

FIG. 2. Experimental deodorizing apparatus. (A) Vacuum pump, (B) manometer, (C) crushed ice, (D) 300 ml flask, (E) water cooler, (F) 2000 ml flask, (G) sample oil, (H) mantle heater (I) thermometer, (J) electric heater, (K) micropump for water, and (L) distilled water.

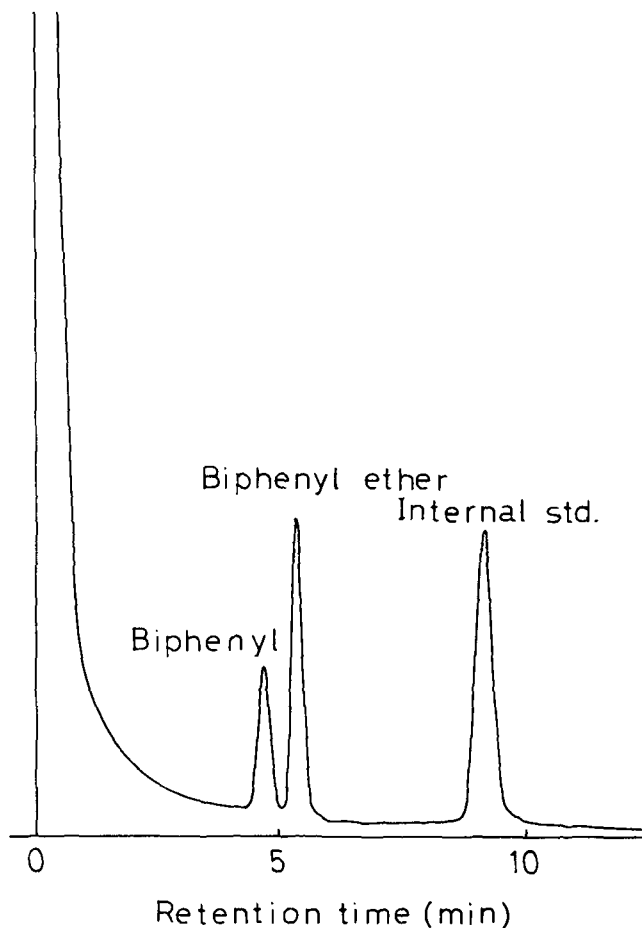


FIG. 3. Gas chromatogram for standard Dowtherm A.

contain less than 10 ppm, recovery ratios of rapeseed oil containing 5 ppm and 1 ppm Dowtherm A were examined.

Detection of Dowtherm A in Secondary Products

Mayonnaise (oil content 70%), salad dressing (oil 40%), and french dressing (oil 40%) using rapeseed oil containing 10 ppm Dowtherm A were produced experimentally by the same formulas and procedures as commercial products, and the recovery ratios determined.

Detection of KSK Oil 260

KSK Oil was analyzed by the same procedure as Dowtherm A.

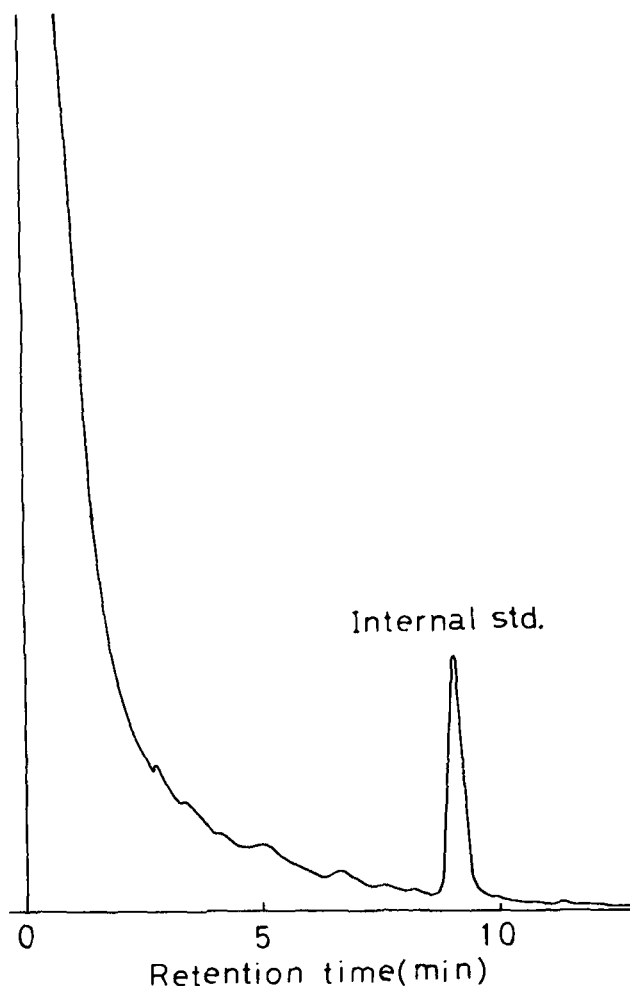


FIG. 4. Gas chromatogram for control rapeseed oil.

