

## Chocolate Authenticity Control Concerning Compliance with the Conditions for Adding Cocoa Butter Equivalents as Laid Down by Directive 2000/36 EC

IVAN BOHAČENKO, ZDENKA KOPICOVÁ and JITKA PINKROVÁ

*Food Research Institute Prague, Prague, Czech Republic*

### Abstract

BOHAČENKO I., KOPICOVÁ Z., PINKROVÁ J. (2005): **Chocolate authenticity control concerning compliance with the conditions for adding cocoa butter equivalents as laid down by Directive 2000/36 EC.** Czech J. Food Sci., **23**: 27–35.

Chocolate samples (22 in total, including 11 samples of milk chocolate) were bought from retail store in Prague and tested for their CBEs contents in relation to the declaration of CBE addition on the product labels. The modified method of Padley and Timms was employed for determining selected triglycerides (C50, C52 and C54). The presence of CBEs in chocolate was evaluated using the following relationship:  $\%C50 < 44.095 - (0.737 \times \%C54)$ . The content of CBE in chocolate was determined using the method of Young, modified by the replacement of the original graphical procedure with the numerical processing of the results. 19 samples i.e. 90% of the total, satisfied the requirements of Directive 2000/36/EC. In view that no official methods for CBE detection and quantification in chocolate have been published up to now and older methods were used in this work, the results published here should be considered as indicative and satisfying the requirements for screening only.

**Keywords:** chocolate; authenticity; cocoa butter equivalent; triglycerides

For its specific physical properties, cocoa butter (CB) is considered the most important chocolate component that determines its texture, melting behaviour, gloss, snap, etc. It also improves the process of dispersion of other chocolate ingredients. The unique properties of CB are due to the composition of its major triglyceride fractions, i.e. the 2-oleylglycerols of palmitic and stearic acids (POP, POS and SOS) that develop a stable  $\beta$ -polymorphic crystal structure in the course of chocolate processing.

In view of the fact that CB is the most expensive chocolate component whose price and availability often change, there have been long term efforts to replace it, fully or in part, with other vegetable fats,

so-called cocoa butter alternatives, which would be cheaper but would not influence the resulting sensoric properties of chocolate. However, it should be noted that, in certain cases, the addition of other vegetable fats might have favourable effects on the chocolate quality, e.g. its resistance to bloom, increased milk tolerance, improvement on the function of some CBs of lower quality, etc. (SHUKLA 1997). Nevertheless, the reasons for using CB alternatives are primarily economical.

Depending on their functional compatibility with CBs, vegetable fats added to chocolate are classified as follows (LIPP & ANKLAM 1998a):

- Cocoa butter equivalent (CBE): non-lauric plant fats, which are similar in their physical and

chemical properties to cocoa butter and miscible with it in any amount without altering the properties of cocoa butter.

- Cocoa butter replacer (CBR): non-lauric fats with the distribution of fatty acids similar to cocoa butter, but with a completely different structure of triglycerides; only in small proportions compatible with cocoa butter.

- Cocoa butter substitutes (CBS): lauric plant fats, chemically totally different from cocoa butter, with some physical similarities; suitable only for 100% substitution of cocoa butter.

Former Council Directive 73/241/EEC concerning cocoa and chocolate products did not contain any legislative requirements concerning the type and level of addition to chocolate of vegetable fats other than cocoa butter. In the EU Member States, various national regulations existed, permitting additions of 5–10% of other fats in the production of chocolate (in Austria, Denmark, Finland, Ireland, Portugal, Sweden and the United Kingdom). Different regulations caused problems in the trade in chocolate and chocolate products which led to the unification of legislation applicable to that alimentary commodity. That was why Directive 2000/36/EC (so-called Chocolate Directive) was issued, laying down the maximum permissible content of 5% CBEs in the final product and forbidding any addition of CBRs and CBSs. Its Annex II lists the raw materials that can be used for CBEs production. The list includes six vegetable fats plus their mixtures, i.e. illipe butter, palm oil, sal fat, shea butter, kokum gurgi and mango kernel fats. The said directive states that the addition of CBEs must be clearly indicated on the product label for the consumer not to be misled.

However, no methods are given or referred to for CBEs detection and quantification that would allow the monitoring of the product wrongful labelling and protect the consumer from fraud. The said Directive was implemented in the Czech legislation through the issue of Vyhláška 76/2003 Sb., fully compatible with the available EU legislation.

