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Microchemical Investigation of Medicinal Plants. VII*

Proposed Structure and Crystal Forms of Yellow Compound I from the Catalpa Seed

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With 2 Figures

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Introduction

In previous communications1–3 it has been reported that microchemical investigation of the seeds of C. bignonioides resulted in the isolation of p-hydroxybenzoic acid and an unknown yellow solid designated as Yellow compound I. By means of organic elemental and functional group analysis4, as well as mass spectrometry, the empirical formula of Yellow Compound I was shown to be C_{17}H_{14}O_{6} with two hydroxyl and two methoxyl functions in the molecule. The IR and NMR spectra indicated the presence of the carbonyl function on a 6-membered ring besides the cyclic ether linkage for one of the oxygen atoms.

In the present paper we report the continuing work on Yellow Compound I. The procedure for isolating this compound has been simplified with concomitant increase in yields. Two crystal forms have been observed. Based on its chemical reactions and spectral characteristics, and through

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* For Part VI see Mikrochim. Acta [Wien] 1969, 1100. Inquiries and requests for reprints of this series of papers should be addressed to Prof. T. S. Ma, Department of Chemistry, City University of New York, Brooklyn, N. Y. 11210, U. S. A.

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literature research\textsuperscript{5-7}, we submit that Yellow Compound I is the flavonoid 5,6-dihydroxy-7,4'-dimethoxyflavone, previously unknown as a natural product.

![5,6-Dihydroxy-7,4'-dimethoxyflavone](image)

**Experimental**

*Isolation of the Yellow Compound*

The procedure previously described\textsuperscript{1} can be modified as follows in order to obtain Yellow Compound I from catalpa seeds without prior defatting. 50 g of the pulverized seeds are transferred into a bag made of thin cloth which is placed in a 1.5-liter beaker containing 400 ml of water at 35-40°C. The bag is open to permit free stirring of the seeds so that a paste results; it is then closed and the neck is twisted to express the extract. This process is repeated 4 times.

The combined aqueous extract is filtered, and 50 ml of conc. HCl are added to the filtrate. The solution is then heated over a steam bath for 1 hour, allowed to cool, and set aside for 24 hours. The dark brown precipitate so formed is then collected on Büchner funnels using Whatman No. 1 filter paper and washed thoroughly with water to remove the acid. After being dried under vacuum, the residue is extracted with 4 x 200 ml of boiling diethyl ether. The combined yellow extract is filtered to remove ether-insoluble materials and subsequently concentrated to yield Yellow Compound I (43–52 mg).

*Preparation of the Crystals*

When the yellow ethereal solution is allowed to evaporate slowly, needle shaped amber-color crystals are formed (see Fig. 1). Recrystallization from anhydrous methanol, however, produced crystals in the form of thin flat rhombic plates (Fig. 2). There is no significant difference in melting point between these two crystal modifications.

**Discussion**

Yellow Compound I is a stable substance which can be obtained from fresh pods and from dry catalpa seeds even after long storage. Its structure as 5,6-dihydroxy-7,4'-dimethoxyflavone was deduced on the basis of the following evidence.