

after redistilling. For this reason the aqua distillata sterilisata of the next pharmacopœia is open to the same criticism, since unless the freshly distilled water is *collected aseptically*, the subsequent boiling to sterilize it will as likely produce a vaccine as when made from an older distilled water, only in a lesser degree.

Here we have an apparatus that is inexpensive and does the work. Pharmacists should get busy if they want to meet the demands of up-to-date practitioners, who require *freshly distilled* water for preparing or having the pharmacist prepare salvarsan, physiological salt and other solutions for intravenous use.

If one cares to put up ampoules wherein such water is always used as a solvent, I think it will pay to do so—if not directly, then indirectly, in added prestige with physicians who direct their prescriptions towards your establishment. Examples of such ampoules are sodium cacodylate, a substance which has almost superseded Fowler's and Pearson's solutions in chorea and anemia—iron and arsenic are used particularly in pernicious anemia. Emetine hydrochloride in amebic dysentery and pyorrhea has brought wonderful results, and as a consequence, the price of ipecac is soaring. There are too many substances used in ampoule form to mention all—I simply call attention to the above medicaments because the demand for them is increasing.

The technique necessary for this work is to use sterile utensils. Ampoules must be of hard glass, either Jeno or Non-Sol. *Hard glass is very essential*, soft glass being more or less soluble, especially when sodium chloride is used to make the solution isotonic, the powerful sodium and chlorine ions acting on soft glass. Solutions must be neutral. Ampoules may be filled by using a burette with rubber connection with glass tube drawn to a fine point or with a hypodermic syringe. The filled ampoules should be heated to the boiling point before sealing in the flame, so that they may be placed in boiling water for thirty minutes each day, for three successive days.

A METHOD FOR THE DETERMINATION OF CARBON DIOXIDE IN BAKING POWDER AND CARBONATES.*

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The following is the result of an attempt to devise a method for the determination of the available carbon dioxide in baking powders, which is simple in principle, requires an apparatus easy to construct, and manipulate, consumes little time and gives reasonably accurate results. The method was devised for the use of a class of girls in household chemistry. It is adapted not only to the determination of carbon dioxide in baking powder but also in carbonates, bicarbonates and minerals such as limestone and dolomite.

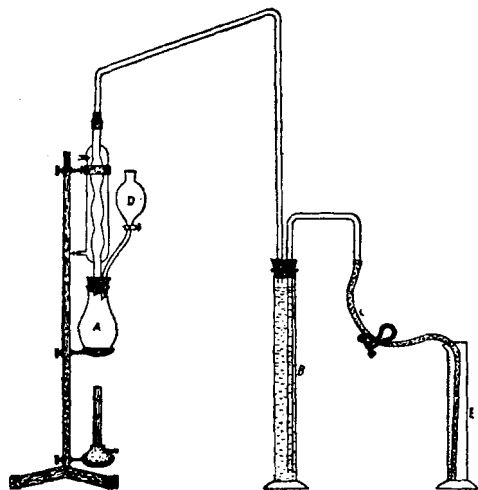
The principle of the method, in brief, is the liberation of the carbon dioxide,

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causing it to displace its own volume of a saturated solution of sodium chloride into a graduated cylinder and measurement of the volume of the solution displaced. A saturated solution of salt is used because carbon dioxide is not readily dissolved in it and tests show that under the conditions of the experiment the amount of absorption is so small that it may be neglected. A saturated solution of salt is also used, instead of water, in the decomposition of the baking powder. The principle of collecting carbon dioxide over a saturated solution of sodium chloride is used by Jago in determining the strength of yeast.

PROCEDURE.

One gram of the baking powder is placed in the flask *A*. Water is circulated through the condenser for several minutes. The cylinder *B* is nearly full and the delivery tube *C* is full, to the open end, of a saturated solution of salt, the pinch clamp being closed. The stopcock in the funnel *D* is now closed and exactly 25 cc. saturated solution of salt placed in the funnel. The pinch clamp on the delivery tube is now opened and the salt solution run in from the funnel by opening the stopcock, care being taken not to admit any air. The carbon dioxide begins to be liberated at once and the salt solution is driven over into the graduated cylinder *E*. The decomposition of the baking powder is completed by heating with the gas burner until the solution has boiled for two or three minutes or until the rise of the salt solution in the cylinder *E* is seen to have stopped. The flame is then turned out and the apparatus allowed to stand



until it has cooled to room temperature again. The cooling of the flask *A* can be hastened, if desired, by lowering the ring and bringing a beaker of cold water up under the flask until it is immersed in the water. When the apparatus has reached room temperature the salt solution in *E* and *B* is leveled up by raising *E*. The pinch clamp is then closed; the cylinder *E* is lowered to the table, the delivery tube removed and the volume of salt solution in *E* read. Subtract 25 cc. from this volume to compensate for the 25 cc. of salt solution which were run into the flask *A* to decompose the baking powder. Correct the remaining volume to 0° C. and 760 mm. pressure. As 5.1 cc. of carbon dioxide gas at 0° C. and 760 mm. pressure weigh 0.01 gm., which is one percent of 1.0 gm., the percent of carbon dioxide in the baking powder may be found by dividing the corrected volume by 5.1.

NOTE: In making the correction for aqueous tension I have used that for pure water which is two or three mm. too high in each case. The effect of the small error thus introduced into the results here recorded is to make them too low.

