Rancidity in Flaked Breakfast Cereals

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IN THE merchandising of cereal flakes, particularly wheat flakes, conditions are occasionally encountered in warehouse storage or elsewhere in transit, such as to induce an objectionably rancid odor which is encountered on opening the package. With the introduction during recent years of more impervious package liners, the development of rancidity has been more frequently apparent. This is attributed to the fact that such liners are not only more impervious to moisture vapor, but also to the organic vapors responsible for rancid odors. Furthermore, the inhibition of moisture absorption by the cereal product, which inhibition is necessary to prevent toughening of the product, can of itself accelerate the development of rancidity as will be shown in what follows.

As early as 1924 Holm and Greenbank (5) showed that small amounts of moisture serve to inhibit the absorption of oxygen by butter fat. In 1928 Fine and Olsen (3) showed that white patent flour would develop rancidity relatively rapidly when dried to a moisture level substantially less than 5%, whereas, at its normal moisture content, in the neighborhood of 10%, it could be kept for several years without developing any rancid odors. Triebold and Bailey in 1932 (7) showed that crackers when dried down to moisture levels in the neighborhood of 1% or less, developed rancidity more rapidly than at their normal moisture level of 5 to 6%. Present experience with cereal flakes is in complete harmony with the foregoing since it has been found that, in the range from about 2% to roughly 6% moisture, there is a pronounced increase in resistance to rancidity development with increasing moisture content.

In a recent patent, Wilder and Lindow (8) point out the fact that cereal flakes processed without salt are noticeably less prone to develop rancidity than those made in the normal manner involving the addition of salt to the cooker batch. They attribute this difference to the liberation of free hydrochloric acid in the case of cereal grains cooked with salt, and further remark that, "Under the further influence of the heat in the cooking operation and in the toasting operation this small quantity of hydrochloric acid, produced during the cooking and subsequent toasting operation, probably acts upon the fatty substances present in the food tending to promote acid hydrolysis of the fat with a consequent production of those decomposition products which accelerate the development of rancidity." Wilder and Lindow go on to propose that salt can be incorporated in the finished cereal products by spraying a moderately strong brine on the products after the final toasting step, and that flakes prepared in this way are substantially more stable than those made with salt present during the cooking operation. The author's experience fully substantiated the first of the foregoing points, namely, it has been consistently observed that flakes made without salt are more resistant to rancidity development, other things being equal, than are flakes made in the normal way with salt. However, it is not possible to accept their explanation of the mechanism whereby salt exerts a rancidity promoting effect. No substantial fat splitting effect can be conceived as resulting from the minute amount of free hydrochloric acid which could exist in a medium at the pH prevailing in a cereal grain, namely approximately pH 5.5. Furthermore, the cereal grain system is so well buffered that any fat splitting reaction resulting from an acid environment is likely to proceed just as extensively whether or not salt is present. It cannot be shown that the exclusion of salt substantially alters the pH of the product. It is also impossible to show that the addition of salt to the flakes in the form of a spray at the end of the manufacturing process is any less destructive of stability than the addition of salt at any other stage.

Although the effects of moisture and of salt on the stability of cereal flakes have been clearly demonstrated, it is not easily practical to control these variables in such a way as to solve the problem of commercial distribution of cereal flakes in a stable form. There is a practical upper limit to the moisture content which can be tolerated in a ready-to-eat cereal product which is set by the crispness of the product. Tests have shown that a reduction in the concentration of the salt employed does not materially improve the stability of the product until the salt level decreases to zero. Complete elimination of salt, of course, would result in a very unpalatable product.

The work to be described in this paper was undertaken for the purpose of providing, first, a more complete understanding of the mechanism whereby salt and moisture produce their respective effects, and second, in the hope of developing some means of improving the stability of the product by the incorporation of either synthetic or natural antioxygens.

