procedure was as follows:

   The berries, leaves or other materials were pulverized in a Waring blender. The resulting material was extracted with a buffer solution, then centrifuged. To the extract, cold acetone or methanol was added to precipitate the enzyme.

   The precipitate was dissolved in water and dried by a freeze-drying operation.

   The details of experimentation for the different materials for extraction are given in Table XXIII.

2. Preparation of Substrate--The general procedure was as follows. See also Table XXIII.

   The berries, leaves, or other materials were boiled in water to inactivate the enzymes. The mixture was cooled and filtered and preserved by either freezing, oven-drying, or freeze-drying.

3. Incorporation into Food Bar--

   The enzymes and substrates were incorporated into the formula for Bland Food Bar Formula #20 as given in Table XXII, and compressed.

4. Preliminary Evaluation of Enzyme Systems in Food Bars

Blueberry--Enzyme preparations from fresh blueberry and from dry leaves were tested on fresh blueberry substrate. The amount of flavor enhancement was not sufficient for incorporation in the Bland Food Bar, therefore no further work was done with blueberry. The emphasis was shifted to pineapple which was tested extensively as discussed in a later section of this report.

Horseradish--In the Bland Food Bar, a good horseradish flavor was obtained, but the appeal of a "horseradish bar" was not too high. Therefore, the enzyme and substrate was incorporated into a "shrimp-cocktail bar", containing dehydrated shrimp and spices. This product developed an off-flavor very quickly on aging due to deterioration of the shrimp. This enzyme system might be more effective in a meat food bar.

Watercress--Good odor was obtained when 0.010 g of enzyme, 1.5 g substrate and 2 grams of food bar were combined. However, this flavor effect was obtained only at very high concentrations. It is possible that the watercress system would be more effective when combined with a lemon-fish flavor, or with a salad flavor.

B. Pineapple Enzyme Studies

The main effort on the study of flavor enzymes for flavor stabilization of food bars was done with pineapple. Pineapple was selected because it is generally available and cheap, it has high flavor acceptance, and it has strong flavor character which is necessary for the food bar.

The object in the pineapple enzyme research program was to isolate and utilize an enzyme or enzymes from the pineapple plant and fruit which would serve to create a pineapple flavor from odorless, tasteless substrates.

1. Procedure for Extraction of Pineapple Enzymes--

The basic approach was to extract various portions of the plant and fruit under different pH conditions and with specific solvents to isolate the protein fraction. The particular enzyme systems readily isolated under the selected extraction procedure were precipitated out of solution by using suitable media such as acetone, ethanol, and ammonium sulfate, usually at reduced temperatures. The precipitated enzyme fraction was

carefully slurried in water (after additional purification) and freeze-dried into a fine powder. Alternately, the extract could be freeze-dried without pre-treatment. If treated properly, it will not lose its specific functional qualities through heat treatment or subsequent denaturization. The details for the preparations of enzymes and substrates for pineapple are given in Tables XXIV and XXV.

2. Preparation of Food Bars

Pineapple flavored food bars were prepared utilizing Formula No. 20 base mix which was prepared specifically for its blandness, with Substrate E and Enzyme 9 in a 2.5-inch die. See Tables XXIV and XXV. The die, containing 25 grams of base mix No. 20 and 4 percent flavor level, was placed in a Carver Press and 10,000 psi pressure was applied for 20-30 seconds. These compression conditions produced a food bar of the desired qualities which measured 2-1/2 inches in diameter and 1/4 inch in thickness.

3. Test for Specificity of Enzymes

To test the specificity of the enzymes, the substrate and enzyme (0.5 grams of Substrate E and 0.05 grams of Enzyme 9) were placed in water. A high level of enzyme and substrate was used against respective blanks to definitely assure that any enzyme activity present would be observed.

C. Discussion of Pineapple Enzyme Studies

A prototype pineapple food bar has been prepared using pineapple Enzyme 9 and Substrate E at the 4 percent flavor level. Smaller pencentages of the flavor level were employed, but the flavor, though present, was too weak to be acceptable. The final enzyme-to-substrate level ratio tested was 1:5.

Future work should be directed towards development of enzyme-substrate systems which would give a more concentrated flavor which is needed for the food bar application. This is not surprising since most natural flavors are weaker than imitations.

VII. SIX-MONTH STORAGE - NEW FLAVORS

The contract required storage tests in two phases for a total of not less than 15 flavors. Twelve of these flavors were tested in the first phase and four new flavors were tested in the second phase. In this, the latter phase, sun-dried fish and soya hydrolysate were specifically requested. In addition, strawberry and orange were included in this phase because of anticipated poor stability of several of the flavors out of the twelve tested in the first phase. This would guarantee a minimum of 15 flavors studied in both phases.

The four new flavors were prepared in Formula 18 as described on page 11. See Table XXVI.

In this storage test the best method for flavor stabilization was used which was encapsulation in Carbowax 6000, and anti-oxident (BHA) in the base mix. The contract allowed for exclusion of those methods which were not satisfactory in the first storage series.

Samples of each flavor of food bar was packed for storage under three sets of conditions: (1) air-packed in tin cans, (2) nitrogen-packed in tin cans, (3) pouch packed in air. In order to determine flavor transfer, flavored bars were can-packed with unflavored bars. Storage was at 100°F, 70°F and 40°F and cycled twice per week.

A considerable number of panels were run in order to determine the hedonic preference and flavor changes for the food bars after storage.

A. Sensory Panel Evaluation of Flavored Food Bars - New Flavors

Six trained members of the Evans Research Sensory Panel evaluated the bars organoleptically, giving hedonic scale rating (hedonic scale = 0 to 9) to the food bars. It should be restated that the score given to the basic bland bar was 4.1. The results are presented in tabular form in Tables XXVII and XXVIII. Each of the different flavored food bars are discussed individually.

1. Sardine Bar (Carbowax Encapsulated)

After six months of storage, the sardine food bars were stabilized by the Carbowax method and were rated acceptable under all three conditions of storage, i.e., nitrogen-packed in tin cans, air-packed in tin cans, and pouch-packed in air. For this natural material, all packaging systems appear to be acceptable. Little or no flavor transfer was organoleptically noted. See Table XXIX for preparation of sardine extract.

2. Strawberry (Carbowax Encapsulated)

All samples after six months of storage were rated acceptable with ratings about that of the 4.1 score of the unflavored food bar. All packing systems appear to be acceptable, with little or no flavor transfer organoleptically noted.

3. Soya (Carbowax Encapsulated)

The soya flavored food bars, after six months of storage, were rated acceptable under all conditions of storage and packaging. No flavor transfer was noted in the food bars packed in cans or pouches.

4. Orange (Carbowax Encapsulated)

The orange flavored food bars were organoleptically rated as acceptable under all systems of storage and packaging. No flavor transfer was noted by the panel members.

B. Discussion of Results of Six-Month Shelf Life Tests-New Flavors

1. Packaging Conditions

In the six-month storage tests all methods of packaging yielded organoleptically acceptable food bars. Food bars packed in cans were rated first while those packed in pouches were rated second. Little or no difference was organoleptically noted between those food bars packed in air or nitrogen.

2. Stabilization

The food bars tested, stabilized by the use of Tenox IV at 0.1 percent levels of flavor and fat content and the Carbowax method, were found to be stable and satisfactory.

VIII. EFFECT OF FLAVOR ADJUNCTS ON FLAVOR

A. Selection of Flavor Adjuncts and Flavors

The contract required a description of the changes, if any, caused by the presence of flavor adjuncts which were specifically given as sugar, sodium chloride, food acid such as citric acid, protein hydrolysate, and flavor enchancers such as inosinic

acid derivatives. These adjuncts were to be tested at various concentrations consistent with consumer acceptability.

In addition, the contract required that the food bars containing these adjuncts be evaluated for changes in intensity and quality of flavor after six-month storage. The evaluations were to be done by a trained sensory panel and correlated by an objective method.

The flavor adjuncts were to be tested in representative chemical types of flavor. Curry - representative of a natural spice, Lemon - representative as an essential oil, Cherry - representative as an artificial type. These were to be incorporated into a bland compressed food bar and Formula 18 was used for this purpose.

The materials selected as adjuncts for testing were nonfat dry milk solids, carboxymethylcellulose, gum arabic, dextrin (50 percent soluble - 50 percent insoluble), salt, citric acid, monosodium glutamate product (95 percent with 5 percent disodium guanylate), disodium guanylate, and lactose.

B. Preparation of Samples

Solution of the above adjuncts and flavors were made and processed in a Bowen Laboratory Spray-Drier. The formulas used, the ratio of flavor to flavor adjuncts, and the spray-drying designation number (the "E" number) are presented in Table XXX (Curry), Table XXXI (Lemon), and Table XXXII (Cherry). Condition and results of spray-drying are presented in Tables XXXIII, XXXIV, and XXXV, Curry, Lemon, and Cherry, respectively. Spray-drying was utilized as a process test as it involved solution and heat processing of flavor and ingredient components. In addition, it is an important working tool of the flavor and food field.

The above flavors and flavor adjuncts as an homogeneous spray-dried powder were incorporated into a bland bar (Formula 18) and subjected to six months of storage (under packaging materials and temperature conditions specified previously). Controls were prepared consisting of spray-dried flavor without the adjuncts using gum arabic and carboxymethylcellulose.

C. Evaluations - Accelerated Tests

As a preliminary step, accelerated storage tests at 120°F were utilized to gain a rapid insight into the changes brought about

by aging. Organoleptically, the accelerated tests produced the following results after 4 weeks:

1. Curry - The gum arabic control sample remained acceptable, with little or no change noticeable by the panel. The general result of the addition of flavor adjuncts was that a raisin-wheat aroma, particularly with the protein fractions was developed. In the sample containing citric acid, the curry flavor was modified with the predominant flavor note being citrus in character and the coriander flavor note from the curry being more dominant than in the control sample.

2. Lemon - As was found in the curry samples, the gum arabic control gave very good results as did carboxymethylcellulose control with no noticeable changes in the flavor. The nonfat dry milk solids masked (entrapped)some of the flavor and aroma of lemon samples. In the sample containing dextrin, there was a noticeable loss or entrapment of flavor. The sample containing lactose yielded a lemon flavor that is sweet and similar to the product known as "Realemon".

3. Cherry - Again, the gum arabic control functioned the best, while the proteins and other ingredients all induced changes in flavor and aroma intensity and character. The changes in the cherry flavor ranged from a modified flowery type cherry note to a protein-cherry flavor.

Prior to instrumental studies to determine the intensity and quality of the changes in flavor, the above samples were evaluated by an experienced flavor panel. The organoleptical ratings are to be found in Tables XXXVI, XXXVII, and XXXVIII. Where the flavor panel found differences, the samples were then subjected to instrumental studies. All of the samples evaluated were found to be acceptable, with hedonic rating above that of the bland unflavored food bar.

IX. INSTRUMENTAL STUDIES

The contract specified that food bars containing flavors and flavor adjuncts were to be evaluated by an objective method to correlate with sensory panel tests. Gas-liquid chromatography was used in these objective studies.

The purpose in testing the combinations of flavors and flavor adjuncts was to determine what influence or effect flavor adjuncts had on flavor after storage.

A. Preliminary Investigations

Gas chromatography, employing solid sampling, dry vapor sampling, and wet vapor sampling techniques were used to investigate the effects of three adjuncts (gum arabic, non-fat dry milk solids and sodium chloride) on spray-dried cherry and/or lemon flavors. Although this was only a preliminary investigation, the information obtained demonstrated the unique value of gas chromatography in such studies.

Based on these preliminary studies, it was found that it was not possible to operate the instrument routinely under experimental conditions which reproducibly detected the flavoring materials in 10 milligram samples of food bar. Possibly the amount of flavoring material initially applied was lost during sample preparation, or the component materials of the food bar actually retain most of the flavoring materials. In either case, even more sensitive detector conditions were required, and more elaborate extraction techniques must be used to recover the flavor possibly entrapped by a single or by several bar components.

The effort to develop the solid sampling technique for routine use was due to the inherent advantages of this technique which makes it especially promising for the analysis of volatile flavor components in food bars. A very small amount of material, about 5 grams for each food bar sample, was available for analysis. Therefore, replicate analyses must be obtained by using aliquots of this 5 grams of material. The nature of the chemical flavors used, the use of adjuncts, the use of a matrix composed of complex interferring substances and relatively low concentration of any one component of the complex chemical flavor, all indicate that the sample will require preliminary heating to release the volatile flavor components from the bulk of the

sample. The solid sampling technique uses a 10 milligram aliquot of the original sample sealed in a glass capsule. This glass capsule can be heated in the injection port of the gas chromatograph at a selected temperature for a selected time. Then, the glass capsule is crushed, releasing the volatile components of the sample for analysis by gas chromatography.

B. Investigation of Spray-Dried Lemon Flavor Plus Adjunct

1. Effects of Adjuncts on Lemon Flavor

The following spray-dried lemon flavor plus adjunct samples were examined for lemon flavor by gas chromatography:

Number	Flavor	Adjunct
E 6813	Lemon (40%)	Gum Arabic
E 6815	Lemon (44%)	Non-Fat Dry Milk Solids
E 6818	Lemon (42%)	Sodium Chloride

The percentages refer to the hypothetical lemon flavor content. They are not a measure of the actual lemon flavor content of the final, spray-dried flavor plus adjunct. At a later stage of this report it will be shown that these experiments have suggested a gas chromatographic approach whereby the actual lemon flavor content could be determined.

