a hardened filter paper or a funnel with a sintered glass disc, washed with water, and pressed in order to squeeze out most of the water. It is not necessary to wash it free from $SO_4^{=}$. For the subsequent step, the wet cake, weighing from 109–143 g., is used.

2-(4-Hydroxybenzoyl)-benzoic Acid.—The wet, yellow cake of "phenolphthalein oxime" is placed into a flask or a beaker of 500 ml., fitted with a stirrer and a thermometer. One-hundred ml. 5 *N* of sulfuric acid, prewarmed to 90–104°, is then added in such a way as to wash down any oxime adhering to the neck of the flask or the side of the beaker. The stirrer is started and the yellow slurry rapidly heated. Within three minutes after the temperature has reached 96–102°, a dark yellow solution is formed. After one to three minutes from this point on, the solution first becomes turbid and then crystals of the acid appear. The reaction mixture is stirred for an additional ten minutes at 97–103°, gradually becoming brown. After cooling to 20°, the sandy crystals of 2-(4-hydroxybenzoyl)-benzoic acid are filtered off and washed with three portions of 20 ml. of water. After drying at 100–120°, the acid, of moderate greenish yellow color, weighs from 22.1–23.0 g. and melts at 209–213° cor. (sl. dec.); (yield 91.3–95.0% based on the phenolphthalein).

If a purer product is required, 20 g. of the acid, 0.5 g. of decolorizing carbon (Norit-A) and 1 liter of water are refluxed for fifteen minutes, filtered through a preheated funnel and the filtrate cooled to 5°. From 18.0–18.4 g. of acid, which melts at 211–215° cor. (sl. dec.), is obtained. This purified acid dissolves in ethanol (1 g. in 20 ml.) with but faint yellow color; its solution in 0.1 *N* sodium hydroxide (0.2 g. in 20 ml.) is clear and either light yellowish or faintly red.

From the filtrate of the acid, together with 75 ml. of wash-water, there can be obtained, after neutralization with 80 ml. of 5 *N* sodium hydroxide and about 60 ml. of 20% sodium sulfite solution and subsequent extraction with ether, 8.6–9.6 g. of *p*-aminophenol (m. p. 182–187°).

Equally good yields are obtainable with batches from 0.5 to 1.0 mole of phenolphthalein.

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RECEIVED JANUARY 3, 1946

Habit Modification of Ammonium Oxalate Monohydrate Crystals During Growth from Solution

BY HARRY J. KOLB[1] AND JOSEPH J. COMER[2]

In a previous paper,[3] the habit modification of ammonium dihydrogen phosphate was described. Additional investigations on the habit modification of ammonium oxalate monohydrate are presented here.

Procedure.—Ammonium oxalate monohydrate crystals were grown in the large water-bath described previously.[3] Each of the tanks was filled with 7.5 liters of ammonium oxalate solution saturated at 40°. Two crystal seeds, about 0.3 sq. cm. in the *z*-plane and 2 cm. along the *z*-axis, were suspended in each tank and grown, as before, by reduction of the temperature in the thermostat until the seeds were 4-cm. long. The cations which were added to the saturated oxalate solution containing the growing crystals are the eight listed in Table I. The concentrations of the cations were 0.1 g./liter of solution. Modification tests were also made at the following concentrations of magnesium ion: 0.001, 0.005, 0.01, 0.03, 0.05, 1.0, 2.0, 5.0 g./liter.

Results.—The ammonium oxalate monohydrate crystals normally show the (001), (110) and (010) planes. The modifications of the growing crystal by the cations are listed in Table I, *i. e.*, development of (021), (110) and (111) planes.

TABLE I

Development of planes	Zn^{++}	Cu^{++}	Mg^{++}	Sc^{+++}	Co^{++a}	Ni^{++a}	Cd^{++}	Mn^{++}
(110)	+	0	0	0	0	0	+	+
(111)	+	+	+	+	0	0	0	0
(021)	+	+	+	+	+	+	0	0

[a] Crystal tapered toward *z*-axis, obscuring the identity of individual planes.

In the series of experiments using magnesium ion, the (021) and $(0\bar{2}1)$ planes became more fully developed as the metal ion concentration increased until the (001) plane was finally eliminated. At the magnesium ion concentration of 5 g./liter, the growth of the crystal seed was completely inhibited.

The habit modification may be due to adsorption of foreign metal ions at the crystal–solution interface. This adsorption with subsequent habit modification could be explained by the insolubility interpretation (oxalate in this case) suggested in the previous paper.[3]

The oxalate reticular densities of the crystal planes were calculated and found to be (110), 3.54; (010), 2.87; (111), 1.83; (021), 1.71; (001), 1.06. All are relative to the (011) plane as 1.00. Thus, the appearance of the (021) plane rather than the (011) plane could be accounted for by the density sequence. The removal of the (001) plane could also be explained. In cases where the (110) plane was not developed (given in the table), no growth was noted on either the (110) or (010) planes. The cessation of all growth at high foreign cation concentrations supports a strongly adsorbed ion interpretation. However, it must be stated that we have no direct evidence of an adsorbed metal ion layer.

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RECEIVED SEPTEMBER 24, 1945

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[3] Kolb and Comer, THIS JOURNAL, **67**, 894 (1945).

The Molecular Structure of Trimethyl Silicon Chloride

BY R. L. LIVINGSTON[1] AND L. O. BROCKWAY

The silicon–carbon bond distances have been determined in several compounds and found to be less than the value of 1.93 Å. obtained for tetramethyl silicon.[2] For example, in hexamethyl disilicon[3] and in SiC,[4] the silicon–carbon distance

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[2] L. O. Brockway and H. O. Jenkins, THIS JOURNAL, **58**, 2036 (1936).

[3] L. O. Brockway and N. R. Davidson, *ibid.*, **63**, 3287 (1941).

[4] Ewald and Hermann, "Structurbericht," Akademische Verlagsgesell., 1931, 146.