Review

Reference Materials for the Determination of Contaminants in Milk and Milk Products

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Abstract

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In this review, the latest information is given about the reference materials for the determination of contaminants in milk and milk products. Certified reference materials for the determination of contaminating chemical elements, organochlorine pesticides, polychlorinated biphenyls, dibenzodioxins, dibenzofurans, and aflatoxin M_1 are presently available. Properties of these reference materials and possibilities for their use are discussed together with the maximum tolerances of contaminants in milk and milk products. The basic information about the producers and distributors of the reference materials in this area is provided.

Keywords: reference materials; contaminants; milk; milk products

The use of the reference materials is currently one of the necessary conditions to assure quality systems in analytical chemistry laboratories. Taking into account the great importance of the reference materials for chemical and physical measurements, the Reference Materials Committee (REMCO) was established by the International Organization for Standardisation (ISO) in 1975 as an international authority in this field. The aim of the Committee is to ensure a broad international effort for the harmonisation and promotion of the reference materials and their production and applications. The Committee compiled and issued an underlying document ISO/REMCO – The role of reference materials in achieving quality in analytical chemistry (1997).

The determination of contaminants in foods is a specific area of analytical chemistry, posing great demands on the accuracy of chemical measurements of very low concentrations of analytes. The choice of a suitable reference material is substantially influenced by the type of the analysed matrix. One of our tasks as a part of the grant project *Development of methods for determination of risk chemical elements and some organic contaminants in milk and milk products and their harmonization with EU methods* was to review the information on the reference materials used in this field. As milk analyses are carried out in many analytical laboratories, we believe that it is useful to present information on the existing variety of the reference materials and their potential use for the determination of contaminants in milk and milk products.

General characteristic of the reference materials

The basic definitions and terms used in connection with the reference materials have been listed in ISO

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Guide 30 – Terms and definitions used in connection with reference materials (1992). The explanations of these definitions and terms in Czech language and other information on the reference materials have been published in specialised literature dealing with the validation of analytical methods (Suchánek *et al*. 1997) and chemical metrology (Mestek & Suchánek 1998; Terminological Commission 2000).

Reference material (RM) is defined as a material or a substance of which one or more property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (ISO Guide 30). This general definition covers all types of RMs that are usually classified in accordance with the purpose of their use as reference materials of chemical composition (pure substances, standard solutions, matrix RMs), reference materials of physical properties, and microbiological reference materials.

As regards metrological quality, there are two basic categories of reference materials used in analytical laboratories: the certified reference materials (CRMs), and other reference materials (called e.g. laboratory, internal or in-house reference materials). CRM is a reference material accompanied by a certificate; each certified value must be traceable to an accurate realisation of the measuring unit and must be accompanied by an uncertainty at a stated level of confidence (ISO Guide 31 – Reference materials – Contents of certificates and labels 1996).

The Czech Metrological Institute or an authorised metrological centre are legally permitted to carry out the certification of reference materials in the Czech Republic (Metrology Act No. 505/1990 with its latest amendments, especially Act No. 119/2000 and others). An imported reference material that was certified in another country by a competent international organisation or by a certification authority of another country is also a certified reference material (according to the Decree No. 262/2000 of the Ministry of Industry and Trade ensuring uniformity and accuracy of measuring instruments and measurements). The producers or importers distributing the certified reference materials and other reference materials are obliged to indicate their metrological characteristics in the accompanying documents.

Besides the certified values, so called indicative (uncertified) values are given for some analytes in the case of most CRMs. In general, these are the values that have not been certified or have not complied with all certification requirements. Uncertified values are usually given different names in producer catalogues, e.g. "reference", "recommended", "indicative" or "information" ones. The level of reliability of these values should be assessed from the respective certification report. The established terminology of the National Institute of Standards and Technology (NIST) uses two categories of uncertified values: reference values (values with indicated uncertainty approaching certified values by their quality) and information values (provided only as additional information on chemical composition of a reference material). International Atomic Energy Agency (IAEA) uses similar categorisation (recommended and information values).

