Afdeling Koolhydraat/Vetchemie Mei 1985

RAPPORT 85.42 Pr.nr. 505.3090

Onderwerp: Methode-ontwikkeling voor het bepalen van het gehalte aan cacaoboterequivalente vetten in chocoladeprodukten.

Verzendlijst: direkteur, direktie VKA, sektorchef, afdeling KVC, medewerkers, projektbeheer, projektleider, bibliotheek (2x), circulatie, WVC dhr De Koe, VAAP dhr Van Slooten.

Afdeling Koolhydraat/Vetchemie Mei 1985

RAPPORT 85.42 Pr.nr. 505.3090

Projekt: Normalisatie en harmonisatie van onderzoekmethoden voor oliën en vetten, vette produkten en oliezaden.

Onderwerp: Methode-ontwikkeling voor het bepalen van het gehalte aan cacaoboter equivalente vetten in chocoladeprodukten.

Doel:
Vaststellen van een analysemethode voor bepaling van het gehalte aan cacaoboter-alternatieve vetten in chocoladeprodukten, vanwege een onderzoek in de EEG naar de bepalingsmogelijkheid van 5% CBA.

Samenvatting:
De Europese Commissie (EC) bestudeert de mogelijkheden om cacaoboter-alternatieve vetten (CBA) toe te staan in chocolade tot een gehalte van 5% van de waar zonder declaratieverplichting. Voorwaarde is de controleerbaarheid met een betrouwbare analysemethode. De CAOBISCO ontwikkelde een triglyceridemethode waarmee cacaoboter equivalente vetten kunnen worden bepaald. Dit verslag gaat in op de CAOBISCO-methode en de daarop gebaseerde EEG ringtest. Tot slot wordt het resultaat vermeld van onderzoek naar de samenstelling van Nederlandse chocolade geanalyseerd met deze CAOBISCO-methode.

Conclusie:
Analytisch levert de methode weinig problemen. Voor het bepalen van 5% CBE is de methode o.i. geschikt. Bepaalde CBE's die buiten de CBE band liggen kunnen met deze methode niet worden geanalyseerd. De standaardisatie van met name C 54 is een kritisch punt in de analyse.
De verwerking van de resultaten is momenteel mogelijk met een spectrum sinclair homecomputer met omnicale-2 spreadsheet software.
Als spreiding op het analyseresultaat is gevonden 3% absoluut + 15% relatief (op vetbasis) met als voornaamste oorzaak de natuurlijke variatie van CBE's.
Onze voorkeur gaat uit naar uitdrukking van het resultaat op vetbasis in plaats van op de totale waar.
In enkele merken Nederlandse chocoladerepen is 15-20% CBE aangetoond op vetbasis (4-6% op de waar).

Verantwoordelijk : drs B.G. Muuse
Medewerkers/samenstellers: H.J. van der Kamp, drs B.G. Muuse
Projektleider : drs B.G. Muuse

Inleiding

Cacaoboter (CB) onderscheidt zich van andere vetten door haar specifieke fysische eigenschappen. In chemisch opzicht is alleen de triglyceride-compositie specifiek voor de soort.

Cacaoboter behoort tot de duurste oliën en vetten, in 1984 ca. £ 2 per kg zie bijlage I, reden waarom reeds lang gezocht wordt naar cacaoboteralternatieven. De vereisten voor deze zgn. CBA's zijn:

1. Een steile dilatatiecurve ofwel een klein temperatuurverschil tussen zacht worden en smelten.
2. Mogen niet onder 30°C en niet boven 37°C smelten.
3. Met cacaoboter gemengd mag er geen smeltpuntverlaging optreden.

Met name dit laatste vereiste is moeilijk te realiseren en reden waarom de CBA's zijn onderverdeeld in cacaoboter equivalente vetten (CBE) en lauric en non-lauric cacaoboter replacers (CBR) (1,10).

De CBE's hebben zowel chemisch als fysisch dezelfde eigenschappen als CB's en zijn in elke verhouding mengbaar zonder fysische verandering. CBE's zijn vrijwel altijd plantaardige vetten. De CBR's daarentegen wijken qua vetzuursamenstelling af van CB. De laurinezuur typen wijken fysisch niet af van CB, de laurinezuurvrije typen wijken zowel chemisch als fysisch af van CB. Voor de CBR's geldt dat ze niet in alle verhoudingen mengbaar zijn met CB zonder verandering van de typische fysische eigenschappen van CB, zodat voor CBR's een beperkt specifiek toepassingsgebied bestaat.

Onderverdeling van CBA vetten

Fincke (6) geeft een overzicht van CBE's en gaat daarbij uit van de fysische gelijkenis met CB. Hij noemt daarin zeven groepen met vnl. gefraktioneerde vetten zoals palmolie-mid-fraktie, sheastearine en salstearine uit India, de illipe groep en bepaalde mengsels van genoemde vetten.

Op sheastearine lijkende vetten komen ook voor in Afrika zoals kokumbutter en allanblackia vet. Tot de illipe groep wordt ook gerekend tenkawang (borneotalg). De illipegroep omvat de zeer sterk op CB lijkende vetten die vrijwel niet van CB zijn te onderscheiden. Dit geldt ook voor Procter en Gamble CBE, een synthetisch bereid triglyceride mengsel (vermoedelijk dierlijk vet). De CB replacers van het laurinezuur type zijn afkomstig van geharde en gefraktioneerde cocos of palmpitvetten.

De non-lauric CBR's zijn afkomstig van geharde palm-, arachide- of katoenzaadolie.

Standpunten van EEG lidstaten t.a.v. toelating van CBA in chocolade

In ons land is het gebruik van CBA's gekoppeld aan de benaming chocolade niet toegestaan (PBO verordening 1974 E3A).
In Engeland is sinds 1977 CBA toegestaan tot 5% van de waar.
Gedurende een aantal jaren is in de codex alimentarius en de EEG een discussie gaande om 5% CBA toe te staan, indien overschrijding daarvan kwantitatief is vast te stellen en het vet aan bepaalde specifieke eisen voldoet.
Denemarken, Ierland en Engeland ondersteunen dit streven. Duitsland en Frankrijk lieten een duidelijk neen horen terwijl de overige lidstaten alternatieve vetten wel willen toelaten doch onder de nodige voorwaarden, zoals deklaratie van de aanwezigheid van CBA (hetgeen ook analyse van 0-5% nodig maakt!). De voornaamste argumenten tegen toelating zijn handhaving van de produktidentiteit en benadeling van de oorsprong landen in hun afzet m.n. ivoorkust en Brazilie zie bijlage I. Daarbij dient te worden bedacht dat de 5% regeling overeenkomt met ongeveer 20% minder CB verbruik.
De CAOBISCO (Vereniging van de chocolade-biscuit-, beschuit- en suikerwerkindustriën in de EEG) heeft inmiddels een analysemethodiek ontwikkeld waarmee het mogelijk zou moeten zijn om CBE in chocoladeprodukten vast te stellen. De ontwikkeling hiervan is door Fincke (4,5,6,7,8) gepubliceerd. Resultaten met deze methodiek worden ook beschreven door Padley en Timms (9) en Young (11).
Deze methodiek heeft de EEG in studie genomen door onlangs een ringtest te organiseren, waarbij in ons land om deelname verzocht is aan CIVO, De Keuringsdienst van Waren te Nijmegen en RIKILT.

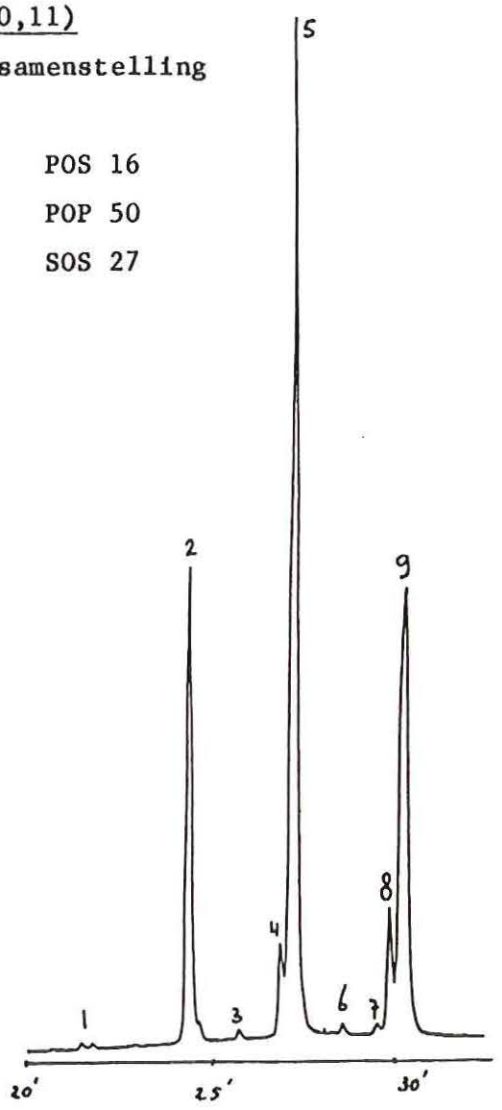
Analysemethoden

De CAOBISCO heeft de volgende kriteria opgesteld voor CBE grondstoffen ter onderscheiding van CBR's zie ook bijlage II:
Het vet dient plantaardig te zijn,
symetrische triglyceridegehalte $\geq$ 65%,
onverzadigde vetzuren totaal $\leq$ 45%,
onverzadigde vetzuren in 2-positie $\geq$ 85%,
meervoudig onverzadigde vetzuren $\leq$ 5%,
trans vetzuren $\leq$ 2%,
laurinezuur $\leq$ 1%.

Hiervoor zijn de volgende analysemethoden nodig:
De analyse van vetzuren waarvoor goed gestandaardiseerde internationaal aanvaarde methoden bestaan,
de vetzuur 2-positie-analyse,
de sterolanalyse en de transvetzuuranalyse.
Voor de vaststelling van het CBE-gehalte in cacaoboter, naast eventueel melkvet, is de op triglyceride-analyse gebaseerde methode van de CAOBISCO nodig.
Voor de overige analysemethoden voor chocolade zoals vet, as, vocht, onverzeepbaar, en zuurtegraad hebben Judd et al. (2) methoden gepubliceerd die acceptabel zijn, uitgezonderd de aan kritiek onderhevige Philips en Sanders methode voor de bepaling van het melkvetgehalte.

Samenstelling van cacaoboter (3,5,6,10,11)

Vetzuursamenstelling		Triglyceridesamenstelling		
C16:0	25-26	C48	0,3	
C18:0	34-36	C50	18,0	POS 16
C18:1	34-35	C52	46,0	POP 50
C18:2	2-3	C54	33,5	SOS 27
		C56	1,2	



Principe van de CBE-bepaling middels triglyceride-analyse

Met de triglyceride (TG) analyse wordt de samenstelling van C40-C56 bepaald. Vervolgens worden de voor melkvet triglyceriden gecorrigeerde en tot een totaal som van 100% herleide waarden van C50, C52 en C54 berekend met behulp van korrektiefaktoren die uit standaardreeksen van CB's, CBE's en melkvet moeten worden vastgesteld. Voor melkvet wordt C50 t/m C54 gesteld op 28,0%, dit komt overeen met onze bevindingen (12). Voor hard gefraktioneerd melkvet vinden wij echter 29,1.
Met de herleide waarden van C50 en C54 wordt het CBE tracee vastgesteld wat aangeeft met welk type CBE de cacaoboter werd gemengd. Voor de 6 waarden A t/m F die dit tracee omvatten (zie bijlage II fig. 3) wordt vervolgens de C52 waarde berekend. Met deze zes C52 waarden, het C52 gehalte van het monster en de C52 waarden van 3 vaste waarden op de cacaoboterlijn wordt de mengverhouding van CB en CBE bepaald en de middelwaarde als eindresultaat berekend, weergevende het percentage CBE in het totale vet. Voor de totale methode zie bijlage II.
Vanwege de bijzonder complexe en arbeidsintensieve rekenkundige bewerkingen die voor de vaststelling van het CBE gehalte nodig zijn, is gecomputeriseerde verwerking onontbeerlijk. Als software is een spreadsheet programma nodig dat voor de meeste homecomputers verkrijgbaar is doch vreemd genoeg niet voor grote computersystemen.
Voor grafische interpretatie van de resultaten zie de figuren achterin bijlage II.

Analyseresultaten

De ringtestmonsters van de EEG bestonden uit 6 diverse cacaoboters, 6 mengsels van een cacaoboter met diverse hoeveelheden melkvet en 18 mengmonsters van CB, CBE en melkvet waarvan in 6 gevallen de samenstelling bekend is gemaakt.
Analyse van de 6 CB's gaf een lineair verband tussen de monsters (cacaoboterlijn) volgens de vergelijking: $C50 = -0{,}776 \cdot C54 + 45{,}2$.
Analyse van de 6 mengsels van CB met melkvet gaf een melkvetfaktor FMF van 4,38. Hiermee is het percentage melkvet in het vet te berekenen uit de waarde van C40 t/m C44.
Ook korrektiefaktoren voor C50 t/m C54 per procent melkvet zijn uit de 6 mengsels met melkvet berekend. De methode geeft waarden voor C50-C54 van respektievelijk 0,12, 0,13 en 0,08. Gevonden werden waarden van respektievelijk 0,11, 0,09 en 0,08.
Voor een overzicht van de analysecijfers zie bijlage III.

Vergelijking van de melkvetbepaling via de triglyceride-analyse en via de vetzuuranalyse geeft marginale verschillen te zien (5% relatief). Met name de basis kengetallen C40 - C44 = 28 respektievelijk C4 = 4,0 zijn bepalend voor het melkvetgehalte en een eventuele systematisch fout daarin.
Tabel 1 geeft een overzicht van de resultaten die gevonden zijn met beide methoden. Een aparte vetzuuranalyse voor de bepaling van melkvet is o.i. niet nodig.

