Gas chromatographic analysis of free fatty acids and fatty acid salts extracted with neutral and acidified dichloromethane from office floor dust

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Abstract

A gas chromatographic method for estimation of fatty acid salts (FASs) in house dust has been developed. The difference between results for acidic and neutral extractions gives the FAS content of a sample. The method was designed to handle the inhomogeneity of the dust and to avoid false positive results due to low concentrations. The absolute recoveries of the model compounds octadecanoic acid and sodium octadecanoate were 91% and 77%, respectively. Problems with the internal standard and the inhomogeneity of floor dust, however, resulted in large variations and somewhat underestimated concentrations. The content of total FASs in eight samples of office floor dust were up to at least 0.5% in the fine particle fraction. © 1998 Elsevier Science B.V. All rights reserved.

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1. Introduction

In recent studies, correlations between self-reported eye irritation and objective changes of the tear film that covers the eye have been demonstrated [1,2]. These objective changes resemble those experimentally produced by installation of silicone emulsion [3,4] or benzalkonium chloride [5] in the human eye. Therefore, it has been postulated that the office environment may have a surface-active/lipophilic effect on the eyes and in this way produce a physicochemical eye dryness [1,6,7]. Cleaning agents are potential sources of surface-active compounds (detergents) in the indoor environment. Detergents are nonvolatile compounds and thus may be components of dust in houses. In addition, there is increasing evidence that dust is an important factor in the sick building syndrome (SBS) [8,9]. The physical properties of dust are probably not solely responsible for this; the chemical composition of the dust may also be important. The potential irritative effects of detergents [10] and possible adjuvant effect (any material that can increase an immune response [11]) [12] may be factors in SBS as well. The aim of this study, and the first step in testing the hypothesis was to show the existence of detergents in the indoor environment. Since the highest concentrations are probably found in floor dust and the most common detergents in floor cleaning agents in Denmark are fatty acid salts (FASs) [13], the aim was to develop a method for estimation of FASs in dust and apply it to floor dust samples. Three papers

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describe the development of gas chromatographic (GC) methods for determination of FASs in environmental samples. They are all concerned with organic compounds in marine aerosols. One includes a very detailed validation of the procedure [14], while another uses essentially the same method but gives very limited documentation [15]. The third article [16] offers little validation of a method slightly different from that in [14]. The method developed here differs from the others reported in the literature especially in the extraction step. The method was applied to eight floor dust samples from eight different office buildings.

2. Experimental

2.1. Chemicals and materials

The solvents used were dichloromethane (stabilised with ~20 ppm 2-methyl-2-butene), hexane, acetic acid, diethyl ether and methanol (analytical-reagent grade, Merck, Darmstadt, Germany), heptane, and 2-propanol (LiChrosolv, Merck). Trifluoroacetic acid (Uvasol, Merck) was used to acidify dichloromethane for the acidic extractions while methylation was catalyzed with boron trifluoride in methanol (20%, synthesis grade, Merck). Sodium chloride and anhydrous sodium sulphate (analytical-reagent grade, Merck) were used to minimize water solubility losses and for drying the organic phases. The internal standards were eicosanoic acid (C_{20} acid) ('puriss.' grade, Fluka, Buchs, Switzerland) and undecanoic acid (C_{11} acid) (99%, Sigma, St. Louis, MO, USA). For recovery studies sodium octadecanoate (Na-C_{18}) and octadecanoic acid (C_{18} acid) ('purum' grade, Fluka) were used. Desiccated coconut obtained from a supermarket was used as a fat surrogate. Calibration standards were methyl eicosanoate, methyl undecanoate, methyl oleate, methyl linoleate (99%, Sigma), methyl dodecanoate (99.5%, Sigma), methyl decanoate, methyl tetradecanoate, methyl hexadecanoate, methyl octadecanoate ('puriss.' grade, Fluka). Clean air was charcoal filtered (Gas-Clean, Chrompack, Middelburg, The Netherlands) compressed air. For solid-phase extraction of fatty acids (FA) aminopropyl Bond Elut® cartridges (100 mg, Varian, Harbor City,

CA, USA) were used. Soxhlet thimbles (10×50 mm) were obtained from Schleicher & Schuell (Dassel, Germany).

