# NMKL

c/o National Veterinary Institute PB 750 Sentrum, N-0106 Oslo, Norway.

# NMKL Newsletter Nordic Committee on Food Analysis

# www.nmkl.org

# New leader of the NMKL Norwegian National Committee

#### PhD Urd Bente Andersen

(photo), is employed at AS Vinmonopolet, the Norwegian Wine and Liquor Monopoly, as Accreditation Technical and Quality Manager for the monopoly's sensorial selection process.

Urd Bente is Cand.Agric. and Dr. Scient. in food science from the Norwegian University of Life Sciences (UMB). She has worked at Bergen University College (HiB), the Norwegian Institute for Food and Environmental Analysis (now Eurofins), the Norwegian Food Safety Authority, Matforsk (now Nofima Food), UMB, the Norwegian Defence and at the Municipal Food Control Authority. Urd Bente has worked extensively with sensorial analysis, but also with food safety (HACCP, labelling, food additives) and laboratory analysis of food and drinking water. For her PhD she worked within instrumental texture measurements (Instron) for measuring the quality of farmed salmon fish. She has also taught the subject

food and health and been an external examinator for master degrees and courses at UMB.

In 1998 Urd Bente Andersen joined NMKL, sub committee 4, sensory analysis. She has been the study director/referee of NMKL method No. 183, Sensorial quality control test for drinking water, the project leader of NMKL Procedure No. 11, Procedure for sensory analysis of drinking water, and has been involved in the elaboration of NMKL Procedure No. 17, Guidelines for requirement specifications for food analyses. She has also contributed in the elaboration of other NMKL procedures within sensory analyses.

Urd Bente Andersen gives the following statement: "NMKL conducts important work, which is appreciated by users in many countries also outside the Nordic countries. For us members, NMKL is of great importance as research community and network. As the Leader of the Norwegian



National Committee I wish to contribute to maintaining the good atmosphere in NMKL. I am aware that I am the first person from the committee 4 to become leader of one of the national committees, and I hope my experience will be of use to the NMKL executive committee as well as for committee 4. Further, I look forward to learning more about the other committees of NMKL and how they work. I am grateful for the confidence given to me as the new leader of the Norwegian National Committee. I look forward to getting started on the work for the organisation."

NMKL would like to thank **PhD Tone Normann Asp (photo)**, for her extensive contribution to NMKL as the leader of the Norwegian National Committee. She has led the committee since 2002. Tone Normann Asp is associated professor at the Norwegian School of Veterinary Science, Department of Food Safety. NMKL is very pleased to learn that Tone will continue as a dedicated member of the Norwegian National committee and of NMKL sub committee 3 (chemistry). Her technical and social skills and qualities are highly appreciated.



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# NMKL Method No. 66, 5th Ed., 2009: Coagulase positive staphylococci. Enumeration in foods.

Staphylococci are widely distributed; their main reservoir is skin and mucosal surfaces of humans and warm-blooded animals. *Staphylococcus aureus* is the most common pathogenic bacteria of the coagualse positive staphylococci, however food poisoning caused by *S. intermedius* has also been reported.

Studies have shown that 20-50% of the population are long term carriers of *S. aureus*, and the infection can be transferred to food by handling. *S. aureus* food poisoning is very unpleasant, but usually brief. After an incubation time of one to six hours, symptoms occurs with nausea and vomiting, often with stomach pain and in some cases also diarrhoea. Serious complications can occur.

Coagulase positive staphylococci (most importantly *S. aureus*, *S. intermedius* and *S. hyicus*) are, in this method, defined as Gram positive, catalase positive cocci that form typical or suspicious colonies on Baird Parker agar, and are coagulase positive, or form typical colonies on Baird Parker agar with Rabbit Plasma Fibrinogen.

Coagulase positive staphylococci are enumerated by surface inoculation of decimal dilutions of a specified quantity of the food sample on the selective agarmedium Baird Parker (BP) and/or Baird Parker agar with a Rabbit Plasma Fibrinogen supplement (BP+RPF). After incubation, typical and atypical colonies are counted. When using BP, a selection of

colonies must be confirmed by the coagulase test. When using BP+RPF, the production of coagulase is tested directly on the agar plate.

The method was validated in a collaborative study in 2001. Three types of food samples, cheese, boiled ham and pre-cut lettuce, were inoculated at two levels using two different strains of *Staphylococcus aureus*. Two parallels of each material were analysed, as well as a blind sample of each of the foods. The laboratories analysed a total of 15 test samples. The standard deviation of reproducibility varied between 0.07 and 0.49 log cfu/g.

There are not many changes from the 4<sup>th</sup> edition, and the most important are as described below:

- The title has changed from *Staphylococcus aureus* to Coagulase positive staphylococci.
- Blood agar cannot be used as the only isolation medium, but it is advisable to use it as a supplementary medium.
- It is important that the Baird Parker Rabbit Plasma Fibriongen agar (BP-RPF) is read after both 24 and 48 hrs.
- Typical and suspicious colonies from Baird Parker shall be confirmed by Gram staining, catalase and coagulase test. It is also recommended that typical colonies on BP-RPF are subjected to Gram staining and catalase test.
- If further identification is necessary, commercial test kits validated for use on food isolates should be used.
- It is recommended that more than one isolate from a positive sample is tested for toxin production and toxin genes, and that this is performed by an expert laboratory.

