U.D.C. 615.733.07 : 545.821

23. Naofumi Ōi : Analyses of Drugs and Chemicals by Infrared Absorption Spectra. I. Quantitative Analyses of Santonin, Thymol, Tetrachloroethylene, and Ascaridol in Mixed Drugs.

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The quantitative analyses of a mixture of santonin, thymol, tetrachloroethylene, and ascaridol (main component in chenopodium oil) always presented a difficult problem. As the procedure of analysis by common chemical method is complicated and it takes a long time and a large amount of samples, attempt was made to assay the four components simultaneously by means of infrared absorption spectrophotometry.

The infrared absorption spectra of santonin, thymol, tetrachloroethylene, ascaridol, and chenopodium oil in carbon disulfide are shown in Figs. 1 to 5.

As can be seen from these curves, santonin has a strong band at 5.56 μ, thymol at 2.78 μ, tetrachloroethylene at 12.90 μ, and ascaridol in chenopodium oil at 14.42 μ. These were chosen as the analytical key bands, because there is a minimum of absorption due to other three components.

Maruyama[1] chose the band at 10.69 μ for the analytical key band of ascaridol in chenopodium oil, but in this case the other three components also have some absorption in this region and it seemed more appropriate to choose the band at 14.42 μ.

The spectra of components except ascaridol in chenopodium oil, are shown in Fig. 6 by differential method and it is very convenient for the analysis that they have no absorption at 5.56, 2.78, 12.90, or 14.42 μ.

From preliminary experimentation, it was found that the most suitable concentration was in the range of 0.5~1.5 mg./cc. for santonin, 2~6 mg./cc. for thymol, 4~8 mg./cc. for tetrachloroethylene, and 4~8 mg./cc. for ascaridol in carbon disulfide.

Absorbancy was then plotted against concentration for each of the four components at 5.56, 2.78, 12.90, and 14.42 μ. As can be seen from Figs. 7 to 10, the relationship was found to be linear in above concentrations.

In such a solution, santonin and thymol form a hydrogen bond and the absorbancy at their key bands decreases in a mixture, as shown in Figs. 7 and 8 (dotted line), but in a dilute solution their effect is very small and very low concentrations can be used.

In analysis the following method was used : 520 mg. of a sample (equivalent to 2 capsules) is accurately weighed, transferred quantitatively to a 10-cc. measuring flask, and dissolved in exactly 10 cc. of carbon disulfide. Using this solution (Solution A) as

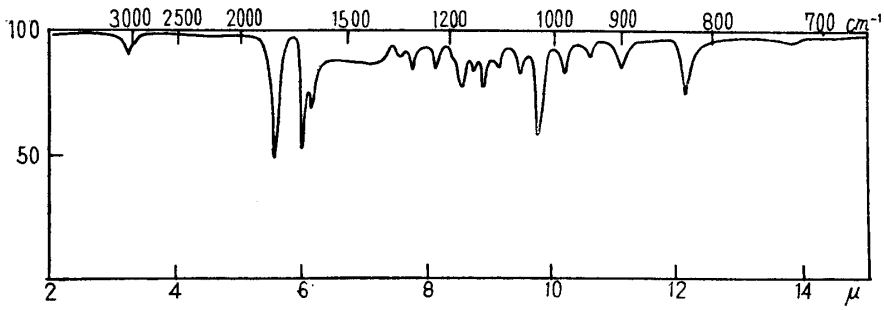

Fig. 1. Infrared Spectrum of Santonin

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1) M. Maruyama : J. Pharm. Soc. Japan, **72**, 927(1952).

