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# THE REFRACTIVE INDEX OF SHELLAC.

By

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The value of the use of the refractive index as an analytical constant for establishing the identity or purity of resins has been pointed out by Bradley (1). The rapidly increasing number of resins which are now being synthesised and coming into commercial use lends importance to such a constant.

The refractive index of a paint medium has considerable influence on the appearance of the film. The use of pigmented shellac varnishes, interest in which appears to be growing, makes it desirable to have reliable information about this property of shellac.

There appears to be no reason why the light coloured shellacs now available should not find use in certain cases as 'mounting media' in microscopy; knowledge of the refractive index for this use is, of course, essential.

Published values for this constant for shellac are somewhat conflicting. Bradley (1) gives a figure  $1.516 \pm .003$  for superfine orange shellac and  $1.534 \pm .003$  for dry bleached shellac. Chamot and Mason (2) give a figure 1.54. Hanstock (3) gives 1.518 for T. N. shellac, 1.517 for Palas shellac and 1.520 for A. C. garnet lac.

It was considered desirable therefore that a survey should be made of the possible variation in the refractive indices of a fairly wide range of shellac samples.

**Experimental.**--Fresh sticklacs were collected from various reliable sources. Many samples were taken from the experimental plantation attached to the Indian Lac Research Institute ; others were sent by members of the Indian Forest Service ; and others by reliable dealers. These samples comprised sticklacs from different host trees, grown in different seasons, from different localities and collected in different stages of maturity. Samples which had been stored for known periods were also obtained. These sticklacs were manufactured into shellac by the country process in an experimental factory attached to the Institute. It may be stated confidently that this range of shellacs covers the majority of the material which goes to make the pure shellac of commerce.

Several possible methods of determining the refractive index of the samples were investigated. Microscopical methods utilising the Becke's effect or the oblique illumination method were tried using two types of immersion media. One series consisted of mixtures of clove and mineral oil, and the other consisted of solutions of mercuric iodide in potassium iodide. It was not found possible to get reliable values by these methods. The former medium gave a figure  $1.525 \pm .003$  while the latter give  $1.518 \pm .003$ . It is clear, therefore,

that this method cannot be considered satisfactory.

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The method finally adopted was substantially that used by Hanstock (3). The sample of shellac was melted and flowed between two thin double convex lenses of focal lengths 20 and 25 cms. The alteration in the focal length of the combination lens system so made, due to the displacement of air by the shellac was measured on an optical bench.

The lenses and bench were calibrated as follows:—Several samples of mixtures of clove and mineral oil were chosen with a range of refractive index for the D sodium line of  $1\cdot505-1\cdot535$ . These values were carefully determined in a previously calibrated Abbé refractometer correct to  $\pm \cdot 0005$ . These mixtures were in turn placed in the space between the lenses and the distance 1 between the conjugate focal positions was determined on the optical bench. The distance between the image screen and the object (a ground glass plate ruled with pencil lines) was carefully measured. The refractive indices of the liquids were plotted against  $\frac{d^2 - 1^2}{4d}$  and a straight line drawn through the points. From this curve the refractive index of a shellac sample could be determined by measuring 1 when the resin was fused between the lenses.

All determinations were made at laboratory temperature which was  $23^{\circ}C \pm 3^{\circ}C$ .

The results obtained for pure shellacs are summarised in Table I.

### TABLE I.

* Type of shellac.		Refractive Index $\pm$ .001
Schleichera trijuga (Kusum) 1990	9. <b>.</b>	1.515 - 1.518
Zizyphus Jujuba (Ber)	10.20	1.516 - 1.519
Butea frondosa (Palas		1.516 - 1.519
Shorea talura	·	1.517 and could new add
Assam Host		1.518
Albizzia species		1.517
Schleichera trijuga (Kusum) on Acacia Catechu		1.518
Flemingia congesta daga de ano		1.517
Ficus glomerata		1.516
Schleichera trijuga (Kusum, Burma)		1.516
Zizyphus Xylopyra (Ghont)		1.519

\* For an explanation of the types of shellac see 'A Manual of Lac Cultivation' by P. M. Glover; Indian Lac Research Institute publication.