In this experiment gas chromatography was used to determine the approximate amount of lemon flavor that could be detected by examining aliquots of each of the above samples. This will not determine the total lemon flavor content of the samples. Due to sorption, each of these adjuncts retains a greater or lesser amount of lemon flavor on its surface. To the extent that the adjunct makes the lemon flavor unavailable for detection, it has masked the lemon flavor. The total lemon flavor content of each spray-dried lemon flavor plus adjunct sample was equal to the lemon flavor content as determined in this experiment, minus the lemon flavor content made unavailable for detection due to retention by the adjunct. Indirectly, the ability of adjuncts to reduce lemon flavor was measured.

Indication that adjuncts were reducing the amount of lemon flavor actually available for detection would be

presumptive evidence that materials in the base mix Formula No. 18 might be further reducing the lemon flavor actually available for detection. Still more losses or changes due to storage might further reduce the amount of lemon flavor available for detection to a concentration too small for detection by this very sensitive method. Analysis of these spray-dried samples of lemon flavor plus adjunct simplified the problem since the lemon flavor content is higher at this stage than in the final food bar, and there are no complications due to any interactions with components of base mix Formula No.18. Demonstration of this point would explain the failure of the solid sampling and dry vapor sampling techniques to detect lemon flavor in food bars, although calculations based on available data indicated that theoretically a 10 milligram sample did contain sufficient lemon flavor for detection.

For the reasons cited above, three samples of lemon flavor plus adjunct were analyzed by the solid sampling technique, although sufficient sample was available to permit use of a vapor sampling technique. A description of the procedure followed in the solid sampling technique will be included in a later section of this report.

2. Results and Discussion - Lemon Flavor Plus Adjunct

Lemon flavor was detected in approximately 11 milligram aliquots of all three samples. The three major components of lemon flavor were present at such concentrations that it was necessary to operate the instrument at less sensitive conditions in order to detect these components as on-scale peaks. Reproducibility was greater than the usual $\pm$ 5 - 10% expected from liquid or vapor sampling techniques. Differences were demonstrated in the relative amounts of lemon flavor detected for each of the three spray-dried lemon flavor plus adjunct samples.

The results obtained indicated that the past failures to detect flavor in food bars was due to the availability of much less than the theoretically expected amount of lemon flavor for detection. In part the discrepancy was caused by some adjuncts sorbing much less flavor than others. The exact losses of flavor due to retention of part of the lemon flavor by other adjuncts under these conditions of gas chromatography still remains unknown. Losses of flavor due to reaction through physical

and chemical means can only be inferred at this time. In a later section of this report such losses will be demonstrated. It should be noted that even with spray-dried powders there still was not completely satisfactory homogenity of the sample, leading to poor reproducibility. As mentioned before, the differences between the samples were so large, the poor reproducibility did not destroy the reliability of the results in this case.

When interpreting the chromatograms (Figures 1, 2, 3) it was evident that three peaks contained the bulk of the lemon flavor detected. Use of one or all three of these peaks furnished a means of comparing the amount of lemon flavor in the three samples. These peaks are designated by the letters X, Y and Z. The heights of peaks X, Y and Z were measured in inches, on a given chromatogram. The peak height in inches was multiplied by the Range Product setting of the instrument for that peak. The resulting number is the peak height in inches, if it were possible to operate the instrument at its most sensitive setting namely, a range setting of one and an attenuation setting of one. This fortunate condition was never realized in practice. At best, a range product setting of 16 was used, and sometimes 32 or 64 had to suffice. This idealization device is one arbitrary way to standardize and simplify the presentation of data from many chromatograms obtained at various levels of instrumental sensitivity, in order to obtain useful information and generalizations.

The following data was obtained by this method:

Sample	Flavor	Adjunct	Idealized Peak Height in Inches at Range Product = 1		
			X	Y	Z
E 6813	Lemon (40%)	Gum Arabic	576	3,840	580
E 6815	Lemon (44%)	NFDMS	3,040	21,120	3,520
E 6818	Lemon (42%)	Sodium Chloride	80	658	106

In each of these samples, the peaks X, Y and Z had essentially the same retention times as corresponding peaks detected in liquid lemon flavor. As further substantiation of the same identity of each of the peaks in the three samples, the peaks X, Y and Z exist in all three samples in the same relative proportions. Peak Y was much larger (about 7 times) than Z and X. Peak Z was only slightly larger than X and for practical purposes these two peaks can be considered as essentially equal.

Dividing the amounts of X, Y and Z detected in sample E 6818 into the corresponding values for these peaks in samples E 6813 and E 6815, presents the data in a more meaningful way.

Sample	Flavor	Adjunct	Idealized Peak Height Relative to Sample E 6818		
			X	Y	Z
E 6813	Lemon (40%)	Gum Arabic	7.2	5.9	5.5
E 6815	Lemon (44%)	NFDMS	38.0	32.2	33.3
E 6818	Lemon (42%)	Sodium Chloride	1.0	1.0	1.0

When non-fat dried milk solids (NFDMS) was the adjunct, it released approximately 32 times as much lemon flavor during analysis as did sodium chloride as the adjunct. The gum arabic released approximately 6 times as much lemon flavor as the sodium chloride adjunct. By an extension of the above procedure, the NFDMS released approximately 6 times as much lemon flavor as the gum arabic adjunct.

This experiment clearly demonstrated that under these conditions the amount of lemon flavor detected by gas chromatography in spray-dried lemon flavor plus adjunct decreased with the adjuncts as follows: non-fat dried milk solids, gum arabic, and sodium chloride. Figures 1, 2, 3 are copies of representative chromatograms obtained in this experiment.

C. Investigation of Spray-Dried Cherry Flavor Plus Adjunct

The following spray-dried cherry flavor plus adjunct samples were examined for cherry flavor by gas chromatography:

Sample No.	Flavor	Adjunct
E 6854	Cherry	Gum Arabic
E 6855	Cherry	Non-Fat Dry Milk Solids
E 6858	Cherry	Sodium Chloride

In this experiment the effect of adjuncts on cherry flavor was determined by two sampling techniques which were intended to be analogous to smelling and tasting the samples. In the dry vapor sampling technique, a one-gram aliquot of a given sample was heated for 10 minutes at 90°C in a 10 cc Erlenmeyer flask sealed by an odorless rubber serum cap and 5 cc of headspace vapors were removed and analyzed by gas chromatography. In the wet vapor sampling

technique, 5 cc of boiling distilled water was added to another gram aliquot of the same sample which was heated for 10 minutes at 90°C in a 10-cc Erlenmeyer flask sealed by an odorless rubber serum cap and 5 cc of heapspace vapors were removed and analyzed by gas chromatography.

The dry vapor sample would detect the cherry flavor released from the spray-dried cherry flavor plus adjunct by the heating step. The wet vapor sample would detect the cherry flavor released from the spray-dried cherry flavor plus adjunct by solution in the water (an extraction) as well as by the heating step. This procedure was felt to simulate in a very crude way the role of saliva in the mouth releasing flavor from the adjunct. At some future time it was planned to examine the water itself for cherry flavor by gas chromatography.

Results and Discussion - Cherry Flavor Plus Adjunct

The benzaldehyde peak was detected in all samples at concentrations necessitating the use of less than the usual sensitive conditions. Significant differences were detected between all three samples. Important differences were detected between the same sample by dry and wet vapor sampling

Sample No.	Flavor	Adjunct	Idealized Peak Height in Inches at Range Product = 1	
			Dry	Wet
E 6854	Cherry	Gum Arabic	1,719	5,549
E 6855	Cherry	NFDMS	6,617	1,719
E 6858	Cherry	Sodium Chloride	277	1,455

Using the dry vapor sampling technique most cherry flavor was detected for the NFDMS adjunct, gum arabic was intermediate and sodium chloride was lowest. Using the wet vapor sampling technique the previous sequence was changed, i.e., most cherry flavor was detected for the gum arabic adjunct, NFDMS was intermediate but not very significantly greater than sodium chloride. Although more cherry flavor was detected by the wet vapor sampling technique for gum arabic and sodium chloride adjuncts, the reverse was true for the NFDMS adjunct.

The above data was converted to make it relative to cherry flavor detected in the sodium chloride adjunct sample.

Sample No.	Flavor	Adjunct	Idealized Peak Height at Range Product = 1 Relative to No. 6858	
			Dry	Wet
E6854	Cherry	Gum Arabic	6.1	3.8
E6855	Cherry	NFDMS	23.9	1.2
E6858	Cherry	Sodium Chloride	1.0	1.0

By the dry vapor sampling technique, NFDMS adjunct released approximately twenty-four times as much cherry flavor as did sodium chloride adjunct and approximately four times as much cherry flavor as did gum arabic adjunct. Gum arabic adjunct released approximately six times as much cherry flavor as did sodium chloride adjunct as determined by the dry vapor sampling technique. By the wet vapor sampling technique gum arabic adjunct released approximately four times as much cherry flavor as NFDMS adjunct and sodium chloride adjunct, the latter two being approximately equal in effect.

In the case of cherry flavor the dry vapor sampling technique demonstrated that NFDMS adjunct was most efficient in releasing cherry flavor, gum arabic adjunct was significantly less efficient and sodium chloride was least efficient.

This data agrees well with the conclusions reached earlier for lemon flavor with these adjuncts. In effect both experiments measured the ability of mild heating to desorb flavors from adjuncts. An additional unknown amount of cherry flavor still remained sorbed to each of the adjuncts in differing amounts.

The wet vapor sampling technique was used to determine the extent to which the water would replace the cherry flavor from the active sites of the adjuncts. It was assumed that the amount of flavor detected in all cases should be greater than for corresponding samples analyzed by the dry technique. The results obtained indicated this was true for gum arabic adjunct, which now released the most cherry flavor. It was also true for the sodium chloride adjunct which by the wet technique released almost as much cherry flavor as the NFDMS under the same wet technique.

Comparing data for the same adjunct when examined by the dry and wet techniques, the wet technique accomplished an increase of approximately 5 times for the sodium chloride adjunct, approximately 3 times for the gum

arabic adjunct, and a decrease of almost four times for the NFDMS adjunct. Solution of the sodium chloride in water with release of the cherry flavor probably resulted in the large increase for this adjunct. Gum arabic is used for spray-drying by the industry because it does readily release flavor in solution. In the case of the NFDMS, it is possible that the resulting solution was more effective in absorbing the cherry flavor than was the dry NFDMS.

The data suggest a complementary relationship exists between the data obtained by the dry and wet techniques. The dry NFDMS released most flavor and the wet gum arabic released most flavor. The numerical values obtained are very similar and probably not too significantly different. The cherry flavor released by wet NFDMS and dry gum arabic had identical numerical values by coincidence. Both wet and dry, the sodium chloride values were always lowest. It is possible that the NFDMS and gum arabic adjuncts actually sorbed essentially the same amount of cherry flavor during the spray-drying treatment. The NFDMS released most of the sorbed flavor on dry heating. The gum arabic released most of the sorbed flavor on solution. With this information as a starting point, it should be possible to utilize both techniques to develop a method to permit measurement of the total amount of flavor initially present on the adjunct after spray-drying or any other charging treatment.

D. Analysis of Food Bars

Evaluations made by the sensory panel detected flavor differences between food bars incorporating Lactose-Lemon, Mertage 5-Lemon and Guanylate-Lemon which had been stored at 40°F as control samples, and identical food bars which had been stored at 100°F. Therefore, these lemon flavored samples were selected for analysis by GLC to determine if a correlation between sensory panel and instrumental results could be demonstrated. Cherry-flavored food bars would have been preferred for this comparison due to the comparative ease and speed of analyzing for the major flavor component, benzaldehyde, by gas chromatography as opposed to the difficulty and two hours required to resolve the many components of the complex lemon flavor. Since none of the cherry-flavored food bars demonstrated any detectable flavor differences between those stored at 40°F and the others stored at 100°F, as determined by the sensory panel, it was necessary to use the lemon flavored food bars.

1. Procedure

For each of the six lemon flavored food bar samples, all of the food bars remaining from the panel tests were ground to the finest powder obtainable on the Wiley Mill. It was reported earlier that such a finely milled powder is essential to obtaining a more homogeneous sample, which in turn is critically important when using the solid sample technique in order to obtain more reproducible and hence reliable data. Approximately five grams of powder were obtained for each of the six food bar samples to be examined, viz; Lactose-Lemon 40°F, Lactose-Lemon 100°F, Mertase 5-Lemon 40°F, Mertase 5-Lemon 100°F, Guanylate-Lemon 40°F, and Guanylate-Lemon 100°F. A five gram total sample was insufficient material to permit use of the vapor sampling (dry or wet) technique with enough replicate samples. Therefore, the only recourse was to use the more sophisticated and far less convenient solid sampling technique, since this technique required aliquots of only 10-20 milligrams per analysis.

A tared capillary tube sealed at one end is filled to the appropriate level with the ground powder. The tube is weighed and sealed. The capillary is placed in the solid sampler device, which is positioned in the injection port of the gas chromatograph. After heating for the selected time at the selected temperature, the plunger of the solid sampler is depressed, crushing the glass tube and releasing the volatiles into the injection port of the gas chromatograph for analysis.

2. Results and Discussion

Peaks were detected for each of the six samples. Much more material was usually detected in a few of the samples, such as Lactose-Lemon 40° and Mertose 5-Lemon 40°, than in the other samples. Good reproducibility was not achieved. Replicate adiquots of the same sample gave peaks ranging in size from one inch to several inches. The bulk of the material detected appeared to be higher boiling compounds not detected in the original lemon flavor itself. These peaks in many cases were far larger than the peaks corresponding to the original liquid lemon flavor.

