

# A Wide-Plastic Range Spread for the Canadian Armed Forces<sup>1</sup>

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BUTTER AND MARGARINE are very hard at sub-zero temperatures and very soft at warm temperatures. Therefore they are not entirely suitable as components of ration packs because the Canadian armed services have to operate in the low temperatures encountered in the Arctic regions and in the warm temperatures encountered in summer. For biscuits and bread, a spread of high chemical and physical stability that has a plastic range over all Canadian temperatures is needed.

Wide-plastic-range spreads can be made by blending high and low melting components. Jones, Dutton, and Cowan developed such a spread containing a liquid vegetable oil and a saturated monoglyceride (9). In later papers pilot-plant production and the beneficial effect of tempering the spread on its consistency and "get-away" in the mouth were described (7, 10).

Feuge, Gros, and Vicknair found that a material having a wide plastic range was obtained by blending liquid acetoglycerides with hard fat (5). Gros and Feuge reported in a later paper that margarine-like products of wide plastic range can be prepared by acetylating lard, cottonseed, soybean, or peanut oils to an acetyl content ranging from 12–20% and blending with 18.5% of hydrogenated cottonseed oil. As the acetyl content of the acetoglyceride increased, the consistency characteristics of the mixtures improved (6).

Baur has described the preparation, properties, and uses of acetin fats. The most striking effect of the introduction of the acetyl group into the glyceride molecule is the lowering of the melting point, permitting the preparation of low-melting-point fats and oils of a high degree of saturation and a significantly increased oxidative stability (3).

The objective of the present work was to develop a formula for an anhydrous spread of wide plastic range, utilizing Canadian oils. A spread of the saturated monoglyceride-cottonseed oil type of Jones, Dutton, and Cowan (9) was tried first. The plastic qualities of this spread were good, but some trouble with flavor deterioration was experienced. Then too cottonseed oil is not a Canadian oil. Soybean oil is produced in Canada, but it did not seem advisable to substitute it for the cottonseed oil owing to its tendency to flavor deterioration.

This study therefore was concerned with the acetoglyceride type of spread of Feuge, Gros, and Vicknair (5). Canadian oils can be utilized since a stable liquid oil can be made by acetylating hydrogenated soybean oil of a very low linolenic acid content.

## Materials and Methods

Acetoglycerides were prepared from several oils by the interesterification reaction described by Baur (3). A large excess of triacetin (six moles to one mole of

oil) was used to insure the production of a high concentration of diacetoglycerides. The mixture of triacetin and oil was dried by heating to 105°C. under vacuum with a little dry nitrogen bubbling through. After 1 hr. the mixture was cooled to 60°C., and the vacuum was reduced to atmospheric pressure with nitrogen. An amount of finely powdered sodium methoxide equal to 0.2% of the weight of the oil was added in a fine stream from a funnel to the agitated mixture with nitrogen bubbling through it. Usually the mixture became homogeneous within a few minutes. After 45 min. a slight excess of dilute phosphoric acid was added to neutralize the catalyst. Excess acid was removed by washing with a 15% sodium chloride solution. After drying, bleaching, and filtering, the excess triacetin was removed by vacuum distillation. Finally the acetoglyceride was steam-deodorized.

The acetoglycerides used were made from lard, butterfat, cottonseed oil, hydrogenated soybean oil of iodine value 70, and hydrogenated peanut oil of iodine value 60. All were liquid oils at room temperature. The acetyl contents were 85% of the theoretical values for diacetoglycerides. The linolenic acid content of the hydrogenated soybean oil of I.V. 70 was 0.5%, determined by the method of Brice *et al.* (4).

Gros and Feuge found that, by decreasing the degree of hydrogenation of the hard fat component, an acetoglyceride spread softer at all temperatures could be produced (6). Since they found that hydrogenated cottonseed oil should have an iodine value lower than 55 and that variations in iodine value from 1 to 29 did not have a great influence on the consistency-temperature relationship, for the present study cottonseed oil was hydrogenated to an intermediate iodine value of 37.3 and melting point of 54°C. Other hard fats used were hydrogenated soybean oil of iodine value 41.2 and hydrogenated beef tallow.

