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A. B. CONNER, DIRECTOR
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DIVISION OF CHEMISTRY

The Estimation of Salt and Molasses in Mixed Feeds

AGRICULTURAL AND MECHANICAL COLLEGE OF TEXAS
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A statement of the percentage of salt in mixed feeds is required by the feed laws of practically all states, and the accuracy of this statement needs to be checked on some samples, especially where excessive quantities are supposed to be present or when salt appears to be present and is not declared. The percentage of molasses in feeds containing low-grade ingredients is also declared in Texas, and in some other states, and the accuracy of the guarantee also requires checking from time to time.

This Bulletin describes an improved method for estimating chlorides in feeds, calculated to salt, based upon the use of picric acid as a precipitant for interfering substances and titration with silver nitrate. It also describes a method for molasses, based upon the estimation of sugars in the mixed feeds. As practically all feeds contain sugars, allowance must be made for the quantity present in the other ingredients in mixed feeds besides the molasses. This is provided for in the method discussed.

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BULLETIN NO. 425 MAY, 1931

THE ESTIMATION OF SALT AND MOLASSES IN MIXED FEEDS

G. S. FRAPS

It is sometimes necessary to estimate salt (sodium chloride) in mixed or unmixed feeds. A statement of the percentage of salt in commercial mixed feeds is required by the feed laws of practically all states, and the accuracy of this statement needs to be checked on some samples. The estimation of molasses is especially necessary in Texas and a few other states whose feed laws require a statement of the percentage of ingredients in case the mixture contains roughage or low-grade materials. Sometimes excessive quantities of salt are present. The estimation of salt is based upon the estimation of chlorides, and allowance must be made for that naturally present in the feed. A discussion of the matter, with analyses of unmixed feeds for chlorides, and a rapid method for chlorides (salt) in feeds, is given in Bulletin 271 of this Station (The Salt, or Sodium Chloride, Content of Feeds). This Bulletin presents a method which is shorter than the method given in Bulletin 271.

The molasses in mixed feeds may be lower than the quantity claimed to be present. The molasses content of feeds is estimated from that constituent of molasses present in the largest quantity, namely, the sugars. As all feeds contain sugars, it is necessary to take into consideration the quantity of sugars derived from ingredients other than the molasses. As the quantity of sugar varies both in feeds and in molasses, the estimation of the quantity of molasses is not exact. The object of the estimation of the molasses should be taken into consideration in the interpretation of the results. If the object is to determine if the feed comes up to the guarantee made under a feed law, it is necessary to give the guarantor the benefit of any doubt.

THE ESTIMATION OF SALT

The method described in Bulletin 271 referred to above, consists briefly in extracting the salt from 5.85 gm. of the feed with about 200 cc. water, purifying the extract by means of carbon black or lead acetate, making up to 200 cc., adding an excess of 0.1 N silver nitrate to 50 cc., and titrating back with 0.1 N sulphocyanate, with ferric sulphate indicator. The volume of silver nitrate used by the salt (in cc.) X 0.4 equals the percentage of sodium chloride.

The method described in this publication is based upon the method of E. R. Theis, published in the Chemist-Analyst, No. 41, 1924, and consists in extracting the salt with a solution of picric acid, neutralizing with calcium carbonate, and titrating an aliquot of the filtrate with silver nitrate, using potassium chromate as an indicator. The picric acid renders the protein insoluble and takes the place of the carbon black or lead acetate used in the other method. Preliminary

work showed that the end point was uncertain and results are inaccurate unless the acidity of solution was neutralized before the titration with silver nitrate.

DESCRIPTION OF METHOD FOR SALT

The method as finally used is described in full as follows:

Saturated solution of picric acid. Add 4000 cc. of water to 70 gm. of picric acid. Let stand 24 hours, shaking from time to time. Use the clear liquid.

