

ments resulted in the formation of 93.5 g. of *n*-propyl nicotinate (b. p. (1 mm.) 80–82°). This represented a yield of 56.7%.

Nicotine and β -picoline behaved in a similar manner and gave yields of the esters in direct proportion to their ease of oxidation to nicotinic acid.³

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Dehydration of Tetrahydrofurfuryl Alcohol¹

By CHARLES HOWARD KLINE, JR.,² AND JOHN TURKEVICH

In the course of study of the catalytic synthesis of pyridine from furfural derivatives, the dehydration of tetrahydrofurfuryl alcohol was investigated. Paul³ has found that the dehydration of tetrahydrofurfuryl alcohol over alumina results in the formation of 1,2-dihydropyrene, C₈H₈O, but did not investigate in detail the yields produced due to variation in temperature, contact body or lifetime of the catalyst.

The materials and experimental procedure were the same as those described by the authors in the publication of the catalytic synthesis of pyridine.⁴

Over alumina it was found that tetrahydrofurfuryl alcohol is unattacked at 250° but is readily dehydrated at 300 and 350°. The latter temperature is the optimum temperature for the dehydration and yields about 70% dihydropyrene. At 400° and above, high boiling material is chiefly formed and much of the feed alcohol is lost either as cracked gases (19% carbon dioxide, 57% unsaturates, 13% hydrogen and 11% saturated hydrocarbons) or catalyst deposit. Condensation of the tetrahydrofurfuryl alcohol residues appears to predominate at these temperatures. The alumina catalyst did not lose activity at 300° and at 350° the activity fell only moderately during four days of continuous operation. In all cases carbonaceous material was left on the surface of the catalyst. It is of further interest that at 300° the dehydration of tetrahydrofurfuryl alcohol poisoned the subsequent dehydration of absolute ethanol even though it did not poison further dehydration of the tetrahydrofurfuryl alcohol. Undoubtedly the tetrahydrofurfuryl alcohol poisons the catalyst for ethanol dehydration by being strongly adsorbed on the surface. This view is confirmed by the fact that only fifteen minutes after the start of the reaction, does the product come out of the catalyst exit tube. Further confirmation is the forty-degree rise in temperature of the catalyst bed on the introduction of the tetrahydrofurfuryl alcohol. This is to be contrasted with a nine degree rise when the ammonia is simultaneously introduced.

Over silica, tetrahydrofurfuryl alcohol is stable up to 400°, at which point some decomposition

sets in. Dihydropyrene was not the only product of the reaction but there was material boiling below and above this substance. At 450° about 20% water was formed and most of the non-aqueous product appeared as high boiling still residues.

Over thoria, tetrahydrofurfuryl alcohol was stable up to 450°. The low-boiling material collected during the 550° run polymerized on standing and had a sharp odor. These facts and its boiling point suggest that it may have consisted of pentenes and pentadienes. It should be pointed out that the recovery of tetrahydrofurfuryl alcohol was high. The inactivity of the thoria for the dehydration was not due to its general lack of catalytic activity. After revivification following the 450° run, the catalyst could be used to dehydrate anhydrous ethanol. One is thus faced with the interesting fact that on alumina the efficient dehydration of tetrahydrofurfuryl alcohol poisons the alumina for the dehydration of ethanol, while on thoria the inefficient dehydration of tetrahydrofurfuryl alcohol does not poison the dehydration of ethanol.

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A Relation between Viscosity and Refractive Index

By R. T. LAGEMANN

During an attempt to estimate the viscosity of higher members of some homologous series, it was noted that the viscosity and the refractive index for liquids are functionally related. A linear relation is found to hold for every homologous series if the molecular refraction R is plotted against the viscosity constant I , which is defined by Souders¹ as

$$I = \frac{M}{d} \{ \log_{10} (\log_{10} \eta) + 2.9 \}$$

where η is the viscosity in millipoises. For such a plot and for determining the constants of the equations, data for I may be obtained from Souders¹ and values of R from Eisenlohr,² Herz³ and the Landolt-Börnstein tables.

In Table I are given the values of the constants a and b as calculated for several series by the method of least squares on the assumption that $I = aR + b$. From this table it may be seen that the slopes are very nearly identical for all the series examined, while the intercepts vary. That linear curves represent the relations very well may be deduced from the fact that use of the constants of Table I allows one to calculate values of I averaging within $1/8$ of one per cent. of the experimental values. As a consequence, if the density and molecular weight of a liquid belonging to any of the series of Table I are known, the viscosity

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(2) F. Eisenlohr, *Z. physik. Chem.*, **76**, 585 (1910).

(3) W. Herz, *Z. anorg. allgem. Chem.*, **179**, 211 (1929).