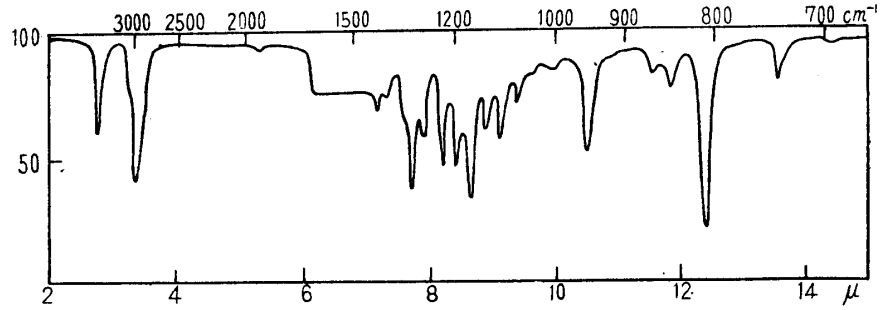

Fig. 2. Infrared Spectrum of Thymol

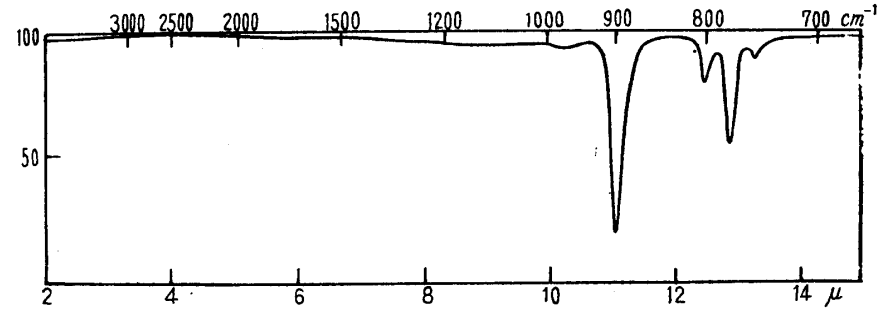

Fig. 3. Infrared Spectrum of Tetrachloroethylene

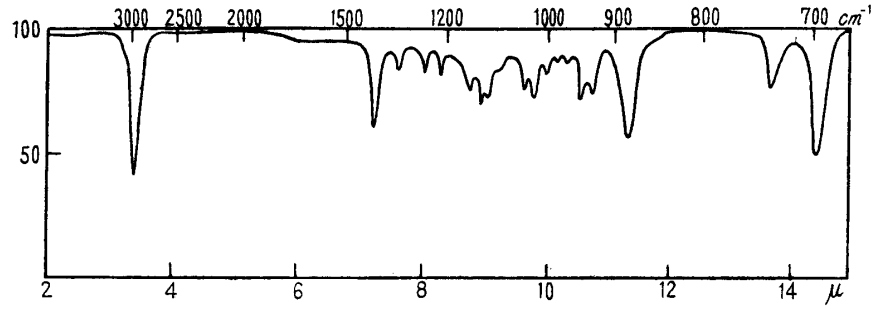

Fig. 4. Infrared Spectrum of Ascaridol

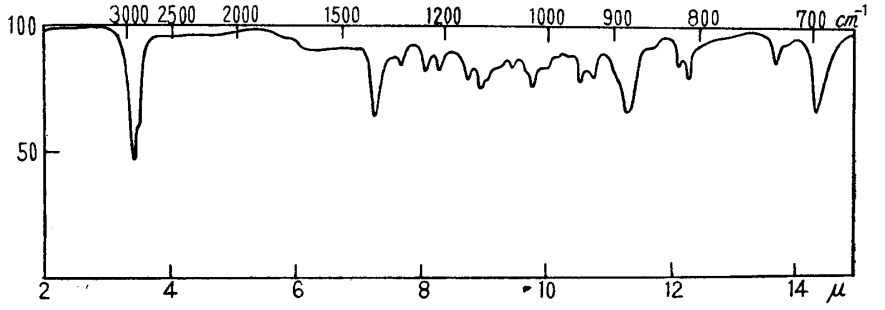

Fig. 5. Infrared Spectrum of Chenopodium Oil

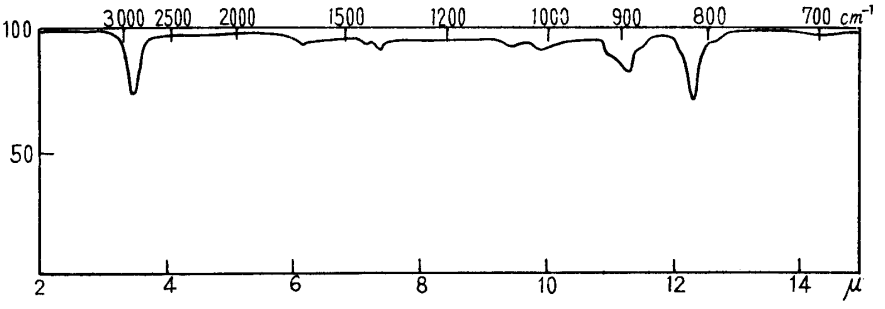

Fig. 6. Infrared Spectrum of Chenopodium Oil (Differential Method)

