

NOTE ON THE ESTIMATION OF CARBONIC ACID IN SELF-RAISING FLOURS AND BAKING-POWDERS.

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In analysing self-raising flours and baking-powders it is customary to estimate the total and available carbonic acid. The former is the carbonic acid liberated from the mixture by the addition of an acid—*e.g.*, hydrochloric acid—and the accurate estimation of its quantity is readily carried out by any of the customary methods.

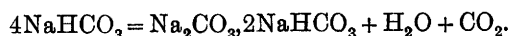
The available carbonic acid is generally assumed to be that liberated on adding water to the mixture, and to be entirely due to the decomposition of the sodium bicarbonate by the acid material present in the mixture. This definition takes no account of the carbonic acid liberated from the excess of sodium bicarbonate (almost invariably present in these mixtures) when heated to the baking temperature. The author considers the true available carbonic acid to be that which is liberated under the actual conditions of baking. This would include the gas liberated by the acid material present, *plus* that liberated by any acid present in the flour, milk, or other ingredients used, together with that liberated by the action of heat on the excess of sodium bicarbonate. Obviously it is practically impossible to estimate carbonic acid liberated in all these ways, as we are dealing with three unknown factors—*viz.*, the amount of acid present in the other ingredients, the time, and the temperature of the actual baking—which will affect the extent of the decomposition of the excess of sodium bicarbonate. Time may vary from five minutes to as many hours. Temperature will probably be anything from under boiling-point to 150° C. or over. In order to determine how much carbonic acid was liberated from sodium bicarbonate by boiling, a number of experiments were made, and a summary of these is given in the following table :

1 gm.	boiled for	6 minutes	required	4 c.c.	$\frac{N}{2}$ HCl	{ = 0.044 gm. CO ₂ .
						= 8.41 per cent. of total CO ₂ .
1	"	"	10	"	"	= 0.0924 gm. CO ₂ .
						= 17.64 per cent. of total CO ₂ .
1	"	"	20	"	"	= 0.1122 gm. of CO ₂ .
						= 21.38 per cent. of total CO ₂ .

On boiling for a further—

10 minutes	required	2.0 c.c.	$\frac{N}{2}$ HCl	= 0.022 gm.	CO ₂	= 4.20 per cent. of total CO ₂ .
20	"	"	1.8	"	"	= 3.7

It will be seen from these figures that for the first ten minutes the liberation of carbonic acid is very rapid, and that for each succeeding ten minutes additional boiling about 4 per cent. carbonic acid is obtained. So long as the conditions of the experiment were the same (*i.e.*, size of flask, etc.), practically identical results were obtained. It is rather remarkable that, when boiled for thirty minutes, 25.58 per cent. of the total carbonic acid is obtained, and this corresponds with the following equation :



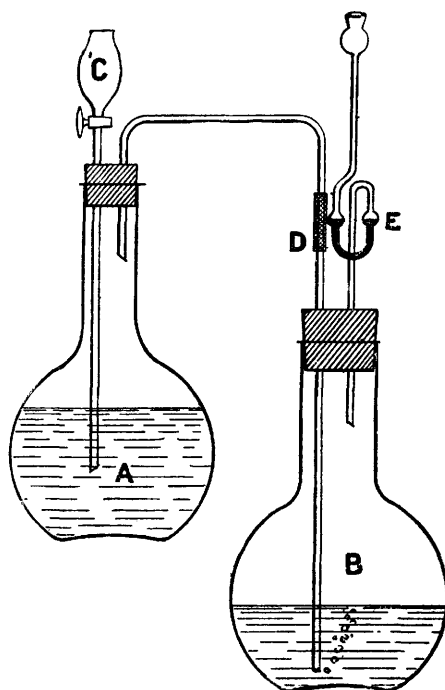
That is, the bicarbonate rapidly decomposes to form the sesquicarbonate, thus liberating 25 per cent. of the total CO_2 .

The author suggests that, for the valuation of baking-powders and similar preparations, the "apparent" available carbonic acid should be estimated, and he would define this as the amount of carbonic acid liberated on boiling with water only for thirty minutes—*i.e.*, the carbonic acid liberated by the action of the acid material, together with 25 per cent. of the carbonic acid present in the excess of bicarbonate.