2.2. Equipment

The gas chromatograph (HRGC 5300, Carlo Erba, Milan, Italy) was equipped with a cold on-column (OC) injector, a flame ionisation detection (FID) system and a capillary column: wall-coated open tubular (WCOT) fused-silica, CP Sil 5 CB (100% dimethylsiloxane), 50 m×0.32 mm I.D., 0.13 μm film (Chrompack). The GC system was cooled with liquid nitrogen. The TURBOCHROM system (PE Nelson, Cupertino, CA, USA) combined with the personal computer (PC) spreadsheet QUATRO PRO for Windows (Borland, Scotts Valley, CA, USA) were used to acquire, integrate and handle the FID data. A Fourier transform infrared (FT-IR) spectrometer (IFS 85, Bruker, Karlsruhe, Germany) was coupled to the GC via a heated transfer line and a light pipe (for details, see [17]). The PC software OPUS (Bruker) was used to control, acquire and manipulate data from the FT-IR spectrometer. The infrared gas phase libraries from EPA (Environmental Protection Agency, USA) (supplied by Bruker) and NIST/EPA (the merged data collections from National Institute of Standards and Technology, Gaithersburg, MD, USA and EPA) were used for identification.

2.3. Sampling

Floor dust sampling was done with a specially designed vacuum cleaner HVS3 (Cascade Stack Sampling Systems, OR, USA) [18]. HSV3 was modified to ensure a more constant suction pressure and volume before dust was sampled from locations selected as being representative of eight office buildings (A to H) [19]. Recently, the design and use of the HSV3 has been standardized [20]. The samples were stored in vials at ambient temperature.

2.4. Sample preparation

The dust samples were divided into particle and fibre fractions with a sieve (pore size 1.25 mm). The fraction that did not pass through the sieve was called fibre. The particle fractions were further

sieved (pore size 0.5 mm) to assure homogeneity. Approximately 500 mg of this fraction was transferred to a Soxhlet thimble, spiked with 2.00 ml internal standard (~0.8 mg C_{20} acid) in dichloromethane, and extracted for 6 h (corresponding to 24–36 cycles) with 25 ml of a 0.06% (v/v) trifluoroacetic acid in dichloromethane in order to protonate and thus solubilize FASs (acidic extraction). The extract was spiked with 2.00 ml of a second internal standard (~0.8 mg C_{11} acid in dichloromethane). The extract was washed three times with 10 ml water to remove excess trifluoroacetic acid, dried with 0.5 g sodium sulphate, filtered, and evaporated to ~10 ml. The extracted FAs were isolated using a Bond Elut column [21]: the column was washed twice with 1 ml hexane (the column must not be completely dry), 3 ml of the extract was applied to the column under weak suction followed by two portions (0.8 ml) of dichloromethane–2-propanol (2:1, v/v), and the eluent was rejected. Finally, the column was eluted with 2 ml acetic acid–diethyl ether (1:20, v/v), and the eluent was evaporated to dryness under a gentle stream of clean air at ambient temperature. The residue was methylated according to a standard method [22]: 1 ml of boron trifluoride–methanol and 1 ml methanol were added, refluxed for 30 min, 2 ml heptane was added, and the mixture refluxed 10 min more. After cooling, ~50 ml saturated sodium chloride solution was added to float the heptane solution of methyl esters into the neck of the flask. A 1-ml volume of the upper heptane solution was transferred to a vial, dried with sodium sulphate, and diluted to appropriate concentration for injection of 1 μl on-column. The content of free FAs in the dust samples was estimated by extraction of approximately 500 mg dust with dichloromethane without addition of trifluoroacetic acid (neutral extraction) and otherwise using the identical analytical procedure. The procedures was repeated to obtain duplicate or triplicate estimations of the total content of free FAs and FASs in the dust samples.

2.5. Analytical conditions

The GC oven was held at 20°C for 2 min and temperature-programmed to 100°C at 20°C/min, from 100 to 280°C at 10°C/min, and held for 18 min at 280°C. The FID temperature was 330°C. The carrier and make-up gas was helium at 1.8 ml/min and 32 ml/min at 20°C, respectively. A 1-μl volume of the solution was injected onto the analytical column and every extract was analyzed at least in triplicate. The peaks were identified by retention times and by comparison of FT-IR spectra with library spectra. The heated transfer line from the GC system to the spectrometer and the light pipe were held at 280°C. Make-up gas (helium) was applied to the light pipe (for more details, see [17]).