Analysis of Contaminated Oil

Seventeen lots of contaminated rapeseed oil stored in our laboratory and 42 samples of mayonnaise using this rapeseed oil were analyzed by the above procedure.

Evaporation of Heat Transfer Media under Deodorizing Conditions

The evaporation of heat transfer media under the usual deodorizing condition was examined, using a pilot deodorizing apparatus shown in Figure 2. The deodorizing conditions were as follows: temperature, 230 C; vacuum, 10 mm/Hg; injected steam, 1 ml/min as water; sample oil, 500 g; concentration of heat transfer media in sample oil, 50 ppm; and deodorizing time, 3, 7, 10, 15, 30, 45, and 60 min.

RESULTS AND DISCUSSION

The gas chromatograms for standard Dowtherm A, control rapeseed oil, and rapeseed oil containing 1 ppm Dowtherm A are shown in Figures 3-5. Recovery ratios of Dowtherm A from rapeseed oil containing 10 ppm are shown in Table II. Recovery ratio of more than 90% was obtained by distillation for 120 min, which was much longer than the time required for determination of biphenyl in orange as reported by Hayashi, et al. (1).

It was assumed that the difference was caused by the fact that the heat transfer media in our study were dissolved in oil. This made them more difficult to remove than the biphenyl in oranges which was emulsified in water. An increase in the quantity of sample oil to obtain a higher detection limit was not suitable as a quantitative

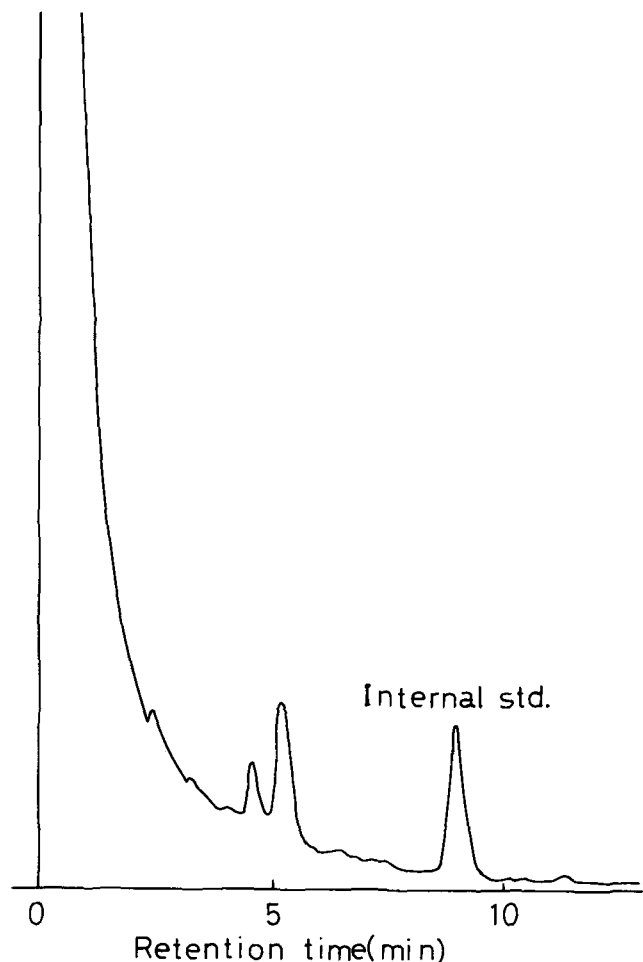


FIG. 5. Gas chromatogram for rapeseed oil with 1 ppm of Dowtherm A.

method, because the recovery ratio decreased. Recovery ratios more than 90% also were obtained from the oils containing 5 and 1 ppm heat transfer media, and the detection limit of this method seemed to be 0.2 ppm or less, considering the ht of the peak on the gas chromatogram. This method was used as a practical analytical procedure.

Recovery ratios of Dowtherm A in secondary products are shown in Table III. The recovery ratio from the products containing rapeseed oil was similar to that obtained from the oil. From a french dressing containing several kinds of spice oil, many interfering peaks were observed on the gas chromatogram, but little trouble was encountered, because these peaks appeared earlier than the peaks of the heat transfer media.

The gas chromatogram of standard KSK Oil 260 is shown in Figure 6. The peaks on the chromatogram of KSK Oil 260 were smaller than those produced by Dowtherm A,