The other ingredients of the spreads were commercial fluidized lecithin, antioxidant,  $\beta$ -carotene, artificial flavor, and a pure grade of salt pulverized in a hammer mill.

Spread mixtures were solidified in the laboratory by two methods:

a) About one pound of melted spread at a temperature of 50°C. was poured into the large bowl of a Hobart C-100 Mixer immersed in water at 10°C. to a depth of 3½ in. The spread was agitated at medium speed for 4 min., scraped down, and agitated for 10 min. at low speed. Salt, flavor, color, etc., were incorporated during the second mixing. Nitrogen was blown over the spread during the operation.

b) A large cake tin was chilled to about -20°C. by floating it on an ice-and-salt freezing mixture, or to -65°C. by utilizing a dry ice-acetone mixture. The melted spread was poured into the tin in such a manner as to cover the bottom of the tin with a layer thin enough to solidify quickly. The other ingredients were blended with the solidified fat in the Hobart Mixer at room temperature.

In the Canada Packers pilot plant, acetoglycerides were prepared in a tank that could be operated under vacuum and that was equipped with an agitator and steam coil. The product was washed with brine, dried,

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and bleached in the same tank. Excess triacetin was removed, and the acetoglyceride was deodorized in a Girdler 400-lb.-batch pilot plant deodorizer. No attempt was made to recover the excess triacetin. The melted spread with salt and other ingredients added was solidified in a Girdler 400-lb.-per-hour pilot-plant Votator.

Plasticity was measured by means of a grease penetrometer, the weight of the cone and spindle being 50 g. Solid fat indices were determined dilatometrically by a method similar to the A.O.C.S. Tentative Method Cd 10-57.

### Experimental

The first spreads contained 20 parts of the hard hydrogenated cottonseed oil, 79 parts of the various acetoglycerides, and 0.5 parts of fluidized lecithin. They were solidified by the Hobart Mixer method already described.

Although all of these were spreadable over a wider range than butter or margarine, those containing butter and lard acetoglycerides had a poor flavor and were eliminated. As cottonseed and peanut oils are not produced in Canada, it was decided to use hydrogenated soybean oil acetoglyceride. For the same reason the substitution of hydrogenated soybean oil of iodine value 41.2 and melting point 58.3°C. for hydrogenated cottonseed oil was tried.

It was observed that the spreads were quite sloppy after the mixing period in the Hobart Mixer. If put into a refrigerator directly, they hardened but became quite sloppy again when warmed to room temperature. If they were allowed to temper at room temperature for 24-48 hrs. however, they became firmer, with a more stable consistency.

To determine the time required for tempering, a spread containing 78 parts of hydrogenated soybean oil acetoglyceride (I.V. 51.2), 20 parts of hydrogenated soybean oil (I.V. 41.2), and 0.5 parts of fluidized lecithin was solidified in the Hobart Mixer and poured into a number of jars which were at once put into storage at 4°C. After one week they were transferred in pairs to a 21°C. temperature. Penetration readings were made on the sample pairs in the jars at intervals by using the grease penetrometer. There was a pair of jars for each time interval. The average penetration for each pair is given in Table I. The initial penetrations were quite high. After 4 hrs. at 21°C. the spreads were pourable. After 14 hrs. they were obviously firmer and would not pour. Maximum firmness occurred at 32 and 48 hrs.