Silver nitrate solution, 0.1 N. Dissolve 34.5 grams of pure crystallized silver nitrate and make up to 2000 cc. Determine the exact strength of solution by titrating against 10 cc. of 0.2 N hydrochloric acid, which has been neutralized with pure powdered calcium carbonate, using potassium chromate as indicator. Adjust so that 20 cc. of the silver nitrate solution equals 10 cc. of the 0.2 N acid. Keep in an amber or dark-colored bottle.

Potassium or sodium chromate. Dissolve five grams in 100 cc. of distilled water.

Estimation

Weigh 5.85 gm. of the feed into a 200 cc. volumetric flask. Make up to volume with picric acid solution. Let stand an hour, or longer, shaking 3 or 4 times. To 50 cc. of the clear liquid, filtered if necessary, add about 0.5 gm. calcium carbonate, or enough to make the solution slightly milky, and 1 cc. potassium chromate solution. Titrate with 0.1 N silver nitrate solution until the red coloration appears, keeping the solution by the side of a solution of picric acid or a portion of the same solution, so as to readily detect the change in color.

If the solution of the feed is brownish in color, as may occur with molasses feeds and some other feeds, it should be decolorized with carbon black. To 70 to 80 cc. in a dry beaker, add 1 gram of carbon black, mix by giving the beaker a rotary motion, so as to wet the carbon black completely, and let stand an hour. Stir well and filter through a dry 11 cm. filter into a dry beaker. Reject the first few cc. Titrate 50 cc. as directed above. 1 cc. of 0.1 N silver nitrate equals 0.4 per cent of sodium chloride.

Test of the Method

The method was tested by three analysts on solutions of known content of salt added to feeds and by comparisons with the sulphocyanate method mentioned above on samples of mixed feeds. One of the analysts preferred the sulphocyanate method, for the reason that the end point was sharper to his eyes. The other two preferred the picric acid method because it was shorter and took less time.

Table 1 contains tests of the picric acid method, on known amounts of a solution of salt added to wheat gray shorts. The salt solution was measured out by one analyst and the estimation run by another who had

no knowledge of the quantities of salt present. Satisfactory agreement is found between the salt used and the salt determined by the method.

Table 1.—Test of picric acid method, using known amounts of salt added to wheat gray shorts.

Portion No.	Salt used (per cent)	Salt found (per cent)
1	1.28	1.30
2	1.52	1.54
3	1.26	1.31
4	0.94	0.98
5	2.01	2.04
6	1.74	1.82
7	1.25	1.26
8	2.30	2.23
9	2.49	2.49
10	2.04	2.09
11	2.36	2.47
12	0.75	0.79

A comparison of the picric acid method with the sulphocyanate method on some samples of commercial mixed feeds is given in Table 2. The agreement between the results by the two methods is satisfactory.

Table 2.—Percentage of salt in mixed feeds as determined by two methods.

Feed Number	Picric acid method	Sulphocyanate method
41560	1.72	1.64
41563	1.09	1.00
41638	1.20	1.13
41639	1.70	1.65
41652	1.77	1.81
41654	1.30	1.31
41656	1.07	.94
41659	3.57	3.57
41663	1.58	1.57
41666	1.64	1.59
41684	.71	.71
41693	1.32	1.32

Interpretation of Results

Chlorides occur in certain ingredients of mixed feeds. Many feeds contain very small percentages of chlorides, but there are some, used especially in chicken feeds, which contain appreciable amounts. The feeds which contain much salt are alfalfa, cocoanut meal or cake, dried beet pulp, molasses, and animal by-products, such as fish meal, tankage, dried buttermilk, dried blood, and meat scraps. A summary of some analyses is given in Table 3.

