Proceedings of the Iowa Academy of Science

Volume 1 | Part 2, 1890-1891; (1887) - Article 31

1890

A New Distilling Flask for Use in the Kjeldahl Process

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A NEW DISTILLING FLASK FOR USE IN THE KJELDAHL PROCESS.

By G. E. Patrick and D. B. Bisbee.

The only serious drawback to the Kjeldahl method of nitrogen determination is the breakage of distilling flasks, and in laboratories where many determinations of albuminoid nitrogen are made by the Stutzer process this breakage is often a matter of much annoyance and considerable expense, since only the best quality of flasks will long stand the requirements of the process.

Some months ago the breakage in a certain lot of flasks purchased for this laboratory having become unendurable, the writers hit upon the idea of distilling from copper flasks; and upon trial, the results have been so satisfactory that we can with confidence recommend the plan to other chemists. The copper flasks used were the ordinary one pint oxygen retorts, minus caps, delivery tubes and clamps.

At first, trials were made by distilling ammonia from a solution of pure ammonium chloride and NaOH, to assure ourselves that no ammonia was retained by the copper. These results were made comparative by distilling from both glass and copper flasks. Exactly 10 c.c. of an ammonium chloride solution of known strength were used in all following tests. The results, after deducting for error found by blank experiment, were as follows:

	IN GLASS FLASKS.	IN COPPER FLASKS.
No. of C.C. of decinormal acid neutralized	14.4	14.25
" " " "	14.23	14.23
" " " "	14.2	14.30
" " " "	14.28	14.25
" " " "	14.3	14.25
" " " "	14.15	14.30
Mean of six	14.26	14.26

Next a salt of mercury was added to the ammonium salt in the flask, and K_2S sufficient to precipitate the mercury was added before liberating the ammonia and distilling. The following were the results after deducting for the error in the blank:

	IN COPPER FLASKS.
No. of C.C. decinormal acid neutralized	14.13
" " " "	14.25
" " " "	14.25
" " " "	14.2
" " " "	14.25
" " " "	14.25
Mean of six	14.22

These results compare favorably with those from glass just reported.

Next, to imitate the condition of Stutzer's process, copper hydrate, as well as a mercuric salt and K_2S, was added. Results after deducting the blank were as follows:

	IN COPPER FLASKS.
No. of C.C. of decinormal acid neutralized	14.2
" " " "	14.2
" " " "	14.3
" " " "	14.25
" " " "	14.25
Mean of five	14.24

Here again, the results were practically identical with those obtained by distilling from glass.

The plan was then tried upon the product of the Kjeldahl digestion in fodder analysis, both in total and albuminoid nitrogen determination, the results in all cases being in substantial agreement with those obtained by distilling from glass; and now we use the metallic flasks in the regular analytical work of the laboratory. A few results will suffice to illustrate:

SUBSTANCE TAKEN.	RESULTS—IN COPPER.	IN GLASS.
Shorts, total Nitrogen	2.81 per cent.	2.81 per cent.
Shorts, Albuminoid Nitrogen	2.26 "	2.26 "
Cream Gluten Meal, total Nitrogen	6.28 "	6.27 "
Cream Gluten Meal, Albuminoid Nitrogen	6.18 "	6.24 "
	6.21 "	6.22 "
Sugar Meal, total Nitrogen	3.33 "	3.19 "

(Determinations made two months apart.)

We employ 200 c.c. of water in transferring the contents of the digestion flask into the distilling flask, using about half of it in diluting and cooling the acid licquid before actually transferring. We are also in the habit of introducing 30 c.c. of the K_2S solution, instead of 25 c.c. as is usually directed. This may not be necessary, but the fact that the residual liquid after distillation is always free from (binary) sulphur, the excess being removed by the flask itself, seems to render a little extra sulphide advisable. This action between the sulphide and the copper will doubtless in time destroy the flasks; but long before that time arrives, they will have saved in glassware many times their cost.

The flasks are heated by rather small, naked flames; a large flame under the one pint flask will boil the charge over. The receiving flasks are marked at the 200 c.c. level to show when the operation is finished. No zinc or pumice is required to prevent "bumping;" otherwise, the arrangements are as usual.

The distillation is completed *within thirty minutes;* so the saving of time is very great.

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