Reproducibility probably was not achieved for aliquots of the same powdered sample due mostly to a lack

of sufficient homogeneity in the sample. As was noted previously, in earlier work better reproducibility was achieved when the food bars were powdered to the finest extent possible on the Wiley Mill as opposed to simple pulverizing by means of a pestle and mortar. Apparently treatment in the Wiley Mill and subsequent stirring of the fine powder to produce an evenly colored product still did not mix the sample sufficiently well so that aliquots taken at random were exactly equivalent in composition. A further step must be added to the sample preparation procedure in order to achieve the requisite homogeneity. Perhaps mixing on the roller mill for a sufficient time might achieve satisfactory homogeneity without any appreciable loss or change of the volatile flavor components. It is not likely that instrumental sensitivity fluctuated to such an extent during a day or even several days.

The compounds having much greater retention times than those for the major constituents of the lemon flavor could be due to a number of factors. These compounds may be due to components of base mix Formula No. 18 itself or of the adjunct. The peaks detected may represent such components either unchanged or changed by storage at 40°F and 100°F, respectively. If these peaks are due to lemon flavor, they would be the result of changes in the original flavor due to storage at 40°F and 100°F, since they are not the major components detected in the initial liquid lemon flavor.

The difficulty with reproducibility and lack of sufficient time precluded further work on this phase of the project. Assuming the reproducibility problem can be solved, the technique shows promise in determining the effects on the initial added flavor due to adjunct, base mix and storage conditions.

Developing a solid sampler capable of accommodating 100 milligram samples would permit operation of the instrument at less sensitive conditions thus reducing interference from extraneous sources and providing better instrumental stability.

E. Comparison of the Dry Vapor Sampling Technique and the Solid Sampling Technique

The procedure described for the dry vapor sampling of spray dried cherry flavor plus adjunct was applied to one

set of spray dried lemon flavor plus adjuncts. These samples were examined by the solid sampling technique as reported on Page 25. The data from both techniques are here compared to determine the more sensitive technique.

Idealized Peak Height
in Inches at Range Product = 1

Adjunct	Solid Sampling (10 milligrams)			Dry Vapor Sampling (1 gram)		
	X	Y	Z	X	Y	Z
Gum Arabic	576	3,840	580	11,904	39,680	13,696
NFDMS	3,040	21,120	3,520	75,528	272,640	65,920
Sodium Chloride	80	658	106	1,080	5,376	992

Generally speaking, especially for the Y peaks of the lemon flavor, the dry vapor sampling technique yielded values that were approximately ten times those of the solid sampling technique. The solid sample was 1/100 the size of the sample for the dry vapor technique. This cancels the ten-fold advantage in peak size detected by the dry vapor sampling technique. Further, it indicates that the solid sampling technique is approximately ten times as sensitive as the dry vapor sampling technique.

TABLE I

COMPOSITION AND ANALYSIS OF UNFLAVORED FOOD BARS

Formula No. 15

Composition:	Rice Cereal	46.10%
	Hydrogenated Vegetable Shortening	7.70%
	Nonfat Dry Milk Solids	15.40%
	Confectionery Sugar	15.40%
	Promine D (clarified)	15.40%
Analysis*:	Carbohydrate	57.00%
	Protein	25.00%
	Fat	15.00%
	Ash	3.00%

*On a dry basis

Formula No. 17

Composition:	Nonfat dry milk solids	35.27%
	Hydrogenated vegetable shortening	8.82%
	Lactose	27.34%
	Promine D (clarified)	10.93%
	Ground corn flakes	17.64%
Analysis*:	Carbohydrate	59.74%
	Protein	26.22%
	Fat	10.06%
	Ash	3.98%

*On a dry basis

Formula No. 18

Composition:	Nonfat dry milk solids	35.27%
	Cottonseed stearine*	8.82%
	Lactose	27.34%
	Promine D (clarified)	10.93%
	Ground Corn flakes	17.64%
Analysis**:	Carbohydrate	59.74%
	Protein	26.22%
	Fat	10.06%
	Ash	3.98%

*Stabilized with Tenox IV
**On a dry basis

TABLE II

FORMULATIONS OF TABLETS - FOR STORAGE TESTS

Cinnamon-Apple

The following changes were made in base mix 17:	Wt/Grms
Nonfat dry milk solids (20)*	35.27
Hydrogenated vegetable shortening (18)	8.82
Apple flour (23)	27.34
Promine-D (clarified)	10.93
Ground corn flakes (9)	7.64
Sugar, confectionery (1)	10.00
Cinnamon (11)	8.00

Chocolate

Base mix	200
Cocoa - 22% fat (10)	15
Vanilla Nodes - spray dried (11)	15
Sugar, confectionery (1)	10

Spaghetti Spice

Base mix	200
Spaghetti spice	5
composed of:	
Tomato powder (10)	40.0
Onion (8a)	11.3
Paprika (6)	4.0
Garlic (8a)	3.1
Basil (8a)	1.0
Monosodium glutamate (17)	0.8
Pepper (8a)	0.6
Celery (8a)	0.5
Oregano (11)	0.5
Rosemary (8a)	0.1

*Figures in parentheses correspond to suppliers listed in Table VI

TABLE II (Continued)

	Wt/Grms
Rice Spice	
Base mix	200
Rice spice	5
composed of:	
Onion (8a)	21.00
Salt	4.35
QM Curry* (8a)	2.45
Celery (8a)	2.00
Chicken fat (14)	2.00
Thyme (8a)	1.35
Parsley (8a)	0.03
Bay (8a)	0.01
Beef-Tomato	
Base mix	100
Freeze dried beef (17)	30
Tomato powder (10)	10
Monosodium glutamate (17)	2
Protein hydrolysate (20)	2
Garlic powder (8a)	1
Onion salt (8a)	1
Mustard (5)	0.5
Curry	
Base mix	200
Curry	3
composed of:	
Coriander (8b)	250
Cumin (11)	100
Fenugreek (15)	100
Black pepper (8a)	60
Cardamon (8b)	50
Mace (11)	40
Allspice (11)	35
Cinnamon (8b)	35
Mustard (5)	20
Paprika (6)	15
Ginger (8b)	10
Celery (8a)	5
Orange oil (7,19,21)	5
Cayenne (8a)	0.5

*Blend made according to Federal Specifications EE-P-600.

TABLE II (Continued)

	Wt/Grms
Tomato Spice	
Base mix	200
Tomato spice	5
composed of:	
Dehydrated tomato (10)	43.8
Onion (8a)	16.1
Garlic (8a)	4.9
Paprika (6)	4.5
Basil (8a)	1 3
Monosodium glutamate (17)	0.9
Oregano (11)	0.5
Pepper (8a)	0.5
Celery (8a)	0.4
Chicken Spice 2	
Base mix	150
Freeze-dried chicken (22)	30
Carrot powder (4)	3
Celery (11)	2
Chicken extract powder (3)	1
Disodium inosinate (12)	1
Monosodium glutamate (17)	1
Protein hydrolysate (20)	1
Spice mix for chicken* (8a)	1
Onion salt (8a)	0.5
Thyme (11)	0.5
Banana	
Base mix	90
Banana crystals (16)	10
Chili	
Base mix	200
Chili spice	8
composed of:	
Chili pepper (6)	11.20
Dehydrated tomato (10)	6.44
Paprika (6)	4.48
Cumin (11)	1.68
Coriander (8a)	1.40
Red pepper (6)	1.40
Oregano (11)	0.84
Celery (8a)	0.56

*Blend made according to Federal Specifications EE-P-600.

TABLE II (Continued)

	Wt/Grms
Lemon	
Base mix	200
Lemon flavor	0.75
composed of:	
Exchange lemon oil (24)	100
Lemon oil 5x (19)	50
Veltol - 2% in benzyl alcohol (17)	10
Terpeneless lemon oil (8c)	5
Coffee	
Base mix	200
Sugar (1)	10
Instant coffee	6
Vanilla	
Base mix	200
Spray dried vanilla nodes (13)	10
Candy Cherry	
Base mix	200
Cherry flavor (10% benzyl alcohol)	2
Cherry Flavor Formula	
A. Ethyl Oenanthate	1.2
Candy base mix	8.5
Tolyl aldehyde	12.5
Benzaldehyde N.F.	55.8
B. Candy base mix	
Eugenol	2.0
Anisyl acetate	9.0
Anisyl aldehyde	9.0
Amyl cinnamic aldehyde	15.0
Absolute jasmin	1.0
Vanillin	24.0
Ethyl tolylglycidate	25.0
Bacon and Tomato	
Base mix	100
Dehydrated tomato powder	1.0
Hickory smoked yeast	0.5
Bacon fat (stabilized)	1.0

TABLE III

HEDONIC SCALE RATINGS OF FLAVORED

COMPRESSED FOOD BARS

Flavor	Individual Ratings										Average
	A	B	C	D	E	F	G	H	I	J	
Cinnamon Apple	8	9	8	8	9	8	7	9	-	-	8.3
Chocolate	6	8	6	8	7	8	7	7	-	-	7.1
Spaghetti Spice	8	7	8	8	8	2	7	7	8	-	7.0
Rice Spice	7	7	8	8	7	6	6	6	-	-	6.9
Beef-Tomato	7	6	7	8	6	6	6	7	-	-	6.6
Curry	7	7	8	4	7	4	7	8	7	-	6.6
Tomato Spice	5	5	5	6	6	7	8	7	-	-	6.1
Chicken Spice 2	6	6	5	7	6	7	5	6	-	-	6.0
Banana	6	6	7	7	3	6	6	6	6	-	5.9
Chili	5	7	1	5	9	7	7	6	-	-	5.9
Lemon	5	6	5	7	7	4	7	6	-	-	5.9
Coffee	6	5	8	6	7	7	4	2	7	-	5.8
Vanilla	5	7	6	3	8	7	4	3	5	-	5.3
Chicken Spice 1	6	5	4	2	1	5	2	7	-	-	4.0
Bland Food Bar 17	5	3	6	3	4	3	7	2	3	5	4.1

TABLE IV

COMPOSITION OF FLAVORS AND FLAVOR-TO-BASE RATIOS FOR REFORMULATED FLAVORS

Chocolate	Original Formulation	Modified Formulation
Base mix	200*	200*
Cocoa (22% fat)	15	5
Spray-dried vanilla	15	0
Sugar, confectionery	10	0
Saccharin sodium	0	0.02
Imitation vanilla	0	2
Coffee		
Base mix	200	200
Sugar	10	0
Instant coffee	6	3
Imitation vanilla	0	0.1
Saccharin sodium	0	0.02
Chicken Spice		
Base mix	150	100
Freeze-dried chicken	30	0
Carrot powder	3	0
Celery dehydrated	2	0
Chicken extract powder	1	1
Disodium inosinate	1	0
Monosodium glutamate	1	0
Protein hydrolysate	1	0.2
Spice mix for chicken	1	0.5
Onion salt	0.5	0
Thyme	0.5	0
Onion dehydrated	0	0.3
Chicken fat	0	2
Disodium inosinate and disodium guanylate	0	0.1

*Parts by weight.

TABLE V

HEDONIC SCALE RATING OF

COMPRESSED FOOD BARS CONTAINING

REFORMULATED FLAVORS

Flavor	Individual Ratings										Average
	A	B	C	D	E	F	G	H	I	J	
Coffee	4	6	7	8	6	3	6	4	-	-	5.5
Chocolate	5	6	6	5	6	7	6	6	-	-	5.9
Vanilla	6	6	6	7	5	4	6	5	-	-	5.6
Bacon & Tomato	6	5	7	8	6	7	8	5	-	-	6.5
Beef Tomato	5	7	6	5	7	7	5	6	8	6	6.4
Chicken Spice	7	6	6	5	6	5	7	7	7	5	6.1
Cherry	6	5	6	6	7	5	7	6	6	7	6.1
Bland Food Bar	5	3	6	3	4	3	7	2	3	5	4.1

TABLE VI

SOURCES OF MATERIALS USED IN FORMULATION OF FLAVORED FOOD BARS

1. American Sugar Refining Company, New York
2. Armour & Company, Chicago
3. Beatrice Foods, Inc., Chicago
4. California Vegetable Concentrates, Modesto, Calif.
5. Durkee Famous Foods, Cleveland, Ohio
6. Gentry, Glendale, Calif.
7. Haarmann & Reimer Corp., Union, N.J.
8. Chas. L. Huisking & Co., Inc., Lyndhurst, N.J.
    a) Saromex S
    b) Saromex D
    c) Essential Oils
9. Kellogg Company, Battle Creek, Mich.
10. Milton Klein Company, Inc., Jamaica, N.Y.
11. McCormick & Co., Inc., Baltimore, Md.
12. Merck & Co., Inc., Rahway, N.J.
13. Norda, New York
14. Ocoma Foods Co., New York
15. S. B. Penick & Co., New York
16. Plant Industries, Inc., Plant City, Fla.
17. Chas. Pfizer & Co., New York
18. Procter & Gamble Co., Cincinnati, Ohio
19. Pierre Robertet, Inc., New York
20. Sheffield Chemical, Norwich, N.Y.
21. Taconic Natural Oils Co., Inc., New York
22. United Fruit Co., New York
23. Vacu-Dry, Oakland, Calif.
24. Warner-Jenkinson Mfg. Co., St. Louis, Mo.

