## Determination of chloride in meat products

OCTAVIO VENEGAS and GUSTAVO ANDUJAR

Food Industry Research Institute, Havana, Cuba

Mohr's method for the determination of chloride gives excessively high results when applied to meat and meat products. However, it is simpler and faster than the Volhard procedure, which is generally accepted as the standard method.

In this paper a comparison is made between results obtained by both methods on samples of 11 assorted meat products, the chloride contents of which ranged from 1,20 to 4,64 %, expressed as NaCl.

Mean differences between methods for each product varied from 0,17 to 0,67 % NaCl. Results were compared by the sensitivity coefficient method, and the corresponding regression equation was obtained, from which a correction equation was derived:

$$\% \text{ NaCl (Volhard)} = -0{,}20 + 0{,}94 \ (\% \text{ NaCl (Mohr)})$$

The effect of several components of sample extracts on the observed interference is considered, as well as the practical consequences of these results, with regard to the precision of the estimation.

It is concluded that, although the value of the sensitivity coefficient is rather low (0,328), it is possible to use Mohr's method as an alternative for a simple, approximate estimation of the chloride content, provided that the results are conveniently corrected.

## Bestimmung des Chloridgehaltes in Fleischprodukten

OCTAVIO VENEGAS und GUSTAVO ANDUJAR

Institut für Forschung der Lebensmittelindustrie, Havanna, Kuba

Die Mohrmethode ergibt höhere Werte, wenn sie bei Fleisch und Fleischprodukten angewendet wird, hat aber zweifellos Vorteile in Bezug auf Einfachheit und Schnelligkeit im Vergleicht zur Volhardmethode, die allgemein als Standard angenommen wurde.

Diese Arbeit vergleicht die Resultate beider Methoden von Proben aus 11 verschiedenen Fleischprodukten, deren Chloridgehalt, ausgedrückt als Sodiumchlorid, zwischen 1,20 und 4,64 % schwankte. Die Mittelwertsdifferenzen pro Produckt und Methode schwankten zwischen 0,17 und 0,67 % NaCl.

Die Resultate beider Methoden verglich man durch die Methode des SensibilitÄtskoeffizienten, daraus leitete sich die entsprechende Regressionsgleichung ab und von dieser die Korrekturgleichung

$$\% \text{ NaCl (Volhard)} = -0{,}20 + 0{,}94 \ (\% \text{ NaCl (Mohr)})$$

Die Effekte der verschiedenen Extraktkomponenten wurden hinsichtlich der beobachteten Störung und der praktischen Verwicklungen der Vergleichresultate vom Standpunkt der SchÄtzungsprÄzision diskutiert und man ist zur Schlussfolgerung gekommen, dass die Mohrsche Methode als schnelle Alternative für annähernde SchÄtzungen angewendet werden kann, obwohl der gewonnene Wert für den SensibilitÄtskoeffizienten relativ niedrig ist (0,328).

5.12

## Determination du contenu de chlorure dans les produits de viande

OCTAVIO VENEGAS et GUSTAVO ANDUJAR

Institut de la Recherche pour l'Industrie Alimentaire, La Havanne, Cuba

La méthode de Mohr donne des résultats excessivement hautes quand on l'emploie en viande et produits de viande, mais elle présent des avantages de simplicité et rapidité par rapport a la méthode généralment acceptée comme réference, celle de Volhard.

On compare dans ce travail les résultats obtenus par tous deux méthodes dans les échantillons d'once produits de viande différents, dont les contenus de chlorure oscillent entre 1,20 et 4,64 %, exprimés comme NaCl.

Les différences moyen par produit entre méthodes oscillent entre 0,17 et 0,67 % NaCl. On a comparé les résultats par la méthode du coefficient de sensibilité, obtenant l'equation de regression correspondant, et, a partir d'elle, l'equation de correction:

$$\% \text{ NaCl (Volhard)} = -0{,}20 + 0{,}94\ (\% \text{ NaCl (Mohr)})$$

On discute les effets de quelques composants des extraits des échantillons sur l'interference observée, et les implications pratiques des résultats depuis le point de vue de la precision de l'estimation. On arrive a la conclusion qu'il est possible, malgré la valeur relativement basse du coefficient de sensibilité (0,328), appliquer la méthode de Mohr comme méthode alternative, simple, pour estimations approchées, avec la correction nécessaire.