The problems of the detection and determination of foreign vegetable fats added to chocolate have been investigated for years. The overview of the analytical methods employed, including their critical evaluation, is given in reviews (LIPP & ANKLAM 1998b; ULBERTH & BUCHGRABER 2003). The last review cited was already worked out taking into due account the requirements of the EU Directive of 2000. From the review it follows that there

are numerous methods available for the purpose, based particularly on gas chromatography (GC) and high-performance liquid chromatography (HPLC). As to the analysed substances in question, special attention is paid to fatty acids (FAs), triglycerides (TGs), and minor fat constituents (sterols, sterol degradation products and terpens). Further, it is pointed out that, for the unequivocal identification of CBEs in CBs and their reliable quantification, specific combinations of various methods will most probably have to be used, followed by chemo-metrical evaluation of the complex data thus acquired.

Taking into account our instrumentation and previous experimental experience, we decided to employ the determination of TGs by means of gas chromatography for the estimation of the addition of CBEs.

In principle, either GC or HPLC technique may be used for determining the TG profile in cocoa butter or chocolate. The GC methods are, however, questioned in view of the assumed degradation of unsaturated components at elevated temperatures ( $> 300^{\circ}\text{C}$ ) employed (MAREŠ & HUŠEK 1985; MAYER & LORBEER 1997). Nevertheless, BUCHGRABER *et al.* (2000) demonstrated that both capillary GC with FID detection and HPLC using an evaporative light-scattering detector (ELSD) are equivalent methods for the determination of TG spectra in CB, particularly if the analytical conditions for GC are optimised (BUCHGRABER *et al.* 2004b). Moreover, CBs do not contain any higher proportions of polyunsaturated TGs that would be readily liable to degradation at the high temperatures involved.

Initially, the determination of the TG profile in CBs by high temperature GC was carried out in short columns (0.5 m) packed with a non-polar stationary phase (FINCKE 1980a; PADLEY & TIMMS 1980). Later, BARCAROLO and ANKLAM (2001) used a non-polar capillary column for this purpose. In both cases (non-polar columns), particular TGs fractions are separated according to their acyl-C-number, i.e. the total number of carbon atoms in the FA chains. Three major TG fractions, identified as C50, C52 and C54, were found in CBs and CBEs. Moreover, medium-polar capillary columns coated with phenyl-methyl silicone stationary phase are able to separate TGs on the basis of both their acyl-C number and the number of double bonds in the molecule (GEERAERT & SANDRA 1987).

However, the issue becomes more complex if the analytical data obtained are to be interpreted

and the decision based on the TG profile determined by the analysis, independent on CBEs having been added or not to the chocolate sample. An older calculation to demonstrate CBEs presence was proposed independently by FINCKE (1980b) and PADLEY and TIMMS (1980). The evidence is based on the fact, supported by the experimental results, that the contents of C50 and C54 found in genuine cocoa butter samples will show a linear relationship, after the contents of three major TGs fractions have been normalised ( $C50 + C52 + C54 = 100\%$ ). This can be expressed by the following equations:

$$\%C50 = 43.8 - 0.737 \times \%C54 \text{ (Padley \& Timms)}$$

$$\%C50 = 44.9 - 0.768 \times \%C54 \text{ (Fincke)}$$

Further, it was demonstrated that both the pure and commercially produced cocoa butter equivalents show lower C52 contents than genuine CBs and, therefore, in CBs/CBEs mixtures the CBEs will cause a deviation from the above given relationships. Taking the error of the determination into account, PADLEY and TIMMS (1980) proposed the following relationship for the qualitative detection of CBE presence in chocolate:

$$\%C50 < 44.095 - 0.737 \times \%C54$$

CHAVERON and VERDOIA (1984) carried out successful inter-laboratory testing of this relationship and confirmed its validity. From its interpretation it follows that the addition of CBE to chocolate is indicated if the content of C50 determined by the analysis is higher than that calculated according to the above equation. Illipe butter makes the only exception to the rule, as its TG profile is very similar to the TG profile of CBs.

The above given relationship may be used also for detecting CBEs addition to milk chocolates. The methodology employed is based on the subtraction of C50, C52 and C54 fraction quantities contained in milk fat from their respective total contents in chocolate.

The authors also proposed an equation for the quantitative determination of CBE additions with the provision that the composition of its TG fractions was known. However, the importance of this method for practical use is quite marginal because the above requirement cannot be met in absolute majority of cases as the manufacturers are not obliged to declare the name or composition of the CBE used.

