

# Toward a Test of Overall Migration from the Coated Face of a Recycled Paperboard Food Contact Material into Fatty Food Simulants

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The main aim of the present work was to evaluate the suitability of recycled paperboard coated on its internal face for use in containers that contact fried foods. Suitability was assessed in terms of overall migration into fatty food simulants and in terms of the residue extracted into *n*-heptane. Testing of the packaging was carried out following the guidelines laid down by the EU for plastic packaging, and also those established by the FDA for the extractive testing of paper and paperboard for use in food contact. With a view to simplifying the test procedures, the results of the official tests were compared with those obtained using alternative fatty food simulants under comparable sets of test conditions. The overall migration test using olive oil as the fatty food simulant was an adaptation of the European Committee for Standardization (CEN) test developed for plastic materials. Two methods for determination of the amount of olive oil absorbed by the paperboard during the tests were compared: a gravimetric method and a modification of the CEN gas chromatographic method. Finally, the packaging materials were extracted with solvents of several different polarities in order to develop a test allowing preliminary identification of major potential migrating components in paperboard contributing to EU overall migration levels and FDA extractive levels.

**Keywords:** Food safety; recycled paperboard coated for food contact; EU overall migration tests; FDA extractive tests; fatty food contact

## INTRODUCTION

**Use of Recycled Paperboard in Food Packaging.** Paper and paperboard are in widespread use as food-packaging materials, frequently in forms adapted for direct contact with foodstuffs. To conserve and protect the environment, it is desirable that paper and paperboard food-packaging materials be manufactured largely from reclaimed fiber; however, the suitability of recycled materials for food contact is questionable on health and safety grounds (Overton, 1994; Nordic Council of Ministers, 1994). A major concern is the wide range of recycled fiber sources and the implications of this on the quality of the packaging material. In the European Union (EU), a system of classification of waste paper quality is currently being developed (Council of Europe, RD 16/25, 1994; Nordic Council of Ministers, 1994). Meanwhile, waste paper is selected for use by reference to lists classifying card and paper fiber in terms of previous use and technical parameters relevant to international trade (e.g. the list prepared by the European Confederation of Pulp, Paper and Board Industries (CEPAC)). However, such lists take no account of the potential health risks posed by the use of waste paper in the manufacture of food-contact materials.

In the United States, the Food and Drug Administration (FDA) has produced its own Code of Federal Regulations (CFR) for paper and paperboard in contact with food (FDA-Code of Federal Regulations). Within the European Union, Germany and Holland are some of the countries with specific regulations (BGA-Bundesgesundheitsamtes; Warenwet). In the EU as a whole, however, there is as yet no legislation for nonplastic food-packaging materials (EEC Directive 89/109/EEC,

1989), with the exception of ceramics and regenerated cellulose films. Currently, the Council of Europe is working on a draft document (CoE-RD 5/26, 1994; CoE-RD 32, 1994) that brings together proposals from several member states. Among these proposals is the recommendation that recycled paper and board should not be used for packaging of foods with a high fat or moisture content because of the health risk posed by the migration of packaging constituents into such foods (Nordic Council of Ministers, 1994).

**Importance of EU Overall Migration and FDA Extractive Tests.** Monitoring of the potential of food packaging constituents for migration into foodstuffs is achieved by means of overall or global migration (GM) tests. The aim of these tests is to guarantee the inertness of food-packaging materials and thus avoid unacceptable changes in the compositions of packaged foodstuffs (90/128/EEC; Piringer, 1994). The overall migration value covers all of the components of the food packaging except volatiles and affords some idea of the degree of alteration (toxic and/or organoleptic) of the food to be expected, and of the likely behavior of the food packaging material (Feigenbaum and Rossi, 1991). As yet, the only EU legislation covering the use of paper and paperboard in food contact materials is Directive 93/10/EEC (EEC, 1993), which covers regenerated cellulose films for food contact. Gilbert *et al.* (1994) have suggested that a list of the potential contaminants in paper and paperboard be drawn up in order to identify areas where these contaminants should be restricted (CoE-RD 32, 1994), and have underlined the need for appropriate tests for the evaluation of overall migration from coated card into olive oil.

