

# The Pilot Plant Ammoniation of Dried Sugar-Beet Pulps

H. C. Millar<sup>1</sup>

Sugar-beet pulp is an important livestock feed in the western part of the United States. Part of it is fed in the wet stage, part is fed dried, while still another portion is mixed with molasses during the drying process to make a product known as molasses pulp. Sugar-beet pulp is low in protein and a means of making it a more important nitrogenous food seems desirable.

The nitrogen in a number of non-protein nitrogen compounds has been shown to be available to ruminants (14), (13), (9), (6), (4), (5). This fact is recognized by the Feed Control Officials in their 1941 action which: (1) "Resolves that urea and ammonium salts of carbonic acid are acceptable ingredients in proprietary cattle, sheep and goat feeds only; that these materials shall be considered to be adulterants in proprietary feeds for other animals and birds • that the protein equivalent of combined urea and ammoniacal nitrogen be no greater than one-third of the total crude protein nitrogen." If urea and an ammoniacal salt of carbonic acid are acceptable, then there is the question as to whether ammoniated plant materials and in particular, ammoniated beet pulp would likewise be available.

In our laboratory we have found (11) that beet pulp is easily ammoniated. The temperature of the pulp during ammoniation was found to increase as the ammonia pressure increased and the nitrogen content of the pulp increased with the ammoniation temperature.

Because of the ease with which sugar-beet pulp ammoniates and its possible utilization in ruminant nutrition, it seemed desirable to investigate the problem further in a pilot plant with larger amounts of pulp than were used in the earlier study.

## Experimental Procedure

The products used in this study were commercial plain and molasses beet pulps. The plain pulp was obtained from beets grown in Ohio, Michigan, and Minnesota.

**Equipment.**—The ammoniation was performed in a pilot plant (figure 1), an ammoniation gun, and an ammoniation cylinder.

The pilot plant was a sphere having an internal diameter of 4 feet and a volume of 33.5 cubic feet. It was provided with a jacket which surrounded the entire unit except the door and the space occupied by the header. The entire unit was built for 200 pounds operating steam pressure. It was designed with inside and outside stuffing

<sup>1</sup>Research Laboratory, The Quaker Oats Company, Chicago, Illinois.

<sup>2</sup>Figures in parentheses refer to Literature Cited.

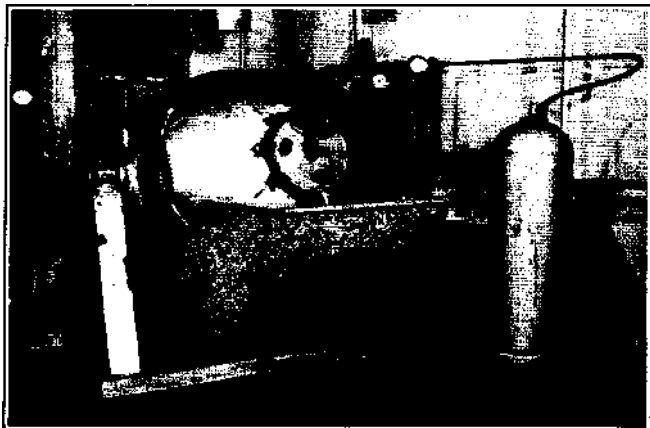


Figure 1.—The ammoniation equipment.

boxes so that independent steam pressure could be maintained on the jacket or inside chamber. A flexible ammonia hose connected the ammonia-storage cylinder to the pilot plant.

The ammoniation cylinder was a steel cylindrical chamber with internal dimensions of 6-inch diameter and 31-inch length. It was closed by a flanged plate at each end. The plates were fitted with quarter-inch inlet and outlet ammonia lines, and each line was equipped with a needle valve. The outlet plate held a thermometer in a thermometer well which extended to the center of the reaction mass. The outlet line near the chamber contained an ammonia gage. The apparatus was rotated by mounting it on two motor-driven trunnions. The amount of ammonia added was determined by adjusting to the desired pressure.

The ammoniation gun was especially designed for short-time, high-temperature experiments. It was a steel cylinder of 6-inch diameter and 30-inch length, which revolved in an insulated oven. A gas burner at the bottom of the cylinder heated the cylinder and oven. The temperature was regulated by an automatic temperature controller—a recorder of the gas-filled type. An electric contact temperature indicator and the ammonia line entered the gun through a stuffing box at the rear. The front of the gun was fitted with a door which was opened instantly by throwing a lever when the cyl-

inder was under pressure. This sudden release in pressure at the end of the ammoniation period forced the charge from the cylinder into a steel cage.

