CORNELL UNIVERSITY; AGRICULTURAL EXPERIMENT STATION. BULLUTIN 37. ALL DIVISIONS. DECEMBER, 1891. SUNDRY INVESTIGATIONS MADE DURING THE YEAR, PP. 357-405

Published @ 2017 Trieste Publishing Pty Ltd

ISBN 9780649296071

Cornell University; Agricultural Experiment Station. Bullutin 37. All divisions. December, 1891. Sundry investigations made during the year, pp. 357-405 by Various

Edited by Trieste Publishing Pty Ltd.
Cover @ 2017

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VARIOUS

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Trieste

CORNELL UNIVERSITY

Agricultural Experiment Station.

BULLETIN 37.

ALL DIVISIONS.

DECEMBER, 1891.

SUNDRY INVESTIGATIONS MADE DURING THE YEAR.

PUBLISHED BY THE UNIVERSITY,
ITHACA, N. Y.
1891.

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CHEMICAL DIVISION.

DETERMINATION OF FATS IN FODDERS BY DIRECT WEIGHING OF THE ETHER-EXTRACT AND BY LOSS OF WEIGHT OF THE SUBSTANCE.

The present official method for the determination of fats in fodders requires that the substance should be dried in a current of dried hydrogen, at the temperature of boiling water, for four hours to remove all the moisture; the dried substance is then extracted with ether and the extract dried to constant weight under the same conditions.

In order to meet all these requirements Prof. Caldwell described in 1888 a "New Apparatus for Drying Substances in Hydrogen and for the Extraction of the Fat." A reference to the cut and the description[1] will show that these tubes serve the double purpose of glass stoppered weighing tubes, as well as drying tubes. This apparatus was made use of in the work described in this paper in the manner indicated in the bulletin, except that the current of hydrogen was conducted in the opposite direction. This change was found to be preferable, to prevent mechanical losses by particles of the substance being carried along with the current, and also to prevent the loosening of the asbestos filter from the metallic disk supporting it.

In the same bulletin Prof. Caldwell says:—"When the extraction is completed there seems to be no reason why the amount extracted cannot be determined by weighing the dried contents of the tube as well as by weighing the substance extracted: and the drying in hydrogen is much more easily managed in the former case than in the latter; but I have not yet had time to test this variation of the method."

[1] Bulletin No. 12 Cornell University Agr. Exp. Station.

At the seventh annual convention of the Association of Official Agricultural Chemists Mr. Anderson reported on two substances that the percentage of fat by loss of weight was a little larger than by weighing the extract. This he accounted for by saying that a portion of the fat was volatilized during the extraction.

In order to obtain some idea as to the favorable or unfavorable conditions for the loss of volatile fats during the extraction process, a short thermometer, 12½ c. m. long, was imbedded in the fodder tubes when ready for the extraction. The thermometer was of such length that it would be entirely inclosed in the extraction apparatus. Another thermometer was inserted in the flask. A number of temperature determinations were made in this way. The temperature of the substance in the extraction tubes ranged from 33° to 35° centigrade, the temperature of the contents of the flask registered 41°+43° C.

The substance before and after extraction, and the fat, were all dried at the temperature of boiling water and it would hardly seem possible that the lower and intermediate temperature of 43° would be the cause of a loss of a portion of the extract by volatilization. During the extraction the fat is in solution, and the conditions are less favorable for volatilization than in either the previous or subsequent drying at the temperature of boiling water.

A number of comparisons were made by the two methods. The tubes were dried first for the official four hours to remove the moisture, but the data obtained in this way were discordant ; with some substances the agreement was close, but with others not so, there being a general tendency for higher results by the loss of weight. The work was repeated on the same substances in the following manner : instead of drying four hours, the drying was continued until duplicate weighings of the tubes agreed within one milligram. This required a variable length of time which always exceeded four hours, and in the case of gluten meal exceeded six hours. When the fats were determined by loss after drying in this way the agreement was much more satisfactory.

From the results thus obtained it appears that the first drying of four hours was not sufficient to remove all of the moisture, and the drying after the extraction removed the moisture left on the first drying ; so that the loss of weight of the tubes also included

a variable amount of moisture with the ether-soluble matter. Hence the larger results by loss of weight.

In the following table the results on the same line are from the same weighed portion. The tubes were dried in hydrogen both before and after the extraction, except that tube c in samples Nos. 6, 7, and 8, and tube b of No. 11 were dried in air after the extraction.

ETHER-EXTRACT.