To study the effect of tempering on plastic range, untempered spreads, spreads tempered for 48 hrs. at 21°C., and, for comparison, a commercial oleomargarine were stored at -20°, 3°, 20°, and 33°C. for

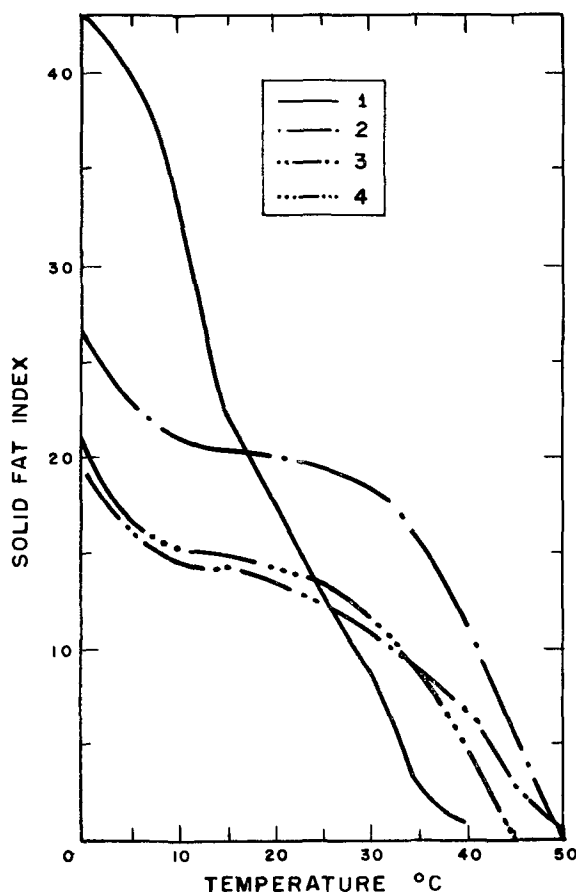


FIG. 1. Relationship between solid fat indices and temperature for 1. margarine, 2. the first pilot-plant spread, 3. a laboratory spread containing hard hydrogenated cottonseed oil and hydrogenated soybean oil acetoglyceride, and 4. the second pilot-plant spread.

24 hrs. The results of penetration measurements on these spreads are given in Table II. The tempered spreads had the widest plastic range.

Tempered laboratory spreads containing hard hydrogenated soybean oil and hydrogenated soybean oil acetoglyceride, together with 1% pulverized salt and artificial flavor, were reasonably satisfactory both in plastic range and organoleptic acceptability.

TABLE II  
Plastic Range of Tempered and Untempered Acetoglyceride Spread

Temp. °C.	Penetration (m.m.)		
	Untempered	Tempered	Margarine
-20.....	2.7	3.3	1.4
3.....	34.7	18.6	3.4
20.....	too soft	19.6	10.4
33.....	too soft	23.8	too soft

TABLE I  
Effect of Tempering at 21°C. on the Consistency of  
Acetoglyceride Spreads

Time at 21°C. (hours)	Penetration (m.m.)
0.....	34.7
4.....	too soft
6.....	37.0
14.....	19.3
16.....	21.0
24.....	20.8
32.....	15.8
48.....	15.8
64.....	20.2
126.....	18.7

At this time a pilot-plant preparation of the spread was attempted. The necessary acetoglyceride was prepared from hydrogenated soybean oil of iodine value 69.1 and had an iodine value of 50.9 and a saponification value of 340. A quantity of soybean oil was hydrogenated to iodine value 41.4 for use as the hard component. A blend of 20 parts of the hard fat and 78.5 parts of the acetoglyceride was deodorized for 4 hrs.; the maximum temperature was 400°F. This treatment caused the loss of some of the acetoglyceride by distillation. While the melted

blend was agitated in the feed-tank of the Votator, 0.5% lecithin,  $\beta$ -carotene, flavor, propyl gallate, citric acid, and 1% pulverized reagent grade salt were added. Nitrogen was incorporated into the spread during solidification in the Votator, and the product was canned in an atmosphere of nitrogen. The cans were tempered at 70°F. for 48 hrs. and were then refrigerated.

