Newsletter for The Nordic Committee on Food Analysis

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The NMKL Method Collection now offered online

Online Method Collection = Up to Date Method Collection

As of 2006, the NMKL method collection will be made available on the Internet. This means that subscribers will have access to a complete and updated collection of methods at all times. The general secretariat of NMKL is responsible for keeping the collection up to date, and for notifying subscribers when new methods are published. The methods will be available as pdf files. In order to gain access to the files from the NMKL home page, the subscribers are given a password to log on with. NMKL will retain the copyright on their methods.

In order to benefit from this service, you must notify the NMKL general secretariat, either via the web shop or by e-mail, that you would like this type of subscription instead of, or in addition to, your existing subscription.

Prices:

Online method collection/ continuous updates for 1-3 users: For subscribers: NOK 1500 For new subscriptions: NOK 2500 The password will be issued when payment has been received.

Subscription for individual methods (approx. 6 methods per year) Paper edition: NOK 1000 Pdf files by e-mail: NOK 750.

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|---------------|---------------------|-----|
| | The NMKL Home Page: | |
| \rightarrow | www.nmkl.org | |
| | e-mail: | |
| | nmkl@vetinst.no | |

ADDRESS: NMKL, Secretary General Hilde Skår Norli, National Veterinary Institute, PB 8156 Dep., N-0033 Oslo, Tel: +47 64870046.

The 59th NMKL Annual Meeting was held on the Danish Island of Rømø, 26 - 30 August 2005

About 55 NMKL members from Denmark, Finland, the Faroe Islands, Iceland, Norway and Sweden (see list on the right) participated in the 59th NMKL Annual Meeting. At the annual meeting, methods and procedures were discussed in the committees for chemistry, microbiology and sensory analysis. The work load was extensive, with approximately 50 projects listed in the working programme. (The NMKL working programme is available on the NMKL home page.) The annual meetings of NMKL are always held on extended weekends, so as to not keep the experts away from their workplaces for too long.



From the plenary discussion, where NMKL's cooperating partners informed us about their activities.

In conjunction with the NMKL annual meeting, the Nordic accreditation bodies arranged a harmonisation meeting, in which they, among many other topics, discussed the practices of the respective countries within flexible scope, use of accreditation marks, accreditation of sub-contractors, visit frequencies, reporting, and measurement uncertainty.

Margareta Hägg, FINAS, gave a summary of this meeting at the NMKL Annual Meeting. Both NMKL and the Nordic accreditation bodies see a continuing need for harmonisation meetings, and they may well be arranged in conjunction with the annual meetings of NMKL.



Photo: Roald Nilsen, NA, Margareta Hägg FINAS, Lisbeth Tillge Lund DANAK.

Referee of the Year: Urd Bente Andersen, Norwegian Institute for Food

and Environmental Analysis

The referees are NMKL's key resource. They lead the work process connected to developing / elaborating methods and arranging collaborative studies (method performance studies). In the last few years, NMKL has invited a referee to the annual meeting to give a presentation of the work being a referee entails. This year, Urd Bente Andersen from the Norwegian Institute for Food and Environmental Analysis was invited to present the first performance study of a sensory method.

Urd Bente Andersen and the Norwegian Institute for Food and Environmental Analysis have invested a significant amount of resources in establishing and collaboratively studying a method for sensory quality control of drinking water. This is the first sensory method to have been validated in a collaborative study. The method is quick and efficient, and is primarily meant to be a tool for water works and laboratories performing quality control of drinking water. The method is used to detect water samples with a deviating odour and taste, thus enabling the administration/customers to assess which measures should be taken, if any. 10 laboratories participated in the method performance study. At the meeting, Urd Bente Andersen shared with her fellow NMKL members, her experiences with preparing samples and sending them to participating laboratories. She also presented the results of the study, which proved the method to be suitable for its purpose. On a final note, Urd Bente Andersen told the meeting that she found the referee work to be very inspiring. For additional information on the method, see page 8.

Urd Bente Andersen and the Norwegian Institute for Food and Environmental Analysis have agreed to assist NMKL in holding courses in sensory control of drinking water in 2006, in the various Nordic countries. Information on where and when these courses will be held, will be published on the NMKL home page.

