Determination of ash content in meat products

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The ISO and AOAC methods for ash determination in meat and meat products are compared with the method approved for the Cuban meat industry and a fourth method using a glycerol-ethanol mixture for hastening the combustion.

Samples of several meat products were simultaneously analyzed using the four methods. Their precision ($2s$) and repeatability ($2s\sqrt{2}$) were evaluated, and recovery tests were carried out spiking the samples with NaCl.

The ISO method gave significantly higher results ($P<0,01$). Its recoveries did not differ significantly, at the 5% level, from those achieved by the AOAC and glycerol-ethanol methods. The method approved for industry gave significantly lower results and recoveries ($P<0,01$), due to the excessively high ashing temperature ($650^{o}C$). Other significant differences amongst methods were probably due to losses of material caused by inflammation of the samples.

All four methods exhibited good precision and repeatability. The differences found, although significant, were rather small in all cases, amounting to only 1,5 - 4 % of the ash content.

The adoption of the ISO method is recommended.

Bestimmung des Aschegehalts in Fleischprodukten

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In dieser Arbeit wurden die Methoden ISO und AOAC mit der Standardmethode für die Fleischindustrie Kubas und einer Methode, die auf der Zugabe einer Mischung von Glyzerin-Ethanol zur Probe basiert, die das Verfahren beschleunigt, verglichen.

Die Proben verschiedener Fleischprodukte wurden auch gleichzeitig mit den vier Methoden analysiert. Ausserdem wurden die Präzision ($2s$) und die Wiederholbarkeit ($2s\sqrt{2}$) der Methoden ausgewertet und Versuche zur Wiedergewinnung durch Addition von NaCl zu den Proben durchgeführt.

Die ISO Methode ergab höhere signifikante Werte im Vergleich zu den anderen ($P<0,01$) und ihre Wiedergewinnungswerte zeigten keine signifikanten Unterschiede mit denen, die bei der AOAC-Methode und bei der Mischung Glyzerin-Ethanol gewonnen wurden. Die Irrtumswahrscheinlichkeit betrug 5%. Die in der Industrie standardisierte Methode ergab Resultate und Wiedergewinnungswerte, die signifikant niedrig waren, im Vergleich zu den anderen ($P<0,01$), was der vorgeschlagenen hohen Veraschungstemperatur ($650^{o}C$) zugeschrieben wird. Andere signifikante Unterschiede der Resultate sind wahrscheinlich durch Materialverschleppung bei der Entflammung der Proben verursacht worden.

Die vier verglichenen Methoden zeigten gute Präzision und Wiederholbarkeit. Die gefundenen Differenzen, wenn auch signifikant, waren in allen Fällen gering und lagen zwischen 1,5 und 4 % des Aschegehalts.

Es wurde die Aufnahme der ISO-Methode vorgeschlagen.

5.17

Determination du contenu de cendres dans les produits de viande

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On compare dans ce travail les méthodes de ISO et AOAC pour la détermination du contenu de cendres dans la viande et les produits de viande, avec la méthode normalisée pour l'Industrie de la Viande à Cuba, et une méthode basée sur l'addition d'un mélange glycérine-ethanol pour accélérer le procédé.

On a analysé, en même temps, des échantillons de différents produits de viande par les quatre méthodes. On a evalué aussi la précision (2s) et la répétibilité ($2s\sqrt{2}$) des mêmes, et on a fait des essais de recuperation ajoutant NaCl aux échantillons.

La méthode ISO a apporté des résultats significativement plus hautes ($P<0,01$). Ses recuperations ne different significativement pas, au niveau du 5 %, des obtenus par les méthodes de AOAC et de mélange glycerine-ethanol. La méthode normelisée dans l'Industrie a donnée des résultats et recuperations significativement plus bas ($P<0,01$) que ceux du reste des méthodes, ce qui est attribué a la temperature d'incineration excessivement haute ($650^{o}C$). Autres differences significatives entre resultats peuvent s'expliquer probablement par le traînement du matériel par inflammation de l'échantillon.

Les quatre méthodes comparées ont donné bonne precision et repetibilité. Les differences trouvées, quoique significatives, ont été dans tous les cas plus bien petites, representant 1,5 - 4 % du contenu de cendres.

On recommande l'adoption du méthode ISO.

Определение содержания золы в мясных продуктах

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В этой работе сравниваются методы ISO и AOAC со стандартным методом для мясной промышленности Кубы и методом, основанным на применении смеси глицерин-этанола с целью ускорения процесса.

Анализированы одновременно разные мясные продукты по вышеуказанным методам. Кроме того оценивается их точность $/2\sigma/$ и повтороность $/2\sigma\sqrt{2}/$. Для того, чтобы восстановить остаток добавляется в образцы NaCl.

