

Development and validation of an HPLC-GC-FID analytical method for the determination of mineral oils (MOSH and MOAH) in edible oils.

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Mineral oils hydrocarbons (MOH) are a class of compounds constituted by a complex mixture derived from crude oil, mainly composed by mineral oil saturated hydrocarbons (MOSH – open and branched chains or cyclic hydrocarbons) and mineral oil aromatic hydrocarbons (MOAH – mono or polycyclic aromatic hydrocarbons). These compounds are not naturally present in food like edible oils, being incorporated into them mostly by bad manufacturing practices. This has become a concern because of their possible carcinogenicity and liver inflammatory potential. The official method for quantification of MOSH and MOAH (CEN EN 16995:2017) allows users to reach a limit of quantification of 10 mg/Kg, for both MOSH and MOAH. This work describes the development of an improvement in the official method and method validation to reduce the limit of quantification and quantify amounts of MOSH and MOAH in edible oils in the concentration of 1 mg/Kg, using a sample cleanup previous to sample preparation in an automated system and high-performance liquid chromatography coupled to gas chromatography and flame ionization detection (HPLC-GC-FID). The separation of MOSH and MOAH fractions in HPLC was achieved using an Allure Silica column at room temperature using a gradient between hexane and dichloromethane. Each fraction was separated in a MXT®5 column using a heating ramp in the GC and detected by flame ionization detection. The fractions were quantified using internal standard. Reference materials were used to evaluate precision, trueness and uncertainty of the method. A limit of quantification of 0.6 mg/Kg was achieved for both MOSH and MOAH. Repeatability and Intermediate reproducibility were below 20% with recoveries between 70% and 120% for MOSH and MOAH. The results showed that the method fits for its purpose. To our knowledge, this is the first work developed describing the validation and quantification of MOSH and MOAH in edible oils in Brazil.

Keywords: fats and oils contaminants; method validation; mineral lubricants.