NMKL Newsletter 79

Nordic Committee on Food Analysis
www.nmkl.org

No. 79, December 2011

Contents

AOAC Europe / NMKL / NordVal International Symposium on Rapid Methods - chemical, microbiological and sensory analysis of foods

7 - 8 May 2012
at IDA Mødesenter, Copenhagen,
Denmark

Language: English

Program:

Day 1 : Plenary session regarding rapid methods

Day 2: Three parallel sessions, chemistry, microbiology and sensory, respectively.

Both days: Poster sessions and exhibition

The symposium is a cooperation between AOAC Europe and NMKL/NordVal.

We would like contributions from as many as possible.

Would you like to contribute with presentations, posters or exhibition material?

Topics

- Experiences with the use of rapid methods
- PCR vs. Elisa
- Multi targeting methods
- Validated methods for:• Allergens• Biotoxins• Mycotoxins• Marine toxins• Veterinary drugs• Chemical contaminants
- Development of fast GC
- Development of rapid methods on sensory analysis (napkin)
- Reference materials within sensory methods
- Test kits for sensory analysis
- Non-destructive methods such as IR, FTIR, NMR
- Multivariate data analysis
- Rapid methods in connection with food adulteration
- Relevant methods for detection of pathogenic bacteria

Deadlines: 10 December 2011 - Abstracts, Oral presentations
5 January 2012 - Abstracts, Poster presentations

Do you wish to participate actively, please contact nmkl@vetinst.no.

For more information, please visit

http://www.nmkl.org/AOAC-NMKL2012/AOAC-NMKL2012.htm

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Determination of net content and drained weight of food

NMKL Method No. 55, 3. Ed., 2011

Astrid Nordbotten, Norwegain Food Safety Authory, referee of the method

This method describes the determination of net content and drained weight in foods with liquid packing medium, such as water, juice, brine, oil or syrup. The method is updated and harmonised with AOAC 968.30 and methods used in Codex.

The determination is based on the weight of the packaging before and after emptying, and the weigh of solids after draining by the use of a sieve.

Astrid Nordbotten, Norwegian Food Safety Authority, served as referee on this method.

A validation of the method was carried out by Arne Højgaard Jensen, Danish Veterinary and Food Administration, Denmark.

Gram-negative bacteria in pasteurized milk and cream. Detection of recontamination. NMKL Method No. 192, 2011

Anders Christiansson, Swedish Dairy Association, referee of the method

Gram-negative bacteria do not survive high temperature short time (HTST) pasteurization (i.e. a minimum heat treatment at 72 °C for 15 seconds). The presence of Gram-negative bacteria in pasteurized milk and cream therefore indicates post pasteurization contamination. Although the level of contamination is very low, often only a few bacteria per liter, growth of Gram-negative bacteria can limit the shelf life of the milk and cream.

The method that is intended to detect recontamination by Gram-negative bacteria in milk and cream is described. Generally, *Pseudomonas* spp dominate. This method has been developed for Gram-negative bacteria in milk and cream but could be applicable to other heat-treated food matrices.

A package of milk or cream is incubated at 25 °C for 24 hours or at room temperature for 28 hours, in order to allow bacterial growth to reach detectable levels. The package is opened and a sample (10 µl following incubation at 25 °C or 100 µl following incubation at room temperature) is streaked on the surface of a VRBG agar plate. The plates are incubated at 30 °C for 24 ± 2 hours. Growth of 5 or more bacterial colonies, irrespective of colony morphology or color, indicates the presence of Gram-negative bacteria in the package. The KOH test can be used to confirm the presence of Gram-negative bacteria.

This method has been elaborated by Anders Christiansson, Swedish Dairy Association and Madeleine Ripsweden, Arla Foods Sweden.Contact persons have been: Niels Skovgård (DK), Anu Surakka (FI), Franklin Georgsson (IS) and Kari Dommarsnes (NO).

Do you have subscription on NMKL methods?

With an online subscription, the newest version of the method is only a key-click away.

Call for laboratories !

Would you like to participate in a collaborative validation on:

- Histamine. Determination by HPLC in fish

Laboratories that participate must have the equipment needed. The method tested has to be followed to the letter. It is the method that is being tested, not the laboratory.