This spread proved to be unsatisfactory. It was firm when taken from a refrigerator, but it became quite sloppy when it had warmed to room temperature. It did not become much firmer on further tempering. Its solid fat indices over the temperature range of 0°–50°C. were higher than those of laboratory spreads, probably because of the loss of acetoglycerides on deodorizing (Figure 1). The sloppy consistency was undoubtedly caused by the coarse crystalline structure. Microscopic examination revealed masses and clusters of large crystals, probably of tristearin from the hard hydrogenated soybean oil.

The unsatisfactory characteristics of the first pilot-plant spread necessitated further consideration of solidification and of the solid component of the spread.

There was some indication that the lecithin in the acetoglyceride spreads affected crystallization, and this was investigated first. Blends of 78.5 parts of hydrogenated soybean oil acetoglyceride and 20 parts of hydrogenated soybean oil (I.V. 41.2) were made up with the lecithin content varying from 0 to 0.8%. Similar blends were made, using 20 parts of hydrogenated cottonseed oil (I.V. 37.3). All of these were allowed to solidify at room temperature. At first the lecithin-containing blends were harder than those without lecithin. After a few days solid and liquid phases separated in the blends containing hydrogenated soybean oil with 0.4 and 0.8% lecithin, and later in those blends with lower lecithin contents. No such separation occurred in any of the blends containing hydrogenated cottonseed oil. Microscopic examination of the solid phase from the hydrogenated soybean oil spreads containing lecithin revealed the presence of large, almost spherical clusters of needle-like crystals, probably of tristearin. The blends without lecithin contained many needle-like crystals, but they were not in clusters. While the hydrogenated cottonseed oil spreads did not have a fine structure, there were few of the needle-like crystals.

All of the above blends were melted, then solidified quickly by pouring into a cake tin floating on a dry ice-acetone bath. After the solidified blends had warmed to room temperature, they were stirred until a homogeneous plastic mass was obtained. They were allowed to stand first at room temperature, then at 30°C. for several days. No separation into solid and liquid phases took place in any blend. Microscopic examination showed that the crystalline structure was very fine. This experiment indicated that the effect of lecithin on the crystallization of solid fat from spreads containing hard hydrogenated soybean oil depends on the temperature of solidification.

Other experiments indicated that lecithin was necessary for the proper tempering of spreads solidified in the Hobart Mixer at 10°C. Blends containing 0.5% lecithin reached maximum firmness after about 30 hrs. of tempering at 21°C. while blends without lecithin became firmer very slowly over a period of 10 days. However when these blends were solidified by the cake-tin method at about -65°C., lecithin had

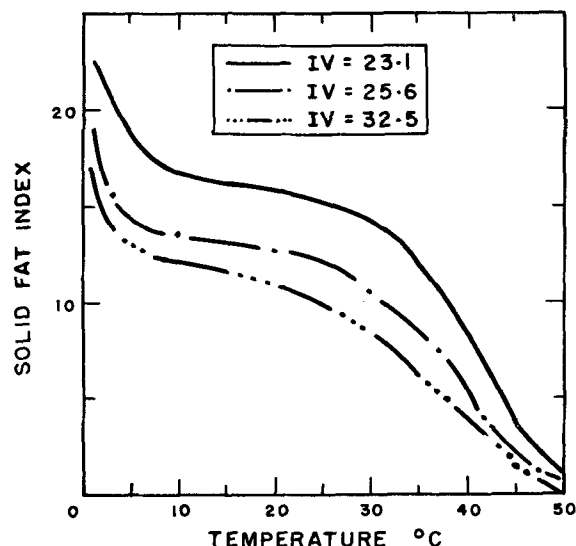


FIG. 2. Effect of the hardness of hydrogenated tallow on the relationship between solid fat indices and temperature for hydrogenated tallow-hydrogenated soybean oil acetoglyceride spreads. Iodine values of hydrogenated tallow were (1) 23.1, (2) 25.6, (3) 32.5.

no effect on tempering. In fact, spreads solidified in this manner and stored at 4°C. without tempering did not become sloppy when warmed to room temperature as did those solidified at 10°C. Furthermore the spreads solidified at the low temperature had a fine microscopic structure, similar to that of a commercial margarine, whereas the spreads solidified by the Hobart Mixer method were coarser in structure. The former however did not have as good a mouth-feel as those solidified at 10°C. Apparently lecithin has little effect on the properties of the spread if it is solidified quickly enough to achieve a very fine crystalline structure.