TABLE VII

BREAKDOWN OF TOTAL NUMBER OF TABLETS PRODUCED WITH FORMULAS 17 AND 18

Method of Stabilization	Storage Conditions			
	35°C	70°F	100°F	Cycling*
Metalized Polyethylene Polyester Pouches:	180 Pouches Containing 5,400 Tablets			
Granulation	420	420	420	420
Dispersion in Fat	420	420	420	420
Encapsulation	420	420	420	420
Control**	90	90	90	90
Tablet Subtotal	1350	1350	1350	1350
Cans - Air Packed:	168 Cans Containing 10,080 Tablets***			
Granulation	420	420	420	420
Dispersion in Fat	420	420	420	420
Encapsulation	420	420	420	420
Control**	1260	1260	1260	1260
Tablet Subtotal	2520	2520	2520	2520
Cans - Nitrogen Packed:	168 Cans Containing 10,080 Tablets***			
Granulation	420	420	420	420
Dispersion in Fat	420	420	420	420
Encapsulation	420	420	420	420
Control**	1260	1260	1260	1260
Tablet Subtotal	2520	2520	2520	2520
GRAND TOTAL	25,560 Pouches Containing Flavored Tablets			

*Cycling between 0° and 35°F.
**Unsealed metalized polyethylene polyester pouches.
***Each can contained two glassine pouches each containing 30 tablets.

Table VIII

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

BANANA

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	7	7	8	7	6	6	41	6.8	None
High - 100°F Air-packed in a tin can*	Granulation	8	8	8	7	7	6	44	7.3	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	6	6	7	8	8	7	42	7.0	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	5	6	7	8	8	7	41	6.8	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	5	6	7	8	8	7	41	6.8	-
Recycling Pouch-packed (air) flavored bars	Granulation	6	6	6	8	7	7	39	6.5	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

TOMATO SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	6	6	5	6	5	5	33	5.5	None
	Carbowax	6	6	5	6	6	6	35	5.8	None
	Cottonseed Stearine	4	7	6	5	5	8	35	5.8	None
High - 100°F Air-packed in a tin can*	Granulation	7	6	6	6	5	6	36	6.0	None
	Carbowax	6	6	5	6	6	6	35	5.8	None
	Cottonseed Stearine	5	7	5	5	5	6	33	5.5	None
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	5	6	8	2	8	34	5.7	-
	Carbowax	6	5	6	5	4	7	33	5.5	-
	Cottonseed Stearine	6	5	7	7	3	5	33	5.5	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	7	6	5	7	7	4	36	6.0	-
	Carbowax	7	7	5	5	6	4	34	5.7	-
	Cottonseed Stearine	7	7	6	7	8	4	39	6.5	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	5	6	5	5	7	35	5.8	-
	Carbowax	7	5	5	5	7	5	34	5.7	-
	Cottonseed Stearine	7	6	7	7	6	6	39	6.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	5	7	7	5	6	35	5.8	-
	Carbowax	5	6	6	7	5	5	34	5.7	-
	Cottonseed Stearine	6	5	6	7	4	8	36	6.0	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table X

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

CHILI

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	6	5	6	5	8	4	34	5.7	None
	Carbowax	6	5	7	6	8	5	37	6.2	None
	Cottonseed Stearine	7	5	6	6	6	6	36	6.0	None to slight
High - 100°F Air-packed in a tin can*	Granulation	7	5	6	5	7	5	35	5.8	None to slight
	Carbowax	7	5	6	5	7	5	35	5.8	None
	Cottonseed Stearine	6	5	6	5	6	5	33	5.5	None
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	6	7	4	5	3	30	5.0	-
	Carbowax	6	6	6	4	2	4	28	4.7	-
	Cottonseed Stearine	4	5	5	2	3	2	21	3.5	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	5	7	4	4	8	34	5.7	-
	Carbowax	6	5	6	4	4	6	31	5.2	-
	Cottonseed Stearine	5	5	7	4	4	7	32	5.3	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	5	5	6	5	7	4	32	5.3	-
	Carbowax	5	5	6	5	5	3	29	4.8	-
	Cottonseed Stearine	5	4	6	5	8	5	33	5.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	5	7	4	4	9	34	5.7	-
	Carbowax	7	4	6	4	4	9	34	5.7	-
	Cottonseed Stearine	6	5	7	4	6	8	36	6.0	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XI

COFFEE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	7	5	7	5	5	7	36	6.0	Slight to none
	Carbowax	8	6	7	7	5	5	38	6.3	Slight to none
	Cottonseed Stearine	8	6	6	6	6	7	39	6.5	Slight to none
High - 100°F Air-packed in a tin can*	Granulation	7	5	7	6	6	6	37	6.2	Slight to none
	Carbowax	8	6	6	5	7	6	38	6.3	None
	Cottonseed Stearine	8	7	6	5	8	5	39	6.5	None to slight
High - 100°F Pouch-packed (air) flavored bars	Granulation	4	3	5	4	3	5	24	4.0	-
	Carbowax	7	5	5	6	6	6	35	5.8	-
	Cottonseed Stearine	6	4	7	6	7	3	33	5.5	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	5	6	5	5	6	33	5.5	-
	Carbowax	7	4	8	7	8	4	38	6.3	-
	Cottonseed Stearine	7	5	8	7	9	7	43	7.2	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	5	7	7	5	5	36	6.0	-
	Carbowax	7	4	8	8	4	6	37	6.2	-
	Cottonseed Stearine	8	5	7	8	4	6	38	6.3	-
Recycling Pouch-packed (air) flavored bars	Granulation	3	7	7	5	6	7	35	5.8	-
	Carbowax	5	4	7	6	8	7	37	6.2	-
	Cottonseed Stearine	3	4	8	6	8	6	35	5.8	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XII

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

CHICKEN SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	5	8	6	7	8	6	40	6.7	Slight to none
	Carbowax	6	8	6	6	7	7	40	6.7	Slight to none
	Cottonseed Stearine	6	6	7	7	8	6	40	6.7	None
High - 100°F Air-packed in a tin can*	Granulation	5	7	6	7	7	7	39	6.5	Slight to none
	Carbowax	5	6	6	7	7	6	40	6.7	Slight
	Cottonseed Stearine	5	6	6	7	7	5	36	6.0	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	6	6	7	3	6	6	34	5.7	-
	Carbowax	6	6	6	4	8	6	36	6.0	-
	Cottonseed Stearine	7	7	5	4	9	5	37	6.2	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	7	7	8	7	5	6	40	6.7	-
	Carbowax	7	6	7	7	7	6	40	6.7	-
	Cottonseed Stearine	5	6	7	8	6	7	39	6.5	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	5	7	5	8	6	6	37	6.2	-
	Carbowax	6	7	6	8	5	7	39	6.5	-
	Cottonseed Stearine	6	6	6	8	7	6	39	6.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	7	8	6	7	7	40	6.7	-
	Carbowax	6	7	7	7	5	8	39	6.5	-
	Cottonseed Stearine	5	6	7	6	7	6	37	6.2	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

TABLE XIII

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

CHERRY

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	6	4	5	5	4	6	30	5.0	None
	Carbowax	5	5	3	7	6	6	32	5.3	None
	Cottonseed Stearine	6	6	3	5	6	4	30	5.0	Slight
High - 100°F Air-packed in a tin can*	Granulation	6	5	4	5	5	4	29	4.8	Slight
	Carbowax	6	5	4	7	6	5	33	5.5	Slight
	Cottonseed Stearine	6	6	3	5	6	4	30	5.0	Slight
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	4	5	2	2	5	23	3.8	-
	Carbowax	4	4	8	2	1	3	22	3.7	-
	Cottonseed Stearine	5	3	4	2	1	7	22	3.7	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	4	7	3	4	4	28	4.7	-
	Carbowax	4	4	7	4	7	6	32	5.3	-
	Cottonseed Stearine	4	4	6	3	8	3	28	4.7	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	6	6	7	7	3	4	33	5.5	-
	Carbowax	3	5	8	7	4	5	32	5.3	-
	Cottonseed Stearine	4	4	8	4	4	3	27	4.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	2	5	7	3	4	6	27	4.5	-
	Carbowax	6	5	6	4	3	8	32	5.3	-
	Cottonseed Stearine	4	4	5	3	5	7	28	4.7	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XIV

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

VANILLA

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	8	6	7	7	5	6	39	6.5	None
	Carbowax	7	5	7	6	6	5	36	6.0	Slight
	Cottonseed Stearine	7	6	6	6	5	5	35	5.8	Slight to none
High - 100°F Air-packed in a tin can*	Granulation	7	5	7	6	5	6	36	6.0	Slight to none
	Carbowax	7	5	7	6	6	5	36	6.0	Slight
	Cottonseed Stearine	7	6	6	6	5	5	35	5.8	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	4	5	5	4	4	27	4.5	-
	Carbowax	6	5	5	4	3	3	26	4.3	-
	Cottonseed Stearine	4	5	5	5	5	2	26	4.3	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	7	8	5	6	9	7	42	7.0	-
	Carbowax	7	6	6	6	6	8	39	6.5	-
	Cottonseed Stearine	6	7	5	5	8	7	43	7.2	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	6	5	6	7	4	8	36	6.0	-
	Carbowax	6	5	5	6	5	8	35	5.8	-
	Cottonseed Stearine	6	5	6	6	5	7	35	5.8	-
Recycling Pouch-packed (air) flavored bars	Granulation	4	5	6	5	6	7	33	5.5	-
	Carbowax	5	4	7	6	8	8	38	6.3	-
	Cottonseed Stearine	4	4	7	4	7	6	32	5.3	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XV

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

BACON-TOMATO SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	5	4	4	7	5	4	29	4.8	None
	Carbowax	5	5	5	6	5	5	31	5.1	None
	Cottonseed Stearine	5	4	4	7	5	4	29	4.8	None
High - 100°F Air-packed in a tin can*	Granulation	5	5	5	7	5	4	30	5.0	None
	Carbowax	5	6	6	7	5	4	33	5.5	None
	Cottonseed Stearine	5	5	5	6	5	4	30	5.0	None
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	4	5	5	4	4	27	4.5	-
	Carbowax	3	4	5	5	6	5	28	4.7	-
	Cottonseed Stearine	1	4	5	2	3	3	18	3.0	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	5	3	5	6	4	4	27	4.5	-
	Carbowax	4	3	4	7	4	2	24	4.0	-
	Cottonseed Stearine	5	3	5	5	4	3	25	4.2	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	5	6	6	6	6	6	35	5.8	-
	Carbowax	5	6	6	7	5	6	35	5.8	-
	Cottonseed Stearine	6	5	5	5	5	7	33	5.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	4	6	6	6	3	7	22	5.3	-
	Carbowax	5	7	7	6	6	6	37	6.2	-
	Cottonseed Stearine	4	5	6	4	3	7	29	4.8	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XVI

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

SPAGHETTI SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in á tin can*	Granulation	7	7	5	7	7	7	40	6.7	None
	Carbowax	6	8	5	7	6	6	39	6.5	None
	Cottonseed Stearine	7	7	5	7	6	7	39	6.5	None
High - 100°F Air-packed in a tin can*	Granulation	7	7	6	7	6	6	39	6.5	None
	Carbowax	5	7	5	6	6	7	36	6.0	None
	Cottonseed Stearine	4	6	5	6	5	6	32	5.3	None
High - 100°F Pouch-packed (air) flavored bars	Granulation	7	6	5	7	6	9	40	6.7	-
	Carbowax	5	7	6	6	5	8	37	6.2	-
	Cottonseed Stearine	3	3	7	5	5	6	29	4.8	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	7	6	6	6	5	8	38	6.3	-
	Carbowax	6	5	5	6	5	7	34	5.7	-
	Cottonseed Stearine	5	6	5	5	4	6	31	5.2	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	6	7	7	7	6	40	6.7	-
	Carbowax	7	5	8	5	5	6	36	6.0	-
	Cottonseed Stearine	6	5	7	6	6	5	35	5.8	-
Recycling Pouch-packed (air) flavored bars	Granulation	6	6	6	8	5	8	39	6.5	-
	Carbowax	5	5	6	7	5	7	35	5.8	-
	Cottonseed Stearine	5	5	5	5	4	8	32	5.3	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XVII

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

CHOCOLATE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	7	5	9	8	5	6	40	6.7	Slight
	Carbowax	7	7	9	7	6	8	44	7.3	Slight to none
	Cottonseed Stearine	8	7	8	7	7	7	44	7.3	Slight to none
High - 100°F Air-packed in a tin can*	Granulation	7	6	6	6	6	5	36	6.0	Slight to none
	Carbowax	8	7	6	7	5	7	40	6.7	Slight
	Cottonseed Stearine	8	8	6	6	5	7	40	6.7	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	6	4	7	4	1	5	27	4.5	-
	Carbowax	6	7	6	5	5	8	37	6.2	-
	Cottonseed Stearine	4	6	6	5	2	6	29	4.8	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	6	7	6	7	7	39	6.5	-
	Carbowax	7	7	8	8	8	8	46	7.7	-
	Cottonseed Stearine	7	7	8	7	7	6	42	7.0	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	6	8	6	6	7	40	6.7	-
	Carbowax	6	7	8	8	4	6	39	6.5	-
	Cottonseed Stearine	6	6	6	8	6	7	39	6.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	7	5	4	7	8	36	6.0	-
	Carbowax	4	7	7	4	6	8	36	6.0	-
	Cottonseed Stearine	4	7	6	4	7	9	37	6.2	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XVIII

