

A CHEMICAL EXAMINATION OF CERTAIN BAKING POWDERS.

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## OUTLINE.

- I. Introduction.
- II. General Discussion of Classes of Baking Powders.
- III. Discussion of Results Obtained by Others.
- IV. Qualitative Analysis of Five Powders.
- V. Quantitative Determinations of Carbon Dioxide in Five Powders.
  1. Description of apparatus.
  2. Manipulation of apparatus.
  3. Results obtained.



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### A Chemical Examination of Certain Baking Powders.

The necessity of having bread preparations raised quickly for immediate baking led to the use of chemical agents for that purpose. In all of these agents, the gas, which expands, is obtained by the decomposition of a carbonate which is introduced with an acid constituent to act upon it, directly in the flour. When water is added to the flour to make the dough, the chemicals are dissolved and the action takes place, carbon dioxide and water being formed, the carbon dioxide acting as the expansive gas. Many people suppose that nothing remains in the bread from that reaction, that it is all driven off during the baking. There is a residue left which varies with the different powders, and is more or less objectionable depending on the powder used.

There are some combinations which leave a minimum amount of residue and of the least objectionable character, while others are the reverse. Most people do not know the healthful character of some of these combinations used and do not realize the danger of using cheap baking-powders when used to any great extent.

The essential constituents of a good baking-powder are first; a carbonate or bicarbonate of an alkali metal and, second; an acid constituent capable of combining with the alkali metal forming a salt, and liberating the carbon dioxide. For the alkaline constituent, sodium bicarbonate is chiefly used, though sometimes ammonium bicarbonate (acid ammonium carbonate) is substituted for it. For the acid constituent various substances are used. Potassium bitartrate, free tartaric acid, an acid phosphate, or an alum, are the most common ones. The acid and alkaline constituents are about in the proper proportions for combination, and starch or flour is added to them so they will not react in the dry state but may be kept indefinitely.

Baking powders may be classified into three classes:

1. Tartrate powders, in which the acid constituent is potassium bitartrate

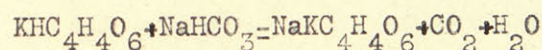


or free tartaric acid.

2. Phosphate powders, in which the acid constituent is an acid phosphate.
3. Alum powders, in which the acid constituent is an alum.

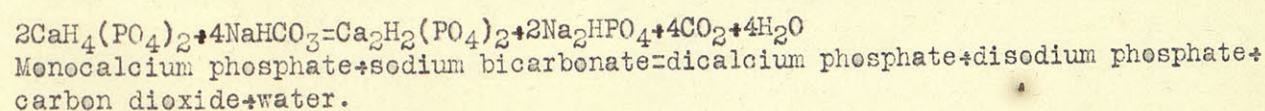
Many powders are combinations of two of these classes.

Under the first we find the bitartrate of potassium used much more as the acid constituent than free tartaric acid. It interacts with the acid sodium carbonate forming the double tartrate of sodium and potassium and giving off carbon dioxide. This double salt is Rochelle salt and is probably the least objectionable of the residues left by baking powder. The equation for this reaction is;



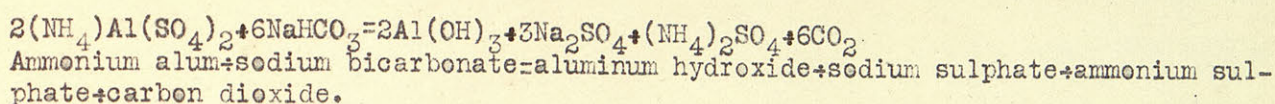
Potassium bitartrate+sodium bicarbonate=sodium potassium tartrate+carbon dioxide+water.