		By loss of weight of extraction tube.	Average.	By weighing of ether-extract.	Average.
No. 1, Brewers' grains	a	7.34		7.26	
	b	7.35	7.35	7.47	7.36
No. 2, Timothy hay	a	2.61		2.59	
	b	2.50	2.55	2.52	2.55
No. 3, Red clover hay	a	3.31		3.27	
	b	3.15	3.22	3.18	3.22
No. 4, Gluten meal	a	10.80		11.24	
	b	11.12		11.15	
	c	11.20	11.16	11.16	11.18
No. 5, Ensilage	a	2.22		2.19	
	b	2.28	2.25	2.32	2.25
No. 6, Cotton-seed meal	a	9.35		9.33	
	b	9.38		9.45	
	c	9.44	9.39	9.42	9.40
No. 7, Corn meal	a	4.04		3.92	
	b	3.92		4.04	
	c	3.85	3.93	3.85	3.93
No 8, Ship stuff	a	4.31		4.37	
	b	4.72		4.67	
	c	4.56	4.53	4.46	4.50
No. 9, Ensilage	a	2.41		2.45	
	b	2.12	2.27	2.28	2.36
No. 10, Ensilage	a	3.20		3.20	
	b	2.73	2.97	2.73	2.97
No. 11, Ensilage	a	2.06		2.57	
	b	2.50	2.55	2.66	2.61
No. 12, Ensilage	a	lost		2.41	
	b	2.43		2.57	

The total average by weighing the fat was .006 per cent. higher than by the loss of weight, a difference which is insignificant.

The tubes are more convenient for drying than flasks, and no additional or expensive drying apparatus is required as for the

flasks. The drying of the tubes after the extraction can be done much more quickly and efficiently than drying the flasks, for the gas passes through the substance rather than over its surface, thereby requiring less hydrogen. More than this, the results show that the use of hydrogen in this second drying can be dispensed with.

On the whole, therefore, it is more economical as to time, apparatus and hydrogen, to determine the fats by loss of weight of the substance, while there is no loss in accuracy.

THE DETERMINATION OF ALBUMINOID NITROGEN.

In the determination of nitrogen by the Kjeldahl method directions are given for the addition of potassium sulphide to precipitate all mercury from the solution in order to prevent the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution.

The determination of albuminoid nitrogen according to the present official method, requires the use of 0.7 to 0.8 grm. of copper hydroxide previous to the determination of the nitrogen by the Kjeldahl method.

The copper hydroxide is carried along in the determination, and is freely soluble in the sulphuric acid; no provision is made for its removal, and when the potassium sulphide is "added to precipitate all mercury in solution," copper sulphide is alike precipitable in the acid solution, with the mercury. The copper present is more than enough to combine with all the potassium sulphide; the result is that neither metal is completely precipitated, a large and variable portion of the mercury is left in the solution, and all the conditions are favorable for the formation of mercuro-ammonium compounds. The work given to the potassium sulphide is doubled and the object for which it is added is not attained.

In order to determine to what extent the mercury so left in the solution would affect the results, a number of determinations were started; to some the official "20 c.c. of potassium sulphide" were added, and to others a sufficient quantity of the same solution to precipitate all the copper as well as all mercury in solution. The

large amount of precipitate formed in the latter case caused so much bumping that only one determination out of six could be completed.

In order to obviate this difficulty the operation was carried on exactly as required by the official method, except that previous to distillation the acid solution was made up to 200 c.c. and divided into two equal portions. One portion was distilled with the relative official amount of potassium sulphide, and the second with enough of the same solution to make the precipitation of both mercury and copper complete. This gives a more satisfactory basis for the comparison of results, since all the conditions previous to the distillation are the same as to the degree of oxidation and the amounts of mercury and copper in solution.

The following results were obtained:.

	50 c.c. K_2S.	30 c.c. K_2S.	Difference.
Ensilage No. 1	1.28 per cent.	1.12 per cent.	0.16 per cent.
" "	1.25 "	1.14 "	0.11 "
" " 2	.66 "	1.53 "	0.13 "
	.66 "	1.60 "	0.06 "
" " 3	1.36 "	1.27 "	0.09 "
	1.20 "	1.15 "	0.05 "
" " 4	1.52 "	1.45 "	0.07 "
	1.54 "	1.45 "	0.09 "
" " 5	1.18 "	1.08 "	0.10 "
	1.23 "	1.12 "	0.11 "
Average			.096

The constant larger proportion of nitrogen yielded in these results with the 50 c.c. of potassium sulphide must be due to the incomplete precipitation of all the mercury in solution when only 20 c.c. is added. The average difference in the ten portions amounts to nearly a tenth of one per cent.

Blank determinations with both 20 and 50 c.c. portions of potassium sulphide gave no nitrogen. A difference of a tenth of one per cent. when multiplied by the protein factor would make a difference of over six tenths per cent. of protein, which is too great to be overlooked.

Some change must therefore be made in combining Stutzer's and Kjeldahl's methods. The following proportions of substance and solutions have given me satisfactory results. Take 0.7 grm. of substance instead of 1 grm., a quantity of copper hydroxide and glycerine solution equivalent to .5 to .6 grms. of the hydrox-