The method describes extraction of histamine from a homogenised sample with 0,6 M perchloric acid. A specific amount of internal standard is added on dilution of the extract. For separation, HPLC is used with gradient elution and post-column derivatisation with o-phtaldialdehyd (OPA).

OPA derivates can be detected by fluorescence, which gives increased sensitivity compared to UV detection and with assumed less interferences. It is previously reported that OPA derivatives may be a bad choice due to their instability . However, with the use of a post column derivatisation these problems are negligible.

For detection, fluorescence is used with an excitation wave length of 365 nm and an emission wave length of 418 nm.

Jarle Wang Andersen, Nofima Ingredients, Bergen, Norway, is the study director of this method.

More laboratories are needed in the study.

If you would like to participate, please contact Jarle Wang Andersen on jarle.wang-andersen@nofima.no

- Methylmercury. Determination with Isotope Dilution-GC-ICPMS in foodstuffs

This method is elaborated for the quantitative determination of mono-methylmercury (MMHg) (10-5000 µg/kg dry weight), mainly in marine biota samples.

The sample is spiked with an appropriate known amount of Hg-isotope enriched MMHg and extracted using tetramethylammonium hydroxide (TMAH). After pH adjustment, derivatisation and extraction, the organic phase is analysed using GC-ICPMS. The GC separates the different mercury species before MMHg is atomised and ionised in the high temperature of the ICP. The ions are extracted from the plasma by a set of sampler and skimmer cones and transferred to a mass spectrometer where the ions are separated by their mass/charge ratio and determined by a pulse-count and/or analogue detector.

This method is also elaborated as a CEN method. EU is funding the method validation study, as mandated work under CEN/TC 275 WG 10.

Stig Valdersnes, National Institute of Nutrition and Seafood Research, NIFES, is the study director of the method.

If you would like to participate, please contact Stig Valdersnes on sva@nifes.no

The following NMKL methods have been withdrawn and should no longer be referred to:

- No. 4, 192, 2. Ed.: Inefficient cleansing. Chemical method for detection on cups, dishes and plates
- No. 20, 1982, 2. Ed.: Methods for microbiological examinations of butter.

New NMKL Protocol:

NMKL Protocol No. 5, 2011: Analytical Quality Control - Guidelines for the Publication of Analytical Results of Chemical Analyses in Foodstuffs

These guidelines describe analytical quality control procedures that are intended to help ensure the quality and reliability of published analytical data. They are based on quality criteria such as sampling procedures, method selection and description, use of certified reference materials and participation in proficiency testing. In addition, they include some advice on how to organise the description of the quality assurance in the manuscript. The quality assurance criteria are summarised in a Check-list, shown to the right.

The protocol is intended for use by all parties involved in the publishing process, i.e. manuscript authors, journal editors and manuscript reviewers/referees. The check list can be of assistance to the persons involved in order to simplify/standardise the reviewing process.

The protocol is elaborated under the leadership of Lars Jorhem, National Food Administration, Sweden, and can be downloaded free of charge under Protocols on NMKL's homepage. The protocol is available in English.

Paragraph in protocol	Analytical parameter	Reviewer comments	
4.1	SAMPLING DESCRIPTION	Sufficient	Insufficient
4.1.1	*Sampling representativity*		
4.1.2	*Sampling procedure, transport, storage*		
4.2	METHODOLOGY		
4.2.1	*Appropriate choice of method*		
4.2.2	*Clarity/transparency of description*		
4.2.3	*Awareness of interferences*		
4.2.4	*Determination of recovery*		
4.2.5	*Handling of sample blanks*		
4.2.6	*Reporting limit*		
4.2.7	*Definition of the reporting limit*		
4.2.8	*Number of replicates*		
4.3	CERTIFIED REFERENCE MATERIALS Available and used in study	Yes	No
4.3.1	*Relevant matrix*		
4.3.2	*Relevant concentration*		
4.3.3	*Clearly referenced*		
4.4	PROFICIENCY TESTING Available and used in study		
4.4.1	*Relevant matrix*		
4.4.2	*Relevant concentration*		
4.5	MEASUREMENT UNCERTAINTY Described/presented		
4.5.1	*Random error (standard deviation)*		
4.5.2	*Systematic error (bias)*		
4.6	UNEXPECTED RESULTS		
4.6.1	*If yes in 4.6, verified by other method*		
4.6.2	*If yes in 4.6, verified by other laboratory*		
5	ACCEPTABLE ORGANISATION OF AQC		
5.1	*Description of AQC procedures acceptable*		
5.2	*Presentation of AQC-results acceptable*		

