

## NOTE

**Note on the Purification of Potassium Dihydrogen Phosphate.—**

It is a matter of common observation in chemical laboratories that many solutions deposit sediments on standing in glass bottles for any length of time. Besides being unsightly this phenomenon causes the worker to doubt the purity of his reagents and the actual concentration of the solutions. The fifth molar solution of potassium dihydrogen phosphate used in preparing Clark and Lubs' standard buffer solutions is usually seen to deposit a sediment on the bottom of the stock bottle. Because of the fact that it is of prime importance that the reagents used in the preparation of regulatory buffer mixtures be of exceptional purity and constant composition, some work has been done in this Laboratory to determine the nature of the sediment and factors influencing its deposition.

It has been found that the material of the container has no effect; soft and hard glass, paraffin and silver were used, and all give equal amounts of sediment when an equal amount of solution is placed in each. The deposition is found to be more rapid at higher temperatures and more complete the less the concentration of salt in the solution. At a temperature of about 85° (on a water-bath) the sediment is thrown out completely in about twenty-four hours, while at room temperature the time taken is considerably longer. If the concentration of salt is greater than 0.2 *M* the sediment is found to stay in suspension. Even if three times recrystallized the salt always contains some impurity, for the sediment forming impurity is soluble in hot saturated  $\text{KH}_2\text{PO}_4$  solutions and cannot be removed completely by filtration.

On analysis, the sediment from a 0.2 *M* solution of a "specially purified and standardized" commercial salt has been found to be largely a mixture of colloiddally aggregated aluminum and ferric compounds. The occurrence of aluminum as an impurity was first pointed out to us by the La Motte Chemical Products Company.<sup>1</sup> Sediments from solutions of salts obtained from other sources contained alkaline earth metals in addition. No silicon has been found present in any of the sediments. It is interesting to note that no alkali phosphate salt containing an ammonium ion, for example, sodium diammonium phosphate,  $\text{Na}(\text{NH}_4)_2\text{PO}_4$ , gives a sediment from solution. During the commercial preparation of the salts, the iron and aluminum impurities of the phosphoric acid have probably been thrown out as phosphates. However, one commercial sample of potassium dihydrogen phosphate, of "C. P." quality, was found to be free from any sediment producing impurity. In all, salts from four different sources were examined.

For the purification of a potassium dihydrogen phosphate salt which may

<sup>1</sup> McCrumb, private communication, January, 1928.

contain the impurities mentioned above, the following procedure is recommended. Make up an approximately fifth molar solution of the salt, let it stand in a flask, sealed to prevent excessive loss of water, for twenty-four hours on a water-bath at 75–85°. Then filter through a very retentive paper or asbestos filter and recrystallize by the usual methods the residue left on evaporation. By this means a salt of higher purity can be obtained in one recrystallization than can be obtained by the three recrystallizations from water recommended by Clark.<sup>2</sup> In place of evaporation to complete dryness, the salt may be precipitated from a cold saturated solution (obtained by partially evaporating and cooling the filtered solution) to which is added an equal amount of cold 95% ethyl alcohol. This gives very small crystals which may be used as such or which may be further recrystallized from water.

CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT  
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RECEIVED APRIL 27, 1928  
PUBLISHED JUNE 5, 1928

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## CATALYTIC OXIDATION OF FURFURAL IN THE VAPOR PHASE

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RECEIVED OCTOBER 8, 1927

PUBLISHED JUNE 5, 1928

### Introduction

During the course of recent investigations upon the catalytic oxidation of furfural in aqueous solutions,<sup>1</sup> it was found that by use of various metallic oxide catalysts, different products could be obtained. Among these products were fumaric acid, mesotartaric acid and oxalic acid. The purpose of the present investigation was to determine what products are formed when furfural is oxidized in the vapor state with a similar catalyst.

### Experimental Part

To prepare the catalyst, vanadic acid was precipitated upon asbestos by adding hydrochloric acid to a solution of ammonium vanadate, the asbestos filtered, washed, dried and ignited to free it from ammonium chloride and to convert the vanadic acid into vanadium pentoxide.

The catalytic mass showed a tendency to become less active during the course of the experiments, undoubtedly due to some form of poisoning. For regeneration, it was necessary only to redissolve the vanadium pentoxide in ammonia and reprecipitate with hydrochloric acid.

The furfural used in these experiments was the ordinary commercial article, which is dark colored and contains about 1% of water. It was employed without redistillation.

<sup>2</sup> Clark, "The Determination of Hydrogen Ions," 2nd ed., Williams and Wilkins, Baltimore, Md., 1922, p. 100.

<sup>1</sup> Milas, *THIS JOURNAL*, **49**, 2005 (1927).