Under the second class the salt most commonly used is monocalcium phosphate. It is made chiefly from bone by the action of sulphuric acid, so that some calcium sulphate is formed in the reaction, and is probably present to some extent in the powder. The amount of residue left is very large but it is not very objectionable. Acid phosphate of sodium is also used in phosphate powders. The probable equation when calcium acid phosphate is used is:



Under the third class we find potassium and ammonium alums as the most common alums from which the sulphuric acid is liberated as the acid constituent. These constituents can be gotten readily from various sources, i.e., from alum stone, direct combination of constituents, etc. It is the thought that the alum would be better if the water of crystallization was driven off. Whether it is driven off, and to how large an extent, varies with different manufacturers. The aluminum is precipitated as aluminum hydroxide from these powders, while the alkali metal, which is in combination with aluminum forming the double salt of sulphuric acid, remains the same. The probable equation for the reaction in this class of powders is:





As stated before there are mixed powders containing acid constituents of more than one class. One combination, which is thought by several of the chemists of different experiment stations to be harmful, is the addition of tartaric acid to an alum powder. The most common combination is alum and phosphate powders and it is thought that this combination improves the powders.

The following is a brief survey of the results obtained by several investigators who worked in experiment stations or in the United States Department of Agriculture. In analyses made by Professor Cornwall, given in The Report of the Dairy Commissioner of New Jersey for 1888, we find that out of thirty-nine powders he examined twenty-five were alum powders. He seems to think tartrate powders are the best and suggests that the powders should be guaranteed to yield a certain per cent of carbon dioxide. Fifty-six percent of the samples tested by the North Carolina Experiment Station as given in Station Bulletin No. 155 were alum powders and eighty-one per cent of them contained alum. The United States Department of Agriculture also made some investigations upon a large number of brands. The carbon dioxide was estimated quantitatively by Kuorr's apparatus in which it absorbed in potash bulbs. The largest number of the samples they used were alum powders.

Professor Cornwall's average yield of carbon dioxide for twenty samples of alum and phosphate powders (no straight alum powders included) is 8.97%; for eight samples of tartrate powders, 11.60%. Professor Weber's report from the Annual Report of the Ohio State Dairy and Food Commissioner for 1887 gives an analysis of thirty brands. Of these nineteen were alum powders and the average percentage of carbon dioxide was 7.58; for eight samples of tartrate powders 11.20%. In the analyses by the United States Department of Agriculture, the average for twenty samples of both alum and alum and phosphate powders is 8%; for eight samples of tartrate powders, 10.10%.



It has been found by many that upon long standing the powders gradually lose carbon dioxide. A good baking powder should yield ten or twelve per cent of carbon dioxide. This brief statement of results obtained by various investigators shows that the tartrate powders yield a higher percentage of carbon dioxide than the alum powders.

The chief substances used for filling in baking powders are starch and flour. The powder having the least amount of filling will probably yield the most carbon dioxide, but if there is not enough present the strength would probably rapidly deteriorate. When baking powder is dissolved, a small quantity of starch gives a certain opacity to the solution but if it excess a paste may be formed. In tartrate powders the percentage of starch varies from about ten to twenty-five per cent, the average being about fourteen per cent. In powders containing free tartaric acid there is a larger percentage, usually from forty to forty-five per cent. In phosphate powders the percentage varies from about twenty to twenty-five. The highest percentage is found in alum powders varying from thirty to fifty-five per cent, the average being about forty. This probably causes the lowness of the available carbon dioxide in these powders which theoretically should yield the highest percentage.

#### I. Qualitative Analysis.

##### 1. Manhattan Baking Powder.

The powder was dissolved in dilute hydrochloric acid and an insoluble residue of starch or flour was left upon filtration. It was tested with hydrochloric acid and hydrogen sulphide for the following metals: silver, mercury, lead, bismuth, copper, cadmium, arsenic, tin and antimony. None of these metals were present. No precipitate forming upon the addition of ammonium molybdate to a small amount of the original solution indicated the absence of phosphoric acid. Ammonium chloride was added to the solution, heated until it boiled, and then ammonium hydroxide was added. A moderately heavy gelatinous precipitate formed which was filtered while hot. The filtrate may contain members of subsequent



groups of metals, and the precipitate may consist of hydroxides of aluminum, chromium and iron, in case phosphoric acid is not present; or, in case that it is not present, it may contain the phosphates of magnesium, aluminium, chromium, iron, zinc, manganese, cobalt, nickel, barium, strontium, and calcium.