Gro S. Johannessen, Tone M. Fagereng and Hannah J. Jørgensen, National Veterinary Institute, Oslo, Norway, have revised this method.



S. aureus on Baird Parker agar with Rabbit Plasma Fibrinogen. Photo: Hanna J. Jørgensen.



# Do you want a subscription for NMKL methods?

NMKL offers method subscription either by PDF files, hard copies or online (using user ID and password) for NOK 2500 (EUR 300) annually.

If you are a new subscriber of NMKL methods, the first time fee for online subscription is NOK 5000 (EUR 600), which corresponds to the price of a compiled method collection.

With access to the online method collection, the newest version of the NMKL methods are only a click away.

#### NMKL Method No. 95, 5th Ed., 2009: *Clostridium perfringens.* Determination in foods, feed and environmental samples.

The method is a horizontal method for determination of *Clostridium perfringens* in food, feed and environmental samples.

The revision of this method consists only of an extension of the field of application. The previous version was restricted to determination in heated foodstuffs. No technical changes are made.

The revision has been carried out by a project group under the lead of Prof. Niels Skovgaard, Denmark.

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*Cl. perfringens* is quantitatively determined by spreading known amounts of sample on mCP agar and/or on TSC agar. The plates are incubated anaerobically for  $24 \pm 3$  hours at  $37.0 \pm 1.0$  °C. Colonies of *Cl. perfringens* are yellow-grey on mCP agar, 3 - 6 mm in diameter, and produce a yellow colour change in the agar around the colonies. An acid phosphatase test is done with ammonia, and a

positive reaction is visualised as a red colour change.

*Cl. perfringens* colonies are black on TSC agar. Non-motile Gram-positive bacteria producing black colonies on TSC agar and acid and gas from lactose may be identified as *Cl. perfringens*. Additional information is obtained from anaerobic incubation of blood agar plates for 24 ± 3 hours at 37.0 ± 1.0 °C. Colonies of 1 - 6 mm surrounded by a double haemolytic zone, may be considered as *Cl. perfringens*. In some cases, the haemolysis is not seen until after 48 ± 4 hours.

The results from the collaborative study showed acceptable results for minced meat and "lobscouse"; a heat treated meat dish.

# Verification of sulphite reducing bacteria

NMKL Method No. 56 refers to NMKL Method No. 95 for verification of sulphite reducing bacteria. This was corrected in NMKL Newsletter No. 69, where the reference to NMKL Method No. 95 was deleted.

NMKL has received questions about how to verify sulphite reducing bacteria. Prof. Niels Skovgaard has responded to the question. He concludes that for the purpose of the method it is sufficient to perform Gramstaining for demonstration of Gram-positive. If this is not considered sufficient, enrichment of the suspicious colony in a suitable broth has to be carried out. This may take days. Thereafter, cultivation on a solid agar, for instance blood agar, must be performed, which again may take several days.

Prof. Skovgaard's complete answer is available on request from nmkl@vetinst.no.

# NMKL Method No. 143, 3rd. Ed., 2009 Ochratoxin A. HPLC determination in cereals and coffee.

Ochratoxin A (OTA), a toxin produced by *Aspergillus ochraceus* and *Penicillium verucosum*, is one of the most abundant food-contaminating mycotoxins in the world. Human exposure occurs mainly through consumption of improperly stored food products, particularly contaminated grain, coffee, and grapes.

The method is applicable for determination of OTA in cereals and roasted coffee. This method has been validated for OTA in barley from  $0.1 - 4.5 \mu g/kg$  and for roasted coffee from  $0.2 - 5.5 \mu g/kg$ .

OTA is extracted from barley by blending with aqueous acetonitrile. The extract is purified by passing it through an immunoaffinity column. OTA is extracted from roasted coffee by blending with methanol and sodium hydrogen carbonate. The extract is cleaned up by passing it through an immunoaffinity column. If interferences occur, which can be seen in the chromatogram, the extract is cleaned up by passing it through a silane column before the immunoaffinity column. Such clean-up process is described in this method. OTA is separated by reverse-phase



HPLC and determined by fluorescence.

The method was validated in two collaborative studies in 1998 and 1999, respectively. The studies were a part of the Standards, Measurements and Testing Projects coded SMT4-CT-96-2045, EUR 18954 EN and EUR 19504 EN. The reports from these SMT projects are not available to the general public. Project leader of the work package dealing with Ochratoxin A in barley and roasted coffee was A. C. Entwistle from the Leatherhead Food Research Association ( Leatherhead, UK). 15 laboratories participated, each analysing duplicate samples at five levels for barley and roasted coffee, respectively. The precision of the results was satisfactory, all results had HorRat values below 2.