**Analytical Methods.**—All nitrogen results are expressed on the moisture-free basis. Moisture was determined by the Bidwell (2) method. The analytical procedure of Fraps (3) when applied to molasses ammoniated pulps was found to give a gel at the point wherein the lead acetate was added. Since the A.O.A.C. (9) method did not produce the gel it was used for all analyses on such pulps.

**Feeding Tests.**—Forty weanling rats varying in weight from 52 to 63 grams were selected for the rat-feeding experiment. Five rats were fed each of the diets shown in table 4. Each rat was fed in a separate cage. The starch and sugar content was varied to bring each ration to 100 percent after the desired amount of nitrogenous material was included. Care was taken that no feed was wasted, yet each rat was given all that it would eat and was weighed at the end of each week.

The palatability tests were made by feeding about 1.5 tons of ammoniated pulp as a separate supplement to 12 Guernsey cows. The milk and butter from 6 of these cows were tested over a period of 1 month for off-flavors.

#### Ammoniation in the Pilot Plant

The ammonia was added to the pilot plant in the gaseous or liquid form and was measured by the loss in weight of the cylinder. The addition of large amounts of gaseous ammonia was not practical because of its cooling effect on the ammonia storage cylinder. This caused such a low pressure that the required quantity could not be added. The difficulty was avoided by withdrawing the ammonia in the liquid form.

The ammonia entered the header and was delivered to the bottom of the pilot plant by 2 steel fingers. When pulp was added to the hot pilot plant the following procedure was used. The empty unit was heated to about 20° C. higher than the desired temperature. All condensate was then removed from the jacket and the unit was rotated until the temperature dropped to the desired value. The pulp was then added and the door was closed immediately. Fifty pounds of the cold pulp always caused a temperature drop of 20° to 30° C. unless the jacket carried steam. After the ammonia was added, the temperature always rose and went through a maximum in about 15 to 30 minutes.

The data in table 1 show that the addition of 2.5 pounds of NH<sub>3</sub> and 50 pounds of plain pulp to the cold digester raised the temperature to 40° C. and after 1 hour gave a grayish-white product containing 3.25 percent nitrogen. Under similar conditions (experiments 25 and 1), except that the pilot plant was 90° 0. when the pulp was

Table 1.—Pilot plant ammoniation of dried sugar-beet pulps.

Orig-inal	Type	Amount	Moisture	Temp. of pilot plant when pulp was added	NH <sub>3</sub> added	Ammoniation		Time to reach max. temp.	Total time	Total nitrogen	Portion of NH <sub>3</sub> fixed by pulp	Color	Palatability for Guernsey milk cow
						Maximum	Pressure						
		lb.	percentage	°C	lb.	°C	lb. gage	min.	min.	percentage	percentage		
3	Plain	50	9.0	25	2.5	40	20	10	51	1.33	...	Gray	Yes
25*	Plain	50	9.0	90	3.0	93	25	30	60	3.90	40.0	Grayish white	Yes
1	Plain	30	9.0	90	2.5	103	20	15	50	4.00	...	Light brownish gray	Yes
6	Plain	30	9.0	110	3.0	103	21	22	30	4.03	36.4	Brownish gray	Yes
27	Plain	50	9.0	90	3.0+	103	40	18	60	4.10	...	Dark Brown	Yes
7	Plain	30	9.0	120	3.0	109	26	15	30	4.12	...	Brownish gray	Yes
33*	Plain	300	8.0	70	15.0	112	45	20	60	4.04	60.8	Dark medium brown	Yes
10	Plain	50	2.0	125	4.0+	130	42	15	30	4.07	...	Cocoa brown	Yes
15	Plain	50	9.0	125	4.0+	133	25	15	60	4.61	...	Dark cocoa brown	Yes
14	Plain	50	9.0	125	4.0+	125	55	20	25	4.31	...	Cocoa brown	Yes
8	Plain	450	9.0	70	27.5	147	150	25	30	5.08	...	Cocoa brown	No
21	Plain	25	9.0	150	2.0	150	40	10	10	2.50	...	Black	Yes
25	Plain	25	9.0	200	2.0+	150	100+	10	10	4.02	...	Black	No
24	Plain	25	9.0	170	4.0+	155	50	8	8	4.20	...	Dark brown	Pool

Table 1.—Continued.

