A NEW METHOD FOR DETERMINATION OF TOTAL NITROGEN IN MEAT AND MEAT PRODUCTS

HANS BERG

Swedish Meat Research Institute
PO Box 504, S-244 00 Kävlinge,
Sweden

SUMMARY
The Leco FP228 is a new instrument for rapid determination of total nitrogen in meat and meat products using the pyrolysis technique. The sample is combusted in oxygen according to Dumas.
The correlation coefficient between corrected Leco values and Kjeldahl values was 0.998 and the standard error of estimation 0.053 for samples containing 0.4-4.3% nitrogen.

INTRODUCTION
During recent years, the need for rapid methods of determination of nitrogen in meat and meat products has progressively increased. The lack of main emission lines for nitrogen in the UV-VIS range does not make it possible to use the most frequent methods of element analysis, namely atomic absorption spectroscopy. Because of this, a number of other methods of analysis are used. Some of the most important methods of determination of nitrogen are;

- Kjeldahl
  Since J. Kjeldahl first described a method of total nitrogen determination in 1883 (J. Kjeldahl, 1883) this has become the most commonly used and most widely accepted method for meat and meat products. In the Kjeldahl method, the sample is heated in concentrated sulphuric acid with salt and a metal catalyst. By adding alkali, organic nitrogen is converted to ammonia, distilled and measured. The method includes work with concentrated acid, alkali, salt and sometimes even toxic metal catalysts. Therefore, the procedure creates an unpleasant and hazardous atmosphere. The time of analysis is approximatly 1 hour.
  Near Infrared Reflectance Analysis (NIR)
  By using molucular spectroscopy in the near infrared region (1100-2500 nm), it is possible to measure the nitrogen content in many types of materials. The use of NIR-measurements on meat and meat products is still a problem due to low precision compared with other nitrogen methods. This lower precision depends partly on the fact that water has a high absorption in this area and therefore interferes with the measurement of other elements such as nitrogen. This problem does not exist in connection with analyses on low-moisture material like grain. The major advantage of this method is a short time of analysis. Results are obtained within 1 minute.
  A disadvantage is the time-consuming and complicated calibration.

In this paper, Leco FP228, a new instrument for nitrogen determination in meat and meat products is described and compared with the Kjeldahl method.
The new approach to nitrogen determination by Leco is the dry combustion of the sample at very high temperatures (Dumas, 1831) followed by nitrogen measurement in a thermal conductivity cell. Until now, it has not been possible to analyse meat products using this method due to very small amounts of sample, less than 100 mg (McKenzie, 1988). The Leco instrument makes it possible, using amounts of sample

down to 0.9 g. This amount is large enough to assure homogenous samples of meat products.

MATERIAL AND METHODS

In total 95 samples of meat and meat products have been analysed using both the conventional Kjeldahl and the Leco method. The samples consisted of beef and pork and different kinds of emulsion products with varying nitrogen contents (0.4 to 4.3%).

Sample preparation

The samples were cut into small pieces. The pieces were passed twice through a chopper with a plate opening of 2 mm and mixed after each grinding. The samples were stored in airtight containers at 4°C until analysis.

The Leco principle

Duplicate samples of 0.9 g were weighed into ten capsules. The weights are automatically transferred to the instrument. The capsules were transmitted into the Leco autosampler. Up to 20 capsules can be placed in the sampler. The sampler places the samples in the combustion chamber where they are combusted in excess oxygen at 1050°C.

The combustion gases are passed through a water absorption tube and then collected in a ballast volume of 4.5 l. 10 ml of the collected combustion gases are passed through water and carbon dioxide absorption tubes followed by a reduction tube containing copper at 650°C. The total elemental nitrogen of the sample is measured in a conductivity cell. Helium and oxygen are used as carrier-gases (Figure 1).

The nitrogen content in the sample is printed out on the built-in printer. The analysis time is about 3 minutes.

Unlike the conventional nitrogen methods, this procedure is free from hazard and harmless to the environment.