The SMT project resulted in the standard EN 14132:2003: Foodstuffs – Determination of ochratoxin A in barley and roasted coffee – HPLC method with immunoaffinity column clean-up. This NMKL method harmonises with this EN standard. NordVal Certificate No. 010 for Salmonella ELISA Test OPTIMA / RayAl Salmonella OPTIMA

# Salmonella ELISA Test OPTIMA, RayAl Salmonella OPTIMA

Manufactured by: Bioline Aps Fredericiavej 414 7080 Børkop Denmark Supplied by: RayAl Ltd Mansfield i-centre Oakham Business Park Hamilton Way Mansfield NG18 5 BR United Kingdom

Bioline Aps Fredericiavej 414 7080 Børkop Denmark

Salmonella ELISA Test OPTIMA / RayAI Salmonella OPTIMA fulfils the requirements of the NordVal validation protocol. The method has been validated according to ISO 16140 against the reference methods ISO 6579:2002 and NMKL 71. The results document no statistical difference in the performance of the methods.

#### PRINCIPLE AND FIELD OF APPLICATION

Salmonella ELISA Test OPTIMA / RayAl Salmonella OPTIMA is an immunoenzymatic test using a microtiter plate coated with specific antibodies directed against Salmonella, and ready-to-use reagents. The test allows the detection of Salmonella after enrichment steps (for about 40 hours) and a heat shock releasing any Salmonella antigens that might be present in the sample. The antigens are detected by a sandwich ELISA (Enzyme Linked Immuno Sorbent Assay). Confirmation of positive samples is not necessary, unless it is required by law.

#### **COMPARISON STUDY**

The Salmonella ELISA Test OPTIMA/ RayAl Salmonella OPTIMA was compared to the reference method on food and animal feed. The following results were obtained:

> Relative accuracy: 99% Relative specificity: 99% Relative sensitivity: 99% Detection level: 1 CFU/ 25 grams. Inclusivity: 55 strains of *Salmonella* were detected out of 55 tested. Exclusivity: 30 strains not belonging to *Salmonella* were all negative; no interferences.

#### COLLABORATIVE STUDIES

Collaborative studies were conducted in 2001, 2004 and 2008.

More than 10 laboratories participated The results showed that there were no significant differences between the reference and the alternative method, as the following results were reported:

Sensitivity: 100% Specificity: 100% Relative accuracy: 100%



![](_page_3_Picture_17.jpeg)

# NordVal Certificate No. 020 for RAPID'E.coli2

# RAPID'E.coli2

Manufactured and supplied by: Bio-Rad Laboratories, 3 Blvd Raymond Poincare, 92430 Marnes-la-Coquette, France

RAPID'E.coli2 fulfils the requirements of the NordVal validation protocol. The reference methods were ISO 4832:1991 for enumeration of coliforms in foods at 37 °C, and ISO 16649-2:2001 for enumeration of *E.coli* in foods at 37 °C and 44 °C.

NordVal has studied the enclosures to the application and evaluated the results obtained in the validations conducted by AFNOR in accordance with ISO 16140. The results document no statistical difference in the performance of RAPID'E.coli 2 Agar and the reference methods.

#### PRINCIPLE OF THE METHOD AND FIELD OF APPLICATION

RAPID'E.coli2 is based on simultaneous detection of two enzyme activities. RAPID'E.coli2 totally inhibits growth of Gram-positive bacteria and of the principal Gram-negative bacteria other than *Enterobacteriacae*. *Escherichia coli* specificity is obtained by means of simultaneous detection of ß-D-Glucuronidase (GLU) (specific to *E. Coli*) and ß -D-Galactosidase (GAL) enzymatic activities.

The medium contains 2 chromogenic substrates:

- one substrate specific to GLU, causing pink coloration of colonies positive for this enzyme, and
- one substrate specific to GAL, causing blue coloration of colonies positive for this enzyme.

Coliforms other than *E. coli* (GAL+/GLUC-) form blue colonies, and *E. coli* (GLU+/GAL+) form violet to pink colonies.

The method is applicable to the determination of *E.coli* and total number of coliforms in food.

# COMPARISON STUDY - COMPLIANCE BETWEEN RAPID'E.COLI 2 AND THE REFERENCE METHODS

A comparison study performed in 2004 on 5 food product/strain combinations. The samples were analysed in duplicates with each of the two methods, at levels from 10 to 10.000 cfu/g. The precision of the results for all food products were similar to the result of the collaborative study for the meat products, illustrated in the graph to the right. As the results lie within 2 times the standard deviation of the reference method, given as the acceptance criteria, there is no significant difference between the results of the alternative method and the reference method.

#### **COLLABORATIVE STUDY**

The collaborative study was conducted in November 2004. The analyses were carried out on duplicate samples of pasteurised milk artificially contaminated, with an *Enterobacteria-cae cloacae* strain isolated from powdered milk and an *E.coli* strain isolated from a pastry. Eleven laboratories reported results. However, one laboratory was excluded as they had not followed the instruction carefully.