Exp. No.	Dried pulp				Ammoniation					Ammoniated product			
	Type	Amount	Moisture	Temp. of pilot plant when pulp was added	NH <sub>3</sub> added	Maximum		Time to reach max. temp.	Total time	Total nitrogen	Portion of N <sub>11</sub> fixed by pulp	Color	Palatability for Guernsey milk cow
						Temperature	Pressure						
	lb.	percentage	°C	lb.	°C	lb. gage	min.	min.	percentage	percentage			
Original	Molasses	...	...	...	...	...	...	...	...	2.00	...	Brownish gray	Yes
11	Molasses	50	8.2	90	19.5+	78	130	30	30	3.85	...	Dull brown	Yes
30	Molasses	50	8.2	90	3.0	90	25	32	60	3.66	...	Light brown	Yes
28	Molasses	50	8.2	90	3.0	90	25	53	60	3.70	...	Medium brown	Yes
19	Molasses	50	8.2	90	2.5	93	30	25	60	3.90	...	Cocoa brown	Yes
12	Molasses	50	8.2	90	2.5	94	28	25	30	3.65	...	Dull brown	Yes
2	Molasses	50	8.2	90	2.5	95	25	17	60	3.98	47.4	Dull brown	Yes
10	Molasses	300	8.2	90	19.0	95	60	20	30	3.65	...	Dull brown	Yes
9	Molasses	50	8.2	70	2.5	98	25	25	30	3.50	...	Dull brown	Yes
32	Molasses	100	8.5	90	3.0+	104	25	44	60	3.55	32.68	Greenish gray	Yes
26	Molasses	100	8.2	100	5.0+	121	45	20	30	4.18	...	Dark brown	Fair
13	Molasses	50	8.2	125	3.0	140	60	30	30	3.91	...	Dark brown	Poor

\*Pulp was less than 1 month old when ammoniated. All other pulps were 9 to 12 months old.

+Steam on jacket after NH<sub>3</sub> was added.

=When no steam was on jacket the temperature dropped 20 to 30° C. and then started rising and went through a maximum.

++Pressure went to 40 pounds gage when the NH<sub>3</sub> was added. Steam was then added to pulp to give 60 pounds gage.

added and reached a maximum temperature of 103° C, the nitrogen content of the pulp was about 4 percent. The nitrogen values of the plain pulps increased with the ammoniation temperature and in most cases fixed as much nitrogen in 30 minutes as in 1 hour. In experiment 33, a 500-pound charge of plain pulp was brought to 70° C. in the pilot plant. All the steam was then removed from the jacket and 15 pounds (weight) of liquid ammonia was added to the pulp. After 20 minutes this raised the temperature to 112° C. A palatable brown product containing 4.04 percent nitrogen was obtained and 96 percent of the added ammonia was fixed.

In experiment 23, steam was added to the pulp immediately after the ammonia was added. The steam caused 1.4 percent more nitrogen to be fixed than in experiment 21, where no steam was used, but it also made the product very much darker.

In experiments 11 and 12, molasses pulps were subjected to similar conditions, except that the former received a large excess of ammonia. The cooling effect of the excess ammonia in the pilot plant prevented the temperature of experiment 11 from becoming as high as that in experiment 12. This large excess of ammonia caused only 0.20 percent more nitrogen to be fixed in the pulp of experiment 11 than in that of experiment 12. At about 94° C. only 0.25 percent more nitrogen was fixed in dried molasses pulp in a 60- than in a 30-minute ammoniation period.

The production of dark-colored products in the pilot plant when high temperatures were employed led to a short-time, high-temperature study in the ammoniation gun. The conditions employed and the results obtained are shown in table 2. The data for a 1-minute

Table 2.—Ammoniation of plain dried sugar-beet pulp in the ammoniation gun.