The following institutions were represented at the annual meeting:

Denmark:

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- Danish Meat Research Institute
- Danish Environmental Protection Agency, Water
- Danish Institute for Food and Veterinary Research
- Danish Institute for Fisheries Research
 - Scanpharm A/S
- Regional Veterinary and Food Control Authority of Århus
- Chr. Hansen A/S
- DANAK
- Regional Veterinary and Food Control Authority of Vejle
- SSI (Statens Seruminstitutt)
- Danish Institute of Agricultural Sciences

The Faroe Islands:

 Levnedsmiddel, miljø og veterinærstyrelsen (Institute for food, environmental and veterinary issues)

Finland:

- National Veterinary and Food Research Institute
- National Food Administration

Iceland:

- Umhverfisstofnun (Environment and Food Agency)
- Norðurmjólk (Dairy Company)
- Rannsóknaþjónustan Sýni h.f (Syni Laboratory Service)
- Rannsóknastofnun fiskiðnaðarins (Icelandic Fisheries Laboratories)

Norway:

- Norwegian Institute for Food and Environmental Analysis
- AnalyCen
- National Veterinary Institute
- National Institute of Nutrition and Seafood Research
- Norwegian Food Research Institute
- Norwegian Food Safety Authority, regional office at Ås
- The Norwegian School of Veterinary Science
- Labnet

Sweden:

- National Food Administration
- Iggesund Paperboard,
- Procordia Food AB

In addition to updates from the Nordic Committee of Senior Officials for Food Issues and its working groups, NordVal and CEN, the NMKL members also received some interesting information on the extensive EU project called:

"PathogenCombat"

Vicki Lei from the Royal Veterinary and Agricultural University (KVL) in Denmark, talked about the project, which is a part of the EU's 6th framework programme, and is coordinated by KVL. The full title of the project is "Control and prevention of emerging and future pathogens at cellular and molecular level throughout the food chain".

The project counts 44 participants from 15 European countries and Australia. The project stretches over 5 years and is expected to gain a prominent position within European food research. Vicki Lei is the project manager and Professor Mogens Jacobsen (KVL) is the coordinator. In this project, the entire food chain is studied in an attempt to develop methods for predicting the pathogenic effect of various microorganisms at the time of consumption of the finished product. At the same time, efforts are being made to develop new technologies to detect the pathogenic microorganisms and prevent them from growing in food. An important part of the project is to develop methods for use in the food production industry, as well as informing food production companies of the new developments.



In October, the PathogenCombat project established its own home page. See www.<u>pathogencombat.com</u> for more information about the project.

Ole Bjørn Jensen, Denmark, re-elected as chairman of NMKL

In the many years NMKL has existed, there has been great stability in the chairmanship of the organisation. Ole Bjørn Jensen has been the chairman of NMKL since 1989, and the organisation was extremely pleased when he agreed to put himself up for re-election for another period of 4 years. Mr. Jensen has been an active member of NMKL since 1976, making this year's annual meeting on Rømø, the 30th meeting he has attended. Mr. Jensen is the Managing Director and owner of Scanpharm A/S, an all round medical firm which deals with everything from development, production, clinical testing and laboratory control to marketing and export and import of medicine and health food products.

Ole Bjørn Jensen is an active listener, with a unique ability to see the bigger picture, draw conclusions and think visionary. He is a great asset to NMKL – and to many others. In addition to running Scanpharm A/S and being chairman of NMKL and the Danish National Committee of NMKL, Ole Bjørn Jensen has a number of other important posts, such as:

Chairman of BFID (the association of pharmaceutical industrial businesses in Denmark), Chairman of DANAK (the Danish Accreditation and Metrology Fund), Chairman of CCURE ApS, Vice President of Europharm (SMC), Board member of Eurolab Danmark (the association of laboratories in Denmark) and Board member of DFM (Danish Fundamental Metrology)



Research Manager, Dr. Philos. Kåre Julshamn, Norway,

re-elected as chairman of the chemical committee of NMKL - Sub Committee 3.

Research Manager, Dr. Philos. Kåre Julshamn, has also been involved in NMKL's work since 1976. Mr. Julshamn became an active member of NMKL in 1977. At the annual meeting on Rømø, he was re-elected as chairman of the chemical committee of NMKL for another period of 4 years. Thus, NMKL may benefit from Mr. Julshamn's extensive competence, experience and leadership skills through his 5th period as chairman of this committee. In the years 1990-1998, Mr. Julshamn was also chairman of the Norwegian National Committee of NMKL.

Kåre Julshamn is head of the programme for documentation and monitoring at the National Institute of Nutrition and Seafood Research (NIFES), previously known as the Institute of Nutrition, Directorate of Fisheries. Furthermore, Mr. Julshamn has part-time employment as a professor of food chemistry at the University of Bergen.

Mr. Julshamn and NIFES have been particularly active in NMKL, and have been in charge of the elaboration and collaborative studies of several NMKL methods and procedures. His list of scientific publications is too long to count. Mr. Julshamn is a great asset to NMKL and to many other organisations. Several methods which Mr. Julshamn has elaborated for NMKL, have also been adopted by AOAC INTERNATIONAL and by CEN/TC 275. Mr. Julshamn participates actively in WG 10 of CEN/TC 275 (trace elements / heavy metals). Furthermore, he has been appointed as an expert in two working groups in the Norwegian Scientific Committee for Food Safety, where one of his most important tasks is to focus on the uncertainty and quality of the data in connection with conclusions that are drawn.