In the United States, however, the FDA (FDA, 21 CFR 176, 1994) has established a series of extractive tests that evaluate the suitability of paper and paper-

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board for food contact. FDA extractive tests for paper and paperboard in contact with aqueous and fatty foods (21 CFR 176.170) are for currently regulated paper and paperboard adjuvants, paper and paperboard coatings, and paper and paperboard coating adjuvants. Unregulated substances for use in paper and paperboard that contact food must undergo extraction protocols as per the FDA's Recommendations for Chemistry Data for Indirect Food Additives, June 1995. The time and temperature conditions used in these tests, and the food simulant/extractant (distilled water, *n*-heptane, or 8 or 50% (v/v) ethanol), vary according to the conditions of use. Recycled paper and paperboard formed from pulp from reclaimed fiber is regulated under 21 CFR 176.260. Under section (b) (1) and (2), any paper and paperboard that bears or contains any poisonous or deleterious substance which is retained in the recovered pulp and that migrates to food is excluded from food contact use. Many companies proposing to use recycled paper and paperboard (coated and uncoated) in contact with food have chosen to submit analytical protocols to detect possible contaminants in the recycled paper and paperboard.

#### Problems with the Testing of Overall Migration from Plastics and Paperboard into Fatty Foods.

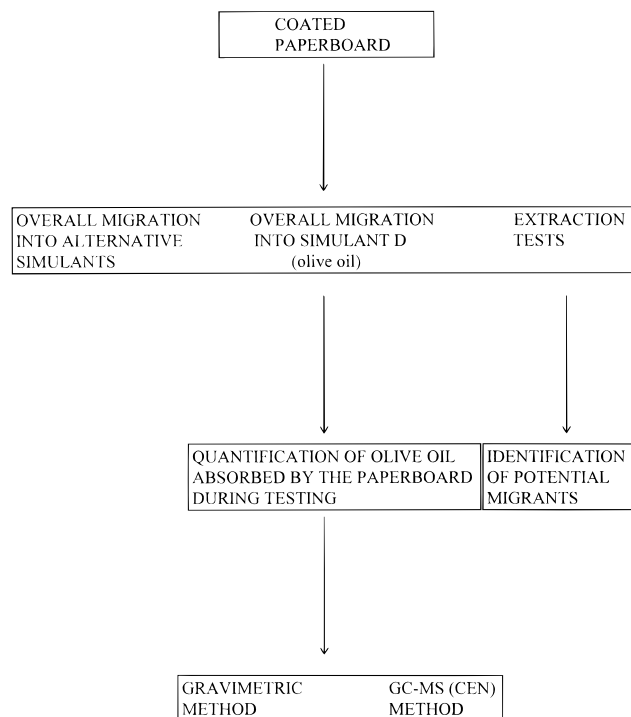
The European Committee for Standardization (CEN) has laid down procedures for evaluation of overall migration from plastic materials into food simulants (CEN, 1991a,b). For fatty food simulants such as olive oil, the CEN method is complicated by the nonvolatility of the simulant, and recourse to indirect methods of measurement is necessary, making these methods very labor and time intensive. A further complication, and one particularly relevant to the testing of paperboard, is that overall migration is calculated in terms of the loss of sample mass during the test, and so errors may be introduced if the sample moisture content is altered by contact with the olive oil (Tice, 1994). One solution to this problem is thorough vacuum-drying of the sample before each weighing (CEN, 1991a). Alternatively, especially for hygroscopic plastics, determination of the water transferred from the olive oil to the plastic may be carried out by means of Karl-Fischer titration (Castle *et al.*, 1992). In view of these problems (Tice, 1988), many investigators in this field consider that an alternative simulant to olive oil must be sought (de Kruijf *et al.*, 1983; Gramiccioni *et al.*, 1986; Piringner, 1990; Baner *et al.*, 1992; Rijk *et al.*, 1992) and existing test methods simplified (Feigenbaum *et al.*, 1994; Gilbert *et al.*, 1994; Baner *et al.*, 1994).

**Work Plan.** The work scheme followed in the present work is outlined in Figure 1.

#### EXPERIMENTAL PROCEDURES

**1. Paperboard Samples.** The packaging material evaluated was a 330  $\mu\text{m}$  thick Triplex paperboard of European quality code GT2, which contained at least 80% recycled fiber (ca. 10–15% from industrial waste and 80–85% from consumer waste). This paperboard has been certified for use in packaging for dry, fatty foodstuffs by the German authorities (BGA Recommendation XXXVI). To improve the appearance and performance of the paperboard in the final packaging application, it is subjected to the following treatments by means of offset printing.