Stability and homogeneity are the basic requirements for the reference materials. Information on these characteristics and methods used for their testing should be given in the certification report. Instructions for use and storage should be also included in documents accompanying every CRM. Sometimes instructions for CRM handling are not respected by their users which may lead to serious errors.

Two basic requirements should be respected if CRM suitable for the validation of an analytical method is to be selected: (1) CRM matrix should be similar to the matrix of the analysed samples and (2) the analyte concentration in CRM should agree with the concentration range of the analysed samples.

Although CRM supply on the world market is large, sometimes it is difficult to select a suitable CRM, especially with respect to its similarity with the analysed matrix. If any laboratory fails to buy suitable CRM that will meet its needs, they can try to develop their own (internal) reference material. Development and use of internal RM should guarantee metrological traceability and eliminate systemic errors that would influence general analytical uncertainty (Suchánek *et al*. 1997).

Information sources on reference materials

The largest computer database of the reference materials is COMAR (Code of Reference Materials), which has been recommended by ISO/REMCO as a basis of international information system on reference materials (Mestek & Suchánek 1998). More than ten thousand reference materials are

registered in COMAR database and classified into eight main sections according to the type of matrix. The reference materials for food analysis are included in the section "RMs for the Quality of Life". COMAR database is administered in the Czech Republic by EURACHEM-CZ (http://www.eurachem.cz); specialists of this organisation can carry out computer retrieval on request. Further information on COMAR can be found on the website http://www.comar.bam.de.

Catalogues of the leading world producers represent another important source of information on the up-to-date variety of reference materials. Besides the production of their own reference materials, some producers specialise in the distribution of the reference materials of other manufacturers (e.g. LGC Promochem in Europe). A survey of the producers and distributors of the reference materials discussed in this review is presented below.

Reference materials are recommended to be purchased only through mediation of authorised distributors. Reference materials in the Czech Republic are distributed and sold by several specialised companies; more detailed information is available on the website of "Laboratory Guide" catalogue (http://www.labo.cz). RM distributors are obliged to co-ordinate their activities in accordance with the latest guidelines of ISO/REMCO and respective laws (Metrology Act No. 505/1990 with its latest amendments and Decree No. 262/2000).

Survey of the producers and distributors of RMs (cited in this review)

- Agrifood Research Finland (ARC), FIN-31600 Jokioinen, Finland; e-mail: jorma.kumpulainen@mtt.fi; http://www.mtt.fi/english;
- Institute for Reference Materials and Measurements (IRMM/BCR), Reference Materials Unit, Retieseweg, B-2440 Geel, Belgium; e-mail: bcr.sales@irmm.jrc.be; http://www.irmm.jrc.be/mrm.html;
- International Atomic Energy Agency (IAEA), Analytical Quality Control Services, Agency's Laboratories, A-2444 Seibersdorf, Austria; e-mail: aqcs@iaea.org; http://www.iaea.org/programmes/aqcs;
- LGC Promochem (authorised distributor of IRMM/BCR), Mercatorstrasse 51, D-46485 Wesel, Germany; e-mail: de@lgcpromochem.com; http://www.lgcpromochem.com;
- National Institute of Standards and Technology (NIST), Standard Reference Materials Program, Building 202, Gaithersburg, MD 20899, USA; e-mail: srms@nist.gov; http://ts.nist.gov/srm;
- National Research Centre for Certified Reference Materials (NRC-CRM), Office of CRMs, Beijing, China; e-mail: nrccrm@public3.bta.net.cn.

Maximum tolerances of contaminants in milk and milk products

The need of reference materials in analytical food laboratories is closely connected with the existence of maximum tolerances for contaminants in foods. This area is legally guided by the Act No. 110/1997 on Foodstuffs and Tobacco Products followed by the Decree No. 322/1999 laying down maximum limits of pesticide residues in foodstuffs, Decree No. 273/2000 of the Ministry of Health laying down the maximum permitted residues of veterinary medications and biologically active substances used in livestock production in foodstuffs and foodstuff raw materials, and Decree No. 53/2002 of the Ministry of Health laying down chemical requirements on health safety of particular foodstuffs and foodstuff raw materials, and conditions for the use of foodstuff additives.