PODLAHA *et al.* (1984) arrived at a very similar conclusion concerning the linear relationship between the contents of C50 and C54 ( $\%C50 = 43.9 - 0.73 \times \%C54$ ), but on the basis of TGs profiles in CBs determined by HPLC. SIMONEAU *et al.* (2000) published a little different equation: ( $\%C50 = 36.047 - 0.503 \times \%C54$ ) and explained the difference from other authors' results by the much larger set of CBs analysed. In addition, they stated that their method could not always distinguish genuine cacao butter from CB samples with CBEs content, particularly if illipe butter was involved.

YOUNG (1984) proposed a procedure for determining the addition of unspecified CBE to chocolate of unknown CB composition. This graphical method relies on the already proved linear relationship between the contents of C50 and C54 in genuine CBs (so-called CB line) and brings an additional finding that a CBE band may be plotted where the intersection points of the contents of C50 and C54, present in the prevailing majority of available CBEs, will fall in. The accuracy of the determination of  $\pm 1.5\%$  is achieved at the 5% level of CBE addition in chocolate. The method is designed particularly for routine laboratory control, as the CB line required for determining the content of an unknown CBE can be plotted using only three different samples of demonstrably genuine cocoa butter.

A similar procedure may be used for milk chocolates after subtracting the contents of major TGs fractions in milk fat from their total contents in milk chocolate samples. In the calculations, account is taken of the fact that milk fat (butter) contains, on average, the following percentages of particular triacylglycerol fractions: C50 = 12%, C52 = 13%, C54 = 8%. Nevertheless, this author recommended the correction of this factor according to actual experimental results.

Other procedure for distinguishing CBs from CB/CBE mixtures, based on the determination of TGs fractions (C50, C52, C54) using non-polar capillary columns of 5 m length, was proposed by BARCAROLO and ANKLAM (2001, 2002). By computing the C54/C50 ratio and  $(C54/C50 \times C52)$  product followed by 2-dimensional plotting of these values, identification was possible of various CBs model samples containing either 0.25–0.50% of pure CBEs or 5% of commercial mixtures of CBEs.

Other procedures employed for detecting and quantifying CBEs in CBs are aimed at the evaluation of the analytical results obtained by high-

temperature gas chromatography using capillary columns with a stationary phase of medium polarity. Primarily, the contents of major TG fractions (POP, POS, SOS, or even POO, SOO, PLS and SLS) are employed in the calculations based on various chemo-metrical methods (GEERAERT & SANDRA 1987; SIMONEAU *et al.* 1999, 2000). However, all these procedures have been applied to model mixtures only and are characterised by the use of sets comprising high numbers of CBE and CB samples. For instance, in the cluster analysis for systematic grouping of CBs and CBEs, 74 CB and 75 CBE samples were used (BUCHGRABER *et al.* 2004a).

The possibility of detection and quantification of CBE additions to chocolates was the object of the DG III-E-1 Project, co-funded by the European Commission (LIPP *et al.* 1999). Twelve analytical methods proposed for CBE detection were tested on the set of 41 CB samples, 21 CBE samples, and about 400 model mixtures prepared from these samples. The method of TG profile determination by high-resolution capillary gas chromatography (HR-GC) with the evaluation of the results by multiple linear regression analysis was found as the most appropriate. The procedure, described in the Technical Annex, allows to determine CBE content in chocolate with absolute uncertainty of  $\pm 2\%$ , i.e.  $\pm 2$  g/100 g chocolate (assuming the fat content in chocolate to be 20%). Nevertheless, unreliable results may be obtained in some cases, over- or underestimating the true CBE content, particularly in chocolates made of CBs of other than average composition, or if CBEs containing illipe butter have been added. The results may be improved by employing as large data sets as possible on the composition of various CBs and CBEs.

The aim of our work was to obtain basic information on the compliance with Vyhláška 76/2003 Sb. in the sale of chocolates, including milk chocolates, of domestic and foreign origins bought from retail stores in Prague. The contents of CBEs in the chocolates and the respective correct labelling of the additions of vegetable fats to cocoa butter were checked. Already at this point would we like to note that the data presented here should be considered as indicative only, due to the reasons specified under Results and Discussion below.

