THE INVESTIGATION OF SIX METHODS FOR THE RAPID DETERMINATION OF SALT IN MEAT AND MEAT PRODUCTS

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Summary

Methods for the rapid determination of salt in meat and meat products have involved the measurements of electrical conductivity, electrical resistivity, and the use of the chloride electrode. The possibility of using techniques involving radioactive substances has also been investigated.

The results indicate that electrical conductivity and resistivity can be related to the salt content of meat, but that the relationship was affected by a number of factors, including moisture, and fat content. If these factors are reasonably constant for any one product then an instrument which can measure conductivity (or resistivity) can be used for the routine quality control of salt content.

Broadly similar conclusions were arrived at from the studies involving the use of the chloride electrode.

Introduction

The growth of bacteria can be inhibited by salt, and the beneficial effect of adding salt to foodstuffs has been utilized throughout historical times. In meat products salt is used to inhibit the growth of both spoilage organisms and pathogens thus increasing the microbiological stability and safety of these products. The inhibitory effect of salt is related to its concentration.

A second function of salt in meat products is that of flavour, i.e. saltiness, and in this case the concentration required for flavour will depend on the consumer's preference. However, with any single product, the consumer will expect the same degree of saltiness each time the product is purchased. This need for uniformity is becoming of increasing importance with the increase in the retailing of prepacked materials under branded names.

Thus, since the consumer' requires both microbiological stability and safety, and uniform saltiness, the necessity of carefully controlling the salt content becomes obvious.

In most manufacturing processes, the amount of any ingredient in the final product can be controlled by the addition of a known weight of the ingredient. However, this is only true if the ingredient is readily dispersible or is soluble in the product. This is not always possible, as in the case of the salt content of certain canned meat products, in which the salt uptake has taken place either via a cover brine or from a site of injection. The salt uptake is affected by a number of factors, including the intrinsic properties of the meat, which are subject to considerable variation. This variability in meat makes it difficult for the salt content of the final product to be predicted withany accuracy, and it therefore can only be determined in the end product.

A considerable number of analytical methods have been developed for the determination of salt, most of them depending on an extraction procedure followed ba determination as chloride. Such methods are destructive, time consuming, and require a certaine amount of experimental skill. The ideal alternative would be an inexpensive, rapid, preferably non-destructive, accurate method requiring little or no technical skill. In order to meet these specifications some from of instrumentation will obviously be required. Although such a method would be the final objective, it does not preclude the consideration of modifications of established methods which could lead to an increase in the ease and speed of carrying out salt determinations.

It is not the purpose of this paper to review the different methods which have been developed for determining salt concentration, nor to discuss the merits of the various ways in which the results can be expressed, but to describe some investigations which have been carried out at the B.F.M.I.R.A. and which have taken the form of an assessment of a number of instruments which were either specifically designed for or which could be used for salt determination.

The object of the work was to study those easily measurable parameters of meat products which can be related to their salt content. It was not the intention to carry out a comprehensive study of each individual method for every possible product, but to demonstrate the feasibility of the particular approach and to provide some support for its practical application.

The methods which have been investigated include conductivity and resistance measurements, electromagnetic radiation absorption, and the use of the chloride electrode.

A pre-requisite for the investigation of these methods is the possibility of producing a suitable test sample of uniform salt and moisture content which can be used to provide a series of test samples. The preparation of such a material was carried out by mincing lean raw meat freed from visible fat through a 4.8 mm plate. A known weight of the minced meat was placed in a mixing bowl, the requisite amount of salt was added and the whole mixed for five minutes with a mechanical stirrer. The efficiency of this procedure is demonstrated in Table I which shows the salt content, determined by the Volhard method, and the moisture content, determined by loss on drying at 105°C, on five samples taken at random from the mix.

TABLE I
Variation in Salt Content of Minced Pork containing Added Salt

Sample	% salt	% moisture	% salt/100 g moisture
A	5.08	69.8	7.29
B	5.13	69.8	7.36
C	5.11	69.2	7.39
D	5.05	69.6	7.27
E	5.09	69.9	7.28

The F.I.R.A. Salinity Tester

This instrument is based on the original salinity tester developed by B.F.M.I.R.A.

