

b. The gradient tank may afford a means of studying "habit" formation and also of investigating compounds or combinations of drugs which may produce the desired therapeutic effect with least danger of creating an appetite. Results with fishes may differ from those obtained with man, but they may readily afford information which can serve to interpret results with higher forms.

ZOOLOGICAL LABORATORIES,  
UNIVERSITY OF ILLINOIS.

---

## RAPID GENERAL ASSAY METHOD FOR ALKALI SALTS OF ORGANIC ACIDS.

BY A. B. LYONS.

Ignition of salts of organic acids is a tedious operation, even if one is satisfied merely with thorough carbonization of the organic compound. In the official general assay process (U. S. P. IX, p. 589) this is all that is required, yet the plan of dissolving out from the carbonized residue the alkali carbonate with aid of volumetric acid and heat, filtering out the carbon and washing out all the residual acid, calls for a considerable expenditure of time and labor.

It has been suggested by E. Elvove that conversion of the salt into a *sulphate* is to be preferred to simple ignition resulting in the formation of a carbonate, and no doubt equally good results can be obtained by this procedure. There is, however, no gain in time consumed, but by a very simple modification of the process it is made far more rapid than either of the other methods, with no sacrifice of accuracy in the results.

The following is the new procedure: Weigh accurately in a small beaker about 0.5 Gm. of the salt, add 20 mils of alcohol, to which has been previously added 10 drops of strong sulphuric acid, from a pipette delivering 60 drops to the mil. Stir the mixture well and let it stand a few minutes, then decant the alcoholic solution into a platinum or quartz dish. Wash the residue in the beaker by decantation with two successive portions of alcohol (5 and 3 mils), adding the washings to the dish. Set fire to the alcohol and allow it to burn off, then ignite the residue at a temperature not exceeding dull redness. Since the residue consists almost wholly of the organic acid of the salt, together with a little free sulphuric acid, the carbon will burn off, in a very short time, the ignition requiring no attention meanwhile.

Dissolve the residue of alkali sulphate in the beaker in a little hot water, and when the carbon has been practically all burned off from the first residue, cool the dish and add to it the sulphate solution, together with rinsings from the beaker. Evaporate the solution in the dish to complete dryness and ignite at a red heat until white. Cool in a desiccator and weigh as alkali sulphate.

Since the sulphate has not been in contact with carbon to any appreciable extent during the ignition it may be considered to consist (barring impurities in the salt) wholly of alkali sulphate. However, if there is any doubt about this, the salt may be dissolved in a little hot water, a drop or two of sulphuric acid added, the solution evaporated, and the residue once more ignited.

It is assumed in these general assay processes that the salt is substantially free from impurities. The Pharmacopoeia in each case provides tests which exclude most of the impurities which would affect the result of the official assay. The question arises whether in an assay based on conversion of the salts into sulphates the result will be equally guarded by the pharmacopoeial purity requirements. It is evident that an impurity which increases the weight of the sulphate vitiates the result of the assay by the sulphate method, while the presence of the same impurities in the carbonate residue of the official assay does not affect the result unless they increase or diminish the alkalinity of the residue. Hence, if the sulphate method is resorted to, the question of the possible presence in the salt of fixed impurities not excluded by pharmacopoeial purity requirements must be kept in mind. The impurities most likely to be present in alkali salts for the detection of which pharmacopoeial tests are wanting or inadequate are sodium and potassium, and not infrequently magnesium and calcium. These impurities (not likely to be present in any large proportion) will affect the result of an assay either by the carbonate or the sulphate method, the former to a greater degree than the latter. One must bear in mind, however, the fact that where such impurities are present the result reached by the U. S. P. assay does not accurately represent "the amount of salt contained in the sample," as is assumed in the rubric of purity of the respective salts.

## FACTORS FOR CONVERTING SULPHATE INTO ORGANIC SALT.

Lithium Citrate Cryst., $\text{Li}_3\text{C}_6\text{H}_5\text{O}_7 + 4\text{H}_2\text{O}$ .....	1.709
Lithium Salicylate, $\text{LiC}_7\text{H}_5\text{O}_3$ .....	2.619
Lithium Benzoate, $\text{LiC}_7\text{H}_5\text{O}_2$ .....	2.328
Potassium Acetate, $\text{KC}_2\text{H}_3\text{O}_2$ .....	1.115
Potassium Benzoate, Cryst., $\text{KC}_7\text{H}_5\text{O}_2 + 3\text{H}_2\text{O}$ .....	2.458
Potassium Benzoate, Anhydrous, $\text{KC}_7\text{H}_5\text{O}_2$ .....	1.838
Potassium Bitartrate, $\text{KHC}_4\text{H}_4\text{O}_6$ .....	2.158
Potassium Citrate, Cryst., $\text{K}_3\text{C}_6\text{H}_5\text{O}_7 + \text{H}_2\text{O}$ .....	1.241
Potassium Citrate, Anhydrous, $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$ .....	1.172
Potassium Lactate, $\text{KC}_2\text{H}_3\text{O}_3$ .....	1.471
Potassium Salicylate, $\text{KC}_7\text{H}_5\text{O}_3$ .....	2.022
Potassium and Sodium Tartrate, Cryst., $\text{KNaC}_4\text{H}_4\text{O}_6 + 4\text{H}_2\text{O}$ .....	1.619
Potassium and Sodium Tartrate, Anhydrous, $\text{KNaC}_4\text{H}_4\text{O}_6$ .....	1.206
Potassium Tartrate, Cryst., $\text{K}_2\text{C}_4\text{H}_4\text{O}_6 + \frac{1}{2}\text{H}_2\text{O}$ .....	1.350
Potassium Tartrate, Anhydrous, $\text{K}_2\text{C}_4\text{H}_4\text{O}_6$ .....	1.298
Sodium Acetate, Cryst., $\text{NaC}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$ .....	1.916
Sodium Acetate, Anhydrous, $\text{NaC}_2\text{H}_3\text{O}_2$ .....	1.155
Sodium Benzoate, $\text{NaC}_7\text{H}_5\text{O}_2$ .....	2.028
Sodium Bitartrate, $\text{NaHC}_4\text{H}_4\text{O}_6 + \text{H}_2\text{O}$ .....	2.676
Sodium Citrate, Cryst., $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 + 2\text{H}_2\text{O}$ .....	1.380
Sodium Citrate, Anhydrous, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ .....	1.211
Sodium Lactate, $\text{NaC}_2\text{H}_3\text{O}_3$ .....	1.577
Sodium Salicylate, $\text{NaC}_7\text{H}_5\text{O}_3$ .....	2.253
Sodium Tartrate, Cryst., $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 + 2\text{H}_2\text{O}$ .....	1.619
Sodium Tartrate, Anhydrous, $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6$ .....	1.366