## Определение содержания хлористого натрия в мясных продуктах

ОКТАВИО ВЕНЕГАС и ГУСТАВО АНДУХАР

Исследовательский институт пищевой промышленности, Гавана, Куба

Определение хлористого натрия в мясе и мясных продуктах по методу "Мор" даёт чрезмерно высокие результаты, но несомненно представляет некоторые преимущества по быстроте и простоте в сравнении с методом "Волхард".
В этой работе сравниваются результаты, полученные по обоим методам. Образцы взяли из 11 разных мясных продуктов, у которых содержане хлористого натрия находилось в пределе 1,20–4,64%.
Средняя разница по продукту между методами колеблется между 0,17–0,67% NaCl.
Результаты обоих методов сравнивались по методу чувствительного коэффицента, получая соответствующее регрессивное уравнение, и из него поправочное уравнение:

$$[\text{NaCl}]\ /\text{Волхард}/ = -0{,}20 + 0{,}94\ [\text{NaCl}]\ /\text{Мор}/$$

Обсуждаются эффекты различных компонентов экстрактов на наблюдаемую интерференцию и практические результаты предлагающегося исправления с точки зрения точности оценки.
В заключении можно сказать, что,несмотря на низкий коэффициент чувствительности/0,328/, можно использовать метод Мора для быстрого определения хлористого натрия в мясных продуктах.

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OCTAVIO VENEGAS and GUSTAVO ANDUJAR

Food Industry Research Institute, Havana, Cuba

## Introduction

The traditional volumetric methods of Mohr and Volhard are still today those most commonly used for the determination of chloride content. Volhard's method, with eventual variations, is the standard method almost universally accepted for this determination in meat and meat products (ISO, 1970; AOAC, 1970).

Mohr's method, on the other hand, has the advantages of a simpler procedure and greater speed, although its application to meat and meat products is limited by various inconvenient interfering factors.

The interference of proteins and related substances in $AgNO_3$ titration was reported long ago (Dyer, 19[illegible], and is particularly noticeable in neutral or slightly alkaline solution, as used in Mohr's method. Earlier attempts have been made to overcome this inconvenience by correcting the results, but no thorough study in this sense has been reported, and the approach is sometimes too simple (Rudischer, 1974). The absolute error of the titration would be expected, firstly, not to be constant throughout, but to increase with the chloride concentration of the sample and secondly to include a stochastic component, as the quantity and quality of the interfering substance depend on sample composition and not solely on chloride concentration.

In this paper both methods, Mohr's and Volhard's, are compared in order to derive a correction equation whereby acceptable results may be obtained more simply and quickly, using Mohr's method.

## Material and methods

All samples were homogeneized by passing them three times through a meat mincer provided with a 3 mm plate, mixing thoroughly after each operation. Homogeneized samples were stored in completely filled, tightly closed bottles, at about -18°C until used.

Analyses by the Volhard method were carried out according to the standard ISO procedure (ISO, 1970). A 10 g sample is extracted with water over a boiling water bath. The extract is deproteinized with potassium ferrocyanide and zinc acetate. The pH is adjusted to 7,5-8,3, and after convenient dilution to a given volume, it is filtered. An aliquot of the filtrate is acidified and made to react with excess $AgNO_3$, and this excess is back titrated with 0,1 N KSCN, in the presence of ferric ammonium sulphate.

The Mohr procedure established in the standard for the Cuban meat industry was used (Cuba, 1969). A 10 g sample is extracted with water for 1 h. The pH is then adjusted at 7,0-8,0. The extract is diluted to 250 ml, filtered, and an aliquot of the filtrate is titrated with 0,1 N $AgNO_3$ in the presence of potassium chromate as indicator.

Recovery tests were carried out, spiking samples with NaCl solution just prior to the extraction stage. Mean percentage recoveries were compared by Student's "t" test.

10 samples of each of 11 different meat products were analyzed by both methods, and the results were compared by the method of the sensitivity coefficient (Zukal et al, 1970).