Basically it consists of a probe and a measuring instrument. The probe consists of two hardened steel rhodium tipped spikes set a fixed distance apart. Provision is made for measuring the depth to which the probe can be inserted into the material under test. The measuring instrument consists of a low frequency oscillator and a resistance bridge circuit coupled to a shock-proof galvanometer. The instrument as used records a current on A and has not been calibrated to read salt content directly.

Calibration of the instrument was carried out by placing 5 cm cubes of fresh pork into a series of brine solutions of strengths ranging from 1 to 7%, these brines then being covered to prevent loss of moisture. They were then allowed to stand for 8 weeks at 45°F.

The cubes were then carefully wiped and their resistance measured, using three cubes from each brine and making several determinations on each. The plots of the instrument readings against (a) % salt, and (b) % salt per 100 g water showed that the scatter of the respective points was of the same order in each case, suggesting, contrary to earlier findings, that the instrument could be used for measuring either perameter.

Experiments were then carried out using bacon as the test material to confirm the validity of the calibration, and to determine which salt parameter did in fact give the better correlation with instrument readings. Table 2 records the data obtained.

TABLE 2
The Salt Content of Bacon determined by
the F.I.R.A. Salinity Tester

Sample	F.I.R.A. Salinity Tester			Chemical Analyses	
	Mean reading (A)	% salt	% salt/100g water	% salt	% salt/100 g water
A	95.50	5.05	5.85	4.97	6.22
B	85.00	3.25	3.90	4.12	5.31
C	84.17	3.15	3.15	4.01	5.25
D	93.20	4.60	5.35	4.85	6.30
E	94.17	4.75	5.55	6.92	9.18
F	93.00	4.60	5.35	4.93	6.23
G	88.82	3.85	4.50	6.18	8.31

Inspection of the data indicates that agreement between the instrumental and chemical methods is not particularly good, reasonable agreement only being obtained in three (A, D and F) of the seven samples investigated. Thus on the whole, the data do not support earlier claims that results obtained with this instrument are within 0.6% of those determined by chemical means.

In general, instrumentally determined results were lower than those determined chemically. This lack of agreement cannot be due to the presence of nitrate in the bacon since it is known that this gives rise to higher instrument readings. Hence any correction for the nitrate content would only reduce the values still further.

Subsequent experiments, in which the technique of measurement was modified, and in which the material used for chemical analysis was taken from the vicinity of the probe, showed less variation in the individual test samples but consistently lower instrumental estimates for salt content were still obtained.

It must be concluded therefore that the method used for the calibration of the instrument was not valid when used to determine the salt content of bacon. A possible alternative for obtaining a valid calibration curve may however be obtained by using data such as occurs in Table 2 as calibrant data.

One explanation of the non-applicability of the calibration curve when used for determining the salt content of bacon is the difference in moisture content. This was investigated by preparing protein water slurries of constant salt content but which the moisture content varied from 59 to 74%. Table 3 records the data obtained.

TABLE 3

The effect of the moisture content of protein slurries on their apparent scale content

Samples No.	% NaCl added	% H_2O	% NaCl/100 g H_2O	Mean instrument reading
1	2.5	59.0	4.24	70.0
2	2.5	64.9	3.85	81.4
3	2.5	69.3	3.61	81.8
4	2.5	75.0	3.33	83.8
5	2.5	74.1	3.37	83.8

This table clearly shows that the response of the salinity tester is markedly influenced by the moisture content.

Previous work had indicated that sides of bacon with a higher fat content gave higher readings than those with lower fat content though the salt content was the same in both cases. Furthermore it was found that there was an appreciable difference in the instrument reading, depending on the position of the probe in the material, e.g. side, under test.

It is well known that electrical resistance varies with temperature. It follows therefore that unless an instrument is equipped with a temperature compensating device its usefulness will be severely limited.

It is clear therefore that a number of considerations have to be taken into account when using the F.I.R.A. Salinity Tester in order that meaningful results may be deduced from any data obtained.