THE STABILITIES OF WHEAT GERM AND WHEAT BRAN OILS

One of the first steps to be taken in approaching the problem of rancidity in cereal products was to determine the fundamental stabilities of the component fatty oils. Hexane extracts of commercial grades of flaked wheat germ and of wheat bran were prepared, the solvent removed by evaporation at low temperature and by aspiration with nitrogen, and the resulting oil extracts were subjected to accelerated oxidation under an atmosphere of pure oxygen at 90° C. in the apparatus described in an earlier publication from this laboratory (1). Briefly, the method consists of agitating a 5 to 10 cc. sample of the oil in a 50 cc. bulb immersed in a thermostat at 90° C. The bulb is connected by means of capillary tubing to a gas measuring burette which, in turn, is connected with a mercury reservoir in such a way that as oxygen is absorbed, mercury flows in from a constant level reservoir to maintain the system at constant pressure. The bulb, gas burette, and connecting tubing are filled with pure oxygen at atmospheric pressure at the beginning of the run. The volume

of mercury pulled over into the burette, which measures the volume of oxygen absorbed, is plotted against time and the length of the oxygen absorption induction period is determined graphically. A large number of measurements on a variety of oils has established the fact that the induction period determined graphically by the oxygen absorption method coincides in every case with the induction period as determined by the appearance of organoleptic rancidity in the sample undergoing accelerated oxygen absorption. The oxidation induction periods observed by this method are compared in Table 1 with similar values obtained on a few other edible oils listed for the purpose of comparison. The most striking point about these results is the fact that it is virtually impossible to make wheat bran oil go rancid, its stability being comparable with that of cocoa butter and paraffin wax. On the other hand, wheat germ oil is to be grouped among the least stable oils. Since this marked difference in the stabilities of wheat germ and wheat bran oils is not reflected in any significant differences between the ordinary fat constants for the two oils, it seems probable that the bran oil contains a small amount of a very potent natural antioxygen,

TABLE 1

Oxidation Induction Periods of Representative Fats and Oils at 90°C. Under One Atmosphere Oxygen Pressure

Fat or Oil	Induction Period hours
Cod Liver Oil	1.0—1.5
Peanut Oil, Refined	10
Lard	10
Green Coffee Oil	10—15
Sesame Oil, Refined	12
Wheat Germ Oil	6—20
Refined Corn, Cottonseed, Coconut Oils	20—25
Roasted Coffee Oil	30
Crude Corn, Cottonseed Oils	35—45
Crude Coconut Oil	95
Crisco, Primex	70—106—296
Paraffin Wax	250—500
Cocoa Butter	400—450
Wheat Bran Oil	Greater than 300

probably associated with its unsaponifiable fraction. To test this idea a series of five mixtures was set up, ranging from 100% wheat germ oil to 100% wheat bran oil, through three intermediate proportions. When these mixtures were submitted to the accelerated oxidation test, it was found that the oxygen absorption induction period increased systematically with increasing content of wheat bran oil, thus supporting the hypothesis that the bran oil contains a natural antioxygen. As a negative control for this group, it was shown that for two similar series prepared by mixing wheat germ oil with paraffin oil, in one case, and wheat germ oil with cocoa butter, in another, the induction period remained substantially identical with that of pure wheat germ oil as long as the mixture contained any wheat germ oil, showing that neither paraffin nor cocoa butter contain natural antioxygens which can function in stabilizing wheat germ oil. These observations at once suggested that the incorporation of additional small portions of wheat bran oil in the cooker batch might serve to improve the stability of the finished product. However, experimental batches of wheat flakes, prepared with as much as 2% added wheat bran oil, failed to show any improved stability. Since the initial oil content of whole wheat is of the order of 2% this failure of a bran oil supplement amounting to 100% of the original oil, implied that the bran oil of whole wheat is already exerting its full effect in inhibiting the greater rancidity tendency of wheat germ oil in a whole wheat cereal. This conclusion was further substantiated by preparing flakes from hand degerminated wheat and comparing the stability of the product with flakes made from whole wheat. No difference in stability was found. On the other hand, flakes prepared from the germ end of the wheat berry were far less stable than those made either from whole wheat or from degerminated wheat. Finally, the hexane extract from finished wheat flakes showed much the same properties as wheat bran oil in the accelerated oxidation test. Oxygen absorption in this case was very slow and approximately linear in character. That is, there was little indication of an induction period and in the great majority of cases no definite rancid odor developed after prolonged oxidation. These results make it appear that the instability of processed cereal flakes is related to the physical state of the oil fraction, its dispersion in the fiber structure of the flakes, the effect of salt interfaces, etc., rather than to any far-reaching changes occurring in the oil itself during processing or to the inherent instability of wheat germ oil. Incubation tests on dried cooked "grits," that is, the fully cooked and flavored wheat berry taken from the process just prior to flaking and dried down to the moisture content of the finished flake, have shown a remarkably high stability for this product in comparison with the flaked product. In parallel tests it has been shown that ordinary wheat farina, which normally is a very stable product, can be made to develop rancidity very rapidly after cooking by merely passing it over the flaking roll and drying down to 2 to 5% moisture. Independent tests had shown that the amount of metallic contamination normally encountered in the flaking operation could not account for the observed effects. When cooked grits were flaked by hand, using a porcelain roller on plate glass, the product was no more stable than a portion of the same batch flaked on steel rolls in the factory. These results support the view that it is the physical extrusion of oil from the oil cells and the resultant smearing of the fat constituents of the grain over a relatively large surface, greatly increasing their exposure to atmospheric oxygen, which has the greatest effect of all of the processing operations in accelerating the development of rancidity in flaked cereal products.