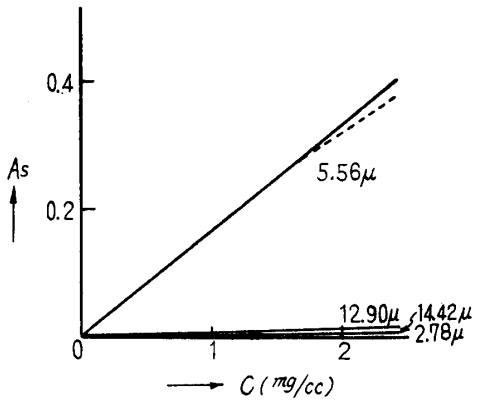

Fig. 7. Calibration Curve for Santonin

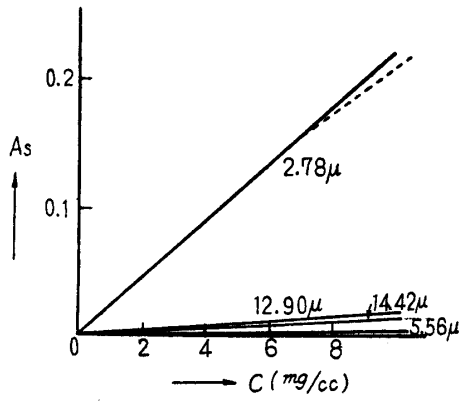

Fig. 8. Calibration Curve for Thymol

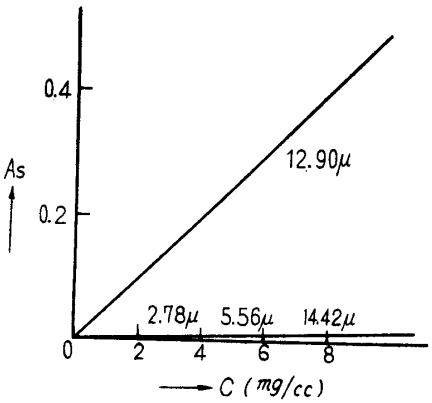

Fig. 9. Calibration Curve for Tetrachloroethylene

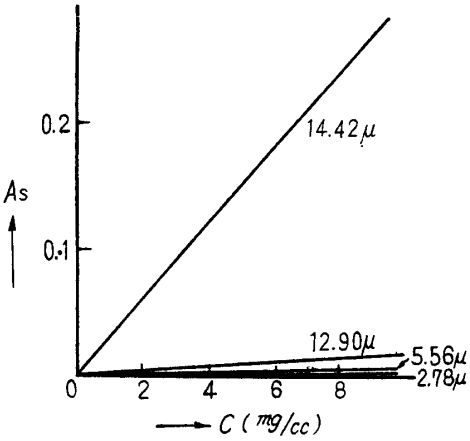

Fig. 10. Calibration Curve for Ascaridol

Table I. Analyses of Weighed Mixtures

Sample No.	Santonin Weighed (mg.)	Santonin Found (mg.)	Santonin Recovery (%)	Thymol Weighed (mg.)	Thymol Found (mg.)	Thymol Recovery (%)
1	18.0	18.25	101.4	47.0	45.7	97.2
2	18.0	17.6	97.8	47.0	46.0	97.9
3	18.0	17.8	98.9	47.0	46.8	99.6
4	18.0	17.6	97.8	47.0	46.0	97.9
5	18.0	18.0	100.0	47.0	47.7	101.5
6	18.0	17.45	97.0	47.0	46.8	99.6
7	18.0	18.1	100.6	47.0	47.2	100.4
	mean deviation		1.5%			1.4%
	max. deviation		3.0%			2.8%

Sample No.	Tetrachloroethylene Weighed (mg.)	Tetrachloroethylene Found (mg.)	Tetrachloroethylene Recovery (%)	Ascaridol Weighed (mg-)	Ascaridol Found (mg.)	Ascaridol Recovery (%)
1	150.0	150.0	100.0	28.8	28.3	98.3
2	150.0	147.6	98.4	28.8	29.2	101.4
3	150.0	152.0	101.3	28.8	28.8	100.0
4	150.0	149.1	99.4	28.8	29.0	100.7
5	150.0	149.4	99.6	28.8	29.0	100.7
6	150.0	148.4	98.9	28.8	28.5	99.0
7	150.0	150.5	100.3	28.8	29.3	101.7
	mean deviation		0.8%			1.0%
	max. deviation		1.6%			1.7%

Table II. Analyses of Commercial Capsules

Sample No.	Santonin Theoretical (mg.)	Found (mg.)	Label claim (%)	Thymol Theoretical (mg.)	Found (mg.)	Label claim (%)
1	18.0	18.1	100.6	47.0	46.0	97.9
2	18.0	18.5	102.8	47.0	46.0	97.9
3	18.0	18.5	102.8	47.0	47.2	100.4

Sample No.	Tetrachloroethylene Theoretical (mg.)	Found (mg.)	Label claim (%)	Ascaridol Theoretical (mg.)	Found (mg.)	Label claim (%)
1	150	141	94.0	—	31.7	—
2	150	139	92.7	—	33.2	—