Данные, полученные по методу ISO , были значительно выше $/P < 0,01/$, чем по остальным методам, а результаты остатков незначительно отличаются на уровне 5% от остатков, полученные по методу AOAC и смеси глицерин-этанола. Результаты, полученные по стандартному методу в мясной промышленности, были значительно ниже $/P < 0,01/$ результатов, полученных по остальным методам, что можно отнести за счёт предлагаемой температуры сжигания, которая чрезмерно высокая $/650^{o}C/$. Другие значительные различия между результатами можно вероятно объяснить утратой материала при воспламенении образца.

Все четыре сравниваемых метода показывают хорошую точность и повторность. Найденные различия, и смотря на свою значительность, были во всех случаях, главным образом, низкими, в пределе I,5 - 4% от содержания золы.

Рекомендуется применение метода ISO .

5.17

Determination of ash content in meat products

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Introduction

The "ash" of meat products may be defined as the inorganic residue remaining after the combustion of the sample in the presence of air at atmospheric pressure. It is composed of the mineral constituents of the sample in the form of oxides, sulphates, phosphates, silicates and chlorides, the proportions of which depend on the initial composition of the sample and the ashing conditions.

Methods for ash determination in meat and meat products are generally based on the drying and charring of the sample followed by its combustion until white or light gray ashes are obtained. There are differences, though, amongst the various recommended methods, as to charring procedure, ashing temperature and whether or not to add reagents in order to hasten the combustion. Modifications of this sort are present in the ISO (1978) and AOAC (1970) methods, a method described by Lees (1971) and the method approved for the Cuban meat industry (Cuba, 1969). These and many other such modifications have been reviewed by Joslyn (1970).

In this paper the above mentioned methods are compared in order to choose the most suitable one from the standpoint of speed, accuracy and reliability.

Material and methods

Sample preparation: Samples weighing more than 250 g each were passed three times through a meat grinder provided with a 3 mm plate, thoroughly mixing after each operation. Homogeneized samples were stored in completely filled, tightly closed bottles, at about -18°C, until used.

ISO method (1978): In this, as in every other method, empty dishes were heated in the muffle furnace at the temperature prescribed in each case, for about 20 minutes, and weighed to ±0,1 mg, after cooling in a desiccator.

About 5 g of the sample are weighed into the dish to the nearest 0,1 mg. 1 ml 15% (m/v) magnesium acetate solution, the residue of which after ashing is known to ±0,1 mg, is pipetted in, spreading it as uniformly as possible over the sample. The dish is kept over a steam bath for 30 minutes, and the sample is charred on a heating plate or low flame. It is then placed in the muffle furnace, at 550°-600°C, for not less than 30 minutes, cooled in a desiccator and weighed to ±0,1 mg. The ashing procedure is repeated until two successive weighings do not differ by more than 1 mg.

AOAC method (AOAC,1970): A 5 g to 10 g sample is weighed to the nearest 1 mg, and dried by putting the dish over a boiling water bath. A few drops of vegetable oil are added, and the dish is gently heated over a low flame or under IR lamp until frothing ceases. Ashing is carried out at 525°C until white ashes are obtained. The dish is weighed to ±0,1 mg, the ashes moistened with a few drops of water, dried over steam bath or heating plate, and muffled again, until constant weight is attained.

Lees' method (Lees,1971): A 5 g sample is weighed to the nearest 1 mg, and dried over a steam bath. 1 ml of an ethanol-glycerol 50% (v/v) solution is added, and the sample is then charred over a low Bunsen flame, followed by ashing at 550°-570°C for 1 hour. The ashing phase is repeated until constant weight is attained.

Standard method for the Cuban meat industry (Cuba,1969): A 5 g sample is weighed into a porcelain crucible and charred on a heating plate. Ashing is performed at 650°C for 2 hours, after which the dish is cooled in a desiccator and weighed.

15 samples of 5 different products were analyzed by each method, in duplicate. Results were compared by analysis of variance. An additional comparison was carried out with 10 replicate analyses by each method on portions of the same sample. These results were also compared by analysis of variance, and from them the

precision (2s) and repeatability ($2s\sqrt{2}$) of each method were evaluated.

As NaCl is the main component of the ash of meat products, the percentage recovery of the four methods was determined by spiking the samples with differents amounts of a 10% (m/v) NaCl solution of exactly known concentration. Results were evaluated by analysis of variance.

Results and discussion

Mean results for each method and product are shown in Table 1. The corresponding analysis of variance, summarized in Table 2, shows a highly significant difference amongst methods as well as amongst samples ($P < 0,001$). The reasons for the later are obvious, since samples of products varying widely in ash content were analyzed. Table 3 presents the results of the comparison of the 4 methods by means of Duncan's multiple range test (Duncan,1955). The ISO method gave significantly higher results ($P < 0,01$) than the rest of the methods.

Table 4 shows the mean values and measures of variation - standard deviation(s), coefficient of variation ($100 s/\bar{x}$), precision (2s), and repeatability ($2s\sqrt{2}$) - of the results of 10 replicate analyses by the four methods on portions of the same sample. All methods exhibited a low variation of results, the coefficient of variation being about 1% or less, and consequently, good precision and repeatability.