The precision of the 40 obtained results is given in the table below:

Contamination level	Referenc	e method	Alternative method		
(log cfu/g)	Repeatability limit, r	Reproducibility limit, R	Repeatability limit, r	Reproducibility limit, R	
50	0.20	0.31	0.19	0.22	
500	0.16	0.31	0.16	0.30	
5000	0.19	0.38	0.20	0.26	

The repeatability limit may be interpreted as the amount within which two determinations performed at the same laboratory should fall 95% of the time. The reproducibility limit may be interpreted as the amount within which two separate determinations conducted in different laboratories should fall 95% of the time.

#### CONCLUSION

Both the alternative method and the reference method have satisfactory precision. Based on the comparison study and the collaborative study no statistical differences were found between the results of the RAPID'E.coli 2 Agar and the reference methods; ISO 4832:1991 and ISO 16649-2: 2001.

![](_page_4_Picture_21.jpeg)

![](_page_4_Figure_22.jpeg)

# NordVal Certificate No. 018 for Hygicult® TPC

# Hygicult® TPC

Manufactured and supplied by: Orion Diagnostica Oy, PO. Box 83, 02101 Espoo, Finland

![](_page_5_Picture_3.jpeg)

#### PRINCIPLE AND FIELD OF APPLICATION

Hygicult® TPC is a hinged dip-slide culture method for the detection of total microbial count on surfaces. The slide is covered on both sides with Total Plate Count Agar which supports rapid growth of the most common bacteria and fungi. Hygicult® TPC slides are intended for rapid monitoring of microbiological hygiene in different types of materials, both solid and liquid. The test can be performed onsite, or the slides can be used as convenient transport media for samples.

<u>Note:</u> The limit values for microbial count in normal drinking water are too low to be detected by the Hygicult method.

#### **COLLABORATIVE STUDY**

A full collaborative study on total aerobic bacterial count was conducted to validate Hygicult® TPC against NMKL Method No. 5 (contact plates and swabbing), using stainless-steel surfaces artificially contaminated with different microbes at various levels. Twelve laboratories participated in the collaborative study, analysing a total of 108 samples. The study was organised by VTT Biotechnology, Finland, in 1999. The following results were obtained:

![](_page_5_Picture_9.jpeg)

NordVal Validation

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![](_page_5_Picture_11.jpeg)

	Low level		N	Medium level		High level			
	Ref. method		Ref. method Altern. Ref. method meth.		Altern. meth.	R	ef. method	Altern. meth.	
	Contact plate	Swab- bing	TPC	Contact plate	Swab- bing	TPC	Contact plate	Swab- bing	TPC
Mean of theoreti- cal yield (cfu/cm <sup>2</sup> )	1.41	1.41	1.41	10.7	10.7	10.7	43.6	43.6	43.6
Mean of surface yield (cfu/cm <sup>2</sup> )	0.43	0.43	0.35	1.91	2.17	2.07	7.12	9.09	8.03
Recovery (%)	30	30	25	18	20	19	16	21	18
Repeatability, s <sub>r</sub>	0.17	0.32	0.15	0.70	0.81	1.45	1.94	3.04	2.51
Repeatability limit, r	0.49	0.90	0.42	1.96	2.26	4.07	5.43	8.50	7.02
Reproducibility S <sub>R</sub>	0.21	0.38	0.20	1.29	1.45	2.02	2.99	5.07	4.29
Reproducibility limit, R	0.59	1.07	0.56	3.60	4.06	5.64	8.34	14.2	12

The Hygicult® TPC dip-slide, contact plate and the swabbing methods gave similar results at all the 3 microbial levels tested. There were no significant differences between results obtained at different incubation temperatures (25 and 30 °C) or incubation times (48 and 72 h) for all 3 methods.

## CONCLUSION

Based on the collaborative study, it may be concluded that the Hygicult® TPC dip-slide does not differ in practical terms neither in yield nor in precision, compared with the reference method.

## NordVal Certificate No. 017 for Campylobacter real-time PCR

Eurofins Steins, Denmark, has applied for renewal of the NordVal certificate for the *Campylobacter* real-time PCR for detection of thermotolerant *Campylobacters* in raw chicken meat, faeces on cloacae swabs and on disposable shoe covers with chicken faeces. The method has been compared against ISO/DIS 10272-4 and NMKL No. 119: Thermotolerant *Campylobacter*. Detection, semi-quantitative and quantitative determination in foods and drinking water.

#### SCOPE AND FIELD OF APPLICATION

This real-time PCR method is applicable to the detection of human pathogenic thermotolerant *Campylobacters* (*C. jejuni, C. coli* and *C. lari*) in raw chicken meat, cloacae swabs and faecal samples collected from disposable shoe covers in rearing houses. Thermotolerant *Campylobacters* are qualitatively determined with a sensitivity of 1-10 cfu/ 25 gram raw chicken meat and 100-1000 cfu/ml dilution of cloacae swabs and faecal samples collected from disposable shoe covers.

#### PRINCIPLE

For raw chicken: 25 g sample is incubated in 225 ml of Bolton broth according to NMKL No. 119. After 24 hours 1 ml of the enriched meat buffer is transferred to a 1.5 ml Eppendorf tube.

