PART V

Attempted synthesis of thiazolo-benoxadiazine derivatives (p. 126-130)

76. Attempted condensation of o-aminophenol(CXXI)
with 2-chloro-4-phenyl-thiazole; attempted formation
of 2-(o-hydroxy anilino)-4-phenyl thiazole(CXXIX)

o-Amino phenol (1.09 g, 0.01 mole) and 2-chloro-4-phenyl
thiazole (1.95 g, 0.01 mole) in phenol (5 ml.) and conc. HCl
3-4 drops were heated in an oil bath at 120-140° for 4-6 hours.
Anhydrous ether was then added to remove phenol and the
remaining semi solid sticky product treated with sodium carbonate
solution and then washed with water. But repeated attempts to
procure a pure sample failed.

77. Preparation of o-hydroxy-phenyl thionures (CXXIII)

This was prepared by reacting together o-aminophenol
and potassium sulphonylureide in presence of hydrochloric acid,
crystallised from hot water, m.p. 161°(192).

78. Condensation of o-hydroxy-phenyl thionures with -bromo
oestophenones (CXXIII); formation of 2(o-hydroxy anilino)
4-(p-substituted phenyl) thiazoles (CXXIX)
General Procedure

A mixture of equivalent amounts of o-hydroxy phenyl thiourea and appropriate ω-halo ketones was heated in ethanol on a steam bath. Separation of a crystalline product started within 15 minutes. It was refluxed for another 15 minutes cooled and product collected by suction. These were crystallised from appropriate solvent (Table XIV, p. 123).

79. Attempted ring closure of 2(2'-hydroxy anilino)thiazoles (CXXIX)

1) To a known weight of (CXXIX) in dry ether was added excess of silver oxide and the mixture gently refluxed for 10 hours. On working up only the unchanged 2(2'-hydroxy anilino)thiazole was recovered in quantitative yield.

2) To a known weight of (CXXIX) dissolved in hot acetic acid in an iodine flask, a known excess volume of 0.1 N potassium bromate - potassium bromide solution was added. To this was then added a few ml. of 5 per cent potassium iodide solution to avoid any loss of bromine. The flask was heated on a water bath at 40° for 2 hours. To the contents was added 30 ml. 5 % potassium iodide and the iodine liberated thus, was titrated against standard sodium thiosulphate solution.

Each mole of the compound consumed equivalent to three mole of bromine. However, the product of bromination could not be crystallised.