Decree No. 53/2002 lists eleven chemical elements among the contaminating substances in foodstuffs (Appendix No 2), nitrates, aliphatic chlorinated hydrocarbons, polycyclic aromatic hydrocarbons, polychlorinated biphenyls, mycotoxins and some other groups of contaminants. Three types of tolerance values are used in the above decree to limit the contents of contaminating substances:

(a) maximum admissible tolerance (MAT); if it is exceeded, the foodstuff is excluded from circulation,

(b) admissible tolerance (AT); conditions for the foodstuff assessment according to this value are defined in the Appendix No 2,

(c) special tolerance (ST) is laid down for foodstuffs for special diets; if this value is exceeded, the foodstuff cannot be used for the intended special purpose.

Table 1 shows the maximum tolerances of the contaminating chemical elements in milk and milk products according to the Decree No. 53/2002. If the decree does not specify tolerances for some elements (e.g. chromium or tin) in milk and milk products, the tolerances valid for foods in general are used (group A or B of foods in general). Group A includes milk and milk-based baby and infant food,

Table 1. Tolerances of contaminating chemical elements in milk and milk products (all values in mg/kg according to the Decree No. 53/2002)

Element	Milk	Milk-based baby and infant food	Milk products (yogurts, curds etc.)	Soft and processed cheeses	Hard cheeses	Cream	Butter
	(MAT)	(ST)	(AT)	(AT)	(AT)	(AT)	(AT)
Al	1.0	1.0	–-	–	–	–	–
As	0.05	0.05	0.2	0.2	0.5	0.1	0.1 (MAT)
Cd	0.01	0.005	0.05	0.05	0.06	0.05	0.05
Cr	0.2* (AT)	0.2* (AT)	4.0*	4.0*	4.0*	4.0*	4.0*
Cu	0.4	0.8	2.5	20.0	20.0	2.5	0.4 (MAT)
Fe	50.0* (AT)	50.0* (AT)	80.0*	80.0*	80.0*	80.0*	5.0
Hg	0.01	0.003	0.02	0.02	0.02	0.01	0.01
Ni	0.1	0.1	6.0*	6.0*	6.0*	6.0*	6.0*
Pb	0.02	0.02	0.3	0.5	0.7	0.1 (MAT)	0.1 (MAT)
Sn	100.0* (AT)	100.0* (AT)	200.0*	200.0*	200.0*	200.0*	200.0*
Zn	10.0	12.0	20.0	60.0	80.0	20.0	5.0

*Tolerance for foods in general; MAT = maximum admissible tolerance; AT = admissible tolerance; ST = special tolerance

group B covers other types of milk products (e.g. curds, cream, butter, cheeses).

For the assessment of dried or concentrated food products, the tolerances for the original raw materials should be used adjusted by a coefficient expressing the respective loss of water. Thus for milk powder, the tolerances established for liquid milk should be multiplied by coefficient 8 (at average dry matter content 12.5%, Kratochvíl *et al*. 1985). Tolerances for baby and infant food are indicated for products after reconstitution, i.e. after mixing with water according to the manufacturer's instructions. The calculation of tolerances for such products in the form of powder will be similar to that for milk powder (e.g. the manufacturer's instructions for Sunar recommend to mix 13 g of powder with 90 ml of water).

In the group of milk products, the tolerance of nitrates is given only for the milk-based baby and infant food: 15 mg/kg (ST). The admissible tolerances for aliphatic chlorinated hydrocarbons (as the sum of eight substances) are laid down for milk (0.001 mg/kg), milk products (0.002 mg/kg), hard cheeses (0.003 mg/kg) and butter (0.01 mg per kg). The admissible tolerances for polycyclic aromatic hydrocarbons (individual compounds) are 0.002 mg/kg for fats, 0.001 mg/kg for group A of foods and 0.01 mg/kg for group B of foods in general. The total content of polycyclic aromatic hydrocarbons should not exceed tenfold tolerances for the individual compounds. The tolerances for polychlorinated biphenyls and aflatoxin M_1 are listed in Table 2.