For cloacae swab: Samples are collected in tubes with 3 ml 0.9% NaCl. 3 ml of sample preparation is transferred to a small filter bag. 1 ml of the filtrate is transferred to a 1.5 ml Eppendorf tube. Alternatively, the tube containing the swab is lightly centrifuged, in order to spin down remains of faecal material. 1 ml of the supernatant is transferred to a 1.5 ml Eppendorf tube.

<u>For disposable shoe covers with faeces:</u> Samples are weighed and washed in 0.9% NaCl and homogenized using a stomacher before 1 ml is transferred for the preparation of DNA to the real time PCR analysis.

The real time PCR is performed on a part of the prepared volume. The primers are targeting a *C. jejuni* 16S rRNA sequence. An internal amplification control (IAC) is analyzed with the samples to detect false negative responses.

#### THE COLLABORATIVE STUDY

Nine laboratories participated in a study analysing pellets from 18 coded 1-ml samples including 6 chicken neck skin samples, 6 shoe cover samples and 6 cloacae swab samples. The samples were spiked in duplicate with C.*jejuni* CCUG 11284 at three levels. The laboratories were also given a positive DNA control, a ready-to-use PCR mixture with added IAC, and reagents for the magnetically based DNA extraction. A second collaborative study, comprising eight participating laboratories, was subsequently performed on shoe cover samples. The results of the studies are given in the table below:

	Speci- ficity %	Sensitivity %		Accuracy %		
Matrix	-	Low	High	-	Low	High
Neck skins	100	100	100	100	100	100
Shoe covers	94	100	100	100	100	100
Cloacae swabs	100	92	100	100	92	100

The results of the studies show that the PCR method performs satisfactorily, and that there is no statistical significance between the results of the methods tested.

#### THE COMPARISON STUDY

The comparative study of real-time PCR was performed by comparing to two reference culture methods (NMKL 119 and ISO/DIS 10272-4) on naturally contaminated samples; 99 shoe covers, 101 cloacae swabs, 102 neck skins from abattoirs and 100 neck skins from retailers. Culturing included enrichment in both Bolton and Preston broths followed by isolation on Preston agar and mCCDA. The results obtained by the real-time PCR and the reference culture method were as follows:

		% Value	
Matrix	Accuracy	Sensitiv- ity	Specificity
Shoe covers	96	82	95
Cloacae swabs	95	88	96
Neck skin Abattoir	100	100	98
Retailer	100	100	100
Total	98	95	97

The <u>degree</u> of agreement quantified as kappa was found to be 0.98. As kappa is above 0.80, there is good agreement between the alternative method and the reference method. Further, the requirement for a sensitivity of 95% or better is fulfilled.

# New NordVal Protocol for the validation of alternative microbiological methods

A revised version of the NordVal Protocol has been elaborated. The NordVal Protocol describes the technical procedures for validation of alternative methods for microbiological analyses of food, water, animal faeces, feed, samples from equipment and processing areas and environmental samples.

# The Protocol may be downloaded from the NordVal pages under www.nmkl.org.

The validation of qualitative and quantitative methods comprises two phases:

- A: A method comparison study of the alternative method against a reference method (performed by an expert laboratory).
- B: A collaborative study of the alternative method (organised by an expert laboratory).

The test characteristics for alternative methods are shown in the following table:

Qualitative methods	Quantitative methods
1. Selectivity (inclusivity/ exclusivity)	1. Selectivity (inclusivity/ exclusivity)
2. Relative accuracy	<ol><li>Lowest validated level with satisfactory precision</li></ol>
3. Limit of detection	3. Repeatability
4. Relative sensitivity	4. Reproducibility
5. Relative specificity	
6. The agreement between the methods, $\kappa$	

![](_page_7_Picture_8.jpeg)

NMKL Method No 2, 3rd Ed., 1979: Benzoic acid. Determination in foods, is <u>withdrawn</u> from the NMKL method collection.

For analysis of Benzoic acid, NMKL recommends NMKL Method No. 124, 2nd Ed., 1997: Benzoic acid, sorbic acid and p-hydroxybenzoic acid esters. Liquid chromatographic determination in foods. This method is also recommended by Codex Alimentarius (as a type II method).

Alternative: NMKL Method No. 103, 1984: Benzoic acid and sorbic acid in foods. Quantitative determination by gas chromatography.

# **Available NMKL Methods**

Lists of NMKL publications are available at www.nmkl.org.

For information about specific methods, please use the web shop and search for the particular method or analyte of interest. You will then find information about the method's scope, field of application, principle and performance.

# **NordVal Certificates**

List of methods certified by NordVal is available under NordVal at www.nmkl.org. Here you also find the NordVal certificates.

![](_page_7_Picture_17.jpeg)

![](_page_8_Picture_0.jpeg)

We will invite participants from the Nordic countries, as well as from other countries. All presentations will be held in English.