Margrét Geirsdóttir, Deputy Chairman

of the Icelandic National Committee of NMKL

Margrét Geirsdóttir will be the Deputy Chairman of the Icelandic National Committee for the coming 2 years, as Chairman Franklin Georgsson has been given leave of absence to work in Mozambique.

Margrét Geirsdóttir is a project manager at the microbiological laboratory at Umhverfisstofnun (Environment and Food Agency) in Iceland, where she mainly works with pathogenic bacteria and food poisoning. She is a contact person for WHO, works with elaboration of methods and participates in various projects within the area of microbiology. Furthermore, she is the laboratory quality manager. Margrét Geirsdóttir has been an active member of NMKL since the beginning of 2002, when she joined the organisation as a secretary for the Icelandic National Committee. Ms. Geirsdottír has also for several years been a member of the Nordic consultation group for proficiency testing programmes which is headed by the National Food Administration of Sweden.

The NMKL Annual Meeting decided to **withdraw** the following method: NMKL Method No. 151: Shigella bacteria.

Detection in foods.

The method is withdrawn as most laboratories use NMKL Method No. 174, 2nd Ed. 2002: "*Shigella spp.* PCR method for detection in foods." NMKL Method No. 174 is considered to better than No. 151. Withdrawing the method means that the method is taken out of the method collection and should no longer be referred to as an NMKL method.



Validation of test kits

NMKL does not validate test kits. In the Nordic countries, NordVal evaluates these methods. For information on which test kits have been approved by NordVal, how to obtain an approval and who to contact, see the NordVal home page: www.nmkl.org/nordval.

New NMKL methods

NMKL Method No. 40, 2nd Ed., 2005:

Fat content. Determination in milk using a butyrometer - the Gerber method.

The method describes the so-called Gerber method for butyrometric determination of fat in both non-homogenized and homogenized milk. The method can be used for whole milk (3-5%) as well as semi-skimmed milk (1-3%). A given amount of the sample is transferred to a butyrometer containing sulphuric acid. The acid breaks down the protein. The fat content is then separated from the hydrous phase by centrifuging. The separation is facilitated by adding *iso*-amyl alcohol. After some minutes in a water bath the scale of the butyrometer for the top and the bottom of the fat column are read. This gives the % fat content.

Compared to the previous edition of NMKL method No 40, published in 1960, this method is more up to date. The reading temperature is among others lowered from 67 ± 2 °C to 65 ± 2 °C, which is in accordance with the DIN and ISO standards. This somewhat lower temperature results in somewhat lower results. Further the ratio between the two iso-amyl alcohols is stated to be: $91\% \pm 2\%$ 3-methyl-butan-1-ol and $9\% \pm 2\%$ 2-methyl-butan-1-ol, in order to give the optimal results. The method is not validated collaboratively. The repeatability and reproducibility given in the method is related to the scale of the butyrometer with examples given in percent.

The referee of this method is Dr. Erik Wolthers, Danish Institute for Food and Veterinary Research. To support the referee in the work the following contact persons were appointed: Timo Lukkarinen (City of Helsinki, Environment Centre, Finland), Þorsteinn Karlsson (Osta- og smjörsalan sf., The Icelandic Dairy Produce Marketing Association), Øyvin Stray Pedersen and Gudmund Bråthen (The Norwegian Institute for Food and Environmental Analysis).



NMKL Method No. 110, 2nd Ed., 2005:

Total Solids (Water). Gravimetric determination in milk and milk Products.

The method describes a gravimetric method for determination of the total solids content (water content) of milk and cream (including fermented products), all types of preserved milk, casein and caseinates, butter, ice cream and all types of cheese with the exception of whey cheese. A known quantity of sample is dried at constant temperature (102 °C) to constant mass. The loss in mass of the sample after drying is determined, which is the amount of total solids.

Dr. Erik Wolthers, Danish Institute for Food and Veterinary Research has revised this method. The revision consisted of an update of the methods references and the list of literature. The following contact persons were appointed to support Wolthers in revising the method: Sonja Latvakoski, (Valio, Finland), Kristín Halldórsdóttir (Norðurmjólk, Iceland), Gudmund Bråthen (The Norwegian Institute for Food and Environmental Analysis), Karin Bergkvist, Per Persson, (Arla Foods, Sweden). The method is not validated collaboratively.



NMKL Method No. 144, 3rd Ed., 2005: *Enterobacteriaceae*. Determination in foods and feeds.

The revision of this method consisted of including the results from the collaborative validation, conducted already in 1993, where 13 laboratories participated. The samples included in the study were Freeze-dried cultures of *Enterobacteriaceae* strains, which were mixed with normal flora from minced pork meat, chicken, ox liver and sprouts.

