Method of analysis for the determination of lead and cadmium in fresh meat.

Summarry

This report comprises the result of the RIKILT of an intercomparison on the determination of lead and cadmium in bovine liver and bovine kidney.
The aim of this round robbin was to check a wet ashing procedure followed by a flame AAS determination as described too in EEC doc. 2266/VI/77.
Special attention has been given to the latest version of this method, i.e. "Revision 3".
In the tables 1a-3a and 1b-3b (a for Pb, b for Cd) all measuring data, including absorbances and blank values have been collected. In tables 4a (Pb) and 4b (Cd) the results of the various analyses have been given.
The overall results using the calibration curve of the applied method, and the calibration curve of Revision 3 respectively (see text) are as follows.

Element	Sample	Calibration curve applied method		Calibration curve Rev. 3 in all cases	
		$\bar{x}$	c.v.	$\bar{x}$	c.v.
		mg/kg	%	mg/kg	%
Lead	Kidney	3.0	48	2.8	46
	Liver	1.6	48	1.5	50
Cadmium	Kidney	2.7	11	2.5	5
	Liver	0.28	43	0.20	24

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The EEC Scientific Veterinary Commission needs a method of analysis for the determination of lead and cadmium in fresh meat, to control the Directive for import from third countries.
In a meeting at 1979-10-18 the experts of the ad-hoc subgroup IV in principle have adopted two methods: a dry ashing method, doc. 3027/VI/79 and a wet ashing method.
The wet ashing method was origniating from the Netherlands National Institute of Public Health (Schuller, Vaessen), doc. 2266/VI/77. This method was redrafted by Andersen, doc. 2266/VI/77 Rev. 1 and Rev. 2. Recently at a meeting on 1980-05-08 Andersen, De Ruig, Schuller and Wolf redrafted the method again, doc. 2266/VI/77 Rev. 3.
To check the method, in the meeting on 1979-10-18 the experts decided to a small intercomparison, with two samples:
- bovine kidney (NIPH 71 601)
- bovine liver (NIPH 74 228).

Experimental

We have analysed the samples using varied wet ashing procedures. Six samples could be destructed at the same time.

Series 1. According to doc. 2266/VI/77, Rev. 1. Wet destruction in Kjeldahl flasks, extraction with NaDDC/MIBK, measuring MIBK solution.
The NaDDC solution is not extracted with MIBK before using it.
A calibration curve was obtained by standard solutions treated throughout the whole procedure.

Series 2. Wet descruction in Thielepape apparatus extraction with NaDDC/MIBK, measuring MIBK solution. In fact this series conforms to 2266/VI/77 Rev. 3, which at that time, however, not yet was formulated. For calibration the standard solutions were treated throughout the whole procedure, unlike Rev. 3.

Series 3. According to doc. 2266/VI/77, Rev. 3. Wet destruction in Thielepape apparatus, extraction with NaDDC/MIBK, measuring MIBK solution.
Calibration curve obtained by standard solutions treated starting with the extraction step, also without the destruction step.

Results

The results with all experimental data of Series 1 are collected in Table 1a,b and Graph 1a,b for Series 2 in Table 2a,b and Graph 2a,b and for Series 3 in Table 3a,b and Graph 3a,b.

It is observed that the calibration points in graph 1a and 2a are relatively bad. The calibration graphs 3a and 3b, where the standard solutions are not destructed are more regular.

Therefore the results from Series 1 and Series 2 are calculated also with use of calibration curves 3a and 3b.

In Tables 4a and 4b the results of all series are collected:

a = result of series 1

b = result of series 2

c = result of series 3.

The mean value of a, b en c is the mean value of all series, each calculated with their own calibration curve.

e = results of series 1, but calculated with calibration curve of series 3.

f = results of series 2, but calculated with calibration curve of series 3.

The mean value of e, f en c is also the mean value of all series, when a calibration curve is used as stated in Rev. 3.

It is noted that in case of Cd the coefficient of variation has become much better.

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Table 1a Analytical data series 1, Lead.

Sample	No.	Weight	Absorbtion (100xscale exp) vol = 20 ml	Abs-blank	Content mg/kg
Kidney	1	2.886	0.353	0.225	1.06
Kidney	2	2.946	0.479	0.351	1.60
Liver	3	3.074	0.408	0.280	1.24
Liver	4	3.011	0.207	0.079	0.37
Blank	5		0.128		
Blank	6		(0.208)		
2.5 ug Pb	7		0.209	0.163	
5.0 ug Pb	8		0.455	0.409	
10 ug Pb	10		0.806	0.760	
15 ug Pb	11		1.206	1.160	
Blank	12		0.046		

Table 2a Analytical data series 2, Lead.

Sample	No.	Weight	Absorbtion (100xscale exp) vol = 20 ml	Abs-blank	Content mg/kg
Kidney	1	2.002	0.673	0.588	4.13
Kidney	2	1.983	0.573	0.488	3.45
Liver	3	2.051	0.552	0.467	3.20
Liver	4	2.096	0.260	0.175	1.18
Blank	5		0.074		
Blank	6		0.093		
2.5 ug Pb	7		0.188	0.115	
5.0 ug Pb	8		(0.164)	(0.091)	
10 ug Pb	9		(0.565)	(0.492)	
15 ug Pb	10		1.152	1.079	
Blank	11		0.072		
Blank	12		0.073		

Table 3a Analytical data series 3, Lead.