The other feeds examined usually contain less than .2 per cent chlorides calculated to salt and include barley chop, velvet beans, brewers' dried grains, corn bran, corn chop, corn feed meal, ground corn and cobs, ear corn chop with husk, whole-pressed cottonseed, cottonseed meal or cake, feterita, Graham flour, rye flour, hominy feed, kafir chop, kafir head stems, linseed meal, milo chop, milo heads, oats, oat groats, oat

hulls, oat meal mill by-product, peanut feed, peanut hulls, peanut meal or cake, whole-pressed peanuts, rice bran, rough rice, rice polish, sorghum fodder, wheat, wheat bran, wheat brown shorts, wheat gray shorts, wheat mixed feed, wheat white shorts, as well as some samples of hay and grasses. The guarantee of salt in a mixed feed should, of course, give the total quantity present, regardless of source.

Table 3.—Percentage of chloride expressed as sodium chloride in feeds high in chlorine.

Name of feed	Average	Maximum	Minimum	Number of samples
Alfalfa (western)	.98	1.89	.42	14
Beet pulp, dried	.87	1.47	.37	3
Blood, dried	.75	.....	.....	1
Buttermilk, dried	2.38	2.94	1.64	3
Cocoanut meal or cake	1.11	1.73	.84	14
Corn gluten feed (one manufacturer)	2.25	2.30	2.17	3
Fish meal	.57	.76	.48	3
Meat meal	2.77	2.95	2.58	2
Meat scraps	1.31	3.37	.58	8
Meat and bone scraps	1.54	1.58	1.49	2
Molasses	1.12	1.41	.90	14
Tankage	1.53	2.99	.23	8

If it is desired to estimate the amount of salt which has been added to a mixed feed, it is necessary to deduct from the total quantity of salt found, the amount of salt furnished by the feed ingredients which carry salt. This estimate should be based on the nature of the ingredients and the kind of feed, if a knowledge of the percentage of ingredients of the feed is not available.

The percentage of salt in the feeds mentioned above should also be taken into consideration when these feeds are used in compounding mixed feeds. Otherwise the mixture may contain more salt than is desired, or supposed to be present.

THE ESTIMATION OF MOLASSES

The method used for the estimation of molasses in mixed feeds consists in estimating the sugar and (A) calculating the percentage of molasses from the amount of sugar left after deducting for that carried by the other ingredients of the feed or (B) comparing the quantity of sugars found with the quantity calculated from the ingredients. Feeds usually contain small amounts of reducing sugar, but most of the sugar is present as compound sugars, such as sucrose, which must be completely or partly converted into reducing sugar before the estimation is made. The method of conversion conveniently followed is that used for sucrose or cane sugar. This does not completely hydrolyze all of the compound sugars found in the plants. Raffinose, for example, which occurs in cottonseed meal, is hydrolyzed to two molecules of reducing sugar, while complete hydrolysis would change it to three. It is thus necessary to make the calculations of the sugar in the ingredients of the feed on the basis of the amount found by the method of analysis and not

on the amount which would be found if all the sugar is completely inverted. The number of feeds which contain sugar not completely hydrolyzed is small. Most feeds contain sugars which are completely hydrolyzed by the method referred to.

Description of Method for Sugar

The method as used for mixed feeds is described as follows:

Place 5.333 gm. of the substance in 100 cc. of water and let stand two hours, stirring thoroughly every 15 minutes. Filter and wash into a 200 cc. graduated flask nearly to volume. Add 5 cc. lead subacetate solution as used in the A. O. A. C. method for sugars; make up to mark and shake well. Filter and measure 75 cc. with a pipette into a 200 cc. flask; add 5 cc. sodium sulphate (10 per cent solution) and shake; then 5 cc. copper sulphate (10 per cent solution). Make up to mark and shake. Filter through a fluted filter into a dry beaker.

Place 50 cc. (equals 0.5 gram substance) in a beaker, add 5 cc. of concentrated hydrochloric acid, and set aside for 24 hours at a temperature not below 20° or for ten hours at 25° C. Neutralize with dry sodium carbonate and determine total sugar, by the Munson and Walker method (A. O. A. C.). Calculate copper to invert sugar.

Sugar in Molasses

The percentage of sugar in molasses varies according to the degree of extraction of the sugar from the syrup and the amount of concentration of the molasses. The percentage of sugar in some samples of molasses is given in Table 4. On the basis of this table, it seems fair to estimate that molasses should contain not less than 50 per cent sugar, after inversion by the method mentioned above.