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

BEEF-TOMATO SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	5	8	5	5	7	5	35	5.8	None
	Carbowax	5	8	6	4	7	6	36	6.0	Slight to none
	Cottonseed Stearine	5	9	7	4	8	6	39	6.5	Slight to none
High - 100°F Air-packed in a tin can*	Granulation	5	8	5	6	8	4	36	6.0	Slight to none
	Carbowax	5	6	6	6	7	5	35	5.8	Slight
	Cottonseed Stearine	5	5	6	5	8	6	35	5.8	Slight
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	6	5	6	6	7	35	5.8	-
	Carbowax	6	5	6	5	7	7	36	6.0	-
	Cottonseed Stearine	3	4	8	3	4	6	28	4.7	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	6	7	8	5	2	34	5.7	-
	Carbowax	6	5	6	7	5	3	32	5.3	-
	Cottonseed Stearine	6	6	7	6	5	2	32	5.3	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	6	7	6	6	2	34	5.7	-
	Carbowax	7	7	7	7	7	2	37	6.2	-
	Cottonseed Stearine	7	6	8	5	6	3	35	5.8	-
Recycling Pouch-packed (air) flavored bars	Granulation	6	6	8	8	6	5	39	6.5	-
	Carbowax	5	7	6	8	5	4	35	5.8	-
	Cottonseed Stearine	4	6	7	8	6	8	39	6.5	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XIX

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

LEMON

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	5	8	7	5	5	7	37	6.2	None
	Carbowax	6	7	6	6	5	6	36	6.0	Slight
	Cottonseed Stearine	6	6	7	6	6	7	38	6.3	Slight to none
High - 100°F Air-packed in a tin can*	Granulation	5	8	8	5	5	6	37	6.2	Slight
	Carbowax	6	6	8	6	5	7	38	6.3	Slight
	Cottonseed Stearine	7	7	7	7	5	6	39	6.5	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	5	5	5	3	5	6	29	4.8	-
	Carbowax	4	6	7	4	8	7	36	6.0	-
	Cottonseed Stearine	5	6	5	6	6	8	36	6.0	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	6	7	8	5	7	6	39	6.5	-
	Carbowax	5	6	7	6	7	4	35	5.8	-
	Cottonseed Stearine	7	6	7	6	5	3	34	5.7	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	6	6	7	7	7	4	37	6.2	-
	Carbowax	7	7	7	7	6	4	38	6.3	-
	Cottonseed Stearine	7	6	6	6	5	3	33	5.5	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	6	7	6	9	4	37	6.2	-
	Carbowax	7	7	8	7	9	5	43	7.2	-
	Cottonseed Stearine	4	6	6	7	8	3	34	5.7	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XX

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

CURRY

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	6	6	6	5	8	5	36	6.0	None
	Carbowax	7	5	6	5	8	6	37	6.2	None
	Cottonseed Stearine	6	5	6	6	8	5	36	6.0	None
High - 100°F Air-packed in a tin can*	Granulation	7	6	5	5	8	5	36	6.0	None
	Carbowax	7	5	5	5	7	6	35	5.8	None
	Cottonseed Stearine	7	5	5	6	6	5	34	5.7	None
High - 100°F Pouch-packed (air) flavored bars	Granulation	1	4	6	5	6	9	29	4.8	-
	Carbowax	1	4	7	5	2	8	27	4.5	-
	Cottonseed Stearine	6	5	5	4	3	6	29	4.8	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	4	5	5	6	7	8	35	5.8	-
	Carbowax	2	5	5	6	6	6	30	5.0	-
	Cottonseed Stearine	5	4	5	6	7	8	35	5.8	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	5	6	7	6	6	8	38	6.3	-
	Carbowax	5	6	6	6	5	8	36	6.0	-
	Cottonseed Stearine	5	7	6	6	5	8	37	6.2	-
Recycling Pouch-packed (air) flavored bars	Granulation	5	6	6	6	5	9	37	6.2	-
	Carbowax	5	6	5	6	5	8	35	5.8	-
	Cottonseed Stearine	5	6	5	6	5	9	36	6.0	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

Table XXI

SIX-MONTH STORAGE EVALUATION OF FOOD BARS

RICE SPICE

Storage Temperature and Method	Method of Stabilization	Testers							Average Hedonic Reading	Average Flavor Transfer
		1	2	3	4	5	6	Total		
High - 100°F Nitrogen-packed in a tin can*	Granulation	6	7	7	7	7	5	39	6.5	None
	Carbowax	6	6	6	6	6	5	35	5.8	Slight to none
	Cottonseed Stearine	5	8	7	6	6	5	37	6.2	None
High - 100°F Air-packed in a tin can*	Granulation	6	7	6	6	7	5	37	6.2	None
	Carbowax	6	6	7	6	6	5	36	6.0	None
	Cottonseed Stearine	5	6	7	6	6	5	35	5.8	Slight to none
High - 100°F Pouch-packed (air) flavored bars	Granulation	6	6	7	4	4	7	34	5.7	-
	Carbowax	6	6	4	3	4	6	29	4.8	-
	Cottonseed Stearine	5	4	6	3	2	8	28	4.7	-
Medium - 70°F Pouch-packed (air) flavored bars	Granulation	7	5	7	4	6	8	37	6.2	-
	Carbowax	3	5	6	6	4	7	31	5.2	-
	Cottonseed Stearine	5	5	6	5	5	7	33	5.5	-
Low - 35°F Pouch-packed (air) flavored bars	Granulation	7	7	7	6	5	3	35	5.8	-
	Carbowax	6	6	8	6	5	2	33	5.5	-
	Cottonseed Stearine	7	6	7	6	5	3	34	5.7	-
Recycling Pouch-packed (air) flavored bars	Granulation	6	6	7	6	5	7	37	6.2	-
	Carbowax	5	6	6	4	6	6	33	5.5	-
	Cottonseed Stearine	5	5	7	6	5	8	36	6.0	-

*Containing one open pouch of flavored bars and one open pouch of unflavored bars.

TABLE XXII

COMPOSITION OF FORMULA NUMBER 19

		%
Composition:		
	Lolac	14
	Cottonseed Stearine*	15
	Lactose	23
	Promine D	3
	Non-Fat Dry Milk Solids	35
Analysis:**		
	Protein	24.3
	Fat	17.0
	Carbohydrate	50.4

COMPOSITION OF FORMULA NUMBER 20

		%
Composition:		
	Lolac	50
	Cottonseed Stearine*	15
	Lactose	35
Analysis:**		
	Protein	25
	Fat	15
	Carbohydrate	50

COMPOSITION OF FORMULA NUMBER 21

		%
Composition:		
	Lolac	30
	Cottonseed Stearine*	15
	Lactose	45
	Promine D	4
Analysis:**		
	Protein	19.7
	Fat	16.0
	Carbohydrate	57.4

* Stabilized with Tenox IV
** On a dry basis

TABLE XXIII

METHODS FOR PREPARATION OF ENZYMES AND SUBSTRATES

Blueberry, Horseradish, Watercress

1. Blueberry

(a) Extraction of Enzymes from blueberries

500 grams of the berries were pulverized in a Waring Blendor with dry ice. This was extracted with 500 ml of 1.7% sodium borate (pH 4.5 adjusted to pH 8.0 with 30% sodium hydroxide), with stirring for two hours at room temperature (final pH = 7.4). The mixture was centrifuged and precipitated with an equal volume of cold acetone. After a 10-minute wait, the mixture was centrifuged and the precipitate slurried in water. The resultant preparation was freeze-dried.

(b) Substrate preparation from berries

Fresh frozen blueberry substrate was prepared by boiling 400 grams of the berries for 10 minutes with 400 ml water. The mixture was cooled and filtered through cheesecloth. The filtrate was stirred for 15 minutes with 5% Nuchar, filtered and frozen.

(c) Extraction of Enzymes from Blueberry Leaves

60 grams of the leaves were treated in a Waring Blendor with 450 cc of phosphate buffer (pH 7.0) for two hours at room temperature. The resultant material was filtered through cheesecloth and filtrate precipitated with an equal volume of cold acetone. The precipitate was centrifuged and freeze-dried.

(d) Substrate preparation from leaves

Blueberry leaf substrate was prepared by boiling 87 grams of dry leaves with about 800 cc water for 5 minutes. The mixture was cooled, run through a Waring Blendor and filtered through cheesecloth. The filtrate was stirred for 15 minutes with 5% Nuchar, filtered and frozen.

TABLE XXIII
(Continued)

2. Horseradish

(a) Substrate

Horseradish substrate was prepared from both fresh and commercial dehydrated material. The fresh horseradish was cut into strips, boiled for 15 minutes with water, then dried in a forced draft oven for 3-1/2 hours at 80°C. The dried strips were then ground in a Waring Blendor. The commercial dehydrated powder was boiled for 15 minutes and then lyophilized.

(b) Enzyme

Fresh horseradish was pulverized in a Waring Blendor with dry ice. The powder was extracted with an equal volume of cold distilled water for 1 hour at 5°C. Following extraction, the solids were removed by filtering through cheesecloth, and the filtrate further clarified by centrifugation. An equal volume of cold acetone was added to the supernatent and the resultant precipitate was centrifuged. The precipitate was slurried in water and lyophilized.

3. Watercress

(a) Substrate

100 grams of powdered dehydrated watercress was boiled for 15 minutes in water. The material was centrifuged and lyophilized.

(b) Enzyme

Myrosinase enzyme, prepared from white mustard seeds was used. 600 grams of white mustard seeds were ground with dry ice in a Waring Blendor and extracted with 2400 ml of cold water for 1 hour. This was filtered through cheesecloth and centrifuged. The supernatent was precipitated with an equal volume of 90% ethanol and centrifuged. The resulting precipitate was washed with 70% ethanol, centrifuged, suspended in 2 liters of water, filtered and lyophilized.

TABLE XXIV

PINEAPPLE ENZYME STUDIES

ACTUAL EXPERIMENTAL PROCEDURES FOR PREPARATION OF NEW ENZYMES

The pineapple used was fresh fruit obtained locally. An effort was made to purchase a quantity of fruit suitable for experimentation so that variance due to crop and ripeness of fruit was minimal. The fruit was bought in large batches and stored in a refrigerator at approximately 45°F. The actual history, i.e. age, variety, processing, and storage conditions, of the pineapple purchased is not known. During the experiment it was observed that the fruit was ripe and possessed a delightful aroma and characteristic flavor.

The fruit was divided by hand into its various sections, such as core, fruit, peel or skin, and leaves. Immediately after sectioning, the fruit was processed and/or frozen to minimize deterioration.

The freeze-drying was accomplished in a Model 15 RePP sublimator according to suggested operating procedures. The liquid samples were placed in suitable trays (small or large trays were used depending upon the amount of sample) and frozen in the freeze-drier until a temperature of approximately -50°F was obtained. The condensors were then activated until their temperature reached -40°F or lower. The vacuum pump was turned on. When the McLeod Gauge showed a pressure of 50 microns or less, the shelf heat was turned on at setting of 80°F.

The dried fruit substrate or enzyme was removed when the product temperature was equal to the shelf temperature. The shelf temperature was kept low to prevent any possible deterioration or inactivation of the sample due to excessive heating.

The following enzyme preparations were made:

1. The juice of two fresh pineapples was extracted through a Juice X Extractor to yield one liter of juice. The juice was centrifuged, acetone precipitated with an equal volume of -30°C acetone, and again centrifuged. The precipitate was slurried in water and freeze-dried to yield 1.5 grams of Enzyme 1.

TABLE XXIV

(Continued)

2. One fresh pineapple was peeled, and 1000 grams was extracted in a Waring Blendor with 400 ml of 1.7 percent sodium tetraborate at 5°C. The solution, having an initial pH 9.5 before the addition of the pineapple, had a pH 4 after mixing. The resulting pH was not readjusted. The solution was extracted for 5 minutes, filtered through cheese cloth, and centrifuged. The extract was precipitated with an equal volume of -30°C acetone, and the precipitate was slurried in water and freeze-dried to yield 1.5 grams of Enzyme 2.

3. Another fresh pineapple was extracted with 400 ml of phosphate buffer (pH 8). After mixing in the Waring Blendor, the pH became 2 and was then adjusted to pH 6.0 with 2N NaOH. The solution was filtered through cheese cloth, centrifuged, precipitated with acetone, and freeze-dried as above to yield 1.4 grams of Enzyme 3.

4. 100 grams of pineapple leaves were extracted with 200 ml of a 1.7 percent solution of sodium tetraborate, filtered, and centrifuged. The extract was pH 8 and was precipitated with acetone as above to yield 0.5 grams of Enzyme 4.

5. 100 grams of pineapple leaves were extracted with 0.1M citrate phosphate buffer (pH 5.6), filtered, centrifuged, and precipitated with acetone as above to yield 0.4 gram of Enzyme 5.

6. 300 grams of pineapple peel were extracted with 200 ml of a 1.7 percent sodium tetraborate as above, filtered, and centrifuged. The extract (pH 7.0) was precipitated with acetone to yield 1.7 grams of Enzyme 6.

7. 300 grams of pineapple peel were extracted with a citrate phosphate buffer (pH 5.6), filtered, centrifuged, and precipitated with acetone as above to yield 0.6 grams of Enzyme 7.

8. Pineapple peel was extracted with 30 percent sodium carbonate and then neutralized with acetic acid. This was freeze-dried without acetone precipitation to yield 43.1 grams of Enzyme 8.

9. Pineapple core was extracted with 40 ml of 30 percent sodium carbonate and neutralized with acetic acid and freeze-dried to yield 28.2 grams of Enzyme 9.

TABLE XXIV

(Continued)

10. 189 grams of core were extracted with an equal weight of cold water with 10 ml of 30 percent sodium carbonate. The extract was filtered, precipitated with acetone, and freeze-dried as in preparation to yield 0.45 grams of Enzyme 10.