THE EFFECTS OF ADDED ANTIOXYGENS

Although it has been shown that the wheat bran lipids contribute a potent natural antioxygen to whole wheat flakes, it was felt that other synthetic or natural antioxygens might be found which would have more far-reaching specific effects on the oil as it exists in the finished flakes, that is, it was hoped that we could find an antioxygen which would offset the specific effect of the flake surface which had been shown to be the principal factor contributing to the instability of the finished product. The antioxygens studied in this connection can be classified into three general groups: First, products derived from food sources without alteration in chemical composition, such as lecithin and raw oat flour (Avenex); second, a group of synthetic materials now being exploited in the food field either on the basis of demonstrated lack of toxicity or on the basis of identity with normal food components. This group includes such substances as isoascorbic acid, gluco-ascorbic acid, amino glucose, glucamine, alkali phosphate, methyl salicylate and a synthetic phenolic compound which is being exploited on the basis of its identity with a constituent of Star anise oil (Fries Brothers "MEH"). As a subgroup under this

class may be included also sarcosine, cysteine, and hydroquinone. In spite of its known toxicity hydroquinone has been used to a limited extent in cod liver oil at very low concentrations. The toxicities of sarcosine and of cysteine are not known definitely but are assumed to be quite low on account of their existence in many proteins. The third group comprises a series of heterocyclic imino compounds which have recently been shown to include some of the antioxygenic materials developed in the roasting of coffee (2). Many of these compounds are undoubtedly toxic and could not be used in a cereal product in more than trace amounts.

The results of incubation tests carried out on cereal flakes prepared with small additions of the above compounds to the cooker batch, as listed in Table 2, show that with four exceptions all of them are completely ineffective in delaying the onset of rancidity. Among the exceptions, piperidine at a concentration of 0.1% and hydroquinone at concentrations of 0.5% and 3.0%, are of no practical importance since the toxicity of these compounds precludes their use at such high concentrations.

To demonstrate that these compounds are, on the whole, definitely antioxygenic in vegetable oils and in particular in wheat germ oil, oxygen absorption experiments were carried out with wheat germ oil treated with small amounts of each of the antioxygens. The degree of protection, as determined by the length of the induction period, was found to be quite high in general as indicated by the results tabulated in Table 2.

TABLE 2

The Protective Effects of Added Antioxygens on Dry Wheat Germ Oil, on Wheat Germ Oil Cooked with Brine, and on Wheat Flakes

