Rapid Method for Determining Fat Content in Meat by Nuclear Magnetic Resonance

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SUMMARY: Rapid methods are needed for the in-line control of quality characteristics (e.g. fat or moisture content) of meat in the meat-processing plants. Recently, Nuclear Magnetic Resonance (Newport Analyser MK III.A) technique has been applied for determination of fat content in meat. The disturbing moisture of meat was removed by microwave drying, the dried residue was transferred quantitatively into the NMR-tube. The total time of determination needed approximately 35 minutes. Experiments were performed with different pork, beef, lard, tallow samples and with their combinations (lard-tallow; pork-beef; lard-pork; tallow-beef; lard-beef; lard-tallow-beef; lard-tallow-pork, etc.). It was established that the regression equations of pork and beef didn't widely differ, however, it was a substantial difference between the equations of lard and tallow. Moreover, both differed from the equations of pork, of beef and of meat-fat combinations. The variability in the fatty acid composition at the same type of fat may presumably influence the stability of the calibration curves. The sensitivity of the NMR-signal to the quality of the fat seems to interfere with the quantitative determination of the fat content in meat.

INTRODUCTION: The common methods for in-line quality control are either time-consuming or unreliable. Different instrumental techniques have been used to determine the fat content of food products (X-ray, near infra-red reflectance, nuclear magnetic resonance, etc.). Nuclear magnetic resonance (NMR) spectrometry can each provide rapid and simple method and it has been used succesfully for a long time in the Hungarian oil and confectionary industry (KŐVÁRI,1990). There are two types of instruments for analytical purposes: continuous wave (CW) NMR and pulsed (P) NMR.

The CW-NMR investigations of NILSSON et al. (1974) showed that even if the correlation was good there was a slight difference as the NMR-method in the mean gave an 0,3 p.c. higher fat content than the standard method in meat products. CASEY et al. (1974) measured the fat content of lean meat by NMR and established that there were linear relationships between the fat contents measured with NMR and those determined by two standard chemical methods; the residual standard deviations being 0,2 and 0,16 p.c. for total and extractable fat, respectively.

RENOU et al. (1985) applied pulsed NMR spectrometry to estimate the fat content in meat samples. The NMR results correlated well with chemical data, the mean standard deviation of the difference between calculated(NMR) and chemically assessed fat content was 0,8 p.c.

Later, RENOU et al. (1987) determined the fat and the moisture content of pork and beef by an "home built" pulsed NMR spectrometer. The NMR-ratios correlated closely with those determined by standard analysis (the correlation coefficient being 0,9977), but it was established that different fats may lead to a $-CH_2$-signal intensity error of around 1 p.c.

The aim of the present work was to determine how accurately and precisely could the NMR-technique predict the fat content in pork and beef.

MATERIALS and METHODS: The measurements were carried out in 24 different pork-samples and in 16 different beef-samples. Besides three different meat-fat mixtures were prepared:

- lean pork mixed with lard (about from 2 to 30 p.c. w/w),
- lean beef mixed with tallow (about from 1 to 25 p.c. w/w),
- lean pork mixed with lean beef (1:1) and also mixed with lard:tallow=1:1 (about from 1,5 to 30 p.c. w/w).

The relationship between NMR-signal and quantity of the rendered fat was investigated with lard, tallow and lard:tallow=1:1.

The measurements were carried out with a Newport Analyser MK III.A CW-NMR spectrometer using a 40 cm^3 sample tube. About 20 g of homogenized sample was dried in a microwave oven (23 minutes). The dried residue was then transferred quantitatively into the NMR-tube. The fats were dehydrated with desicc. Na_2SO_4. The NMR-signal was obtained in 2-3 s. Results obtained with NMR-technique (calibrations with pork, beef, lard, tallow, lard-tallow, pork-beef, lard-pork, etc.) were compared with those determined by Soxhlet extraction.