Enterobacteriaceae is determined by inoculating specific volumes of dilutions of foods in violet red-bile-glucose agar (RVG). Bacteria belonging to the family *Enterobacteriaceae* form pink to red colonies with or without a precipitation zone. After incubation at 37 °C for 22-26 hours a representative selection of colonies are confirmed by the oxidase test. The results of the collaborative study are given in the method. No false positive results were obtained, but four false negative results. The repeatability standard deviation the results were estimated to 0.18 log cfu/ml and the reproducibility standard deviation to 0.79 log cfu/ml.

Per Norberg, National Food Administration, Sweden, arranged the collaborative validation in the 90ties and has supported the revision of this method.

NMKL Method No. 152. 2nd Ed., 2005: *Penicillium verrucosum –* Ochratoxin A producing. Detection in food and feed.

Ochratoxin A (OTA) is a mycotoxin with carcinogenic, nephrotoxic, teratogenic, immunotoxic and possibly neurotoxic properties. It occurs naturally in a variety of plant products, such as cereals, coffee beans, cocoa beans, and dried fruit, but may also be

carried over to products of animal origin via contaminated feed. The main contributors to dietary intake of OTA are cereal and cereal products. Therefore The Commission of the European Communities has established a maximum limit on 5 μ g/kg of OTA on cereal grains. On dried vine fruit the max. limit is 10 μ g/kg

Reported fungal OTA producers belong to Aspergillus and *Penicillium* species such as *A. ochraceus*, *A. sulphureus*, *A. niger*, *A. carbonarius*, *Neopetromyces muricatus*, *Petromyces alliaceus*, and *P. verrucosum* and *P. nordicum*.

The two OTA producing *Penicillium* species have different habitats. *P. nordicum* is most often found on meat and cheese whereas *P. verrucosum* is only found on plants. On cereals *P. verrucosum* is the only known producer of OTA and detection of *P. verrucosum* on cereals indicates a risk of OTA contamination.

NMKL Method No. 152, 1995 proposed to use DRYES [dichloran



rose bengal yeast extract sucrose agar] as a medium for detecting *P. verrucosum*. However, in 1992 a new medium DYSG [dichloran yeast extract sucrose 18% glycerol agar] was introduced. Later investigations have shown that DYSG is an even more convenient medium compared to DRYES in detecting *P. verrucosum*. Results from a collaborative study comparing DYSG and DRYES have shown no significant differences on counts of *P. verrucosum*.

Normally it is recommended to use surface disinfection of food such as grains before direct plating to permit enumeration of fungi actually invading the food. However, results from a recent investigation showed that infection of *P. verrucosum* often was zero when using surface disinfection in cereal samples with considerable amounts of OTA – whereas *P. verrucosum* was found in high number by direct plating without treatment with surface disinfection. Until more results concerning surface disinfection are generated it is therefore recommended to include untreated grains (food particles) without surface disinfection when fungi are determined in food and feed.

Using DYSG and either dilution or direct plating (or both) it is possible to count the number of colony forming units (cfu) per gram in particles of food or the percentage infection of *P. verrucosum* in surface disinfected particles. Using non-surface disinfected particles the percentage infestation is counted. The dilution plate count of *P. verrucosum* is obtained by a surface plating technique. After homogenizing a specified quantity (40 g), a serial dilution is carried out. Of each dilution 0.1 ml is spread plated onto the culture medium in duplicate. After incubation at 20.0 ± 1.0 °C for 7 days, the colonies with a characteristic appearance are counted. The number of cfu of *P. verrucosum* per gram of food is calculated. The direct plating of *P. verrucosum* is performed by placing 100 food pieces (often kernels) on the medium, 5-10 on each 9 cm Petri dish (this procedure may be repeated with surface disinfected food pieces). The number of food pieces from which at least one colony of *P. verrucosum* grows out is recorded and the percentage infestation (or infection) is calculated.

Flemming Lund, National Food Administration, Sweden has elaborated the method. Assisting persons on this method have been Liisa Vanne (VTT, Bioteknik, Finland), Margrét Geirsdóttir (Environment and Food Agency, Iceland), Ida Skaar (National Veterinary Institute, Norway), Marianne Boysen (National Food Administration, Sweden). The method is not validated collaboratively.

NMKL Method No. 173, 2nd Ed., 2005 Ash, gravimetric determination in foods.

As NMKL had several gravimetric methods for determination of ash in foods, NMKL determined to harmonise these. The following methods are replaced by NMKL method No. 173, 2. Ed., 2005:

- NMKL Mthod No. 7, 2. Ed., 1987: Ash. Determination in grain and flour.
- NMKL Method No. 23, 3. Ed., 1991: Moisture and ash. Gravimetric determination in meat and meat products.
- NMKL Method No. 108, 1984: Ash. Determination in milk and milk products.
- NMKL Method No. 128, 1989: Ash. Gravimetric determination in foods.
- NMKL Method No. 173, 2002: Ash. Gravimetric determination in foods.