2.6. Recovery and blank studies

Absolute recoveries were estimated by weighing portions of ~0.02 g Na-C_{18} directly into an empty extraction thimble. The thimble was Soxhlet extracted with dichloromethane with and without trifluoroacetic acid (0.06%, v/v) as described above. The internal standard was added to the extracts when the extractions were terminated. The same procedure was used for portions of ~0.02 g C_{18} acid. The dust sample extracts were spiked with C_{11} acid as a second internal standard in order to estimate the absolute recovery of the first internal standard (C_{20} acid) which were spiked directly onto the weighed dust sample in the Soxhlet thimble. Portions (0.02 g) of desiccated coconut were extracted as a fat surrogate with 0.06% (v/v) trifluoroacetic acid in dichloromethane. The background contamination levels were estimated by extractions of empty Soxhlet thimbles.

2.7. Calibration

Stock solutions were prepared by dissolving the calibration standards in heptane. One containing the conversion product of the internal standard (methyl eicosanoate) and one containing the other calibration standards (the FA methyl esters). Four standard solutions were made by mixing the two stock solutions in varying ratios covering the ratios of analytes and internal standard found in the samples. The standard solutions were each analysed in triplicate.

2.8. Data treatment

Calibration curves were generated by plotting the

ratios of the GC area counts for the actual methyl ester and the methyl ester of the internal standard (C_{20} acid) versus the molar ratios and estimating the parameters by linear regression. The molar concentration of the FAs (the neutral extraction) or total FAs and FASs (the acidic extraction) in the dust was estimated using the equation

$$m = n/M_{dust} = n_{I.S.} A/A_{I.S.} - \alpha)/\beta/M_{dust} \tag{1}$$

where m = molar concentration in the dust of extracted material measured as FA methyl ester, n = mol of methyl ester, M_{dust} = mass of extracted dust A = area counts of the actual methyl ester, $A_{I.S.}$ = area counts of methyl ester of the internal standard, α = intercept of the calibration curve, β = slope of the calibration curve (relative molar response factor for actual methyl ester). The molar concentration of FASs in the dust was estimated as the difference between the results of the acidic and the neutral extraction

$$m_{salt} = m_A - m_N \tag{2}$$

where m_{salt} = molar concentration in the dust of FASs released by trifluoroacetic acid, m_A = molar concentration in the dust of FAs extracted by acidified dichloromethane (mean of duplicate or triplicate extraction) and m_N = molar concentration in the dust of FAs extracted by neutral dichloromethane (mean of duplicate or triplicate extraction). The molar concentrations were converted into w/w concentrations by multiplication by the molecular mass.

Confidence limits for the concentrations of both free FAs and FASs in the dust were estimated using the standard deviation obtained by pooling of the standard deviations of the acidic and the neutral extractions (F-test was performed). Confidence limits for the concentration of FAs and FASs in the

dust were estimated using Eqs. (3) and (4), respectively:

$$C_H \pm t \cdot s_{pool}/(N_N)^{\frac{1}{2}} \tag{3}$$

$$C_{Na} \pm t \cdot s_{pool}[(N_A + N_N)/(N_A N_N)]^{\frac{1}{2}} \tag{4}$$

where C_H = concentration of FAs, t = the value of the t-distribution for the number of degrees of freedom given by s_{pool}, s_{pool} = pooled standard deviation, and N_N = number of neutral extractions, C_{Na} = concentration of FASs in equivalents of the sodium salt, and N_A = number of acidic extractions.

3. Results

The absolute recoveries of the test compounds and the absolute mean recovery of the internal standard from the individual extractions of all samples are shown in Table 1. The relative mean recoveries were estimated to 82% for C_{18} acid and 70% for Na-C_{18} for the acidic extraction by correcting the recoveries of C_{18} acid and salt for the recovery of the C_{20} acid shown in Table 1. No hydrolysis of fat could be detected during acidic extractions of desiccated coconut. The blank chromatograms contained only a few insignificant peaks from background contaminants. The typical levels were below ~0.01 mg/g dust in equivalents of C_{18} acid in the 0.5 g standard amount of dust extracted. The background contaminants were not identified and no blank correction of the results was performed.

