**INTRODUCTION**

The contamination of foods with mineral oil saturated hydrocarbons (MOSH) is a common occurrence. MOSH, as well as mineral oil aromatic hydrocarbons (MOAH), derive from crude oil, with the former fraction containing linear and branched alkanes, as well as cyclic ones (naphthenes). The Joint FAO/WHO Expert Committee on Food Additives (JECFA) published a list of admissible daily intake (ADI) values for white mineral oils: the lowest value was 0.01 mg/kg for a high viscosity one. In 2009, ESFA (European Food Safety Authority) published an opinion on the use of high viscosity white mineral oils, establishing an ADI value of 12 mg/kg bw/day. A limit of 0.6 mg/kg for MOSH (from C10 to C25) contamination in foods, has been recently proposed by the German Federal Ministry of Food, Agriculture and Consumer protection (BMLV).

Heart-cutting LC-GC, with a flame ionization detector (FID), is a prime choice for the quantification of MOSH in samples, and the LC fractions, relative to the most contaminated samples, were collected off-line and then analyzed by using comprehensive two-dimensional GC-quadrupole mass spectrometry (LC-GC-qMS). The results herein reported are important; moreover, the use of LC-GC-qMS appears to be an interesting option to “see and identify” what is beneath anonymous LC-GC-FID hydrocarbon humps.

**EXPERIMENTAL**

**Samples and sample preparation**

All the sixteen baby-food samples were purchased from supermarkets located in Messina (Italy). All the samples (500 mg) were extracted for three times with hexane (1 mL each, for 15 min), and then each time filtered. After the filtrates were added together and dried under a gentle nitrogen stream. The extract was then weighed (to derive the % of extract) and then diluted to a final concentration of 25% v/v in hexane. Apples, sugar and corn starch were purchased from a supermarket located in Messina. Quantification was achieved through a calibration curve, constructed through external standardization.

**LC-GC-FID analyses**

All samples, reported in Table 1, were analyzed by using an LC-GC system (Shimadzu, Kyoto, Japan).

**GC-MS analyses**

All GC-GC applications were carried out on a Shimadzu GC/GC-MS system. The primary column, a SLB-5ms 30 m × 0.25 mm ID × 0.25 µm column, was connected to an uncoated capillary segment (1.0 m × 0.25 mm ID, used to create a double-loop). The uncoated capillary was then connected to a segment of BPX50 (50% phenyl polisilphenylene-siloxane) 1.5 m × 0.10 mm ID × 0.10 µm column (SGE). Loop-type modulation was carried out every 6 sec. MS parameters: the sample was analyzed in the full scan mode, using a mass range of 40-460 m/z; spectra generation frequency: 25 Hz; GC oven temperature program: 80°C to 360°C at 10°C/min. Carrier gas, helium, was supplied at an initial pressure of 250 kPa (constant linear velocity). Injection temperature: 350°C. Injection mode and volume: split (1:10), 4 µL. Data were collected by the GCMS Solution software (Shimadzu), bidimensional visualization was carried out by using the ChromoSquare v.1.5 software (Shimadzu Europe, Duisburg, Germany).

**RESULTS AND DISCUSSION**

Twelve homogenized solid baby foods, containing either meat or fish (and vegetable oil), were subjected to analysis (Table 1). The vegetable oil used as ingredient was, in all cases, sunflower oil. As can be observed in Table 1, the meat and fish baby foods were all contaminated. Also reported in the table are MOSH concentrations considering the upper alkane boundary (MOSH up to C25). After quantification, the MOSH “humps” were subjected to a qualitative investigation. A four-dimension off-line LC-GC-qMS experiment was carried out as follows: the 175-µL MOSH fraction was collected, dried, and then the residue was solubilized in 10 µL of hexane. The LC-qPolarGC-polarGC-qMS results are shown in Figure 1 and 2 (most contaminated samples). The zones defined by the ellipses in Figure 1, show the compounds contained in the first and second hump, namely n-alkanes, branched alkanes (continuous line between the n-alkanes) and cyclic alkanes (continuous line above the linear and branched alkanes). A mass spectrum derived from the first “cyclolokzane” is shown in Figure 1. The “cyclics” were identified on the basis of i) bidimensional chromatogram location, and ii) information reported in the literature.

At this point, it was decided to extend the study to fruit-based baby foods. Four samples were subjected to analysis, and found to contain MOSH concentrations in the range 0.3-10.5 mg/kg (Table 1). As a consequence of such a finding, an apple baby food was lab-produced using the ingredients reported on the jar label, and then analyzed; the LC-GC result indicated the presence of mineral oil saturated hydrocarbons. The necessity arose, at this point, to analyze each ingredient (apples, sugar and starch): a MOSH contamination was pinpointed in the commercial corn starch (7.3 mg/kg) and sugar (4 mg/kg). All the baby foods analyzed in the present investigation were found to be MOSH contaminated, with the levels observed not to be considered as low (average value: 3.8 mg/kg). It is clear that the origin of the MOSH contamination observed, in all samples, cannot be exactly pinpointed, though the vegetable oil probably plays a role. The hydrocarbon molecular-weight range also varied, meaning that the contamination most certainly came from different sources.

**ACKNOWLEDGMENTS**

The Project was funded by the “Italian Ministry for the University and Research (MIUR)” within the FIRB “Future in Research” Program. Project n. RBFR09R80K “Advanced analytical techniques for the analysis of contaminants in food products.”