Exp. No.	Sample size		Time	Pressure	Ammoniated product		
	lb.	o c			Total nitrogen	Moisture	Color
					percent- age	percent- age	
Original ..					1.70	10.2	Gray
115	4	149	1	75	3.17	5.5	Greenish gray
113	4	149	10	75	4.75	5.3	Light brown
114	4	149	15	75	4.89	3.9	Medium brown
116	4	149	30	75	5.21	4.1	Medium brown
109	4	204	1	100	3.47	5.0	Light brown
104	4	204	35	100	4.48	3.0	Dark brown
110	4	216	0.5	100	3.52	6.4	Light brown
108	4	216	2	100	4.00	5.0	Medium brown
118	4	216	10	120	5.48	3.3	Dark brown
127	4	260	2	70 to 170	3.64	4.0	Medium brown
128	4	260	3	70 to 240	4.11	2.9	Dark brown
129	4	260	4	70 to 240*	5.05	1.0	Black
130	4	260	6	70 to 120*	5.15	3.0	Black

\*The ammonia pressure was released and new ammonia was added twice.

period at a temperature of 149° 0. and 75 pounds pressure show that the nitrogen content of the pulp was about twice that of the original pulp. For each ammoniation temperature and pressure the amount of nitrogen fixed by the pulp increased with time. Furthermore, the amount of nitrogen fixed also increased with temperature.

The pulps in table 3, except number 30, were ammoniated in the ammoniation cylinder. The temperatures shown were those developed by subjecting the pulp to the corresponding pressures.

The ammoniation temperature and the nitrogen content of the pulps increased as the pressure was raised. The fat values differed little with ammoniation temperature changes and were about the same as that of the original product. The fiber values increased slightly with increased temperature and were from 1 to 2 percent above that of the original pulp. The ash was the same in each ammoniated pulp and was increased slightly over that of the original material.

The molasses pulp ammoniated at 90 pounds pressure and a similar moisture value did not reach as high a temperature or nitrogen value as the plain pulp. The amount of ammonia fixed in the molasses product was not increased appreciably by grinding it to pass a 30-mesh sieve. Furthermore, its moisture value when ammoniated did not greatly influence the amount of nitrogen fixed. The data show that the molasses pulps carry slightly more molasses after ammoniation than before. Apparently some reducing compounds are formed by the process.

### Results of Feeding Experiments

The results in table 1 on pulps ammoniated in the pilot plant show that the lighter-colored, ammoniated, plain pulps were palatable for cows. The dark-brown products were not liked and the black ones were refused.

The ammoniated molasses pulps prepared at ammoniation temperatures below about 115° C. were very palatable, while those prepared at temperatures above this value were not. The ammoniated molasses pulp was more palatable than the ammoniated plain pulp. Except for the darker products, little difference was noted between the palatabilities of the ammoniated or unammoniated pulps, either molasses or plain. There were no off-flavors or odors in the milk or butter from 6 Guernsey cows fed 1 ton of ammoniated pulps during a period of 1 month.

The pulps prepared in table 3 were also found to be palatable for cows. Sample 30-A represented the most palatable sample prepared that contained above 5 percent nitrogen.

The rat diets and growth are shown in table 4. The results show, as was expected (7), that the rats were not able to use the nitrogen added to the pulp by ammoniation. Apparently such nitrogen was not toxic to the rats, for no deaths occurred.

Table 3.—The influence of certain ammonia pressures on the temperature and analyses of sugar-beet pulps, (Data on moisture-free basis).

Exp. No.	Kind	Amount	Ammoniation						Ammoniated product						
			Moisture when ammoniated	Maximum pressure	Maximum temperature	Time	Total nitrogen	Nitrogen as protein	H <sub>2</sub> O insoluble nitrogen	Fat	Fiber	Ash	Nitrogen-free extract	Molasses	Color
Original	plain	---	---	---	---	---	1.79	11.19	1.46	0.23	21.63	2.40	64.09	---	Gray
32-A	plain	9.5	8.1	50	95	60	4.16	26.00	1.73	0.13	22.80	2.73	48.25	---	Brown
31-A	plain	9.5	8.1	90	106	60	4.82	28.98	1.95	0.04	22.95	2.84	45.20	---	Brown
30-A	plain	9.5	8.1	175	122	90	5.25	32.31	1.59	0.14	23.67	2.84	40.57	---	Brown
Original	molasses	---	---	---	---	---	1.92	12.00	---	---	---	5.64	---	32.31	Brownish gray
43*	molasses	9.5	10.2	90	85	60	4.34	27.12	---	---	---	---	---	---	Cocoa brown and gray
44	molasses	9.5	10.2	90	85	60	4.17	26.06	1.37	---	---	5.93	---	33.70	Cocoa brown and gray
46	molasses	9.5	20.6	90	106	60	4.30	28.88	1.20	---	---	---	---	35.90	Dark brown and gray
Original	molasses	---	---	---	---	---	1.93	12.06	---	---	---	---	---	27.90	Brownish gray
30**	molasses	50.0	6.8	25	94	60	3.66	22.88	---	---	---	---	---	29.00	Light brown

\*Ground to pass a 30-mesh sieve.