Several FA methyl esters were identified with GC–FT-IR in the extracts of the dust samples. They were FA esters with even and odd chain lengths, as well as some unsaturated FAs. Based on a prelimin-

Table 1
Absolute recovery percentages (S.D.) from Soxhlet extraction of three compounds

Extraction	Octadecanoic acid in empty thimble (%)	Sodium octadecanoate in empty thimble (%)	Eicosanoic acid spiked on the dust samples in thimbles (%)
Acidified dichloromethane	90.8 (5.5) ($n=3$)	77.3 (6.0) ($n=5$)	111 (11) ($n=16$)
Neutral dichloromethane	–	N.D. ($n=2$)	108 (13) ($n=18$)

n = Number of extractions, N.D. = not detected.

Table 2
Identification of the peaks in Fig. 1 and the relative molar response factors (β) estimated by linear regression ($\pm$95% confidence limits)

Peak		β
(1)	Decanoic acid methyl ester	0.71 ± 0.02
(2)	Undecanoic acid methyl ester (from internal standard)	
(3)	Dodecanoic acid methyl ester	0.87 ± 0.01
(4)	Tetradecanoic acid methyl ester	0.92 ± 0.01
(5)	Hexadecanoic acid methyl ester	0.99 ± 0.01
(6)	Linoleic acid methyl ester	0.86 ± 0.01
(7)	Oleic acid methyl ester	1.00 ± 0.01
(8)	Octadecanoic acid methyl ester	1.01 ± 0.01
(9)	Eicosanoic acid methyl ester (from internal standard)	

Eicosanoic acid was internal standard.

ary examination of an unspiked subset of two samples, the amount of C_{20} acid was estimated to be less than 2% of the amount added as internal standard. Except for decanoic acid, only the major compounds were quantified. All calibration curves (except for linoleic acid methyl ester) had an intercept of zero (5% significance level) and thus $\alpha=0$ was used in Eq. (1). The estimated relative molar response factors are listed in Table 2.

Fig. 1 shows chromatograms of methylated FAs extracted from floor dust with and without trifluoroacetic acid in the dichloromethane. The measured concentrations of FAs and FASs in the fine particle fraction of dust from the eight office buildings (A to H) estimated by the present method are shown in Figs. 2 and 3, respectively.

4. Discussion

4.1. Recoveries and interferences

The absolute recovery of 91% of C_{18} acid (Table 1) is high compared to the absolute recovery of 63% of 3-methyloctadecanoic acid obtained probably by spiking unused glass fibre filters [14]. The same authors reported relative recoveries from 90 to 101% for the C_{20}–C_{30} FASs with 3-methyloctadecanoic acid as internal standard [14]. The absolute recovery of the C_{20}–C_{30} FASs can be estimated to be between 57 and 64% (using the absolute recovery of 3-methyloctadecanoic acid). These absolute recoveries are low compared to the 77% found for Na-C_{18} (Table 1). This may be due to the much smaller

amounts used by Peltzer and Gagosian, to the different chemical nature of the paper thimble and the glass fibre filter, and to the different methods (they treated the filters with 0.1 M HCl–methanol prior to extraction with hexane). However, the 85% recovery of Na-C_{18} relative to C_{18} acid (calculated from the figures in Table 1) is lower than the values reported by Peltzer and Gagosian. Again, this may be due to methodical differences.

The absolute recoveries of C_{20} acid (the internal standard) from the samples (Table 1) were surprisingly high compared to an expected recovery of about 90% or lower. The high recoveries are probably due to a relatively high content of C_{20} acid in the samples. The other possibility is the particularly low recovery of C_{11} acid which was used to estimate the recoveries of C_{20} acid. However, this appears to be unlikely since C_{11} acid was added to the extracts after the extractions, which presumably is the high loss stage in the analytical procedure.