Table 4.—Composition of molasses used in Texas feeds.

Laboratory number	Total sugar as invert	Reducing sugar	Protein	Water	Ash
9265	60.68	29.73	2.47	25.59	5.75
9266	55.36	24.51	3.29	32.61	5.83
9263	62.32	31.12	2.25	28.45	5.62
9264	60.04	29.30	2.16	29.89	5.51
9341	58.20	10.68	3.21	20.43	7.59
17427	55.96	32.60	2.75	29.75	6.53
17428	52.09	23.39	4.84	27.36	7.26
17439	52.36	17.64	4.68	26.57	8.50
17461	58.96	25.12	2.75	25.41	4.28
17462	54.96	19.32	3.50	28.95	7.13
17456	60.84	21.72	2.82	22.49	5.06
17486	61.28	23.44	2.94	23.58	5.29
17713	56.20	22.28	4.58	28.25	6.20
17757	57.60	21.32	3.37	27.08	6.11
17792	53.12	33.04	5.22	30.61	7.47
17801	47.33	27.69	4.63	29.06	5.56
17802	52.32	19.38	4.00	29.56	.38
17793	47.82	10.96	3.98	26.37	13.01
17798	58.18	19.72	3.75	24.24	6.03
17794	54.20	18.40	3.83	23.84	5.73
22252	42.59	26.25	3.62	35.19	8.46

Sugar in Feeds

The amount of sugar in feeds which should be allowed for in calculating the molasses present depends upon whether it is desired to ascertain if the feed contains too little molasses or too much. If it is desired to ascertain if the feed contains too little molasses, the sugar assumed to be present in the other ingredients should be a little below the average. This gives the manufacturer the benefit of the doubt in this direction. If it is desired to determine if the feed contains too much molasses the assumed amount of sugar in the other ingredients should be above the average, as this would give the manufacturer the benefit of the doubt in the other direction.

The usual object of the analysis is to ascertain if the mixed feed contains too little molasses. The amounts of sugar in feeds proposed for use in this case are given in Table 5. As stated above, the amounts of reducing sugar and inverted sugar taken together are slightly below the minimum. The analyses on which this table is based are published in Bulletins 196, 290, and 418 as well as in unpublished work of the Experiment Station. There is of course some variation in the quantity of sugar present in feeds as well as in molasses.

Table 5.—Percentage of sugar, as invert sugar, for use in calculating molasses in mixed feeds.

Feed	Sugar
Alfalfa meal	4.0
Alfalfa, chopped	4.0
Barley chop	2.2
Beet molasses	50.0
Beet pulp, dried	8.0
Blood, dried	0
Buttermilk, dried	.....
Bone meal	0
Brewers' dried grains	0.2
Cocoanut cake or meal	7.5
Corn bran	2.0
Charcoal	0
Corn chop	1.5
Corn grits—see corn chop	.....
Corn cob	1.5
Corn feed meal	1.5
Distillers' corn grains	0.2
Corn, ear chop	1.5
Corn germ oil meal	1.5
Corn gluten feed	2.0
Corn, ear chop with husk	1.5
Corn husk	1.0
Cottonseed chop	2.0
45% protein cottonseed meal or cake	5.0
43% protein cottonseed meal or cake	5.0
Whole pressed cottonseed	2.0
Whole pressed cottonseed	2.0
Cottonseed hulls	0.2
Feterita chop	1.2
Fish meal	0
Feterita head chop	1.0
Hominy feed or meal	2.5
Flax plant by-product	2.0
Kafir chop	1.0
Kafir head chop	1.0
Kafir head stems	0.5
Linseed meal, new process	4.0
Linseed meal, old process	4.0
Meat and bone scraps	0

Table 5.—Percentage of sugar, as invert sugar, for use in calculating molasses in mixed feeds —Continued.