De EEG ringtest gaf niet aan met welke cacaoboter de zes monsters voor vaststelling van de melkvetkorrekties is bereid. Ook is niet tevoren aangegeven welke uitvoeringsprocedure gevolgd moest worden en hoe de resultaten opgegeven moeten worden. Enkele storende fouten in het rekenmodel van de methode maakten de ringtestopzet tenslotte niet ideaal.

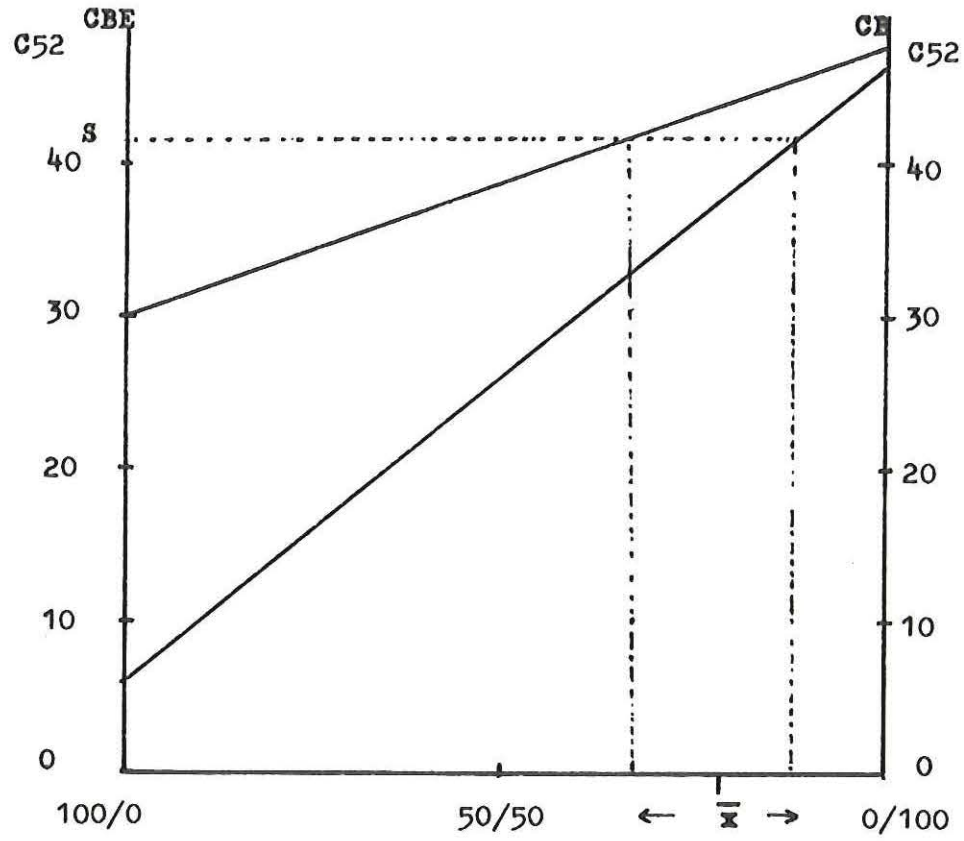
Tabel 1. Melkvetgehalte bepaald via de triglyceride-analyse en via de vetzuuranalyse in de EEG-ringtestmonsters.

Monsters	% melkvet via		Verschil
	Triglyceriden	Vetzuren	%
G	17,7	19,8	2,1
H	12,3	13,5	1,2
I	24,0	26,8	2,8
J	10,2	10,5	0,3
K	20,3	23,0	2,7
L	15,0	16,3	1,3
1	0	0	0
2	14,6	16,0	1,4
3	0	0	0
4	0	1,0	1,0
5	0	0	0
6	14,8	16,8	2,0
7	16,0	17,0	1,0
8	15,4	15,5	0,1
9	16,1	19,0	2,9
10	15,6	15,8	0,2
11	15,9	16,2	0,3
12	15,9	16,8	0,9
13	0	0	0
14	0	0	0
15	15,3	14,09	-0,7
16	14,3	14,8	0,5
17	15,3	12,3	-3,0
18	15,6	15,2	-0,4

Uit heranalysen is gebleken dat met name het gehalte aan hoogmoleculaire triglyceriden C54 en C56 meer dan de andere triglyceriden afhankelijk zijn van de gaschromatografische omstandigheden. Er worden eerder te lage waarden gebonden dan te hoge en daardoor een CB-lijn die niet voldoet aan de kriteria in de methode.

Het eindresultaat van de methode berust op het verschil in C52 (na alle korrekties en normalisaties) tussen zuivere CB en het CBE gebied dat gemarkeerd wordt door de punten A t/m F. Figuur 1 illustreert deze korrelatie. Tevens blijkt hieruit welke mate van spreiding is te verwachten. Deze spreiding is lineair afhankelijk van het CBE gehalte in het vet en komt in de praktijk overeen met 3% absoluut + 15% van het CBE gehalte in het vet.

Figuur 1: Massafraktie diagram voor C52 van CB en CBE waaruit het percentage CBE kan worden vastgesteld en waarmee de resultaatspreiding wordt geillustreerd.



Voor het grensgebied van 20% CBE in chocoladevet (5% op de waar) is een spreiding om de gemiddelde waarde te verwachten van 3 + 3 = 6% absoluut.

Deze spreiding wordt veroorzaakt doordat in het monster de soort CB en de soort CBE niet bekend worden verondersteld en kunnen varieren binnen vastgestelde grenzen. Deze grenzen worden bepaald door CB1-CB3 en de CBE gebiedsmarkeringen A t/m F (zie figuur 3 in bijlage II). Uit de ringtestresultaten zal moeten blijken welke bijdrage de triglyceride-analyse levert aan de spreiding. Verwacht wordt dat de geschetste natuurlijke variatie vele malen groter is dan de analysespreiding.
Daar de spreiding niet van analytische aard is, is o.i. sprake van overschrijding van de 20% grens (= 5% op de chocoladewaar) indien de gemiddeld gevonden waarde deze grens overschrijdt.
Toepassing van de voorgestelde methode op chocoladerepen van de Nederlandse markt gaf voor 4 van de 14 merken (6 van 24 monsters) aanwezigheid van 15-20% cacao- en melkvreemd vet waarvan we aannemen dat het CBE vetten zijn. Op produktbasis komt dit overeen met 4,5-6%.
Het type CBE is in alle gevallen identiek en duidt in het algemeen op een mengsel van palmolie mid faktie met sheastearine en enig vet uit de illipe groep. Bijlage IV geeft een identifikatie grafiek van CBE soorten (11). In nog eens 2 gevallen (van verschillend merk) bestaat twijfel over bijmenging met CBE. Voor grafische weergave van de resultaten zie bijlage V.

Conclusies

Het EEG proposal op basis van de caobisco methode biedt goede mogelijkheden om meer dan 2-3% CBE in chocolade vast te stellen. CBE's die dicht bij de CB lijn liggen zijn met deze methode niet te bepalen (m.n. Procter en Gamble en Illipé vetten). De methode is rekenkundig dermate complex en bewerkelijk dat routine onderzoek niet redelijk uitvoerbaar is zonder computerverwerking met spreadsheet software.
De CBE kriteria van de caobisco zijn niet controleerbaar in mengsels.
De spreiding van de analyse wordt veroorzaakt door natuurlijke variatie en bedraagt + en - 3% absoluut + 15% relatief op basis van vet! Op produktbasis komt dit overeen met 0,75% ABS + 15% relatief.
Indien 5% CBE op basis van produkt wordt toegestaan dan dient o.i. de gemiddeld gevonden waarde voor CBE maatgevend te zijn.
Als controle op de juistheid van de analyse is een vereiste dat voldaan is aan de kriteria waarbinnen de cacaoboterlijn moet liggen.

Het is o.i. eenduidiger om het CBE percentage te baseren op het vet of het chocoladedeel van de waar en niet op de totale waar zoals in de ontwerp verordening. 5% toestaan op de chocolade-waar betekent dat voor chocolade 28%; voor melkchocolade 23%; voor volle melkchocolade 24% en voor chocolade puur 19% mag worden vervangen door CBE.
Het percentage melkvet is vast te stellen met de triglyceride-analyse. Er zijn slecht marginale verschillen gevonden t.o.v. de bepaling via de vetzuursamenstelling.
De melkvetwaarde van 28% voor de som van C40 t/m C44 is ook voor normaal Nederlands melkvet gevonden. Voor gefraktioneerd hard botervet is deze waarde 29%.
Geen onderzoek is gedaan naar de invloed van migratievetten uit de niet-chocoladewaar op de analysespreiding!
Daar gehalten onder 2%-3% CBE in de waar niet nauwkeurig te bepalen zijn, lijkt een deklaratieverplichting tot 3% inhoudsloos.

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Bijlagen

I Wereld produktie en consumptie van cacaobonen.
II Caobisco methode voor de bepaling van CBE in chocolade.
III Analyseresultaten van de EEG ringtestmonsters.
IV C50-C54 diagram met CB lijn en CBE band volgens Young (11).
V Grafische weergave van de resultaten van Nederlandse chocoladerepen.

WORLD COCOA PRODUCTION AND GRINDINGS DATA
Reproduced from Gill and Duffus Market Report dated May 1984
(Thousand Metric Tonnes)

PRODUCTION	1980/1	1981/2	1982/3 (Estimate)	1983/4 (Forecast)
Africa	999	1043	856	854
America	528	520	515	480
West Indies	44	50	52	51
Asia and Oceania	92	113	116	135
WORLD TOTAL	1663	1726	1539	1520
Principal Countries				
Ghana	258	225	178	158
Nigeria	155	181	156	115
Ivory Coast	403	457	355	400
Cameroun	120	122	106	108
Equatorial Guinea	8	8	10	10
Brazil	349	314	336	299
Ecuador	81	85	55	60
Dominican Republic	32	40	40	39
Venezuela	14	17	18	18
Colombia	39	42	40	41
Mexico	30	41	42	40
Papua New Guinea	28	30	28	29
Malaysia	43	60	65	80
GRINDINGS				
Western Europe	598	620	626	623
Eastern Europe and USSR	211	201	228	214
North America	207	215	210	210
Central and South America	349	322	348	341
Australia and New Zealand	16	13	12	12
Asia	90	106	107	105
Africa	125	126	120	125
WORLD TOTAL	1596	1603	1651	1630
Principal Countries				
United States	190	199	194	194
Germany (Federal Republic)	167	175	180	182
Netherlands	141	148	157	158
United Kingdom	85	88	77	80
USSR	120	120	137	130
France	52	52	53	50

PRICES

Month-end prices for nearby shipment Ghana cocoa have been:

		£			£
1982	July	976/tonne	1983	July	1691/tonne
	August	1008/tonne		August	1590/tonne
	September	1080/tonne		September	1567/tonne
	October	1019/tonne		October	1502/tonne
	November	1052/tonne		November	1710/tonne
	December	1171/tonne		December	2097/tonne
1983	January	1348/tonne	1984	January	1965/tonne
	February	1301/tonne		February	1713/tonne
	March	1341/tonne		March	1889/tonne
	April	1316/tonne		April	1998/tonne
	May	1521/tonne		May	2215/tonne
	June	1639/tonne		June	1910/tonne

The market has risen fairly steadily in the light of two successive deficits in the supply/demand situation. Gill and Duffus are not yet making any forecasts for 1984/5.

CAOBISCO

ASSOCIATION DES INDUSTRIES DE LA CHOCOLATERIE, BISCUITERIE-BISCOTTERIE ET CONFISERIE DE LA CEE • ASSOCIATION OF THE CHOCOLATE-, BISCUIT- AND CONFECTIONERY INDUSTRIES OF THE EEC • VERENIGING VAN DE CHOCOLADE-, BISCUIT-, BESCHUIT- EN SUIKERWERKINDUSTRIEËN IN DE EEG • BRANCHEFORENINGEN FOR CHOKOLADE-, BISCUIT- OG KONFEKTURE INDUSTRIEN I EF • ASSOCIAZIONE DELLE INDUSTRIE DEL CIOCCOLATO, DELLA BISCOTTERIA-PASTICCERIA E DELLA CONFETTERIA DELLA CEE • VERBAND DER SCHOKOLADE-, DAUERBACKWAREN- UND ZUCKERWARENINDUSTRIEN DER EWG • ΣΥΝΔΕΣΜΟΣ ΒΙΟΜΗΧΑΝΙΩΝ ΣΟΚΟΛΑΤΟΠΟΙΙΑΣ, ΜΠΙΣΚΟΤΟΠΟΙΙΑΣ - ΦΡΥΤΑΝΟΠΟΙΙΑΣ ΚΑΙ ΖΑΧΑΡΩΔΩΝ ΠΡΟΪΟΝΤΩΝ ΤΗΣ Ε.Ο.Κ.

7301/2621 - WC/CC
Septembre 1984

TRANSLATION (orig : D)

Method for determining cocoa butter equivalents and certain other fats in cocoa butter and chocolate fats

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BIJLAGE II

1. Purpose

The method describes a procedure for determining the amount of certain non-cocoa-butter fats (NCBF), in particular cocoa butter equivalents (CBEs), in cocoa butter and chocolate and milk chocolate fats. The method is based on the gas chromatographic separation and determination of the triglycerides by number of C atoms.

See note 1. 01, 23

2. Definitions

2.1. Cocoa butter: Fats produced from cocoa raw materials that meet the standards of the EC Cocoa Directive for "Cocoa butter".

2.2. Milk fat: Fat obtained from milk and having its natural composition.

2.3. Specified non-cocoa butter fats (NCBF): Fats whose triglyceride data are in accordance with the criteria given in "3. Scope of application".

2.4. Cocoa butter equivalents: Fats whose composition meets the following criteria, suggested by CAOBISCO:

(a) Vegetable fats

(b) Content of β-SOS triglycerides ≥ 65% by weight.

(c) Content of unsaturated fatty acids in the 2-position of the triglycerides ≥ 85% by weight.

