

Titanium/IV/ oxide as an optimum catalyst for the single-sample chemical classification of meats and meat-industry products

GYÖRGY SELMECI and FERENC CSEH

County Institute for Food Inspection and Chemical Analysis Szeged, Hungary

At the 26th Congress of European Meat Researchworkers /Colorado Springs, USA/ we reported the use of zirconium/IV/ oxide as a catalyst in the simultaneous analysis of the biologically important constituents of meats and meat-industry products. In a medium of high H^+ ion activity, Zr^{4+} ions react with phosphate anions to give an insoluble precipitate of $Zr_3(PO_4)_4$, and hence the total phosphorus content and P number of meat products could be determined only indirectly, with a separate destructive decomposition involving the use of a Se catalyst. In contrast with the simultaneous analysis with zirconium/IV/ oxide, the use of titanium/IV/ oxide has simplified the procedure considerably.

We first made an experimental comparison of the catalytic activities of the TiO_2 , ZrO_2 and $CuSO_4$. For this investigation, samples of lyophilized bovine musculus longissimus dorsi, Bologna sausage and salami were used; these had fat contents in the range 3-44%, which covers the interval generally found in meat-industry products. The results:

Sample	Destruction time /min/		
	TiO_2	ZrO_2	$CuSO_4$
Lyophilized beef /m.long.dorsi/	50-55	50-55	120
Bologna sausage	20-25	20-25	120
Salami	45-50	40-45	120

Thus, it was concluded on the basis of the "destruction time" that the catalytic procedure with titanium/IV/ oxide is equally as efficient as that with zirconium/IV/ oxide. Previous experiments with zirconium/IV/ oxide have demonstrated that there is no need for "post-boiling": the proteins are broken down completely during the experimentally established destruction period. A "post-boiling" was not found to be necessary with titanium/IV/ oxide either if the destruction was carried out for 20-50 min in the various cases. The tabulated data show titanium/IV/ oxide to require substantially shorter destruction times than those with copper/II/ sulphate. Table I reveals that the total protein contents $/N \times 6.25/$ found by the titanium/IV/ oxide and copper/II/ sulphate procedures agree well; at a level $p = 0.05$, the calculated F values are in all cases smaller than the tabulated F values.

The data of Stamm and Gertz on meat products, published in 1980, indicate that the results of Kjeldahl destruction with titanium/IV/ oxide as catalyst practically coincide with those obtained with the AOAC 2049 procedure with mercury/II/ oxyde.

Table II shows that the catalytic effect of titanium/IV/ oxide is not influenced by the Sn^{2+} ions used in the determination of hydroxyproline.

The use of titanium/IV/ oxide as catalyst permits a simpler one-sample analysis than in the case of the zirconium/IV/ oxide method:

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|--|---|--|
| <p>1. <u>Water determination</u></p> <p>Dry matter content determination /dehydration at $180^\circ C/$</p> | <p>2. <u>Partial hydrolysis</u>
$/Sn^{2+}/H_2SO_4/$</p> <p>2.1 Fat content determination /tetrachloroethylene extraction/</p> <p>2.1.1 Total protein content determination $/N \times 6.25/$ /solvent removal; TiO_2 catalysis/</p> <p>2.1.1.1 Total P content determination</p> | <p>3. <u>Complete hydrolysis</u>
$/Sn^{2+}/H_2SO_4/$</p> <p>Hydroxyproline determination</p> <p>2.1.1.2 Ca content determination /complexometry/</p> <p>2.1.1.3 Na content determination /flame-photometry/</p> |
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Course of simultaneous analysis:

1. Dehydration /water content determination/

10 g homogenized meat /meat product/ is dried for 25 min at 180 °C.

2. Partial hydrolysis

The sample dehydrated in /1/ is washed into a 250 cm³ flask with 50 cm³ 30% sulphuric acid containing 1 g SnCl₂·2H₂O. The acidic solution of the sample is refluxed for 10 min.

2.1 Extraction /fat content determination/

100 cm³ tetrachloroethylene at 60 °C is added to the solution obtained in /2/, and the mixture is refluxed for 15 min. After cooling, the two phases are separated in a separating-funnel; the lower portion is run off into a 200 cm³ volumetric flask and the extraction is repeated with 95 cm³ tetrachloroethylene. The solvent is removed and the fat content is measured gravimetrically.

2.1.1 Total protein content determination /N x 6,25/

The acidic aqueous phase obtained in /2.1/ is taken in a 250 cm³ beaker and the solvent is removed on a water-bath. The solution is washed into a 100 cm³ volumetric flask and the volume made up to the mark with 30% sulphuric acid. 20 cm³ of the resulting stock solution is pipetted into a 250 cm³ Kjeldahl flask, and 10 g K₂SO₄, 0,15 g CuSO₄·5H₂O, 0,15 g TiO₂ and 30 cm³ conc. sulphuric acid are added. The solution is boiled until it clears. Depending on the protein and fat contents of the sample, the "destruction time" is 15-55 min. The resulting solution is washed into a 200 cm³ volumetric flask, the volume is made up to the mark with water, and the amount of ammonia formed from a 20 cm³ aliquot is determined with a Parnass-Wagner apparatus.

$$\text{Total protein content} = \frac{F \times f \times 1.4008 \times 6.25 \times 10}{G} \times 100$$

where F = consumption of 0.1 N H₂SO₄ /cm³/; f = factor of 0.1 N H₂SO₄;

G = mass of sample taken /mg/.