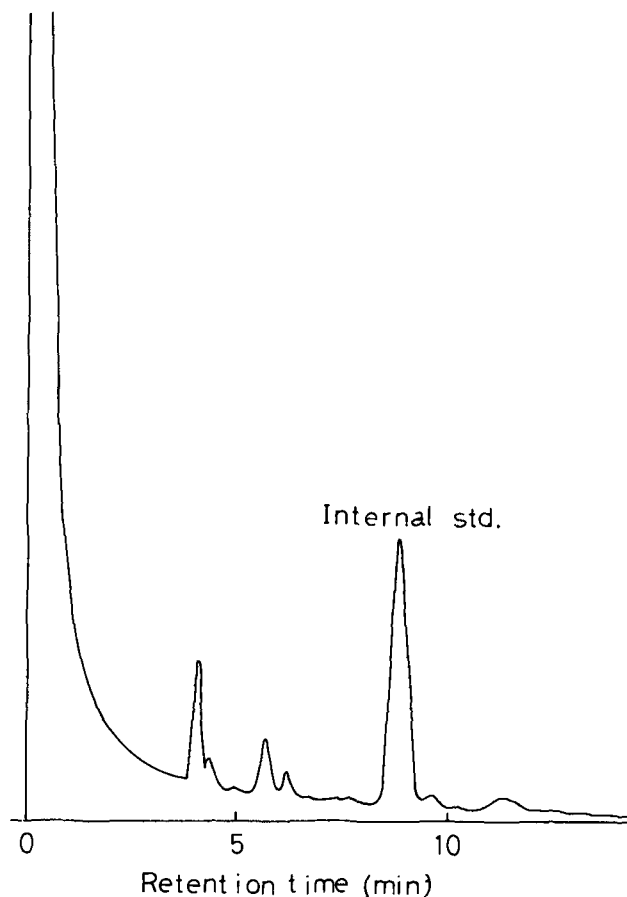


FIG. 6. Gas chromatogram for standard KSK Oil 260.

so the detection limit would seem to be 0.5 ppm or less. The recovery ratio of KSK Oil 260, 87.6%, was less than that of Dowtherm A.

Takagi and Aoyama (4) stated that the analysis of biphenyl by distillation could be performed with relative ease, even if emulsifiers were present in the sample, and, in addition, there was little interference from reagents or solvents. This was found to be true in our study in which, not only biphenyl, but also other components were analyzed.

In spite of the fact that a pin hole actually was observed in the coil of the second tray of the deodorizer of the refinery (Fig. 7) and the heat transfer media must have contaminated the oil, no heat transfer medium was detected in either the sample oil stored in our laboratory or the products made from this oil. The same result was reported by other laboratories which analyzed other lots of this oil by different methods. The evaporation of heat transfer media under deodorizing condition was examined. The results are shown in Table IV. No heat transfer medium was detected in oil containing added 50 ppm of the media and then deodorized experimentally for 15 min at 230 C and 10 mm/Hg. This treatment is milder than the actual conditions in the refinery. In the plant deodorizer, the heat

TABLE II

Percent Recovery of Dowtherm A from Rapeseed Oil Containing 10 ppm

Wt (g)	Distillation time (min)					
	15	30	60	90	120	180
10	33.5	45.9	67.9	83.1	91.8	93.1
10 ^a	---	---	---	---	94.0	---
10 ^b	---	---	---	---	90.9	---
25	---	---	---	---	54.8	---
50	---	---	---	---	35.0	---

^a5 ppm.

^b1 ppm.

TABLE III

Recovery Ratio of Dowtherm A from Secondary Products^a

Products	Recovery ratio (%)
Mayonnaise (oil 70%)	87.8
Salad dressing (oil 40%)	93.5
French dressing (oil 40%) A	92.4
French dressing (oil 40%) B	92.9

^aQuantity of sample: 10 g; distillation time: 120 min; concentration of Dowtherm A in sample oil: 10 ppm.

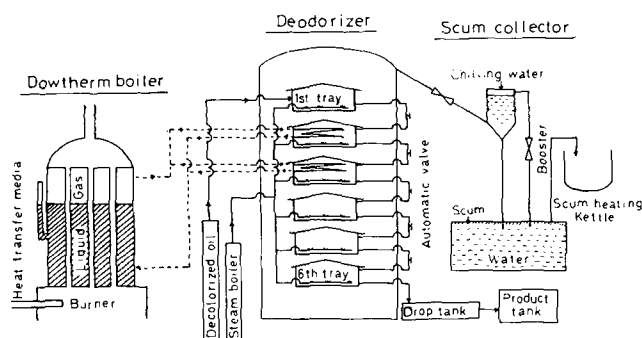


FIG. 7. Deodorizing process in the refinery. Volume of Dowtherm boiler, 1170 liter; volume of heat transfer media, 800 liter. Temperature of heat transfer media gas, 280-300 C; temperature of oil at first tray, 150 C; temperature of oil at second-fifth trays, 270-280 C. Vacuum in deodorizer, 2-4 mm/Hg. Volume of water, each 400 liter. Holding time in each tray, 16-20 min.

transfer media contaminated rapeseed oil in the second tray was heated to 270-280 C at a vacuum of 2-4 mm/Hg, and the temperature and vacuum were held for ca. 70 min until the oil was discharged into the sixth tray. Thus, the reason no heat transfer medium was detected in the oil samples

TABLE IV

Evaporation of Heat Transfer Media under Deodorizing Condition					
Retained heat transfer media (ppm) ^a	Deodorizing time (min)				
	0	3	7	10	15
	45.2	8.3	2.9	1.0	---

^aRecovery ratio is not considered.

apparently was due to sufficient heat treatment in the deodorizer to remove the contaminants effectively.

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