Feed	Sugar
Meat scraps (variable)	0
Milo chop	1.5
Milo head chop	1.0
Molasses, blackstrap	50.0
Oat hull clippings (clipped oat by-product)	0.2
Oats, ground or whole	1.5
Oat screenings	1.0
Oat hulls	0.5
Oat, middlings or shorts or meal	2.0
43% protein peanut meal or cake	8.0
Whole-pressed peanuts	6.0
Peanut vines	3.0
Whole-pressed peanuts	6.0
Peanut hulls	1.2
Peanut meats	3.5
Rice bran	3.5
Rice polish	4.0
Rice, ground rough	1.0
Rice hulls	0.5
Rice straw	0.5
Rye chop	3.5
Tankage	0
Sorghum bagasse	3.0
Sorghum head chop	1.0
Sorghum leaves	5.0
Velvet beans	7.0
Velvet bean pods	0.5
Wheat bran	5.0
Wheat chop	3.0
Wheat mixed feed	5.0
Wheat gray shorts	4.5
Wheat brown shorts or middlings	5.0
Wheat screenings	2.0
Wheat white shorts	3.0
Wheat flour (low grade, red dog)	3.0

Method of Calculation

If the percentage of the ingredients of the feed claimed to be present by the manufacturer is known, the percentage of sugar which should be present could be compared with the percentage found by the analysis. A few comparisons of this kind are shown in Table 6.

If the percentage of the ingredients of the feed is not known, it is necessary to make some estimate of the approximate amount present. This will naturally lead to greater error than if the ingredients are known, as the time is usually lacking to make a complete estimation of the ingredients. However, an examination of Table 5 shows that many feeds containing comparatively small quantities of sugar are closely related in this respect and could be grouped together. This renders less difficult the comparison of the amount of sugar which should be present with that which is found. The errors in estimating the sugar which should be present in the ingredients, other than molasses, are to some extent neutralized and also to some extent low, due to the fact that molasses usually contains more than the 50 per cent of sugar used as a basis in the calculations.

Table 6.—Percentages of invert sugar calculated and found in some mixed feeds.

Laboratory Number	Found	Calculated
31569	5.46	6.48
31571	4.74	5.57
31573	8.36	7.35
31574	6.08	5.60
31582	6.00	7.98
31618	21.90	17.80
31648	7.72	9.45
31649	6.64	7.08
31650	9.92	8.74
31564	4.38	4.39
31566	8.08	8.18
31682	9.22	9.39
31687	4.64	6.71
31688	1.46	8.35
31694	17.94	18.23
31714	9.30	9.61
31715	9.86	11.11
31003	9.18	11.76
31118	20.72	20.10
31176	10.24	11.70
31206	5.62	4.41
31214	6.16	4.62
31221	9.98	14.25
31230	9.10	13.18
31231	12.70	11.68
31251	10.28	11.76
31307	25.78	27.00
31336	10.38	16.55
31344	8.08	9.38
31345	7.64	7.84
31349	8.82	9.11
31477	11.96	13.69
31479	6.26	5.33
31490	9.46	7.07
31494	27.18	22.15
31497	10.38	9.13

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SUMMARY

Chlorides may be estimated more rapidly in feeds by the picric acid method here described than by the sulphocyanate method previously described.

A guarantee of salt should of course include the total amount present. If the amount of added salt is to be estimated, allowance must be made for the chloride naturally present in appreciable amounts in certain ingredients of mixed feeds. These include alfalfa, dried beet pulp, cocoanut meal or cake, molasses, dried buttermilk, and animal by-products, such as fish meal, meat meal and tankage. Most other feeds contain little chloride.

Molasses in mixed feeds is estimated from the invert sugar present after inversion of the sugars in a purified extract by the usual method for cane sugar.

Molasses is assumed to furnish 50 per cent of its weight as invert sugar. Allowance must be made for the sugars naturally present in feeds. A table is given containing the percentages of invert sugar in a number of feeds, which may be used in this calculation.