(d) Content of unsaturated fatty acids in the total fatty acids ≤ 45% by weight.

(e) Content of unsaturated fatty acids with 2 or more double bonds in the total fatty acids ≤ 5% by weight.

(f) Content of lauric acid in total fatty acids ≤ 1% by weight.

(g) Content of trans fatty acids in total fatty acids ≤ 2% by weight.

3. Scope of application

3.1. The method can be applied only to mixtures of cocoa butter, milkfat and those non cocoa butter fats, especially CBE, whose % C5o and % C54 contents (normalized to % C5o + % C52 + % C54 = 100,0 %) lie within a defined range (the CBE band ; see Figure 1 and sections 14.3 and 14.4).

3.2. It follows from the definition of the CBE band (see 14.3. and ~~14.4.~~) that the method can give useful results only when the normalized triglyceride composition (% C5o + % C52 + % C54 = 1oo,o%) of a non-cocoa butter fat satisfies the following inequality:

$$1oo,o - \%\ C54 - (84.o - o,894 \cdot \%\ C54) \leq \%\ C52 \leq 1oo,o - \%\ C54 - (7o - o,875 \cdot \%\ C54)$$

3.3 The definition in (2.2.) excludes fractionated and hardened milk fats as these lead to analytical complications that cannot be assessed at present.

3.4 Compliance with the criteria of (2.4.) can be checked by means of the following analytical methods:

a) Determination of sterol content: ISO 6799-1983 (E); IOCC No. 16-E/1973

b) Content of β-SOS triglycerides: IOCC (in preparation); M.S.J. Dallas and F.E. Padley, Lebensm. Wiss. u. -Technol. 10 (1977) 328

c) [illegible], M. Kaemmerer, F.E. Padley, M.S.J. Dallas, [illegible] B.A. Jorgensen, [illegible] Fett. Ind. [illegible] 74 (1981) 488

d) Unsaturated fatty acids in the 2-position: IOCC (in preparation); ISO Draft Proposal 6800 ISO/TC 34/SC 11; IUPAC Standard Methods for the Analysis of Oils, Fats and Derivatives No. 2.210.

e),f), g) Fatty acid composition: IOCC 17a-E/1973; IOCC 17b-E/1973; ISO 5500-1978 (E); ISO 5508-1978 (E).

h) Trans-fatty acids: IOCC (in preparation).

3.5 At present there is only one commercially available CBE that meets the CAOBISCO definition (2.4.) but does not have a triglyceride composition in accordance with the inequality of (3.2.) for the normalized triglyceride values for % C50, % C52 and % C54. The triglyceride data for this fat does not lie within the CBE band (14.3.1.). This method gives a CBE value too low for cocoa butter or chocolate fats containing such fats. This applies to fats of the "illipe group" and cocoa butter equivalents containing a high level of these fats, as well as to cocoa butter equivalents that may be produced in future by enzyme-controlled interesterification (J. Am. Oil Chem. Soc., 59 (1982) 295 A).

At present, however, the commercially available cocoa butter equivalents meet both the CAOBISCO criteria and the inequality of (3.2.).

3.6 Some non-cocoa butter fats (NCBF) satisfy the inequality quoted in (3.2.) although they do not meet the CAOBISCO criteria and are not suitable as cocoa butter equivalents. These fats include some vegetable oils (e.g. hazelnut oil, soyabean oil) that may be present in chocolate fats in consequence of the addition of hazelnuts and soya lecithin (14.3.).

As these fats can simulate the presence of cocoa butter equivalents, their presence or absence generally has to be established by additional analyses.

The following techniques may be of particular service for this purpose

a) Determination of the fatty acid methyl ester composition: see Note 3.2. e)-g).

b) Isolation and analysis of the more highly unsaturated triglycerides on silver nitrate-silica gel TLC plates: C. Litchfield: Analysis of Triglycerides, Academic Press, New York 1972, p50-67; D. Chobanov et al: J. Am. Oil Chem. Soc. 53 (1976) 48.

c) Determination of the more highly unsaturated triglycerides (e.g. triolein, di-oleolinolein) by HPLC: H. Rehfeldt, E. Schulte and L. Acker, in preparation.

d) Isolation of isomerized unsaturated fatty acid methyl esters on silver nitrate-silica gel TLC plates followed by gas chromatographic analysis see Note 2.2.h).

e) Detection and determination of isomerized unsaturated fatty acids by gas chromatography of the fatty acid methyl esters on capillary columns (≥ 25m): R.P. D'Alonzo et al: J. Am. Oil Chem. Soc. 58 (1981) 223; H.T. Slover and E. Lanza, J. Am. Oil Chem. Soc. 56 (1979) 933.

f) Detection of triglycerides with isomerized unsaturated fatty acids on silver nitrate-silica gel TLC plates: see Note 2.2.b).

g) Isolation and analysis of the unsaponifiables (Composition of the sterols, detection of characteristic constituents, e.g. butyrospermol): IOCC No. 14-E/197o; A. Fincke: Dtsch. Lebensm. Rdsch. 71 (1975) 284; M. Derbesy and M.T. Richert: Oleagineux 34 (1979) 4o5.

h) Immunochemical or electrophoretic detection of proteins that can derive from fat-containing substances added to chocolates, e.g. hazelnut, groundnut or soya protein: Official Collection of Analytical Methods in accordance with § 35 LMBG (in preparation).

4. Principle of the method

4.1. Set up analytical procedure for triglyceride analyses by GLC.

4.2. Analyse standard cocoa butters and define the "cocoa butter line". Check that this agrees with the standard "cocoa butter line".

4.3. Separate chocolate fats and determine the triglycerid composition (classification by carbon numbers).

4.4. Perform additional analytical procedures (i.e. determination of fatty acid methylesters if necessary).

4.5. Correct C5o-, C52- and C54-peaks if milk fat or other fats like or soyaoil are present.

4.6. Plot analytical datas on the cocoa butter - CBE - graph and determine the amount of CBE (and certain other fats) present.

4.7. Apply additional corrections to 4.5. (i.e. due to hazelnutoil) and repeat 4.6.

5. Reagents

(a) Chloroform, analytical grade.

(b) Reagents for determining the fat content of chocolate; IOCC No. 8a-E/1972.

(c) Reagents for determining the C4 methyl ester content of fats containing milk fat; ISO 55o9; ISO 55o8.

(d) At least 6 different cocoa butter samples of different triglyceride composition (see 9).

(e) Samples of different CBEs (see 1o).

(f) Helium or nitrogen (carrier gas).

(g) Hydrogen and purified air.

6. Apparatus and equipment

(a) Gas chromatograph (with FID; programmable oven heating and heated injector).

(b) Suitable separating columns:

either packed columns (glass; length o.5- 1 m; diameter 2 - 4 mm; 1.5 - 3% OV-1 or OV-1o1 or SE-3o on Gaschrom Q 1oo/12o or Chromosorb W 8o/1oo or Chromosorb WHP 1oo/12o or Chromosorb G/AW/DMES 8o/1oo);
or glass capillary columns (length 5-6 m; diameter o.3 mm, OV-1o1 or OV-1).

(c) Recorder and integrator.

(d) Oven

(e) µl syringes.

(f) Normal laboratory glassware.

7. Preparation of the sample

7.1. Cocoa butter and other fats

7.1.1. Cocoa butter and other fats are completely melted, well mixed and filtered in the dark (see IOCC No. 8e-E/1973) before gas chromatographic analysis.

7.1.2. The amount of sample required for gas chromatographic analysis is transferred in the clear melted state (6o° C) into a suitable container (measuring flask, Erlenmeyer flask; 1 drop of melted cocoa butter~15 mg), if necessary weighed after cooling and dissolved in the required amount of chloroform (see 8.2. and 8.3.).

7.2. Chocolate

7.2.1. The fat content of the chocolate is determined by IOCC Method 8a-E/1972.

7.2.2. The chocolate fat for chromatographic triglyceride analysis is obtained from the chocolate sample in accordance with IOCC 8a-E/1972. The method used here differs from the IOCC method in that the petroleum ether extract is evaporated down under vacuum with a rotary evaporator (water bath temperature 6o° C) and the solvent-free residue is dried (water bath temperature 6o° C, about 2o minutes).

7.2.3. The chocolate fat should be stored in the refrigerator, in the dark.

8. Procedure for triglyceride analysis by gas chromatography

8.1. Preliminary remarks

It is not sensible to prescribe obligatory fixed analytical conditions because of the different types of gas chromatographs and separating columns used. Every analyst must determine the optimal working procedure for his particular combination of equipment himself by analysis of suitable test samples (see 8.4., 9 and 1o). The analytical conditions given in 8.2. and 8.3. should therefore be regarded as providing only general guidance.

In every case, the analytical conditions must be chosen to give as complete separation and recording as possible of triglycerides in the range C4o-C56.

8.2. Use of packed columns

Separating columns: See 6 (b)

Temperatures: Injection block: 35o - 38o° C
Separating column: Temperature programme starting at 21o-28o° C
Final temperature: 3oo-35o° C
Temperature rise 2-6° C/min
Detector: 35o-39o° C

Carrier gas: Helium or nitrogen, 2o-1oo ml/min

Amount injected: ~o.5 µl of a 3% solution of the fat in chloroform.

8.3. Use of glass capillary colu..

Separating columns:	See 6 (b)
Temperatures:	Injection block: 36o-38o° C
	Separating column: Temperature programme starting at 21o-29o° C Final Temperature: 33o-36o° C Temperature rise: 2-6° C/min
	Detector ~ 38o° C
Carrier gas:	Helium or nitrogen, pressure o.3-1.2 bar
Amount injected:	~1 µl of a 2% solution of the fat in chloroform

8.4. Assessment of the resolution of neighbouring peaks

8.4.1. The resolution, i.e. the efficiency of separation, of the triglyceride peaks C5o and C52 is assessed by calculating the resolution (see Figure 2):

$$\text{Resolution} = \frac{2\,\Delta}{W_1+W_2}$$

where Δ is the distance in mm between the C5o and C52 peak maxima; and W_1, W_2 are the widths in mm of the C5o and C52 peaks respectively at the baseline as given by constructing triangles using the tangents at the points of inflexion.

The resolution should be at least 1, 3.

8.4.2. When glass capillary columns are used, separation of the C5o and C52 peaks is so complete that the small C51 peak appears as a distinct, separate peak between the two. It is therefore not necessary to test the efficiency of separation.

8.5. Calculation of the triglyceride composition

8.5. In the case of analysis on packed columns, the peaks with C numbers 4o, 42, 44, 46, 48, 5o, 52, 54 and 56 (and sometimes 58 as well) are recorded (area %) and used in the calculation. The triglyceride range C3o-C46 is practically blank for pure cocoa butter.

See also Note 2.

8.5.2. If necessary, the peak areas % are normalized so that

% C4o + % C42 + % C44 + % C46 + % C48 + % C5o + % C52 + % C54 + % C56 = 1oo,o %.

The triglyceride data normalized in this way enable non-cocoa butter triglycerides in the range C4o-C46 to be detected. When fats of the coconut/palm kernel fat group are not present, the milk fat content can be calculated from the sum of the triglycerides % C4o + % C42 + % C44 (see 11 and 14.1.).

8.5.3. The triglyceride data determined in accordance with 8.5.1. are subjected to a second normalisation so that

% C5o* + % C52* + % C54* = 1oo,o %.

The triglyceride values normalized in this way are indicated by placing an asterisk (*) after them. They are needed to find the "cocoa butter line" (9) and to calculate milk fat corrections (1o) and the NCBF (CBE) content.

8.5.4. The peaks of triglycerides with an odd number of carbon atoms are also separated and recorded by analysis using capillary columns. In calculating the triglyceride composition the peak area % values for odd-numbered triglyceride peaks are added to the values for the preceding even-numbered triglyceride peaks and the sums are given as the peak area % for the even-numbered triglyceride peaks (example: % C5o + % C51 is quoted as "% C5o").

The simplified triglyceride data so obtained are normalized and evaluated as described in 8.5.2. and 8.5.3.

9. Determination of the "cocoa butter line"

9.1. The "cocoa butter line" is defined as the straight line obtained by linear regression of the % C5o* values (y) on the corresponding values of % C54* (x) for different cocoa butter samples.

The "cocoa butter line" can be described by the following equation:

$$\% C5o^* = a + b \cdot \% C54^*,$$

where a is the point at which the cocoa butter line cuts the % C5o*(y)-axis, and b is the slope of the cocoa butter line.

9.2. The position of the "cocoa butter line" in the % C54*(x) - % C5o*(y) system of coordinates (see Figure 1) is found by analysing 6 authentic cocoa butter samples by the method of 8.2. or 8.3. and normalizing the results as described in 8.5.3. or 8.5.4.

The samples of cocoa butter should be selected so that the difference between the highest and lowest value of % C54* is such that

$$\% C54^*_{max} - \% C54^*_{min} = 6 \pm 1\%.$$

The mean value for % C52* calculated for the six samples of cocoa butter should be about 47 ± o,5%.

9.3. The intercept on the axis a and the gradient b (in the equation given in 9.1.) should be calculated from the % C5o* and % C54* data for the 6 cocoa butter samples as further checks that the "cocoa butter line" is in the correct position. The values obtained should be in the following ranges:

$$b = -o,66 \ldots -o,8o$$

$$(17,61 - 35,66 \cdot b) + o,6o \geq a \geq (17,61 - 35,66 \cdot b) - o,6o$$

The residual standard deviation should not be greater than o,2.

Substantial departures from these criteria can adversely affect the correctness of the calculated NCBF (CBE) values. If this occurs the analyst will have to make appropriate changes to his chromatographic conditions.

9.4. Ageing of the separating columns can cause changes in the conditions of analysis that may affect the position of the "cocoa butter line". The constancy of this line should therefore be checked routinely.

