made up samples combining wood rosin with tallow fatty acids and tested them with results as shown in TABLE V.

The indications are that *after* saponification, the results obtained are about a percent lower than *before* saponification.

Five Brands of yellow laundry soaps purchased on the market were tested by the Wolff and McNicoll Methods. Data are indicated in TABLE VII. The results by the McNicoll Method are in line with those obtained by the Wolff Method.

The figures as shown in TABLE V indicate that the McNicoll Method is equally as accurate for wood as for gum type rosin.

In making all of the foregoing tests, the various samples were prepared by adding the desired quantity of rosin to pure tallow fatty acids. Inasmuch as the rosin undergoes no saponification in this manner, it was thought advisable to determine the effect of saponification of the rosin-tallow mixtures on the McNicoll determination. Hence, two samples containing definite amounts of rosin-tallow fatty acids were completely saponified, then split with the mineral acid and the mixed fatty acids separated by washing, filtering and drying. The results of the McNicoll rosin determination before and after saponification are indicated in TABLE NO. VI.

On the basis of the data obtained, the following facts have been established with regard to the McNicoll Method.

1. It is rapid and easy to run; a decided advantage in this respect over both the Wolff and Twitchell Methods.
2. It produces reliable and consistent results.
3. It is not appreciably influenced by the grade or type of rosin contained in the soap.
4. It yields satisfactory results when applied to laundry soaps containing rosin.

References:

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THE IODINE VALUE OF TUNG OIL

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THE fact that the apparent iodine value of Tung oil varies with the conditions under which it is determined was apparently first noted in 1909 by Boughton[1] who mentions temperature, time of reaction, concentration of reagent and weight of oil as controlling factors. Notable attempts at standardising the test include that of A. C. Chapman[2], who proposed the use of 0.1 gram of oil dissolved in 20 ml. of purified carbon tetrachloride with 30 ml. of 0.2—N Wijs solution and allowed the reaction to proceed for three hours in the dark.

Thus it will be seen that the difficulties of the problem have been recognised for about 30 years and standardisation of the test in one form or another has been practised for nearly as long.

Evidence in our possession in 1930, but not mentioned in our paper, indicated clearly that the "instantaneous" figure obtained from the 30 minute and 3 hour figures is independent of the excess of reagent and of the temperature of reaction within the limits of ordinary laboratory temperatures.

Reference to our paper will show that there is additional proof that the "instantaneous" figure corresponds to the two double bond absorption, since otherwise percentages of elaeostearic acid calculated from it and the Tom's bromine value[5] would not correspond with those obtained by our polymerisation method.

We therefore feel that the following method, which has stood the test of eight years' application, is preferable to that put forward by von Mikusch:

It has however been realized only in comparatively recent years — as J. D. von Mikusch points out in this journal[3] — that such standardisations gave empirical figures which did not correspond to any definite degree of halogenation.

We ourselves were concerned some eight years ago with the problem discussed by von Mikusch at a time when we were searching for methods of determining elaeostearic acid in the oil. It is interesting to note that we approached the problem on similar lines and, although we did not discuss the effect of temperature, we dealt with the effect of varying the times of contact of oil and different halogenating reagents in a paper read before the Society of Public Analysts in 1930.[4]

The conclusions we then came to are now confirmed bv von Mikusch[3] who agrees that the iodine value corresponding to absorption by exactly two of the three double bonds of elaeostearic acid may theoretically be obtained by absorption for zero time.

In our experiments we found that certain irregularities developed when the time of reaction was limited to less than 5 minutes. On this account we preferred to make two determinations — one of 30 minutes duration, and the other of 3 hours — and to extrapolate from these to the figure for zero time. Alternatively we found that a single figure obtained with the quantities of reagents described by Chapman[2] and only 20 minutes time of reaction is sufficiently close to the "instantaneous" figure for practical purposes.

METHOD

Weigh out 0.08 to 0.10 gram of filtered tung oil into a 300 ml. flask. Add 20 ml. of cp. chloroform or carbon tetrachloride and 30 ml. of 0.2-N Wijs solution.

Close the flask with a watch glass and allow to stand at laboratory temperature for 30 minutes. Add 20 ml. 10% KI solution, shake and add 100 ml. of distilled water. Titrate with 0.1-N $Na_2S_2O_3$ solution using starch solution as indicator.

Repeat the test using the same quantities of reagents but allowing 3 hours for the absorption.

Carry out blank tests without oil alongside each determination.

Calculate in the usual manner:—

I.V. $_{30\ min.}$ = the Wijs iodine value obtained after 30 minutes' absorption.

I.V. $_{3\ hours}$ = the Wijs iodine value obtained after 3 hours' absorption.

Then the "instantaneous" iodine value is given by the expression: I.V. $_{30\ min.}$ — 1/5 (I.V. $_{3\ hours}$ — I.V. $_{30\ min.}$)

We may add that closure of flasks by watch glasses during iodine value determinations has been practised in this laboratory for over 25 years with completely satisfactory results. If any halogen is lost during the period of standing we have yet to obtain proof that it can in anyway affect the results, while the procedure avoids irregularities which may arise from the presence of an aqueous solution in close proximity to the reagents.

In our experience iodine values so determined agree with those carried out with the greatest care in stoppered flasks.

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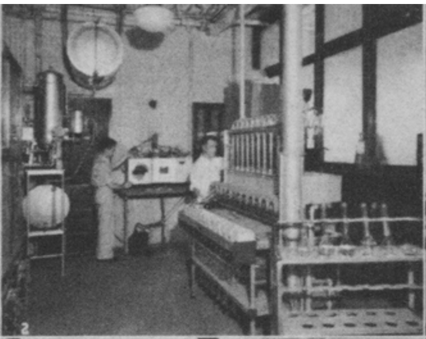

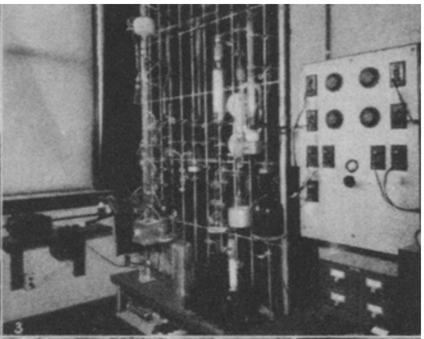

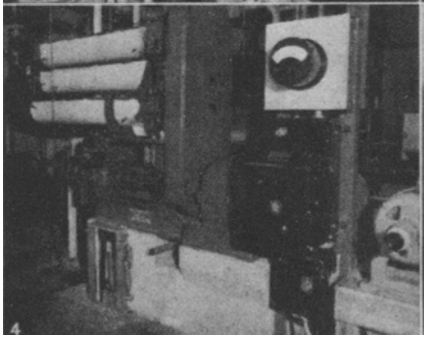

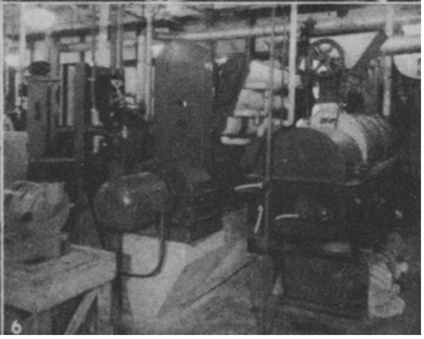

1. *Research on soybean meal and oil*
2. *Analytical equipment*
3. *High vacuum molecular stills and vacuum pumps*
4. *Small expeller, Engineering Section*
5. *Constant temperature-humidity room showing Sanderson drying meter and film spinner used in paint and varnish studies*
6. *Rotary drier, expeller and laboratory mill*