The ash is determined by ashing the sample to constant weight, if necessary after previous drying. The ashing is carried out in a muffle furnace at 550 ± 25 °C. For determination of ash in milk and milk products the temperature has to be 525 ± 25 °C.

The method was collaboratively validated in 1986/87 and in 1999. It is validated for ash content from 0.07 to 8.0 g/100g. For the method validated in 1987 (NMKL Method No. 128, 1989), a stricter temperature control on furnace was described, $(550 \pm 5 \,^{\circ}C)$ then the method studied in 1999 (NMKL Method No. 173, 2002). The results from both studies are given in the new edition of NMKL Method No. 173. The collaborative study in 1999 demonstrated that there is no need for the strict temperature control of $\pm 5 \,^{\circ}C$. Both the validation studies were conducted by 14 laboratories and are together validated on 14 foodstuffs with satisfactory results except from the validation performed on apple purée.

Birthe Mortensen, Danish Meat Research Institute arranged the collaborative study in 1986/87 and Arne Højgård Jensen, Danish Veterinary and Food Administration, Regional Veterinary and Food Control Authority, Århus, Denmark arranged the validation study in 1999 in connection with the elaboration of NMKL Method No. 173.

NMKL Method No. 182, 2005: PSP toxins. Determination of paralytic shellfish poisoning toxins in shellfish by HPLC/ fluorescence.

The method is intended for quantitative determination of paralytic algae toxins that can be present in shellfish. They are causing the syndrome called Paralytic Shellfish Poisoning (PSP). The method can determine the PSP-toxins STX, NEO, GTX1,4 (together), GTX2,3 (together), dcSTX, dcGTX2,3 (together), C1,2 (together) and C3,4 (together).

PSP toxins constitute a group of at least 18 structurally related toxins with varying toxicity. They are neurotoxins and are mainly produced by the algae of the genus *Alexandrium*. The toxins accumulate in shellfish feeding on the algae with no apparent negative effect on the shellfish. However, humans and animals eating the shellfish, experience neural symptoms of poisoning and severe cases have to be treated in a respirator.

The toxins are extracted from the shellfish tissue with 1% acetic acid. The extract is cleaned up on a C₁₈ solid phase cartridge and a solid phase ion exchange cartridge (weak cation exchange) is used to fractionate the toxins into 3 groups. The toxins are oxidized into fluorescence compounds that are separated on a C₁₈ HPLC-column and detected with a fluorescence detector.

The method is elaborated by Dr. J.F. Lawrence et.al, Health Candada. Health Canada has also arranged the collaborative study. The results of the study are given in the method and are also published in the Journal of AOAC International.





18 laboratories from 14 different countries participated in the collaborative study. A total of 21

different samples, consisting of 3 blanks, 5 spiked samples, and 12 naturally PSP contaminated samples. The matrices included were mussels, scallops, clams and oysters with toxin concentrations ranging from ca. 5 μ g/kg to 2500 μ g/kg. Homogenized mussel tissues were spiked at levels between 50 μ g/kg and 1500 μ g/kg and the recovery in these samples were from 53% to 94%. Of the reported results no outliers were obtained and the precisions were satisfactory, with HorRat values below 1.7.

Associating professor Tone Norman Asp, Norwegian School of Veterinary Science has elaborated this method for NMKL. Contact persons involved have been: Kevin Jørgensen (Danish Institute for Food and Veterinary Research), Carola Ranta (Customs Laboratory, Finland), Helga Gunnlaugsdóttir (Icelandic Fisheries Laboratories), Håkan Johnsson (National Food Administration, Sweden).

Corrections to

NMKL Method No. 98, 4th Ed., 2005: Moulds and yeasts. Determination in food and feed.

In section 5.3 on the preparation of dichloran rose bengal agar (DRBC) and in section 5.5, Trace metal solution, the water in the salt complexes were omitted. A correction paper was therefore forwarded to the NMKL subscribers in August.

In 5.3: Magnesium sulphate should be replaced with Magnesium sulphate heptahydrate (MgSO₄ 7H₂O).

In 5.5: Zink sulphate should be replaced with Zink sulphate <u>heptahydrate ($ZnSO_4 7H_2O$)</u>

and Copper sulphate should be replaced with Copper sulphate pentahydrate (CuSO₄ 5H₂O)

NMKL Method No. 183, 2005: Sensory quality test of drinking water.