3	150	139	92.7	—	32.7	—

the standard, solutions of 1/2 and 1/4 concentrations (solutions B and C) are prepared. Compensation cell is filled with carbon disulfide and the sample cells with these three solutions and absorbancies at 2.78 μ (solution B), 5.56 μ, 12.90 μ (solution C) and 14.42 μ (solution A) are read.

From the above calibration curves, using a graphical method, four components can be analyzed easily. This manipulation is simple and rapid to perform and a determination can be completed within one hour.

In Table I are listed a series of determinations on known mixtures. The mean deviations are within ±2% and maximum deviation is within ±3%.

In Table II are listed a series of determination on commercial preparations of santonin, thymol, tetrachloroethylene, and ascaridol.

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Experimental

The instrument used was a Perkin-Elmer Model 21, automatic recording spectrophotometer equipped with NaCl prism. The two cells employed were 0.5 mm. thick with NaCl windows.

Four standard substances (santonin, thymol, tetrachloroethylene, and chenopodium oil) for the analysis were commercial products, and the standard ascaridol was obtained by distillation of chenopodium oil as $b.p_{1.5}$ 79~80°, n_D^{25} 1.4715, d_4^{20} 1.0115. *Anal.* Calcd. for $C_{10}H_{16}O_2$: C, 71.38; H, 9.58. Found: C, 71.24; H, 9.35.

Summary

An infrared method has been developed for the simultaneous determination of santonin, thymol, tetrachloroethylene, and ascaridol (main component of chenopodium oil) in a mixture. The manipulations are simple and rapid to perform, and accuracy and precision of analyses are good.

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