1o. Check on the position of the CBE band

The position of the CBE band in the % C54*(x) - % C5o*(y) coordinate system is predetermined with this analytical method (see Figure 1 and 14.3. and 14.4.). If the position of the "cocoa butter line" meets the conditions given in 9.2. and 9.3., it may be assumed that the position of the CBE band as defined in this procedure is also in conformity with the analytical conditions. If necessary, the position of the CBE band can be checked by analysing suitable CBE samples.

11. Determination of the factor F_{MF} for calculating the milk fat content from the sum % C4o + % C42 + % C44

11.1. Mixtures of known milk fat content (about 2o%) are made by melting a cocoa butter of known triglyceride composition (normalisation: % C4o + % C42 + ... + % C56 = 1oo,o%) together with any milk fats, the mixtures are analysed by the methods of 8.2. or 8.3. and the results are normalized in accordance with 8.5.2.

11.2. The factor

$$F_{MF} = \frac{\%\ MF}{\%\ C4o + \%\ C42 + \%\ C44}$$

where % MF = milk fat content of the mixture in % by weight and % C4o, % C42, % C44 are the peak areas %, is calculated to two places of decimals for each of the fat mixtures. The factors are averaged and quoted to one place of decimals; they should lie between 4 and 5.

11.3. At least 6 mixtures with different milk fats should be analysed to calculate a mean value for the factor F_{MF}.

12. Milk fat corrections of % C5o*, % C52* and % C54* prior to calculating the NCBF (CBE) content

12.1. It is convenient to determine the correction using the same triglyceride analysis data as were used to calculate the factor F_{MF} (see 11.1.).

12.2. As the calculation is made for the normalized triglyceride values % C5o*, % C52* and % C54*, it is necessary to assume a mean value for the content of % C5o, % C52 and % C54 triglycerides in the milk fats; the mean % C5o +

% C52 + % C54 content of the κ fats is taken to be 28%.

12.3. The milk fat correction for the triglycerides % C5o*, % C52* and % C54* for each 1% milk fat are calculated from the following formula (taking % C5o* as example):

$$\frac{\%\ C5o^*_{CB+MF} \cdot (a \cdot b + c \cdot d) - \%\ C5o^*_{CB} \cdot a \cdot b}{\%\ MF} = \%\ C5o^*K$$

where $\%\ C5o^*_{CB+MF}$ = Peak area % C5o* for the cocoa butter/milk fat mixture

$\%\ C5o^*_{CB}$ = Peak area % C5o* for the cocoa butter in the mixture

% MF = Milk fat content of the mixture in % by weight

a = $\frac{\%\ C5o_{CB} + \%\ C52_{CB} + \%\ C54_{CB}}{1oo}$

($\%\ C5o_{CB}$, $\%\ C52_{CB}$ and $\%\ C54_{CB}$: peak areas % of the corresponding cocoa butter triglycerides; normalisation: % C48 + ... % C58 = 1oo,o%)

b = $\frac{1oo - \%\ MF}{1oo}$ = cocoa butter content of mixture in % by weight

c = % MF/1oo

d = o,28 = $\frac{\%\ C5o_{MF} + \%\ C52_{MF} + \%\ C54_{MF}}{1oo}$

($\%\ C5o_{MF}$, $\%\ C52_{MF}$ and $\%\ C54_{MF}$: peak areas % of the corresponding milk fat triglycerides; normalisation: % C~~26~~40 + ... % C56 = 1oo,o%)

$\%\ C5o^*_K$ = Correction for the triglyceride value % C5o* for each 1% milk fat.

The corrections $\%\ C52^*_K$ and $\%\ C54^*_K$ are calculated by replacing $\%\ C5o^*_{CB+MF}$ in above formula by $\%\ C52^*_{CB+MF}$ and $\%\ C54^*_{CB+MF}$, respectively, and $\%\ C5o^*_{CB}$ by $\%\ C52^*_{CB}$ and $\%\ C54^*_{CB}$, respectively. The corrections are calculated to three places of decimals.

12.4. The corrections are averaged for each type of triglyceride and the means are quoted to two decimal places.

13. Determination of the milk fat content of chocolate fats

13.1. The milk fat content of a chocolate fat can be calculated from the sum % C4o + % C42 + % C44 (normalisation % C4o + ... % C56 = 1oo,o%) using the factor F_{MF} (see 11), providing the chocolate fat contains no other fats containing C4o, C42 and C44. In cases of doubt the milk fat content should be found by determining the content of C4 methyl ester in the methyl esters of the total fatty acids; other fats do not interfere with this determination.

13.2. The milk fat content is calculated from the triglyceride data as

$$\% MF_{TG} = (\% C4o + \% C42 + \% C44) \cdot F_{MF}$$

Relative to the total fat content of a chocolate, the milk fat content calculated in this way may be expected to deviate from the true milk fat content by up to ± 1o% (relative).

13.3. The IUPAC method ... (in preparation) is used to determine the milk fat content from the C4 content of the total fat.

The C4 methyl ester content in the total fatty acid methyl esters of milk fat is taken as 4,o ± o,4 %.

The milk fat content determined by this method is denoted "% MF_{C4}".

14. Calculation of the NCBF (CBE) content of cocoa butter and chocolate fats

14.1. Milk fat corrections

14.1.1. The % $C5o^*_S$, % $C52^*_S$ and % $C54^*_S$ values of the milk-fat-containing chocolate fat to be analysed are corrected using the corrections for each 1% milk fat calculated as in 12. The milk fat content is determined in accordance with 13.

The milk-fat-free values of % C5o*, % C52* and % C54* are calculated using the following formula (for % $C5o^*_S$ as example)

$$\frac{\% C5o^*_S - (a_S \cdot b + c \cdot d) - \% MF_S \cdot \% C5o^*_K}{a_S \cdot b} = \% C5o^*_{SK}$$

where % $C5o^*_S$ = Peak ar... C5o* for the ~~chocolate~~ milk fat containing ~~milk~~ chocolate

% $C5o^*_K$ = Correction per 1% milk fat for % C5o*

% MF_S = Milk fat content of the chocolate fat in % (either % MF_{TG} or % MF_{C4}; see 13)

a_S = o,98 (estimated value of the quantity used in 12.3., whose precise value is not known in this case)

b = $\frac{1oo - \% MF_S}{1oo}$ = "cocoa butter" content of the chocolate fat, % by weight — % oil removed

c = % MF_S/1oo

d = o,28 (see explanation of terms in the formula of 12.3.)

% $C5o^*_{SK}$ = Peak area % C5o* in the milk-fat-free component of the chocolate fat

14.1.2. The corrected triglyceride values % $C52^*_{SK}$ and % $C54^*_{SK}$ are calculated by replacing the value % $C5o^*_S$ in the above formula by % $C52^*_S$ and % $C54^*_S$, respectively, and the value % $C5o^*_K$ by % $C52^*_K$ and % $C54^*_K$, respectively.

14.1.3. The corrected peak area % values for % $C5o^*_{SK}$, % $C52^*_{SK}$ and % $C54^*_{SK}$ are calculated to one decimal place.

See also Note 3.

14.1.4. Approximate corrected peak area % values for % $C5o^*_{SK}$, % $C52^*_{SK}$ and % $C54^*_{SK}$ can also be calculated by the following method:

The terms % MF_S · o,12, % MF_S · o,13 and % MF_S · o,o8, respectiveley, are subtracted from the triglyceride data of the chocolate fat: % $C5o^*_S$, % $C52^*_S$ and % $C54^*_S$. The corrected values for the three triglycerides are normalized in accordance with % C5o* + % C52* + % C54* = 1oo,o%, so giving approximate values for the corrected values % $C5o^*_{SK}$, % $C52^*_{SK}$ and % $C54^*_{SK}$ calculated by 14.1.

Example of calculation

Milk fat content of the chocolate fat phase (% MF_S) = 2o,o%.

Corrections for milk fat:

% C5o* = 2o,o · o,12 = 2,4o
% C52* = 2o,o · o,13 = 2,6o
% C54* = 2o,o · o,o8 = 1,6o

	% C5o*	% C52*	% C54*
Chocolate fat containing milk fat	22,4	41,8	35,8
Corrections for milk fat	- 2,4	- 2,6	- 1,8
	2o,o	39,2	34,2
Milk-fat-free chocolate	2o,o/o,934 = 21,4	39,2/o,934 = 42,o	34,2/o,934 = 36,6

14.2. Other corrections

14.2.1. Chocolate and milk chocolate normally contain up to o,5 % soya lecithin, and commercial soya lecithins contain about 4o% soyabean oil. Chocolate fat can therefore contain about o,5-1% soyabean oil depending on the fat and lecithin content of the chocolate. The soyabean oil content of chocolate fat arising from added lecithins may be several times this level in certain chocolate products like chocolate flakes for which a phosphatide content of up to 1% is permitted and in chocolate with an extremely low fat content. In such cases the triglyceride datas of the chocolate fat must be corrected to allow for its possible soyabean oil content. Each 1% of soyabean oil contributes o,2 % C52* and o,8% C54. The corrections are made by the same procedure as in 14.1.4. They are best applied after a correction for milk fat. It should be noted that any soyabean oil content in the milk-fat-free triglycerides % $C5o^*_{SK}$, % $C52^*_{SK}$ and % $C54^*_{SK}$ must be multiplied by $\frac{1oo-\%\ MF_S}{1oo}$ to obtain the soyabean oil content of the total fat.

The soyabean oil content of chocolate fat can be estimated from an analysis of the fatty acid methyl esters (% C18:2 for cocoa butter: about 3,5%, % C18:2 for soyabean oil: about 52%). It may be useful to determine the phosphatide content of the chocolate as well, because this provides information on any addition of lecithins and hence on a possible soyabean oil content.

14.2.2. Chocolates made with added hazelnuts, almonds, peanuts, soya flour and/or other foods that contain fats will also contain these fats. Appropriate corrections to the triglyceride values can be made if it is possible to determine the level of these fats in the total fat of the chocolate.

Chocolates to which such foods are added contain the corresponding proteins as well as the fats, so the fats present can be identified indirectly by means of specific detection of the proteins concerned (e.g. by immunochemical methods).

14.2.3. In the case of the shells of filled chocolates with fillings that contain fat, migration of the fat from the filling into the chocolate coating is a regular occurrence. This can change the triglyceride composition of the total fat in the coating so much that it is not possible to use triglyceride analysis to determine a CBE content.

14.3. Graphical evaluation of the triglyceride analyses (see Figure 3)

14.3.1. The CBE band is drawn in a % C54*(x)/% C5o*(y) coordinate system. The end-points of the upper and lower boundaries of the CBE band are given by the following coordinates:

Lower boundary: % C54* = o; % C5o* = 7o
% C54* = 8o; % C5o* = o

Upper boundary: % C54* = o; % C5o* = 84
% C54* = 94; % C5o* = o

14.3.2. The "cocoa butter line" (see 9) is also entered in the coordinate system. This is done by plotting the 6 pairs of values "% C5o*; % C54*", calculated as described in 9, in the coordinate system. The best straight line (the "cocoa butter line") as judged by eye is drawn through the 6 points.

Three points (CB2, CB1, CB3) are marked on this best straight line:

(a) mean value % C54* = % $C54^*_{CB2}$

(b) mean value % C54* + 3% = % $C54^*_{CB1}$

(c) mean value % C54* - 3% = % $C54^*_{CB3}$

The corresponding values for % $C5o^*_{CB2}$, % $C5o^*_{CB1}$ and % $C5o^*_{CB3}$ are read off on the % C5o* - axis and noted.

14.3.3. The corresponding values of % C52* are then calculated and noted:

(a) $\%\ C52^*_{CB2} = 100,0 - \%\ C54^*_{CB2} - \%\ C50^*_{CB2}$

(b) $\%\ C52^*_{CB1} = 100,0 - \%\ C54^*_{CB1} - \%\ C50^*_{CB1}$

(c) $\%\ C52^*_{CB3} = 100,0 - \%\ C54^*_{CB3} - \%\ C50^*_{CB3}$

14.3.4. The pair of values "$\%\ C50^*_S$; $\%\ C54^*_S$" (or the triglyceride values corrected as described in 14.1 and 14.2) are plotted (S) and the corresponding content of $\%\ C52^*_S$ noted.

14.3.5. Straight lines are drawn from the points CB1, CB2 and CB3 on the cocoa butter line through the point S and extended to cut the upper and lower boundaries of the CBE band. The points of intersection are denoted A,B,C,D,E and F, as shown in Figure 3.

14.3.6. The $\%\ C50^*$- and $\%\ C54^*$-coordinates at each point of intersection are read off from the diagram and noted ($\%\ C50^*_A$, $\%\ C54^*_A$; $\%\ C50^*_B$, $\%\ C54^*_B$... $\%\ C50^*_F$, $\%\ C54^*_F$), and the corresponding values of $\%\ C52^*$ are calculated:

$$\%\ C52^*_A = 100 - \%\ C54^*_A - \%\ C50^*_A$$

$$\%\ C52^*_B = 100 - \%\ C54^*_B - \%\ C50^*_B$$

$$\%\ C52^*_C = 100 - \%\ C54^*_C - \%\ C50^*_C$$

$$\%\ C52^*_D = 100 - \%\ C54^*_D - 1\%\ C50^*_D$$

$$\%\ C52^*_E = 100 - \%\ C54^*_E - 1\%\ C50^*_E$$

$$\%\ C52^*_F = 100 - \%\ C54^*_F - 1\%\ C50^*_F$$

14.3.7. It may happen in some cases that one or more of the points of intersection A-F lie outside the first quadrant, i.e. left of the $\%\ C50^*$-axis or below the $\%\ C54^*$-axis. These points of intersection have negative (and therefore meaningless) $\%\ C54^*$- or $\%\ C50^*$-coordinates. The following procedure is adopted:

(a) The coordinates of points of intersection with the lower or upper boundaries of the CBE band lying to the left of the $\%\ C50^*$-axis are replaced by the coordinates of the end-point of the CBE-band boundary concerned (lower boundary: $\%\ C50^* = 70,0$; $\%\ C54^* = 0$, from which we have for the calculations in 14.3.8.: $\%\ C52^* = 30,0$; upper boundary: $\%\ C50^* = 84,0$; $\%\ C54^* = 0$, from which we have for the calculations in 14.3.8: $\%\ C52^* = 16,0$).