It was decided to adopt the cake-tin method of solidification in the remaining laboratory experiments. It was found however that a temperature of -20°C., achieved by floating the cake-tin on an ice-salt mixture, was sufficiently low to give a product with a fine structure. It was felt that the poorer mouth-feel of spreads solidified at a low temperature might be improved by lowering the melting point of the spread.

Although part of the trouble with the first pilot-plant spread may have been caused by the loss of acetoglyceride on deodorizing referred to previously, and to the high melting point of the hydrogenated soybean oil, it was felt that it would be safer to use a hard fat with a tristearin content as low as that found in hydrogenated cottonseed oil. Hydrogenated beef tallow, readily available in Canada, was suggested as a substitute for hydrogenated cottonseed oil if the latter were not available. Tallow has an even higher content of palmitic and myristic acids than cottonseed oil. As little hydrogenation is required to get a product hard enough for an acetoglyceride spread, it should contain even less tristearin than the hydrogenated cottonseed oil.

For the remainder of the investigation, spreads were compared by determining dilatometrically the solid fat indices over a range of temperatures. In Figure 1 solid fat index curves are compared for a commercial margarine, the first pilot-plant spread, and a laboratory spread containing hydrogenated

cottonseed oil, I.V. 37.3 as the solid component. The curve for the margarine is markedly different from those for the wide plastic range spreads.

It was found that the solid fat index curve of a spread can be varied at will over the range 0° to 45°C. by varying the melting point of the hard component, the degree of saturation of the hydrogenated soybean oil acetoglyceride, and the ratio of solid fat to acetoglyceride. As the spread containing hydrogenated cottonseed oil as the hard component seemed to be satisfactory, an attempt was made to prepare a spread of similar solid fat index-temperature relationship by using hydrogenated beef tallow as the solid component. Hydrogenated tallow of various iodine values was blended with the regular hydrogenated soybean oil acetoglyceride. Three of these spreads are compared in Figure 2. The solid fat index curve for Spread No. 2 resembled that of the hydrogenated cottonseed spread most closely. It had a solid fat index greater than 5 at 40°C., which is considerably higher than that of a commercial margarine at this temperature. One would expect therefore that the spread would not melt in the mouth quite as readily as margarine or butter. This could be remedied easily by using less hydrogenated tallow, but there would be greater danger of the spread melting or separating at warm temperatures, with subsequent settling out of salt. Laboratory spreads with the same composition as Spread No. 2, solidified by the cake-tin procedure, seemed to be quite satisfactory in respect to low temperature spreadability and general acceptability.

A second pilot-plant preparation was now made. The hydrogenated tallow used for the hard component had an iodine value of 28.6 and a capillary melting point of 53.5°C. The acetoglyceride prepared from hydrogenated soybean oil of iodine value 71.4 had a saponification value of 350 and iodine value of 52.0. This time the hard fat and the acetoglyceride were deodorized separately in order to reduce the loss of diacetoglyceride.