Reference method

All the Leco results were compared with the Kjeldahl results. Kjeldahl is the official method in Scandinavia (Annon, 1976).

RESULTS AND DISCUSSION

The correlation between Leco and Kjeldahl values in meat and meat products is shown in Table 1.

The samples contained 0.4-4.3% nitrogen, according to Kjeldahl. The Leco mean values are 0.016% of a unit higher than the Kjeldahl values. The standard error of estimation and the correlation coefficient indicates that the two methods are of almost equal value. A paired t-test showed a significant difference between the two methods at a confidence level of 95%.

This significant difference between the two methods probably depends on the fact, that the Kjeldahl method measures the protein 'nitrogen' while the efficient Leco combustion liberates all chemically-bound nitrogen.

If all the Leco values were reduced by 0.016% of a unit no significant difference between the two methods would be able to be detected using the paired t-test (Table 2).

The high water content in meat samples causes problems with moisture removal after combustion. This problem can probably be solved by using a water condensor instead of the first water absorption tube.

Figure 2 shows the linear regression line between Kjeldahl and reduced Leco values. The good agreement between the two methods is evident.

CONCLUSIONS

The following conclusions can be drawn:

1. The Leco method is very fast and harmless to the environment.
2. In comparison with Kjeldahl, the Leco nitrogen results were constantly 0.16% of a unit higher. The statistical calculations were made using the corrected Leco values.
3. The corrected Leco values were in close agreement with the Kjeldahl values.

REFERENCES

Annon (1976):
Nordic Committee on Food Analysis, no 6.

Dumas, I.B.A. (1831):
Ann. Chim. Phys. 47, pp 198-205.

Kjeldahl, J. (1883):
Z. Anal. Chem. 22, pp 366-382.

McKenzie, H.A. (1988):
Chapter 41 in: Nitrogen, Quantitative Trace Analysis of Biological Materials, (Eds) McKenzie, H.A. and Smythe, L.E., ELSEVIER, pp 689-706.

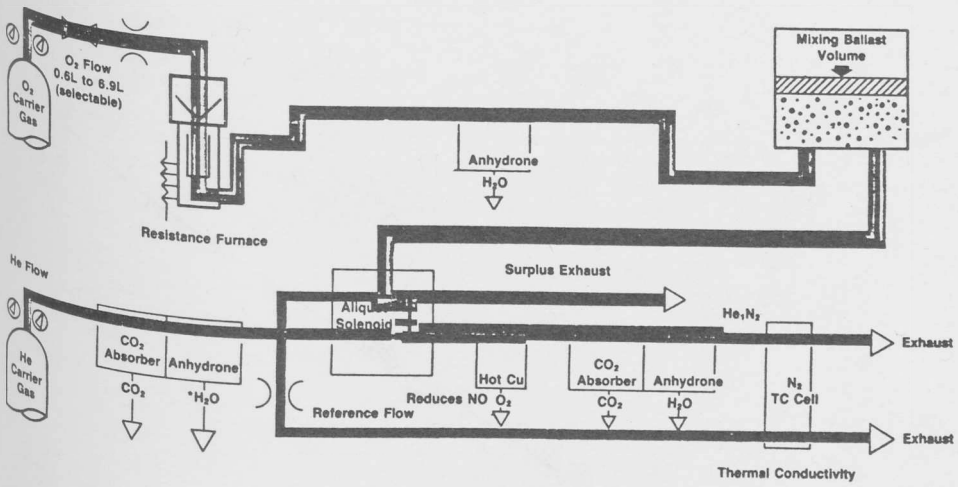

Figure 1. Leco FP228 Flow diagram.

Table 1. Statistical summary of Leco and Kjeldahl values.

Number of samples	Leco mean value	Leco min	Leco max	Kjeldahl mean value	Kjeldahl min	Kjeldahl max	Linear r	regression SEE	Paired t-test
95	2.478	0.406	4.326	2.462	0.461	4.302	0.998	0.053	XX

Table 2. Statistical summary of corrected Leco and Kjeldahl values.