11. 406 grams of pineapple peel were extracted with an equal weight of ice water and 30 percent sodium carbonate was added until a pH 8.0 was obtained. The extract was precipitated with acetone as above to yield 2.5 grams of Enzyme 11.

12. 1019 grams of pineapple were extracted with an equal weight of ice water and buffered to pH 8 with 30 percent sodium carbonate as above. The extract was filtered and precipitated with acetone to yield 0.75 grams of Enzyme 12.

13. Eight hundred grams of unprocessed core and peel from a fresh pineapple frozen overnight was ground in a Waring Blendor with one liter of water and 60 ml of 33-1/3 percent sodium carbonate solution, so that the slurry was positive to phenolthalein. The sample was filtered through 8-fold cheesecloth and Whatman No. 4 filter paper, acidified to pH 5, and freeze-dried to obtain Enzyme 13.

14. Four hundred fifty-six grams of fresh pineapple core and an equal weight of ice was placed in a Waring Blendor. To the mixture was added 200 milligrams of Pectionol 10M. The mixture was stirred and allowed to stand overnight. The sample was then heated to 180°F with stirring, cooled immediately with ice, and filtered through Whatman No. 4 and then No. 2 filter paper to yield 1400 ml of filtrate. To the filtrate was added 15 ml of 33-1/3 percent sodium carbonate solution. The solution was stirred, and 1400 ml of cold acetone (0°C was added in small increments with constant stirring. The mixture was placed in a freezer overnight. The preparation was centrifuged at 2000 R.P.M. for 20 minutes and the precipitate was slurried in water and freeze-dried to yield Enzyme 14.

15. To the residue from the filtration in Enzyme 14 preparation was added 600 ml of water and 2 ml of 33-1/3 percent sodium carbonate solution. The sample was filtered through Whatman No. 2 filter paper. The filtrate was saturated with sodium chloride and again filtered through Whatman No. 2 filter paper to yield residue which is Enzyme 15.

TABLE XXIV

(Continued)

16. Five hundred grams of pineapple skin (cold) was blended in a Waring Blendor with 600 ml of water and stirred for 2 hours. The sample was filtered through 8-fold cheesecloth, Whatman No. 4, No. 1, and No. 2 filter paper in sequence, to yield approximately one liter of filtrate. An equal volume of cold 95 percent ethanol was added, and the material was centrifuged at 2000 R.P.M. for 20 minutes. The residue was slurried in water, and the residual ethanol evaporated. The enzyme slurry was freeze-dried to yield 1.7 grams of Enzyme 16.

17. Two hundred twenty-seven grams of pineapple leaves and 900 ml of water and ice were ground in a Waring Blendor (final pH of 4.0). The material was filtered through 8-fold cheesecloth. Five grams of Nuchar was added, and the mixture was stirred for 10 minutes and filtered through Hyflo Supercel. The solution was freeze-dried to yield 5.7 grams of Enzyme 17.

18. One hundred nine grams of pineapple core and 100 ml of water were mixed in a Waring Blendor and brought to pH 8.5 with 30 ml of 33-1/3 percent sodium carbonate solution. The sample was filtered through cheesecloth, neutralized to pH 7.0 with hydrochloric acid, and again filtered through cheesecloth. The volume of the filtrate was 175 ml. This filtrate was freeze-dried to yield 11.2 grams of Enzyme 18.

TABLE XXV

PINEAPPLE ENZYME STUDIES

ACTUAL EXPERIMENTAL PROCEDURE FOR PREPARATION OF SUBSTRATE*

The substrate was prepared in a similar, but not as complex, procedure. The substrates made from the fruit can be freeze-dried if desired, but in commercial practice the processed foods themselves are the basic substrates of flavor precursors. While it is difficult to isolate the proper enzyme systems, care must also be taken to select the proper flavor substrates for the flavor enzyme associated with the total flavor desired.

1. One hundred forty-one grams of blanched pineapple fruit slices were freeze-dried and then ground to form a powder. The yield was 25 grams of Substrate A.*

2. One hundred nine grams of blanched pineapple core plus 100 ml of water were mixed in a Waring Blendor and brought to pH 8.5 with 30 ml of a 33-1/3 percent sodium carbonate solution. The sample was filtered through cheesecloth, neutralized to pH 7.0 with hydrochloric acid, and again filtered through cheesecloth. The volume of the filtrate was 175 ml. This filtrate was freeze-dried to yield 11.2 grams of Substrate B.

3. One hundred forty grams of blanched pineapple was comminuted in a Waring Blendor, then frozen and freeze-dried to yield 21.6 grams of Substrate C.

4. Two hundred thirty grams of blanched pineapple were put through a Juice X Extractor to yield 130 ml of liquid, which was then freeze-dried to yield 13.6 grams of Substrate D.

5. Seven hundred grams of pineapple was ground in a Waring Blendor. The material was heated to 140-145°F with constant stirring; Pectionol 10M was added to the material. The mixture was stirred for 2 hours and brought to 180°F in 20 minutes to inactivate the enzymes. The material was then filtered through Whatman No. 4 and through Whatman No. 2 filter paper. Two grams of Nuchar activated charcoal was added, and the mixture was stirred for 20 minutes and filtered through Hyflo Supercel and freeze-dried to yield 28.7 grams of dry Substrate E.

6. Substrate E (approximately 1/2 of Substrate E) was rehydrated and frozen to yield 250 ml of Substrate F.

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*Henceforth, substrates of which the preparation is reported in this section will be referred to as "Substrate A", "Substrate B", etc.

TABLE XXVI

COMPOSITION OF NEW FLAVORS AND FLAVOR-TO-BASE RATIOS

Sardine	Parts by Weight
Base Mix Formula No. 18	96
Sardine Extract (Freeze-dried)	4

Strawberry	
Base Mix Formula No. 18	90
Strawberries (Freeze-dried)	10

Soya	
Base Mix Formula No. 18	85
Soy Sauce Mix (Freeze-dried)	15

Soy Sauce Mix consists of:

Lactose	150 gm.
Soy Sauce	450 gm.
Wine Vinegar	30 gm.

Orange	
Base Mix Formula No. 18	85
Orange Crystals (McKees)	15

TABLE XXVII

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVORED FOOD BARS STORED IN SEALED METAL CANS

Flavor	Storage Temperature and Media	Testers							Average Hedonic Rating	Flavor Transfer
		1	2	3	4	5	6	Total		
Sardines	100°F-Oxygen	6	8	6	5	5	5	35	5.8	None
	100°F-Nitrogen	6	7	7	5	6	5	36	6.0	None
	70°F-Oxygen	5	8	6	5	6	6	36	6.0	None
	70°F-Nitrogen	6	8	6	5	6	6	37	6.1	None
	40°F-Oxygen	6	8	7	6	7	7	41	6.8	None
	40°F-Nitrogen	6	7	8	6	7	7	41	6.8	None
	R* - Oxygen	6	7	6	6	7	7	39	6.5	None
	R* - Nitrogen	6	8	6	6	7	7	40	6.6	None
Strawberry	100°F-Oxygen	7	9	8	6	7	7	44	7.3	None
	100°F-Nitrogen	7	7	7	7	7	7	42	7.0	None
	70°F-Oxygen	8	9	8	7	8	9	49	8.1	None
	70°F-Nitrogen	8	9	8	7	8	9	49	8.1	None
	40°F-Oxygen	9	9	7	7	9	9	50	8.3	None
	40°F-Nitrogen	9	9	7	7	9	9	50	8.3	None
	R* - Oxygen	9	9	8	7	9	9	51	8.5	None
	R* - Nitrogen	9	9	8	7	9	9	51	8.5	None
Soy	100°F-Oxygen	6	8	7	6	6	6	39	6.5	None
	100°F-Nitrogen	6	8	7	7	6	7	41	6.8	None
	70°F-Oxygen	7	8	8	6	6	6	41	6.8	None
	70°F-Nitrogen	7	8	7	7	6	7	42	7.0	None
	40°F-Oxygen	7	8	7	7	6	7	42	7.0	None
	40°F-Nitrogen	7	8	7	7	6	7	42	7.0	None
	R* - Oxygen	7	8	7	7	6	7	42	7.0	None
	R* - Nitrogen	7	8	8	7	6	7	43	7.1	None
Orange	100°F-Oxygen	5	6	8	6	6	6	37	6.1	None
	100°F-Nitrogen	5	7	7	6	5	6	36	6.0	None
	70°F-Oxygen	6	7	7	7	6	6	39	6.5	None
	70°F-Nitrogen	6	6	7	7	5	7	38	6.3	None
	40°F-Oxygen	6	7	6	7	6	7	39	6.5	None
	40°F-Nitrogen	6	7	6	7	5	8	39	6.5	None
	R* - Oxygen	6	7	6	7	6	7	39	6.5	None
	R* - Nitrogen	7	7	6	7	5	7	39	6.5	None

*Recycling

TABLE XXVIII

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVORED FOOD BARS PACKED IN ALUMINUM FOIL POUCHES

Flavor	Storage Temperature	Testers							Average Hedonic Rating
		1	2	3	4	5	6	Total	
Sardines	100°F	6	6	5	5	6	5	33	5.5
	70°F	6	6	6	6	6	5	35	5.8
	40°F	6	6	6	6	6	5	35	5.8
	Recycling	6	6	6	5	6	5	34	5.6
Strawberry	100°F	6	7	7	6	7	5	38	6.3
	70°F	6	7	7	7	7	6	40	6.6
	40°F	7	7	8	8	8	8	4	7.6
	Recycling	7	7	8	8	8	7	4	7.5
Soy	100°F	6	5	6	7	6	5	35	5.8
	70°F	6	6	6	7	6	6	37	6.1
	40°F	6	6	6	7	7	6	38	6.3
	Recycling	7	6	6	6	7	6	38	6.3
Orange	100°F	6	5	5	6	5	6	35	5.8
	70°F	6	7	5	6	6	7	37	6.1
	40°F	7	7	7	6	6	7	40	6.6
	Recycling	7	7	7	6	5	7	39	6.5

TABLE XXIX

PREPARATION OF SARDINE EXTRACT

The sardine extract was prepared from commercially canned sardines which were thoroughly drained of the oil used in packing. The sardines were ground in a variable-speed Waring blendor and extracted twice with ethyl alcohol (95%) to remove the oil fractions (approximately 500 grams of ethyl alcohol were used per 675 grams of ground sardines. A slurry was prepared using 500 ml of water to 675 gms of ground sardines and then heated to 80°C and allowed to simmer for four hours under constant agitation. The remaining liquids were decanted and freeze-dried. The freeze-drying was accomplished in a Model 15 RePP sublimator; the liquid extract was placed in stainless steel trays and frozen in the freeze-dryer until an internal temperature of -50°F was obtained. The condensers were then activated until their temperature reached -40°F, when the vacuum pump was turned on. When the McLeod Gauge showed a pressure of less than 50 microns, the shelf heat was turned on at a setting of 70°F.

The dried extract was removed when the product temperature was equal to the shelf temperature, which was kept low to prevent any possible deterioration of the sample. The dried water extract had a very good fish flavor and aroma; the alcohol extract was high in fish aroma and very low in fish flavor. The remaining extracted fish residue was lacking in fish flavor and aroma.

TABLE XXX

COMPOSITION OF FLAVOR ADJUNCTS AND FLAVOR

CURRY FLAVOR COMPOSITION

Curry Concentrate	Parts by Weight
Coriander Powder	750
Cumin Powder	300
Fenugreek Powder	300
Black Pepper Powder	180
Cardamon Powder	150
Mace Powder	120
Allspice Powder	105
Cinnamon Powder	105
Mustard Powder	60
Paprika Powder	45
Ginger Powder	30
Celery Powder	15
Cayenne Powder	15
Orange Oil	15

Base Mix To Flavor - Adjunct Ratio

	Base Mix Formula No. 18	85
	Curry/Lactose Mix	15
	consisting of:	
Lactose Adjunct:	Lactose	200
	Curry Flavor	50
	Carboxymethylcellulose 7HOP	10
	Base Mix Formula No. 18	92.5
	Curry/Salt Mix	7.5
	consisting of:	
Salt Adjunct:	Salt	200
	Curry Flavor	130
	Carboxymethylcellulose 7HOP	3
	Base Mix Formula No. 18	95.8
	Curry/Citric Mix	4.2
	consisting of:	
Citric Acid Adjunct:	Citric Acid	67
	Curry Flavor	200
	Carboxymethylcellulose 7HOP	2.5

TABLE XXX

(Continued)

COMPOSITION OF FLAVOR ADJUNCTS AND FLAVOR

CURRY FLAVOR COMPOSITION

(Continued)

	Base Mix Formula No. 18	95.8
	Curry/Mertaste 5' Mix	4.2
	consisting of:	
Mertaste 5 Adjunct:	Mertaste 5'	50
	Curry Flavor	150
	Carboxymethylcellulose 7HOP	3
	Base Mix Formula No. 18	96.8
	Curry/Disodium Guanylate Mix	3.2
	consisting of:	
Disodium Guanylate Adjunct:	Disodium Guanylate	4
	Curry Flavor	200
	Carboxymethylcellulose 7HOP	2

TABLE XXXI

COMPOSITION OF FLAVOR ADJUNCTS AND FLAVOR

LEMON FLAVOR COMPOSITION

Lemon Concentrate	Parts by Weight
Exchange Lemon Oil	1000
Lemon Oil 5X	500
Veltol - 2% in Ethyl Alcohol	100
Terpeneless Lemon Oil	50