It was necessary to use an acid as internal standard to put it through the entire analytical procedure. The use of C_{20} acid as internal standard was assumed to be a good choice because of its low content in the two test samples, and because it should be a relatively rare FA in plant lipids except peanut oil [23]. In addition, C_{20} acid is readily available in analytical grade quality. The relatively high content of C_{20} acid found in the remaining the samples is probably due to a the large fraction of human skin lipids.

A large proportion of house dust consists of desquamated human skin [19,24]. Human surface lipids usually contain a large fraction of free FAs (up to ~30%) [25]. About 40% of these are saturated
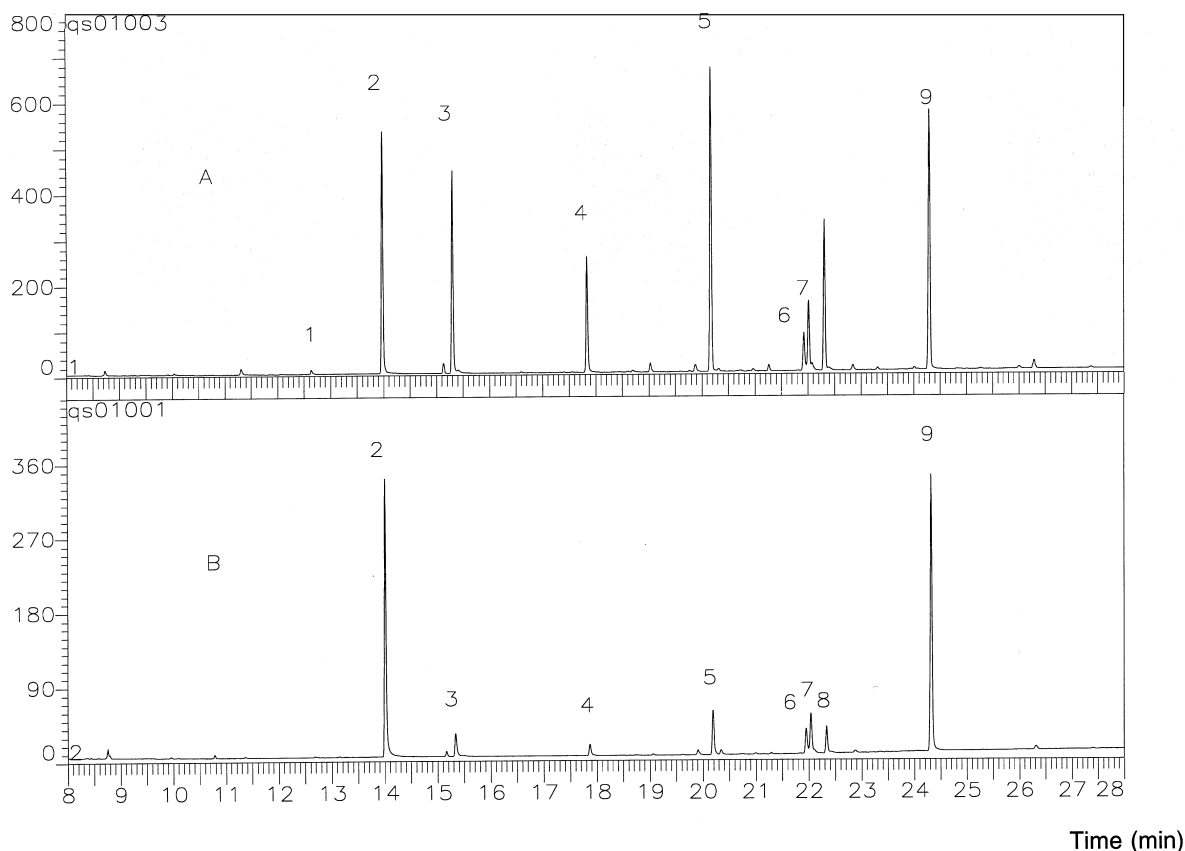
Fig. 1. OC-GC–FID chromatograms of methylated FAs extracted from the fine particle fraction of floor dust from office building F (peak identification is shown in Table 2, peaks 2 and 9 are internal standards): (A) Extraction with acidified dichloromethane (0.06%, v/v) trifluoroacetic acid which protonates the FASs), (B) extraction with neutral dichloromethane.

straight chain FAs with an even number of carbon atoms [26]. C_{20} acid has been found from one male to constitute 0.1% [27] and in a pool from several males to constitute 1.1% [28] of the FAs (C_{12}–C_{26}) in saponified scalp lipids. This indicates a large variation and thus individually higher contents than 1.1% for some of the males in the pooled sample. A large variation in content probably explains the large standard deviations for C_{20} acid recoveries in Table 1. The 1.1% corresponds to 7% of the C_{20} acid added to the samples extracted with neutral dichloromethane and 10% for the acidic extractions.

