

located in the urban areas (more than 15,000 population) although, no doubt, some will be found in smaller communities. Using an average family size of $3\frac{1}{2}$ members, the home softeners will affect approximately 12 million people or, in other words, 7% of the total population. Assuming that the softeners are installed whenever the water is 200-p.p.m. hardness and over, this will enable us to raise the accumulated total below this hardness by 7%. Therefore, going back to the original distribution in Figure 2, we can conclude that 87% of the total population will use water of less than 200 p.p.m. hardness and that 97% of the population, on the average, will use water under 250-p.p.m. hardness in their homes. This makes the approach toward a universal product very promising.

In the event that it would be desirable to use the two-product approach, the states listed in Table II would be singled out for distribution of a soft-water product which would be necessary in water under 100-p.p.m. hardness. The balance of the states would find the hard-water product satisfactory.

Conclusion

A study has been made of the hardness of the water throughout the United States in regard to its distribution by states and by total population. The

purpose was twofold: a) to determine the range of performance necessary for a soap or synthetic detergent product in order to be satisfactory to the majority of the population and b) to determine the areas of distribution for products of varying performance characteristics in respect to water hardness. The study has taken into consideration municipal water-treatment for the urban population, the distribution of rural population, and the distribution of home water-softeners. The mean water-hardness found in the United States, ignoring the home softening-units, was estimated to be 136.6 p.p.m. with a standard deviation of 90.9 p.p.m. Twenty-one states, including the District of Columbia, were found to have a weighted average hardness under 100 p.p.m. In general, the hardest natural water is found in a narrow belt covering the states of South Dakota, Iowa, Illinois, Indiana, and Ohio.

Acknowledgment

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Bulk Sampling of Soybean Oil Meal

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DRAWING A SAMPLE from a carload of meal which will reflect the true composition of the car is a problem with which every processor is faced daily. Further it is a problem of wide applicability to industries dealing in bulk materials such as feeds, fertilizers, and chemicals.

This study was made to determine whether a continuous flow sample taken during the loading of the car is as representative of the contents as the official loading sample, which is taken by probing after loading. This study was conducted as a preliminary to a more detailed study which should be made to assess the variability of sampling and the degree of stratification, if any, which exists in a bulk car. From such a study minimum sample sizes and the most economic sampling method could be determined.

Sampling Methods. The official method of sampling soybean meal has been designated by the National Soybean Processors Association (1).

The main features of Chapter I of the Grain Inspector's Manual (revised, effective July 1, 1942) provide that for sampling bulk shipments the sample shall be taken with a standard double tube, 11-compartment bulk grain probe. At least five probes must be taken in different sections of the car as follows:

- (1) probe in center of the car;
- (2) probe from 2 to 4 ft. back from the doorpost toward the end of car and approximately 2 ft. out from one side of the car;
- (3) probe from 2 to 4 ft. from same end of the car

and approximately 2 ft. from the opposite side of the car as in (2);

- (4) and (5) probe same as in (2) and (3) in opposite ends and sides of the car.

The probe shall be inserted at an angle of about 10 degrees from the vertical, with the slots closed. The slots shall be faced up when the probe is opened. While the slots remain open, give the probe about two slight up-and-down motions so that all the openings may be filled, close slots, and withdraw the probe, placing the contents of the probe full length on a sampling cloth.

Individual probe samples shall be inspected to check on uniformity. The individual probe samples are then composited into one sample, representing the entire lot.

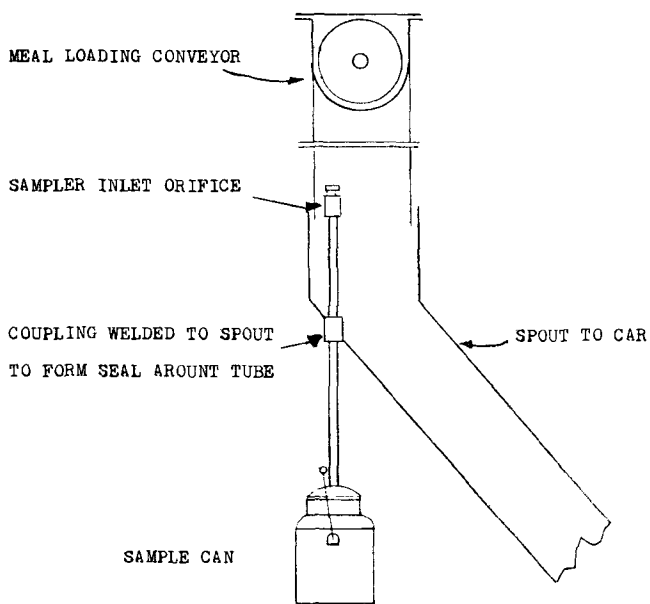
The official method has particular advantage in that it allows the purchaser to sample the car before unloading. Presumably the method should be used by the vendor to draw his sample before shipment. Unfortunately the method is liable to misuse by both vendor and vendee unless the sampler is carefully supervised; the practical difficulties in the use of probes are well known to those with experience in sampling meal cars. A method which is not subject to variation from a human source is desirable. Fortunately the vendor can take a continuous flow sample during the loading of the car which is not subject to the vagaries of human nature.