(1) A damping solution is applied to selected areas during printing to prevent them from absorbing the ink. The solution comprises organic buffers, complexing agents, humectants, preservatives, corrosion inhibitors, polyols, glycolic ethers, and carbohydrates in 10% (v/v) aqueous 2-propanol.



**Figure 1.** General scheme followed for the evaluation of the suitability of the coated paperboard for contact with fatty foods.

(2) Printing inks comprising phenolic and aldehyde resins, vegetable oils, waxes, high-boiling mineral oils and organic pigments are applied: the internal face coming into direct contact with the foodstuff is treated with a yellow ink, and the external face is treated with this ink and a second red ink.

(3) A water-based lacquer comprising an acrylic acid-styrene copolymer, waxes, humectants, and antifoaming agents is applied to the external face.

(4) The internal face coming into direct contact with the food is coated with a film that repels oil and fat. This film is made of two formulations: (a) a water-based lacquer comprising acrylic acid-styrene copolymer, waxes, humectants, and antifoaming agents; (b) a hydroalcoholic solution based on perfluorinated compounds (50–52% (w/w) fluorine in the dry solid) that repels oil, fat, and nonpolar compounds. This product conforms to FDA 21 CFR 176.170 and BGA (teil A, XXXVI, Papiere, Kartons und Pappen für den Lebensmittelkontakt).

**2. Migration Cells.** Glass migration cells were made according to a previously published design (Simal-Gándara *et al.*, 1993). Their airtight seal was made by means of a metal bracket and two Teflon washers placed either side of the disk-shaped (12.7 cm diameter, 1.26 dm<sup>2</sup> area) paperboard sample to be tested.

**3. Fatty Food Simulants.** The foodstuff studied, French fries, is classified under Directive 85/572/EEC as a fried or roasted foodstuff, for which the recommended simulant is rectified olive oil (simulant D). The alternative fatty food simulants examined were 95% (v/v) ethanol and isooctane. The FDA Code of Federal Regulations (21 CFR 176, 1994) classifies French fries in Food Type IX, corresponding to dry solids with free fat or oil at their surface, for which the recommended extractant is *n*-heptane.

**4. Test Duration and Temperature.** French fries are typically fried at 175–180 °C, and then the excess oil is allowed to drip off before they are introduced into the packaging. During this process, the temperature of the French fries falls to approximately 80–90 °C, which is thus the real contact temperature. The fries typically remain in the package for no longer than 30 min. The test conditions were chosen to reflect these real conditions, the test with olive oil being carried out at 100 °C and lasting 30 min (EEC, 1993a). The equivalent contact conditions for isooctane are 60 °C and 30 min (Commission of the European Communities, Practical Guide N.1.,

**Table 1. Test Contact Conditions Considered in This Study for the Overall Migration Tests**

test simulant	time, min	temp, <sup>a</sup> °C
olive oil	30	100
isooctane	30	60
ethanol 95% v/v	30	60
<i>n</i> -heptane	15	49 (120 °F)

<sup>a</sup> In initial tests it was noted that the cell temperature generally fell during filling and assembly outside the oven, so it was heated to 8 °C above the prescribed temperature to compensate for this decrease before setting up the experiment.

1993); the EU does not specify equivalent contact conditions at those temperatures for 95% (v/v) ethanol, so these were carried out under the conditions used for isooctane.

The FDA regulations classify French fries in the group of foods described as hot-filled or pasteurized above 150 °F (65 °C) and set the packaging test conditions accordingly as extraction with *n*-heptane at 120 °F (49 °C) for 15 min (21 CFR, 176.170, 1994). These test conditions are summarized in Table 1. In initial tests it was noted that the cell temperature generally fell during filling and assembly outside the oven, so it was heated to 8 °C above the prescribed temperature to compensate for this decrease before setting up the experiment. A similar approach has been used in other work (Rijk and de Kruijf, 1993).