Publication Series in NMKL:

- **NMKL Methods** (available as a subscription and single copies)
- NMKL procedures (available as single copies)
- NMKL protocols (may be downloaded for free at www.nmkl.org)
- NordVal Certificates (may be downloaded for free at www.nmkl.org)

Now also available in Finnish:

NMKL Procedure No. 11, 2010:

Sensory analysis of drinking water

Leena Lilleberg, Finnish Food Safety Authority Evira, has translated the procedure into Finnish. This procedure gives a general description of how to perform sensory analysis of drinking water. It describes the selection and training of assessors, preparations for the analyses, instructions for the assessors, requirements for the testing area and equipment, and how to treat and prepare samples. This procedure is meant to serve as a guide for how a laboratory or any other business should carry out assessment of drinking water. It is mainly written for analyses performed by a sensory panel in a laboratory. But it may also give examples for those who perform analyses in the field, e.g. at a tapping point in the mains system. The procedure was also described in NMKL Newsletter No. 74.

Foodproof® *E.coli* O157 Detection Kit - NordVal Certificate 026

NordVal has renewed the certificate for **food**proof® *E.coli* O157 Detection Kit in combination with **food**proof® Short-Prep II Kit for PCR analysis.

The Kit is manufactured and supplied by Biotecon Diagnostics GmbH, Germany.

There are no statistical differences in the results obtained by this method and the compared reference method, EN ISO 16654:2001: Horizontal method for the detection of *Escherichia coli* O157**.**

The method is applicable to the detection of *E.coli* O157 DNA isolated from enrichment cultures prepared by various valid methods. *E.coli* O157 is determined by PCR. The method is applicable to foods.

Method performance characteristics:

Inclusivity: 100%. 60 different strains of *E.coli* O157 tested were positive.

Exclusivity: 100%. 73 non-*E.coli* O157 serovars and 47 non-*E.coli* strains tested were negative.

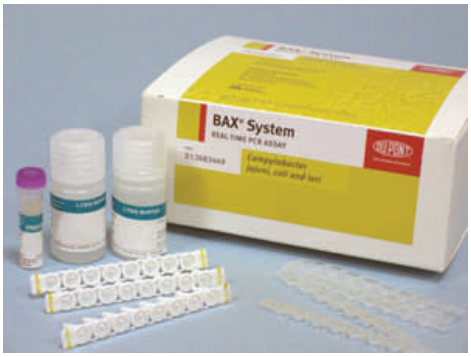
Relative sensitivity: 100%

Relative specificity: 96.2%

Relative accuracy: 98.8%

Limit of detection: 1-10 cells/25 g

BAX Q7 for the determination of *Campylobacter jejuni, coli* and *lari* - NordVal Certificate 039



BAX® Q7 *Campylobacter jejuni, coli* and *lari* is manufactured by DuPoint Qualicon, USA and distributed by Thermo Fisher Scientific.

BAX® Q7 *Campylobacter jejuni, coli* and *lari* is compared against the reference method EN ISO:10272-1:2006: Horizontal method for detection and enumeration of Campylobacter spp. -- Part 1: Detection method.

Bax Q7 is NordVal approved for testing on poultry faeces.

The method

- Detects and quantifies all three species in the same sample
- Has < 90 minutes processing time
- Gives same-day results for highly contaminated samples without enrichment
- Has a detection level of 100 cfu/g
- Has satisfactory sensitivity and selectivity

There is no statistical significant difference between the results obtained with Bax Q7 and the reference method.

The selectivity is a measure of

a) **the inclusivity:** detection of the target microorganism from a wide range of strains, and

b) **the exclusivity**: the lack of interference from a relevant range of non-target microorganisms.

