

Gm. of 95% purity (0.21 mole), was crushed and suspended in a mixture of 150 ml. of absolute ether and 50 ml. of absolute tetrahydrofuran contained in a 2-L., three-necked, round-bottom flask fitted with a dropping funnel, stirrer, condenser, and drying tube. The suspension was heated under reflux for 1 hour, cooled and 10.2 Gm. (0.05 mole) of 3-aza-1,5-dicyano-6,6-dimethyl-2,4-dioxo-bicyclo-(3.1.1)-heptane dissolved in 350 ml. of absolute tetrahydrofuran was added with stirring. After the addition was completed, stirring and reflux were continued for 9 hours. The reaction mixture was cooled and the complex decomposed by the cautious addition of 9 ml. of water, 9 ml. of 15% sodium hydroxide solution, and 27 ml. of water. The granular precipitate that had formed was filtered and washed with ether, the washings being added to the previous filtrate which was then dried with anhydrous sodium sulfate. The solvent was removed under reduced pressure and the residue of 3.4 Gm. distilled. The fraction boiling between 109–110°/0.16 mm. was collected and weighed 1.8 Gm. (20%).

The distilled material solidified at room temperature and attempts to prepare solid derivatives from it resulted in hygroscopic compounds which could not be purified.

**3-Aza-1,5-diaminomethyl-6-ethyl-6-methyl-bicyclo-(3.1.1)-heptane Tripicrate.**—Reduction of the bicyclo Guareschi imide derived from 2-butanone yielded 16% of the product. However, the material was too hygroscopic to have analyzed. A picrate derivative was readily formed but decomposed into picric acid and the triamine when recrystallization from conventional solvents was attempted. A stable tripicrate was prepared by dissolving 0.5 Gm. of the triamine in 1 ml. of absolute ethanol and slowly adding 22 ml. of a saturated solution of picric acid in absolute ethanol. The precipitated derivative was collected and purified by washing it with chloroform and decanting the solvent. This process was repeated six times using ethanol as the washing

medium and finally the solid derivative was dried in a vacuum, m.p. 245–248° (decompn.).

#### SUMMARY

1. Five Guareschi imides were prepared by known procedures.
2. Selective reduction of the imide grouping of the Guareschi imides with lithium aluminum hydride uniformly resulted in ether-insoluble semisolids from which no pure product could be isolated or characterized.
3. Total reduction of the imide grouping and the nitrile groups of the Guareschi imides gave similarly poor results.
4. A series of four bicyclo Guareschi imides was prepared from the monocyclic Guareschi imides.
5. The four bicyclo Guareschi imides prepared readily underwent N-methylation.
6. Lithium aluminum hydride reduction of the four bicyclo Guareschi imides and their N-methyl analogs yielded the expected totally reduced products.

#### REFERENCES

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#### ERRATUM

In the paper titled "Proposed Method of Assay for Diaphene" (1), the formula for calculating the per cent of Diaphene in paragraph three at page 45 should read:

$$\text{per cent Diaphene} = \frac{A \times 50 \times 100}{I \times V \times W} \times 100$$

(1) Soliman, S. A., and Harris, L. E., *THIS JOURNAL*, **52**, 43(1963).