Maximum limits for pesticide residues (MLR) are laid down by the Decree No. 322/1999. The MLR value for the purpose of the decree is maximum admissible toxicologically acceptable content of pesticide residues in a plant product, crop or foodstuff. MLR values for milk and milk products with the fat content of up to 4% are given for the entire product, if the fat content is higher than 4%, they are given for fat. Table 2 shows MLR values for organochlorine pesticides (DDT and its derivatives, hexachlorobenzene, lindane and other HCH isomers). Most of these substances have not been used in the Czech Republic for many years, and they are called "extraneous residues" in the decree.

Besides chlorinated substances mentioned above, the decree sets MLR values for the more than forty pesticides in milk which are presently used. These are e.g. some organophosphates (fenitrothion, chlorpyriphos), urea derivatives (linuron, tribenuron), triazine herbicides (atrazine, metribuzin, prometryn) and other compounds. The MLR values of these substances in milk are very low (in most cases a hundredth mg/kg).

Table 2. Tolerances of some organic contaminants in milk and milk products (all values in mg/kg according to the Decree No. 322/1999 and Decree No. 53/2002)

Compound (group of compounds)	Milk and milk products (with fat content up to 4%)	Milk products (with fat content higher than 4%)
DDT (sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE, *p,p′*-TDE)	0.05	1.0*
Hexachlorobenzene	0.008	0.2*
HCH isomers (except lindane)	0.005	0.1*
γ-HCH (lindane)	0.01	0.2*
PCB (sum of congeners 28, 52, 101, 118, 138, 153, 180)	0.1 (MAT)* 0.05 (ST)*	0.1 (MAT)* 0.05 (ST)*
Aflatoxin M_1	0.00005 (MAT)	0.00005 (MAT)

*Value related to fat

Veterinary drugs are categorised into four groups according to the Decree No. 273/2000. In the first group, in which maximum limits of residues are laid down under permanent validity in accordance with the regulations of European Union, more than thirty MLR values are listed for milk as a food raw material. These limits include mainly those for antibiotics (penicillins, tetracyclines, cephalosporins), chemotherapeutics (sulfonamides) and antiparasitics (benzoimidazoles, diazinon). The MLR values range from thousandths to tenths mg/kg with respect to different properties and biological effects of these substances.

Current assortment of the reference materials for the determination of contaminants in milk and milk products

1. RMs for the determination of contaminating chemical elements

The available reference materials for the determination of elemental contaminants in milk and milk products are listed in Table 3. The table includes all reference materials registered in COMAR database which can be designated as milk or milk product, and where more than one certified or reference value are indicated for some of the elemental contaminants listed in the Decree No. 53/2002. All data were verified in the latest catalogues of the respective producers (IRMM/BCR, IAEA, NIST, LGC Promochem) on their internet pages and in the literature cited below. The same method was used to describe RMs for the determination of other contaminants (organic compounds).

For each reference material, Table 3 shows the code number (producer code), the material name and its weight per package. The data shown include the certified, reference and information values for eleven contaminating elements listed in the Decree No. 53/2002. The values for other elements are not included in the table, additional information is mentioned in the text below. The reference materials in Table 3 can be classified according to their matrix compositions into the following groups: low-fat milk powder (six), whole milk powder (two), milk-based infant food (one) and whey powder (one).

To assess the analytical methods for the determination of the trace elements in milk within a wider concentration range, the group of three reference materials "Skim milk powder" (BCR-063R, BCR-150 and BCR-151) can be recommended. BCR-063R is a material with natural contents of the certified analytes. Besides the elements indicated in Table 3, the values (mass fractions) for the major chemical elements and iodine were also certified for this material (Quevauviller *et al*. 1994). Materials BCR-150 and BCR-151 were spiked with Cd, Cu, Fe, Hg and Pb; these elements were added in the form of certified solutions to liquid milk before its further processing. More detailed information on the preparation of these materials and analytical methods used for their certification is given in the certification report (Griepink *et al*. 1984). BCR-150 is a very suitable material in the analytical practice for the determination of the contaminating trace elements in milk powder; it was used as a certified standard ensuring traceability to the internal reference material "Low-fat milk powder ELIGO" prepared in the Trace Element Laboratory of the Czech University of Agriculture in Prague (Mader *et al*. 2000).