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certainty and quality control. Another important aim is to harmonise the way we work with quality, and to support laboratories in improving quality systems, making them more effective, based on competence and acceptance by all personnel.

> This NMKL/TrainMiC is organized by National Food administration and SP Technical Research Institute of Sweden in cooperation with the NMKL and IRMM

<ul> <li>Thursday 17 September 2009</li> <li>09:00 Registration and coffee</li> <li>09:30 Introduction and presentation of participants (Magnusson)</li> <li>09:45 Presentation of NMKL (Engman)</li> <li>10:00 Statistics and terminology © TrainMiC (Magnusson)</li> <li>11:00 Presentation of example; Pesticide residues © TrainMiC (Engman)</li> <li>Exercise: Traceability of measurement results (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Single laboratory validation of measurement procedures © TrainMiC (Engman)</li> <li>Exercise: Recovery and bias estimation (Magnusson)</li> <li>15:00 Coffee</li> <li>15:20-17:00 Uncertainty of measurement - Principles and the Nordtest approach (Magnusson)</li> <li>19:00 Dinner</li> <li>Friday 18 September 2009</li> <li>08:30 Internal quality control – QC © TrainMiC (Magnusson)</li> <li>09:30 Internal quality control – QC © TrainMiC (Engman)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Single laboratory comparisons © TrainMiC (Engman)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>			
<ul> <li>Disconstruction and presentation of participants (Magnusson)</li> <li>Disconstruction and presentation of participants (Magnusson)</li> <li>Disconstruction and presentation of participants (Magnusson)</li> <li>Disconstruction and presentation of NMKL (Engman)</li> <li>Disconstruction and presentation of example; Pesticide residues © TrainMiC (Engman)</li> <li>Exercise: Traceability of measurement results (Magnusson)</li> <li>Disconstruction and presentation of measurement procedures ©</li> <li>TrainMiC (Engman)</li> <li>Exercise: Recovery and bias estimation (Magnusson)</li> <li>Disconstruction of measurement - Principles and the Nordtest approach (Magnusson)</li> <li>Dinner</li> <li>Friday 18 September 2009</li> <li>Dinner</li> <li>Exercise: Evaluation over time (Magnusson)</li> <li>Dinson</li> <li>Dinner</li> <li>Evaluation of your own method validation and measurement uncertainty</li> <li>Magnusson)</li> <li>Dinner</li> <li>Evaluation of use of reference materials © TrainMiC (Engman)</li> <li>Diconson (Magnusson)</li> <li>Disconson (Magnusson)</li> <li>Evaluation to the learning evaluation, and coffee</li> <li>Disconson (Magnusson)</li> <li>Disconson (M</li></ul>		ay 17 September 2009 Registration and coffee	Speakers
<ul> <li>Administration, Sweden. Analytical chemist working with food control of heavy metals.</li> <li>15:20-17:00 Uncertainty of measurement - Principles and the Nordtest approach (Magnusson)</li> <li>19:00 Dinner</li> <li>Friday 18 September 2009</li> <li>08:30 Internal quality control – QC © TrainMiC (Magnusson)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Engman)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Engman)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>	09:00 09:30 09:45 10:00 11:00 12:00 13:00	Registration and coffee Introduction and presentation of participants (Magnusson) Presentation of NMKL (Engman) Statistics and terminology © TrainMiC (Mangusson) Presentation of example; Pesticide residues © TrainMiC (Engman) Exercise: Traceability of measurement results (Magnusson) Lunch Single laboratory validation of measurement procedures ©	Speakers Bertil Magnusson, SP, Sweden National TrainMiC Team Leader, industrial analytical chemist with focus on measure ment quality and Swedish rep- resentative in metrology organ sations such as CCQM, Eurachem and Euromet. Joakim Engman, National Food
<ul> <li>Friday 18 September 2009</li> <li>08:30 Internal quality control – QC © TrainMiC (Magnusson) Exercise: Evaluation over time (Magnusson)</li> <li>09:30 Inter-laboratory comparisons © TrainMiC (Engman)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>	15:00 15:20-1 19:00	Exercise: Recovery and bias estimation (Magnusson) Coffee 7:00 Uncertainty of measurement - Principles and the Nordtest approach (Magnusson) Dinner	Administration, Sweden. Ana- lytical chemist working with food control of heavy metals. Works with validation of meth- ods and standardisation of methods within NMKL and CEN.
<ul> <li>09:30 Inter-laboratory comparisons © TrainMiC (Engman)</li> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>	<b>Friday</b> 08:30	<b>18 September 2009</b> Internal quality control – QC © TrainMiC (Magnusson) Exercise: Evaluation over time (Magnusson)	
<ul> <li>10:30 Coffee</li> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>	09:30	Inter-laboratory comparisons © TrainMiC (Engman)	Evaluation of your own method
<ul> <li>11:00 Selection and use of reference materials © TrainMiC (Magnusson)</li> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> </ul>	10:30	Coffee	validation and measurement uncer-
<ul> <li>12:00 Lunch</li> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> <li>opportunity to send one of your methods for an evaluation by the speakers. They will advise and suggest improvements, or just comment on your good work.</li> </ul>	11:00	Selection and use of reference materials © TrainMiC (Magnusson)	<b>tainty</b> After this workshop, you will have the
<ul> <li>13:00 Exercise: Pesticide residues © TrainMiC (Engman)</li> <li>14:20 Learning evaluation (Magnusson)</li> <li>14:40 Solution to the learning evaluation, and coffee</li> <li>15:00 Closing of workshop</li> <li>ods for an evaluation by the speakers</li> <li>They will advise and suggest improvements, or just comment on your good work.</li> </ul>	12:00	Lunch	opportunity to send one of your meth-
14:20Learning evaluation (Magnusson)They will advise and suggest improve ments, or just comment on your good work.14:40Solution to the learning evaluation, and coffeements, or just comment on your good work.15:00Closing of workshop	13:00	Exercise: Pesticide residues © TrainMiC (Engman)	ods for an evaluation by the speakers.
14:40Solution to the learning evaluation, and coffeeInertis, or just comment on your good15:00Closing of workshopwork.	14:20	Learning evaluation (Magnusson)	They will advise and suggest improve-
	14:40 15:00	Solution to the learning evaluation, and coffee Closing of workshop	work.