Sample	No.	Weight	Absorbtion (100xscale exp) vol = 20 ml	Abs-blank	Content mg/kg
Kidney	1	3.010	0.994	0.972	4.02
Kidney	2	3.000	0.928	0.906	3.76
Liver	3	1.780	0.317	0.295	2.06
Liver	4		0.012		
Blank	5		(0.142)		
Blank	6		0.033		
2.5 ug Pb	7		0.198	0.172	
5.0 ug Pb	8		0.415	0.389	
10 ug Pb	9		0.838	0.812	
15 ug Pb	10		1.231	1.205	
Blank	11		0.032		
Blank	12		0.019		

Table 4a Lead in Kidney (sample 71601)
and in Livei (sample 74228).

Sample	series	Calibration curve series	Content mg/kg	Content mg/kg
a. Kidney	1	1	1.06-1.60	1.33
b. Kidney	2	2	4.13-3.45	3.79
c. Kidney	3	3	4.02-376	3.89
d. Kidney	1	3	0.97-1.48	1.23
e. Kidney	2	3	3.66-3.06	3.36
a. Liver	1	1	1.24-1.37	0.81
b. Liver	2	2	3.20-1.18	2.19
c. Liver	3	3	2.06-	2.06
d. Liver	1	3	1.13-0.32	0.73
e. Liver	2	3	2.83-1.04	1.94

Kidney Over all mean value of a+b+c: $\bar{x}$ 3.00 mg/kg
n = 6
coefficient of variation: 48% n=3.

Kidney Mean value of c+d+e: $\bar{x}$ 2.82 mg/kg
n = 6
coefficient of variation: 46% n=3.

Liver Over all mean value of a+b+c: $\bar{x}$ 1.61 mg/kg
n = 5
coefficient of variation: 48% n=3.

Liver Mean value of c+d+e: $\bar{x}$ 1.48 mg/kg
n = 5
coefficient of variation: 50% n=3.

Table 1b Analytical data series 1, Cadmium.

Sample	No.	Weight	Absorbtion vol = 20 ml	Content mg/kg
Kidney	1	2.886	0.154	2.50
Kidney	2	2.946	0.158	2.51
Liver	3	3.074	0.010	0.16
Liver	4	3.011	0.010	0.17
Blank	5		0.000	
Blank	6		0.000	
2.5 ug Pb	7		0.052	
5.0 ug Pb	8		0.108	
10 ug Pb	10		0.203	
15 ug Pb	11		0.271	
Blank	12		0.000	

Table 2b Analytical data series 2, Cadmium.

Sample	No.	Weight	Absorbtion vol = 20 ml	Content mg/kg
Kidney	1	2.002	0.102	3.12
Kidney	2	1.983	0.097	3.00
Liver	3	2.051	0.008	0.38
Liver	4	2.096	0.009	0.41
Blank	5		0.000	
Blank	6		0.000	
2.5 ug Pb	7		0.026	
5.0 ug Pb	8		(0.037)	
10 ug Pb	9		0.163	
15 ug Pb	10		0.234	
Blank	11		0.000	
Blank	12		0.000	

Table 3b Analytical data series 3, Cadmium.

Sample	No.	Weight	Absorption vol = 20 ml	Abs-blank	Content mg/kg
Kidney	1	3.010	0.162	0.165	2.61
Kidney	2	3.000	0.166	0.169	2.69
Liver	3	1.780	0.008	0.011	0.28
Liver	4				
Blank	5		-0.004		
Blank	6		-0.004		
2.5 ug Pb	7		0.058	0.060	
5.0 ug Pb	8		0.113	0.115	
10 ug Pb	9		0.196	0.198	
15 ug Pb	10		0.274	0.276	
Blank	11		-0.002		
Blank	12		-0.002		

Table 4b Cadmium in Kidney (sample 71601)
and in Liver (sample 74228).

Sample	series	Calibration curve series	Content mg/kg	Content mg/kg
a. Kidney	1	1	2.50-2.51	2.50
b. Kidney	2	2	3.12-3.00	3.06
c. Kidney	3	3	2.61-2.69	2.64
d. Kidney	1	3	2.55-2.56	2.56
e. Kidney	2	3	2.43-2.34	2.39
a. Liver	1	1	0.16-0.17	0.16
b. Liver	2	2	0.38-0.41	0.40
c. Liver	3	3	0.28-	0.28
d. Liver	1	3	0.16-0.17	0.16
e. Liver	2	3	0.19-0.20	0.20

Kidney Over all mean value of a+b+c: $\bar{x}$ 2.74 mg/kg
n = 6
coefficient of variation: 11% n=3.

Kidney Menr value of c+d+e: $\bar{x}$ 2.53 mg/kg
n = 6
coefficient of variation: 5% n=3.

Liver Over all mean value of a+b+c: $\bar{x}$ 0.28 mg/kg
n = 5
coefficient of variation: 43% n=3.

Liver Mean value of c+d+e: $\bar{x}$ 0.20 mg/kg
n = 5
coefficient of variation: 24% n=3.