All extracts were spiked with C_{11} acid, an alternative quantitation standard. However, use of this led to much larger variation of the results of the individual extractions. The variation may reflect the inhomogeneity of the dust samples leading to differ-

ent extraction efficiencies or presence of C_{11} acid in some of the samples. In conclusion, the choice of C_{20} acid as internal standard was disadvantageous and instead a more exotic organic acid should be chosen as internal standard in future work.

Peltzer and Gagosian [14] concluded that wax ester hydrolysis by hydrochloric acid formed by decomposition of dichloromethane was an insignificant process during extraction from glass-fibre filters. Wax ester hydrolysis during the acidic extraction in this study was not tested, but no hydrolysis of coconut fat could be detected. In addition, no simple esters were found in the dust samples, except for very small amounts of isopropyl tetradecanoate [29]. Furthermore, Table 1 shows that no C_{18} acid could be extracted from Na-C_{18} with neutral dichloromethane. Peltzer and Gagosian had significant blank
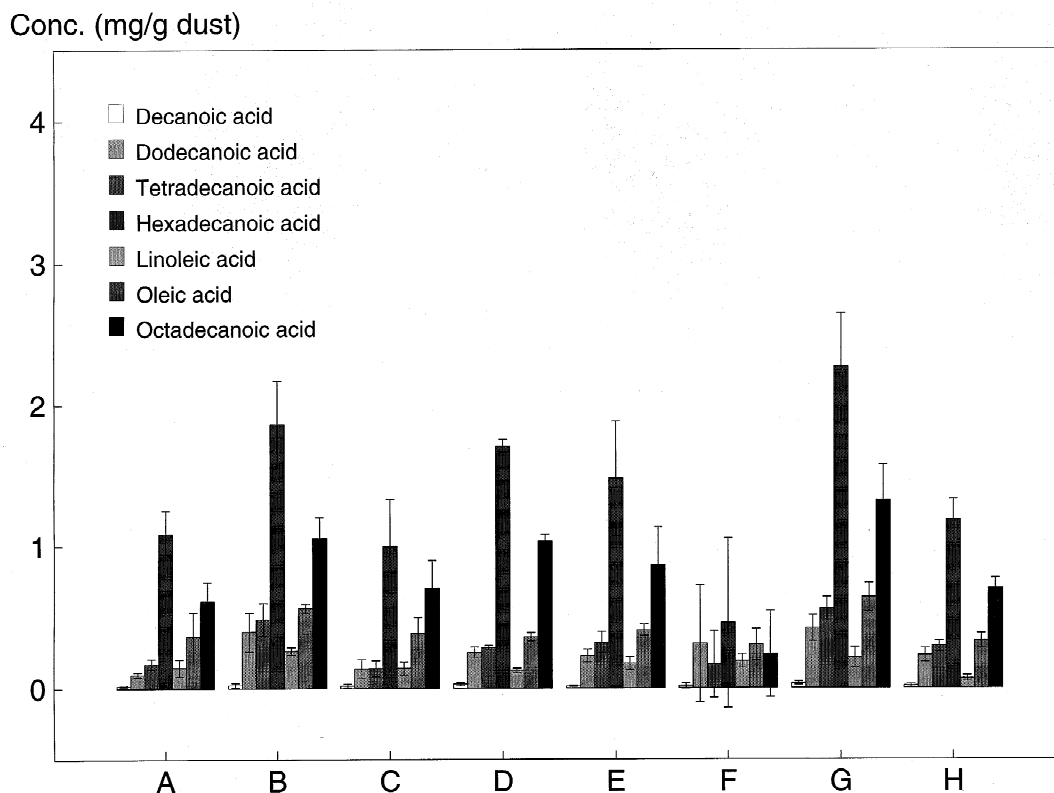
Conc. (mg/g dust)

Fig. 2. Concentrations and 95% confidence limits of the most frequent FAs in the fine particle fraction of floor dust from office buildings A to H.

values of the analytes especially for the FASs. This was not observed in this study and may be due to the much larger amounts used here (approximately 1000 times).