The precipitate was washed into a test-tube and a few bits of sodium peroxide were added. This was boiled until it ceased to effervesce. It was divided into two portions, one of which was tested for chromium, the other for aluminum. Chromium was absent, but a gelatinous precipitate forming on the addition of an excess of ammonium hydroxide, proved the presence of aluminum. Tests were made which indicated the absence of iron, zinc, manganese, cobalt, nickel, barium, strontium, calcium, and magnesium. The filtrate from previous groups was evaporated to dryness, the residue moistened with hydrochloric acid, and tested for potassium and sodium by the flame test. On looking through a blue glass the violet flame of the potassium shows, otherwise it was masked by the yellow flame. Platinic chloride or sodium cobaltic nitrite also showed the presence of potassium by the formation of a heavy yellow precipitate. In testing for ammonia Nessler's reagent ( $K_2HgI_4 + NaOH$ ) was used. Sodium carbonate was added to precipitate the hydroxide of aluminum, chromium, or iron, and the filtrate tested with Nessler's reagent. A yellowish solution formed, showing that only traces, if any, of ammonia were present.

In testing for acids, barium chloride was added to some of the original solution, a moderately heavy, fine white precipitate was thrown down. Fused with sodium carbonate on charcoal, sulphates are reduced to sulphides. If the mass is moistened with dilute hydrochloric acid and placed on a silver coin it will stain it black. The fact that the precipitate obtained with barium chloride was insoluble in acids pointed to the fact that it was probably barium sulphate, which was proven by the test on silver. Carbonic acid may be said to be present in all of these powders as shown by the giving off of carbon dioxide when treated with acids. No other acids present in this powder.



## 2. K. C. Baking Powder.

The same method of analysis used as before. Silver, mercury, lead, bismuth, copper, cadmium, arsenic, tin, and antimony were absent. Phosphoric acid was found present by the ammonium molybdate test. A heavy, white, flocculent precipitate was obtained with ammonium chloride and hydroxide which proved to be aluminum hydroxide. No other metals were present except sodium and potassium.

Phosphoric and sulphuric acids were found to be present by the regular tests. In testing for tartaric acid, sodium carbonate was added to the solution to precipitate the hydroxide of aluminum. Hydrochloric acid was added in excess to the filtrate and it was evaporated to drive off the carbonic acid. The solution must remain acid after boiling, and ammonium hydroxide and calcium chloride were added to it. A white precipitate formed which is tested with sodium hydroxide. A thick precipitate formed and was filtered off. The filtrate was boiled and a slight precipitate formed indicating tartaric acid. It was filtered while hot, washed into a test-tube, a drop of ammonium hydroxide and some silver nitrate added, and warmed. A black precipitate or a silver mirror indicated tartaric acid.

## 3. Royal Baking Powder.

No metals present in this powder except potassium and sodium. Tartaric acid was the only acid present.

