Experimental[4]

Extraction and Isolation of the Alkaloids from *Magnolia parviflora* Sieb et Zucc.—1.3 kg. of the undried bark of *M. parviflora* was coarsely ground, extracted 3 times with warm MeOH, and MeOH extract was treated by the usual method. The quaternary bases present were precipitated as the reineckate, then as the mercuric chloride salt, and finally as the styphnate. The styphnate was obtained as yellow microneedles, m.p. 230~231°(decomp.), from a mixture of water and acetone, and was proved by infrared spectrum to be identical with magnoflorine styphnate. Yield, 10 g. *Anal.* Calcd. for $C_{20}H_{24}O_4N \cdot C_6H_2O_8N_3$: C, 53.24; H, 4.47; N, 9.55. Found : C, 53.21; H, 4.63; N, 10.10.

The iodide formed from MeOH, colorless pillars, m.p. 249°(decomp.), and this was also shown by infrared examination to be identical with magnoflorine iodide. *Anal.* Calcd. for $C_{20}H_{24}O_4NI$: C, 51.18; H, 5.15. Found : C, 51.06; H, 5.12.

The syrupy mother liquor left after separation of magnoflorine styphnate was decomposed with hydrochloric acid and the liberated styphnic acid eliminated by means of ether. The aqueous layer was then treated with lead subacetate, and the filtrate was freed from excess Pb with H_2S. The filtered solution was evaporated to dryness *in vacuo* and the residue was induced to crystallize as the picrate. It formed from a mixture of acetone and EtOH yellow needles, m.p. 181~182°, undepressed on admixture with magnocurarine picrate. Yield, 2.6 g. *Anal.* Calcd. for $C_{19}H_{24}O_3N \cdot C_6H_2O_7N_3$: C, 55.35; H, 4.83. Found : C, 55.09; H, 5.11.

Summary

Two quaternary alkaloids, magnoflorine (I) and magnocurarine (II) were identified from the bark of *Magnolia parviflora* Sieb. et Zucc.

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[4] All melting points are uncorrected. We are thankful to Dr. K. Hozumi, Mr. K. Imaeda, and Miss F. Tanase of the Central Analysis Room of this Institute for the microanalytical data.

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Tatsuhiko Nakano and Mikio Uchiyama : Studies on the Alkaloids of Magnoliaceous Plants*. XVIII.[1] Alkaloids of *Magnolia Kobus* DC. var. *borealis* Koidz.

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Previous investigation[2] disclosed that the bark of *Magnolia Kobus* DC. (Japanese name "Kobushi") contains salicifoline as the quaternary base. Our interest has been directed to the alkaloids of *Magnolia Kobus* DC. var. *borealis* Koidz. (Japanese name "Kita-Kobushi" or "Ezo-Kobushi"), the variety of the same species.

The plant material used in this investigation was made available in November, 1955, in the precincts of the Kanazawa Castle, Ishikawa Prefecture, and the methanolic extract of its bark was worked up in the usual manner, whereby a quaternary base was isolated as the picrate of orange yellow prisms, m.p. 180~181°. The empirical formula represents the composition of salicifoline (I) picrate, and the two substances

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* This constitutes a part of a series entitled "Studies on the Alkaloids of Magnoliaceous Plants" by Masao Tomita.

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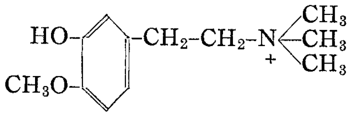
[1] Part XVII. T. Nakano, M. Uchiyama : This Bulletin, **4**, 392(1956).

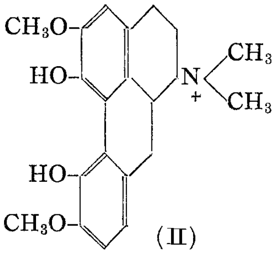
[2] M. Tomita, T. Nakano : J. Pharm. Soc. Japan, **72**, 727(1952).

[3] M. Tomita, T. Nakano : *Ibid.*, **72**, 197, 281(1952).

showed no depression of the melting point on admixture, thus confirming them to be identical. From the mother liquor remaining after removal of salicifoline picrate, another quaternary alkaloid was obtained as the styphnate, $C_{20}H_{24}O_4N \cdot C_6H_2O_8N_3$, of yellow needles, m.p. 230~231° (decomp.), which was shown by infrared spectral determination to be identical with magnoflorine[4] (II) styphnate.



(I)



(II)

The occurrence and distribution of the alkaloids of *Magnolia Kobus* DC. VAR. *borealis* KOIDZ. and *M. Kobus* DC. are compared in Table I, which shows that the latter furnishes a higher percentage of salicifoline than the former although it is devoid of magnoflorine.

TABLE I.

	Salicifoline (I)	Magnoflorine (II)
M. Kobus DC. VAR. *borealis* KOIDZ.	+ (0.04% as the chloride)	+
M. Kobus DC.	+ (0.4% as the chloride)	−

We wish to acknowledge our indebtedness to Prof. Tomita for his interest in this work, and to Prof. Kurono and Assistant Prof. Kimura for providing us with the plant material used in this investigation. We also wish to express our thanks to Messrs. Matsui and Narisada of the Research Laboratory, Shionogi & Co. Ltd., for carrying out the infrared spectral determinations and to the Ministry of Education for a grant in aid of expenses of this work.

Experimental[5]

Extraction and Isolation of the Alkaloids from *Magnolia Kobus* DC. VAR. *borealis* KOIDZ.—1.7 kg. of the undried bark of the plant material was extracted 3 times with boiling MeOH and the isolation of the quaternary bases from this MeOH extract was effected by the hitherto described procedures, whereafter a quaternary base was obtained as its picrate forming from acetone orange yellow prisms, m.p. 181~182°. Yield, 1.2 g. A mixed melting point with salicifoline picrate showed no depression. *Anal.* Calcd. for $C_{12}H_{20}O_2N \cdot C_6H_2O_7N_3$: C, 49.31; H, 5.06. Found: C, 49.03; H, 5.11.

The uncrystallizable mother liquor left after separation of salicifoline picrate was decomposed with HCl, and the liberated picric acid removed by extraction with ether. The aqueous layer was evaporated *in vacuo* to dryness and treated with aq. sodium styphnate. The styphnate was obtained from acetone as yellow needles, m.p. 230~231°(decomp.), which was shown by infrared spetrum to be identical with magnoflorine styphnate. Yield, 0.2 g. *Anal.* Calcd. for $C_{20}H_{24}O_4N \cdot C_6H_2O_8N_3$: C, 53.24; H, 4.47. Found: C, 53.49; H, 4.75.

Summary

Two quaternary alkaloids, salicifoline (I) and magnoflorine (II), were isolated from the bark of *Magnolia Kobus* DC. var. *borealis* KOIDZ.

(Received May 28, 1956)

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[4] T. Nakano: This Bulletin, **2**, 326, 329(1954).

[5] All melting points are uncorrected. We are indebted to Dr. K. Hozumi, Mr. K. Imaeda, and Miss F. Tanase of the Central Analysis Room of this Institute for the microanalyses reported herein.