In order to observe the effect of several components on the interference with Mohr's method, different amounts of solutions of gelatin, cattle serum albumin, glutamic acid, and different phosphates were added to 10 ml portions of pure 0,1 NaCl solution, and these were titrated with 0,1 N $AgNO_3$, at slightly alkaline pH, with potassium chromate indicator.

All chloride contents are expressed as % NaCl.

Results and discussion

Table 1 shows the results of the recovery tests. Student's "t" test showed the difference between methods to be highly significant ($P < 0,01$). This confirms the accuracy of Volhard's procedure, and proves the results of Mohr's method to be excessively high. They are higher, in fact, than the mean percentage recovery suggests, since this is calculated in relation to the original chloride content of the samples, measured by the same method, and consequently also inflated.

Table 2 presents mean results for each product and method, as well as the difference between methods for each product.
Different ranged from 0,17 to 0,67 % NaCl, as compared with 0,25 to 0,45 % reported by Rudisher (1974), proving that his "subtract 0,3 %" correction is inadequate for use with products varying widely in chloride content.
For the application of the method of the sensitivity coefficient, the standard method - Volhard's - was taken as the independent variable. The results of the assessment of the 109 pairs of values can be summarized as follows:

Table 1.- Results of recovery tests

| Method | Mohr | Volhard |
|---|---|---|
| No. samples (n) | 9 | 16 |
| Mean (%) ($\bar{x}$) | 104,7 | 100,3 |
| Std.dev. (s) | 3,706 | 1,076 |
| Variance ratio (F) | 3,90 * | |
| Std. error of dif. (E) | 1,321 | |
| t calc. | 3,33 ** | |
| t crit. ($\alpha = 0.01$; df = 10,93) | 3,11 | |

* $P < 0,05$ ** $P < 0,01$

Table 2.- Mean results for each method and product, and differences between methods for each product.

| Product | Chloride content (% NaCl) Mohr | Volhard | Difference |
|---|---|---|---|
| Veal in sauce | 1,36 | 1,20 | 0,17 |
| Luncheon meat | 2,46 | 1,94 | 0,52 |
| Frankfurters | 2,48 | 2,06 | 0,42 |
| Country sausage | 2,72 | 2,30 | 0,42 |
| Wieners | 2,92 | 2,42 | 0,50 |
| Mortadella | 2,75 | 2,44 | 0,31 |
| Smoked Ham | 2,83 | 2,50 | 0,33 |
| Jamonada | 2,94 | 2,59 | 0,35 |
| Blood sausage | 3,53 | 3,16 | 0,37 |
| Smoked shoulders | 4,20 | 3,90 | 0,30 |
| Spanish sausage | 5,31 | 4,64 | 0,67 |

a) Regression parameters, variances and standard deviations

| Intercept | Slope |
|---|---|
| $\alpha = 0,2188$ | $\beta = 1,0679$ |
| $S_\alpha^2 = 0,001278$ | $S_\beta^2 = 0,0001616$ |
| $S_\alpha = 0,03575$ | $S_\beta = 0,01271$ |

b) Sensitivity coefficient: $E(y/x) = 0,328$

From the regression equation : $y = 0,2188 + 1,0679x$,

the correction equation can be obtained: $x = -0,2049 + 0,9364y$,

which gives estimated Volhard results (x) from Mohr's results (y). Or expressed more simply,

% NaCl (Volhard) = $- 0,20 + 0,94 \left[ \%\ NaCl\ (Mohr) \right]$.

The coefficients of variation of the regression parameters are satisfactorily small: 16.3% for the intercept and 1,19% for the slope.

The sensitivity coefficient, on the other hand, indicates that the Volhard method is roughly 3 times more sensitive than Mohr's method. An estimation of the error of the alternative - Mohr's - corrected method can now be obtained from the error of the standard method, $\sigma_\varepsilon$ . An estimation is made first of carrying out replicate analyses. 10 simultaneous identical replicates gave in this case $\sigma_\varepsilon = 0,0366$

$$S = \frac{\sigma_\varepsilon}{E(y/x)} = \frac{0,0366}{0,328} = 0,11$$

which is not unacceptably large: the ISO (1970) standard Volhard procedure admits differences between identical replicate analyses of up to 0,2% NaCl. This is equivalent to a standard deviation of about 0,07% NaCl, quite comparable to the error figure obtained for the alternative method.