(b) The coordinates of points of intersection with the upper or lower boundaries of the CBE band lying beneath the $\%\ C54^*$-axis are replaced by the coordinates of the end-point of the CBE-band boundary concerned (lower boundary: $\%\ C50^* = 0$; $\%\ C54^* = 80,0$, from which we have for the calculations in 14.3.8.: $\%\ C52^* = 20,0$; upper boundary: $\%\ C50^* = 0$; $\%\ C54^* = 94,0$, from which we have for the calculation in 14.3.8.: $\%\ C52^* = 6,0$).

14.3.8. The values for % NCBF (CBE) in the total fat or in the milk-fat-free component of the total fat corresponding to the 6 points of intersection are then calculated:

$$\frac{\%\ C52^*_{CB1} - \%\ C52^*_S}{\%\ C52^*_{CB1} - \%\ C52^*_A} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_A$$

$$\frac{\%\ C52^*_{CB1} - \%\ C52^*_S}{\%\ C52^*_{CB1} - \%\ C52^*_B} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_B$$

$$\frac{\%\ C52^*_{CB2} - \%\ C52^*_S}{\%\ C52^*_{CB2} - \%\ C52^*_C} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_C$$

$$\frac{\%\ C52^*_{CB2} - \%\ C52^*_S}{\%\ C52^*_{CB2} - \%\ C52^*_D} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_D$$

$$\frac{\%\ C52^*_{CB3} - \%\ C52^*_S}{\%\ C52^*_{CB3} - \%\ C52^*_E} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_E$$

$$\frac{\%\ C52^*_{CB3} - \%\ C52^*_S}{\%\ C52^*_{CB3} - \%\ C52^*_F} \cdot (100 - \%\ MF_S) = \%\ NCBF\ (CBE)_F$$

14.3.9. If the contents of fats other than milk fat in the chocolate fat have also been determined, the expression "$(100 - \%\ MF_S)$" in the equations of 14.3.8. should be appropriately modified. For instance, if a soyabean oil content ($= \%\ SO_S$) has been determined and allowed for by appropriate correction of the triglyceride values, the expression "$(100 - \%\ MF_S) - \%\ SO_S$)" must be used in the calculations.

14.3.10. The arithmetic mean of the 6 NCBF (CBE) values is found:

$$\frac{\%\ NCBF(CBE)_A + \ldots \%\ NCBF(CBE)_F}{6} = \%\ NCBF(CBE)_M$$

and so is the difference between the highest and lowest % NCBF(CBE) values, divided by 2:

$$\frac{\%\ NCBF(CBE)_{max} - \%\ NCBF(CBE)_{min}}{2} = D\ \%$$

The NCBF(CBE) content of the total fat, in % by weight, is th
% $NCBF(CBE)_M$ ± D %

14.3.11. The NCBF(CBE) content of a chocolate with fat content F % is found from

% $NCBF(CBE)_M$ · F %/1oo ± D % · F %/1oo

14.4. Computational evaluation of the triglyceride analyses

14.4.1. If there is a frequent need to evaluate quite large numbers of analyses or if it is desired to improve the accuracy, it is best to use a programmable calculator for the computational evaluation instead of a graphical interpretation.

The principle of this evaluation is described briefly below.

14.4.2. The following quantities are given:

(a) The gradient b and intercept on the axis a of the upper (subscript U) and lower (subscript L) boundaries of the CBE band:

a_U = 84,o; b_U = - o,894

a_L = 7o,o; b_L = - o,875

(b) The % C5o* and % C54* coordinates of the three points CB1, CB2 and CB3 on the "cocoa butter line" (see 14.3.2.).

(c) The % $C5o^*_S$, % $C52^*_S$ and % $C54^*_S$ values for the fat sample S, if necessary corrected for the milk fat content, etc.

14.4.3. The following quantities have to be calculated:

(a) The gradients b_i and intercepts on the axis a_i of the three straight lines (1,2,3) that pass through the points CB1 or CB2 or CB3 on the "cocoa butter line" and the point S (see Figure 3):

$$b_i = \frac{\Sigma xy - \frac{1}{n} \cdot \Sigma x \cdot \Sigma y}{\Sigma x^2 - \frac{1}{n} \cdot (\Sigma x)^2} \quad ; i = 1,2,3$$

$$a_i = \Sigma y/n - b_i \cdot \Sigma x/n \quad ; i = 1,2,3$$

x = % C54*; y = % C5o*; n = 2

(b) The % C5o* and % C5 coordinates of the points of intersection of the lines 1,2 and 3 with the lower and upper boundaries of the CBE band.

For the point of intersection A (see Figure 1):

$$\% C54^*_A = \frac{a_1 - a_L}{b_L - b_1}$$

$$\% C5o^*_A = a_L + b_L \cdot \% C54^*_A$$

% $C52^*_A$ is given by

$$\% C52^*_A = 1oo,o - \% C54^*_A - \% C5o^*_A$$

The remaining points of intersection are calculated in a similar way by substituting a_2, a_3, b_2, b_3 and b_U in the above formulae.

14.4.4. The values for % NCBF(CBE) are calculated using the calculated values of % $C52^*_A$ ... % $C52^*_F$, as indicated in 14.3.8.-14.3.11.

14.4.5. The procedure of 14.3.7. is adopted when the formulae of 14.4.3. give points of intersection with negative % C54* or % C5o coordinates.

14.4.6. The equations given in 14.4.3. cannot be used if it should by chance happen that exactly equal values of % C54* or % C5o* are obtained for the test fat sample S and one of the three points CB1, CB2 and CB3. However, this possibility is practically excluded if the triglyceride analyses are evaluated using a programmable calculator and there is n rounding off until the calculation of the values of % NCBF(CBE).

15. Repeatability r and reproducibility R of the triglyceride analyses

Type of triglyceride	Range	r	R
C 4o	1-2%	o,2	o,6
42	1-2%	o,1	o,4
44	1-2%	o,1	o,4
46	1-2%	o,1	o,6
48	1-3%	o,2	1,o
5o	16-43	o,3	2,o
52	4o-48	o,5	2,6
54	29-36	o,5	2,8
56	1-2	o,2	1,3

16. Hints for the evaluation

16.1. Application of the method described to chocolate fats containing only cocoa butter or only cocoa butter and milk fat gives NCBF(CBE) contents of o ± 3%; for chocolate with a fat content of 3o%, this gives an NCBF (CBE) content of o ± 1% for the chocolate itself. NCBF(CBE) values within this range should be interpreted as "blank values" unless more detailed analyses are performed.

16.2. If the method gives points of intersection with the boundaries of the CBE band (see 14.3.) that lie in the upper left part of the CBE band (% C5o: 7o-8o%, % C54: 2-6%), the CBE present is in all probability a palm oil mid fraction. In such cases the contents of the C48 triglyceride and of the C14 and C16 fatty acids generally also show a distinct increase.

16.3. If the method gives points of intersection with the boundaries of the CBE band (see 14.3.) that lie in the lower right part of the CBE band (e.g. % C5o: 2-1o%; % C54: 75-85%), the NCBF(CBE) present is generally a vegetable oil (e.g. hazelnut oil from finely chopped hazelnuts), but could also be sal fat (CBE) or a mixture of vegetable oils with sal fat. The presence of shea stearine (CBE) in such cases is unlikely, as this CBE is generally used only as a mixture with palm oil mid fraction. Vegetable oils (e.g. hazelnut oil) can be recognised from their high C18:1 and C18:2 contents and their high contents of OOO- and OOL-type triglycerides.

The presence of sal fat results in an increased content of C56 and C58 triglycerides and of C2o fatty acid. Shea stearine can be detected from the butyrospermol content in its unsaponifiable matter.

16.4. If the points of intersection do not lie at the ends of the CBE band, the CBEs present are generally mixtures of palm oil mid fraction and shea stearine and/or sal fat and/or fats of the illipe group, possibly mixed with hazelnut oil or other vegetable oils from other added foods. In such cases the evaluation should be carried out in accordance with 16.2. and 16.3.

16.5. If the analysis for NCBF(CBE) content yields a range which - relative to the chocolate - includes the content of 5%, further investigations are not generally necessary when the object is to determine whether or not the 5% limit has been exceeded.

16.6. Figures 4-6 (triangle diagr.) facilitate the evaluation of triglyceride and fatty acid analyses and give indications of the possible presence of certain fats.

17. Notes

Numbers in round brackets in the notes that follow refer to the correspondingly numbered section of the analytical procedure.

Note 1

The method described in based essentially on the following studies or publications:

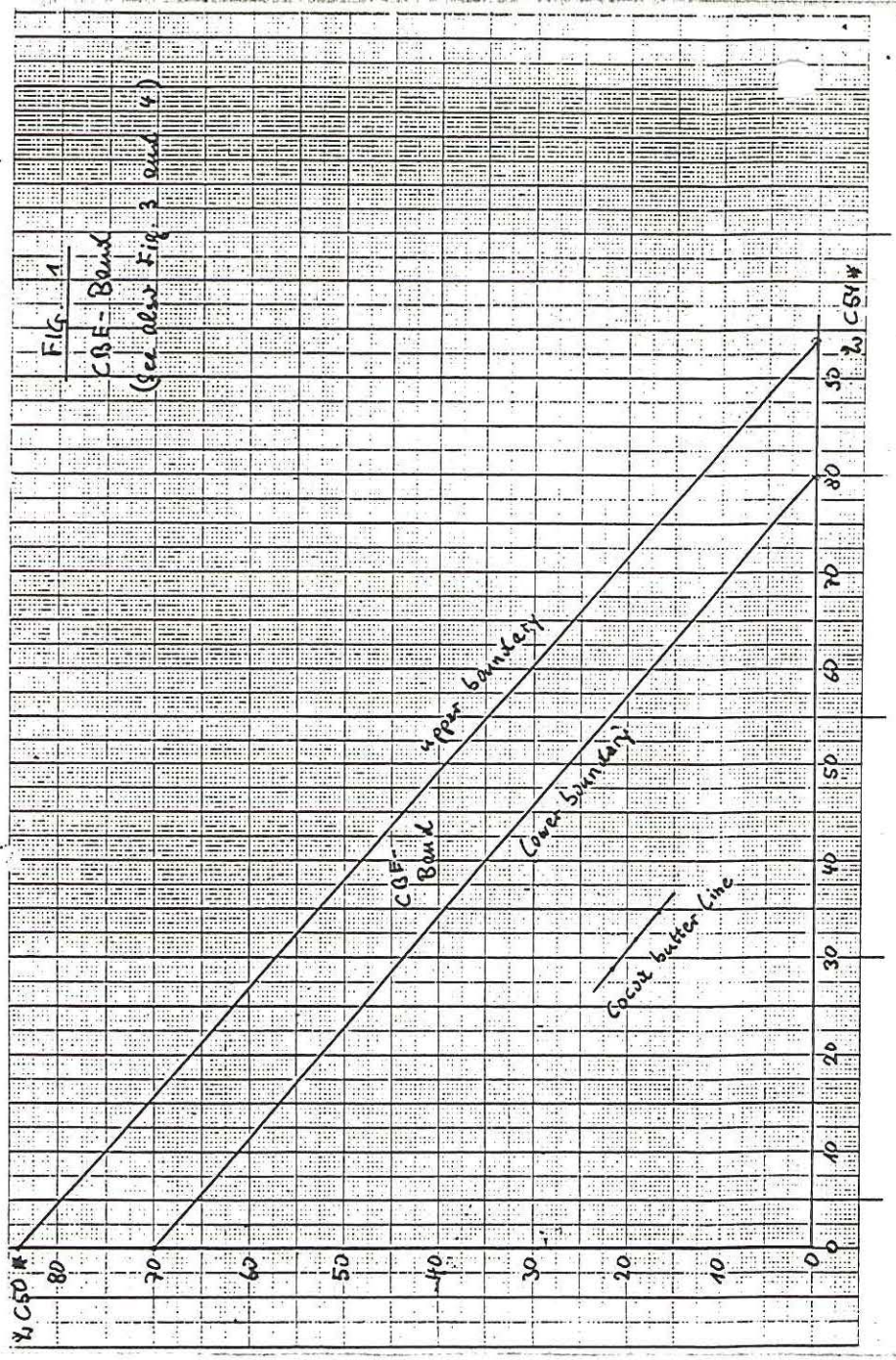
a) C. Young: J. Am. Oil Chem. Soc. 61 (1984) 576
b) F.B. Padley and R.E. Timms: Chem. and Ind. 2. Dec. 1978
c) F.B. Padley and R.E. Timms: J. Am. Oil Chem. Soc. 57 (198o) 286
d) A. Fincke: Dtsch. Lebensm. Rdsch. 76 (198o) 162
e) A. Fincke: Dtsch. Lebensm. Rdsch. 76 (198o) 187
f) A. Fincke: Dtsch. Lebensm. Rdsch. 76 (198o) 384
g) A. Fincke: Dtsch. Lebensm. Rdsch. 78 (1982) 389
h) A. Fincke: GdCh-Fortbildungskurs, Münster, 6./7.1o.1981
i) CAOBISCO, Results of second ring test on the determination of CBE; 1981/82

Note 2

CBEs containing fairly large amounts of palm oil mid fraction generally have a C46 peak. Peaks for C56 and C58 are indicative of the presence of groundnut oil (including hardened groundnut oil) or sal fat. The C6o peak can generally also be detected when groundnut oil is present.

Note 3

The factor a_s = o,98 is based on the assumption that in cocoa butter whose triglyceride data are normalized to "% C4ᵍ + % C5o + % C52 + % C54 + % C56 + % C58 = 1oo,o%", 98% is accounted for by the sum % C5o + % C52 + % C54.



