Determination of Therminol 66 Heat Transfer Fluid in Palm and Coconut Oils

ABSTRACT

A semiquantitative method for determination of low levels of Therminol 66 heat transfer fluid in plam and coconut oils is described. After separation of the unsaponifiable matter in the triglyceride oil, IR analysis is used to determine the fluid level utilizing representative peaks at 700 and 760 cm^{-1}.

INTRODUCTION

Therminol 66 is a heat transfer fluid manufactured by Monsanto and used in steam deodorizers within the oils and fats industry. Chemically, it is a hydrogenated aromatic mixture which consists of modified terpenyls (ortho- and para-terphenyls) (1).

Tests conducted by Monsanto show that the single oral LD_{50} to albino rats is 10.2 g/kg, and the single dermal LD_{50} to albino rabbits is ca. 6.8 g/kg. Subacute feeding studies conducted with albino rats for 3 months at dietary levels up to 1000 ppm produced no significant lesions upon histopathological examination (2).

Even in view of these results, we believe that a rapid method for detection of low levels in edible oils is needed. Work by Monsanto, detailed in a private communication, which utilized a gas liquid chromatograph (GLC) equipped with a flame ionization detector and a 100′ SE-52 SCOT capillary column gave acceptable results at 0.5% Therminol 66 in edible oil. Reaction of the oil with BSA (NO-bis-trimethylsilyl-acetamide) to form the trimethylsilyl ether derivative was necessary prior to injection. Since this method requires special sophisticated equipment, its utility is limited.

The method developed in our laboratory is simple, requires less sophisticated equipment, and is able to detect this heat transfer fluid in edible oils at 0.01% (100 ppm). The two oils utilized in our study were palm oil and coconut oil; however, we believe this method to be applicable to all naturally occurring triglyceride oils.

The heat transfer fluid is not saponifiable and may be concentrated in the unsaponifiable portion (0.5-1.0%) of an oil. On this basis, the relative concentration of 0.01% becomes ca. 1-2% based upon the recovered unsaponifiable matter. At this level, IR detection becomes feasible. An IR spectrophotometer is generally available in most laboratories.

EXPERIMENTAL PROCEDURES

Materials

Crude palm and coconut oils were obtained from shipments of newly arrived oil and were analyzed to ensure identity. Therminol 66 obtained from Monsanto was added to these oils in concentrations of 0.01-1.0%.

Procedure

AOCS method Ca 6a-40(3) was used to separate the unsaponifiable portion of the oils. After determination of the total unsaponifiable matter, the minimum quantity of petroleum ether necessary to redissolve the unsaponifiable matter was added. A drop of this mixture was placed on the surface of a flat salt cell and the petroleum ether evaporated with a gentle stream of dry nitrogen. The cell was closed and placed in a cell holder and then into the IR spectrophotometer. To obtain well defined peaks at 700 and 760 cm^{-1}, a reference peak at 2870 cm^{-1} was used. Prior to full scanning, this peak ht was adjusted, by sample size, to provide an absorption of 90% minimum. At this absorption, well defined peaks in the detection range were