**5. Other Migration Test Requirements.** Since only the internal surface of the multilayer paperboard tested is destined for food contact, this was the surface tested. Disk-shaped samples were placed in contact with 65 mL of simulant, the corresponding area/simulant ratio being 2 dm<sup>2</sup>/100 mL, in accordance with draft CEN standards. Because the packaging has a capacity below 500 mL, the overall or global migration limit (GML) is expressed in mg/dm<sup>2</sup>, as is the case for plastics (EEC, 1990). The reduction factor to be applied to the GML is 5 (EEC, 1985) for both olive oil and alternative fatty food simulants, under both EU and US regulations (Commission of the European Communities, Practical Guide N.1., 1993; 21 CFR 176, 1994).

**6. Overall Migration Tests from Coated Paperboard into Alternative Fatty Food Simulants.** *Reagents and Apparatus.* Analytical grade absolute ethanol, isooctane, and *n*-heptane were all from Panreac. The principal apparatuses were an analytical balance measuring to within 0.1 mg; an incubator (range 30–80 °C) and a thermostatically controlled oven (range 40–250 °C); a water bath; 80 × 45 mm glass dishes; a desiccator and silica gel; and several thermometers covering the range –10 to 110 °C (±0.3 °C).

*Procedure.* The test procedure followed as closely as was possible the Draft CEN Standard test method for overall migration from plastics into aqueous or volatile fatty food simulants (CEN, 1991b). The overall migration value is determined gravimetrically. The results are the means of triplicate determinations, and have a precision of ±0.1 mg. The test was considered valid if the standard deviation of this mean was less than the analytical tolerance (1 mg/dm<sup>2</sup>) set for volatile fatty food simulants.

**7. Overall Migration Test from Coated Paperboard into Olive Oil.** *Reagents and Apparatus.* Olive oil (simulant D) conforming to the characteristics set out in CoE Directive 85/872/EEC and reagent grade (99%) *n*-pentane were used. The principal apparatuses were an analytical balance measuring to within 0.1 mg; a vacuum oven (range 30–210 °C) connected to a vacuum pump via a gas washing bottle containing silica gel; a water bath; 80 × 45 mm glass dishes; a desiccator and silica gel; a 250 mL Soxhlet extractor and 37 × 130 mm extraction thimbles; and a heating mantle.

*Procedure.* The samples of coated paperboard were conditioned by vacuum-drying at a pressure of 10 mmHg and temperature of 60 ± 5 °C for 24 h and then allowing them to cool in a desiccator for 1 h, whereupon their masses were noted. This heating–cooling cycle was repeated (five cycles were usually necessary) until the sample mass varied by less than 2.5 mg (2 mg/dm<sup>2</sup>) between successive cycles. The samples were then exposed to the simulant (CEN, 1991a), after

which they were subjected to a second set of heating–cooling cycles, identical to those just described, until their mass varied by less than the stated value between successive cycles. Any olive oil remaining in the sample was then extracted with *n*-pentane, following as closely as was possible the Draft CEN Standard test method for overall migration from plastics into olive oil (CEN, 1991a).

*1. Gravimetric Method.* The extract in *n*-pentane was transferred to a preweighed weighing dish, concentrated on a water bath at 40 ± 5 °C, and then subjected to heating–cooling cycles until it had a constant mass between successive cycles. The paperboard sample was then extracted a second time with *n*-pentane. If the mass of oil extracted was more than 5% (w/w) of that extracted in the first extraction, a third extraction was carried out. The masses for the various extractions were then summed to give the mass of oil extracted. This mass was compared with that of the residue obtained from an extract of paperboard that had not been exposed to olive oil.

*2. GC–MS Method.* This method is an adaptation of the CEN method for plastic materials and involves saponification of the oil and derivatization of the resulting fatty acids to esters, which are then determined by GC–MS.

*Reagents and Apparatus.* All reagents were of analytical grade. The internal standard was triheptadecanoin (glyceryl trimargarate), which was made up as a 2 mg/mL solution in *n*-heptane. For the saponification and esterification reactions: potassium hydroxide, as an 11.0 g/L solution in methanol, 150 g/L boron trifluoride in methanol, and anhydrous sodium sulfate. The principal apparatuses were 50 mL round-bottomed saponification flasks with reflux condenser and a gas chromatograph equipped with a 5% diphenyl- and 95% dimethylpolysiloxane-fused silica column (0.25 mm i.d. × 60 m; 0.50 μm film) and linked, by a 300 mm interface tube that entered the ionization chamber, to a mass spectrometer and a PC running software for data acquisition and treatment.