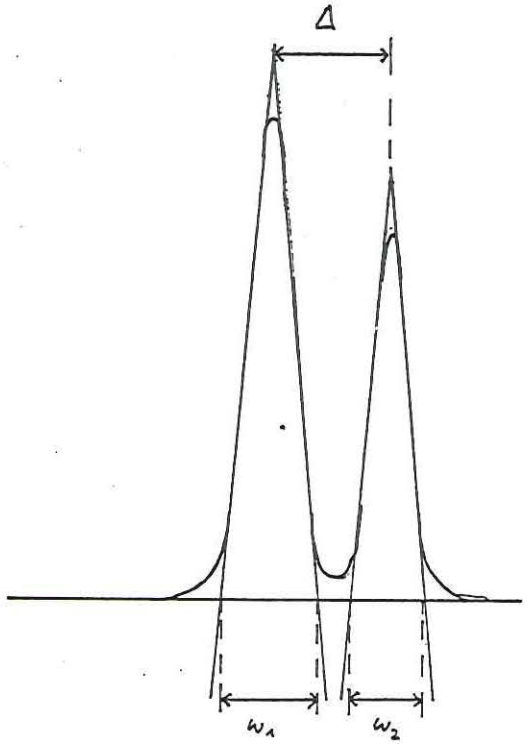


Fig. 2 Determination of the resulution

(see 8.4.)

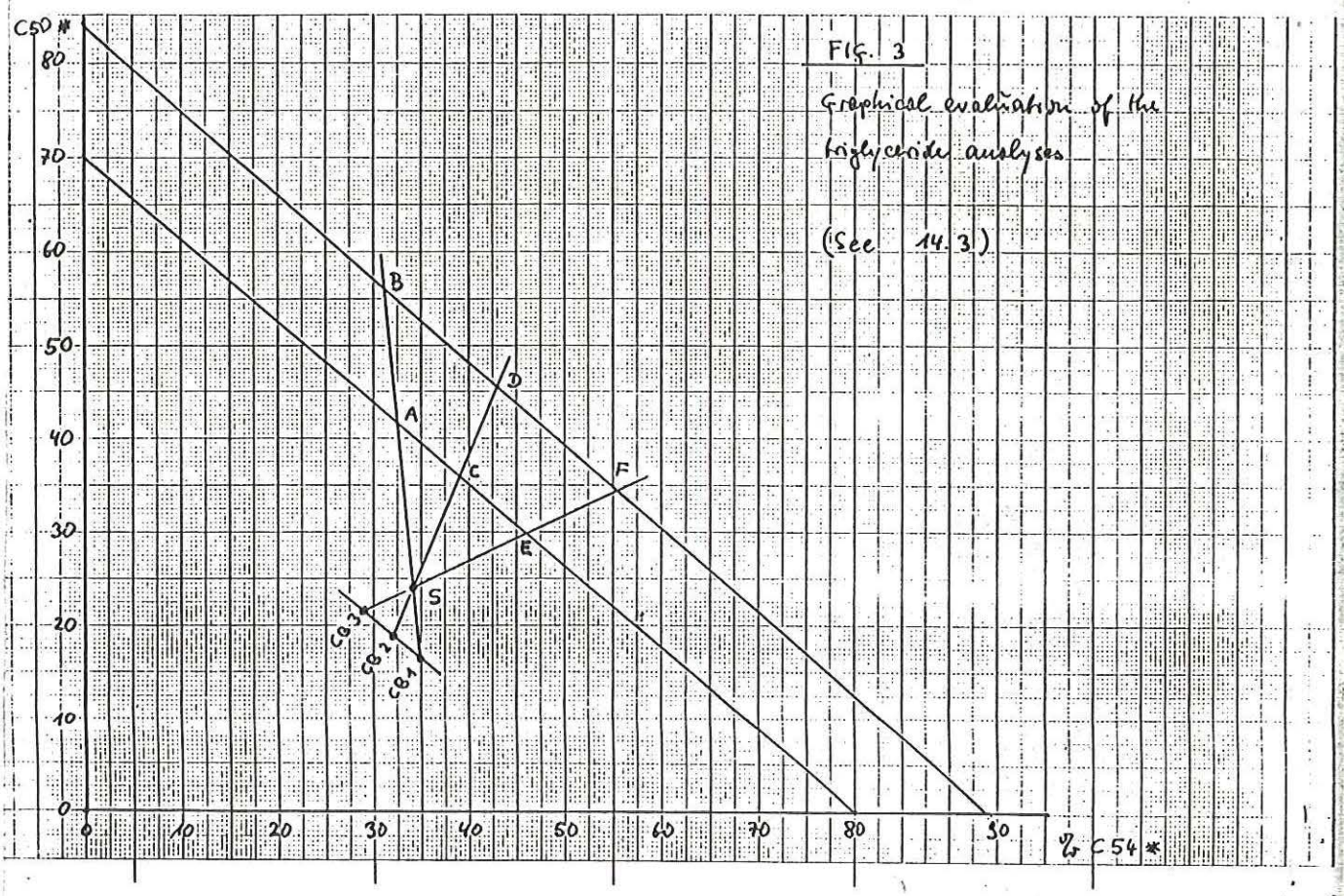


FIG. 3

Graphical evaluation of the triglyceride analyses

(See 14.3)

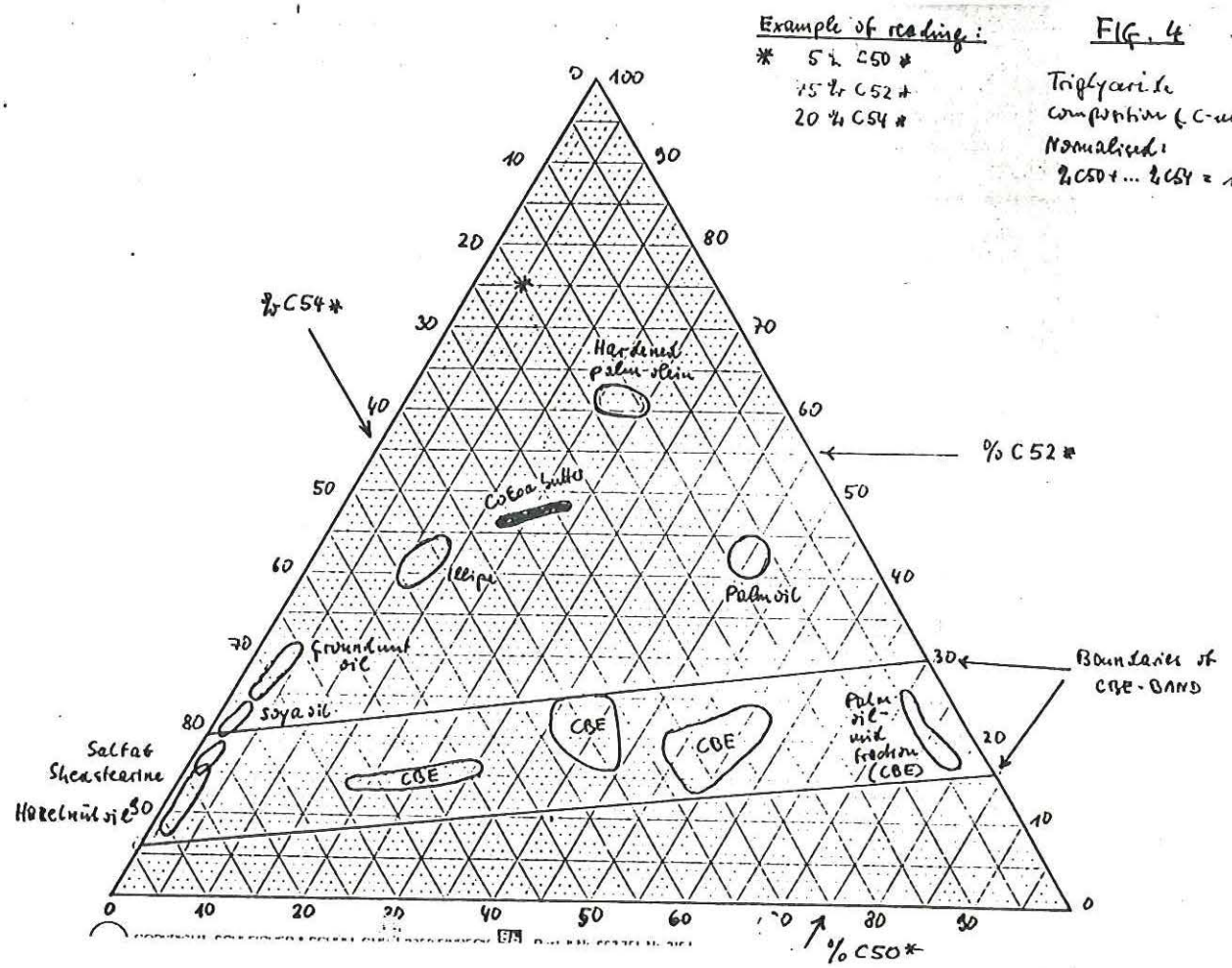


FIG. 4

Triglyceride composition (C-numbers)
Normalized:
% C50 * ... % C54 * = 100 %

FIG. 5

Fatty acid composition

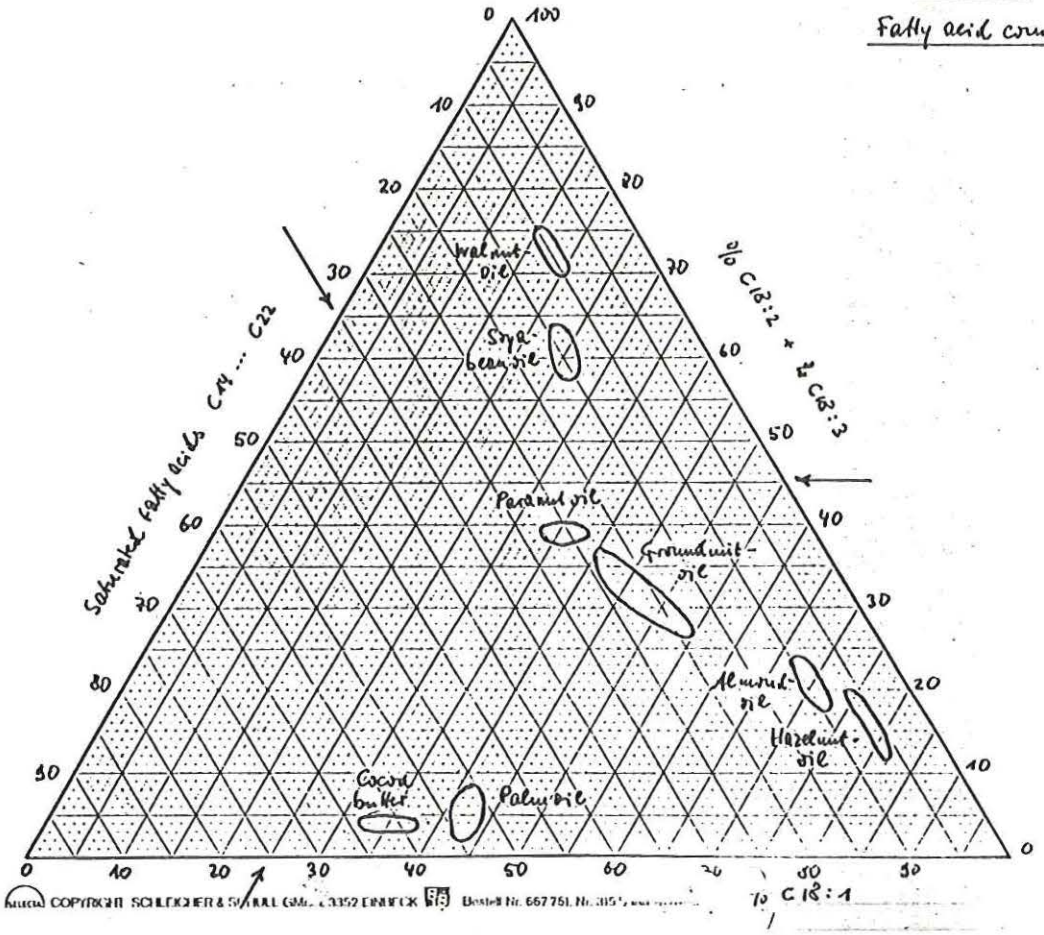
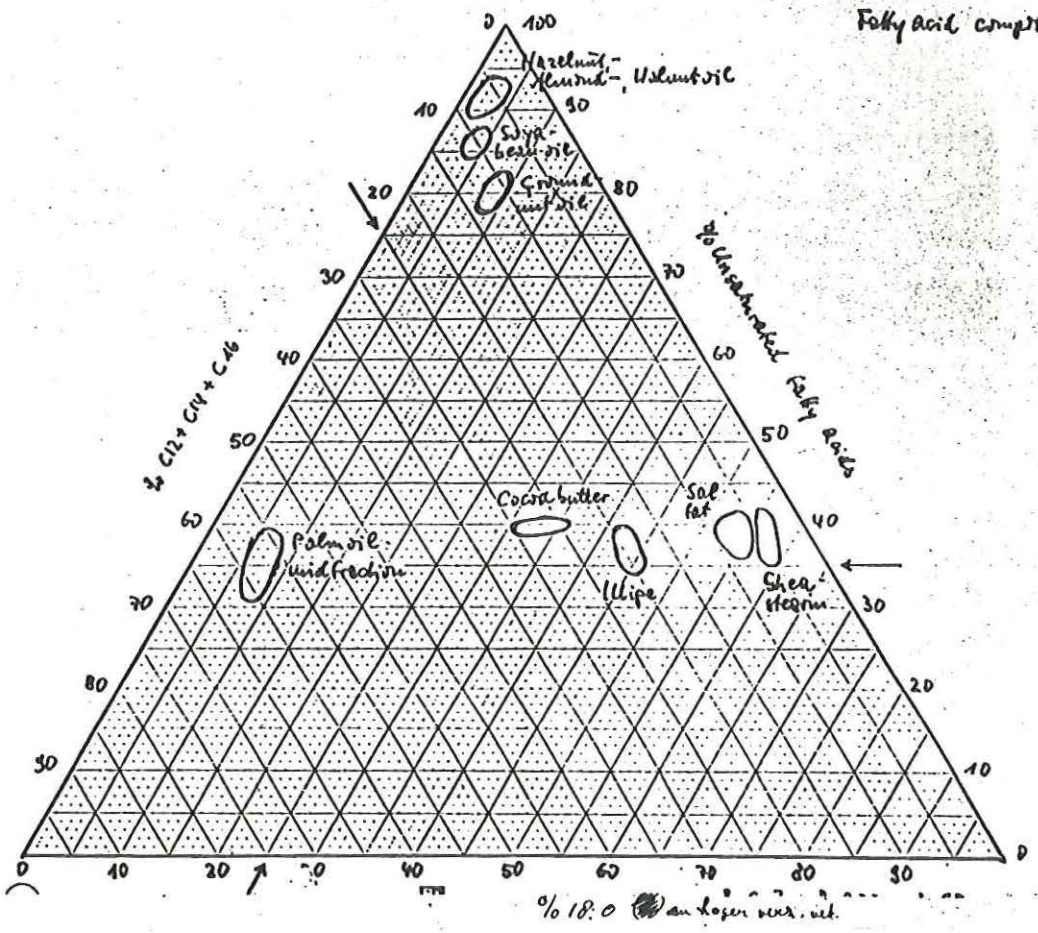


FIG. 6

Fatty acid composition



Results of RIKILT

1. Cocoabutter line: gradient b = -0.776
   intercept a = 45.24

2. Milkfat correction factor F_{MF}:

G	4.38
	4.59
	4.38
	4.16
	4.50
L	4.25

3. Milkfat correction for the triglycerides C50*-54* for each percent milkfat, assuming that the samples G-L are made with a mean mixture of A-F cocoabutter samples!