Base Mix to Flavor/Protein/Salt/Carbohydrate Ratio

Base Mix Formula No. 18	92.5
Lemon/Lactose Mix	7.5
consists of:	
Lactose	200
Lemon Flavor Oil	50
Carboxymethylcellulose 7HOP	5
Base Mix Formula No. 18	96.3
Lemon/Salt Mix	3.7
consists of:	
Salt	200
Lemon Flavor Oil	150
Carboxymethylcellulose 7HOP	8
Base Mix Formula No. 18	98.6
Lemon/Mertaste 5' Mix	1.4
consists of:	
Mertaste 5'	150
Lemon Flavor Oil	450
Carboxymethylcellulose 7HOP	2
Base Mix Formula No. 18	98.1
Lemon/Disodium Guanylate Mix	1.9
consists of:	
Disodium Guanylate	3
Lemon Flavor Oil	150
Gum Arabic	100

TABLE XXXII

COMPOSITION OF FLAVOR ADJUNCTS AND FLAVOR

CHERRY FLAVOR COMPOSITION

Cherry Concentrate	Parts by Weight
Ethyl Oenanthate	1.2
Tolyl Aldehyde	12.5
Benzaldehyde N.F.	55.8
Candy Base Mix	8.5
Ethyl Alcohol	78.0

Candy Base Mix

Eugenol	2.0
Anisyl Acetate	9.0
Amyl Cinnamic Aldehyde	15.0
Absolute Jasmin	1.0
Vanillin	24.0
Ethyl Tolylglycidate	25.0
Anisyl Aldehyde	9.0

Base Mix to Flavor/Protein/Salt/Carbohydrate Ratio

Base Mix Formula No. 18		91
Cherry/Lactose Mix		9
consists of:		
Lactose	400	
Cherry Flavor Concentrate	100	
Carboxymethylcellulose 7HOP	5	
Base Mix Formula No. 18		96.4
Cherry/Salt Mix		3.6
consists of:		
Salt	300	
Cherry Flavor Concentrate	225	
Carboxymethylcellulose 7HOP	4	
Base Mix Formula No. 18		98.2
Cherry/Mertaste 5'		1.8
consists of:		
Mertaste 5'	150	
Cherry Flavor Concentrate	450	
Carboxymethyl Cellulose 7HOP	2	

TABLE XXXII

(Continued)

COMPOSITION OF FLAVOR ADJUNCTS AND FLAVOR

CHERRY FLAVOR COMPOSITION

(Continued)

Base Mix Formula No. 18	97.5
Cherry/Disodium Guanylate Mix consists of:	2.5
Disodium Guanylate	3
Cherry Flavor Concentrate	150
Cum Arabic	100

TABLE XXXIII

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN CURRY-FLAVORED FOOD BARS PACKED IN POUCHES

Flavor Adjunct	Storage Temperature	Testers							Average Hedonic Rating
		1	2	3	4	5	6	Total	
Lactose	100°F	7	6	5	7	7	6	38	6.3
	70°F	6	6	5	7	6	6	36	6.0
	40°F	6	6	5	6	6	7	36	6.0
	Recycling	6	6	5	6	6	7	36	6.0
Salt	100°F	6	6	6	5	5	6	34	5.6
	70°F	6	6	6	6	6	5	35	5.8
	40°F	7	6	6	6	6	7	38	6.3
	Recycling	7	6	6	6	6	6	37	6.1
Citric	100°F	6	6	6	5	6	6	35	5.8
	70°F	6	6	5	5	6	6	34	5.6
	40°F	7	6	6	5	6	6	36	6.0
	Recycling	7	6	6	5	6	6	36	6.0
Disodium Gyanylate	100°F	6	5	5	5	5	6	32	5.3
	70°F	6	6	6	6	6	5	35	5.8
	40°F	6	6	6	6	6	6	36	6.0
	Recycling	5	6	6	6	6	6	35	5.8

TABLE XXXIV

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN LEMON-FLAVORED FOOD BARS PACKED IN POUCHES

Flavor Adjunct	Storage Temperature	Testers							Average Hedonic Rating
		1	2	3	4	5	6	Total	
Lactose	100°F	6	6	6	7	7	6	38	6.3
	70°F	7	6	7	7	7	7	41	6.8
	40°F	7	6	7	7	8	8	43	7.1
	Recycling	7	7	7	7	8	7	43	7.1
Salt	100°F	5	6	5	6	6	6	34	5.6
	70°F	6	6	5	6	6	6	35	5.8
	40°F	6	6	6	6	6	7	37	6.1
	Recycling	5	6	6	7	5	6	35	5.8
Gyanylate	100°F	5	5	5	6	7	6	34	5.6
	70°F	5	6	6	6	6	6	34	5.6
	40°F	6	7	6	6	6	6	37	6.1
	Recycling	7	6	7	6	6	6	38	6.3
Mertaste	100°F	5	5	5	6	5	6	32	5.3
	70°F	5	5	5	6	6	5	32	5.3
	40°F	6	6	7	7	6	6	38	6.3
	Recycling	6	6	6	6	6	7	37	6.1

TABLE XXXV

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN CHERRY-FLAVORED FOOD BARS PACKED IN POUCHES

Flavor Adjunct	Storage Temperature	Testers							Average Hedonic Rating
		1	2	3	4	5	6	Total	
Lactose	100°F	5	6	6	6	7	6	36	6.0
	70°F	6	6	6	6	7	6	37	6.1
	40°F	7	6	6	7	7	5	40	6.6
	Recycling	7	6	5	7	7	6	38	6.3
Salt	100°F	5	6	5	5	6	5	32	5.3
	70°F	5	6	5	6	7	5	34	5.6
	40°F	6	6	6	6	7	5	36	6.0
	Recycling	5	6	5	6	7	6	35	5.8
Disodium Gyanylate	100°F	5	5	5	5	5	5	30	5.0
	70°F	6	6	5	5	6	6	34	5.6
	40°F	6	7	6	6	6	6	37	6.1
	Recycling	6	6	6	6	5	6	35	5.8
Mertaste 5	100°F	6	7	6	6	6	6	37	6.1
	70°F	7	7	6	6	6	6	38	6.3
	40°F	6	7	7	7	6	6	39	6.5
	Recycling	6	6	6	6	7	7	38	6.3

TABLE XXXVI

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN CURRY-FLAVORED FOOD BARS PACKED IN SEALED METAL CANS

Flavor Adjunct	Storage Temperature and Media	Testers							Average Hedonic Rating	Flavor Transfe
		1	2	3	4	5	6	Total		
Lactose	40°F-Oxygen	7	6	5	6	6	6	36	6.0	None
	40°F-Nitrogen	7	6	5	6	6	6	36	6.0	None
	R* -Oxygen	7	6	5	6	6	6	36	6.0	None
	R* -Nitrogen	7	6	5	6	6	6	36	6.0	None
	70°F-Oxygen	6	6	5	7	7	7	38	6.3	None
	70°F-Nitrogen	6	6	5	7	7	7	38	6.3	None
	100°F-Oxygen	7	6	4	8	6	6	37	6.1	None
	100°F-Nitrogen	8	6	4	6	7	7	38	6.3	None
Carboxy Methyl Cellulose	40°F-Oxygen	6	7	5	5	6	6	35	5.8	None
	40°F-Nitrogen	6	7	5	6	5	6	34	5.6	None
	R* -Oxygen	6	7	5	6	6	6	36	6.0	None
	R* -Nitrogen	6	7	5	6	6	6	36	6.0	None
	70°F-Oxygen	6	7	5	6	5	6	35	5.8	None
	70°F-Nitrogen	6	7	5	6	5	6	35	5.8	None
	100°F-Oxygen	6	7	4	5	6	6	34	5.6	None
	100°F-Nitrogen	6	6	5	5	6	6	34	5.6	None
Citric Acid	40°F-Oxygen	5	6	6	7	7	7	38	6.3	None
	40°F-Nitrogen	5	6	6	7	7	7	38	6.3	None
	R* -Oxygen	5	6	6	7	7	6	37	6.1	None
	R* -Nitrogen	5	6	6	6	7	6	36	6.0	None
	70°F-Oxygen	5	6	6	7	7	7	38	6.3	None
	70°F-Nitrogen	5	6	6	6	7	6	36	6.0	None
	100°F-Oxygen	5	5	6	6	7	7	36	6.0	None
	100°F-Nitrogen	5	5	6	7	7	7	37	6.1	None
Mertaste 5	40°F-Oxygen	6	6	6	7	6	6	37	6.1	None
	40°F-Nitrogen	6	6	7	7	6	6	38	6.3	None
	R* -Oxygen	6	6	7	7	5	6	37	6.1	None
	R* -Nitrogen	6	6	6	7	6	6	37	6.1	None
	70°F-Oxygen	6	6	7	7	6	6	38	6.3	None
	70°F-Nitrogen	6	6	6	7	6	6	37	6.1	None
	100°F-Oxygen	6	6	6	7	6	6	37	6.1	None
	100°F-Nitrogen	6	5	5	7	7	5	35	5.8	None
Gyanylate	40°F-Oxygen	7	6	5	6	5	5	34	5.6	None
	40°F-Nitrogen	7	6	5	5	6	5	34	5.6	None
	R* -Oxygen	7	6	5	6	5	6	35	5.8	None
	R* -Nitrogen	7	6	5	5	5	6	34	5.6	None
	70°F-Oxygen	7	6	5	6	6	7	37	6.1	None
	70°F-Nitrogen	7	7	5	7	6	6	38	6.3	None
	100°F-Oxygen	6	7	5	6	5	6	35	5.8	None
	100°F-Nitrogen	6	7	6	6	6	5	36	6.0	None

*Recycling

TABLE XXXVII

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN LEMON-FLAVORED FOOD BARS PACKED IN SEALED METAL CANS

Flavor Adjunct	Storage Temperature and Media	Testers							Average Hedonic	Flavor Transfer
		1	2	3	4	5	6	Total		
Lactose	40°F-Oxygen	8	6	5	7	7	6	39	6.5	None
	40°F-Nitrogen	8	7	5	7	7	6	40	6.6	None
	R* -Oxygen	8	7	5	7	7	6	40	6.6	None
	R* -Nitrogen	6	7	5	7	7	6	38	6.3	None
	70°F-Oxygen	6	6	5	6	6	6	35	5.8	None
	70°F-Nitrogen	6	6	5	6	6	6	35	5.8	None
	100°F-Oxygen	5	7	4	6	5	6	33	5.5	None
	100°F-Nitrogen	5	8	4	5	5	6	33	5.5	None
Salt	40°F-Oxygen	6	6	6	6	6	6	36	6.0	None
	40°F-Nitrogen	6	5	6	7	6	5	35	5.8	None
	R* -Oxygen	6	6	7	6	6	6	37	6.1	None
	R* -Nitrogen	6	6	7	6	6	6	37	6.1	None
	70°F-Oxygen	6	7	7	6	6	6	38	6.3	None
	70°F-Nitrogen	6	7	7	6	6	7	39	6.5	None
	100°F-Oxygen	5	7	6	6	5	7	36	6.0	None
	100°F-Nitrogen	5	7	6	5	5	7	35	5.8	None
Gyanylate	40°F-Oxygen	7	7	7	8	7	6	42	7.0	None
	40°F-Nitrogen	7	7	7	8	8	7	44	7.3	None
	R* -Oxygen	7	6	7	7	7	7	41	6.8	None
	R* -Nitrogen	6	6	7	8	8	7	42	7.0	None
	70°F-Oxygen	8	5	6	6	5	7	37	6.1	None
	70°F-Nitrogen	7	5	6	6	6	6	36	6.0	None
	100°F-Oxygen	6	5	5	5	5	5	31	5.1	None
	100°F-Nitrogen	6	5	5	5	5	6	32	5.3	None
Mertaste	40°F-Oxygen	7	6	6	7	6	7	39	6.5	None
	40°F-Nitrogen	7	6	6	7	6	7	39	6.5	None
	R* -Oxygen	7	6	6	7	6	6	38	6.3	None
	R* -Nitrogen	6	6	7	7	6	7	39	6.5	None
	70°F-Oxygen	6	5	6	7	5	7	36	6.0	None
	70°F Nitrogen	6	5	6	5	5	6	33	5.5	None
	100°F-Oxygen	6	5	5	5	5	5	31	5.1	None
	100°F-Nitrogen	6	5	6	5	5	5	32	5.3	None