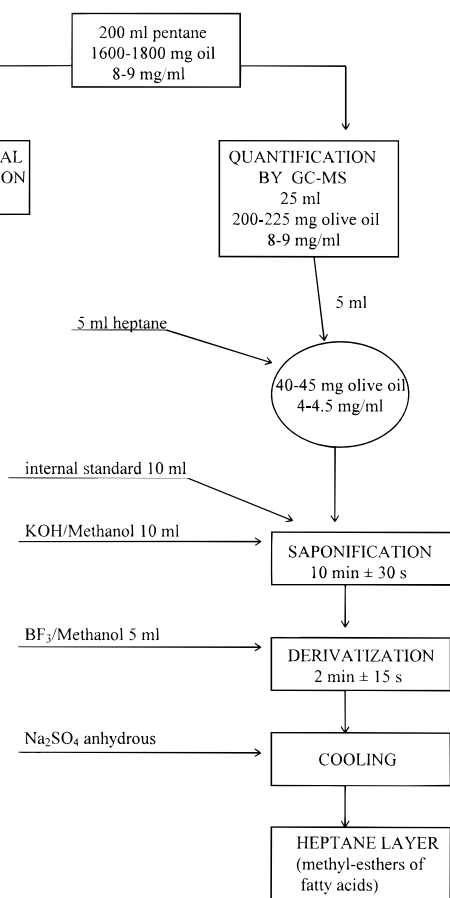
*Chromatography Conditions.* The sample (2 μL) was injected in splitless mode, opening the vent 1 min after injection (split/column flow ratio 10:1). Carrier gas was helium at a column head pressure of 160 kPa (flow rate, 0.8 mL/min). The injection temperature was 250 °C; the oven temperature was 250 °C. The interface linking the detector to the column was also maintained at 250 °C. MS detection (50–300 amu; scan time 0.9 s, interscan time 0.1 s) used electron-impact ionization at 70 eV; the ion source was held at 200 °C. Solvent delay was 5 min.

*Analysis.* Saponification and derivatization of the standards and extracts, and GC–MS determination of the extracted olive oil, followed as closely as was possible the Draft CEN test method (CEN, 1991a). GC–MS analysis was performed under the instrumental conditions described above. The amount of oil extracted was estimated from the ratio of the areas of the peaks due to the methyl esters of the C16 and C18 fatty acids to that of the C17 fatty acid (coming from the glyceryl trimargarate internal standard) by means of interpolation of a calibration line. The sequence is outlined in Figure 2.

*Expression of Results.* The GM value was calculated as the difference of the initial and final masses of the paperboard sample, less the mass of olive oil absorbed by the sample (determined in the pentane extracts). This mass was then divided by the surface area of the sample, and the mean value for three separate tests with each sample was calculated in mg/dm<sup>2</sup>, with a precision of ±0.1 mg/dm<sup>2</sup>. The test result was considered valid if the standard deviation of this mean did not exceed the analytical tolerance of 3 mg/dm<sup>2</sup>.

**8. Rapid Extraction Tests.** *Reagents and Apparatus.* Reagent grade (99%) *n*-pentane and analytical grade ethyl acetate were used. The principal apparatuses were a 250 mL Soxhlet extractor and 37 × 130 mm extraction thimbles; a heating mantle; 80 × 45 mm glass dishes; a water bath; 10 mL graduated glass tubes. Determination of the extracted materials used the GC–MS chromatograph described above (section 7, part 2).

*Chromatography Conditions.* The sample (2 μL) was injected in splitless mode, opening the vent 1 min after injection (split/column flow ratio 10:1). Carrier gas was helium at a flow rate of 0.8 mL/min. Injection temperature was 250 °C.



**Figure 2.** Scheme carried out for the comparison of the two methods used for the quantification of the extracted olive oil after the overall migration tests.

The oven temperature program was as follows: 1 min at 50 °C, increasing at 10 °C/min to 275 °C, at which it was held for 10 min. The interface linking the detector to the column was also maintained at 250 °C. MS detection (45–245 amu; scan time 1.8 s, interscan time 0.2 s) used electron-impact ionization at 70 eV. The ion source was held at 200 °C. Solvent delay was 5 min.