After some trial and error a system was designed for sampling from the loading spout.

Continuous Flow Method (dock). The sampler is a 1-in. standard pipe centered in the stream of meal as it discharges from the overhead conveyer into the vertical section of the loading spout. The upper end

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50% MEAL LOADING
SAMPLER
DECATUR MILL
RLR NO SCALE

Fig. 1.

of this pipe is equipped with a plug which has a $\frac{3}{16}$ to $\frac{1}{4}$ -in. diameter orifice drilled in it. The orifice is large enough to allow the largest particles to pass easily but not large enough to collect more than about a $1\frac{3}{4}$ -gal. sample. The other end of the pipe has the cover of a 2-gal. cream can welded to it and is equipped with a hook for the bale of the cream can. A schematic drawing is shown in Figure 1.

A sample is taken by hooking a 2-gal. cream can on the discharge of this pipe before loading is started. When loading is complete, the can is removed and capped to prevent moisture loss. The whole sample is later mixed and divided to provide a sample for analysis by the laboratory.

Once we built a sampler which appeared to be satisfactory, tests were made to compare the continuous flow sample to the official probe sample to ascertain whether the continuous sample could be substituted for the official sample.

Experimental Design

The continuous sampler was installed on loading spouts used for discharging 50% protein soybean oil meal to cars. The object of the experiment was to determine whether the protein content of the sample from the continuous sampler was higher than the protein content of the sample drawn by the official method and to determine whether the crude fiber content was lower. The questions were phrased in this somewhat peculiar manner because a negative answer would then provide additional protection against customer complaints and a positive answer would require that further work be done to improve the sampling device.

From previous work it was known that the standard deviation of a replicate test for protein content was $\pm 0.19\%$. The standard deviation between laboratories

based on limited data was $\pm 0.221\%$ protein. No information was available which positively defined the error of the Crude Fiber Test Method on 50% meal. However the American Association of Feed Control Officials (2) reported a value of $\pm 0.34\%$ as the standard deviation between laboratories on 44% meal. Because the average crude fiber content of 50% meal is about one-third that of 44% meal, the standard deviation may be lower for 50% meal.

The experiment was designed a) to detect a bias between the two sampling methods if the bias were equivalent in absolute magnitude to the between-laboratory standard deviation and b) to discover this bias, provided it existed with a probability of 0.95, *i.e.*, 19 in 20. This is, the error of the second kind, or the probability that no difference of this magnitude will be detected when in fact it does exist, was set at 0.05. The error of the first kind, or the probability that a difference of this magnitude will be declared to exist when in fact it does not, was also set at 0.05. With these limitations fixed in advance 23 cars would have to be sampled by both methods (3).

The protein and crude fiber data were converted to a moisture-free basis and analyzed by the paired comparison method, which enables the average for all of the cars by both sampling methods to be compared by using the variability of the difference between the two samples taken from each car as a basis for comparison.

Experimental Data

Twenty-four cars were sampled by both methods during regular production. Nine of these samples were taken in one period and 15 during a later period. Table I shows the original data not corrected for moisture content.

Analysis of Data and Discussion

The data were analyzed and the summary is shown in Table II.

For protein, since calculated value of "t" is less than the limiting value of "t" (from the tables), there is not sufficient evidence to conclude that the continuous flow sample protein content is higher. Since the experiment was designed to be large enough to detect a difference if it existed, it is concluded that no difference of consequence exists; or it was not

TABLE I
Protein and Crude Fiber Obtained on 50% Soybean Oil Meal
Using Two Methods of Sampling

Sample No.	% Moisture		% Protein		% Crude fiber	
	Off. probe	Cont. flow	Off. probe	Cont. flow	Off. probe	Cont. flow
1.....	12.50	12.30	50.05	50.90	2.60	2.80
2.....	12.40	12.25	50.55	50.75	2.65	2.82
3.....	12.50	12.40	50.05	50.15	2.53	2.90
4.....	12.65	12.55	51.00	50.22	2.90	2.94
5.....	12.40	12.45	50.45	51.10	2.57	2.69
6.....	11.75	11.70	50.05	50.50	2.74	2.86
7.....	11.70	11.70	51.00	50.76	2.56	2.60
8.....	12.00	11.95	50.75	50.57	2.43	2.25
9.....	12.50	12.55	50.05	50.57	2.45	2.88
10.....	10.94	10.92	50.87	50.62	2.70	2.82
11.....	11.04	10.90	50.87	51.00	2.71	2.72
12.....	10.76	11.02	51.00	50.75	2.62	2.74
13.....	10.50	11.01	51.25	51.00	2.80	2.91
14.....	11.18	11.81	50.87	50.25	2.44	2.64
15.....	10.70	11.31	50.50	50.50	2.57	2.64
16.....	10.18	10.63	50.87	50.75	2.83	2.76
17.....	10.61	10.68	50.38	51.12	2.67	2.71
18.....	10.55	11.03	51.00	50.75	2.61	2.70
19.....	10.42	10.86	50.50	50.50	2.83	2.76
20.....	10.41	10.63	50.00	50.12	2.82	3.05
21.....	10.80	10.64	50.62	50.38	2.77	3.03
22.....	10.61	10.26	50.75	50.62	3.02	2.97
23.....	10.35	11.02	51.70	50.75	2.92	2.65
24.....	10.64	10.85	51.40	51.40	2.98	2.51