Added Antioxygens	Dry Oil		Brine Cooked Oil		Wheat Flakes	
	Concentration*	Protection Factor†	Concentration*	Protection Factor†	Concentration**	Protective Effect
	per cent		per cent		per cent	
Avenex					5.0	0
Lecithin	0.3	1.1			0.5	0
Iso-ascorbic acid	0.2	2.4	0.2	1.9		
			5.0	2.5	0.1	0
Gluco-ascorbic	0.2	2.2	0.2	1.5	0.05	—
acid	0.5	5.3	0.5	1.4		
			5.0	2.3	0.10	—
Amino glucose	0.5	2.1	0.5	1.4		
			5.0	1.4		
Methyl glucamine	0.5	2.2	0.5	0.9		
Methyl salicylate	0.2	1.0			0.1	0
K_2HPO_4			10	0.8	1.0	+
"MEH"	0.2	1.4	0.2	1.9		
	0.5	2.0	0.5	2.2	0.05	—
Sarcosine	0.2	3.1	0.2	0.8	0.5	0
					sprayed	
Cysteine	0.2	1.2	0.2	1.8		
hydrochloride			0.5	4.6		
Hydroquinone					3.0	++
					sprayed	
					0.5	+
Piperidine	0.2	4.1	0.2	1.2	0.1	+
	0.3	6.4	0.5	2.2		
Piperazine	0.2	4.7	0.2	0.8	0.05	0
Pyrrole	0.2	1.6			0.05	+
					sprayed	
					0.05	+
Diethanolamine	0.2	1.9	0.2	0.8	0.5	0
	0.3	3.5			sprayed	
Di-isopropanol amine	0.2	4.0	0.2	0.7		

† Protection Factor $= \frac{\text{Induction period of oil treated with antioxygen}}{\text{Induction period of untreated oil}}$

— = pro-oxygenic effect
0 = no effect
+ = protection factor between 1 and 2
++ = protection factor between 2 and 3
* = Concentrations on the basis of dry oil
** = Concentrations on the basis of finished flakes

Lecithin is an exception, but its failure may be attributed to the relatively high temperature employed in the test, since it is known that lecithin is unstable at such temperatures.

The failure of some of these compounds to exert their protective effects in finished flakes might be attributed to the low oil solubility of the majority of these compounds, resulting in an unfavorable distribution between the fat components and the rest of the cereal product. In order to study this matter of phase distribution, a test procedure was set up in which a portion of wheat germ oil, treated with the material under investigation was shaken with a volume of dilute salt brine such that the relative amounts of salt, water and oil corresponded approximately to their relative distribution in the cooker batch. After shaking, the mixture was subjected to a cooking period of 1½ hours under 15 pounds steam pressure, cooled, shaken again to insure an equilibrium distribution between the oil and water phases. On standing a few minutes the mixture separated into a relatively large layer of brine on which floated a brine-in-oil emulsion. The latter was separated in a separatory funnel and finally clarified in a centrifuge. A portion of the clear oil layer was then subjected to the accelerated oxidation test and its induction period compared with a control portion cooked and separated in the same way. The results of these tests, as listed in Table 2, show that there are, indeed, some otherwise potent antioxygens whose effectiveness is completely nullified and many others whose effectiveness is diminished by selective extraction in the aqueous phase. However, gluco-ascorbic acid and amino glucose present an apparent anomaly in that they show a protective effect on wheat germ oil cooked with an aqueous brine containing them in spite of the fact that both are preferentially water soluble. On the other hand it has been shown that gluco-ascorbic acid is not protective in finished flakes.

A SALT EFFECT REVEALED BY THE BRINE-OIL COOK

In preparing the control samples for the brine-oil cooks described above, it was noted that the stability of wheat germ oil is markedly lowered by the process of shaking and cooking with dilute salt brine. This observation was extended to include brines of varying concentrations and water. The ratio of brine volume to oil volume was also varied from 1:1 to a value of 60:1. The values actually involved in a cooker batch correspond to approximately 2.5% brine and a brine-oil volume ratio of approximately 65:1. The results of these tests, appearing in Table 3 show, first, that as the ratio of brine volume to oil volume increases at the intermediate brine concentrations the wheat germ oil suffers an increasing degree of loss in stability. At a fixed brine-oil ratio of 20:1 or greater, it is clearly apparent that there is a maximum salt effect at a brine concentration in the neighborhood of 5% .These results suggest that there is a natural antioxygen in wheat germ oil which has a preferential solubility in dilute brine, is poorly soluble in concentrated brine, and insoluble in water. Confirming this conclusion it can be shown in some cases that repeated shaking and cooking of a given portion of dilute brine with successive fresh portions of wheat germ oil serves to accumulate in the brine portion a sufficient amount of extracted antioxygen to progressively decrease the magnitude of the salt effect in the successive cooking periods. This effect could be demonstrated on some batches of wheat germ oil but not all, indicating that although the natural antioxygen in all batches of wheat germ oil is soluble in dilute brine it apparently cannot persist in the brine layer throughout successive cooks equally well for all batches of wheat germ oil. On the assumption that a natural antioxygen which has a measurable