The Radiometer Salt Tester Type CDM 21

The CDM Salt tester (V. Howe & Co. Ltd., London) comprises a single needle shaped probe coupled with a conductivity meter calibrated to give a direct reading of salt concentration up to 5%. A temperature compensating device is built in, which allows measurments to be made over a temperature range from -5°to +25°C. It was borne in mind however that the instrument makers emphasize that the calibration operates inly for Wilshire type bacon muscle and that only comparative results can be obtained when the instrument is used with other types of meat products.

In preliminary experiments which were intended to confirm the carrelation between instrument response and salt content of the meat, only relative measurments of the different salt -meat mixtures used were required.

Determinations carried out at 5°C and 17°C showed that there was very little difference between the response readings at either temperature, indicating the efficiency of the temperature controlling device. Further it was found that the relationship between instrument response and salt content was linear, this instrument therefore having an advantage over the F.I .R.A. instrument in this respect which not only has a non-linear relationship but also suffers from poor sensitivity at high salt levels. The observation that there is a linear relationship between the instrument reading and the salt content over the range investigated is of considerable interest since the calibration factor used was derived according to the manufacturers recommendation using Wiltshire sides and not comminuted meat. This suggested that differences between uncomminuted meat and comminuted meat which were found in the case of the F.I.R.A salinity tester did not greatly affect the response of the instrument, and indicated that the recommended calibration factor may not be as limited as the manufacturers had originally supposed.

The influence of moisture content on instrument response was investigated by using protein slurries. From this it was found that, as in the case of the F.I.R.A. salinity tester, the instrument response increases with increase in moisture content. It must be concluded therefore that the validity of any determinations carried out with this instrument will depend on the moisture content of the sample. Nevertheless, salt determinations were carried out on a range of products, a summary of the results being given in the following table:

TABLE 4

Salt content determinations of cured meats using the CDM 21 Salt Tester

Product	Salt content	Chemical analyses	
		% salt	% water
Smoked Wiltshire Shoulder	3.03	3.63	68.6
	4.18	5.27	66.8
	3.30	3.43	71.7
	4.38	4.91	72.4
	3.40	3.88	71.1
Dutch backs	3.5	3.10	-
	3.7	3.15	-
	5.0	4.28	-
	6.5	5.46	-
	7.0	5.42	-
	7.5	7.38	-

Wiltshire sides	2.6	3.41	54.9
	3.6	4.29	56.9
	3.9	4.26	54.4
	4.3	5.39	44.7
	3.7	4.56	52.6
Ham	1.8	2.06	71.6
	2.0	2.12	71.3
	2.4	2.39	75.5
	2.8	2.77	72.6
	3.1	2.49	73.3
	3.5	3.10	73.6
	4.0	3.24	72.9

Inspection of the data given in the table indicates that a correlation exists between the values for the salt content determined instrumentally, and by chemical means. From a statistical analysis of the complete data it can be shown that the 95% confidence limits for any insgrument reading irrespective of the product being investigated is of the order of ± 1%.

The results obtained using Wiltshire sides are of considerable interest. These sides were tested during storage at 21 °C and it was anticipated that the low moisture contents of the sides would give rise to errors in the instrumental determinations. It is seen that in all cases the instrumental value is lower than the chemical value (by an average of 0.6%). These results are taken to illustrate the point that although low meter readings were obtained for samples with low moisture contents, there was a good correlation between meter reading and salt content, thus confirming the manufacturer's claim that the instrument can be used to obtain relative readings for comparable products other than Wiltshire bacon. Further, since the calibration of the instrument can be altered by adjustment of the electrode calibration constant it should be possible to adjust the instrument to give a direct reading for salt content in any meat product.

It would appear therefore that variations due to the effect of interfering factors would preclude the use of the instrument for the accurate determination of salt content in meat products, but it would clearly be of value for obtaining a good indication of the salt level and distribution of salt in such products.

The Radley Semiconductor Resistivity Bridge

This instrument is comprisedof a probe and bridge circuit, the probe having four contacts. A low frequency square wave is applied to the outer pair of contacts. The voltage drop across the outer and inner contacts which occurs when the probe is in contact with the sample of test material will depend on the resistance of the material. The resistance, measured by balancing the voltage between the inner and outer contacts, represents a direct reading of the resistivity of the sample.

