with a dropping funnel, stirrer and thermometer, and cooled with an ice–salt-bath. Bromine (3.2 g., 0.02 mole) was then dropped in with stirring over a period of one-half hour. The solution was filtered and the carbon tetrachloride removed under reduced pressure. There remained a dark-colored oil which solidified to a brown solid (crude yield, 4.9 g.). Several recrystallizations from ethanol with activated charcoal treatment afforded 3.7 g. of colorless crystals with a fruity odor, m. p. 92–93°. A mixed melting point with the product of condensation of chloral and 2-bromothiophene gave no depression.

*Anal.* Calcd. for $C_{10}H_5S_2Br_2Cl_3$: S, 14.08. Found: S, 14.15.

**Attempted Condensations.**—Attempts were made to condense 2-*t*-octylthiophene, 2,5-*t*-butylthiophene, 2,3,5-trichlorothiophene and 2-benzoylthiophene with chloral using conditions reported above for the successful condensations and also using acetic acid as solvent in the reaction. 2-Benzoylthiophene was unaffected by these reaction conditions, being isolated quantitatively unchanged, while the other thiophene derivatives gave dark oils which we were not able to crystallize or to distill under reduced pressure (1 mm.).

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## 2-Ethylpyridine and Derivatives

BY EARL C. GREGG, JR., AND DAVID CRAIG

2-Ethylpyridine has been prepared in this Laboratory by two independent methods; the hydrogenation of 2-vinylpyridine and the reaction of methyl iodide with α-picolyl lithium. Nevertheless, derivatives (picrate[1] and mercuric chloride[2]) reported by others for 2-ethylpyridine do not agree in their properties with our derivatives.

Since our methods of preparation appear unequivocal, we believe that previously recorded preparations of this compound have been impure or unauthentic.

### Experimental

2-Vinylpyridine[3] was hydrogenated at 60 lb. hydrogen pressure at room temperature over old Raney nickel. One-tenth mole of hydrogen was absorbed by one-tenth mole of 2-vinylpyridine in 35 ml. of alcohol. The hydrogenation product distilled at 70–73° at 50 mm. or 144° at atmospheric pressure, $n^{20}D$ 1.4978. The picrate melted at 108.5–110° (uncor.) when crystallized from alcohol instead of 187° (uncor.) reported by Bergstrom.[1] It was established that no other picrate forming substance was present.

### Analytical

Calculated picric acid content for $C_5H_4O_7N_3 \cdot C_7H_9N$ is 68.1%. Picric acid found by polarographic analysis in 0.1 *N* hydrochloric acid solution is 68.1 ± 0.3%.

The chloroplatinic acid derivative melted at 165–166° (uncor.) as reported by Ladenburg.[2] However, the mercuric chloride derivative melted at 113–114° (uncor.) instead of 103–106° (uncor.) reported by Ladenburg.[2] α-Picolyl lithium was prepared according to Walter.[4]

Dry methyl iodide was added dropwise to the α-picolyl lithium in the reaction flask which was immersed in ice. The resulting ether mixture was extracted with water to remove lithium iodide and some of the unreacted α-picoline. The ether layer was extracted with dilute hydrochloric acid to remove the 2-ethylpyridine and remaining unreacted α-picoline. The hydrochloric acid layer was neutralized and the resulting 2-ethylpyridine was drawn off and steam distilled. The 2-ethylpyridine was dried over anhydrous sodium sulfate and fractionated to remove α-picoline at 77–79° at 80 mm. The picrate from the higher boiling (70–73° 50 mm.) fraction melted at 108.5–110° (uncor.). The mixed melting point of the picrates prepared by the two methods was 108.5–110° (uncor.).

The ultraviolet absorption spectrum of the 2-ethylpyridine prepared above was characteristic of a monoalkyl pyridine.

We wish to acknowledge the measurement and interpretation of the ultraviolet absorption spectrum of 2-ethylpyridine by Laura Schaefgen.

A CONTRIBUTION FROM THE
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## Small Angle X-Ray Scattering of Various Cellulose Fibers

BY A. N. J. HEYN

The study of diffraction and scattering of X-rays at small angles by various substances has been undertaken only very recently and may be considered negligible in comparison with the studies at larger angles.

Small angles studies of fibrous material have been mainly confined to proteins[1–7] which give *discontinuous* scattering indicating the presence of a large (super-identity) period, inside the large molecules.

Only very preliminary work has been done on scattering of cellulose fibers, although it has been long known that ramie fiber gives a *continuous* small angle scattering.[2,8] Kratky and collaborators (1938–1942)[9,10] recently ventured a quantitative evaluation of the scattering in this material and related the identity period calculated to the size and distance of the cellulose micelles, which they found to be 50–60 Å. Besides ramie they also studied a special regenerated cellulose product derived from viscose. (Other authors[11–14]

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