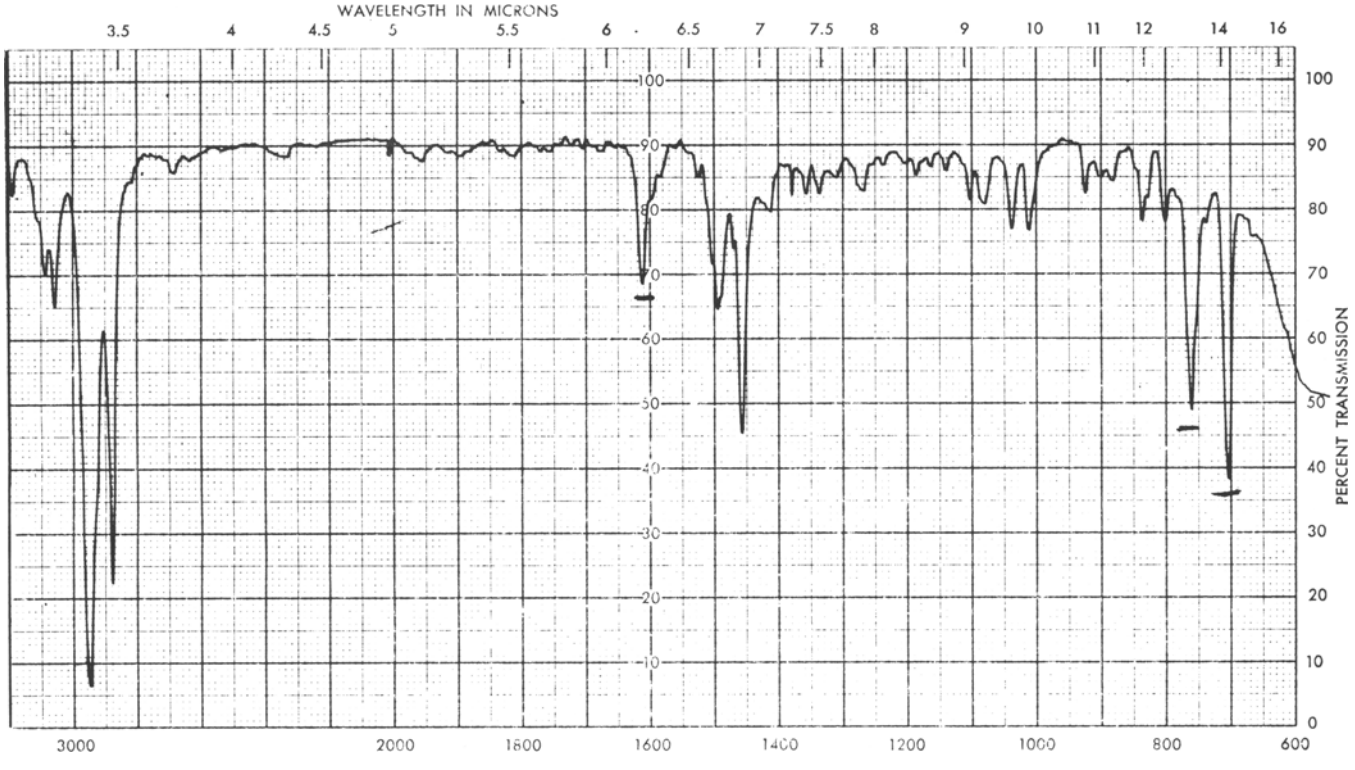

FIG. 1. Therminol 66 liquid between salts.

