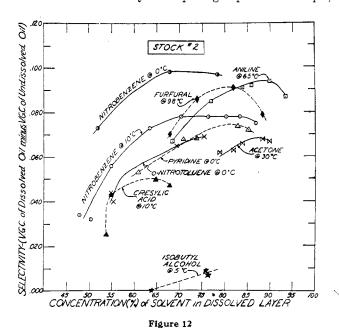
There are, however, many indications pointing to a generalization that solvent action is profoundly affected by certain structural characteristics and the presence of certain groups, most of which are usually called polar groups. For example,



the first nine compounds in Table VII are of ring structure. That cyclic compounds are superior to chains is indicated by the following comparisons in which the polar groups are identical: Phenol is superior to *n*-propyl and isobutyl alcohols; benzaldehyde and furfural are more effective than propionaldehyde; and phenyl acetate surpasses methyl acetate. Furthermore, the addition of aliphatic chains to the ring appears to cut down the efficiency, as is shown by the

fact that nitrobenzene and phenol are, respectively, superior to o-nitrotoluene and cresylic acid.

The group attached to the ring obviously affects the solvent action of the compound. Thus (Table VII) the nitrile group attached to the benzene ring is followed in decreasing order of efficiency by the nitro, aldehyde, acetate, hydroxyl, and amino groups. The above considerations afford qualitative criteria of solvent action, but work now in progress offers some hope of deriving quantitative relationships.

### Acknowledgment

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# A New Varnish from Xylose and Aniline

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BY THE combined efforts of the Federal Phosphorus Company, the Bureau of Standards, the Alabama Polytechnic Institute, and the University of Alabama, xylose has recently been made from cottonseed hulls on a small factory scale at Anniston, Ala. (1). A part of the work assumed by the University of Alabama was to find new uses for xylose. During the progress of this research some new resins were produced, one of which, when dissolved in acetone, provides a good varnish.

## Production of Resin

The resin is made by heating xylose and aniline in equimolecular proportions at 75–80° C. for 10 minutes. When first mixed at room temperature, xylose and aniline give a damp, granular mass similar in consistency to wet sand. With constant grinding the mass becomes more fluid and some resin is formed with slight evolution of heat, but no homogeneous resin could be produced without heating.

<sup>1</sup> Received April 9, 1931. Presented before the Division of Paint and Varnish Chemistry at the 81st Meeting of the American Chemical Society, Indianapolis, Ind., March 30 to April 3, 1931.

At 35-40° C. the mass becomes dark brown and liquid enough to stir easily, but from 45° to 55° C. it becomes stiff and granular and is dark red. From 60° to 80° C. a greater liquidity and darkening of color gradually appear and at 75° C. the color is black and the viscosity from 60 to 65 Saybolt. From this point the rate of heating has a marked effect. If held at about 75-80° C. for 10 minutes, the most satisfactory resin is obtained. If, however, the resin is further heated rapidly on the water bath, 85° C. being reached in from 3 to 5 minutes, the mass suddenly begins to froth and expands from twenty to twenty-five fold in volume. At the same time there is a sudden evolution of heat and the temperature of the resin is several degrees higher than that of the bath. Very slow elevation of heat, from 80° to 100° C. in 25 to 30 minutes, does not result in foaming or frothing, but shortly after 100° C. is reached the temperature of the resin rises to 104° C., with the bath still at 100° C.

Using 150 grams xylose and 92 grams aniline, the molar weights, it was found that in the slow heating process no water is lost up to 75° C., one mol of water is lost from 75° to 100° C., and during the time when the resin, on a 100° C.

water bath, goes through the range from  $100^{\circ}$  to  $104^{\circ}$  C. and back to  $100^{\circ}$  C., a second mol of water is lost. In the rapid heating process approximately the same losses of water were noted, but the solubility in acetone was much less.

#### Production of Varnish

All the products except the granular one at 45-50° C. dissolve to some extent in acetone. The resin made by holding the temperature at  $75-80^{\circ}$  C. gives the best and clearest solution for varnish purposes. Acetone is the only common organic solvent which will dissolve it. The acetone solution flows readily from the brush, has good covering power, and dries in about 30 minutes. When applied to pine wood it ranges from cherry, through mahogany, to dark oak, according to the concentration of the solution. The finished work is of a satisfactory luster and is proof to hot water, alkalies, and acids except nitric, which gives a redder tint without removing the varnish. In making the varnish solution it was found advantageous to dissolve the resin shortly after it was made or to protect it from the air. If left undissolved in contact with air, it does not dissolve so well later, there is an undissolved residue of the resin, and the finished work has less luster. The resins made by the slow process at temperatures higher than 80° C, are much more susceptible to change by air exposure, in the

undissolved state, but they have the advantage of giving darker stains for the same concentrations in acetone solution.

#### Cost

It was found at Anniston that the cost of pure crystallized xylose is much greater than that of an impure product in cake form, the purification and crystallization being the largest items of expense as to time, apparatus, and expert control. The earlier experiments on the xylose-aniline resin were made with purified xylose and laboratory quality aniline. Later work has shown that even better results, at a lower price, can be had with aniline oil and impure cake xylose still containing gums and coloring matter.

While no cost sheets are available for the impure cake xylose employed, it is estimated to be about 2 cents per pound. The finished varnish contains from 2 to 5 pounds of the resin to the gallon, according to the depth of color required for one coat. The heaviest varnish would therefore require about 2 pounds of aniline to the gallon. The raw materials for this varnish, using the unpurified xylose, should not cost more than about \$1.00 per gallon. The equipment for manufacture and the procedure are very simple.

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# The Crystal Form of Paraffin Hydrocarbons

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PURE paraffin hydrocarbons from C<sub>18</sub>H<sub>38</sub> to C<sub>43</sub>H<sub>88</sub> as isolated from petroleum waxes crystallize in plates quite distinct from the characteristic needles formed by samples less carefully purified (1). Four photomicrographs are presented here showing that synthetic normal paraffin hydrocarbons containing 22, 23, 30, and 40 carbon atoms likewise form platelike crystals apparently identical with those of the petroleum waxes.

The hydrocarbon C<sub>22</sub>H<sub>16</sub> was prepared by the electrolysis of lauric acid. Its melting point was 44.3° C. The hydrocarbon C<sub>23</sub>H<sub>48</sub> was prepared by catalytic hydrogenation of laurone. It melted at 47.8° C. The writer is indebted to E. E. Reid and F. O. Cockerille, who furnished these samples

<sup>1</sup> Received April 30, 1931. Contribution 59 from the Experimental Station of E. I. du Pont de Nemours and Co.

and who will describe them in a later publication. The hydrocarbons  $C_{30}H_{52}$  and  $C_{40}H_{52}$  were synthesized at this laboratory by the action of sodium on decamethylene bromide. They have already been described (2). In each case the method of synthesis leaves no doubt as to the structure of the resulting compound. The photomicrographs were taken using polarized light as has been described previously (1). Magnifications are  $\times$  80.

The writer is indebted to H. B. DeVore for assistance in obtaining the photomicrographs.

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C22H46

C23H48

C20H62

C40H82