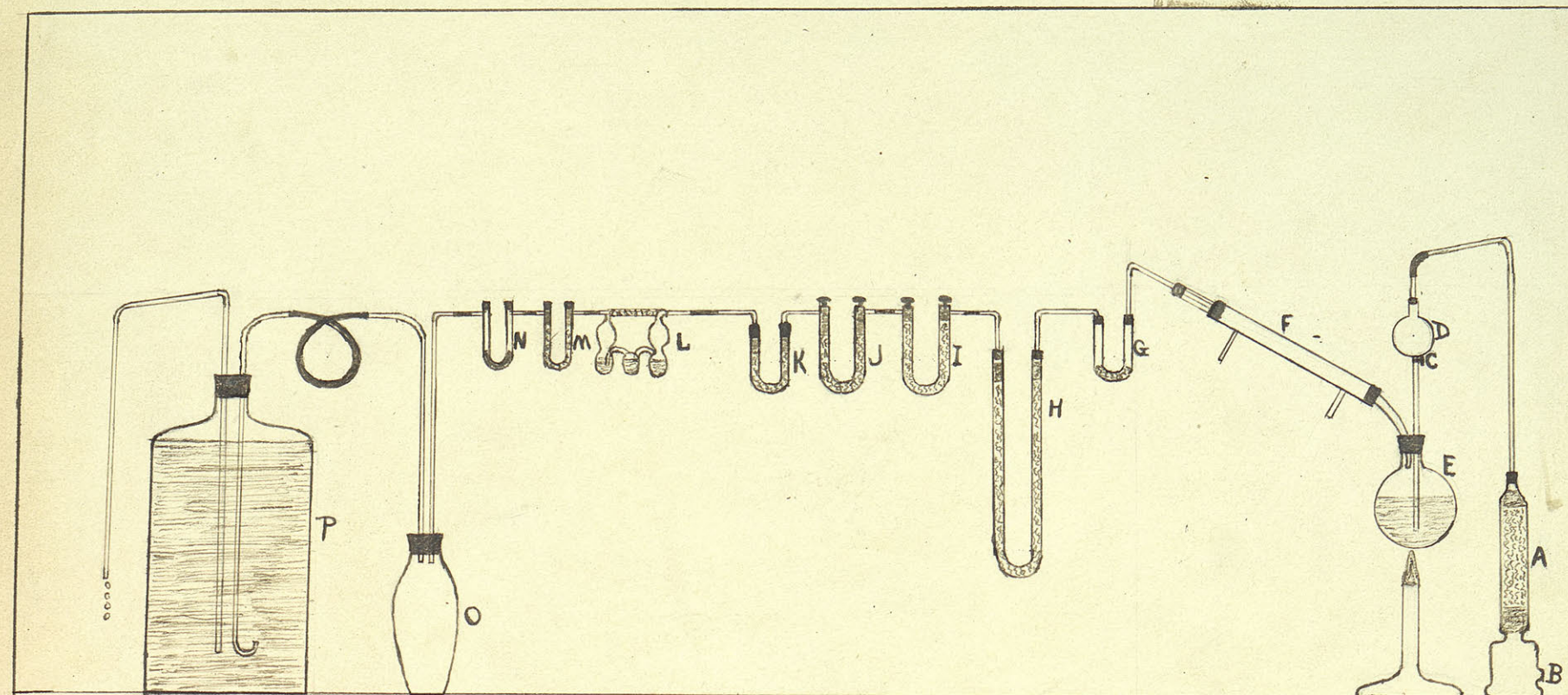
## 4. Calumet Baking Powder.

A moderately heavy white precipitate of aluminum was formed with ammonium chloride and hydroxide. Ammonia was proved present by the formation of a heavy brown precipitate with Nessler's reagent. Potassium and sodium were the only other metals present. Sulphuric, phosphoric and tartaric acids were present.

## 5. Shepard's Baking Powder.

Potassium, sodium, and aluminum were the only metals found present. The precipitate of aluminum hydroxide was moderately heavy. Sulphuric, phosphoric and tartaric acids were present.





Heidenhain's apparatus for the determination of carbon dioxide.



Alum was found in four of the five powders tested, though Manhattan was the only straight alum powder. K. C., Calumet, and Shepard's were alum and phosphate powders, and Royal was potassium bitartrate powder. The alum present in Manhattan was probably potassium alum as no ammonia was present. K. C. contained more aluminum than any other powder examined. It also contained a small amount of tartaric acid. The alum in Calumet was partially ammonium alum, as a large amount of ammonia was proved to be present.

## II. Quantitative Analysis.

The apparatus, used in making quantitative determinations of the amount of carbon dioxide given off by each of the various powders, was Heidenbain's apparatus with one or two slight modifications. This determination gives the leavening power of the powders.

The apparatus consists of first, a soda lime tower, A (lettered in the sketch), which has an opening, B, near the bottom so air can pass up through the soda lime, which moves the carbon dioxide from it. From A a tube extended to, D, a dropping funnel which passed into a flask, E,. The flask was supported so a burner could be placed under it. This flask was the one in which the powder was placed and acid from the funnel was allowed to act upon it. A small condenser, F, was attached to this flask, from F a tube passed into a small U tube, G, which contained a few pieces of calcium chloride, to collect some of the water which passed over through the condenser. The next U tube, H, also contained calcium chloride for drying purposes. The U tube, I, contained punice stone with dehydrated copper sulphate on it, which was to prevent the passing of hydrochloric acid gas into the absorbing tubes. Following this was another U tube, J, of calcium chloride, then came the absorbing tubes. The first absorbing tube was a U tube, K, containing soda lime chiefly, but in the side towards L, some calcium chloride was placed. The second absorbing tube, L, was a Geissler's bulb which contained potassium hydroxide. At the end of this bulb, which passed into another U tube, M, was a small tube containing soda lime which prevented the moisture escaping as air passed