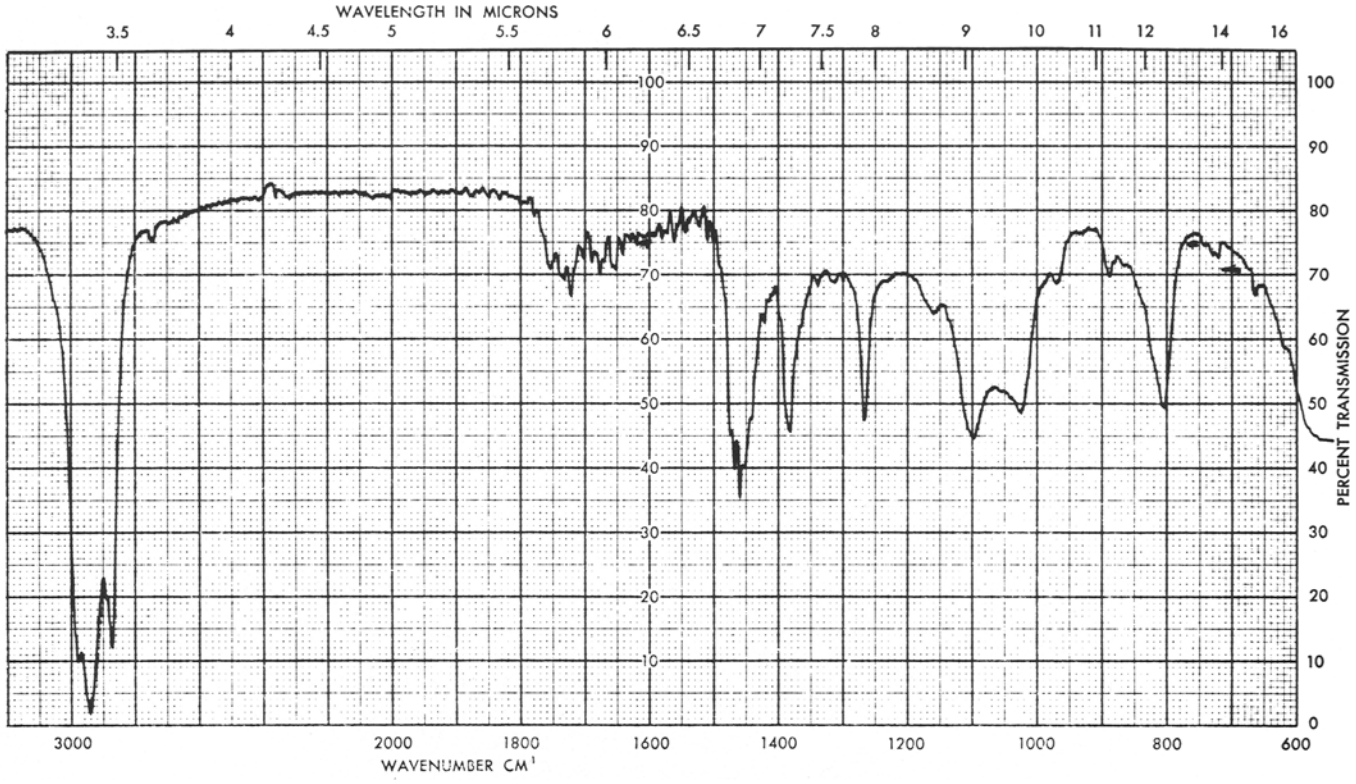

FIG. 2. Unsaponifiable matter from crude palm oil. Film between salts.