## MATERIALS AND METHODS

**Chemicals.** Petroleum ether, p.a. (ACS ISO, Merck); Hexane Suprasolvo (Merck); Methanol,

p.a. (ACS ISO, Merck); Sodium sulphate, anhydrous, p.a. (Lachema Brno); TG standards (Sigma): 1,2-dipalmityl-3-oleyl-glycerol (C50); 1,2-dioleoyl-3-palmityl-glycerol (C52); trioleine (C54); Fatty acid standards (Fluka): methyl myristate, methyl palmitate

**Apparatus.** Extractor 200 ml, Soxhlet type, by Supelco; Rotational vacuum evaporator; HP 5890 II gas chromatograph with HP 7673 auto-sampler, electronic pressure control and FID detection.

**Samples.** In the second half of 2003, within the retail store of Prague, eleven samples of chocolate were purchased of different origin (6× CR, 2× FRG, 1× each from Belgium, Slovakia and UK). In addition, during the first half of 2004 additional 11 milk chocolate samples were acquired in the same way (3× CR, 3× Slovakia, 2× Belgium, 1× each from FRG, Poland and Croatia). Three samples of genuine cocoa butter and 3 samples of CBEs were obtained from industrial sources.

### Determination of total fat in chocolates

**Chocolate.** The total fat content was determined by direct extraction in the Soxhlet apparatus.

**Milk chocolate.** The determination was made according to ČSN 560130, Part A, i.e. the sample was first hydrolysed by boiling in acid solution (HCl) for 15 min and then its total fat content was determined by the Soxhlet extraction method.

### Determination of milk fat

The analysis was based on the content of minor fatty acids (MINFA) after PONTILLON (1995). Minor fatty acids include acids eluted between myristic acid (C14) and palmitic acid (C16). These acids are present in milk fat but not in cocoa butter.

### Determination of fatty acids

Fatty acids contained in fat extracted from chocolate were first converted into methyl esters and then determined by capillary GC by the procedure proposed by BOHAČENKO and KOPICOVÁ (1999).

The content of milk fat was calculated using the following formula:

$$\%MF = (MINFA \times 100)/3.4$$

where: %MF – milk fat proportion in the total fat of the chocolate sample



MINFA – the sum of percentages of these acids in the sample, expressed by using their peak areas

3.4 – the factor determined by analyses made by Pontillon

The calculation is well applicable to fats that are not rich in lauric acid and fulfil the following relationship:

$$(C12 : MINFA) < 1.25$$

### Determination of TGs C50, C52 and C54 in chocolates

The determination was made by the method of PADLEY and TIMMS (1980) using a non-polar capillary column.

Fat (10–20 mg) extracted from chocolate (or milk chocolate) was dissolved in *n*-hexane to obtain the final concentration of 0.2–0.5 mg fat in 1 ml hexane. 1 µl of the solution was injected in the gas chromatograph.

**GC conditions.** Injection: on-column; injector temperature: track on (i.e. higher by 3°C then that of the column); Detection: FID,  $T = 360^{\circ}\text{C}$ ; Column: DB-5HT 30 m, i.d. = 0.25 mm, film thickness = 0.1 µm,  $T_{\text{max}} = 400^{\circ}\text{C}$ ; Temperature programme: initial temperature  $50^{\circ}\text{C}$  (1 min),  $20^{\circ}\text{C}/\text{min}$  up to  $320^{\circ}\text{C}$ , then  $4^{\circ}\text{C}/\text{min}$  to  $360^{\circ}\text{C}$ , total time: 20 min; Total time of analysis: 44.5 min.

Retention times of TGs C50, C52 and C54 were determined using their respective standards.

Repeatability of the method expressed as standard deviation (STD) was calculated from the determination of TGs, repeated eight times with two chocolate and one milk chocolate samples, with the following results:

At the level of C50: 20.4–23.5% STD = 0.20  
C52: 44.2–47.5% STD = 0.08  
C54: 31.7–32.9% STD = 0.25

### Identification of CBE addition to chocolates/milk chocolates

The qualitative determinations of CBE addition were based on the compliance with the relationship proposed by PADLEY and TIMMS (1980):

$$\%C50 < 44.095 - 0.737 \times \%C54$$

The chocolate/milk chocolate samples were considered as containing CBE additions, if the results

of TGs C50 and C54 determinations, normalised to 100% for chocolates, or re-normalised to 100% for milk chocolates (see below), did not fit the given relationship.