\*\*External heat added. The ammoniated pulps were all very palatable for cattle.



Table 4.—Growth response of rats fed ammoniated pulps.

Ingredients	Nitrogen air-dry basis	Group 1		Group 2		Group 3		Group 4		Group 5		Group 6		Group 7		Group 11	
		Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen	Percentage of ration	Percentage of nitrogen
Brewers' yeast	<b>6.89</b>	3.0	0.20	3.0	0.20	3.0	0.20	3.0	0.20	3.0	0.20	3.0	0.20	3.0	0.20	3.0	0.20
Salts*	0.70	4.0	0.03	4.0	0.03	4.0	0.03	4.0	0.03	4.0	0.03	4.0	0.03	4.0	0.03	4.0	0.03
Cod liver oil	0.00	2.0		2.0		2.0		2.0		2.0		2.0		2.0		2.0	
Starch	0.00	42.4		40.85		39.3		32.0		21.6		37.2		32.00		34.15	
Sugar	0.00	42.4		40.85		30.3		32.0		21.6		37.2		32.00		34.15	
Casein	13.76	6.2	0.85	9.30	1.28	12.40	1.70	6.2	0.85	6.2	0.85	6.2	0.85	6.2	0.85	5.86	0.80
Original molasses pulp	1.92					<u>0.0000</u>		20.8	0.40	41.6	0.80						
Ammoniated—molasses pulp	3.84	0.00										10.4	0.4	20.8	0.80	0.00	0.00
Ammoniated—plain pulp	4.75			0.00		0.00				0.00		0.00				16.84	0.80
Total N percentage.....			1.08	0.00	1.51	0.00	1.93		1.48		1.88	0.00	1.48		1.88		1.83
Average rat weight change during 7 weeks (gm.)			31.0	0.00	58.6	0.00	68.2		55.2		61.6	0.00	50.4	0.00	54.0	0.00	38.6

\*Reference (8).

### Discussion of Results

Neither the age of the pulp, its state of division, nor its source had any influence on its ammoniating properties. The color change with ammoniation was probably due to a toasting by the heat involved.

The addition of water to the pulp before ammoniation gave a darker sample than if water was not added. The addition of steam to the pulp immediately after the ammonia was added caused more nitrogen to be fixed but made the product very much darker. The moisture-free weight of the pulps changed negligibly with ammoniation.

Ammoniated sugar-beet pulp varies greatly in nitrogen content, color, odor, texture, and palatability depending upon the ammoniating conditions. Since these characteristics are affected mostly by temperature, there is the problem of fixing appreciable amounts of ammonia and at the same time keeping the other changes at a minimum. This is particularly true if a product having a nitrogen content above 4 percent is desired but is very much less important in the preparation of pulps containing 4.0 percent nitrogen or less. Experiments to secure an ammoniated pulp having a nitrogen value above 5.0 percent and a minimum amount of darkening were made. These involved employing varying temperatures for time periods as short as 30 seconds, using steam with the ammonia, successively adding and removing ammonia to reduce the amount of oxygen present, and using high-ammonia pressure with no external heat source. The product with the lightest color for the largest amount of ammonia fixed was sample 30-A which was prepared in the small chamber by subjecting the pulp to 175 pounds pressure of ammonia with no external source of heat. This procedure appears promising for the preparation of ammoniated pulps of such high-nitrogen values. A difference of 16.5 pounds of ammonia on 50 pounds of pulp in the pilot plant (experiments 11 and 13) had little effect on the nitrogen content of the product.

The pilot plant was considered to be a successful means of ammoniating pulp in large quantities, for it conveniently held 500 pounds of pulp. Such an amount of pulp was heated to 70° C, then 15 pounds of ammonia were added which raised the temperature to 112° C. After 1 hour the product had fixed 96.8 percent of the ammonia added and contained 4.04 percent nitrogen. The results indicate that a 1-hour ammoniation period is much longer than is necessary. Obviously, a minimum time period would be important for commercial operations.