No significant differences were found amongst variances, according to Fisher's "F" test. The analysis of variance, summarized in Table 5, again established significant difference amongst methods ($P < 0,001$). Duncan's test (Table 6) verified that the ISO method gives significantly higher results ($P < 0,01$), but also showed a significant difference between the industrial standard method and the other three, results of the former being significantly lower ($P < 0,01$).

The analysis of variance of the results of the recovery test is shown in Table 7. A significant difference was found amongst methods ($P < 0,01$) , Duncan's test showing that the significance is due to the lower results ($P < 0,01$) given by the industrial standard method (Table 8).

Ashing temperature plays an essential role in this event. Temperatures above 600°C cause volatilization losses of various minerals

Table 1.- Mean ash content for each product and method.

Product	Number of samples	Mean ash content (%) ISO	AOAC	Lees	IAS Std.
Ham sausage	6	4,56	4,41	4,34	4,30
Smoked shoulders	2	5,03	5,00	4,98	4,91
Smoked loins	3	6,48	6,28	6,28	6,38
Ham	1	2,81	2,76	2,72	2,72
"Jamonada"	3	4,10	4,03	4,05	4,12
Mean results		4,80	4,68	4,64	4,66

Table 2.- Analysis of variance of ash content of 15 samples.

Source of Variation	Sum of Squares	Degrees of freedom	Mean Square	F Value
Methods	0,2136	3	0,07119	8,16***
Samples	58,6817	14	4,1916	480,5***
Error	0,3663	42	0,008724	-
Total	59,2616	59	-	-

*** $P < 0,001$

Table 3.- Duncan's test of mean values of ash content.

Method	Mean Result
ISO	4,80[a]
AOAC	4,68[b]
Lees	4,64[b]
IAS Std.	4,66[b]

[a,b] Mean values without common letters differ at $P < 0,01$

Table 4.- Mean values and measures of variation of 10 replicate analyses of the same sample.

Method	ISO	AOAC	Lees	IAS Std.
No. of replicates (n)	10	10	10	10
Mean value ($\bar{x}$)	2,228	2,195	2,184	2,138
Standard deviation (s)	0,0199	0,0158	0,0227	0,0187
c. of variation($100s/\bar{x}$)	0,89%	0,72%	1,04%	0,87%
Precision (2s)	0,040	0,032	0,045	0,037
Repeatability ($2s\sqrt{2}$)	0,056	0,045	0,064	0,053

Table 5.- Analysis of variance of the results summarized in Table 4.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F Value
Methods	0,04153	3	0,01384	36,61***
Error	0,01361	36	0,000378	-
Total	0,05514	39	-	-

*** $P < 0,001$

(Joslyn,1970). This accounts for the fact that the industrial standard method, ashing at 650°C, gives lower recoveries than the other methods, using ashing temperatures of 600°C and below.

Although the AOAC and Lees' methods gave good recoveries, their results for ash content were lower than those of the ISO method (Table 3). This proves that losses are not due in this case to volatilization, but to some other mechanism, probably mechanical. Thus, in the AOAC method, the sample is not totally charred prior to ashing, so that during the first phase of the combustion this might proceed too rapidly. In Lees' method, on the other hand, at the beginning of the charring operation, the alcohol present bursts into flames, and this also may cause sample particles to be blown out of the dish.

It is worth noticing that the differences observed, although statistically significant, are rather small, amounting to a mere 1,5 - 4 % of the total ash content.

Results show that the ISO method gives the best recoveries together with the highest ash values, all this indicating minimal losses of material.

Table 6.- Duncan's test for mean values reported in Table 4.

Method	Mean Result
ISO	2,21[a]
AOAC	2,20[b]
Lees	2,18[b]
IAS Std.	2,14[c]

a,b,c Mean values without common letters differ at P< 0,01

Table 7.- Analysis of variance of recovery test results.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F Value
Methods	64,145	3	21,382	10,08**
Error	25,455	12	2,121	-
Total	89,600	15	-	-

** P< 0,01

Table 8.- Duncan's test for recovery data.

Method	Mean Result
ISO	100,7[a]
AOAC	99,5[a]
Lees	99,3[a]
IAS Std.	95,4[b]

a,b Mean values without common letters differ at P<0,01

This is ascribable to the conditions of operation and the use of magnesium acetate as an auxiliary agent in combustion, the convenience of which had already been indicated by Bailey (1937) in the analysis of flours. The method established by the industrial standard, on the other hand, illustrates the inconveniences of a high ashing temperature, giving results and recoveries significantly lower than the other methods tested.

Conclusions

1.- The ISO method gave significantly higher results than the other methods tested. Its recoveries were among the highest achieved with these methods.

2.- The standard method IAS 2-043 gave lower recoveries and, in one of the experiments, lower results for ash content, than the other methods tested.

3.- The AOAC and Lees' methods gave high recoveries, but their results for ash content were significantly lower than those of the ISO method.

4.- All four methods showed good precision and repeatability, the differences found amongst them being rather small, although statistically significant.

5.- The ISO method is recommended on account of its speed, accuracy and reliability for the determination of ash content in meat products.

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