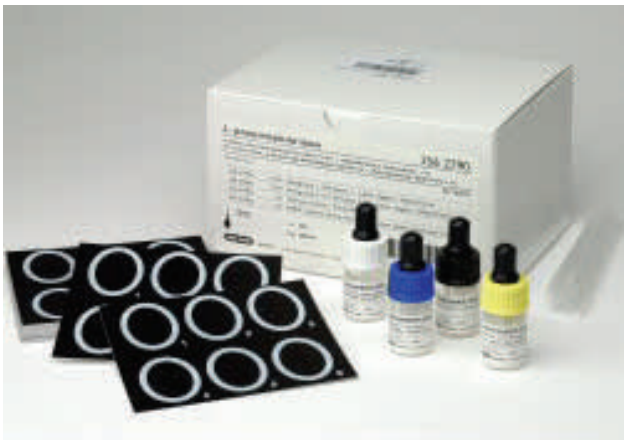
The relative sensitivity is the ability of the alternative method to detect the analyte compared to the reference method.

The relative specificity is the ability of the alternative method not to detect the target microorganism when it is not detected by the reference method.

The relative accuracy is the degree of correspondence between the response obtained by the alternative method and the reference method on artificially inoculated samples.

The limit of detection is the lowest level the method can detect the analyte with a certain probability.

RAPID'*L.mono* - NordVal Certificate 022

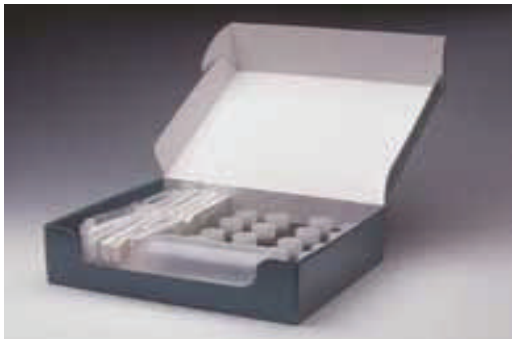


RAPID'*L.mono* from BIO-RAD is applicable to the detection and the enumeration of *Listeria monocytogenes* and to the detection of other *Listeria* species in food and environmental samples.

The principle of the RAPID'*L. Mono* medium relies on the chromogenic detection of the *Listeria monocytogenes* phosphatidylinositol-specific phospholipase C and on the inability of these species to metabolise xylose. After 24 ± 2 hours of incubation, *Listeria monocytogenes* forms characteristic blue (pale blue, grey blue to dark blue) colonies without a yellow halo. Colonies formed by other species of *Listeria* are white, with or without a yellow halo. The selective mixture in the medium allow the inhibition of most interfering flora (Gram-positive and Gram-negative bacteria, yeast and mould). Thus RAPID' L mono detects *Listeria monocytogenes* in 24 hours and other *Listeria* species in 24 and 48 hours.

NordVal has concluded that it has been satisfactorily demonstrated that there are no statistical differences in the performances of *RAPID' L. mono* and the reference method, EN ISO 11290-1(1996/ amendment 2004).

TRANSIA™PLATE *Salmonella* Gold - NordVal Certificate 001



TRANSIA® PLATE *Salmonella* Gold from Biocontrol is based on a on a three-step, sandwich-type ELISA using

- a microtitre plate with divisible strips coated with antibodies specific to *Salmonella,*
- and ready-to use reagents.

The method is tested on foods, feeds and environmental samples.

The method describes:

- Enrichment on buffered peptone water (BPW) incubated for 16-20 h at 37°C ± 1°C
- Inoculation of 0.1 mL of the pre-culture broth in 10 mL of Rappaport Vassiliadis Soya (RVS) broth incubated for 18-24 h at 41.5°C ± 1°C, followed by
- TRANSIA® PLATE *Salmonella* Gold test after heating of 1 to 2 mL of the enrichment broth RVS in boiling water for 20 minutes.
- The reading of the microtitre plate is carried out using a spectrophotometer at a wavelength of 450 nm.

TRANSIA™ PLATE Salmonella Gold was first approved in 2001. Since then the method has been modified. The modifications are validated.

The results show that the method's performance has not been reduced. The limit of detection, sensitivity, specificity and selectivity are all satisfactory.