**Procedure.** The procedure followed was based on that of Baner *et al.* (1994). The packaging material was extracted with solvents of differing polarities in order to identify the nonvolatile organic components contributing to the GM value. *n*-Pentane was used as the representative nonpolar solvent, and ethyl acetate as representative polar solvent. Each sample was composed of three 1.26 dm<sup>2</sup> disks of recycled paperboard coated on its internal face, which were cut into strips for the purposes of extraction. One sample was extracted in a Soxhlet with 200 ± 10 mL of *n*-pentane and another with the same volume of ethyl acetate (a duplicate was run independently for each extractant solvent). The extraction lasted 10 h (±30 min), and the rate of boiling of the solvent was adjusted so that the reservoir filled and emptied at least six times hourly in the course of the test. Following extraction, the extract solution was transferred to a weighing dish and concentrated on a water bath at 40 ± 5 °C for *n*-pentane (bp 36 °C) and 85 ± 5 °C for ethyl acetate (bp 78 °C) (in the case of the ethyl acetate extract, concentration to <10 mL was prevented by precipitation of extracted material). The concentrated extract solution was transferred to a graduated glass test tube. Then, the weighing dishes were rinsed with 2 mL of the same solvent, the washings were mixed with the concentrated extract, and a sample was injected into the GC–MS. Comparison of the GC–MS trace with that for a solvent blank concentrated in the same way allowed preliminary identification of the extracted potentially migrating components.