Certain specially treated shellacs were examined and the results are summarised in Table II.

# TABLE II.

Type of shellac.	History. Refractive	Index $\pm .001$ .
Schleichera trijuga (Kusum) shellac.	Fused with 30% rosin in the laboratory	1.526
Butea frondosa (Palas)	Old factory sample mixed with 10% rosin	1.521
Mixed sample	Old factory sample mixed with 30% rosin and shellac bye-products.	1.525
R. S. Alba	German machine made lac	1.519
Wax-free shellac	Sample from Angelo Bros., Calcutta	1.521
Wax-free shellac	Old sample prepared in laboratory	1.522
Wax-free shellac	Fresh sample prepared in laboratory by cold alcohol extraction.	1.521
Depolymerised shellac (4)	Prepared in laboratory	1.520
Bleached lac	Fresh sample prepared in laboratory	1.520
Ether soluble resin from shellac.	Sample prepared in laboratory	1.503
Pure resin from shellac	Sample prepared in laboratory	1.526
Sulphur treated shellac (5)	5% sulphur treated at 150°C—160°C	1.523

In view of the considerable change which takes place on subjecting shellac to heat treatment the possibility of change in refractive index was examined. A sample of Kusum shellac was heated for several hours at  $105-110^{\circ}$ C a sample being removed at certain intervals. This treatment resulted in considerable change in certain physical properties, *e.g.*, viscosity as measured by the Metropolitan Vickers apparatus at  $25^{\circ}$ C was increased by several fold. The refractive index of the heated sample showed practically no change as shown in Table III.

## TABLE III.

## Heat treated shellac.

 Temp. of treatment.
 Time of heating.
 Refractive index ± .001

 control
 1.517

 105--110°C
 1 hr.
 1.517

 ,,
 2 hrs.
 1.516

 ,,
 3 ,,
 1.518

**Discussion.**—The samples examined in Table 1 are such that they cover the greater part of the materials which go to make the shellacs of commerce. It will be seen that the variation of refractive index from sample to sample is not very large. It would appear therefore that any alleged pure country made shellac with full wax content which gave a refractive index outside the range 1.516—1.519 should be suspected of abnormality.

Bradley (1) gives a figure  $1.534 \pm .003$  for a sample of dry bleached shellac using the immersion method. This figure differs considerably from the value 1.520 recorded in this paper. The uncertainty of the immersion method is a possible explanation of this difference.

The method of determining the refractive index described in this paper is very satisfactory in that the results are obtained with good reproducibility and presumably accuracy, but the method is somewhat cumbersome if only occasional examinations of shellacs are made. In this case there is much to be said for the extreme simplicity of the immersion method. If this method is used the results described in this paper indicate the need for careful choice of immersion medium. Of the two series investigated the solutions of mercuric iodide in potassium iodide give results approximately correct. Mixtures of clove oil and mineral should not be used since for some reason, possibly solubility, they give results much too high.

Summary.—Accurate determination of the refractive index of shellac indicates that all pure country made shellacs should give values within the range 1.516—1.519. Any shellac giving a value outside that range may be suspected of abnormality.

### References.

- 1. Bradley T. F.: Ind. Eng. Chem., Anal. Ed., 3 No. 3, p. 304.
- 2. Chamot & Mason: Hand-book of Chem. Microscopy. J. Wiley & Sons, Vol. I. p. 588.
- 3. Hanstock: Technical Paper No. 35. April 1932. The Paint Research Sta., Teddington.
- 4. Rangaswami & Aldis: "Depolymerisation of shellac" (in preparation).
- 5. Venugopalan : Res. Note No. 10. Nov. 1933. Indian Lac Res. Inst., Ranchi.