

Destructive Dry Distillation of Lac

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Analytical data for the product of dry distillation of lac with hydrated calcium oxide are presented. The presence of carboxyl group, aldehyde and ketone groups, ester group, hydrocarbons and unsaturation in the distillate is indicated.

LAC resin is a mixture of some free mono-, di- and non-hydroxy aliphatic and hydroaromatic acids and their inter-esters^{1,2}. The presence of a free aldehyde group and unsaturation (Koul, G. L. & Misra, G. S., unpublished data) has also been indicated. All these facts provide scope for chemical modification of the resin and also for the possible use of the resin as the starting material for making some useful products by various degradation processes.

The modification of lac resin through the preparation of ketones by thermal pyrolysis of the calcium salts of fatty acids has been tried by some workers³ by distilling lac resin with lime. For every 100 g. of shellac, a distillate consisting of 50 ml. (approx.) of an oily product, 14 ml. (approx.) of water, a considerable portion of uncondensable gases and a charred residue were obtained. The nature of the oily product has not been studied in detail. It was felt that a detailed study might give some idea regarding its possible uses in different fields of application.

A dry intimate mixture of partially dewaxed shellac (ASK grade) with hydrated calcium oxide (lime) in 1:1 proportion by weight was subjected to distillation. The mixture was heated directly by a gas flame in a Pyrex glass vessel. The distillate was collected at 245°C. (max.) in a flask cooled in air, the residual gas being allowed to pass through two traps cooled in ice and finally into a third trap. Most of the distillate collected in the first receiver. In the ice-cold receiver the distillate was thinner in consistency and lighter in colour. In the last trap, a small quantity of light brown semisolid substance collected. All the distillates were mixed together and were 51.2 per cent on the weight of shellac.

The distillate was extracted with ether and the extract was dried over anhydrous sodium sulphate. After removing ether, a dark brown, fluorescent, viscous oily product having a terpenic odour was left; yield, 35 per cent on the weight of shellac. The ether-insoluble portion was water.

During distillation, a good amount of uncondensable gas evolved which burnt with a luminous flame. A charred residue was left in the flask.

The ether-soluble fraction was first qualitatively tested and gave positive indication for the presence of free carboxyl group, unsaturation, aldehyde and ketone, ester group and hydrocarbon. Presence of phenols and quinones was also indicated.

The product had the following characteristics: acid value, 30; saponification value, 129.1; carbonyl value, 90.7; and hydroxyl number, 53.15. It had specific gravity 0.9308 and relative viscosity 5.055 cp.

The oily ether-soluble product was miscible in all proportions with alcohol, acetone, ethyl acetate, carbon disulphide, chloroform, carbon tetrachloride and hydrocarbon solvents.

The values confirm the presence of $-\text{COOH}$, $-\text{COOR}$, $-\text{C}=\text{O}$, $-\text{C}=\text{C}-$ and $-\text{OH}$ groups. The values differ, as expected, considerably from those for original shellac. Lowering of acid and saponification values may be due to the formation of hydrocarbons and ketones, whose presence has been indicated. The hydroxyl value of shellac is about 270 and iodine value 18 as against 53.15 and 90.74 of the pyrolytic product. These changes may be due to the formation of unsaturated bonds by dehydration of $-\text{OH}$ groups with adjacent hydrogen. Increase of unsaturation gives promise for using the distillate for producing useful polymerization products.

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References

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