### Quantitative determination of CBE addition to chocolates/milk chocolates

The method of YOUNG (1984) was used modified by the combination of graphical and numerical calculations. The results were evaluated using EXCEL programme functions. Standard 2D coordinate system ( $x, y$ ) was used, where C54 and C50 values were plotted on the  $x$  and  $y$  axes, respectively, for the determination of the intersection points of the straight lines of concern.

Before the calculation itself, the contents of TGs (C50, C52 and C54) in genuine CBs and chocolates were normalised to 100%. For milk chocolates, the contents of the particular TG fractions in milk were subtracted from their corresponding total values (assuming the average percentages of the fractions in milk fat to be 12%, 13% and 8% of C50, C52 and C54, respectively /Young/), and the “net” values were re-normalised to 100% once more.

The following initial values were employed:

Three genuine CBs of the following (normalised) contents of TG fractions: [C50; C52; C54] = [27.54; 49.10; 23.36], [28.78; 48.69; 22.53] and [30.90; 48.0; 21.10], respectively.

The parallel “CB band” delimited by a pair of straight lines [the lower line intersecting the co-ordinate axes in points (0.70) and (80.0), the upper line in points (0.79) and (89.0)].

Partial CBE contents in chocolate fat (6 values in total, as three different genuine CBs samples were used) were calculated from the following relationship:

$$\%CBE = \frac{[C52(CB) - C52(x)] \times (100 - MF)}{C52(CB) - C52(CBEz)}$$

where:

CBE – partial CBE addition (%)  
C52(CB) – C52 contents in particular genuine CBs  
C52(x) – C52 content in the analysed sample  
MF – milk fat content (%) (for chocolates MF = 0)  
C52(CBEz) – C52 content delimited by the points of intersection of straight lines connecting, in turns, the points of genuine CBs and the analysed sample with the “lower” and “upper” lines

The partial contents of CBE were averaged and converted into the CBE proportions in final prod-

ucts taking account of the total fat contents in the chocolates.

The uncertainty of the determination for chocolate of 2.1% was obtained as the maximum difference between the true and the experimentally determined CBE contents in 18 model mixtures prepared from two CBs using the additions of 3%, 5% and 10% of each of three different CBEs.

The uncertainty of the determination for milk chocolate of 2.2% was obtained as the maximum difference between the true and experimentally determined CBE contents in 8 model mixtures prepared from one CBs using the additions of 10%, 15% and 20% of milk fat and 3%, 5% and 10% of CBE.

## RESULTS AND DISCUSSION

The analyses of chocolate samples bought from retail stores were made by the modified method of PADLEY and TIMMS (1980) based on the use of the non-polar capillary column instead of the packed column. The qualitative evidence of CBE presence in chocolate was based on the fit of the measured data with the relationship expressed as:  $\%C50 < 44.095 - 0.737 \times \%C54$ . The content of CBE was determined using the method proposed by YOUNG (1984), modified by replacing the original graphical evaluation of the results with the more precise numerical form.

The main reason why we used those older methods, which had been questioned in many respects, was the fact that only a limited number of unquestionably genuine CB samples and CBE samples of known provenience were available. We were not able to collect any large set of genuine CBs and various CBEs necessary for the application of new methods using HR-GC with multivariate data evaluation.

On the contrary, the method of Young allows the CBE addition to chocolates (including milk chocolates) to be calculated employing only a limited number of the input data. Moreover, the method was verified with the samples of real chocolates composed of unknown CBs and CBEs. This aspect is very important for controlling the observance of permitted CBE additions in practice, as the manufacturers need not declare both the basic CB type and CBE type added and have to inform only that the chocolate contains vegetable fat(s) added to cocoa butter.

As the newly developed methods are concerned, their results have been presented only at the level

of model experiments employing large numbers of CB/CBE mixtures prepared to simulate chocolate. We have not succeeded in finding any publication describing their practical application on a set of real chocolates. Further, the available literature does not deal with the identification and quantification of CBE addition to milk chocolates.

The CBE contents (in per cent of whole chocolate), as well as the selected analytical data required for their calculation after Young, are given for chocolates and milk chocolates in Tables 1 and 2. Taking account of the determination uncertainty of  $\pm 2.1\%$  for chocolate, resp.  $\pm 2.2\%$  for milk chocolate, the analytical findings of CBE presence as high as exceeding 7.1%, resp. 7.2% were considered the evidence of breaching the limit of 5% CBE, laid down by Directive 2000/36/EC.