Table 3. Reference materials for the determination of contaminating chemical elements in milk and milk products

Code number	BCR-063R	BCR-150	BCR-151	IAEA-153	IAEA-155
Material	Skim milk powder (natural)	Skim milk powder (spiked)	Skim milk powder (spiked)	Milk powder	Whey powder
Weight (g)	50	23	23	50	50
Element	Certified value (mg/kg)				
Al	–	–	–	–	(52.9)
As	–	–	–	–	(0.049)
Cd	–	0.0218	0.101	–	0.016[a]
Cr	–	–	–	–	0.59[a]
Cu	0.602	2.23	5.23	(0.57)	(0.57)
Fe	2.32	11.8	50.1	2.53[a]	(62)
Hg	–	0.0094	0.101	–	0.0026[a]
Ni	–	(0.0615)	(0.056)	–	0.54 [a]
Pb	0.0185	1.000	2.002	–	0.104[a]
Sn	–	–	–	–	–
Zn	49.0	(49)	(50)	39.56[a]	34.3[a]

Code number	SRM 1549 (NIST)	RM 8435 (NIST)	SRM 1846 (NIST)	ARC/CL-MP	GBW 08509 (NRC-CRM)
Material	Non-fat milk powder	Whole milk powder	Infant formula (milk-based)	Skim milk powder	Non-fat milk powder
Weight (g)	100	40	10 × 30	50	50
Element	Certified value (mg/kg)				
Al	(2)	(0.9)	–	–	–
As	(0.0019)	(0.001)	–	–	0.031
Cd	0.0005	(0.0002)	–	<0.005	–
Cr	0.0026	(0.5)	–	–	–
Cu	0.7	0.46 [b]	5.04 [b]	0.59	0.26
Fe	1.78	1.8 [b]	63.1 [b]	4.54	5.18
Hg	0.0003	–	–	<0.001	(0.0005)
Ni	–	(0.01)	–	–	–
Pb	0.019	0.11 [b]	–	0.016	0.034
Sn	(<0.02)	–	–	–	–
Zn	46.1	28.0 [b]	60.0 [b]	41.68	46.8

[a]recommended value according to IAEA; [b]reference value according to NIST; value in brackets = information value

With regard to the qualitative representation of analytes, the certified reference material SRM 1549 (NIST) "Non-fat milk powder" has a great information validity; besides nickel it comprises the certified or information values for all contaminating elements listed (the values of major elements are also certified). It is a material with natural content of certified analytes (Rasberry 1985). For its practical use, it should be taken into account that the concentrations of the contaminating elements are substantially lower than maximum tolerances in most cases, and that the concentrations of some elements (Cd, Hg) may be below the detection limits of analytical methods currently used.

The group of milk reference materials with low fat content includes furthermore the certified reference materials ARC/CL-MP (produced by ARC Jokioinen, Finland) and GBW 08509 (produced by NRC-CRM Beijing, China). The certificates of these materials include the values for copper, iron, lead, zinc, and also arsenic in GBW 08509.

RM 8435 (NIST) "Whole Milk Powder" is a main representative of the milk powder products with a high fat content (declared value 21.3%). It is an uncertified but very carefully characterised reference material (Ihnat *et al*. 1994); the reference values of copper, iron, lead and zinc can be useful for the analysis of these elements. Reference values of proteins, saccharides, fatty acids and vitamins have been also determined for this material (Ihnat 1994). Detailed data on the matrix composition are given also for the certified reference material SRM 1846 (NIST) "Infant formula (milk-based)" with fat content 27.1% (Sharpless *et al*. 1997). This material is intended for the determination of the basic nutrients, vitamins, and major elements. As for trace elements, the reference values are given for copper, iron, and zinc only.

An important position in the variety of RMs for the determination of chemical elements in milk products is held by the reference material IAEA-155 "Whey Powder", which is in this category the only reference material with quite a different type of matrix (high contents of whey proteins, lactose, and very low fat content). Table 3 shows the recommended values for cadmium, chromium, mercury, nickel, lead and zinc.

