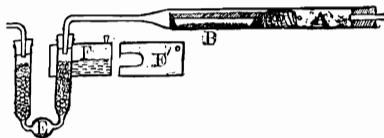Art. XLVII.—*The Determination of Water and Carbonic Acid in Natural and Artificial Salts;* by Thomas M. Chatard.

The following method and apparatus having been used for the analysis of a large number of natural and artificial alkaline carbonates, have been found to give results which are satisfactory not only as to accuracy but also as to ·ease and rapidity.

A is a combustion tube drawn out and bent at a right angle, B is a platinum boat which contains the salt to be examined and rests upon the thin sheet of asbestos paper C used to prevent adhesion between the platinum and the glass when heated. D is a roll of asbestos paper wrapped with thin platinum wire, which fits loosely in the tube and prevents back-currents of air during the heating. The bent end of the combustion tube enters one limb of the U-tube E which is my own form and differs, as shown by the drawing, from those at present on the market, by having an especially large bulb at the bottom. The two limbs of the tube are filled with glass beads wet with strong sulphuric acid. Sufficient acid should be put in to fill the narrow tubes up to the bends, compelling the air-current to bubble through.

In using the apparatus the combustion tube is thoroughly heated and then allowed to cool, a current of perfectly dried air being continually passed through it and the part of the tube where the boat is to be placed being protected by a semi-cylindrical trough of sheet iron lined with asbestos paper. The U-tube, having been weighed, is now attached to the combustion tube as shown and the air current is regulated. The platinum boat, previously heated and then cooled in a desiccator, is weighed and about one gram of the salt put into it and spread evenly along the bottom; the salt should be very finely pulverized to prevent decrepitation. The boat with its contents should, after weighing, be at once inserted into the tube and the asbestos plug D which should have been highly heated and still be hot, shoved in close behind the boat.

The tube is now heated gradually beginning at the plug. As the water in such salts is driven out, in great part, at a low temperature the heating must be cautiously done and the air current must not be too slow else water may condense in the cold part of the tube back of the plug. When heating salts containing sodium bicarbonate, the regulation of the air current is the more difficult since the acid CO_2 of the bicarbonate

is set free simultaneously with the water, but upon this regulation and the gradual heating depends the success of the operation.

The water bath F, of which F' is a top view, is now put into position, as shown, so that one limb of the U-tube fits into the curved recess in the side of the bath. A small alcohol lamp keeps the bath hot so that the water driven out of the salt may not condense in the upper part of the limb. By the use of this bath the time required for the operation is much shortened.

As soon as that part of the tube, which contains the boat, appears free from condensed water, it should be highly heated until all the moisture has disappeared from the drawn out portion. This will generally take from twenty minutes to half an hour; when completed, the water bath is removed and the apparatus allowed to cool, the air current being still kept up. As soon as the tube is cool, the U-tube is disconnected and the boat with its contents removed and placed in a small well stoppered glass tube which has been weighed.

The increase of weight of the U-tube gives the amount of water in the sample, while the weight of the small tube, containing the boat and the calcined salt, if subtracted from the sum of the weights of the tube, the boat and the salt taken, shows a loss which is the water plus the acid CO_2 of the bicarbonate. The calcined salt which should be sintered together but not fused can then be used either for a determination of the residual CO_2 or, as is preferred, dissolved and the alkali determined by standard sulphuric acid, methyl orange being used as the indicator, the same portion being then used for the Cl determination if chlorides are present.

Many attempts were made to collect and weigh the CO_2 driven off by heat, but it was found impracticable to do so without, at the same time, sacrificing the water determination. No matter how carefully the heat and air current are regulated, the CO_2 comes off too rapidly for proper absorption in a potash bulb and if the air current is too slow, water condenses back of the plug and this determination is too low.

When no attempt is made to absorb the CO_2, the results are very satisfactory. A specimen of Urao (Na_2CO_3, $NaHCO_3+$ $2H_2O$) from Owens Lake, Cal., gave the following results:

Salt taken.	Loss of salt.	H_2O.	Difference $= CO_2$.
1·0040	29·51	20·12	9·39
1·0490	29·58	20·09	9·49
1·1583	29·49	20·00	9·49
Average 1·0638	29·53	20·07	9·46

Three closely agreeing determinations of total CO_2 gave an average of 38·13 per cent; two determinations of the alkali

gave 40·28 per cent Na_2O equivalent to 28·58 per cent CO_2 required to make Na_2CO_3; 38·13−28·58=9·55 per cent CO_2 as the acid CO_2 of the bicarbonate present. Hence 0·09 per cent of this CO_2 was not driven off.

In another sample of Urao from same locality :

Salt taken.	Loss of salt.	H_2O.	Diff.=CO_2.	CO_2 in Res.	Sum CO_2.
1·1801	29·27	19·80	9·47	28·69	38·16
1·2297	29·35	19·83	9·52	28·64	38·16
1·2066	29·39	19·85	9·54	28·49	38·03
1·0880	29·26	19·80	9·46	28·67	38·13
1·1761	29·32	19·82	9·50	28·62	38·12

Two determinations total CO_2 gave 38·17 per cent and 38·22 per cent, average 38·19 per cent CO_2; two determinations of alkali gave 40·19 and 40·22 average 40·20 per cent Na_2O equivalent to 28·53 per cent CO_2. Hence as 28·62 per cent CO_2 was found in the residue we have 0·09 per cent acid CO_2 not driven off.

In a sample of Trommsdorff's C. P. sodium bicarbonate then was found :

Salt taken.	Loss of salt.	H_2O.	Diff.=CO_2.	CO_2 in Res.	Sum CO_2.
1·4265	36·68	10·91	25·77		
1·5995	36·81	10·90	25·91		
1·3200	36·71	10·91	25·80	26·25	52·05
1·3723	36·73	10·98	25·75	26·37	52·12
1·4296	36·73	10·92	25·81	26·31	52·09

Two determinations of total CO_2 gave average of 51·98 per cent; two determinations of alkali gave average of 36·88 per cent Na_2O equivalent to 26·17 per cent CO_2. Subtracting 26·17 from 26·31, average amount of CO_2 in residue, we have 0·14 per cent CO_2 not expelled by heating to sintering.

Comparing these amounts of excess of CO_2, retained by the residues of the ignition, with the weight of salt taken we have the following relations.

Sample.	No. of deter- minations.	Average wt. taken.	Average excess.	Relation of excess to weight.
Urao No. 1	3	1·0638	0·09	0·084
Urao No. 2	4	1·1761	0·09	0·077
Bicarbonate	4	1·4296	0·14	0·091
			Average	0·084

Hence we may say that if sodium bicarbonate, or a salt containing a large proportion of it, is heated till it sinters without fusing and is kept at that temperature for one hour, it still retains CO_2 in excess of the amount required to form mono-

carbonate with the alkali present; and that, for quantities of from 1 to 1·5 grams, this excess may be considered as one-tenth of one per cent of the salt taken.

For an analysis of sodium bicarbonate or baking soda, it is therefore sufficient to determine the loss by ignition, the H_2O, and the alkali in the ignited residue to obtain the total amount of CO_2 in the sample, adding to this amount an amount of CO_3 equal to 0·10 per cent of the weight of the samples taken. The method is much more rapid, far easier and quite as accurate as the distillation process, and may prove of value for technical purposes.