To test the ingredients, solid fat indices were determined on two blends of the hydrogenated tallow and acetoglyceride, a) in the ratios of 19.2 to 75.2 parts and b) in the ratio of 19.2 to 79 parts. The solid fat indices were as follows:

	0°	5°	10°	15°	20°	25°	30°	35°	40°	45°C.
Blend a)	21.1	17.4	16.1	15.3	14.7	14.0	12.3	9.1	5.5	0.7
Blend b)	20.3	16.6	15.3	14.7	13.9	13.3	11.8	8.6	5.0	0.5

The difference between these spreads was slight; but as blend b) more closely resembled the hydrogenated cottonseed spread, this ratio was adopted for the pilot-plant spread. The formula was as follows:

- 38.4 lbs. of deodorized hydrogenated beef tallow, I.V. 28.6
- 158 lbs. of acetoglyceride, I.V. 52.0
- 0.5 lbs. of fluidized lecithin
- 2.5 lbs. of pulverized pure sodium chloride

$\beta$ -carotene, commercial antioxidant, and flavor were added.

The agitated blend was kept at 120°F. in the feed-tank and was pumped into the Votator, where nitrogen was incorporated. The solidified product was canned in a nitrogen atmosphere and tempered at 70°F.

The solid fat index-temperature curve for the

second pilot-plant spread is No. 4, Figure 1, and is quite close to that of the hydrogenated cottonseed oil spread.

The consistency of this spread was excellent, and the spreadability was good at temperatures as low as -10°F. and up to 100°F. When tested by the Active Oxygen Method, the product had not noticeably deteriorated after 165 hrs. at 97.7°C. Long-term storage tests have been started.

### Discussion

The hydrogenated tallow-hydrogenated soybean oil acetoglyceride spread appears to be quite acceptable for the purpose for which it is intended. The use of acetoglycerides for edible purposes however has not yet been approved.

The physiological properties of the acetoglycerides have been studied fairly extensively by Ambrose and Robbins, Herting *et al.* and Mattson *et al.* (2, 8, 11). This work has been reviewed recently by Alfin-Slater *et al.* (1). They concluded from work reported up to date that acetoglycerides have no deleterious effect on growth or on reproduction and that the mode of digestion of aceto-oleins and acetostearins does not differ appreciably from that of the corresponding natural fats. As to over-all digestibility, that of aceto-oleins appears to be equal to that of the corresponding normal triglycerides whereas the digestibility of acetostearins is markedly better than the absorption of the normal saturated glycerides. On the basis of these results it may be possible that sufficient data will be forthcoming to qualify the acetoglycerides for use with foods.

### Summary

An acetoglyceride type of spread having a wide plastic range has been developed, utilizing hydrogenated edible tallow for the hard component and acetoglycerides made from hydrogenated soybean oil for the liquid component of the spread. The product had good spreadability over a wide temperature range and good acceptability.

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## Carboxymethylated Soybean Protein<sup>1</sup>

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TWO MAJOR PROBLEMS in the industrial utilization of vegetable proteins involve their dispersibility at a neutral pH and stability to putrefaction. Soybean protein can be modified during its isolation from hexane-extracted flakes to give a product of improved solubility at a neutral pH by treating the meal extract with alkali at pH 11-12 in the presence of sulfite and lime, heating for 0.5 to 2 hrs. at 50°-70°C., and clarifying the dispersion prior to precipitating with acid at pH 4.5-4.8 (2, 5, 12). Besides bleaching, the sulfite reduces the disulfide bonds, which are then readily attacked by the alkali (7, 9), and eliminates the possibility of decreased solubility because of polymerization through disulfide bond formation. At pH 11-12 phytate is dissociated and insolubilized by the lime, hence it may be removed by clarification (5). Removal of the phytate has been shown to improve solubility of the protein (5, 8). In addition, the mild hydrolytic treatment converts a portion of the nonpeptide amide groups of asparagine and glutamine to carboxyl groups; this allows dispersion of the protein at a lower pH.

Ten per cent of the total nitrogen of isolated soybean protein has been shown to be nonpeptide amide nitrogen (6). Calculations based on nonpeptide amide nitrogen and the reported (11) dicarboxylic acid content of acid-precipitated soybean protein indicate that about 50% exists as asparagine and glutamine residues. That increased solubility of proteins at lower pH values can be achieved by hydrolysis of nonpeptide amide groups to carboxylic acids is shown by experiments on zein (4). Zein, which is insoluble in water below pH 11, was rendered soluble at pH 6-7 when about 40% of the nonpeptide amide groups was hydrolyzed to carboxylic acids.