C50*K = 0.11
C52*K = 0.09
C54*K = 0.08

4. Percentage CBE calculated by means of computational evaluation of the triglyceride analysis (Percentages on total fat content.)
and:
Percentage of milkfat content.

Sample	1	2	3	4	5	6	7	8	9
NCBF A	-1.42	-2.00	19.72	12.69	20.98	17.88	11.18	20.17	26.54
NCBF B	-0.93	-0.81	11.18	7.57	12.75	9.73	6.42	12.22	16.72
NCBF C	1.11	1.18	20.28	13.89	22.07	18.80	12.18	21.05	27.34
NCBF D	0.71	0.72	12.66	8.56	13.63	11.35	7.46	12.95	17.40
NCBF E	3.49	2.95	21.94	15.10	23.16	19.74	13.19	21.94	28.13
NCBF F	2.32	1.96	13.56	9.50	14.52	12.13	8.25	13.69	18.45
NCBF GM	0.88	0.66	16.64	11.22	17.85	14.94	9.78	17.00	22.43
D	2.45	2.47	5.38	3.77	5.21	5.01	3.39	4.86	5.71
Milkfat	0.00	14.55	0.00	0.00	0.00	14.76	15.98	15.44	16.11

Sample	10	11	12	13	14	15	16	17	18
NCBF A	48.49	38.77	39.79	18.19	-1.93	24.55	49.71	-1.37	11.55
NCBF B	29.88	24.18	24.83	11.02	-0.78	15.47	30.66	-0.74	6.74
NCBF C	48.99	39.40	40.41	19.32	1.41	25.38	50.21	1.62	12.56
NCBF D	30.44	24.78	25.43	11.92	0.89	16.20	31.22	0.99	7.70
NCBF E	49.48	40.03	41.02	20.45	3.69	26.21	50.70	3.28	13.57
NCBF F	30.99	25.39	26.04	12.83	2.46	17.31	31.78	2.18	8.49
NCBF GM	39.71	32.09	32.92	15.62	0.95	20.85	40.71	0.99	10.10
D	9.80	7.93	8.10	4.72	2.81	5.37	10.02	2.33	3.42
Milkfat	15.55	15.87	15.88	0.00	0.00	15.25	14.25	15.34	15.59

Sample	% CBF	CBE	MF
1	99	1	–
2	84	1	15
3	83	17	–
4	89	11	–
5	82	18	–
6	70	15	15
7	74	10	16
8	68	17	15
9	62	22	16
10	44	40	16
11	52	32	16
12	51	33	16
13	84	16	–
14	99	1	–
15	64	21	15
16	45	41	14
17	84	1	15
18	74	10	16

Cocoabutter line.
Samples A-F.

Omni calc.	CB A		CB B		CB C	
C40						
C42						
C44						
C46						
C48						
C50	16.39	17.45	17.39	17.80	18.45	18.71
C52	41.49	45.05	44.62	46.57	45.59	45.57
C54	29.96	32.88	32.73	33.39	31.62	32.04
C56						
	Totaal	Totaal	Totaal	Totaal	Totaal	Totaal
	87.84	95.38	94.74	97.76	95.66	96.32
	100proc	100proc	100proc	100proc	100proc	100proc
C50*	18.66	18.29	18.35	18.20	19.28	19.42
C52*	47.23	47.23	47.09	47.63	47.66	47.31
C54*	34.10	34.47	34.54	34.15	33.05	33.26

Omni calc.	CB D		CB E		CB F	
C40						
C42						
C44						
C46						
C48						
C50	20.60	20.63	19.17	19.17	16.65	16.51
C52	45.65	45.57	45.63	45.63	44.40	44.58
C54	29.97	29.79	31.27	31.27	35.12	35.25
C56	0.00					
	Totaal	Totaal	Totaal	Totaal	Totaal	Totaal
	96.22	95.99	96.07	96.07	96.17	96.34
	100proc	100proc	100proc	100proc	100proc	100proc
C50*	21.41	21.49	19.95	19.95	17.31	17.13
C52*	47.44	47.47	47.49	47.49	46.17	46.27
C54*	31.14	31.03	32.55	32.55	36.52	36.59

Milkfat faktors.
Samples G-L.

Omni calc.	G	H	I	J	K	L		
C40	1.71	1.21	2.21	1.10	1.89	1.52		
C42	1.03	0.71	1.28	0.60	1.11	0.88		
C44	0.87	0.49	1.29	0.55	0.97	0.77		
C46	1.17	1.02	1.67	0.87	1.30	0.96		
C48	1.99	1.77	2.57	1.51	2.17	1.85		
C50	16.79	16.21	16.33	17.42	16.70	17.06		
C52	38.27	39.77	36.33	40.62	37.22	38.87		
C54	27.51	28.73	25.64	29.23	26.72	28.07		
C56								
	Totaal	Totaal	Totaal	Totaal	Totaal	Totaal		
	89.34	89.91	87.32	9.906	88.08	89.98		
	100proc	100proc	100proc	100proc	100proc	100proc		
	1.91	1.34	2.53	1.19	2.14	1.69		
	1.15	0.79	1.46	0.65	1.26	0.97		
	0.97	0.54	1.47	0.60	1.10	0.85		
	1.31	1.13	1.91	0.94	1.47	1.06		
	2.22	1.97	2.94	1.64	2.46	2.05		
	18.79	18.03	18.70	18.95	18.96	18.96		
	42.83	44.23	41.60	44.20	42.25	43.20		
	30.79	31.95	29.36	31.80	30.33	31.19		
	100.00	100.00	100.00	100.00	100.00	100.00		
% MF:	17.70	12.30	24.00	10.20	20.30	15.00		
C50*	20.33	19.13	20.85	19.96	20.71	20.31		
C52*	46.35	46.95	46.40	46.54	46.15	46.27		
C54*	33.31	33.91	32.74	33.49	33.13	33.41		
							mean:	
C50*K	0.11	0.05	0.11	0.13	0.12	0.12	0.65/6	0.11
C52*K	0.09	0.11	0.10	0.07	0.08	0.07	0.54/6	0.09
C54*K	0.07	0.11	0.06	0.08	0.07	0.08	0.48/6	0.08
	F MF	F MF	F MF	F MF	F MF	F MF	F MF	
	4.38	4.59	4.38	4.16	4.50	4.25	26.28/6	4.38

Mixtures Samples 1-9

Omni calc.	1	2	3	4	5	6	7	8	9
C40		1.38				1.45	1.55	1.51	1.54
C42		0.87				0.87	0.91	0.91	0.86
C44		0.72				0.71	0.79	0.75	0.89
C46		1.04				1.20	1.13	1.14	1.31
C48	0.88	2.00	0.69	0.98	1.33	2.52	2.28	2.47	2.25
C50	17.63	16.99	24.50	20.26	21.22	23.57	19.57	21.08	13.55
C52	43.59	38.48	40.30	40.64	38.82	35.38	35.95	34.45	31.68
C54	31.69	27.79	28.65	30.99	31.55	24.17	26.89	27.58	37.36
Total	93.79	89.37	94.14	92.87	92.92	89.87	89.07	89.89	89.44
C40*	0.00	1.54	0.00	0.00	0.00	1.61	1.74	1.68	1.72
C42*	0.00	0.97	0.00	0.00	0.00	0.97	1.02	1.01	0.96
C44*	0.00	0.80	0.00	0.00	0.00	0.79	0.88	0.83	0.99
C46*	0.00	1.16	0.00	0.00	0.00	1.33	1.27	1.27	1.46
C48*	0.94	2.23	0.73	1.05	1.43	2.80	2.56	2.74	2.51
C50*	18.79	19.01	26.02	21.81	22.83	26.22	21.97	23.45	15.15
C52*	46.47	43.17	42.81	43.76	41.77	39.36	40.36	38.32	35.42
C54*	33.79	31.09	30.43	33.37	33.95	26.89	30.19	30.68	41.77
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
%MF TG	0.00	14.55	0.00	0.00	0.00	14.76	15.98	15.44	16.11
Corr MF									
C50	18.79	17.41	26.02	21.81	22.83	24.60	20.21	21.75	13.37
C52	46.47	41.86	42.81	43.76	41.77	38.04	38.92	36.93	33.97
C54	33.79	29.93	30.43	33.37	33.95	25.71	28.91	29.44	40.48
Total	99.06	89.20	99.26	98.94	98.57	88.35	88.04	88.13	87.83
(Normalised on 100%)									
C50 SK*	18.97	19.51	26.21	22.05	23.17	27.84	22.95	24.68	15.23
C52 SK*	46.91	46.92	43.12	44.22	42.38	43.05	44.20	41.90	38.67
C54 SK*	34.11	33.55	30.66	33.72	34.44	29.10	32.83	33.41	46.09

CB line	C50	C54	C52
CB1	16.80	26.66	46.54
CB2	19.12	33.66	47.21
CB3	21.45	30.66	47.89

Sample lines tangens									
B1	-0.85	-0.87	-1.57	-1.79	-2.87	-1.46	-1.61	-2.42	-0.16
B2	-0.32	-3.79	-2.36	44.94	5.14	-1.91	-4.65	-22.36	-0.31
B3	-0.71	-0.66	-1893.20	0.19	0.45	-4.10	0.69	1.17	-0.40
abcis C50									
A1	48.05	49.89	74.32	82.35	122.29	70.37	75.82	105.73	22.89
A2	29.98	146.50	98.70	-1493.60	-154.03	83.55	175.83	771.84	29.65
A3	43.45	41.91	58067.55	15.46	7.53	147.31	0.21	-14.55	33.80

Mixtures Samples 10 t/m 18

Omni calc.	10	11	12	13	14	15	16	17	18
C40	1.51	1.50	1.51			1.50	1.34	1.48	1.50
C42	0.89	0.93	0.91			0.88	0.89	0.91	0.89
C44	0.80	0.79	0.83			0.75	0.73	0.76	0.78
C46	1.05	1.20	1.14			1.19	1.19	1.14	1.17
C48	2.60	2.30	2.20	1.44	0.92	2.15	2.58	2.22	2.16
C50	22.53	16.58	16.54	21.00	17.80	13.67	22.40	17.11	19.52
C52	28.29	29.23	29.29	39.48	43.71	32.53	28.46	38.49	35.95
C54	32.47	36.33	37.22	31.30	31.77	37.21	33.37	27.84	27.10
Total	90.14	88.86	89.64	93.22	94.20	89.88	90.96	89.95	89.07
C40*	1.67	1.69	1.68	0.00	0.00	1.67	1.47	1.64	1.68
C42*	0.98	1.04	1.01	0.00	0.00	0.98	0.98	1.01	1.00
C44*	0.88	0.89	0.92	0.00	0.00	0.83	0.80	0.84	0.87
C46*	1.16	1.35	1.27	0.00	0.00	1.32	1.31	2.46	1.31
C48*	2.88	2.59	2.45	1.54	0.97	2.39	2.83	2.46	2.42
C50*	24.99	18.66	18.45	22.52	18.69	15.21	24.62	19.02	21.91
C52*	31.38	32.89	32.67	42.35	46.40	36.19	31.29	42.79	40.36
C54*	36.02	40.88	41.52	33.57	33.72	41.40	36.68	30.95	30.42
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
%MF TG	15.55	15.87	15.88	0.00	0.00	15.25	14.25	15.34	15.59
Corr MF									
C50	23.28	16.91	16.70	22.52	18.89	13.53	23.06	17.33	20.20
C52	29.98	31.46	31.24	42.35	46.40	34.82	30.00	41.41	38.96
C54	34.77	39.61	40.25	33.57	33.72	40.18	35.54	29.72	29.18
Total	88.04	87.99	88.20	98.45	99.02	88.53	88.61	88.46	88.33
(Normalised on 100%)									
C50 SK*	26.44	19.22	18.94	22.88	19.08	15.28	26.02	19.59	22.86
C52 SK*	34.05	35.76	35.42	43.01	46.86	39.33	33.86	46.80	44.10
C54 SK*	39.50	45.02	45.63	34.10	34.06	45.38	40.11	33.59	33.03
CB line	C50	C54	C52						
CB1									
CB2									
CB3									
Sample lines									
tangens									
B1	3.39	0.29	0.24	-2.38	-0.87	-0.17	2.67	-0.91	-1.67
B2	1.25	0.01	-0.01	8.48	-0.09	-0.32	1.07	-7.65	-5.95
B3	0.56	-0.15	-0.16	0.41	-0.69	-0.42	0.48	-0.63	0.59
abcis	C50								
A1	-107.74	6.18	8.06	103.99	48.96	23.17	-81.04	50.25	78.08
A2	-23.10	18.82	19.63	-266.44	22.30	30.13	-16.87	276.59	219.53
A3	4.12	26.21	26.59	8.71	42.81	34.29	6.62	40.81	3.11