The reference materials for the determination of radionuclides ^{40}K, ^{90}Sr, ^{134}Cs and ^{137}Cs with code numbers IAEA-152 "Milk powder", IAEA-321 "Milk powder" and IAEA-154 "Whey powder" are also available (they are not included in the table). The recommended values of radionuclides cited above are given in Bq/kg. The materials were produced from cow's milk obtained from animals that had grazed on land contaminated with radioactive fallout resulting from the Chernobyl incident in 1986 (Radecki *et al*. 2002).

2. RMs for the determination of organochlorine pesticides and PCBs

The available variety of reference materials is shown in Table 4. For the determination of organochlorine pesticides (OCPs) two certified reference materials can be used: BCR-187 (milk powder with natural content of pesticides) and BCR-188 (spiked milk powder). Both materials are whole milk powders with the fat content of about 25%. Four and nine certified values, respectively, have been declared for BCR-187 and BCR-188 (Griepink *et al*. 1989). The values are related to dry matter. Minimum sample weight for analysis is 2 g, the recommended storage temperature in a sealed ampoule is max. 5°C. No changes in the analyte contents were demonstrated by long-range tests of stability, not even at higher temperatures (Van

Table 4. Certified reference materials (weight 20 g) for the determination of organochlorine pesticides and polychlorinated biphenyls in milk

Code number	BCR-187	BCR-188	BCR-450	
Material	Natural milk powder	Milk powder (spiked)	Natural milk powder	
Compound	Certified value (μg/kg)		Compound	Certified value (μg/kg)
HCB	1.5	37.4	PCB 52	1.16
α-HCH	1.80	20.0	PCB 118	3.3
β-HCH		12.0	PCB 153	19.0
γ-HCH	5.7	45.4	PCB 156	1.62
β-HEPO	(1.4)	32.0	PCB 170	4.8
p,p′-DDE	6.6	51.3	PCB 180	11.0
Dieldrin	(2.3)	36.1		
Endrin		6.2		
p,p′-DDT		69.0		

Values in brackets are not certified

der Paauw *et al*. 1988). It is BCR-188 that can be recommended for the regular quality control of the respective analytical methods because it comprises certified values for all OCPs listed in the Decree No. 322/1999. BCR-187 is suitable for the assessment of the respective analytical methods at the level of detection limits.

Only one certified reference material is available for the determination of polychlorinated biphenyls in milk: BCR-450 (milk powder with natural content of PCBs). Matrix properties are approximately the same as with BCR-187 and 188 (fat content 25%), recommended weight of analytical sample is 3 g. The values of six PCB congeners are certified (Table 4) including four of seven congeners listed in the Decree No. 53/2002 (congeners 52, 118, 153, 180). The values are related to dry matter. Total content (sum) of certified congeners is about 0.16 mg/kg related to fat. More detailed information is given in the certification report (Maier *et al*. 1994).

3. RMs for the determination of polychlorinated dibenzodioxins and dibenzofurans

PCDDs and PCDFs are global contaminants occurring nearly in all environmental matrices, mostly at ultratrace concentrations. Their maximum tolerances for milk and milk products are not laid down by the Decree No. 53/2002. The development and validation of the analytical methods for the determination of these compounds are a matter of sophisticated specialization. Table 5 shows the reference materials available.

Certified reference material BCR-607 (milk powder with natural contents of PCDDs and PCDFs) is recommended for the validation of an analytical system. The analytical methods used during the certification process involved capillary gas chromatography and high-resolution mass spectrometry (Tuinstra *et al*. 1997). To check the analytical system stability, a set of uncertified reference materials RM 532/533/534 can be used. Details on the preparation of these materials and the results of interlaboratory studies were published (Schimmel *et al*. 1994).