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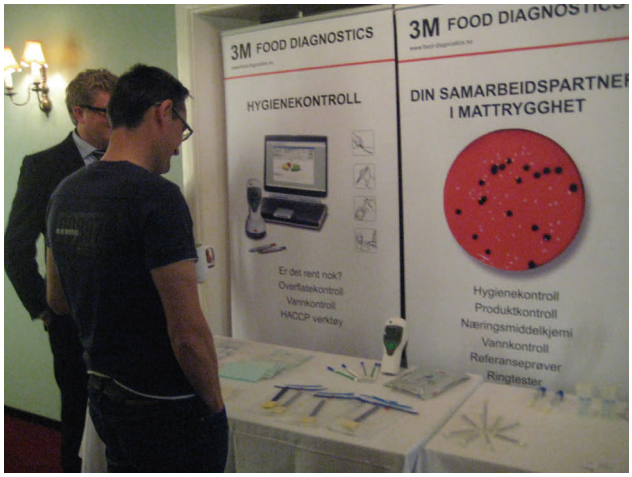
To see the NordVal Certificates and the information about the performance of the methods, click on the blue link under "list of methods" under "NordVal" on www.nmkl.org.

The methods approved by NordVal can be used according to the EU Regulation on microbiological criteria.

The results from the validation may also be used in quality assurance.

Analyte	Certificate of the kit (click on the title to open Certificate):					
Campylobacter	*Campylobacter* real-time PCR					
Campylobacter	BAX ® Q7 *Campylobacter jejuni*, coli and lari chicken cloacae swabs	Oxoid				39
Coliforms	3M Petrifilm. Coliform Count Plate	3M Health Care, USA	All Foods	ISO 4832: 2006 ISO 4831: 2006 NF V08-06	Approval: 01.06.2011 Valid until: 01.06.2013	013
Coliforms/E. coli	Compact Dry EC Method for the Enumeration of Total Coliforms/*E.coli*	HySerVe GmbH & Co. KG, Germany	All Foods	ISO 4832: 2006 ISO 16649-2: 2001	Approval: 01.12.2008 Valid until: 01.12.2012	036
Coliforms/ E.coli	3M Petrifilm *E.coli*/Coliform Count Plate	3M Health Care, USA	All Foods	ISO 4832: 1991 ISO 16649-2: 2001 AOAC 991.14	Approval: 01.06.2011 Valid until: 01.06.2013	014
Coliforms	Compact Dry CF Method for the Enumeration of Total Coliforms	HySerVe GmbH & Co. KG, Germany	All Foods	ISO 4832: 2006	Approval: 01.12.2008 Valid until: 01.12.2012	035
E.coli	RAPID' E.Coli 2	Bio-Rad, France	All Foods	ISO 16649-2: 2001, ISO 4832: 2006	Approval: 01.06.2011 Valid until: 01.06.2013	020
E.coli	3M Petrifilm Select *E.coli* Count Plate	3M Health Care, USA	All Foods	ISO 16649-2: 2001	Approval: 28.04.2006 Valid until: 28.04.2012	027
E.coli O157	food proof® E. coli O 157 Detection Kit in combination with foodproof® ShortPrep II Kit	Biotecon Diagnostics GmbH, Germany	All Foods	ISO 16654: 2001	Approval: 01.11.2011 Valid until: 01.11.2013	026
					Approval:	

Seminar on SAMPLING, held in Bergen 26 August 2011



3M exhibited and presented simple hygiene wipes, swabs and socks for sampling purposes

The Norwegian National Committee of NMKL organized a seminar on sampling, 26 August 2011 at Rick's Stuene in Bergen, Norway. There were about 55 participants from all the Nordic countries.

At the seminar, we learned about laws and regulations in connection with sampling of vegetables, fish and fish products (by the Norwegian Food Safety Authority). Representatives from the food industry (BAMA and Lerøy) held lectures on sampling in practice, and from NIFES (National Institute of Nutrition and Seafood Research), we learned about sampling of fish for research purposes.

The participants were also introduced to the extensive EU Baseline project: "Selection and improving of fit-for-purpose sampling procedures for specific foods and risks". The Norwegian Veterinary Institute is heavily involved in the project dealing with new needs and schemes for food sampling. Food safety objectives (FSOs) and performance objectives (POs) are new terms that will lead to a more risk-based food legislation when implemented. However, before implementation, more knowledge about how risk changes along the farm-to-fork chain is needed, and sampling schemes to detect latent risks at an early stage must be developed. The results will be translated into recommendations to the EC and end users.

The international year of chemistry was marked at the seminar by a lecture given by Leif Sydnes from the University of Bergen.