2.1.1.1 Total phosphorus content determination

10 cm³ of the 200 cm³ stock solution obtained in /2.1.1/ is pipetted into a 100 cm³ volumetric flask, and 10 cm³ is added from a solution of 20 g ammonium molybdate, 1 g ammonium vanadate and 140 cm³ conc. nitric acid made up to 1000 cm³ with distilled water. Photometry is performed against a blank in 1 cm cells at 420 nm. KH₂PO₄ solutions are employed for the calibration plot.

$$\text{Total P content} = \frac{a \times 20 \times 5}{G} \times 100 \text{ mg/100 g}$$

where a = P quantity read off calibration plot /mg/; G = mass of sample taken /g/.

2.1.1.2 Ca content determination

Starting from the 200 cm³ stock solution obtained in /2.1.1/, the quantity of calcium is determined in the normal manner by direct titration with EDTA.

2.1.1.3 Na content determination

The sodium content is determined by flame photometry in the customary way, on the 200 cm³ stock solution obtained in /2.1.1/.

Complete hydrolysis /hydroxyproline determination/

We set out from the residue /80 cm³/ of the solventfree 100 cm³ stock solution obtained in 2.1.1. The solution is kept in a drying oven at 105 °C for 15 hr. /This is preferably done overnight immediately after sample taking./ The determination is subsequently carried out as described at the 26th Congress.

As an example of the simultaneous analysis procedure developed, analytical data on beef m. longissimus dorsi are presented in Table II. It can be seen that, with the recommended procedure, it is possible to determine the following chemical parameters simultaneously on one sample:

- | | | |
|--|---|---|
| 1/ Water content %/
Dry matter content %/ | 5/ Water - fat index | 10/ Meat protein content
free of connective
tissue protein %/ |
| 2/ Feder number | 6/ Fat - protein index | 11/ Total protein /row
protein content %/
/N x 6.25/ |
| 3/ Foreign water content %/ | 7/ Hydroxyproline content %/ | 12/ Total P content %/ |
| 4/ Fat content %/ | 8/ Connective tissue protein
content %/ | 13/ P number |
| | 9/ Relative connective tissue
protein content %/ | 14/ Ca content /mg/100 g/ |

This procedure is comparatively fast and cheap, and permits serial analyses in small and medium-sized plants, whereby 14 chemical parameters can be determined simultaneously on one sample. Similarly to zirconium/IV/ oxide, the titanium/IV/ oxide applied is cheap, available on a technical scale, and non-toxic, and accordingly causes no environmental pollution.

References:

- 1/ Selmeçi, Gy., Cseh, F., Nové, L.: Simultaneous determination of constituents of meat and meat products on one sample. 26th European Meeting of Meat Research Workers, Colorado Springs, 1980, USA.
- 2/ Stamm, H., Hertz, Ch.: Praktische Erfahrungen bei der Verwendung von Titandioxid als Katalysator bei der Stickstoffbestimmung nach Kjeldahl in Fleisch - und Wurstwaren, Lebensmittelchemie u. gerichtl. Chemie, 34 70-72 /1980/.

Table I.

Total protein /row protein/ determination with titanium/IV/ oxide as catalyst

	Mode of analysis	No. of measurements	Mean protein content %/	Error of mean /E/	Square error of mean /E ² /	F /calcd./	F /tabulated/ /p=0,05/
Lyophilized musc. long. dorsi /beef/	I	20	79,27	0,085	0,0072	1,014	2,17
	II	20	79,29	0,084	0,0071		
Bologna sausage	I	5	11,22	0,081	0,0066	3,300	6,39
	II	5	11,22	0,045	0,0020		
Salami	I	5	25,02	0,044	0,0019	1,188	6,39
	II	5	25,03	0,040	0,0016		

I. Titanium/IV/ oxide as catalyst

II. Control: copper/II/ sulphate as catalyst

Table II.

Simultaneous analysis of beef /musc. long. dorsi/ with titanium/IV oxide as catalyst

<u>Parameter</u>		I.	II.
1.	Water content /%/	71,66	71,76
1.1	Feder number	3,44	3,41
1.2	Foreign water content /%/	0	0
2.1	Fat content /%/	3,45	3,71
2.1.1	Water - fat index	20,77	19,34
2.1.2	Fat - protein index	0,17	0,18
2.2	Hydroxyproline content /%/	0,17	0,18
2.2.1	Connective tissue protein content /%/	1,36	1,46
2.2.2	Relative connective tissue protein content /%/	6,53	6,94
2.2.3	Meat protein content free of connective tissue protein /%/	19,47	19,58
2.3	Raw protein content /%/	20,83	21,04
2.3.1	Total P content /%/	0,12	0,13
2.3.2	P number	1,32	1,42
2.3.3	Ca content /mg/100 g/	10,82	10,14
I.	Means of simultaneous analyses with titanium/IV/ oxide		
II.	Controls: means of individual analyses		