Mixtures Samples 1-9

Omni calc.	1	2	3	4	5	6	7	8	9
%C54 A	80.00	0.00	6.22	13.53	26.11	0.64	7.92	23.04	66.46
%C54 C	72.45	26.31	19.27	34.12	37.22	13.04	27.99	32.66	71.77
%C54 E	80.00	80.00	30.65	50.96	47.00	23.93	44.51	41.26	76.68
%C54 B	94.00	94.00	0.00	0.00	19.30	0.00	0.00	14.18	83.97
%C54 D	94.00	21.65	10.00	34.42	39.42	0.00	24.41	32.04	93.51
%C54 F	94.00	94.00	30.64	62.29	56.73	19.71	52.80	47.65	94.00
%C50 A	0.00	70.00	64.55	58.16	47.15	59.44	63.06	49.84	11.84
%C50 C	6.60	46.98	53.13	40.14	37.43	58.58	45.50	41.42	7.19
%C50 E	0.00	0.00	43.18	25.41	28.87	49.05	31.05	33.89	2.90
%C50 B	0.00	0.00	84.00	84.00	66.74	84.00	84.00	71.31	8.93
%C50 D	0.00	64.64	75.06	53.23	48.75	84.00	62.17	55.35	0.39
%C50 F	0.00	0.00	56.60	27.74	33.28	66.37	36.79	41.40	0.00
%C52 A	20.00	30.00	29.22	28.31	26.73	29.92	29.01	27.12	21.69
%C52 C	20.94	26.71	27.59	25.73	25.34	28.37	26.50	29.51	21.03
%C52 E	20.00	20.00	26.17	23.63	24.12	27.01	24.43	24.84	20.41
%C52 B	6.00	6.00	16.00	16.00	13.95	16.00	16.00	14.49	7.10
%C52 D	6.00	13.70	14.94	12.35	11.82	16.00	13.41	12.60	6.08
%C52 F	6.00	6.00	12.75	9.33	9.98	13.91	10.40	10.95	6.00
NCBF A	-1.42	-2.00	19.72	12.69	20.98	17.88	11.18	20.17	26.54
NCBF B	-0.93	-0.81	11.18	7.57	12.75	9.73	6.42	12.22	16.72
NCBF C	1.11	1.18	20.82	13.89	22.07	18.80	12.18	21.05	27.34
NCBF D	0.71	0.72	12.66	8.56	13.63	11.35	7.46	12.95	17.40
NCBF E	3.49	2.95	21.94	15.10	23.16	19.74	13.19	21.94	28.13
NCBF F	2.32	1.96	13.56	9.50	14.52	12.13	8.25	13.69	18.45
Mean NCBF GM	0.88	0.66	16.64	11.22	17.85	14.94	9.78	17.00	22.43

Mixtures Samples 1-9

Omni calc.	10	11	12	13	14	15	16	17	18
%C54 A	41.60	54.80	55.63	22.61	0.00	66.78	42.62	0.00	10.15
%C54 C	43.72	57.90	58.58	35.95	61.11	72.77	44.68	30.49	29.44
%C54 E	45.74	60.84	61.37	47.49	80.00	78.27	46.65	80.00	45.40
%C54 B	44.68	65.74	67.06	13.46	94.00	84.45	46.32	0.00	0.00
%C54 D	49.85	72.19	73.24	37.37	77.17	94.00	51.38	28.51	26.78
%C54 F	54.74	78.22	79.04	57.49	94.00	94.00	56.17	94.00	54.21
%C50 A	33.59	22.05	21.32	50.21	70.00	11.56	32.70	70.00	61.12
%C50 C	31.74	19.33	18.74	38.54	16.52	6.32	30.90	43.31	44.24
%C50 E	29.97	16.76	16.30	28.44	0.00	1.51	29.18	0.00	30.27
%C50 B	44.05	25.22	24.04	71.96	0.00	8.49	42.59	84.00	84.00
%C50 D	39.43	19.46	18.52	50.59	15.01	0.00	38.06	58.51	60.05
%C50 F	35.06	14.06	13.34	32.60	0.00	0.00	33.78	0.00	35.53
%C52 A	24.80	23.15	23.04	27.17	30.00	21.65	24.67	30.00	28.73
%C52 C	24.53	22.76	22.67	25.50	22.36	20.90	24.41	26.18	26.32
%C52 E	24.28	22.39	22.33	24.06	20.00	20.21	24.17	20.00	24.32
%C52 B	11.26	9.03	8.89	14.57	6.00	7.04	11.09	16.00	16.00
%C52 D	10.71	8.34	8.23	12.04	7.82	6.00	10.55	12.97	13.16
%C52 F	10.19	7.70	7652	9.90	6.00	6.00	10.04	6.00	10.25
NCBF A	48.49	38.77	39.79	18.19	-1.93	24.55	49.71	-1.37	11.55
NCBF B	29.88	24.18	24.83	11.02	-0.78	15.47	30.66	-0.74	6.74
NCBF C	49.99	39.40	40.41	19.32	1.41	25.38	50.21	1.62	12.56
NCBF D	30.44	24.78	25.43	11.92	0.89	16.20	31.22	0.99	7.70
NCBF E	49.48	40.03	41.02	20.45	3.69	26.21	50.70	3.28	13.57
NCBF F	30.99	25.39	26.04	12.83	2.46	17.31	31.78	2.18	8.49
Mean NCBF GM	39.71	32.09	32.92	15.62	0.95	20.85	40.71	0.99	10.10

THE DETERMINATION OF CBE

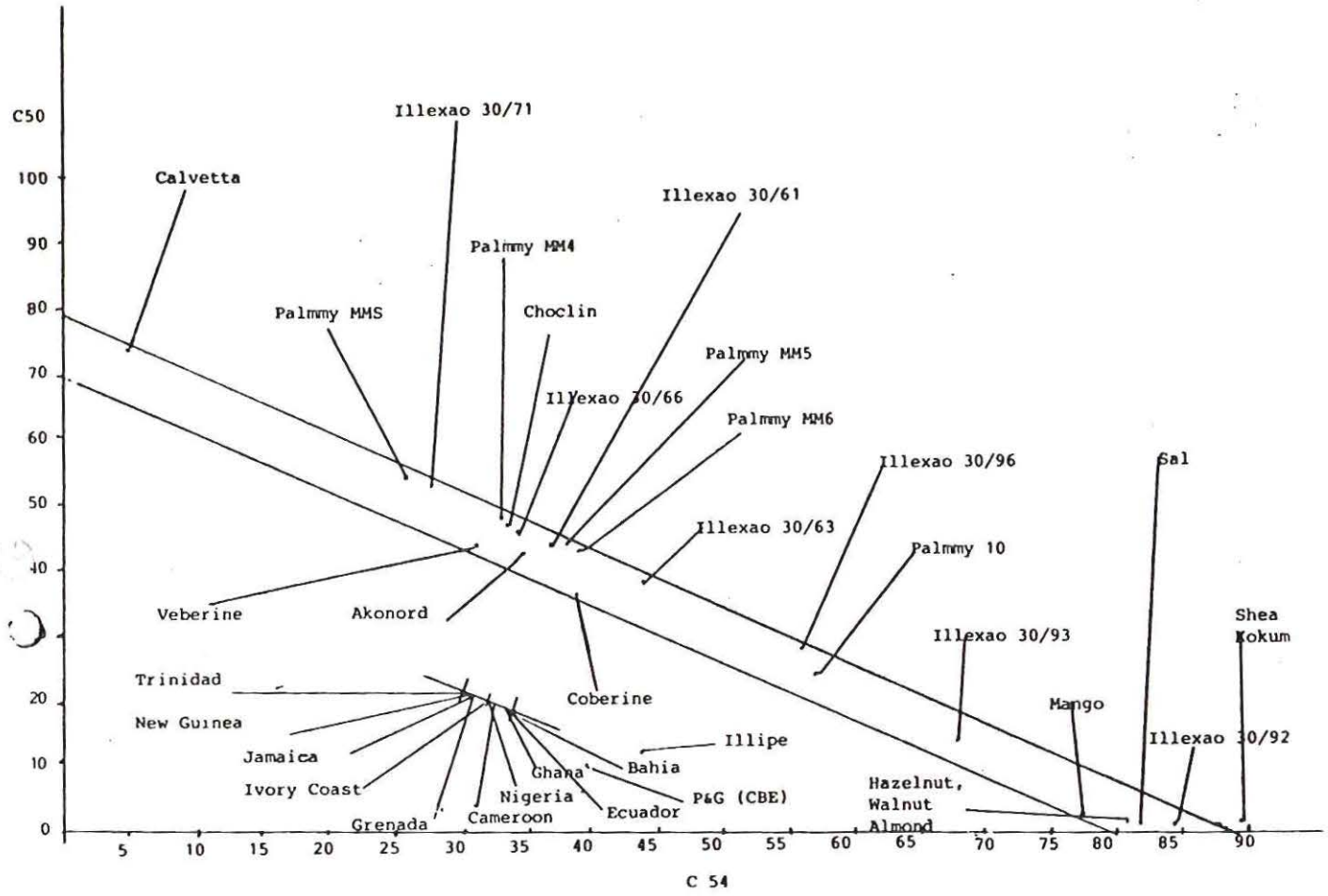
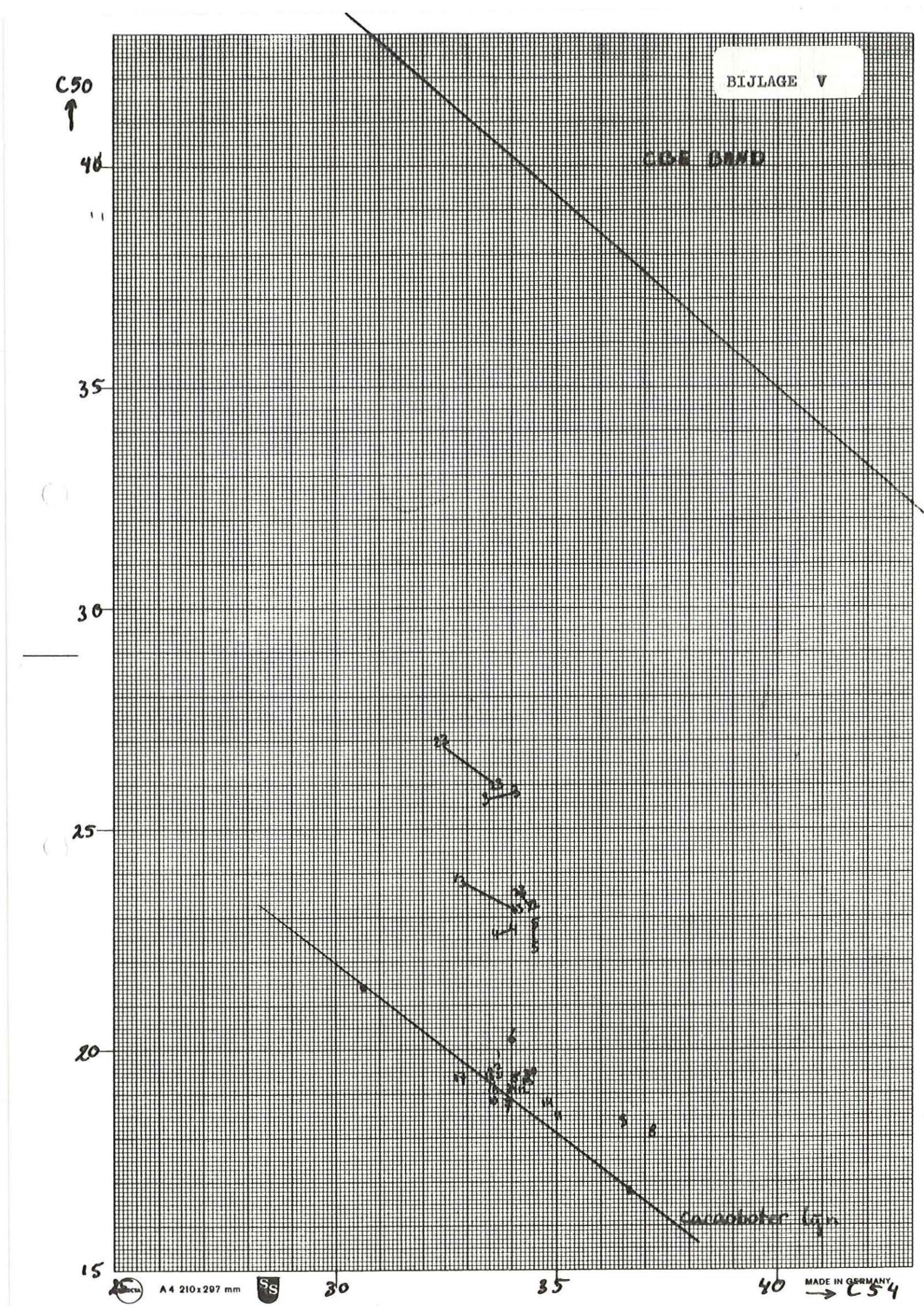


FIG. 1. C_{50} vs C_{54} plot of the GLC triglyceride data of CBE and cocoa butters.


BIJLAGE V
C50
40
35
30
25
20
15
30
35
40
C54
A4 210x297 mm
MADE IN GERMANY