The possibility of using this instrument for determining the salt content of meat products was investigated altough it was realized that the instrument would probably have the same disadvantages as the F.I.R.A. salinity tester and the CDM 21 salt tester.

The response of the instrument was determined by immersing the probe to a constant depth in a solution containing increasing amounts of sodium chloride.

TABLE 5
Response of Resistivity Bridge to Sodium
Chloride Concentration

% Na Cl	Meter Reading
1	17.00
2	11.90
3	8.70
4	6.95
5	5.65
6	4.70
7	4.00
8	3.20

It is seen that as with the F.I.R.A. instrument, there is no linear relationship between meter reading and salt content, and the sensivity decreases rapidly as the sodium chloride concentration increases.

In order to test the possibility of using this instrument to determine the salt content of meat and meat products, a simple correlation test was carried out using bacon, smoked and green bacon being used to demonstrate also the influence of moisture content.

A total of 8 readings per sample were taken, the mean found, and this corrected by a factor depending on the thickness of the sample and the spacing between the probes. The results obtained were as follows:

TABLE 6
Instrument response to bacon of known
salt content

Sample No.	Corrected mean instrument reading	% salt per 100g meat	% H_2O	% salt per 100 g H_2O
1	20.8	4.48	64.20	6.98
2	23.0	4.97	64.43	7.71
3	22.4	4.29	63.81	6.72
4	15.0	4.82	68.88	7.00
5	15.4	5.28	67.51	7.82
6	16.7	4.90	68.71	7.13

It is clear from the above data that there is no obvious correlation between the meter reading and the salt parameters quoted.

Two points of interest emerged from these data.

(1) Samples with a high moisture content gave lower readings than samples with lower readings than samples with lower moisture contents. This is in agreement with data obtained for both the F.I.R.A. and the Radiometer tester.

(2) There is a wide scatter in the estimate of moisture within each group.

If this variation is indicative of the variation in resistance of the meat, then any method for determining salt which depends on resistance or conductivity methods will be subject to variations of this order.

It can be concluded therefore that the Radley Semiconductor Resistivity Bridge will be of very little value for determining the salt content of meat.

Xray Fluorescence Analysis

This technique depends on the emission of Xrays when a sample is excited by a radioactive source. The Xrays emitted from each particular element.

It was suggested that this technique might be useful for determining the amount of salt in meat by determining the amount of chlorine present. However, when this was attempted, it was found that the characteristic Xray energy of chlorine was too low to permit detection. Hence it may be unlikely that this technique can be developed to produce a rapid method for the determination of salt.

Electromagnetic Energy Absorption

This technique depends on the preferential absorption of soft electromagnetic radiation by elements of higher atomic number. Since chlorine has a higher atomic number than carbon, oxygen, hydrogen and nitrogen, the major component elements in meat, then it should be possible to estimate the salt content by measuring the preferential absorption due to this element.

The instrument used in this investigation was the 147L Laboratory Analyser (Nuclear Enterprises Ltd.) which had previously been used for determination of the fat content of meat. The instrument contains a radioactive source (promethium 147, 1/2 life period 2.6 years) which emits radiation, with peak energy of 20 KeV. When this radiation strikes a zirconium target, low energy X radiation passes through the meat sample, some being absorbed by the sample, the remainder being determined by a Geiger-Müller tube.

Results are initially expressed in terms of absorption ratios, these being calculated from the count rate per unit time of a standard, and the count rate from the specimen under test.

By comparing the absorption rations obtained using known weights of materials containing high and low salt contents it was possible to determine the optimum sample size to be used on the instrument.

From the correlation studies which were carried out, the relevant data for which are recorded in table 7, it was shown that a linear correlation existed between the absorption ratio and the percentage of salt in meat, expressed either in terms of % salt per 100 g meat, or % salt per 100 g water. This suggests that the method could be used for estimating either parameter.