solubility in dilute brine may exist more abundantly in the non-fatty portions of the grain than in the fatty oil itself, brine extracts were prepared from whole wheat germ and whole wheat bran. These extracts, when clarified by centrifuging and cooked with wheat germ oil, showed that they had, indeed, extracted from the germ and bran respectively, sufficient quantities of antioxygen to largely offset the salt effect. Once more these results suggested a possible practical application whereby a brine extract of commercial wheat bran or wheat germ could be substituted in the cooker batch for salt and water. Unfortunately, experimental batches of flakes prepared according to this procedure failed to show any significant increase in stability of the product resulting from the presence of the brine extracts of germ or bran.

TABLE 3

Effect of Brine Concentration and Brine Volume on Stability of Wheat Germ Oil Cooked With It At 15 Pounds Steam Pressure For 1.5 Hours

Oil Batch	Brine Concentration	Brine/Oil Volume Ratio	90° Induction Period
	%		hrs.
	—	Dry oil, control	18.0
I	0 (water)	1:1	21.6
	4.8	1:1	23.3
	9.1	1:1	21.8
	20	1:1	21.2
	saturated	1:1	21.2
	—	Dry oil, control	12.8
B	0 (water)	20:1	10.2
	4.8	20:1	1.9
	9.1	20:1	2.9
	20	20:1	4.6
	saturated	20:1	7.7
	—	Dry oil, control	7.9
J-2	0 (water)	60:1	7.6
	2.4	60:1	3.0
	4.8	60:1	1.9
	10	60:1	8.6

THEORETICAL CONSIDERATIONS

To recapitulate, it has been shown that the significant factors affecting the tendency toward rancidity development in cereal flakes are: The moisture content; the presence or absence of salt; and the physical effects of flaking. These effects have been consistently demonstrated in all cases, whereas, the incorporation of antioxygens of demonstrated potency has been uniformly without significant effect. One of the first hypotheses which were advanced to account for the observed facts was to the effect that the natural or added antioxygens are adversely distributed between the oil and non-oil phases as a result of the solvent action of the brine present in the cooker. Facts which support this hypothesis are the demonstration that wheat germ oil cooked with brine is far less stable than when cooked with water and the fact that the majority of the added antioxygens selected from among those successfully used in salad oils and shortenings are predominantly water-soluble compounds. Opposed to this hypothesis are the following facts: To the extent that dilute brine serves to segregate antioxygens from the oil phase during the cooking operation, the subsequent drying and toasting should restore the initial distribution in the substantially dry end product; flakes can be cooked without salt, and thus, without the adverse distribution postulated, yet the addition of salt by spraying on the finished product is just as effective in reducing the stability of the product as if the salt had been added in the cooker batch; brine extracts of wheat germ and of wheat bran when added to the cooker batch failed to upset the postulated unfavorable distribution in the way to be anticipated from the law of mass action; finally the extraction of finished flakes with hexane yields an oil of unusually high stability. To be sure, the hexane may bring together in solution an unstable extruded oil fraction and natural antioxygens which are not necessarily present in the same phase in the flake structure before treating with hexane. However, it was observed that homogenization of the oil fraction by steeping flakes in hexane, followed by its subsequent evaporation, has failed to confer any greater stability on the product than is found in a control portion not so steeped. The inference from this test is that the extruded oil and natural antioxygen are probably not segregated in the finished flakes.

An alternative hypothesis is that the oil in a wheat flake at the higher moisture levels may be present as a stable emulsion. Such an emulsion would, of course, tend to be broken by the incorporation of salt or by drying to lower moisture levels, both of which conditions are known to be associated with lower stability. However, this hypothesis cannot account for the fact that dry flakes of low inherent stability can be restored to a comparatively stable condition by humidification to a higher moisture level. It is difficult to conceive of a separate oil phase being restored to an emulsion by merely raising the moisture content of the system. Furthermore, there is no evidence to indicate that moisture as it exists in such a cereal product is capable of forming emulsions at all. Reporting on studies made on the respiration of raw wheat kernels Gortner (4) states, "It appears probable that at or close to 14.75% moisture all or nearly all the water is bound in the wheat kernel and that appreciable amounts of free water are present only when the moisture content exceeds 14%." Referring to experiments on the viability of wheat kernels after freezing with moisture contents above and below 50.6% Gortner comments, "It appears probable that a large part of the water represented by the 50.6% moisture content is in a bound form." Both of the above quotations refer to raw wheat but cryoscopic measurements of Kruyt and Winkler (6) showed that starch which had been gelatinized by cooking for one hour at 120° C., held approximately 0.80 grams of bound water per gram of starch. On this basis for raw wheat with a starch content of 60%, the moisture bound by the starch in cooking amounts to 32% of the cooked grain. From the foregoing, it is clearly evident that at the moisture levels with which we are dealing in cereal flakes, there can be no free water. As defined by its method of measurement, bound water is water which is incapable of exerting solvent action on any soluble solutes which may be present in the system.