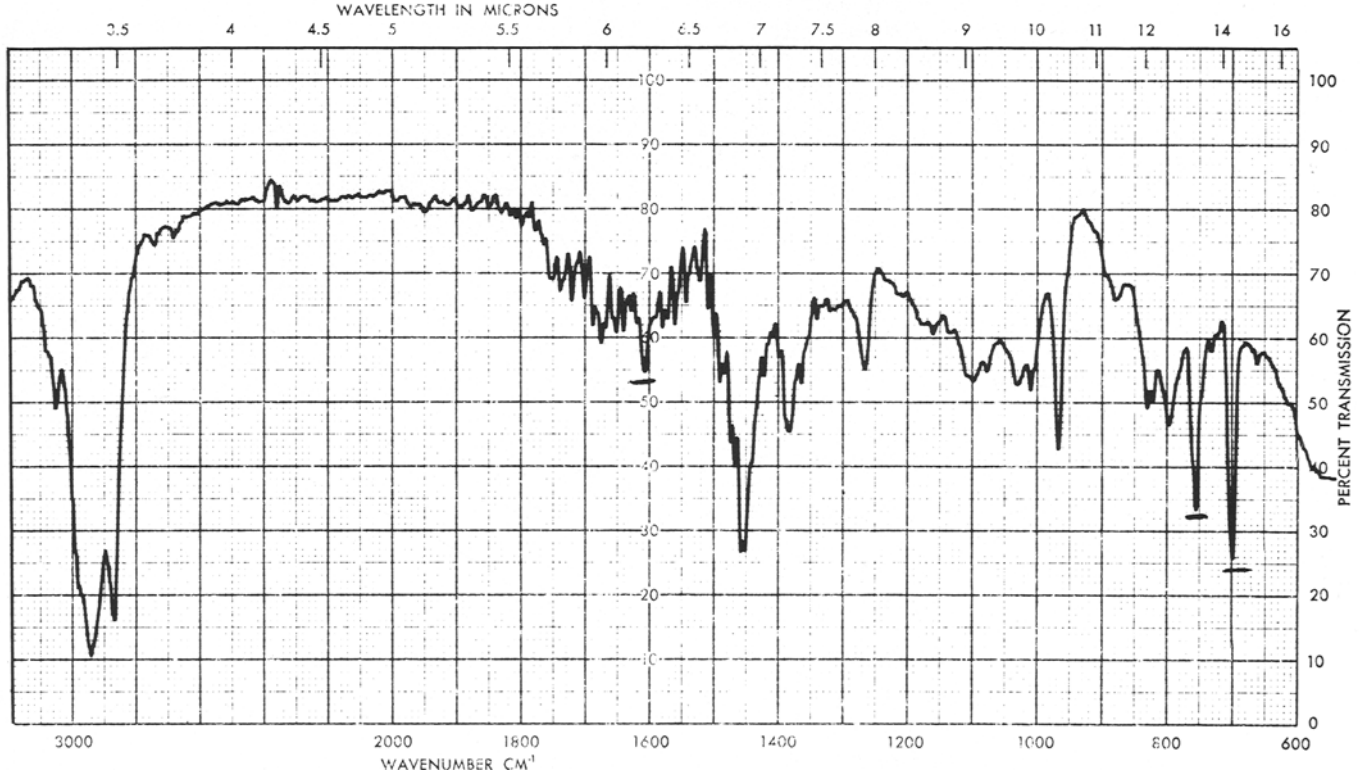

FIG. 3. Unsaponifiable matter from 0.1% Therminol 66 in crude palm oil. Film between salts.

obtained, even at 0.01%. By preparation of spectra at various levels of Therminol 66 in the oil and inspection of peak ht a semiquantitative determination can be made.

Analytical Instrument

A Beckman IR-33 spectrophotometer equipped with standard flat NaCl windows was used for the analysis. No spacers were used with these windows.

RESULTS AND DISCUSSION

The IR spectra utilizing crude palm oil are presented in Figures 1-4. Crude oil was selected for the work, since it contains a higher level of unsaponifiable matter then refined or deodorized oil. It can be seen from Figure 4, that even at levels of 0.01% in the initial crude oil, well defined peaks at 700 and 760 cm^{-1} are visible. Contamination at