# **General Information**

Location: National Food Administration (www.slv.se), Hamnesplanaden 5, Uppsala, Sweden. Fee: SEK 6 000 excl. VAT, including documentation, coffee, lunch and dinner.

Registration: on the SP home page www.sp.se/conf (mätteknik, kemisk mätteknik), or by contacting Johan Seger; johan.seger@sp.se, phone: + 46 (0)10 - 516 52 67 before 26 June.

Accommodation: Please make your own room reservation by e-mail to uplandia@scandichotels.com before 10 August. We have reserved rooms for 16-18 September at SEK 971/night (incl. VAT) at Hotel Scandic Uplandia. The booking code is LIV160909.

# InterAgency Meeting (IAM)

NMKL is a member of IAM, which is a forum for international organisations working on methods of analysis and sampling and associated quality assurance measures. The main purpose of IAM is to promote cooperation between international organisations, and to support Codex Alimentarius and thus the governments' and industries' need for methods and quality assurance guidelines.

IAM held its 21st meeting in Budapest on 6 March, and 15 international organisations were represented. Roger Wood, IUPAC, is the chairman and Richard Cantrill, AOCS, is the secretary of IAM. For further information and minutes, please visit www.aocs.org/meetings/iam.

# International workshop: "Method Performance and the Criteria Approach: Truth and consequences?"

Together with MoniQA (Monitoring and Quality Assurance in the Food Supply Chain - a European Commission funded initiative within the Sixth Framework Programme) members of IAM organised an open workshop prior to the meeting of the Codex Committee on Methods of Analysis and Sampling. The programme was given in NMKL Newsletter No. 70, 2008. The workshop, attended by more than 90 delegates from about 60 countries, was a great success, and the organisations are willing to organise a new workshop same time next year.

![](_page_9_Picture_5.jpeg)

Dr. Roland E. Poms, ICC Secretary General/CEO, Coordinator of MoniQA welcomed the participants to the workshop.

![](_page_9_Picture_7.jpeg)

Dr. Richard Cantrill, AOCS Technical Director. In the background from left: Ralph Joseph, BIPM, Hilde Skaar Norli, NMKL, and Roger Wood, IUPAC.

Lars Jorhem, Sweden (below) talked about relevant method criteria.

![](_page_9_Picture_10.jpeg)

![](_page_9_Picture_11.jpeg)

More than 90 persons from about 60 countries participated in the workshop arranged Sunday, 8 March 2009, in Balatonálmádi, Hungary, prior to the CCMAS meeting.

#### MoniQA

(www.moniqa.org) was the main sponsor of the event. All photos (except one for obvious reasons) are taken by Roland Poms.

## The 30<sup>th</sup> Meeting of the Codex Committee on Methods of Analysis and Sampling (CCMAS). 9 - 13 March 2009, Balatonalmádi, Hungary

About 130 participants from 68 countries and 9 international organisations attended the meeting. NMKL was represented by the Secretary General Hilde Skaar Norli, who also represented AOAC International. Other participating NMKL members were; Astrid Nordbotten Norwegian Food Safety Authority, Marianne T. Werner, National Veterinary Institute, Ulla Edberg, Eva Lønberg and Lars Jorhem, National Food Administration, Sweden. Dr. Arpad Ambrus, Hungary chaired the meeting.

CCMAS aims among other to:

- consider, amend and endorse, as appropriate, methods of analysis proposed by Codex (commodity) Committees
- elaborate, consider and define procedures and guidelines for the assessment of food laboratory proficiency, as well as QA systems for laboratories

This year's meeting made considerable progress. Important papers were finalised such as:

- Analytical terminology/definitions (mostly based on VIM and ISO).
- How to establish numeric values for

method criteria. The criteria, which NMKL has been involved in the elaboration of, give the laboratories freedom to choose any appropriate, collaboratively validated method, as long as it meets the method performance criteria.

