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NOTES ON THE DETERMINATION OF THE CINEOL CONTENT OF EUCALYPTUS OILS.

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IN Schimmel's semi-annual report (1908) some notes are recorded on the resorcinol method for the determination of cineol. Every worker with this method soon realises its unreliability when used on the virgin or ordinary rectified oils, and it is no uncommon thing to meet with samples of oil, containing no cineol, which nevertheless give quite considerable readings. Recently I had occasion to work with a pure white E. Dives oil, free from cineol, which showed, however, 32 per cent. by using the resorcinol test.

The rectification suggested by Schimmel—viz., using for the test only that portion of the oil distilling between 170° to 190° C.—seems to remove this difficulty. Notwithstanding this, the results are always slightly high. This is perhaps negligible when the percentage does not exceed 50; for higher percentages I have found that the difference between the apparent and true percentages warrant some suggestions for overcoming the difficulty, which seems to be due to some retention by the resorcinol solution.

By using a warm 55 per cent. solution of resorcinol instead of a 50 per cent., better results are obtained. It is well to remember that in very cold weather solutions of over 50 per cent. strength are apt to crystallise, and also is always the difficulty that with high percentages of cineol (above 75 per cent.), small flakes of the euco-resorcinol compound separate, and, rising to the surface of the concentrated resorcinol solution, lie at the junction of the two liquids, and prevent an accurate reading.

My experiments show that this method is most reliable when the percentage of cineol does not exceed 40 to 50 per cent. High percentage oils require to be diluted, and for this purpose Schimmel recommends turpentine. Unless great care is taken, the use of turpentine is likely to produce serious errors. Various samples of different qualities were tried, and all were found to contain substances absorbed by resorcinol.

After many trials I found that by using as a diluent the fraction of turpentine distilling between 156° to 160° C., the objection was overcome. To

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confirm this, the first fractions (below 150° C.) were collected and tested with resorcinol, and also the higher fractions (over 160° C.), as well as the residual oil in the distillation flask, and all showed appreciable though varying absorptions, according to the variety of turpentine used. Of these, the first distillate showed least absorption, the higher distillate more, and the residuum by far the greatest, the absorption obviously being due to oxidation products of the terpenes. It is convenient to distil 200 c.c. of ordinary turpentine in a 250 c.c. round-bottom distillation flask, fitted with side-tube, by means of direct flame. The fraction distilling between 156° to 160° C. may be kept indefinitely.

One hundred c.c. of the oil to be tested for cineol are distilled in a flask of 150 c.c. capacity, reserving the distillate collected from 170° to 190° C., and then diluting to 100 c.c. with the distilled turpentine. If a trial shows the percentage of cineol to be above 70 per cent., the distilled cineol is diluted further with the turpentine, so that the percentage is not over 50. The temperature is noted, and 6 to 10 c.c. shaken with warm 55 per cent. resorcinol solution. After five minutes' shaking more solution is added, so as to bring the oil to the graduated neck. It is then cooled, and the volume read.