The method describes a sensory quality control test, the samples are evaluated against odourless and taste free reference water, and the assessors give points for the properties odour and taste. When an assessor detects a deviation from the reference water in a sample, he shall indicate this. For drinking water, it is recommended to use a scale ranging from 0 - 4 points, where 0 points mean that the sample does *not* deviate from the reference water. A sample which is evaluated at 2 points or higher, must always be annotated. The assessors shall use annotations from a nomenclature list which describes known deviations for odour and taste in drinking water.

Ten laboratories participated in the collaborative validation study. Each laboratory analysed 12 samples in two rounds. In addition, all laboratories were given two calibration samples which were used to calibrate the assessors before the evaluation of the actual samples.

The study showed that the laboratories to a great extent agreed on their evaluations both in terms of point scores and descriptions of deviations.

The Nordic Committee of Senior Officials for Food Issues, EK-Livs supported financially the collaborative study arranged at the Norwegian Institute for Food and Environmental Analysis. Urd Bente Andersen, the Norwegian Institute for Food and Environmental Analysis has been the referee on the topic and represented the work at the NMKL annual meeting. To support Andersen in her work, the national committees appointed the following experts: Grethe Hyldig (Danish Institute for Fishery Research), Juhani Airo (City of Helsinki, Environment Centre, Finland), Ása Þorkelsdóttir (Icelandic Fisheries Laboratories), Bengt Dahlberg (Göteborg Water and Wastewater Works, Sweden).



Photo: The referee of the year: Urd Bente Andersen, Norwegian Institute for Food and Environmental Analysis.

New NMKL procedyre:

NMKL Procedure No 4, 2nd version, 2005: Validation of chemical methods.

This NMKL procedure is a revision of the procedure with the same title published in 1996. The Nordic Committee of Senior Officials for Food Issues, EK-Livs has given financial support for this revision. The following experts have been involved in the revision:

Per Lea The Norwegian Food Research Institute Matforsk (author) Harriet Wallin, National Food Administration, Finland (project leader) Inge Meyland, Danish Institute for Food and Veterinary Research Elísabet Jona Sólbergsdóttir, University of Iceland Johan Lindeberg, National Food Administration, Sweden

The NMKL procedure describes validation of methods for different purposes ^L based on possible existing validation materials. The amount of work laid down to test the method before use, depends on the available documentation of the method and what the method is tend to be used for.

Validation of a method means examination and determination of the parameters of the method. This can either be done by a single laboratory (internal validation), or by several laboratories (collaborative study). Verification of a method is an examination of a single laboratory's ability to perform the analysis in accordance with the method parameters established in the validation.



The procedure recommends:

- When no documentation of the method of interest is available (an internal developed method or a method found in the literature with no performance characteristics given) the method should be fully validated.
- A method published in a scientific literature and vital method performance characteristics are given or the method is well established verification or an internal validation should be conducted.

When using a collaborative validated method, but on other matrices or with other instrumentation than included in the described method, a verification of trueness and precision, and possibly also detection limit should be carried out.

> Is the method collaboratively validated, only the trueness and precision need to be verified.

NMKL procedure no 4, can be ordered on the Web shop or by forwarding an e-mail to: nmkl@vetinst.no. The procedure is available in Norwegian and English.

| NMKL PROCEDURE No. 4 (2005) | Validation of chemical analytical methods Page: 1 of 30 Version: 2 Date: 31.10.2005 Accounted: Ole Bjørn Jensen | |
|---|---|---|
| Validation of chemical ana | lytical methods | |
| 1. INTRODUCTION 2. VALIDATION OF A CHEMICAL METHO 3. THE VALIDATION PROCEDURE 2.1 PLAN | D | WEB |
| 3.2. SPECIFICITY 3.3. STANDARD CURVE 3.4. PRECISION 3.4.1. Quantitative determinations 3.4.2. Qualitative determinations 3.5. TRUENESS 3.5.1. Analyses for which certifies materials are available 3.5.2. Analyses for which referer 3.5.3. Analyses for which certifies 3.5.4. Analyses for which certifies | s ed reference materials or other reference ice methods are available sed proficiency testing schemes are available ed reference materials, reference methods or are not available | SHOP |
| 3.6. CONCENTRATION AND MEASU 3.7. LIMIT OF DETECTION 3.7.1. Quantitative determination 3.7.2. Qualitative determinations 3.8. LIMIT OF QUANTIFICATION AN 3.9. RUGGEDNESS 3.10. EVALUATION OF VALIDATION | N RESULTS | The procedures are not included in NMKL subscription. |
| 4. DOCUMENTATION OF VALIDATION AN 5. MONITORING 5.1. CONTINUOUS MONITORING 5.2. MONITORING FOLLOWING CH. | ND VERIFICATION ANGES IN THE PROCEDURE | |
| 6. WORD LISTS 7. REFERENCES AND RELEVANT LITERA | TURE | |

New NMKL procedure:

NMKL Procedure No. 16, 2005: Sensory quality control

This procedure describes how it is possible to measure whether a product has the desired sensory properties, and to specify the nature and size of the deviation from the expected quality. The properties the product should have, are described in a product specification, and the deviation is expressed by way of a fault nomenclature. The quality is measured by means of a point scale. The procedure may be used as a tool for monitoring product quality.