TABLE 7
Determination of % salt in salt - meat mixtures using the 147L Laboratory Analyser

Sample No.	% NaCl	% H_2O	% NaCl per 100 g H_2O	Absorption Ratio
1	0.26	73.7	0.35	0.715
2	1.25	72.7	1.72	0.709
3	2.20	86.1	3.12	0.689
4	2.77	72.3	3.83	0.667
5	4.14	71.1	5.32	0.638
6	5.13	65.5	7.83	0.601
7	5.90	70.1	8.22	0.590
8	7.45	68.8	10.82	0.553

However it had previously been shown that in the determination of fat in comminuted meat, this application is, in effect, a determination of the moisture content, based on the preferential absorption of radiation by oxygen in water molecules. It follows from this that variation in the moisture content of the test sample would have an effect on the chloride being determined.

The effect was investigated using a series of meat samples containing an increasing amount of fat, but maintaining an approximately constant salt level, the absorptions then being determined in the normal way. Table 8 records the results obtained.

TABLE 8
Effect of Moisture Content on the energy absorption by meat-salt mixtures

Sample	% added fat	% NaCl	% H_2O	% NaCl per 100g H_2O	Absorption Ratio
A	0	5.09	70.3	7.24	0.583
B	10	5.13	64.2	7.99	0.618
C	20	5.22	58.6	8.90	0.643
D	30	5.10	51.2	10.18	0.650
E	40	4.92	23.9	11.20	0.710
F	50	5.10	38.9	13.10	0.687

The above table shows that although the salt content remained constant, the absorption ratio, and the % salt per 100 g H_2O increased as the moisture content decreased.

Thus although the results of the investigation showed that there is a direct relationship between the salt content of comminuted meat and the amount of energy absorbed, it has also been shown that the amount of energy absorbed is dependent on the moisture content.

Clearly, however, reproducible results can be obtained in the case of various types of products in which the moisture (and fat) contents remain practically constant. This restriction however at the moment excludes the method from consideration as a rapid means of determining salt content universally in meat and meat products. The possibility of developing the technique in such a way as to allow for the simultaneous determination of salt and moisture should not be overlooked.

The Chloride Electrode

The Orion Chloride electrode model 96 - 17 (E.I.L. Ltd., Richmond) was used in this investigation in conjunction with a Pye Model 290-pH meter (W.G. Pye Co.Ltd.Cambridge).

The electrode consists of a reference, and a solid state chloride sensing electrode in one body. The solid state sensing element consists of a solid silver halide membrane which separates the sample solution from the internal filling solution, the latter containing 1.0 M KNO_3 and 0.1 M KCl, saturated with silver ions by the addition of 0.1 M $AgNO_3$. The potential between the internal solution and the internal reference is fixed, as is the potential between the filling solution and the inside surface of the membrane.

Changes in electrode potential are due to changes in the Ag^+ activity.

Although the test solution may not originally contain silver ions a small number will be provided by the extremely low solubility of the silver halide membrane. The activity of these silver ions will depend on the activity of the chloride ions present in the test solution, and can be calculated from well-known electrochemical equations, and from these an estimate of the salt content of the test material can be made.

In use, the combination electrode is connected to a suitable millivoltmeter, the electrode being immersed in the test solution on placed on the test sample, the resultant potential being measured.

The relationship between electrode response and chloride concentration was established by immersing the electrode in solutions of increasing salt concentration and a subsequent plot of log salt concentration and electrode potential was shown to be linear. The same experiment carried out using salt-meat mixtures gave an identical plot. These results indicate therefore that the chloride electrode can be used to determine directly, either the salt or the moisture content of meat, provided that the appropriate calibration charts are used.

Sine however the relationship between the parameters is logarithmic, only small changes in salt content at the higher levels. Thus the difference between 9.0 and 0.5% salt corresponds to an instrument reading of lmV, and since differences between two sets of readings may well be of this order of magnitude, although the instrument can be used satisfactorily over the lower salt ranges, it is likely that this sensivity restriction will render the instrument less useful at the higher salt levels.