This paper describes the reaction of soybean protein with sodium chloroacetate to produce a protein derivative containing added carboxyl groups, resulting in increased solubility of pH 6-7 and in resistance to putrefaction; dispersions of the derivative do not gel on adding formaldehyde. The purpose of this study was to determine whether soybean protein would react with sodium chloroacetate in an alkaline medium sufficiently mild not to expect extensive alkali degradation of the protein, to determine the extent of the reaction, if any, and to determine whether addition of carboxymethyl groups to the protein would allow increased solubility at a lower pH than the starting protein does. No attempt was made to carry out an extensive study of the reaction conditions to produce

a series of proteins with varying carboxymethyl content for study and evaluation. Such a study should be made on the wet protein curd obtained during its manufacture to avoid drying twice.

### Preparation of Carboxymethylated Protein

**Materials Used.** Protein A was commercial Alpha Protein purchased from the Glidden Company.<sup>3</sup> *Analyses.* P, 0.19%; total N, 15.0; amino nitrogen (Van Slyke), 0.60%; ultracentrifugal analysis: S<sub>20</sub>, 40% at 1.10, 60% at 3.93.

Protein B was prepared in the pilot plant from undenatured hexane-extracted flakes by extracting with lime at pH 9.6 in the presence of 0.1% sodium sulfite at 50°C. The extract was clarified by centrifugation, and the protein was precipitated from the clarified extract by adjusting the pH to 4.2 with sulfur dioxide. *Analyses.* P, 0.79%; total N, 15.5%; amino nitrogen (Van Slyke), 0.63%; neut. equiv. to pH 9.5, 1430 (dry basis); ultracentrifugal analysis: S<sub>20</sub>, 2.41.

**Reaction with Sodium Chloroacetate.** Data for the reaction and isolation of two preparations are shown in Table I. A control for Preparation 1 is included

TABLE I  
Preparation and Analyses of Carboxymethylated Soybean Protein

Preparation steps and analyses	Preparation 1	Control for 1	Preparation 2
Preparation, 100 g. protein:			
Source of protein.....	Protein A	Protein A	Protein B
pH of dispersion.....	10	10	10
Protein conc., %.....	14	14	12.5
ClCH <sub>2</sub> COONa, moles.....	0.1875	0	0.224
Temperature, °C.....	70	70	50
Time, hrs.....	5.5	5.5	10
NaOH used, moles <sup>a</sup> .....	0.139	0	0.150
pH of min. soly.....	3.1	4.5	3.1
Pptd. yield, %.....	86	89	90
Dialysis loss, %.....	12	12	10
Analyses: <sup>b</sup>			
Total N, %.....	13.9	15.0	14.1
Amino N, %.....	0.07	0.60	0.08
-CH <sub>2</sub> COOH, % <sup>c</sup> .....	7.3	0	9.0
Neut. equiv. to pH 9.5.....	590	1250	830
Ultracentrifugal analysis, S <sub>20</sub> .....	1.35	....	2.59

<sup>a</sup> Moles of alkali required to maintain pH 10 indicate chloroacetate reacted.

<sup>b</sup> Analyses on dialyzed product, dry basis.

<sup>c</sup> Calcd. from difference in N content.

in which the protein was carried through all of the reaction steps without the addition of sodium chloroacetate. No control was carried out for Preparation 2 because this reaction was run to compare the reacted with the unreacted protein.

<sup>1</sup> Presented at fall meeting, American Oil Chemists' Society, Cincinnati, O., September 30-October 2, 1957.

<sup>2</sup> Present address: Victor Chemical Works, Chicago, Ill.

<sup>3</sup> Mention of this company and its products does not constitute preference by the U. S. Department of Agriculture over similar products manufactured by other companies.