RESULTS.

The purpose of the condenser in the apparatus is to prevent any water from distilling over into the cylinder *B* and thus diluting the salt solution. Table I shows some results obtained with various baking powders:

Table I—Analyses of Baking Powders for CO₂

Sample	Vol. CO ₂ cc.	Temp. ° C.	Barometer mm.	Percent CO ₂
Baking Powder				
Royal A 1.....	65.0	20	740	11.30
2	66.0	20	740	11.47
Royal B 1.....	66.4	21	742	11.43
Calumet A 1.....	78.3	22	740	13.47
2	77.9	23	740	13.34
Calumet B 1.....	82.0	29	745	13.69
K. C. 1.....	65.0	24	745	11.14
2	64.5	25	745	11.02

In order to ascertain whether an appreciable amount of carbon dioxide is absorbed by the salt solution during a determination, 90 cc. of carbon dioxide were run into the apparatus at 22° C. and 740 mm. and allowed to stand two hours and forty minutes, at the end of which time the volume was 90.3 cc. and the temperature 23° C., the pressure having remained the same. The increase in volume is very close to that which would result from the increased temperature.

In applying this method to the determination of carbon dioxide in carbonates such as sodium carbonate and precipitated calcium carbonate the sample was decomposed by means of a dilute solution of hydrochloric acid which was saturated with sodium chloride. The absorption of carbon dioxide by the acid solution is appreciable and must be allowed for. This is done by decomposing one sample with 10 cc. of the acid solution, then running a second determination, using 15 cc. of acid. The difference in the amounts of carbon dioxide obtained in the two cases being due to the absorption by 5 cc. of acid solution, the proper correction is made on either determination. When decomposing precipitated carbonates with hydrochloric acid no heating is necessary, hence the time consumed in each determination is very short, but two or three minutes being required for the liberation of the carbon dioxide. In running two samples of calcium carbonate in this way, the whole process, including the weighing of the samples, required only ten minutes.

Table II shows some of the results obtained on carbonates. In Determinations 1 and 2 heat was used. In Determination 3 no heat was used. The sample of sodium carbonate used in Determinations 4 and 5 was found to contain 4.46 percent of moisture. The theoretical amount of carbon dioxide in this sample is therefore 39.65 percent. Some of this sodium carbonate was now dried and Determination 6 made upon the dry sample with the result shown. Allowing for the same absorption as in Determination 5, which is 2.97 cc., gives a total volume of 121.17 cc., under the above conditions of temperature and pressure which correspond to 41.38 percent of carbon dioxide.

Determination 7 was made on a sample of limestone. It was necessary to use heat to completely decompose the sample with the acid solution. I had no gravimetric determination of carbon dioxide in this limestone against which to check the result. However, according to the figures of another analyst, the limestone contains 12.28 percent SiO₂, 1.15 percent Al₂O₃+Fe₂O₃, 46.74 percent

CaO and 1.38 percent MgO, making a total of 61.55 percent, which leaves for carbon dioxide 38.45 percent. This makes no allowance for potassium and sodium which may be present in very small quantities. The result is undoubtedly a close approximation to the actual percentage of CO₂ present.

Table II—Analysis of Carbonates for CO₂

Detn. No.	Sample	Wt. taken gm.	Decomposed by cc. acid	Vol. CO ₂ cc.	Temp. ° C.	Bar. mm.	Percent CO ₂	
							Found	Theoretical
1	CaCO ₃	1/4	5	62.5	23	737	43.98	44.00
		1/4	10	61.0	25	737		
2	CaCO ₃	1/4	3	64.0	24	739	43.88	44.00
		1/4	8	63.5	25	739		
3	CaCO ₃	1/2	8	127.0	25	735	43.98	44.00
		1/2	13	124.65	25	735		
4	Na ₂ CO ₃	1/2	7	116.0	25	734	39.95	39.65
		1/2	12	113.88	25	734		
5	Na ₂ CO ₃	1/2	7	116.0	25	734	39.95	39.65
		1/2	12	113.88	25	734		
6	Dry Na ₂ CO ₃	1/2	7	118.2	24	742	41.38	41.50
7	Limestone	1/2	10	111.5	25	744	38.18	38.45 (a)
		1/2	15	113.5	28.3	742		

(a) By difference; see text.

NOTE: Since sending this paper for publication I have found a convenient way of obviating the necessity of making a correction for the absorption of carbon dioxide in those cases where the sample is decomposed by dilute acids. This makes it unnecessary therefore, to decompose more than one sample of the substance for one determination. The procedure is as follows: Just before making the determination pour 30 or 40 cc. of dilute hydrochloric acid (1 acid : 3 water) on 0.5 gm. of precipitated calcium carbonate. When the effervescence has ceased, and the acid solution has become clear, pipette 10 cc. of this into the bulb of the separatory funnel and proceed with the determination. This method of saturating the acid with carbon dioxide requires but a moment and makes it unnecessary to correct for any absorption whether the sample is decomposed by heating or at room temperature. The following determination will illustrate the accuracy of this method of procedure:

Wt. of limestone taken.....	0.5 gm.
Volume of salt solution displaced.....	138.18 cc.
Temperature, 22° C. Barometer.....	739 mm.
Volume corrected to 0° C. and 760 mm.....	112.3 cc.
Percent CO ₂ found.....	44.03
Theoretical percent CO ₂	44.00

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