The regression line fitted is shown in Fig. 1. Average results for each product have been plotted as well.

The observed interference is produced by many different components. Tables 3, 4 and 5 illustrate the effects of two soluble proteins, an amino acid and three phosphates, all of which are commonly found either in meat or meat products. The amounts added correspond to those which might be expected to be found in the aliquot titrated in Mohr's method.

The interference is evident, as well as the individual difference amongst interfering agents - orthophosphate and tripolyphosphate, for instance.

This stresses the dependence of the interference on the composition of the sample, and consequently, its stochastic character.

Table 3.- Recovery of NaCl from pure solution with addition of soluble protein.

| Amount added \ Protein | % recovery of NaCl ($\bar{x} \pm 2s$) Gelatin | Albumin |
|---|---|---|
| 0,01 g | 100,3 ± 0,28 | 100,2 ± 0,20 |
| 0,02 g | 100,7 ± 0,20 | 100,6 ± 0,23 |
| 0,03 g | 100,9 ± 0,20 | 101,0 ± 0,35 |
| 0,04 g | 101,2 ± 0,00 | 101,7 ± 0,00 |

Table 4.- Recovery of NaCl from pure solution with the addition of glutamic acid.

| Amount added | % recovery NaCl ($\bar{x} \pm 2s$) |
|---|---|
| 0,45 mg | 99,93 ± 0,28 |
| 0,90 mg | 100,31 ± 0,30 |
| 1,35 mg | 100,66 ± 0,10 |
| 1,80 mg | 100,78 ± 0,10 |

Table 5.- Recovery of NaCl from pure solution with addition of phosphates.

| Amount added | % recovery of NaCl ($\bar{x} \pm 2s$) Orthophosphate | Pyrophosphate | Tripoly phosphate |
|---|---|---|---|
| 2,5 mg | 100,4 ± 0,20 | 101,7 ± 0,38 | 100,7 ± 0,20 |
| 5,0 mg | 100,7 ± 0,19 | 102,8 ± 0,41 | 102,0 ± 0,30 |
| 7,0 mg | 101,1 ± 0,25 | 104,6 ± 0,32 | 104,6 ± 0,66 |

Conclusions:

1) Mohr's method gives excessively high results when applied to meat and meat products.
2) Acceptable results can be obtained, however, correcting these results by means of the equation:
   % NaCl (Volhard) = -0,20+0.94 [% NaCl(Mohr)]
3) Although, according to the value of the sensitivity coefficient, Volhard's method, is nearly three times more sensitive than Mohr's method, the error of the corrected results of the latter is not much higher than the error admitted for the reference method.
4) Proteins, aminoacids and phosphates are amongst the interfering substances, the intensity of the interference depending on their type and concentration.

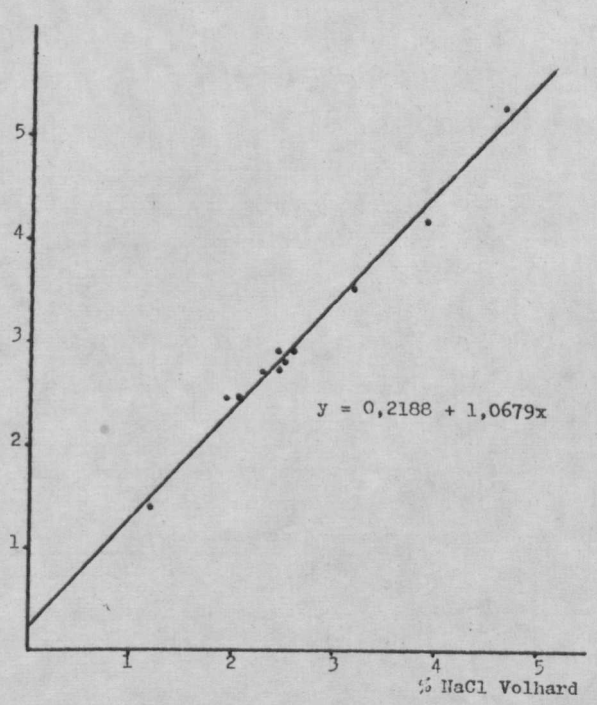


Fig. Regression line. Mean results for each product.

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