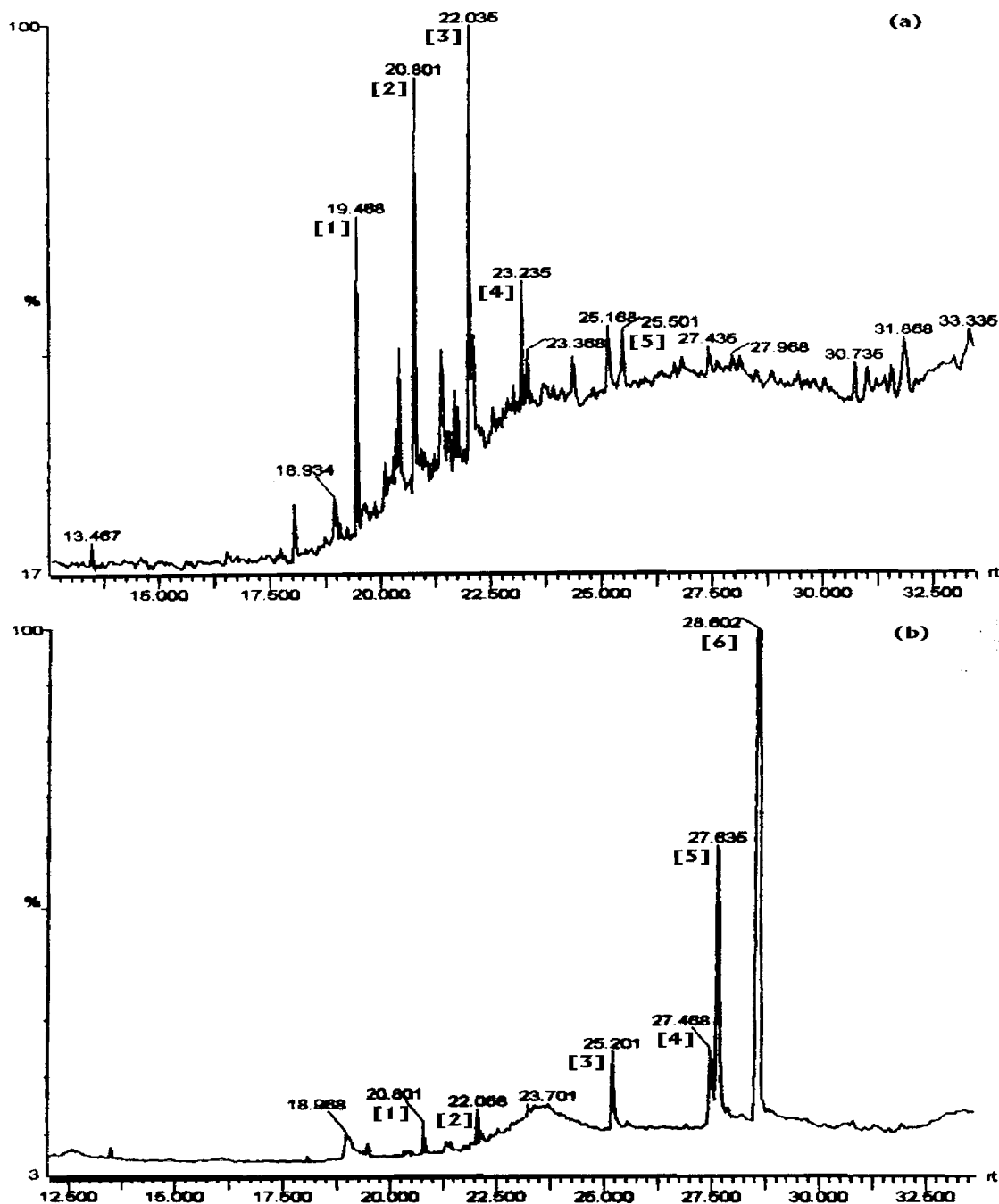
## RESULTS AND DISCUSSION

**Comparative Study of the GM Levels Obtained with Olive Oil and the Alternative Fatty Food Simulants.** After the recommended reduction factor (5 for all simulants) was applied, the mean ± standard deviation results of the overall migration tests carried out on the treated card were as follows: olive oil (simulant D)/100 °C/30 min, 3.4 ± 2.3 mg/dm<sup>2</sup> (*n* = 16); 95% (v/v) ethanol/60 °C/30 min, 1.4 ± 0.3 mg/dm<sup>2</sup> (*n* = 13); isooctane/60 °C/30 min, 0.3 ± 0.1 mg/dm<sup>2</sup> (*n* = 14); and *n*-heptane/49 °C/15 min, 0.3 ± 0.1 mg/dm<sup>2</sup> (*n* = 13). The results with pairs of simulants were compared using the Mann–Whitney *U*-test. This showed that, at the 95% significance level, the test with olive oil gave significantly higher GM values than the tests with the alternative fatty food simulants; that the 95% (v/v) ethanol gave intermediate GM values; and that the isooctane and *n*-heptane gave statistically identical GM values that were much lower than those obtained with the other simulants.

The differences between the results with the EU-recommended simulant, olive oil, and those with the alternative fatty food simulants suggests that, in the case of the latter, the use of more severe test conditions might be necessary. However, this is not recommended since the combination of alternative simulant and test conditions have been carefully selected to reflect the worst-case behavior of real foodstuffs. Alternatively, the same conditions could be used and a smaller reduction factor could be applied; for example one of around 2 in the case of 95% (v/v) ethanol would afford values similar to those obtained with olive oil. One advantage of using 95% (v/v) ethanol as simulant is that the analytical tolerance is lower (±1 as against ±3 mg/dm<sup>2</sup> with olive oil) due to its easier handling, which allows avoidance of the substrate conditioning and simulant extraction steps necessary with olive oil. With regard to the other two alternative fatty food simulants (isooctane and *n*-heptane), the use of smaller reduction factors is ruled out by the fact that the unadjusted GM values are smaller than the adjusted values obtained with olive oil. For the test with *n*-heptane, both the conditions and the reduction factor to be applied are those recommended in the FDA regulations for the testing of paper and paperboard for food contact (FDA, 21 CFR 176.170, 1994). It is noteworthy that the conditions prescribed in the FDA regulations are less severe than those applied in the EU for plastics, while the maximum amount of extractable material permitted by the FDA in paper or paperboard packaging for food contact is 0.5 mg/in<sup>2</sup>, which equates to 7.75 mg/dm<sup>2</sup> (lower than the GML of 10 mg/dm<sup>2</sup> for plastics in the EU).

**Comparison of Two Procedures for the Quantification of the Olive Oil Absorbed by the Paperboard during the Migration Test.** A paired two-tailed *t*-test was used to compare the amounts of olive oil absorbed in the migration test, as determined by the gravimetric and CEN methods, for three pairs of paperboard samples. No significant differences (*p* ≤ 0.05; 2 df) were found between the results obtained by the two methods.

**Rapid Extraction Tests.** These tests have not yet been approved by the EU. They overestimate overall migration from the packaging material because the test conditions are quite severe and extract the main components of the recycled paperboard coated on its internal face for food contact. One advantage of this is that,



**Figure 3.** Chromatogram of the substances extracted from the tested paperboard with (a) *n*-pentane and (b) ethyl acetate (see Table 2).

following identification of these compounds by GC-MS, a profile can be built up of the major potential migrants in the packaging material.