For identifying the addition of CBEs, the tables were complemented with the values of C50 calculated using the formula of Padley and Timms. The addition of CBE to chocolate was demonstrated if the calculated value of  $C50 < \text{analytical value of } C50$ .

In the assessment of the chocolates tested using the above mentioned criteria it was stated that:

(a) CBE additions lower than 5% were determined in samples Nos. 1 to 5 (of CR provenience), using both the method of Young (the determined CBE contents were within the range of 2.27%–4.05%) and the relation of Padley and Timms (the calculated C50 values were always lower than the analytical ones). The addition of vegetable fat to cocoa butter was declared on the labelling of all the products.

(b) No CBE additions were detected by the method of Young in samples Nos. 6 to 9 (of Belgium, Germany and UK); the respective calculated values of C50 were almost the same or higher than the corresponding analytical findings. As to their labelling, no vegetable fat addition was declared on the products of Germany and UK while the Belgian products bore the labels reading without addition of any vegetable fats.

(c) In sample Nos. 11 and 12 (CR and Slovakia) labelled as chocolate for cooking, above-limit CBE additions (10.76% and 13.4%, respectively) were found which was in compliance with the results of calculation according to Padley and Timms. While the Czech sample was labelled as containing an addition of vegetable fats to cocoa butter, no such declaration was found on the Slovak product package.

Table 1. Selected analytical data and CBE additions in the chocolate samples

Country of origin	Czech Republic					Belgium	Germany		United Kingdom	Czech Republic	Slovakia
Sample No.	1	2	3	4	5	6	7	8	9	10	11
Indication of CBE addition [1]	A	A	A	A	A	C	B	B	B	A	B
Total fat (%)	29.44	28.85	27.15	28.20	28.90	42.66	28.45	25.78	26.93	29.30	29.72
TGc	Proportion of the area (%) after standardisation to 100%										
C50	25.07	21.57	26.62	22.78	21.44	20.98	21.01	22.65	21.50	31.83	32.22
C52	44.87	46.13	45.19	44.31	46.36	47.33	47.51	50.12	48.28	39.44	36.78
C54	30.06	32.30	28.19	32.10	32.20	31.69	31.48	27.22	30.22	28.73	31.00
CBE addition in whole chocolate (%)	4.05	2.27	3.76	4.20	2.28	0	0	0	0	10.76	13.40
C50 value calculated after Padley and Timms [2]	21.941	20.290	23.319	19.840	20.364	20.739	20.894	24.034	21.823	22.921	21.248

[1] Indication of CBE addition on the product label: A – contains vegetable fats in addition to cocoa butter; B – addition of vegetable fats to CB not declared, but found; C – no vegetable fats added to cocoa butter

[2] Calculated values of %C50 =  $44.095 - (0.737 \times \%C54)$  (C54 contents being determined by analysis)

From the similar assessment of milk chocolate samples it followed that:

(a) The addition of vegetable fats to cocoa butter was declared on the labels of samples Nos. 1 and 2 (CZ) and 10 (Croatia), even though the respective analytical CBE findings were within the limit of 5% (2.0–6.7% by analysis) and in compliance with the results of calculation after Padley and Timms.

(b) No CBE addition was declared in samples Nos. 3 (CZ), 11 (Belgium), 5 (Germany) and 6 to 8 (Slovakia). The analytical level of CBE in these samples was very low (0.3%–1.0%) and could not be considered as evidence for CBE having been added or not. Neither was it possible to make the decision on the basis of the relationship of Padley and Timms as the corresponding calculated and analytical values of C50 showed very small differences. We concluded that, most probably, no CBEs were added to the milk chocolates in question and that the differences found were due to the uncertainty and errors of the methods employed.

It was not possible to determine the content of CBEs in the Polish sample of milk chocolate. Even though the addition of vegetable fats to cocoa butter was declared there, it was obvious that neither CBS nor CBR were used.

On the basis of the above mentioned assessment, one can conclude that out of 22 chocolate samples (including 11 milk chocolates) tested for the content of CBEs and inspected for the mandatory labelling of vegetable fats addition to cocoa butter, 19 samples (90%) complied with the requirements of Directive 2000/36/EC. Two chocolate samples showed a non-declared CBE addition in excess of the permitted level of 5%, while the addition of other vegetable fats, obviously CBS or CBR, was found in one milk chocolate sample. It should be noted that all the samples taken were the products of renowned manufacturers, expected to observe the Directive.

