

Fig. 1.—Spruce native lignin: curve A, 3% solution in dioxane, 0.127 mm. cell; curve B, 10–15% dispersion in Nujol, smear on salt plate; curve C, film from dioxane–EtOH on salt plate (1900  $\text{cm}^{-1}$ –2600  $\text{cm}^{-1}$  section essentially transparent).

pounds, of lignin derivatives, and of lignin from other sources and as the result of industrial processing is in progress.

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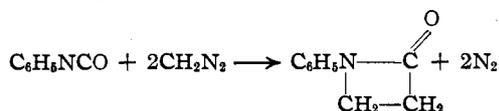
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#### A NOVEL SYNTHESIS OF A $\beta$ -LACTAM

Sir:

In the course of an investigation of amino acid derivatives, a unique synthesis of the  $\beta$ -lactam of N-phenyl- $\beta$ -alanine was discovered. The reaction between an isocyanate and diazomethane does not seem to have been studied previously. We have found that in the case of phenyl isocyanate two methylene units are added in a reaction analogous to the formation of cyclobutane from ketene and diazomethane.<sup>1</sup>



To a solution of 2.38 g. (0.02 mole) of freshly distilled phenyl isocyanate in 25 ml. of anhydrous ether was added 109 ml. of a 0.46 M cold ethereal solution of diazomethane (0.05 mole) dried over sodium. Within about two minutes a vigorous reaction commenced with evolution of nitrogen and the simultaneous deposition of an amorphous orange precipitate. After twenty hours at 0–5° the solution was separated from insoluble material (0.9 g.), and concentrated under reduced pressure to a brown, viscous oil (1.8 g.). Evaporative distillation at 80–120° and 1 mm. gave 0.59 g. (20%) of the colorless crystalline  $\beta$ -lactam of N-phenyl- $\beta$ -alanine, m. p. 74–76°. An analytical

(1) P. Lipp and R. Köster, *Ber.*, **64**, 2823 (1931).

sample, recrystallized twice from acetone–isooctane, was obtained as colorless glistening platelets, m. p. 78–79°.

*Anal.* Calcd. for  $\text{C}_9\text{H}_9\text{ON}$ : C, 73.43; H, 6.16; N, 9.52; mol. wt., 147.2. Found: C, 73.20; H, 6.23; N, 9.58; mol. wt., 139 (Rast).

A suspension of the lactam (0.5 g.) in 5 ml. of *N* sodium hydroxide was warmed with shaking to 70–80°. After ten to twelve minutes the solution became completely clear. From the cooled acidified solution crystalline N-phenyl- $\beta$ -alanine (0.25 g., m. p. 59–60°) was recovered by ether extraction, evaporative distillation, and crystallization from carbon disulfide–ligroin. No depression in melting point was observed for a mixture with authentic N-phenyl- $\beta$ -alanine (m. p. 58–59°), prepared from aniline and  $\beta$ -iodopropionic acid by the method of Bischoff and Mintz.<sup>2</sup>

This appears to be the simplest  $\beta$ -lactam reported; other known representatives of this class of compounds have at least two substituents on the ring.<sup>3</sup> It is of interest to note the ease of hydrolysis in the present case as compared to more highly substituted  $\beta$ -lactams or normal amides. The reaction between diazomethane and other isocyanates and isothiocyanates has been investigated and will be reported shortly.

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(2) C. A. Bischoff and N. Mintz, *Ber.*, **25**, 2351 (1892).

(3) H. Staudinger and S. Jelagin, *ibid.*, **44**, 373 (1911); Staudinger, *ibid.*, **50**, 1038 (1917); H. Breckpot, *Bull. soc. chim. Belg.*, **32**, 424 (1923); H. Gilman and M. Speeter, *THIS JOURNAL*, **65**, 2255 (1943).