A third hypothesis is based on the assumption that adsorption of the wheat oil on the surface of salt crystals predisposes the oil to a rapid development of rancidity. Although it has just been pointed out that the moisture present in cereal flakes even at the higher moisture levels cannot conceivably exert any solvent action on the salt crystals, it may, nevertheless, be pictured as forming a preferentially adsorbed film, thus displacing the oil from the salt surfaces. According to this hypothesis, at the lower moisture level the bound water is held primarily in the fiber and gelatinized starch, leaving the salt surfaces substantially dry.

Still another hypothesis to account for the protective effect of moisture is the one suggested by Holm and Greenbank (5) to the effect that "In the absence of moisture the autoxidation proceeds to the aldehyde stage, while when moisture is present it proceeds directly to the acid stage, thus giving little of the tallowy odor produced by the aldehydes and the other by-

products." This idea is in harmony with the observation by Fine and Olsen (3) that a cereal product which had gone rancid at a level of 2.7% moisture, when rehydrated to the levels of 7.6 and 8.3% moisture had lost all trace of rancidity after a subsequent storage interval. This experience has been repeated and confirmed very recently by the author in connection with another flaked wheat cereal.

The last two hypotheses are in fairly complete harmony with the facts relating to the effects of salt and moisture but they offer no explanation for the failure of the added antioxygens so far tried. This failure might be accounted for on the basis that, as pointed out above, wheat bran oil and the oil extract of whole wheat are both remarkably stable, consequently the addition of more antioxygenic material might well fail to be reflected in any measurable increase in stability. Alternately, it might be postulated that added antioxygens, which necessarily contain one or more highly reactive groups, are altered in the cooker batch by reaction with other components of the grain such for example, as partially hydrolyzed protein, carbohydrates, etc.

In conclusion, it is apparent that a great deal more work must be done before a consistent and clear-cut picture of the mechanism whereby cereal flakes become rancid can be presented. However, it is felt that the work which has already been done demonstrates that this problem is in quite a different category from that confronting the manufacturer of shortening or salad oils. The specifications for antioxygens which can be expected to function satisfactorily in the latter cases are fairly well known. On the other hand it appears that a solution of the rancidity problem in cereal flakes must rest more heavily on a study of the colloidal or physical aspects of the flake surface than on a strictly chemical consideration of autoxidation as influenced by antioxygens.

It is not intended to present this paper as a finished piece of research in any sense, since there are many gaps to be filled in the data presented. The justification for presenting this review is in the hope of stimulating interest in a less familiar aspect of the rancidity problem.

REFERENCES

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Uniform Methods and Planning Committee Report

Fall Meeting — October 2, 3 and 4, 1940

THERE were only two reports submitted to the Uniform Methods and Planning Committee for action at this time. One was received from the Glycerine Analysis Committee and this merely reported progress, so that no action was required.

The Soap Analysis Committee make the following recommendations, which are concurred in by the Uniform Methods and Planning Committee:

"(1) Pyrophosphate in soap—No recommendations for official action. Further studies to be undertaken.

(2) Combined CO_2 in soap—Recommend tentative adoption of Evolution-Volumetric method. Retain present official absorption method (A.O.C.S.) as alternate.

(3) Free alkali in potash soaps—Studies to be undertaken using isopropyl alcohol as solvent instead of ethyl alcohol.

(4) McNicoll method for rosin in soap—Recommend official adoption of this method and deletion of present Wolff method."

These recommendations were voted upon by the Society and adopted.

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