The utilization of simple nitrogen compounds by ruminants has been shown (15) to be possible because of a bacteriological transfer of nitrogen. Since molasses pulp contains an abundance of readily available carbohydrate, it may be that ruminants can use the nitro-

gen in ammoniated molasses pulp more efficiently than in the ammoniated plain product.

The ammoniation gun was very serviceable for extremely short-time, ammoniation experiments. A light-brown product having 3.52 percent nitrogen was obtained with an ammoniation period of only 30 seconds.

The cows to which the products were fed had never eaten beet pulp before and it was 2 or 3 days before they ate the ammoniated products readily. After this time, unless the product was too dark, they ate the ammoniated products exceedingly well, and were always very anxious to receive the ammoniated molasses pulp. They always preferred the ammoniated molasses pulp to the ammoniated plain pulp but, except for the very dark products, seemed to have no choice between the ammoniated and unammoniated pulps, either molasses or plain. The milk and butter from the COWS fed the ammoniated pulp gave no evidence of having any off-flavor.

It has been shown (7) that rats cannot use urea and it was not expected that they could use the nitrogen imparted to ammoniated beet pulp but nevertheless this investigation seemed of value. While the rats could not use such nitrogen for their growth, there was no indication that any toxic compounds were present in the pulp, for not a single rat was lost from any of the feeding groups.

The commercial ammoniation of sugar-beet pulp could probably be done most economically by locating the ammoniation equipment at the point where the hot pulp leaves the dryer. This would make it possible to use the heat added to the pulp for drying purposes in the ammoniation step. If a product having 4.0 percent nitrogen were satisfactory, it would probably not be necessary to have the pulp or ammoniation plant hotter than about 70° C. when the ammonia is added. Under such conditions this would be a very economical process for the equipment could be built to hold large charges, and would need to rotate only very slowly. The ammonia would preferably be added in the liquid form to prevent a cooling of the storage tank and a large drop in pressure. Furthermore, the ammonia can be added in a fraction of a minute by this method. Whether the problem of recovering unreacted ammonia would be necessary would depend upon the operating conditions chosen.

Orange, grapefruit and apple pulps ammoniate in much the same manner as beet pulp. The first experiment in which ammoniated pulp was fed to calves has been concluded (12) and a second experiment on steers is in progress. Experiments to study the feeding value of ammoniated citrus pulp have been started.

### Summary and Conclusions

Sugar-beet pulps were ammoniated in 3 types of ammoniation units and the products were fed to cows.

1. Three units for producing ammoniated beet pulp commercial-ly have been described. These are the pilot plant, the ammoniation gun, and the ammoniation cylinder.

2. The ammoniation temperature greatly affects the nitrogen content, texture, color, and palatability of sugar-beet pulp. The color changes are thought to be a toasting effect.

3. A product having the lightest color for the greatest amount of nitrogen was obtained by subjecting the pulp to 175 pounds pressure of ammonia.

4. Large amounts of the pulp were easily brought to 4.0 percent nitrogen by heating the pulp to 70° C. in the pilot plant and adding liquid ammonia.

5. The molasses pulp ammoniates almost as well as the plain pulp and the ammoniation process has little effect on the fat, ash, fiber, and molasses values.

6. The ammoniation gun was very efficient for short-time ammoniation experiments.

7. The addition of water or steam to the pulp during ammoniation gives a much darker sample than is produced if these are not added.

8. A small excess of ammonia on 50-pound samples in the pilot plant had little effect on the nitrogen content of the product.

9. The ammoniated pulps, unless they were too dark, were palatable to dairy cows and had no influence on milk or butter flavor.

10. Feeding tests with rats showed the products developed no toxic substances during ammoniation, as indicated by the fact that no deaths occurred.

11. A study is needed to determine the exact value for ruminant nutrition of the nitrogen imparted to ammoniated pulps.

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## The Determination of Sulfates in Sugar-Factory Products Employing the Tetrahydroxyquinone (THQ) Reagent

A. H. EDWARDS<sup>1</sup>

A method for the determination of sulfates in beet sugars and sirups by titration using tetrahydroxyquinone as an indicator was developed by my experiments during the 1939 intercampaign and through experiences with the use of it during the 1939-40 campaign. The method adapted and developed for our use has been successfully

<sup>1</sup>Chemist, Great Western Sugar Company, Sterling, Colorado.

This paper was presented by S. J. Osborn, General Chemist, Great Western Sugar Company.