Linear r	regression SEE	Paired t-test
0.998	0.053	-

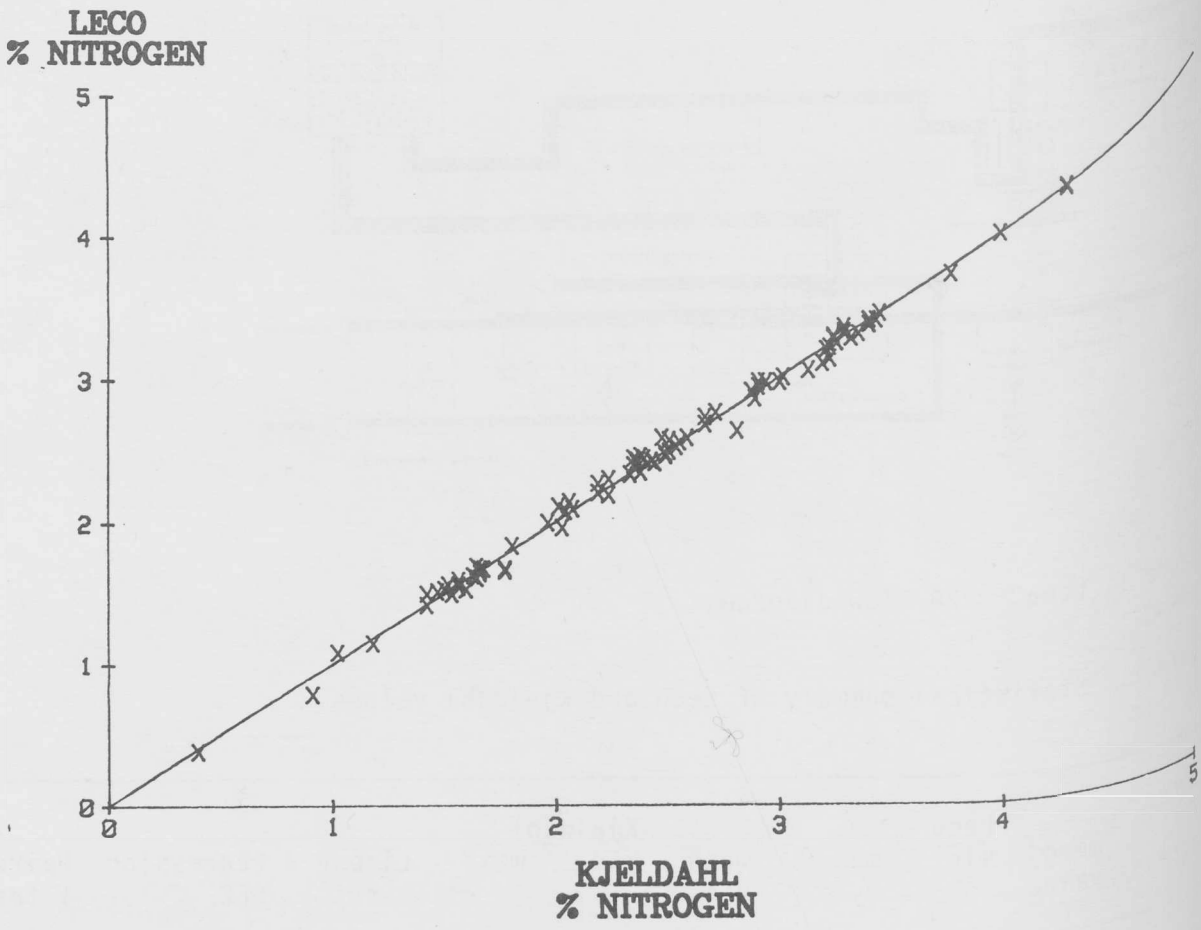

Figure 2. Nitrogen values, as obtained using the Leco and Kjeldahl methods, on the same samples.