the 2nd Internal Clinic of this University. The classification of patients was pulmonary tuberculosis with cavity, bronchitis, and bronchial asthma. Average daily dose of the drug administered orally was 1~2 mg. for the children and 4~6 mg. for adults. The classification of the effect was as follows: For pulmonary tuberculosis (17 patients), remarkably effective (5/17), effective (12/17), ineffective (0/17); for bronchial asthma and bronchitis (20 patients), remarkably effective (16/20), effective (1/20), ineffective (3/20). It seemed that 6 mg. is sufficient to maintain the effect for a day for adults. The antitussive effect of the drug (3~4 mg.) was compared with those of Dextromethorphan (40 mg.) and codeine (60 mg.) in 17 tuberculosis patients. The effect was the most marked with Win-1161-3, followed by Dextromethorphan and codeine. No side-effect (especially tolerance and cumulation) was observed in 4 tuberculosis patients who were given 6 mg. daily dose of the drug for 40~50 days.

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Shigehiko Sugasawa and Seiichi Takano: 2-(2-Pyridyl)indole Methiodide.**

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By heating 2-(2-pyridyl)indole with methanolic methyl iodide solution in a sealed tube at 100° for two hours Sugasawa, *et al.*[1] obtained a crystalline product. When purified from hydrous ethanol this compound formed yellow needles of m.p. 194°(decomp.), having U. V. $\lambda_{max}^{50\%EtOH}$ at 325 mμ(log ε 4.34), which they took for the methiodide of the original base but was later found to be erroneous.

The methiodide of 2-(2-pyridyl)indole was now found to be conveniently prepared by heating the indole with pure dimethyl sulfate in benzene solution to form the methyl methosulfate of the base and then treating it with potassium iodide. The methiodide thus prepared came as faint yellow needles of m.p. 227~228° (decomp.), having U. V. $\lambda_{max}^{95\%EtOH}$ at 252 mμ (log ε 3.95) and 373 mμ (log ε 4.19).

According to the suggestion of Dr. Swan the compound of m.p. 194°(decomp.) obtained by Sugasawa, *et al.* was reinvestigated.

This compound was now obtained as a crystalline solid, which formed faint yellow needles of m.p. 197~199°(decomp.) with the same U. V. maximum as was described before, and was now found to be a mixture of the methiodide and the hydriodide of 2-(2-pyridyl)indole. This mixture could not be separated through crystallization from a variety of solvents, but when hydrous methanolic solution of this compound was basified with sodium hydrogen carbonate, there separated a basic substance, which could be collected in benzene. From this solution colorless rhombic pillars of m.p. 152~153° were recovered, which was found to be identical with 2-(2-pyridyl)indole.

The aqueous layer was extracted continuously with chloroform and thus a compound of m.p. 225~226°(docomp.) was recovered, which was proved to be the methiodide of 2-(2-pyridyl) indole by direct comparison with an authentic specimen prepared as above.

When roughly equal portions of the methiodide and the hydriodide of the base

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** This paper concerns a correction of the erroneous description of 2-(2-pyridyl)indole methiodide published in this Bulletin, **4**, 16(1956) by Sugasawa *et al.* One of the present writers (S.S.) is grateful to Dr. G.A. Swan, Chemistry Department, King's College, University of Durham, Newcastle-upon-Tyne, England, who drew his attention to this error and gave him an opportunity to make a correction by his own hands.

1) S. Sugasawa, M. Terashima, Y. Kanaoka: This Bulletin, **4**, 16(1956).

were mixed and heated, the mixture melted at 196~199° with decomposition.

It is well known that 2-substituted pyridine derivatives usually are resistant to the formation of quaternary salts. Therefore, under such a strenuous conditions as had been adopted by Sugasawa, *et al.* some of methyl iodide suffered hydrolysis, forming hydriodic acid, which combined with 2-(2-pyridyl)indole to form the hydriodide of the base.

Experimental

Methiodide of 2-(2-Pyridyl)indole—The base (150 mg.) in dehyd. benzene (10 cc.) was mixed with freshly purified Me_2SO_4(100 mg.) and the whole was refluxed on a steam bath for 5 hrs. On evaporating benzene there remained a solid substance, which was dried on a porous plate. The crude methyl methosulfate of the indole base thus obtained was a white solid of m.p. 127~128°, which was not hygroscopic and was directly converted to the methiodide. The yield was 180 mg. or 81% of the crude methosulfate salt.

The methosulfate was dissolved in hydr. MeOH, to which solution cold saturated aq. solution of KI (150 mg.) was added, causing a yellow solid substance to separate. After some time this was collected and purified from MeOH, forming faint yellow needles of m.p. 227~228°(decomp.); U.V. $\lambda_{max}^{95\%\ EtOH}$ mμ (log ε): 252 (3.95), 373 (4.19). *Anal.* Calcd. for $C_{14}H_{13}N_2I$: C, 50.0; H, 3.5; N, 8.3. Found: C, 49.7; H, 3.8; N, 8.1.

Separation of the Compound of m.p. 197~199° into Its Two Components—100 mg. of this substance was dissolved in a warm mixture of 4 cc. of MeOH and 10 cc. of H_2O, giving a faint yellow clear solution. To this solution was now added 10% aq. solution of $NaHCO_3$ to distinct alkalinity, causing turbidity. The mixture was shaken with benzene, the benzene layer separated was washed with water, dried, and the solvent was evaporated, leaving a solid substance, which was purified from EtOH, forming colorless rhombs of m.p. 152~153°. The melting point was not depressed when admixed with an authentic sample of 2-(2-pyridyl)indole. The yield of 42 mg. of this base means that ca. 70% of the original substance consisted of the hydriodide of 2-(2-pyridyl)indole.

The faint yellow clear aq. layer was continuously extracted with $CHCl_3$ for 35 hrs. When $CHCl_3$ solution was cooled there separated faint yellow needles of m.p. 225~226°(decomp.), which was identified with an authentic specimen of the methiodide of 2-(2-pyridyl)indole by direct comparison.

The hydriodide of 2-(2-pyridyl) indole was prepared from the base and hydriodic acid. When purified from EtOH this salt formed faint yellow needles of m.p. 207~209°, which on admixture with the methiodide of the base melted at 197~199° with decomposition.

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