through the bulb. These two pieces of apparatus, K and L, were weighed before and after each determination. After they had increased in weight about a gram, the filling was changed in them. Next after the absorbing apparatus, there was another small U tube, M, containing calcium chloride, then a U tube, N, which contained a small amount of glycerine. This tube, N, was used as a trap by which to tell whether the apparatus was air-tight, whether or not air was passing through, and how rapidly it was passing through. A piece of tubing connected N with an empty bottle, O, which was connected with the aspirator, P. The aspirator was a large bottle filled with water. The tubes in it were bent to form a siphon so that as the air passed through the apparatus into the bottle, water was forced out. The empty bottle was a safeguard against the water drawing back into the apparatus, spoiling the determination.

The U tubes were filled as stated in the description of the apparatus. The punice stone was broken into small pieces, heated in a muffle furnace until red-hot, and dropped into a saturated solution of copper sulphate. It was dipped out when it sank to the bottom and heated again in a furnace to 150 degrees C., kept at that temperature for about two hours, allowed to cool in a dessicator, and then put into a tube.

In making a determination, about two grams of baking powder were weighed out from a weighing bottle in to the flask, E. Next, the two absorbing tubes were weighed and connected with the apparatus. The dropping funnel was filled with dilute hydrochloric acid.

The apparatus was tested to make sure that all joints were tight. The powder is put in the flask and the condenser, F, started. The acid and water in the funnel are allowed to drop very slowly so that the gas will not be given off too rapidly to be absorbed. The water used in diluting the hydrochloric acid should be made free from carbon dioxide by boiling. When all the acid has been let into the flask, the stop-cocks, C, is turned so no air can enter. The tower



of soda lime, A, is now attached and B is opened. The liquid in the flask is heated to boiling and kept at that temperature. When the carbon dioxide is being given off from the powder, it drives out air slowly so that the aspirator runs slowly during this part of the operation. When no more air passes N, the aspirator is started with greater speed and when the water stops running, which it does when all the air has been driven out of the apparatus, the stop-cock, C, is opened cautiously. Air is allowed to pass through at not too rapid a rate, so that the short guard-tube on the potash bulb, containing soda lime, will have time to absorb the moisture from it, and not allow the tube to lose in weight. This is kept up until about three liters of water are aspirated away, with continued boiling of the liquid in the flask throughout the entire operation. The operation is stopped, the absorbing tubes removed, capped, and weighed. The apparatus was first tested with calcium carbonate to determine its accuracy. It was found to give results that were slightly too high.

#### Results.

##### 1. Calcium carbonate.

Weighing bottle + $\text{CaCO}_3$	= 14.1347 gm.
" " " "	= 12.7100 "
$\text{CaCO}_3$ used	= 1.4247 "

Wt. of Geissler's bulb + $\text{CO}_2$	= 61.7558
" " " "	= 61.2820
" " $\text{CO}_2$ in Geissler's bulb	= .4738

Wt. of U-Tube + $\text{CO}_2$	= 35.5131
" " " "	= 35.3561
" " $\text{CO}_2$ in U-Tube	= .1570

Wt. of $\text{CO}_2$ in Geissler's bulb	= .4738
" " " " U-Tube	= .1570
Total wt. of $\text{CO}_2$	= .6308

By dividing 44 the molecular weight of carbon dioxide by the molecular weight of calcium carbonate (100.1), the percentage of carbon dioxide in calcium carbonate is obtained, which is .43956.



$$\begin{aligned} \text{Wt. of CaCO}_3 \times .43956 &= \text{Wt. of CO}_2 \\ 1.4247 \times .43956 &= .62624 \end{aligned}$$

This shows that the result obtained, .6308, is about .3 per cent too high.