TABLE II
Statistical Analysis of Data on 24 Shipments

Test	Avg. diff. % cont. flow minus off. probe	Standard deviation of diff.	Calculated value of "t"	0.05 Signifi- cance level for "t" @ 24 degrees of freedom
Protein, moisture-free	+0.067	±0.460	+0.714	+1.71 ^a
Crude fiber, moisture-free	+0.080	±0.217	-1.819	+1.71 ^a
Moisture	+0.138	±0.299	±2.26	±2.06

^a Single-tailed test.

found, a possibility which might occur once in 20 attempts. The test which was made is commonly called a one-sided test, meaning that the continuous flow method of sampling would be rejected only if it had a higher protein content of a definite magnitude and the continuous method would be accepted even if it differed markedly on the low side.

For crude fiber, since the calculated value of "t" is less than the limiting value, there is insufficient evidence that the continuous flow sample has a lower crude fiber than the official sample. Therefore the alternate hypothesis, that the crude fiber in the continuous flow sample is not lower than the official method, is accepted. Here again a single-sided test was used, requiring rejection of the continuous method only if the method obtained low values for crude fiber. Since the sampling method leads to obtaining slightly higher values, the continuous flow sample is satisfactory.

The moisture content was run on the samples as a necessary part of reducing the analysis for crude fiber and protein to a dry basis. It was desirable to know whether the moisture content on the continuously drawn sample differed from that of the official sample. Note that the question was "is the moisture different?" The calculated value of "t" is higher than the limiting value from the tables, and the hypothesis that the two methods differ is accepted. The moisture con-

tent is higher on the continuous sample, as would be expected, since this sample is protected from loss by evaporation whereas the probe sample is not as well protected. In this respect this study therefore indicates that the continuous flow sample is a better sample, hence a safeguard against shipment of high-moisture meal.

Summary

In an effort to avoid the operational errors introduced by the human factor in taking official probe samples of meal, a continuous flow sampler was designed and tested in comparison with the official probe sample on 50% Protein Soybean Oil Meal. The protein content was found not to be higher on the continuously taken sample. The crude fiber content was found not to be lower on the continuously taken sample. The moisture content on the continuously taken sample differed from the official probe sample. The difference was on the high side as would be expected from consideration of the degree of exposure to air.

The continuous flow method described does not conform to the tentative A.O.C.S. Committee recommendation on automatic meal sampling, hence has no official standing. Any such installation as that described should be evaluated to reveal whether its operation is satisfactory. An experiment similar to the one described here should be used in such an evaluation.

These conclusions were reached as the result of objective statistical tests of data provided by a soundly designed experiment. The value of statistical planning for an experiment is demonstrated.

REFERENCES

1. National Soybean Processors Association, "Year Book and Trading Rules," 1956-1957, p. 30.
2. Law, T. C., *et al.*, Report of Collaborative Check Sample Committee," Bulletin of Feed Control Officials, 1956, p. 58-60.
3. Davies, O. L., "The Design and Analysis of Industrial Experiments," New York: Hafner, 1954, p. 31-32.

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Comparison of Lipoxidase Oxidation and Autoxidation of Cottonseed Oil by Rat Bio-Assay¹

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THE FRACTIONATION of lipoxidase enzymes from legumes and studies of the properties of the different fractions (12) led to the work reported in this presentation. Studies on purified fractions showed (12) a rapid loss of enzyme activity with time. As an explanation for this loss of activity, it was of interest to determine whether the products of the enzyme reaction were inhibitory or whether the enzyme was particularly sensitive to the experimental conditions.

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It was also noted that the highly oxidized oil had an odor quite different from both unoxidized and autoxidized oils. At no time was the odor of the enzyme-oxidized oil as offensive as that in autoxidized oils, even those of very high peroxide values. This extreme difference in the odors typical of the two treatments is a strong indication that the oxidation products must differ. The usual analytical procedures for oxidation products are not adequate to account for these observed differences.

A number of workers (3, 8, 9, 10, 11) have reported that thermally oxidized oils have less nutritive value than the comparative fresh oils using the rat bio-assay. It was decided to use a bio-assay to determine whether the differences in odor between the enzyme-