

COMMUNICATIONS TO THE EDITOR

THE CLEAVAGE OF 3-TROPANYL CHLORIDE WITH POTASSIUM CYANIDE

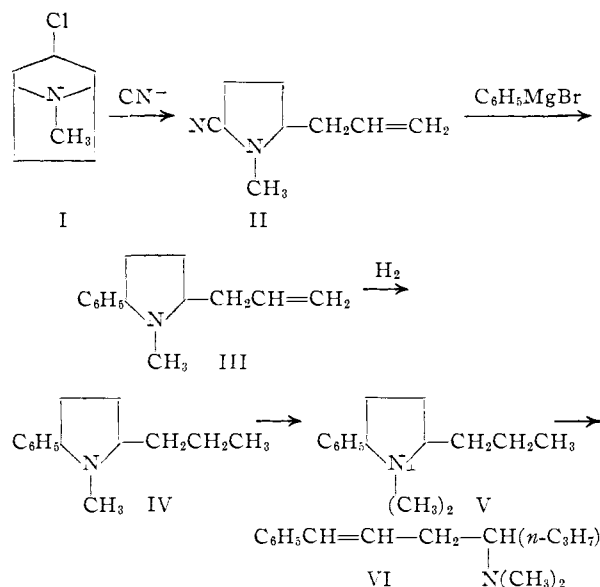
Sir:

When 3-tropanyl chloride¹ (I) was treated with potassium cyanide in alcohol solution a sharp-boiling (b.p. 68–70°, 0.2 mm.) mixture of isomers was obtained whose analysis agreed with the formula $C_9H_{14}N_2$ (I); (*Anal.* Calcd. for $C_9H_{14}N_2$: N, 18.65, N_{AP} ,² 9.33. Found: N, 18.35; N_{AP} ,² 9.38).

The infrared spectrum³ was characterized by the presence of two cyanide bands at 4.50 μ and an intense band at 6.08 μ . When this oil was treated with phenylmagnesium bromide a liquid (b.p. 69–70°, 0.1 mm.), presumably a mixture of isomers also, was obtained, the analysis of which was compatible with the formula $C_{14}H_{19}N$ (III); (*Anal.* Calcd. for $C_{14}H_{19}N$: C, 83.53; H, 9.51; N, 6.96. Found: C, 83.70; H, 9.81; N, 6.84). The infrared spectrum showed that bands due to the aromatic ring system and the 6.08 μ band were present but the absorption at 4.50 μ had disappeared. This displacement of a nitrile function by the radical of a Grignard reagent is manifested by α -di-alkylaminoacetonitriles.⁴ The band at 6.08 μ is indicative of a terminal vinyl group (confirmatory bands at 11.00 μ + 3.27 μ). Catalytic hydrogenation resulted in the uptake of one mole of hydrogen and the bases IV (b.p. 111–113°, 0.4 mm.). (*Anal.* Calcd. for $C_{14}H_{21}N$: N, 6.86. Found: N, 6.90) were isolated. The band at 6.08 μ had disappeared. Structures I–IV illustrate the course of the reactions. The mixtures which persisted throughout were due to the formation of *cis* and *trans* pyrrolidine nitriles in the first step.

The bases (IV) were converted to a mixture of methiodides (V), m.p. 126–130°, the most readily purified of which melted at 173–174° after crystallization from acetone (*Anal.* Calcd. for $C_{15}H_{20}IN$: I, 36.1. Found: I, 36.0). Hofmann degradation of the bases corresponding to V furnished an oil VI (b.p. 125–128°, 0.4 mm.). (*Anal.* Calcd. for $C_{15}H_{23}N$: N, 6.46. Found: N, 6.44) which showed typical styrene absorption, $\lambda_{max}^{95\% EtOH}$ 253 (log ϵ 4.07).

Reduction in the presence of Adams catalyst furnished 4-dimethylamino-1-phenylheptane, which was independently synthesized from 1-phenyl-4-heptanone via oximation, reduction and methylation. The picrates from both samples melted at 92.5–94° (*Anal.* Calcd. for $C_{21}H_{29}N_4O_7$: N, 12.49. Found: N, 12.23) and were otherwise identical in all respects. The 1-phenyl-4-heptanone (b.p. 96–



98°; 0.1 mm.; *Anal.* Calcd. for $C_{13}H_{18}O$: C, 82.06; H, 9.53. Found: C, 82.07; H, 9.43) required for this synthesis was prepared from γ -phenylbutyronitrile and propylmagnesium iodide.

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THYMIDINE INCORPORATION INTO DEOXYRIBONUCLEIC ACID OF RAT LIVER HOMOGENATES

Sir:

The incorporation of radioactive thymidine into DNA¹ of cell-free extracts of *E. coli* was reported in 1956.² Attempts to demonstrate thymidine incorporation in homogenates of chick embryo were unsuccessful,³ although rabbit thymus nuclei have been reported to be active in this respect.⁴ In normal or regenerating rat liver homogenates which actively incorporate radioactive orotic acid into RNA, no radioactivity was found in DNA.⁵ The availability⁶ of very high specific activity tritium labeled thymidine encouraged us to re-examine DNA synthesis in rat liver homogenates. Our experiments have met with some success, and will be described briefly herein.

(1) These abbreviations are used: DNA, deoxyribonucleic acid; RNA, ribonucleic acid; ATP, adenosine triphosphate; and Tris, tris-(hydroxymethyl)-aminomethane.

(2) A. Kornberg, I. R. Lehman and E. S. Simms, *Federation Proc.*, **15**, 291 (1956).

(3) M. Friedkin, D. Tilson and D. Roberts, *J. Biol. Chem.*, **220**, 627 (1956).

(4) M. Friedkin and H. Wood, *ibid.*, **220**, 639 (1956).

(5) E. Herbert, V. R. Potter and L. I. Hecht, *ibid.*, **225**, 659 (1957).

(6) From Schwarz Laboratories, Inc., Mount Vernon, N. Y. Specific activity of the thymidine is reported to be 390 $\mu c./\mu M.$, labeled in position 4; radiochemical purity 100%.

(1) M. Polonovski and M. Polonovski, *Bull. soc. chim.*, [IV] **45**, 305 (1929).

(2) Acetic-perchloric acid titration of basic nitrogen. Analyses were carried out under the supervision of Mr. K. D. Fleischer.

(3) We are indebted to Miss C. M. Martini for determination of the spectra reported here.

(4) P. Bruylants, *Bull. soc. chim. Belg.*, **33**, 467 (1924); *Chem. Abs.*, **19**, 288 (1925).