2. Royal Baking Powder.

Weighing bottle + powder	=	16.3904
" "	=	14.2060
Wt. of powder used	=	<u>2.1844</u>

Wt. of Geissler's bulb + CO <sub>2</sub>	=	63.1317
" " "	=	63.0581
" " CO <sub>2</sub> in Geissler's bulb	=	.0736

Wt. of U-tube + CO<sub>2</sub> = 35.3669  
 " " " = 35.1620  
 " " CO<sub>2</sub> in U-tube = .2049

Wt. of CO<sub>2</sub> in Geissler's bulb = .0736  
 " " " " U-tube = .2049  
 Total wt. of CO<sub>2</sub> = .2785

Wt. of CO<sub>2</sub> + wt. of powder used, x 100 = percentage of CO<sub>2</sub>  
 .2785 ÷ 2.1844 = .1274 or 12.74% of CO<sub>2</sub>

### 3. Manhattan Baking Powder.

Weighing bottle + powder = 13.1035  
 " " = 11.0563  
 Wt. of powder used = 2.0472

Wt. of Geissler's bulb + CO<sub>2</sub> = 63.2424  
 " " " " = 63.1317  
 " " CO<sub>2</sub> in Geissler's bulb = .1107

Wt. of U-Tube + CO<sub>2</sub> = 35.4926  
 " " " = 35.3669  
 " " CO<sub>2</sub> in U-tube = .1257

Wt. of CO <sub>2</sub> in Geissler's bulb	= .1107
" " " " U-Tube	= <u>.1257</u>
Total wt. of CO <sub>2</sub>	= .2364

$$.2364 \div 2.0472 = .1105 \text{ or } 11.05\% \text{ of CO}_2$$

4. K. C. Baking Powder.

Weighing bottle + powder = 12.8937  
 " " = 10.7358  
 Wt. of powder used = 2.1579



Wt. of Geissler's bulb + CO<sub>2</sub> = 61.2065  
 " " " " = 61.1550  
 " " CO<sub>2</sub> in Geissler's bulb = .0515

Wt. of U-tube + CO<sub>2</sub> = 30.8421  
 " " " " = 30.7057  
 " " CO<sub>2</sub> in U-tube = .1364

Wt. of CO<sub>2</sub> in Geissler's bulb = .0515  
 " " " " U-tube = .1364  
 Total wt. of CO<sub>2</sub> = .1879

.1879 ÷ 2.1579 = .0870 or 8.7% of CO<sub>2</sub>

#### 5. Sheperd's Baking Powder.

Weighing bottle + powder = 13.0881  
 " " " " = 11.1890  
 Wt. of powder used = 1.8991

Wt. of Geissler's bulb + CO<sub>2</sub> = 63.3456  
 " " " " = 63.2424  
 " " CO<sub>2</sub> in Geissler's bulb = .1032

Wt. of U-tube + CO<sub>2</sub> = 35.5451  
 " " " " = 35.4926  
 " " CO<sub>2</sub> in U-tube = .0525

Wt. of CO<sub>2</sub> in Geissler's bulb = .1032  
 " " " " U-tube = .0525  
 Total wt. of CO<sub>2</sub> = .1557

.1557 ÷ 1.8991 = .0819 or 8.19% of CO<sub>2</sub>

#### 6. Calumet Baking Powder.

Weighing bottle + powder = 13.9146  
 " " " " = 11.8239  
 Wt. of powder used = 2.0907

Wt. of Geissler's bulb + CO<sub>2</sub> = 61.2691  
 " " " " = 61.2065  
 " " CO<sub>2</sub> in Geissler's bulb = .0626

Wt. of U-tube + CO<sub>2</sub> = 30.9326  
 " " " " = 30.8421  
 " " CO<sub>2</sub> in U-tube = .0905

Wt. of CO<sub>2</sub> in Geissler's bulb = .0626  
 " " " " U-tube = .0905  
 Total wt. of CO<sub>2</sub> = .1531

.1531 ÷ 2.0907 = .0732 or 7.32% of CO<sub>2</sub>