It is evident that the "apparent" available carbonic acid cannot be estimated directly by the Schrötter method, but the Knorr apparatus and method described in Leach might be adapted to the purpose. As the latter process is cumbersome and complicated, the author prefers to use the process described by himself (ANALYST, 1904, 29, 152), which requires no elaborate apparatus, and can be adapted for working with very large or quite small quantities of the sample. In order to overcome the difficulty arising from frothing when such mixtures are boiled, the process has been slightly modified.

For baking-powders 2 to 5 grms. are used, and for self-raising flour 10 grms. In the case of the former, the process as originally described is usually quite sufficient, but it is generally easier to treat both mixtures alike.

The weighed quantity of the sample is transferred to a dry litre flask, A. Four or five pieces of pumice are added, and the flask closed with the cork and fittings. Into the absorption flask B are placed 50 c.c. of a saturated solution of barium hydroxide, and 100 c.c. water (previously well boiled and cooled), and the seal of the safety funnel E is closed with a little barium hydroxide solution coloured with phenolphthaleïn. One hundred c.c. of alcohol are run into A, and the mixture well shaken to break down any lumps. This is followed by 100 c.c. of water, and the



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mixture again well shaken. The absorption flask should also be shaken if any carbonic acid appears to be passing into it. One hundred c.c. of liquid paraffin are added, and the flask and contents heated over an Argand burner, with frequent shaking until the contents boil briskly. The boiling is continued for fifteen minutes, and, without stopping the operation, about 400 to 500 c.c. of *boiling* water are carefully added, and the ebullition continued for a further fifteen minutes. The absorption flask should be kept well shaken during the earlier part of the process, when the gas is passing freely. Under these conditions no carbonic acid will be found in the seal of the safety funnel. A shallow basin of water is placed under the absorption flask when it begins to get hot. At the end of the thirty minutes the flask B is disconnected, and the inlet tube closed, and the contents cooled in running water if necessary. A small funnel is then inserted in the inlet tube, and the excess of barium hydroxide neutralised by adding 20 per cent. hydrochloric acid in slight excess until liquid is slightly acid to phenolphthalein); $\frac{N}{2}$ soda is then run in in sufficient quantity to make the contents just slightly alkaline, and the flask is once more well shaken, to absorb any carbonic acid which may have been liberated. The stopper is then lifted, and the contents of the safety funnel washed in if necessary. The solution in the flask is carefully neutralised with $\frac{N}{2}$ or $\frac{N}{10}$ hydrochloric acid. Methyl orange is added and the titration completed, taking care to wash down the inlet tube with a little of the acid. If the flask has been kept well shaken during the process of absorption, the barium carbonate can be titrated direct, but sometimes a little scale may be formed which cannot be titrated in this way. In such cases it is only necessary to add 3 or 4 c.c. excess of acid, heat for a few minutes, and titrate back.

The same quantity can be used for the estimation of the non-available carbonic acid by adding acid to the residue in A and boiling into another absorption flask.

For the total carbonic acid it is not necessary to use alcohol or oil, as on adding 20 to 25 c.c. of hydrochloric acid, and boiling, little frothing takes place. In other respects the process is the same, but the time of boiling is regulated by the time taken for the last appearance of carbonic acid in the absorption flask. It is better in all cases to continue boiling the flask, after nearly filling it with boiling water, for about three to five minutes.

The following are some results obtained by this process in analysing commercial baking-powders and self-raising flour :

BAKING-POWDERS.					
	I.	II.	III.	IV.	
Apparent available CO ₂ ...	7.52	16.2	11.8	12.8	
Non-available CO ₂ ...	2.33	3.2	1.0	0.5	
Total ...	9.85	19.4	12.8	13.3	
Acid in mixture ...	Tartaric Acid.	Tartaric Acid.	Cream of Tartar.	Cream of Tartar and Tartaric Acid.	

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SELF-RAISING FLOURS.

	I.	II.	III.	IV
Apparent available CO ₂ ...	0·60	0·46	0·57	0·58
Non-available CO ₂ ...	0·08	0·03	0·02	0·02
Total ...	<u>0·68</u>	<u>0·49</u>	<u>0·59</u>	<u>0·60</u>

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