RESULTS and DISCUSSION: Calculations were carried out with the help of Deming's regression (ref. KÖRMENDY et al., 1989). Results are shown in Table 1.

Calculations showed that the relations presented in Table 1., - except no.6. (beef-tallow mixture), could be described with the $\hat{y}=b\hat{x}$ equation, namely the regression lines started from the origin. The slopes (b) of the different regression equations were compared by Student's t-test (HALD, 1962), and results are summarized in Table 2.

As it can be seen from the Tables 1., 2.:

- The slopes (b) of pork (1), of lard (3), and of pork-lard mixtures (5) differ significantly, however, the difference in the slopes of lard (3) and of pork-lard mixtures (5) is not substantial.
- The comparison of the slopes (b) of beef (2), of tallow (4) and of beef-tallow mixtures (6) showes that they do not differ considerably. However, the intercept of equation(6) differs significantly from 0.
- By comparing the slopes of lard (3) with tallow (4) it can be seen, that the values of "b" differ significantly and considerably.
- The calibration curves of pork (1) and beef (2) don't differ significantly.
- The regression equations of meat-fat mixtures (5,6,8) differ significantly.

CONCLUSIONS:

- There is an interaction between the fat and other components of meat, because the calibration curves of rendered fat, of meat-fat mixtures and that of meats differ considerably.
- The fatty acid composition of fats could influence considerably the NMR-signal. This fact is verified by the comparison of lard and tallow, and for this reason, the stability of calibration curves for pork and beef are also questionable. The fatty acid composition (ratio of saturated and unsaturated fatty acids, lenght of aliphatic chains) varies with the type of fat (beef or pork), with the diet and with the anatomical location, too (SZEREDY, 1956).

RENOU (1987) mentioned that different fats may lead to a NMR-signal intensity error of around 1 p.c., but according to his opinion this error falled within the accuracy of the method. This hypothesis should be confirmed by further experiments.

- The regression equations of pork and beef don't differ considerably, however, the calibrations with the mixtures of pork+fat and beef+tallow deviate essentially from the formers. Therefore, it could be concluded, that the CW-NMR technique can't be used for determining the fat content of pork and beef mixtures. So, this technique may only be suitable for pure pork or pure beef. However, the stability of the calibration curves must be verified later, because of the systematic changes which may occur in the chemical composition of fats even of the same origin.

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TABLE 1.: Results of the calculations according to Deming

Sample no.	S a m p l e	R a n g e (g fat per appr. 20 g of sample)	n	a	b	V{b}
1.	Pork	0,3370 - 4,1530	24	0	4,543	0,0022
2.	Beef	0,1230 - 3,2200	16	0	4,641	0,0032
3.	Lard	0,6130 - 8,0127	5	0	5,353	0,00010
4.	Tallow	0,7286 - 12,1654	5	0	4,400	0,00011
5.	Pork-lard mixture	0,4030 - 6,2330	14	0	4,915	0,0021
6.	Beef-tallow mixture	0,1500 - 5,8670	14	1,044	4,553	0,0088
7.	Lard-tallow=1:1	0,2321 - 6,0542	7	0	4,768	0,00062
8.	Pork-beef=1:1 + lard-tallow=1:1 mixture	0,3230 - 6,3200	14	0	5,248	0,00061

"a" and "b" = constants of $\hat{y}=a + b\hat{x}$;
V{b} = variance of "b"
n = sample size

TABLE 2.: Comparison of the slopes with Student's t-test

×	1	2	3	4	5	6	7
1	×	×	×	×	×	×	×
2	NS	×	×	×	×	×	×
3	S	S	×	×	×	×	×
4	NS	S	S	×	×	×	×
5	S	S	S	S	×	×	×
6*	S	S	S	S	S	×	×
7	NS	NS	S	S	NS	S	×
8	S	S	NS	S	S	S	S

S - significant difference ($\alpha < 0,05$)
NS - no significant difference ($\alpha > 0,05$)
α - level of significance
* - "a" differs significantly from 0.