 The "dispute-document", which gives guidance on how to handle dispute situations when the exporting and importing countries do not obtain the same analytical results.

For further information about Codex, and CCMAS in particular, please visit

# NMKL's cooperation with AOAC International through 25 years

At AOAC's 100th Anniversary, the cooperation agreement between AOAC and NMKL was signed. The cooperation started some years earlier, in 1978, when the organisations saw the advantage of cooperation within method elaboration and validation in order to avoid double work. A need for international cooperation was recognised, and the same year IU-PAC called a meeting in London for har-

monising elaboration of collaboratively validated methods. This harmonisation meeting led to, after some roundabouts, the establishment of InterAgencyMeetings, which are held in connection with the meetings of CCMAS. At the first harmonisation meeting, only AOAC and NMKL, of the international organisations, were validating their methods collaboratively followed by statistical evaluation. Gradually, this practice was taken up by other organisations, and definitions and criteria were jointly agreed upon (IUAPC Harmonisation Protocol).

The cooperation with AOAC has existed for 25 years, which means that **AOAC INTERNATIONAL can celebrate its 125th Anniversary. NMKL extends its congratulations**.

![](_page_10_Picture_16.jpeg)

Photo from AOAC's 100th Anniversary, where the cooperation agreement between NMKL and AOAC was signed. From left: Professor Yrjö Mälkki, Finland, the then Chair of NMKL, Dr. William Horwitz, the then Executive Director of AOAC, and NMKL Secretary General Professor Lars-Aake Appelqvist, Sweden.

The cooperation between AOAC and NMKL has resulted in NMKL/AOAC and AOAC/NMKL methods. The organisation mentioned first in the heading arranged the method performance study.

Further, NMKL microbiological methods have been published on a CD distributed by AOAC.

And last, but not least, the networking between the members of the two organisations is very important.

The Secretary General of NMKL is now on the Board of Directors of AOAC International, this should be conducive to further fruitful cooperation.

![](_page_10_Picture_22.jpeg)

2008-2009 AOAC Board of Directors: (seated, I to r) Xiumei Liu, Gayle Lancette, Darryl Sullivan, Sam Page, Russ Flowers, and Hilde Skaar Norli;

(standing, I to r) James Harnly, Mark Coleman, Ronald Johnson, Douglas Hite, Stan Bacler, and Robert Brackett (not in photo: Jonathan DeVries and Barry Titlow).

Available NMKL Procedures (Guides)				
No. 23, 2008	Guide on quality assurance in microbiological laboratories. (Swedish and English)			
No. 22, 2008	Considerations regarding evaluation of immunochemical test kits for food analysis. (Swedish and English)			
No. 8, 4th Ed. 2008	Measurement uncertainty in quantitative microbiological examination of foods. (Norwegian and English)			
No. 21, 2008	Guide for sensory analysis of fish and shellfish. (English)			
No. 20, 2007	Evaluation of results from qualitative methods. (Norwegian and English)			
No. 19, 2007	Guideline for sensorial analysis of food containers/packages. (Swedish and English)			
No. 9, 2nd Ed. 2007	Evaluation of method bias using certified reference materials. (Swedish and English)			
No. 18, 2006	The use of reference materials, reference strains and control charts in a food microbiological laboratory. (Norwegian and English)			
No. 17, 2006	Guidelines for requirement specifications for food analyses. (Norwegian and English)			
No. 16, 2005	Sensory quality control. (Update of references in 2007.) (Norwegian and English)			
No. 4, 2nd Ed., 2005	Validation of chemical analytical methods. (Norwegian and English) (Spanish edition from 1996 as PDF-file)			
No. 1, 2nd Ed., 2005	Calibration and performance checking of laboratory balances. (Swedish and English)			
No. 15, 2004	Temperature control in microbiological laboratories. (Swedish and English)			
No. 14, 2004	SENSVAL: Guidelines for internal control in sensory analysis laboratories. (Norwegian and English)			
No. 5, 2nd Ed., 2003	Estimation and expression of measurement uncertainty in chemical analysis. (Swedish, English and Span- ish)			
No. 13, 2003	Volumetric control. (Danish and English)			
No. 12, 2002	Guide on sampling for analysis of foods. (Norwegian, English, Finnish and Polish)			
No. 11, 2002	Procedure for sensory analysis of drinking water. (Norwegian and English)			
No. 10, 2001	Control of Microbiological Media. (Norwegian, English and Finnish)			
No. 7, 1998	Checking of UV/VIS spectrophotometers. (English and Danish)			
No. 6, 1998	General guidelines in quality assurance for laboratories performing sensory analysis. (Danish and Finnish)			
No. 3, 1996	Control charts and control materials in internal quality control in food chemical laboratories. (Swedish and English)			
No. 2, 1995	Performance check and in-house calibration of thermometers. (Swedish and English)			
No 1, 2nd Ed., 1983	Calibration and performance checking of laboratory balances (Swedish and English)			

## NMKL

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www.nmkl.org

![](_page_11_Picture_3.jpeg)