4. RMs for aflatoxin M_1 determination

Considering a large scale of mycotoxins that can be present in foods as toxic products of metabolism of various mildews, it is most important to monitor milk and milk products for aflatoxin M_1. Its potential presence can result from feed contamination by type B aflatoxins which are mostly transformed to aflatoxin M_1 in milk cow's organism. Because the toxicity of this substance is extremely high, methods with very low detection limits (immunochemical methods, HPLC) are used for its determination. This requirement is

Table 5. Reference materials for the determination of polychlorinated dibenzodioxins and dibenzofurans in milk

Code number	BCR-532 (RM)	BCR-533 (RM)	BCR-534 (RM)	BCR-607 (CRM)
Material	Natural milk powder	Spiked milk powder	Spiked milk powder	Natural milk powder
Weight (g)	60	60	60	100
Compound	Certified value (ng/kg)			
D 48	(0.10)	(0.42)	(0.65)	0.25
D 54	(0.29)	(0.86)	(1.44)	0.79
D 66	(0.17)	(0.56)	(0.80)	0.42
D 67	(0.43)	(1.27)	(1.92)	0.98
D 70	(0.16)	(0.47)	(0.80)	0.34
F 83	(0.16)	(0.39)	(0.65)	0.05
F 94	(0.1)	(0.1)	(0.15)	0.054
F 114	(0.67)	(1.96)	(3.16)	1.81
F 118	(0.34)	(1.06)	(1.75)	0.94
F 121	(0.34)	(1.03)	(1.55)	1.01
F 130	(0.39)	(1.22)	(1.90)	1.07

Values in brackets are not certified

Table 6. Certified reference materials for the determination of aflatoxin M_1 in milk

Code number	BCR-282	BCR-283	BCR-284*	BCR-285
Material	Whole milk powder (very low level)	Whole milk powder (low level)	Whole milk powder (medium level)	Whole milk powder (high level)
Weight (g)	25	25	25	25
Compound	certified value (μg/kg)			
Aflatoxin M_1	< 0.05	0.09	–	0.76

*New batch in certification

met by the available variety of reference materials that are shown in Table 6. These are reference materials BCR-282, BCR-283, BCR-284 (new batch in certification) and BCR-285 "Whole milk powder" with natural content of aflatoxin M_1 in four concentrations (Van Egmond & Wagstaffe 1987). BCR-282 is intended for the verification of the detection limit, other materials are to be used for the validation of analytical methods within the respective concentration range.

Conclusions

The currently available reference materials for the determination of contaminants in milk and milk products can be used for the determination of contaminating chemical elements, organochlorine pesticides, polychlorinated biphenyls, dibenzodioxins, dibenzofurans and aflatoxin M_1. The most frequent type of matrix is homogenized milk powder (skim or whole), other materials are milk-based infant food and whey powder.

There is a relatively large assortment of reference materials for the determination of elemental contaminants in milk powder, but certified values for some elements are missing or their concentration range is not sufficient (tin, aluminium, arsenic, nickel). No reference materials for the determination of elemental contaminants in cheese, cream, and butter are available. New reference materials LGC 7104 "Sterilized cream" and LGC 7106 "Processed cheese" present a contribution to the current assortment but they are mainly intended for the determination of major elements and nutrition constituents (Thurlow 2000).

In the information sources available we did not find any reference material for the determination in milk of veterinary drugs, polycyclic aromatic hydrocarbons, and other pesticides than OCPs although the indicated maximum tolerances cover a relatively large range of these substances. Therefore, the professional discussion should be aimed at proposals for new reference materials and arrangements for interlaboratory studies in this area.

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Souhrn

Táborský J., Hejtmánková A., Dolejšková J. (2003): **Referenční materiály pro stanovení cizorodých látek v mléce a mléčných výrobcích**. Czech J. Food Sci., **21**: 111–120.

V článku je zpracován přehled informací o aktuálním sortimentu referenčních materiálů pro stanovení cizorodých látek v mléce a mléčných výrobcích. V současné době jsou dostupné certifikované referenční materiály pro stanovení kontaminujících chemických prvků, organických chlorovaných pesticidů, polychlorovaných bifenylů, dibenzodioxinů, dibenzofuranů a aflatoxinu M_1 v mléce. Vlastnosti jednotlivých referenčních materiálů a možnosti jejich použití jsou diskutovány v návaznosti na hygienické limity cizorodých látek pro mléko a mléčné výrobky. Uvedeny jsou také základní údaje o producentech a distributorech referenčních materiálů v této oblasti.

Klíčová slova: referenční materiály; cizorodé látky; mléko; mléčné výrobky

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