The extracts were concentrated virtually to the solubility limit of the extracted material in the extractant, in order to detect the largest number of compounds possible. For the same reason, the sensitivity and detection level of the MS was optimized by shortening the scan range as much as possible (preliminary experiments indicated the absence of  $m/z$  fragments  $>245$  amu, and so the scan range was accordingly reduced to 45–245 amu) and decreasing the spectral resolution (kept at around  $\pm 0.5$  amu). In order to avoid compromising the quality of the mass spectrum and thus making component identification more difficult at low concentrations, spectral noise was reduced by adjusting the interscan delay and scan time to longer values than

usual (0.2 and 1.8 s, respectively). Figure 3 shows the chromatograms of the *n*-pentane and ethyl acetate extracts, and Table 2 lists the compounds identified in those chromatograms. Identification of the compounds used the library of ca. 100 000 mass spectra supplied with the spectrometer, which in most cases was only able to classify the compound type. The *n*-pentane mainly extracted long-chain, saturated aliphatic hydrocarbons, while the ethyl acetate extracted compounds such as organic acids, polyols, and esters. A further step in this research would be to look for specific toxicants with more selective and sensitive techniques.

## CONCLUSIONS

**1. Suitability of the Packaging Material Studied for Fatty Food Contact.** The Draft CEN test condi-

**Table 2. List of the Substances Extracted from the Tested Paperboard with (a) *n*-Pentane and (b) Ethyl Acetate (See Figure 3)**

peak no.	<i>t<sub>R</sub></i> (min)	type of compound
(a) <i>n</i> -Pentane		
1	19.46	C20–C25 unbranched saturated aliphatic hydrocarbon (MW = 280-350)
2	20.80	C26–C27 unbranched saturated aliphatic hydrocarbon (MW = 365-380)
3	22.03	C27–C30 branched aliphatic hydrocarbon (MW = 380-425)
4	23.23	branched aliphatic hydrocarbon (chains C17-C25)
5	25.50	phthalates (MW ≈ 445)
(b) Ethyl Acetate		
1	20.80	C26–C27 unbranched saturated aliphatic hydrocarbon (MW = 365-380)
2	22.06	C27–C28 branched aliphatic hydrocarbon (MW = 380-395)
3	25.20	C16–C18 monocarboxylic acid (MW = 255-285)
4	27.46	C14–C20 polyols (MWT = 230-315)
5	27.63	C20–C24 dicarboxylic acid esters (MW = 340-395)
6	28.60	C20–C26 dicarboxylic acid esters (MW = 340-425)

tions for plastic packaging materials and the alternative fatty food simulants (95% ethanol or isooctane) afforded overall migration values for the treated recycled paperboard that are well below the GML for this type of simulants ( $10 \pm 1$  mg/dm<sup>2</sup>). Likewise, the tests using olive oil were well below the corresponding GML ( $10 \pm 3$  mg/dm<sup>2</sup>). The FDA extractive test using *n*-heptane also afforded values below the prescribed maximum limit (7.75 mg/dm<sup>2</sup>).

**2. Comparison of the GM Values for Olive Oil and Those for the Alternative Fatty Food Simulants.** The GM values obtained with olive oil as simulant are significantly higher than those obtained with the alternative fatty food simulants. By applying a smaller reduction factor (2 as against 5) to the GM value obtained with 95% ethanol, a value similar to that for olive oil was obtained. Alternatively, the use of 95% ethanol under more severe test conditions might afford GM values similar to those obtained with olive oil. The FDA test with *n*-heptane as extractant gave a significantly lower GM value, indicating that this test is less severe than the Draft CEN test.

**3. Comparison of Gravimetric and Chromatographic Methods for the Quantification of the Olive Oil Absorbed by the Packaging Material.** The gravimetric method and the chromatographic method gave statistically identical results for the amount of olive oil absorbed by the packaging. Although the gravimetric method is more straightforward, has fewer intermediate steps, and uses simple apparatus, the chromatographic method has the advantage that it quantifies the olive oil specifically and it suffers less from analytical interferences.

**4. Rapid Extraction Tests.** The results of the rapid extraction tests indicate that the specific migration tests should focus on hydrocarbons, organic acids, polyols and esters, when studying those components that are major contributors to overall migration.

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