By investigating salt solutions of concentration ranging from 1 - 10%, and containing phosphate buffer to give solutions of pH values ranging from 5 to 6.5, it was shown that instrument response was independent of pH value. Likewise, similar experiments carried out in the presence of nitrite and of nitrate ions indicated that instrument response was independent of either of these ions.

Certain cured products, e.g. sweet cure bacon can contain sugar, and the possibility arises of the sugar reducing the water activity in such a product, thereby causing an apparent increase in salt content. Investigation of the effect sugar, using model systems, swowed that there was a small increase in response with increase in sugar concentration, but since the amount of sugar incorporated into a meat product will be small, the presence of sugar will be unlikely to give rise to significant errors.

The chloride electrode was then used to determine the salt content of sliced bacon, by direct application of the electrode to material.

Triplicate readings were taken along the eye muscle, which was then dissected out for the chemical determination of salt.

TABLE 9
Determination of the salt content of bacon obtained by
using the chloride electrode

Product	Replicate No.	Mean Electrode Value	% NaCl/100g H_2O
Sweet cure bacon vacuum packed	1	5,5	4.83
	2	5.9	4.86
	3	5.9	4.75
	4	6.1	4.72
	5	6.1	4.62
Dutch collar	1	10.3	10.2
	2	10.8	10.6
	3	12.1	10.4
	4	11.5	10.0
	5	11.6	10.1
Mild cure Vacuum packed	1	9.0	9.1
	2	8.8	8.6
	3	8.4	8.8
	4	9.0	9.1
	5	9.3	9.2
Mild cure back vacuumpacked	1	7.4	6.8
	2	8.2	7,3
	3	8.0	6.9
	4	8.1	6.9
	5	8.1	7.1
Best back	1	10.0	8.0
	2	10.6	9.5
	3	10.3	8.6
	4	11.0	9.8
	5	11.0	9.3

It is seen that good agreement was obtained in 2 samples out of the 5, the electrode giving high results in the other 3.

It was considered that this was due to there being higher concentrations of salt a the surfaces of the samples and this was investigated by carrying out tests on the surface of t slices followed by dissection, mincing, and testing the resultant mince. The results showed that in general, surface determinations give higher results than those from the minced samples, but the values obtained from the minces samples remained higher than those obtained by chemical analyses. This tends to suggest that chloride electrodes cannot be used satisfactorily for the direct determination of salt using sodium chloride solution as thestandard reference.

It can also be deduced from the data obtained that the differences between the el ctrode method and chemical method of determination are not constant.

Thus although preliminary work indicated that there was a direct correlation between electrode potential and % salt as expressed as % per 100 g water, subsequent correlation studies indicated that this was not valid. In general, the electrode gave high estimates of salt content both with intact samples and when used with minced samples. These differences between chemical and electrode estimates, occur both between and within the products tested. The reason for this discrepancy is not known, but it has been suggested that it may b due, to some extent at least, to the fact that the electrode measures activity and not concer tration, and that on these systems these parameters may not be directly related.

Although the electrode cannot be applied universally to the direct determination of salt in meat and meat products, it way well find a role in routine quality control for com rable measurments on samples of the same product, at least in those cases in which the differences between the electrode estimation and chemical estimation is reasonably constant.

Conclusions

Methods based on conductivity are more reliable than those based on resistance measurements since there appears to be direct relationship between this parameter and the salt content of the meat. The Radiometer CDM 21 Salt Tester is capable of giving values wit in ± 1.0% of those obtained by chemical analysis.

Of the other methods investigated, use of the chloride electrode gave the most promising results, but limitations of this method involve the problem of calibration when it i to be used with different meat products. Despite this, it is suggested that the method can be used for routine quality control for any particular product, providing that in that product, other interfering substances are kept fairly constant.

Although salt cannot be measured directly using radiation absorption techniques the possibility arises for the development of a method which could give the simultaneous determination of moisture and salt content of comminuted meat.

The overall results demonstrate that the ideal of a direct rapid method which can find universal application for the determination of the salt content of all meat and fish products is unattainable at present. It is possible however that this may be attainable in the future when further developments in Xray fluorescence techniques have taken place.