

Estimation of Insoluble Lac Resin in Old Lac Samples

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A method has been standardized for the estimation of polymerized lac present in old seedlac and shellac samples. The method is based on the fact that insoluble lac resin dissolves in boiling 95% alcohol in the presence of mineral acid. The method is also applicable for the detection of old lac samples admixed with fresh lac samples, at as low as 2% level.

IT is well known that when seedlac or shellac is stored for a long time, especially under unfavourable conditions, it deteriorates and gradually becomes insoluble in alcohol and infusible. As a result, while the percentage of non-lac impurities remains constant, that of insoluble lac resin in the sample increases gradually. There is no known quantitative method for the determination of the latter. An attempt has, therefore, been made to develop a standard method for the estimation of this insoluble resin.

When the non-volatile matter insoluble in hot alcohol (termed as an impurity) is determined in a sample of old lac, the insoluble portion obtained consists of polymerized lac and non-lac impurities. When polymerized lac is boiled with 95% alcohol in the presence of a mineral acid, the insoluble lac goes into solution¹⁻³. The method for the determination of insoluble lac in any sample reported in this communication was standardized taking advantage of this observation. The method is similar to those recommended by the Indian Standards Institution (IS: 15 & 16: 1956) and the International Organization for Standardization (ISO R55 & 56: 1957), except for the second determination using alcohol-containing mineral acid.

For purposes of this determination, sulphuric acid was preferred to hydrochloric acid as the mineral acid to be used, because of

the fact that the former does not volatilize during dissolution at the boiling temperature. From a series of experiments with various samples of old lacs, the optimum amount of sulphuric acid required to solubilize the insoluble lac resin in a 5 g sample of old lac in 125 ml of 95% alcohol was found to be 1 g or 0.6 ml of concentrated acid of sp. gr. 1.84.

Details of the estimation method⁴⁻⁶

The sample is first ground so as to pass completely through a sieve of aperture 0.425 mm in the case of seedlac and 0.71-0.425 mm in the case of shellac.

An extraction cartridge (26×60 mm) is freed from alcohol solubles by placing it inside the siphon tube and 125 ml of 95% alcohol in the flask, and extracting with alcohol for 30 min. The burner is so adjusted as to ensure one cycle of filling and emptying of the siphon tube every 2 min. The cartridge is then taken out, dried in an oven at 100° ± 2°C for 3 hr and weighed in a tared weighing bottle (40×80 mm) kept in a desiccator over sulphuric acid. Drying and weighing are repeated till a constant weight is obtained.

Through another cartridge, a boiling solution of 95% alcohol (125 ml) containing sulphuric acid (0.6 ml) diluted with an equal volume of water is filtered to get rid of matter, if any, soluble in acidulated

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alcohol. The cartridge is washed with 50 ml hot alcohol from the top downwards, transferred to the siphon tube, extracted with alcohol, dried and weighed in the same way as the first one.

The powdered sample is mixed thoroughly by rolling on paper several times and a 4.5-5.5 g lot is accurately weighed and placed in a 200 ml beaker, to which 125 ml of 95% alcohol is then added. This is then covered with a cover glass and placed on a boiling water-bath. The alcoholic solution is boiled vigorously for 30 min, keeping the volume constant by replenishing from time to time.

A weighed cartridge (first set) is placed inside the filter tube surrounded with hot water, which is maintained at not less than 90°C. The boiling lac solution is decanted into the cartridge followed by the insoluble matter with successive portions of hot 95% alcohol. Finally, the cartridge is washed from the top downwards with a fine jet of hot alcohol. For this transfer, nearly 75 ml of hot alcohol is required.

The cartridge with the insoluble matter is next transferred to the siphon tube in the extraction flask and extracted with alcohol as described earlier for 1 hr with exactly 30 cycles of complete filling and emptying of the siphon tube. The cartridge is removed, drained upright on filter paper, dried in an oven at $100^{\circ} \pm 2^{\circ}\text{C}$ for 2 hr, cooled and weighed. The drying process is repeated till a constant weight is obtained.

The experiment is repeated with another 4.5-5.5 g sample using 125 ml of 95% alcohol containing 1.2 ml aqueous (50:50 by vol.) sulphuric acid for the initial dissolution and using another cartridge (second set) for the filtrate.

The first determination gives the percentage (W_1) of total insolubles, i.e. of non-lac impurities and insoluble (or polymerized) lac and the second (W_2) of the non-lac insolubles alone. The difference between these two values ($W_1 - W_2$) obviously gives the percentage of polymerized lac in the sample.

The determination was repeated a number of times and was found to give reproducible results. Some typical data for fresh and old samples of seedlac are presented in Table 1.

It is seen from Table 1 that in the case of fresh lacs, there is practically no difference in the percentage of insolubles obtained using alcohol, with and without mineral acid, while in old lacs, there is a difference, which is obviously due to the polymerized (insoluble) lac present.

To test the applicability of the method for the detection of old lacs when admixed with fresh lac, an old sample of seedlac was mixed with a fresh one in different proportions and subjected to the estimation. The results given in Table 2 indicate that the incorporation of old seedlac (polymerized lac content 20.89%) in fresh sample to the extent of even 2% can be detected by this method.

Table 1 — Percentage of polymerized lac in old seedlac samples

Sl No.	Variety of seedlac	Age of seedlac year	Hot alcohol insolubles, %		Polymerized lac ($W_1 - W_2$) %
			In alcohol (W_1)	In alcohol containing mineral acid (W_2)	
1	<i>Kusmi</i>	Fresh	2.89	2.92	—
2	<i>Rangeeni (ber)</i>	do	2.76	2.76	—
3	<i>Rangeeni (palas)</i>	do	4.14	4.14	—
4	<i>Rangeeni</i>	do	3.28	3.28	—
5	do	13	19.75	6.79	12.96
			19.77	6.79	12.98
			19.75	6.77	12.98
6	<i>Kusmi</i>	10	18.74	3.20	15.54
			18.76	3.24	15.52
			18.75	3.22	15.53
7	do	12	30.11	3.76	26.35
			30.14	3.72	26.42
			30.14	3.74	26.40
8	<i>Rangeeni</i>	13	20.16	5.06	15.10
9	do	13	28.34	4.40	23.94
10	do	14	14.90	4.94	9.96
11	do	14	24.53	3.64	20.89
12	do	14	26.16	4.29	21.87

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Table 2 — Percentage of polymerized lac in a mixture of fresh and old seedlac samples

(The old seedlac sample contained 20.89% of polymerized lac)

Sl No.	Proportion of seed-lacs in the mixture		Hot alcohol insolubles, %				Polymerized lac	
	Fresh	Old	In alcohol		In alcohol with acid		Found %	Calc. %
			Found	Calc.	Found	Calc.		
1	100	0	3.28	—	3.28	—	—	—
2	98	2	3.72	3.71	3.28	3.29	0.44	0.42
3	95	5	4.38	4.34	3.29	3.30	1.09	1.04
4	90	10	5.44	5.41	3.30	3.31	2.14	2.10
5	80	20	7.56	7.53	3.36	3.35	4.20	4.18
6	70	30	9.67	9.66	3.39	3.39	6.28	6.27
7	60	40	11.77	11.78	3.44	3.42	8.33	8.36
8	50	50	13.93	13.91	3.46	3.46	10.47	10.45
9	0	100	24.53	—	3.64	—	20.89	—

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