4.2. Calibration and quantification

As expected, the relative response factors (Table 2) decreased with decreasing chain length with the exception of linoleic acid. An explanation might be that the peak on the tailing side is incompletely resolved from the peak of oleic acid methyl ester. Because of the drop down integration, a significant part of the peak is cut off resulting in smaller response and significantly negative intercept of the calibration curve.

The absolute recoveries of the single compounds in the specific samples are not known but they are probably similar to or smaller than those of the

model compounds (Table 1). In spite of the apparently low mean recoveries of the model compounds relative to the internal standard the results were not corrected for recovery. Corrections will lead to complicated and ambiguous results. Thus, it may be concluded that the dust concentrations of FAs and FASs determined by the method are somewhat underestimated. In addition, the FASs are quantified as the sodium salts because the counter ions are unknown.

The confidence intervals are estimated by statistics based on the normal distribution though the 'true' distribution is unknown. Especially when the variation is large this may be a problem. To compensate, 95% confidence limits have conservatively been used instead of 90% that corresponds to a one-sided test. This increases the confidence intervals by 1.3–1.5 in this study. Thus, when the confidence intervals do not include zero we assume that the dust sample
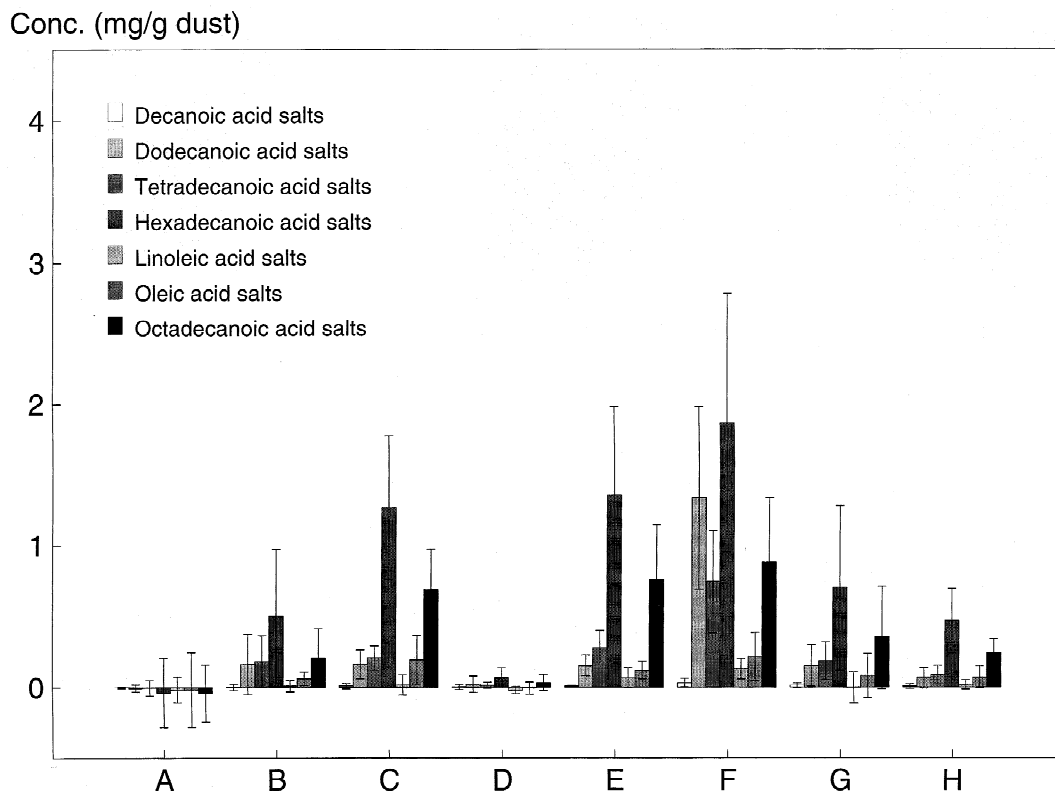
Conc. (mg/g dust)

Fig. 3. Most frequent FASs in the fine particle fraction of floor dust from office buildings A to H. Concentrations and 95% confidence limits are estimated in equivalents of the sodium salts.

contains the compound of concern and we use the result quantitatively. The results should, however, be considered as semi-quantitative because of the large variation, and underestimation of the concentrations and the use of sodium salt equivalents.