It describes how to prepare and carry out evaluations of a product's appearance, odour, taste and consistency. It is vital that the performance of the analysis is supervised by a well qualified person, and that the panel members (assessors) are selected and trained for the relevant task. Following a standardised method also makes it possible to compare results over time.

The project has been supported financially by NMKL and the participants' institutions: Ole Inge Skorbakk, Norway, (project leader), Grethe Hyldig, Danish Institute for Fishery Research, Denmark Leena Lilleberg, The National Veterinary and Food Research Institute of Finland, Ása Þorkelsdóttir, Icelandic Fisheries Laboratories, Steffen Solem, The Norwegian Institute for Food and Environmental Analysis, Karin Hallin-Saedén, Norrmejerier, Sweden.

The Norwegian sensory method committee has contributed to the finalisation of the procedure. The committee consists of:

Urd Bente Andersen, The Norwegian Institute for Food and Environmental Analysis, Marit Rødbotten, The Norwegian Food Research Institute, Matforsk Per Lea, The Norwegian Food Research Institute Matforsk. The procedure is in press.



Translation of NMKL procedures into Spanish The Laboratory of Quality Supervision CUBACONTROL Corp. (LQS)

in Havana, Cuba

The Laboratory of Quality Supervision CUBACONTROL Corp. (LQS) in Havana, Cuba, belongs to the Cuban company of commercial supervision named CUBACONTROL Corp. International Supervision Services. LQS carries out quality supervision by means of rendering independent laboratory services to foreign companies residing in or outside of Cuba, and also to Cuban entities requesting these services. CUBACONTROL is head of the Secretariat of the Cuban Technical Committee of Standardization on Methods of Analysis and Sampling, and is thus involved in the Codex Committee on Methods of Analysis and Sampling. Through Codex, the Cuban delegation has learned about NMKL, and cooperation has been established. CUBACONTROL SA has translated NMKL Procedure No. 4 (method validation), NMKL Procedure No. 5 (measurement uncertainty within chemistry) and NMKL Procedure No 8 (measurement uncertainty within microbiology) into Spanish, and uses these procedures in their standardisation work.



LQS provides services to customers who require commercial supervision of samples for quality purposes, through physicalchemical, microbiological and sensorial evaluation analysis. This independent service is offered by means of customers' applications, coming from exporters, importers or other officers of diverse economic branches. LQS has 48 accredited methods (accredited according to ISO 17025). The laboratory has been accredited since 1997. The staff consists of 27 employees out of whom 13 have graduated from university. For further information about LQS and CUBACONTROL, please visit: www.cubacontrol.com.cu.

Dr. Nelson Fernandez Gil, CUBACONTROL, writes among others to NMKL:

"Through the working sessions of the Codex Committee on Methods of Analysis and Sampling (CCMAS), held in Budapest, the LQS, has established relationships with NMKL since 1997 and during these years of exchange it has received testing methods and procedures of this Nordic institution, which have become a very useful collaboration for the Cuban analytical community in the food branch. The Procedures 4, 5 and 8 about Testing Validation and Measurement Uncertainty have been especially very useful, as much for chemical tests as microbiological ones, which have functioned as fundamental reference, among others, in the elaboration of similar Cuban standards. The NMKL procedures before mentioned have been translated to the Spanish language by the LQS and those versions have been sent to NMKL who places them available for the Spanishspeaking analytical community, through its web site. LQS and CTN-46, as a part of this community before mentioned, wish to thank through the pages of NMKL Newsletter to this subregional European institution and very especially to its General Secretary, Mrs. Hilde Skår Norli, for her interest in the development of these mutually profitable relationships, which we wish to continue strengthening in the future."