In view of no official method for detecting and/or quantifying the CBE content in chocolates having been published, and due to the objective reasons mentioned above, we had to employ older methods for the given purpose. That is why the results presented in this paper should be considered as indicative and meeting the requirements of screening only. However, there remains unsolved the problem whether the newly developed methods, which will probably become obligatory in near future, can be employed in the laboratories (such as our facility) that do not possess the opportunity

Table 2. Selected analytical data and CBE additions in the milk chocolate samples

Country of origin	Czech Republic			Belgium	Germany	Slovakia			Poland	Croatia	Belgium
Sample No.	1	2	3	4	5	6	7	8	9	10	11
Indication of CBE addition [1]	A	A	B	B	B	B	B	B	A	A	B
Total fat (%)	32.52	37.25	33.33	29.73	32.48	27.52	31.86	28.81	30.48	34.10	30.60
Milk fat (%)	5.50	6.92	4.70	5.47	5.55	3.69	5.26	4.12	3.05	4.91	5.32
TGc	Proportion of the area (%) after re-standardisation to 100%										
C50	25.77	19.74	20.13	18.24	18.88	19.03	19.00	20.87	27.43	23.61	20.44
C52	40.72	45.63	46.30	46.05	47.98	46.95	47.03	47.47	54.95	43.03	47.08
C54	33.52	34.63	33.58	35.71	33.14	34.02	33.97	31.65	17.72	33.35	32.49
CBE addition in whole chocolate (%)	6.7	2.0	1.8	1.0	0.0	0.4	0.3	0.3	– [3]	5.1	0.5
C50 value calculated after Padley and Timms [2]	19.391	18.573	19.347	17.77	19.671	19.022	19.059	17.777	31.035	19.516	20.150

[1] Indication of CBE addition on the product label: A – contains vegetable fats in addition to cocoa butter; B – addition of vegetable fats to CB not declared, but found

[2] Calculated values of %C50 =  $44.095 - (0.737 \times \%C54)$  (C54 contents being determined by analysis)

[3] No determination of CBE addition was possible

or capacity to analyse large numbers of different CBs and CBEs and their mixtures. In this respect, it would be very helpful to create and maintain a central readily accessible database for gathering the related data in the way proposed by LIPP *et al.* (1999).

## References

- BARCAROLO R., ANKLAM E. (2001): Development of a Rapid Method for the Detection of Cocoa Butter Equivalents in Mixtures with Cocoa Butter. *Journal of the American Oil Chemists Society*, **54**: 1485–1489.
- BARCAROLO R., ANKLAM E. (2002): Investigation of a threshold for a limit of detection of individual vegetable fats in mixtures with cocoa butter. *Deutsche Lebensmittel-Rundschau*, **98**: 44–49.
- BOHAČENKO I., KOPICOVÁ Z. (1999): Detection of sunflower and soybean oil adulterated with rapeseed oil. *Czech Journal of Food Sciences*, **17**: 182–187.
- BUCHGRABER M., ULBERTH F., ANKLAM E. (2000): Comparison of HPLC and GLC techniques for the determination of the triglyceride profile of cocoa butter. *Journal of Agricultural and Food Chemistry*, **48**: 3359–3363.
- BUCHGRABER M., ULBERTH F., ANKLAM E. (2004a): Cluster analysis for the systematic grouping of genuine cocoa butter and cocoa butter equivalent samples based on triglyceride patterns. *Journal of Agricultural and Food Chemistry*, **52**: 3855–3860.
- BUCHGRABER M., ULBERTH F., ANKLAM E. (2004b): Interlaboratory evaluation of injection techniques for triglyceride analysis of cocoa butter by capillary gas chromatography. *Journal of Chromatography, A*, **1036**: 197–203.
- Council Directive 73/241/EEC of 24 July 1973 on the approximation of the laws of the Member States relating to cocoa and chocolate products intended for human consumption. *Official Journal L228*, 16/08/1973, 23–35.
- CHAVERON H., VERDOIA CH. (1984): Détermination des matières grasses végétales (M.G.V.) dans les chocolats. *Annales des Falsifications, de L'Expertise Chimique et Toxicologique*, **77**: 401–426.
- Vyhláška 76/2003/Sb., kterou se stanoví požadavky pro přírodní sladidla, med, cukrovinky, kakaový prášek a směsi kakaa s cukrem, čokoládu a čokoládové bonbony. Praha.
- Directive 2000/36/EC of the European Parliament and of the Council of 23 June 2000 relating to cocoa and