From the 65th NMKL Annual Meeting

Photo above:
From Voss and the NMKL Annual Meeting

The 65th NMKL Annual Meeting was held at Fleischer Hotel in Voss, Norway. The meeting is for NMKL experts only, and this year 45 members of the National Committees of Denmark, Finland, Iceland, Norway and Sweden participated.

At NMKL meetings, work takes place partly in plenary sessions and partly in sub committees. The plenary sessions included information on NMKL's activities and updates from invited partners such as the Nordic Council of Ministers' Department of Food and its working groups, NordVal, CEN (the European Organization for Standardization) and AOAC International.

This year, the participants were also introduced to a coming NMKL procedure on recovery, as well as emergency response plans and networks for laboratories in Sweden, and the EU project BioTracer: Improved biotraceability of united micro-organisms and their substances in food and feed chains.

NMKL's working program

NMKLs working program is discussed in the following committees:

- Sub 2: Microbiology. Chair: Flemming Hansen, Danish Technological Institute
- Sub 3: Chemistry. Chair: Harriet Wallin, Finnish Food Safety Authority Evira, Finland
- Sub 4: Sensory. Chair: Gunnar Forsgren, Iggesund Paperboard, Sweden

Microbiology:

- *Clostridium botulinum* and botulinum toxin
- Patogen *Yersinia enterocolitica.* Culture method and PCR method for detection in foods
- *Shigella.* Culture method and PCR method for detection in foods
- *Clostridium difficilie.* Detection in foods
- Quality control of PCR analysis
- Comparison of NMKL and ISO methods of relevance for EU 2073/2005
- Verification of microbiological methods
- Cryptosporidium in water

Chemistry:

- Methyl mercury. Determination with isotope dilution GC-ICPMS
- Histamine. HPLC determination in fish
- Sterols and stanols. GC determination in phytosterol fortified foods
- Nitrate and nitrite. Spectrophotometric determination after reduction with zinc and Griess' reaction
- Fat. Determination using NMR in foods
- Forbidden colouring agents in mixed spices
- Folate in milk and milk products
- Recovery. Estimation and expression .
- Calibration of NIR and IR
- PSP toxins. HPLC determination in shellfish

Sensory:

- Quality control of drinking water (toxicological evaluation)
- Guidelines for reporting sensory data
- Measurement uncertainty in sensory analysis
- Sensory analysis of meat and meat products
- Guidelines for sensory evaluation of bread
- Methods for discriminate tests (binomial tests).

Available NMKL Procedures

No 1, 2nd Ed. 2005	Calibration and performance checking of laboratory balances
No 2, 1995	Performance check and in-house calibration of thermometers
No 3, 1996	Control charts and control materials in internal quality control in food chemical laboratories
No 4, 3rd Ed. 2009	Validation of chemical analytical methods
No 5, 2nd Ed. 2003	Estimation and expression of measurement uncertainty in chemical analysis
No 6, 1998	Yleiset ohjeet aistinvaraisten laboratorioiden laadunvarmistukseen (avail. Danish/ Finnish)
No 7, 1998	Checking of UV/VIS spectrophotometers
No 8, 4th Ed. 2008	Measurement of uncertainty in quantitative microbiological examination of foods
No 9, 2nd Ed. 2007	Evaluation of method bias using certified reference materials
No 10, 2001	Control of microbiological media
No 11, 2nd Ed. 2010	Procedure for sensory analysis of drinking water
No 12, 2002	Guide on sampling for analysis of foods
No 13, 2003	Volumetric control
No 14, 2004	SENSVAL: Guidelines for internal control in sensory analysis laboratories
No 15, 2004	Temperature control in microbiological laboratories
No 16, 2005	Sensory quality control
No 17, 2006	Guidelines for requirement specifications for food analyses
No 18, 2006	The use of reference materials, reference strains and control charts in a food microbiological laboratory
No 19, 2007	Guideline for sensorial analysis of food containers/packages
No 20, 2007	Evaluation of results from qualitative methods
No 21, 2008	Guide for sensory analysis of fish and shellfish
No 22, 2008	Considerations regarding evaluation of immunochemical test kits for food analysis
No 23, 2008	Guide on quality assurance in microbiological laboratories (replaces NMKL Report No. 5)
No 24, 2010	Guidelines for quality assurance for food chemical laboratories