CXIX. NOTE ON THE LOSS OF NITROGEN AND SULPHUR ON DRYING FAECES.

BY DAVID PATON CUTHBERTSON
AND ALEXANDER KING TURNBULL.

*From the Biochemical Laboratory of the Royal Infirmary and the
Pathology Department, The University, Glasgow.*

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WHEN the collection of faeces during a metabolic experiment extends over a considerable period of time, difficulties arise in the determination of the total nitrogen and sulphur contents. Sampling of the wet composite specimen is generally unsatisfactory and ordinary methods of drying on a steam-bath result in a considerable loss of both nitrogen (as ammonia) and sulphur (mainly as sulphide).

If nitrogen is the only element under consideration its loss as ammonia can be prevented by acidification with H_2SO_4 before drying. To obviate drying Van Slyke [1932] has recommended sampling from a suspension of the faeces in H_2SO_4 . If sulphur is also being determined, the excess of sulphuric acid which requires to be added masks the sulphur originally present in the faeces, and further, acidification leads to loss of H_2S and certain organic sulphides on drying.

The following experiments indicate the extent of the nitrogen and sulphur loss during drying. A method of preventing this loss is described.

EXPERIMENTAL.

Total N was estimated by the Kjeldahl method; total S by Denis's modification of Benedict's ashing method. Analyses were performed in triplicate, first on the wet specimens and then on aliquot portions subjected to various procedures. The accuracy of the sampling of these wet specimens was confirmed by the close agreement of triplicate estimations.

All specimens of human faeces were analysed within an hour or two of being voided.

Exp. 1. Sample of faeces containing 0.8976 g. total N had after 10 days' continuous drying on a steam-bath 0.8439 g. N. % loss = 5.9.

Exp. 2. Sample of faeces containing 0.616 g. N had after 14 days' drying on a steam-bath 0.558 g. N. % loss = 9.4.

Exp. 3. Sample of faeces containing 1.599 g. N and 0.222 g. S contained after 14 days' continuous drying on a steam-bath 1.531 g. N and 0.116 g. S. % loss N = 4.2. % loss S = 47.

Exp. 4. Fresh wet faeces containing 1.305 g. N were transferred to a vacuum-pan and the temperature of the bath raised to 80°. Ammonia-free air was passed over the faeces and drying was carried out under reduced pressure. In the course of 1 hour during which the temperature of the bath was rising no ammonia came over. In the next 5 hours 0.0286 g. N was trapped in $N/10$ H_2SO_4 . That the loss of N was as NH_3 was confirmed by nesslerisation. During the subsequent 6 hours only 0.0007 g. more N came over. Loss of N = 2.2 % in course of 14 hours' drying.

In an attempt to prevent the simultaneous loss of N and S from faeces by means of a preliminary treatment with substances which would trap NH_3 and H_2S no simple method could be devised. It was eventually determined that the most satisfactory method was to moisten the faeces with excess 2 % copper acetate and trap the ammonia in acid during the process of drying.

Exp. 5. Sample of faeces containing 0.114 g. S contained after drying with copper acetate 0.115 g. S. There was thus no loss of S. When an aliquot portion was dried without any precaution for a similar period, *viz.* 2 days, the dried faeces contained 0.109 g.—a loss of 4.8 % S.

From clinical and bacteriological considerations it is obvious that the amounts of free gases contained in faeces must vary considerably.

Exp. 6. A sample of faeces containing 1.867 g. N was dried in the presence of 2 % copper acetate for 24 hours. Ammonia-free air was passed over the faeces and then through standard H_2SO_4 . Ammonia was estimated by nesslerisation as it was found that acetic acid was also driven over. The total amount of trapped N was 0.275 g. The total residual N was 1.591 g. 1.866 g. N of the original 1.867 g. N were thus accounted for.

The original sample of faeces contained 0.1705 g. S. An aliquot dried without precaution for 24 hours contained 0.147 g. S and one dried for 6 days contained 0.1355 g. S—losses of 14 % and 20 % respectively. An aliquot treated with copper acetate lost no S on drying for 24 hours.

Exp. 7. A sample of faeces containing 2.522 g. N was dried in presence of 2 % copper acetate for 48 hours. Ammonia-free air was passed over the faeces and through a 10 % NaOH trap to absorb the acetic and other volatile acids. The air was finally passed through standard acid. In the final estimation the residual ammonia was distilled over from the NaOH solution and added to that trapped in the acid. The total loss was 0.0339 g. N. The nitrogen content of the dried faeces was 2.495 g. The original 2.522 g. were thus accounted for.

The original sample of faeces contained 0.588 g. S. An aliquot dried without precaution for 24 hours contained 0.581 g. S and one dried for 5 days 0.5675 g. S—losses of 1.2 % and 3.5 % respectively. An aliquot dried with 2 % copper acetate lost no S on drying for 24 hours.

In these experiments allowance was made for the trace of S in the copper acetate by adding aliquot amounts at the appropriate times. It was thus added to ordinary air-dried specimens of faeces after the process of drying was complete but before analysis.

It is seen from these experiments that an accurate analysis of the nitrogen and sulphur of faeces is possible if in the process of drying, ammonia-free air is passed over the faeces to which have been added a dilute solution of copper acetate in excess, the air is then passed through alkali to trap acetic and other volatile acids and then through standard acid. The alkaline fluid traps some ammonia and this is finally distilled into the excess standard acid.

SUMMARY.

1. The process of drying faeces on a water-bath leads to a considerable loss of N (mainly as ammonia) and S (mainly as sulphide).

2. This loss can be prevented by adding an excess of a dilute solution of copper acetate to the faeces before drying them in a closed vessel through which passes a current of dry ammonia-free air. The ammonia liberated from the faeces during drying is trapped in acid.

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REFERENCE.

Van Slyke (1932). Quantitative clinical chemistry, 2, 77. (Baillière, Tindall and Cox, London.)