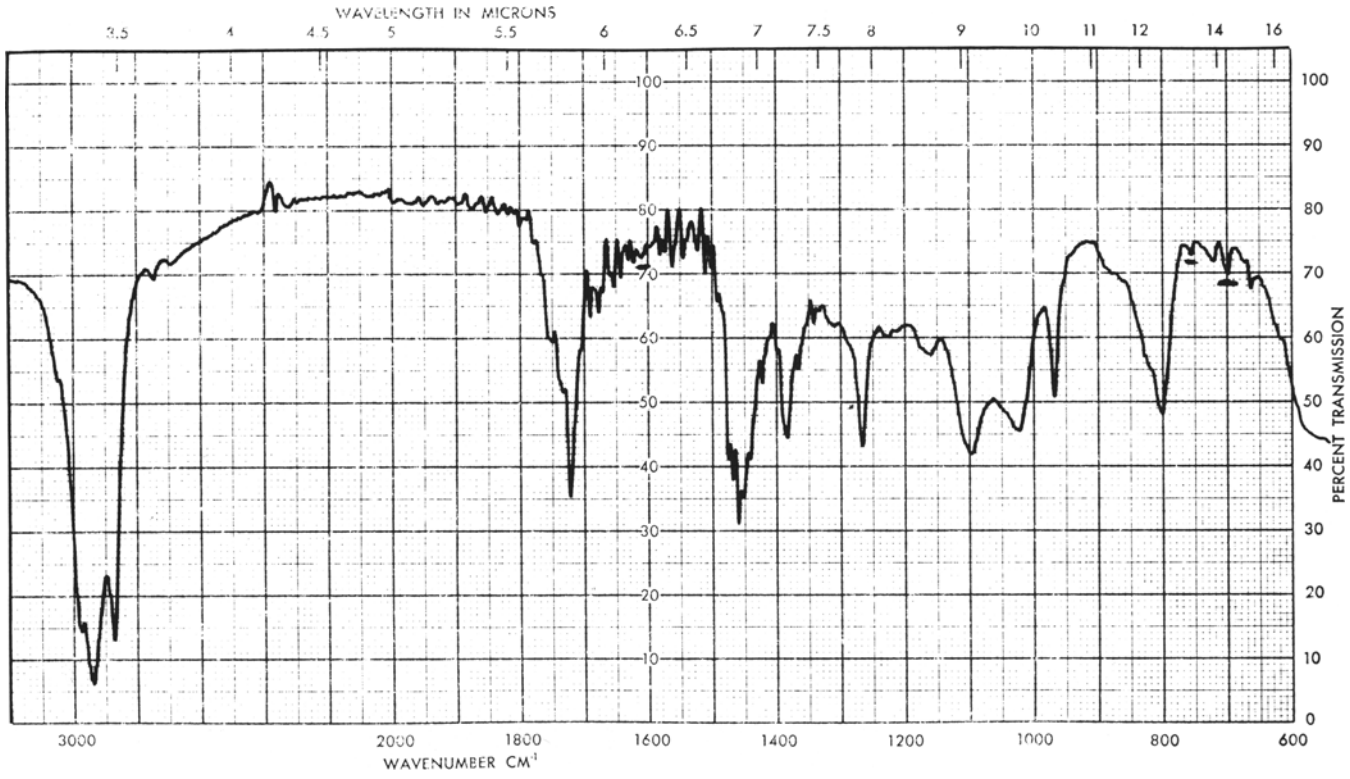

FIG. 4. Unsaponifiable matter from 0.01% Therminol 66 in crude palm oil. Film between salts.

levels between 0.01-0.10% can be approximated by inspection of peak hts and comparison with standard spectra.

To determine whether partially oxidized and polymerized Therminol 66 might affect the results, fluid which had been in use for 5 months at temperatures of 600 F was obtained and added to palm oil at a level of 0.1%. Therminol 66 was degraded further in an open beaker on a hot plate to a product insoluble in palm oil.

The partially oxidized Therminol 66, when recovered in the unsaponifiable portion, gave results similar to that of the fresh fluids.

Since the fully degraded fluid was insoluble, no further work was attempted on this mixture.

It was suggested that substituted aromatic compounds, such as Xylene, might interfere with this method of analysis. Generally, materials of this sort are not used in any edible oil plant; however, this point was investigated. A mixture of 95% palm oil and 5% xylene gave a small peak at 765 cm^{-1}. When 0.1% xylene was added to palm oil and the unsaponifiable recovered, however, only a poorly defined peak at 700 cm^{-1} was observed.

JACK G. MARCUS
The Theobald Industries
Harrison, N.J. 07029

ACKNOWLEDGMENTS

Analyses of unsaponifiable matter and IR spectra were performed mainly by J. Dempsey. R. Hooker, Jr., assisted in these analyses. J. Pullman, Monsanto, provided valuable feedback and suggestions on the method.

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[Received April 29, 1974]

Cucurbit Seeds: III. Ultrastructure of Quiescent Storage Tissues

ABSTRACT

Electron-microscopic examinations of seeds from wild xerophilous cucurbits revealed that their storage tissues ultrastructurally resembled those of commercially important oilseeds such as castor, cotton, peanut, soy, and tung.

INTRODUCTION

A resurgence of interest is developing in the uses of cucurbits, particularly wild, xerophilous species, as sources