Since the pooled standard deviations used in Eqs. (3) and (4) are approximately equal, the N terms of the equations govern the relative size of the confidence intervals. Thus, the confidence intervals of the FAs concentrations will be approximately $\sqrt{2}$ times smaller than those for the concentrations of the FASs.

4.3. Miscellaneous aspects of the method

Even if a suitable internal standard without inter- ference problems was chosen, the problem of in-

homogeneity still remains. This may or may not be resolved by a mechanical homogenisation and siev- ing. Ultimately, the inhomogeneity may necessitate an increased number of extractions. In this study only the particle fraction (sieved) was analysed. Analysis of the fibre fraction would probably lead to even larger inhomogeneity problems.

The estimation of the FASs content in the dust as a difference between the results of the acidic and neutral extraction was used to avoid false positive results. The other published methods [14–16] uses an acidification followed by a second FA extraction. If the first extraction was incomplete or left residues of FAs, this gives a false positive content of FASs in the sample. In addition, the acidic extraction does not appear to result in lower recoveries than the acidifi- cation followed by an extraction and the procedure is one step shorter.

4.4. FAs and FASs in the floor dust

Using the criterion that both the FAs and the FASs can be quantified if the 95% confidence intervals do not include zero, only samples A and D do not contain FASs (see Figs. 2 and 3). The figures also show that the concentrations of both FAs and FASs in the dust samples are in the same order of magnitude. The large confidence intervals of building F in Fig. 2 indicate that only few FAs are present. This is probably not the case and must be due to sample inhomogeneity. Fig. 3 shows that the dust of buildings C, E and F contain FASs whereas the dust of buildings A and D do not contain FASs at statistically significant levels. The dust of the other buildings may contain FASs, but the confidence intervals are very large. The total amount of FASs in the samples (the sum of the measured FASs as the sodium salts) may vary from ~0% to at least ~0.5% with an approximately average content of 0.2%. The maximum content of single FASs in floor dust is at least 1.5 mg/g.

The pattern of the relative abundance of both FAs and FASs appears to be approximately the same in the samples with building F as an exception. The dust concentrations of both dodecanoic acid and dodecanoic acid salts are relatively high in building F. Characteristic of coconut oil is the high content of dodecanoic acid (~50%) [23]. Thus coconut oil may be a major source of FAs and FASs in building F. Coconut oil and soybean oils are the most frequently used oils for soap production.

5. Conclusion

We have developed a method for estimation of FASs in house dust, which takes into account the inhomogeneity of the dust and avoids false positive result at low concentrations. The absolute recoveries of the model compounds octadecanoic acid and sodium octadecanoate extracted from Soxhlet thimbles were 91% and 77%, respectively. This is better than the recoveries (for much smaller amounts) reported for homologous compounds extracted from glass-fibre filters. The choice of eicosanoic acid as internal standard was disadvantageous, probably because of interference from relatively large amounts of this acid in the samples. This resulted in underestimation of the concentrations. Thus, a more exotic organic acid should be chosen as internal standard. A major problem is inhomogeneity of floor dust which results in large variations of the estimated concentration. In conclusion, the results must conservatively be considered as semi-quantitative. Taking into consideration the large variations of the results, statistical analysis was conservatively used to decide whether or not a sample contained FAs or FASs. The method was applied to the fine particle fraction of eight floor dust samples from office buildings. Three of the samples contained clearly significant amounts of FASs, two did not appear to contain FASs, and the dust of the other buildings appeared to contain FASs (the uncertainties were very large). Based on these eight samples it can be concluded that floor dust from offices may or may not contain FASs. The content of total FASs may be up to at least 0.5% in the fine particle fraction of floor dust from offices. It is unknown whether such concentrations of FASs in floor dust can produce or contribute to eye irritation in the indoor environment.

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