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### THE REACTION OF 1,2-BIS(2-PIPERIDYL)ETHYLENE WITH PHOSGENE

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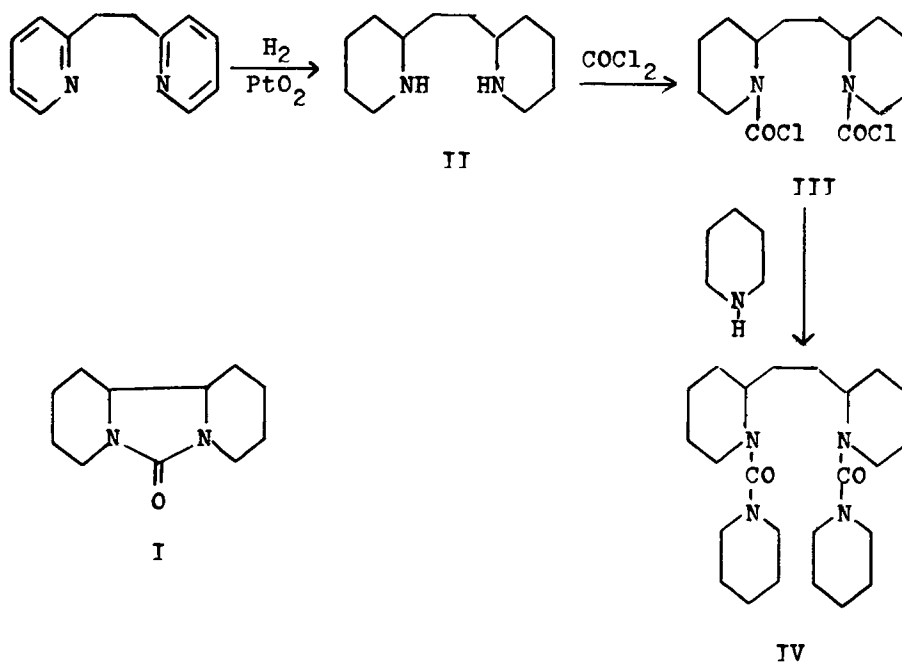
THE REACTION OF 1,2-BIS(2-PIPERIDYL)ETHYLENE WITH PHOSGENE

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It has been reported<sup>1</sup> that 2,2'-bipiperidine reacts with phosgene to give I. In an attempt to extend this synthesis to diazepines, 1,2-bis(2-piperidyl)ethylene (II) was reacted with phosgene under a variety of conditions to give the carbamoyl chloride III as the only product isolated. Reaction of III with piperidine gave the diurea IV.



## EXPERIMENTAL

All melting points were taken in capillaries and are corrected.

Analyses by Spang Microanalytical Laboratory, Ann Arbor, Mich.

1,2-Bis(2-piperidyl)ethylene (II). - A mixture of 25.0 g (0.137 mole) of 1,2-bis(2-pyridyl)ethylene<sup>2</sup> and 1.1 g of platinum oxide in 87 ml of glacial acetic acid and 62 ml of concentrated hydrochloric acid was hydrogenated (40 p.s.i.) in a Parr apparatus. The reaction mixture was filtered and made strongly basic with sodium hydroxide solution. The solid which formed was filtered to give 24.2 g (90%) of II, m.p. 107-108° (from low boiling petroleum ether). IR(KBr): 3215, 1150 cm.<sup>-1</sup>.

Anal. Calcd. for C<sub>12</sub>H<sub>24</sub>N<sub>2</sub>: C, 73.40; H, 12.30; N, 14.26. Found: C, 73.43; H, 12.23; N, 14.21.

Reaction of II with Phosgene. - A cold solution of 1.1 g (0.011 mole) of phosgene in 25 ml of toluene was added dropwise over 10 min. to an ice cooled solution of 1.12 g (0.0057 mole) of II and 2.15 g of triethylamine in 25 ml of toluene. The mixture was then refluxed for 2 hr, washed with water, dried over magnesium sulfate, and concentrated in vacuo to give a brown oil. Trituration with petroleum ether gave 1.17 g (64%) of III, m.p. 116-117° (from low boiling petroleum ether). Treatment with alcoholic silver nitrate gave an immediate white precipitate. IR(KBr): 1730 cm.<sup>-1</sup>.

Anal. Calcd. for C<sub>14</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>: C, 52.34; H, 6.91; N, 8.72. Found: C, 52.63; H, 6.81; N, 8.88.

## THE REACTION OF 1,2-BIS(2-PIPERIDYL)ETHYLENE WITH PHOSGENE

III was the only product isolated from a similar reaction involving equimolar quantities of phosgene and II. Ethyl carbonate did not react with II under the above conditions.

Reaction of III with Piperidine. - A solution of 1.04 g (12.2 mmole) of piperidine in 10 ml of toluene was added dropwise over 5 min. to 0.55 g (1.7 mmole) of III in 25 ml of toluene. The mixture was refluxed for 1.5 hr and allowed to stand at room temperature for 21 hr. Filtration gave 0.38 g of piperidine hydrochloride. The filtrate was washed with water, dried over magnesium sulfate and concentrated in vacuo to give a brown oil which crystallized on standing. Recrystallization from low boiling petroleum ether gave 0.40 g (60%) of IV, m.p. 102.5-104.5°. IR(KBr): 1635  $\text{cm}^{-1}$ .

Anal. Calcd. for  $\text{C}_{24}\text{H}_{42}\text{N}_4\text{O}_2$ : C, 68.86; H, 10.11; N, 13.38. Found: C, 68.99; H, 9.98; N, 13.33.

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