Dr. Nelson Fernandez Gil

NMKL procedures

| No. 1, 2. Ed., 2005 | Performance checking of laboratory balances. NEW in English !! <i>[Available in Swedish and English]</i> |
|------------------------|--|
| No. 2, 1995 | Performance check and in-house calibration of thermometers. [Available in Swedish and English] |
| No. 3, 1996 | Control charts and control materials in internal quality control in food chemical laboratories. [Available in Swedish and English] |
| No. 4, 2. Ed., 2005 | Validation of chemical analytical methods. [Available in Norwegian and English. The 1996 version is available in Spanish]. |
| No. 5, 2. Ed., 2003 | Estimation and expression of measurement uncertainty in chemical analysis. [Available in Swedish, English and Spanish]. |
| No. 6, 1998 | General guidelines for the quality assurance of sensory laboratories. [Available in Danish and Finnish] |
| No. 7, 1998 | Checking of UV/VIS spectrophotometers. [Available in Danish and English] |
| No. 8, 2. Ed., 2002 | Measurement of uncertainty in microbiological examination of foods. [Available in Swedish, English and Spanish]. |
| No. 9, 2001 | Evaluation of results derived from the analysis of certified reference materials. [Available in Swedish and English] |
| No. 10, 2001 | Control of microbiological media. [Available in Norwegian and English] |
| No. 11, 2002 | Procedure for sensory analysis of drinking water. [Available in Norwegian and English] |
| No. 12, 2002 | Guide on sampling for the analysis of foods. [Available in Norwegian, Finnish, Polish and English] |
| No. 13, 2003 | Volumetric control. [Available in Danish and English] |
| No. 14, 2004 | SENSVAL: Guidelines for internal control in sensory analysis laboratories. [Available in Norwegian and English] |
| No. 15, 2004 | Temperature control in microbiological laboratories. [Available in Swedish and English] |
| No. 16, 2005 | Sensory quality control. [In press in Norwegian and English] |
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Workshop: Bromic flame retardants

Time: February / March 2006 (specific date not yet set) Location: Uppsala, Sweden Arranged by: EK-Livs (the Nordic Committee of Senior Officials for Food Issues) / NMKL Language: English

A workshop on bromic flame retardants will be arranged some time during the first quarter of 2006. At this workshop, Nordic and international experts within chemistry and toxicology will meet to identify relevant products / matrices, and, from a toxicology point of view, define and assess various interesting polybromic substances. The purpose of the workshop is an invention resulting in proposals for relevant analysis methods, that can be used in quality control work within food production and research, and also in a more scientific context. The workshop will last 2-3 days.

If you would like to <u>contribute</u> to the workshop programme, please contact the project manager Håkan Johnsson at the National Food Administration of Sweden (e-mail: <u>hajo@slv.se</u>, tel: +46 18 175705) or the general secretariat of NMKL (e-mail: nmkl@vetinst.no). The detailed programme, invitation and registration information will be distributed and published on the NMKL home page as soon as it is available.



Chemistry - Microbiology - Sensory topics

Time: 24 August 2006 Location: Thon Hotel Opera, Oslo Arranged by: The Norwegian National Committee of NMKL Language: Scandinavian languages

In connection with the 60th NMKL Annual Meeting, the Norwegian National Committee of NMKL will arrange a seminar on multivariable analysis. The seminar is a follow-up to a previous seminar, which NMKL arranged at KVL (Royal Veterinary and Agricultural University, Denmark) in 2003. The purpose of the seminar is to show how multivariable techniques are used, and what they can be used for when processing results and developing new methods.

Multivariable methods are used for comparing and finding correlations between large amounts of data. They may be used for large, complex problems, or seemingly simpler tasks, for example describing which properties are important for different varieties of apples or different types of brown bread. They also make it possible to see whether there are any probable correlations between different analysis parameters obtained by chemical, physical, microbiological and sensory measurings.

The seminar will be useful for professionals within research, quality control work, product development and production, who use chemical, microbiological and/or sensory analysis in their daily work or who simply take an interest in the analyses of food and food related products.

It will be possible to participate actively at the seminar. If you would like to <u>contribute</u> to the seminar programme (lectures / other input), please contact the secretary of the Norwegian National Committee, Dag Grønningen, National Veterinary Institute (e-mail: <u>dag.gronningen@vetinst.no</u>, tel: +47 2321 6214) or the general secretariat of NMKL. The detailed programme, invitation and registration information will be distributed and published on the NMKL home page as soon as it is available.

Workshop: Will the changed laboratory structure in the Nordic countries have an impact on food safety?

Time: 1-2 February 2006 Location: Sigtuna, Sweden Arranged by: EK-Livs (the Nordic Committee of Senior Officials for Food Issues) / NMKL

Language: Scandinavian languages

20 years ago, there was a multitude of food laboratories in the Nordic countries. There were public private. and commercial laboratories offering a wide spectre of analysis services within both microbiology and chemistry, and sometimes also within sensory topics, to support risk evaluation, rule development and control. Today, the number of competent laboratories is greatly reduced, and some analysis services can't even be performed within the Nordic counties.

During these two days in February, we will discuss the question of whether the changed laboratory structure in the Nordic countries will have an impact on food safety.

NMKL would like to invite persons from the food control / inspection authorities (central and local government level) as well as trade organisations, to a closed workshop.

would lf you like to participate, or know someone who should definitely be invited to the workshop, please contact the NMKL general Edberg. secretariat or Ulla National Food Administration (e-mail: uled@slv.se).