- chocolate products intended for human consumption. Official Journal L197, 03/08/2000: 19–25.
- FINCKE A. (1980a): Möglichkeiten und Grenzen einfacher gaschromatographischer Triglyceridanalysen zum Nachweis fremder Fette in Kakaobutter und Schokoladefetten. 1. Mitteilung: Verteilung der nach C-Zahlen klassifizierten Triglyceride in Kakaobutter. Deutsche Lebensmittel-Rundschau, **76**: 162–167.
- FINCKE A. (1980b): Möglichkeiten und Grenzen einfacher gaschromatographischer Triglyceridanalysen zum Nachweis fremder Fette in Kakaobutter und Schokoladefetten. 2. Mitteilung: Verteilung der nach C-Zahlen klassifizierten Triglyceride in Kakaobutter-Austauschfetten und anderen Fetten. Deutsche Lebensmittel-Rundschau, **76**: 187–192.
- GEERAERT E., SANDRA P. (1987): Capillary GC of triglycerides in fats and oils using a high temperature phenylmethylsilicone stationary phase. Part II. The analysis of chocolate fats. Journal of the American Oil Chemists Society, **64**: 100–105.
- LIPP M., ANKLAM E. (1998a): Review of cocoa butter and alternative fats for use in chocolate – Part A. Compositional data. Food Chemistry, **62**: 73–97.
- LIPP M., ANKLAM E. (1998b): Review of cocoa butter and alternative fats for use in chocolate – Part B. Analytical approaches for identification and determination. Food Chemistry, **62**: 99–108.
- LIPP M., SIMONEAU C., ULBERTH F., ANKLAM E. (1999): Possibilities and limits to cocoa butter equivalents in mixture with cocoa butter. EUR 18992 EN, European Communities.
- MAREŠ P., HUŠEK P. (1985): Quantitative capillary gas-liquid chromatography of triglycerides on fused-silica column with a chemically bonded stationary phase. Journal of Chromatography, **350**: 87–103.
- MAYER B.X., LORBEER E. (1997): Triacylglycerol mixture for testing capillary columns for high-temperature gas chromatography. Journal of Chromatography, **785**: 235–242.
- PADLEY F.B., TIMMS R.E. (1980): The determination of cocoa butter equivalents in chocolate. Journal of the American Oil Chemists Society, **57**: 286–293.
- PODLAHA O., TÖREGÅRD B., PÜSCHL B. (1984): TG-Type composition of 28 cocoa butters and correlation between some TG-type components. Lebensmittel Wissenschaft und Technologie, **17**: 77–81.
- PONTILLON J. (1995): Determination of milk fat in chocolates by gas-liquid chromatography of triglycerides and fatty acids. Journal of the American Oil Chemists Society, **72**: 861–866.
- SHUKLA V.K.S. (1997): Chocolate – the chemistry of pleasure. INFORM: International News on Fats, Oils and Related Materials, **8**: 152–162.
- SIMONEAU C., HANNAERT P., ANKLAM E. (1999): Detection and quantification of cocoa butter equivalents in chocolate model systems: analysis of triglyceride profiles by high resolution GC. Food Chemistry, **65**: 111–116.
- SIMONEAU C., LIPP M., ULBERTH F. (2000): Quantification of cocoa butter equivalents in mixture with cocoa butter by chromatographic methods and multivariate data evaluation. European Food Research and Technology, **211**: 147–152.
- ULBERTH F., BUCHGRABER M. (2003): Analytical platforms to assess the authenticity of cocoa butter. European Journal of Lipid Science and Technology, **105**: 32–42.
- YOUNG C.C. (1984): The interpretation of GLC triglyceride data for the determination of cocoa butter equivalents in chocolate: A new approach. Journal of the American Oil Chemists Society, **61**: 576–581.

Received for publication December 1, 2004

Accepted December 27, 2004

---

*Corresponding author:*

Ing. IVAN BOHAČENKO, CSc., Výzkumný ústav potravinářský Praha, Radiová 7, 102 31 Praha 10-Hostivař, Česká republika  
tel.: + 420 296 792 379, fax: + 420 272 701 983, e-mail: i.bohacenko@vupp.cz

---