*Recycling

TABLE XXXVIII

RESULTS OF SIX-MONTH STORAGE TESTS ON FLAVOR ADJUNCTS IN CHERRY-FLAVORED FOOD BARS PACKED IN SEALED METAL CANS

Flavor Adjunct	Storage Temperature and Media	Testers							Average Hedonic Rating	Flavor Transfer
		1	2	3	4	5	6	Total		
Lactose	40°F-Oxygen	7	8	7	6	7	6	41	6.8	None
	40°F-Nitrogen	7	8	7	7	7	6	42	7.0	None
	R* -Oxygen	7	8	7	6	7	6	41	6.8	None
	R* -Nitrogen	7	7	7	7	7	5	40	6.6	None
	70°F-Oxygen	6	7	7	7	7	5	39	6.5	None
	70°F-Nitrogen	6	7	7	7	7	5	39	6.5	None
	100°F-Oxygen	6	6	7	6	6	5	36	6.0	None
	100°F-Nitrogen	6	6	7	7	7	5	38	6.3	None
Salt	40°F-Oxygen	5	6	6	5	6	6	34	5.6	None
	40°F-Nitrogen	5	5	6	6	5	6	33	5.5	None
	R* -Oxygen	5	6	6	5	5	6	33	5.5	None
	R* -Nitrogen	5	6	6	6	5	6	34	5.6	None
	70°F-Oxygen	5	6	6	6	5	6	34	5.6	None
	70°F-Nitrogen	5	6	5	6	5	6	33	5.5	None
	100°F-Oxygen	5	6	5	5	5	6	32	5.3	None
	100°F-Nitrogen	5	6	5	5	6	6	33	5.5	None
Disodium Gyanylate	40°F-Oxygen	6	7	6	6	5	7	37	6.1	None
	40°F-Nitrogen	6	7	6	6	5	7	37	6.1	None
	R* -Oxygen	6	7	6	6	5	7	37	6.1	None
	R* -Nitrogen	6	7	7	6	5	7	38	6.3	None
	70°F-Oxygen	6	6	6	6	5	7	36	6.0	None
	70°F-Nitrogen	6	6	7	6	5	7	37	6.1	None
	100°F-Oxygen	6	6	6	6	5	6	35	5.8	None
	100°F-Nitrogen	6	6	6	6	4	6	34	5.6	None
Mertaste 5	40°F-Oxygen	7	7	6	7	6	6	39	6.5	None
	40°F-Nitrogen	7	7	6	7	7	5	39	6.5	None
	R* -Oxygen	7	7	6	6	6	5	37	6.1	None
	R* -Nitrogen	7	7	6	6	7	6	39	6.5	None
	70°F-Oxygen	6	7	6	6	6	5	37	6.1	None
	70°F-Nitrogen	7	7	6	6	6	6	38	6.3	None
	100°F-Oxygen	7	7	6	7	6	6	39	6.5	None
	100°F-Nitrogen	7	7	7	7	6	6	40	6.6	None

*Recycling

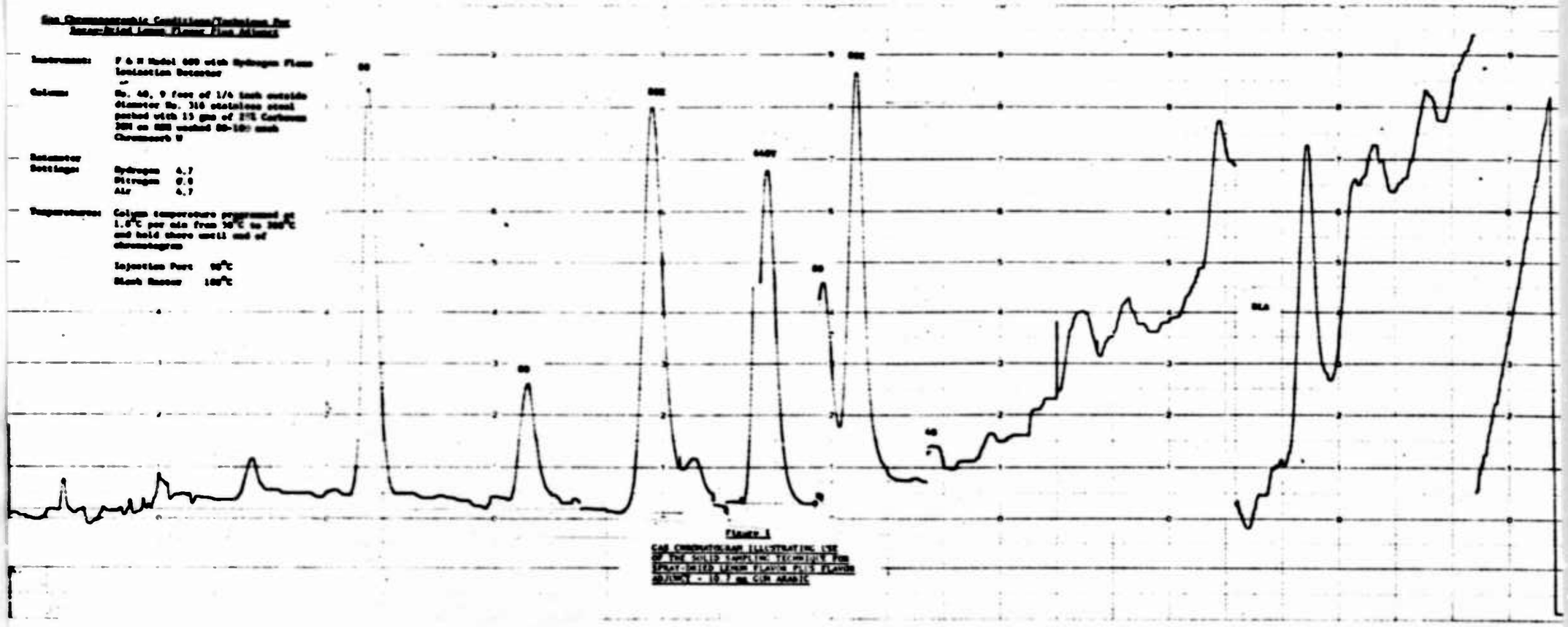

Figure 1

GAS CHROMATOGRAM ILLUSTRATING USE OF THE SOLID SAMPLING TECHNIQUE FOR SPRAY-DRIED LEMON FLAVOR PLUS FLAVOR ADJUNCT - 10.7 mg CIN 464616

Gas Chromatographic Conditions/Technique for Instant Dried Lemon Flavour Flash Aliquots

Instrument: F & M Model 609 with Hydrogen Flame Ionization Detector

Column: No. 40, 9 Feet of 1/4 inch outside diameter No. 316 stainless steel packed with 15 gms of 25% Carbowax 20M on HMDS washed 80-100 mesh Chromosorb W

Rotameter Settings:

Hydrogen	4.7
Nitrogen	0.8
Air	4.7

Temperature: Column temperature programmed at 1.8°C per min from 50°C to 200°C and held there until end of chromatogram

Injection Port 90°C

Block Heater 180°C

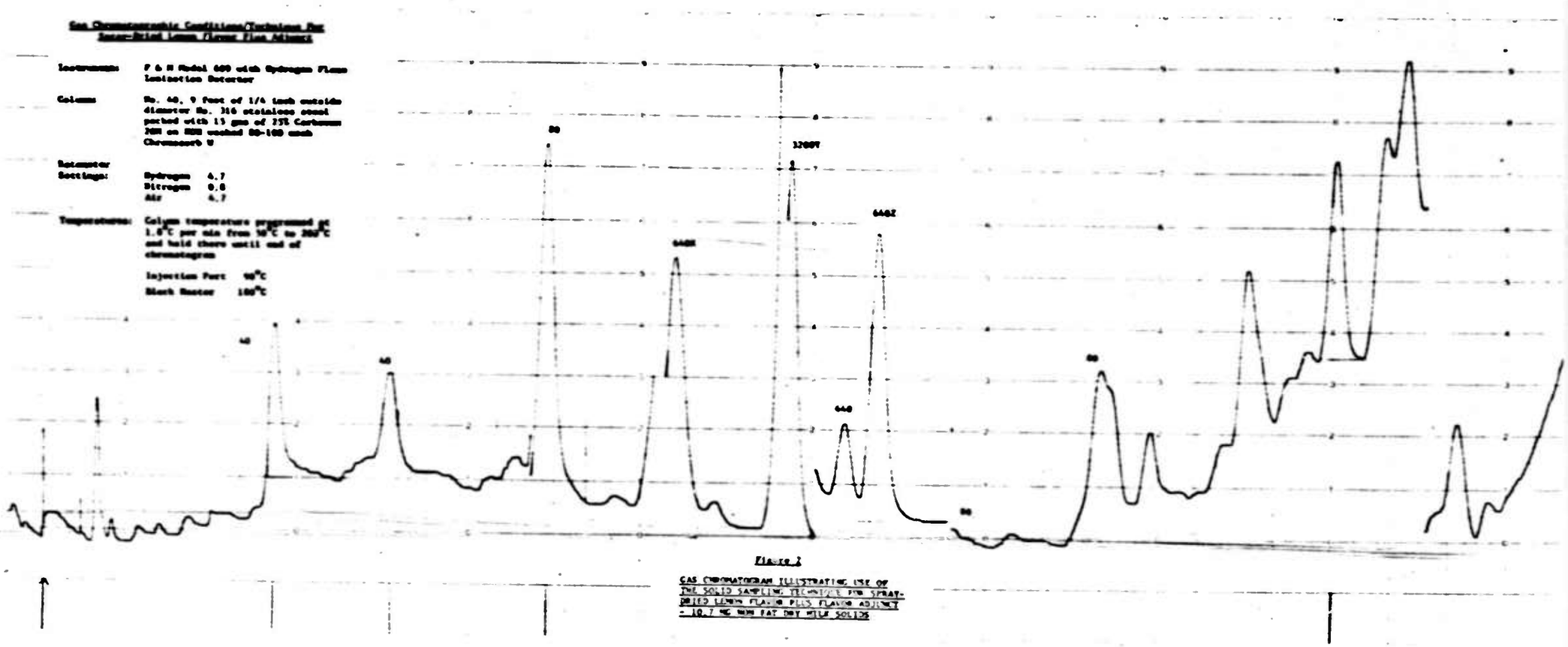

FIGURE 2

GAS CHROMATOGRAM ILLUSTRATING USE OF THE SOLID SAMPLING TECHNIQUE FOR SPRAY-DRIED LEMON FLAVOR PLUS FLAVOR ADJUNCT - 10.7 MG NON FAT DRY MILK SOLIDS

Gas Chromatographic Conditions/Technique For Freeze-Dried Lemon Flavour Fish Analogue

Instrument: F & M Model 609 with Hydrogen Flame Ionization Detector

Column: No. 40, 9 feet of 1/4 inch outside diameter No. 316 stainless steel packed with 15 gms of 25% Carbowax 3-20M on HMDS washed 80-100 mesh Chromosorb W

Detector Gas Flows:

Hydrogen	4.7
Nitrogen	6.0
Air	4.7

Temperatures: Column temperature programmed at 1.8°C per min from 50°C to 200°C and held there until end of chromatogram

Injection Port: 90°C

Block Heater 100°C

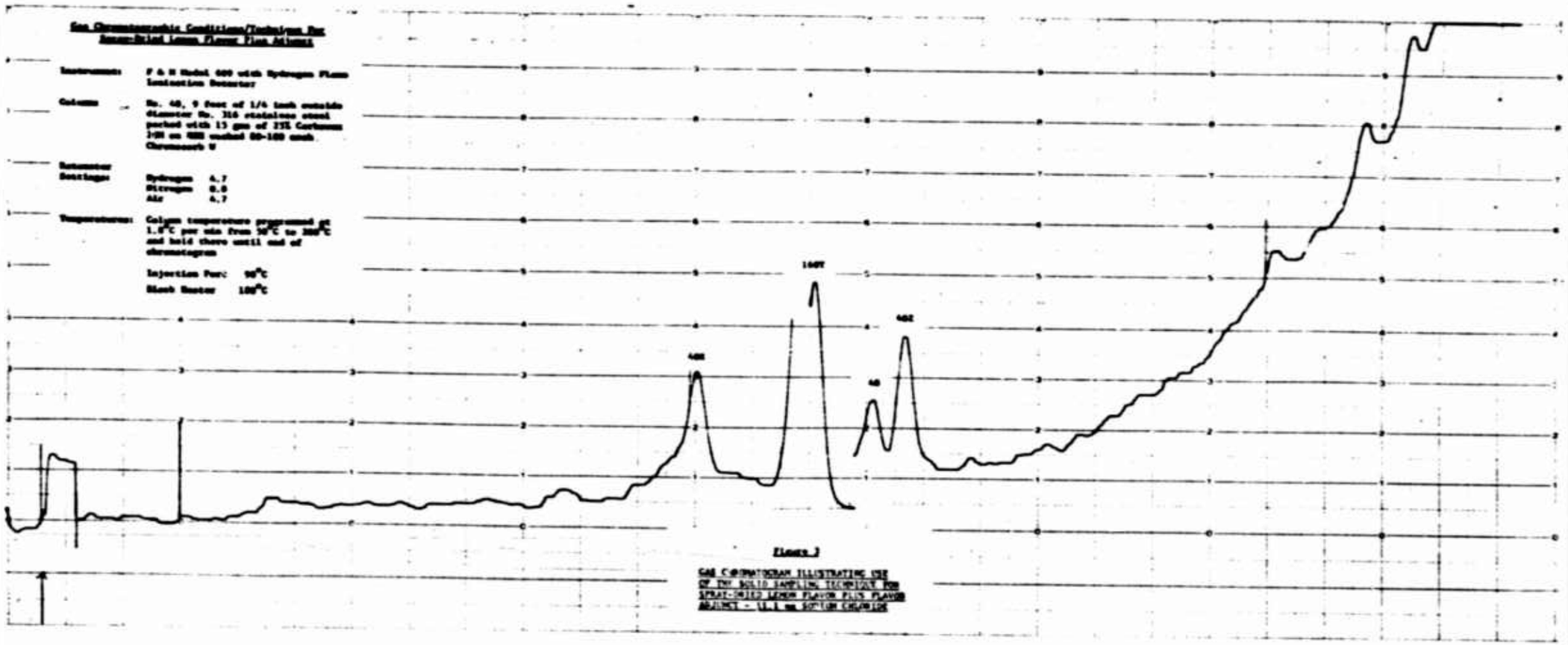

Figure 3

GAS CHROMATOGRAM ILLUSTRATING USE OF THE SOLID SAMPLING TECHNIQUE FOR SPRAY-DRIED LEMON FLAVOUR FISH FLAVOUR ANALOGUE - 15.1 mg SOLIDS INJECTED