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## A STUDY ON A NEW ACIDIC COMPOUND ISOLATED FROM VOLATILE PORTION OF A GREEN ALGA, *ULVA PERTUOSA*

TERUHISA KATAYAMA AND TETUO TOMIYAMA

It has long been noticed that sea weeds on drying generally give off a particular odor just like so-called "isonoka" in apan, which is smelled over every seashore. In the course of isolating this odorous ingredient from *Ulva pertuosa*, a compound of an acidic nature has been isolated. The analyses of its derivatives have shown that the compound has been hitherto unknown. The compound, accordingly, has been named as ulvaic acid.

The present paper deals with the method for isolation of reduced ulvaic acid and the determination of its molecular formula. The study of the constitution of ulvaic acid is under progress.

#### I. METHOD FOR ISOLATION OF ULVAIC ACID

The procedures finally adopted for the isolation of ulvaic acid are as follows. The distillate which has been obtained by subjecting the dried *Ulva* to the steam distillation is extracted with ether. The ethereal layer is shaken with three per cent sodium hydroxide, washed with dilute hydrochloric acid and then with water. The ethereal layer contains a neutral compound having a specific odor. The alkaline aqueous solution having some weak odor is acidified with dilute hydrochloric acid and then extracted with ether. The ethereal extract containing ulvaic acid, on catalytic reduction with hydrogen and palladium black, yields some white crystalline matter. The yield of the reduced ulvaic acid was about 0.0002 per cent of dried *Ulva*. The fractional crystallization from ethanol gave two kinds of crystal which are named for the time being as

crystal X and Y. The yield of crystal Y was very small in most cases compared to that of crystal X.

#### II. PROPERTIES AND ANALYSIS OF CRYSTAL X

The crystal has no smell and shows a white leaf-shaped crystal form which usually combines forming a flower-like conglomerate (Fig. 1). It is insoluble in water, whereas easily soluble

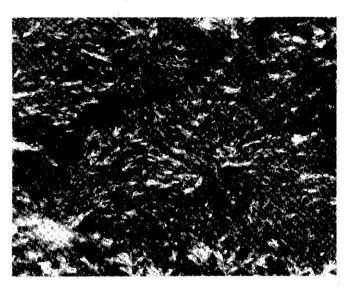


Fig. 1. Crystal X. × 90.

in ether, petroleum ether, 96% hot ethanol, pyridine, and dioxan, and slightly soluble in 96% cold ethanol or acetone. After several recrystallization from ethanol, it shows a constant melting point of 54.5–55.0°C. It dissolves readily in caustic alkaline solution. On adding to this solution a salt of silver, lead, copper, barium, calcium, or magnesium, a sparingly soluble salt in water precipitates.

The molecular weight was determined as shown in the following data to be about 290 by the Rast camphor method, by determining its acid radical, and by analyzing its barium salt.

1) The weights of sample and the camphor were 14.92 mg and 162.30 mg, respectively. The depression in the melting

- point of camphor was observed to be 11.0°C. Hence the molecular weight can be calculated to be 306.
- 2) 12.07 cc of 0.0135 N alcoholic potash solution were required for the neutralization of 46.16 mg of the sample. The molecular weight was calculated to be 284.
- 8) 13.46 mg of barium salt yielded 3.92 mg of barium carbonate on incineration. The molecular weight was calculated to be 281.

Since the crystal does not contain water of crystallization, nitrogen, halogene, phosphorus and sulfur, an experimental formula of  $C_6H_{12}O$  was obtained by the elementary analysis as will be seen in the following data.

Sample taken (mg)	found		calculated			atomic ratio		
	CO <sub>2</sub> (mg)	H <sub>2</sub> O (mg)	C (%)	H (%)	0 (%)	С	Н	o
19.32	51.45	20.95	72.63	12.15	15.22	6.03	12.0	0.97
18.90	50.00	20.50	72.15	12.14	15.71			
calculated for C <sub>6</sub> H <sub>12</sub> O		1.05	71.93	12.08	15.99	1 10 25		

It follows, accordingly, that the molecular formula of crystal X is  $C_{18}H_{38}O_3$ . Because of the fact that the crystal yields salts of various metals, an acidic radical might be a carboxyl group. Determination of the form of one more oxygen in the molecule needs a further experiment.

#### III. PROPERTIES AND ANALYSIS OF CRYSTAL Y

Crystal Y is a white needle crystal (Fig. 2). The solubility in various solvents is qualitatively the same as crystal X. It is, however, less soluble in 96 per cent ethanol than crystal X. It melts at 64.5–65.0°C. The alkaline metal salt is soluble in water, but salts of heavy metal and alkaline earth metal are sparingly soluble.

The mixture of 7.53 mg of the sample and 84.43 mg of camphor shows the depression of 11.0° which corresponds to the molecular weight of 324. Since for neutralization of 9.01 mg of sample was required 1.76 cc of 0.0167 N alcoholic potash, the molecular



Fig. 2. Crystal Y. × 90.

weight should be 306. It will be presumed from this data that crystal Y may be an isomer of crystal X or a preexisting saturated compound similar to crystal X.

#### IV. SUMMARY

- 1) A new acidic compound which is named as ulvaic acid has been isolated from steam distillate of *Ulva pertuosa*.
- 2) The molecular formula of the crystalline reduced ulvaic acid has been determined to be  $C_{18}H_{36}O_3$ .
- 3) Two modifications exist for the reduced ulvaic acid. One of them might be an isomer or a preexisting staturated compound similar to ulvaic acid.

In conclusion the authors wish to express their thanks to Mr. T. Kondo of the laboratory of wood chemistry for his aid in carrying out the elementary analysis. This study was carried out in part with the aid of a grant from the Research Fund of the Ministry of Education.

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