Correspondence: A method for determining total nitrogen . . .

From Professor A. Fleck

I read with interest the paper by Geiger, Dennis *et al.* in the journal's Volume 9, Number 2, pages 72–76: 'A method for determining total nitrogen in Kjeldahl digestion solution using a centrifugal analyser'.

Without wishing to detract from the value and considerable interest of this application of a centrifugal analyser, there are a few points which it is unfortunate that the authors did not comment on or note in their paper.

The digestion method using a small proportion of a strong oxidizing agent perchloric acid seems to have been taken from a 'continuous flow method', which was not designed for discrete digestion and analysis. There is considerable evidence that satisfactory digestion of even fairly refractory substances can be achieved without the use of perchloric acid (1) and it is now usually recommended that perchloric acid digestion should be avoided if at all possible because of the hazards of explosion. There is also considerable evidence that the use of selenium dioxide as a catalyst may lead to incomplete recovery of nitrogen (see [1]). This is also a hazard with temperatures above 400 °C if that was the temperature of the digest [1].

It seems to be a pity that the authors did not explore the possibility of using the rate of colour development to quantitate the ammonia, preliminary observations of which have been reported by Weichselbaum *et al.* [2].

Finally, the authors give no evidence of the recovery of the analyte from real samples.

Thus, although the authors show that, as might be anticipated, the centrifugal analyser can be satisfactorily used for the determination of ammonia in solution; I would strongly recommend that others wishing to use this procedure for determining ammonia in Kjeldahl digests should carefully check on the recommended conditions for the Kjeldahl digestion before following the details of the authors' procedure.

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References

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WEICHSELBAUM, T. E., HAGGARTY, J. C., MARK, H. B. JNR., *Analytical Chemistry*, **41** (1969), 848.

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