

SLBS ENGINEERING COLLEGE
DEPARTMENT OF PETROLEUM ENGINEERING

SESSION : 2016-2020



MANUAL

GAS TESTING LABORATORY

SPE7 Gas Testing Lab

B. Tech. (Petroleum Engineering)

Max. Marks: 100

Hours per Week:2

Practicals:

1. Determination of compositions of Gas with Gas Chromatography.
2. Determination of Reid Vapour Pressure.
3. Determination of % reserve of gas.
4. Determination of Gas gravity.
5. CO₂ detection.

EXPERIMENT - ①

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EXPERIMENT # 13

DETERMINATION OF NATURAL GAS COMPOSITION USING GLC CHROMATOGRAPH

⑥

THEORY

Chromatography is a discontinuous operation widely used in the determination of the composition of a wide range of fluids. Since the composition of natural gas is very important for the economic evaluation of any natural gas field, chromatography has always been a useful tool for the natural gas engineer.

Chromatography is defined as a combination of processes whereby the separation of a mixture of substances can be brought about by taking advantage of the different degrees to which they undergo adsorption, partition, or ion-exchange when passing over a material of a different phase. The mixture to be detected is normally dissolved in a fluid used to carry the mixture through a column of an adsorbent in powder form. Gas chromatography is a term used when the carrier fluid is a gas. Gas liquid chromatography or GLC is a partition kind of chromatography in which the column is packed with granules of inert supporting material (e.g. celtite, firebrick, or glass beads) coated with a non-volatile liquid such as silicon oil, polyethylene glycols, or detergents. The carrier gases normally used are hydrogen, nitrogen, helium, carbon dioxide, and argon.

The detector used in GLC is normally a single one or a series combination of the following two detectors:

- 1- Thermal conductivity detector (TCD): which is a catharometer consisting of a wire heated by an electric current. The temperature and subsequently the resistance of the catharometer varies with the composition of the material flowing over the wire.
- 2- Flame ionization detector (FID).

Equipment and Material

- 1- GLC with a TCD or a TCD connected in series with an FID.
- 2- A 9m chromosorb column for the separation of light hydrocarbons (C_2 to C_5^+) and CO_2 connected in series with a 2m molecular sieve column for the separation of elemental gases (H_2 , O_2 , N_2 , CH_4 , and CO). An empty column should be fitted between the two column to retard the passage of the elemental gases.

- 3- Recorder (an integrator, or a chart recorder).
- 4- Carrier gas (helium) and for the FID air and hydrogen supplies.

PROCEDURE

- 1- Make sure that all the gas lines, fittings, etc. are properly connected.
- 2- Turn on the carrier gas and for FID turn on the hydrogen and air.
- 3- Set the detector temperature control to 100°C in the Readout/Temperature Controller module.
- 4- Turn on the basic equipment power switch and turn on the power switch to the associated equipment.
- 5- Allow time for the detector oven temperature to reach the set temperature as indicated by the red ION DET or TC DET light on the ESP Panel turning off.

- 6- Set the following front panel controls:

COL OVEN to HTR and FAN
COLUMN LIMIT to 200°C
INITIAL TEMP to 25°C
RMT to OFF
INJ A to 20 (200°C)

FID

MODE to ION DET A
OUTPUT to (+)
RANGE to 10⁻¹⁰
ATTENUATOR to INFINITY

TCD CONTROLS:

FIL TEMP to 280
OUTPUT to (+)
RANGE to (0.05)
ATTENUATOR to INFINITY

- 7- Allow 1 hour for the temperature to stabilize.
- 8- Open the sample flow for 1 minute at a low pressure (1.5 to 3 atmos).
- 9- After 25 minutes you will note that no change could be detected in the output device reading. If, however, this is not the case (indicating the existence of C₅⁺) back flush the columns and a final peak will be observed in the output chart indicating the mole % of the C₅⁺ fraction.

10- Put all the temperature controls to room temperature and shut off the ovens.

11- Wait for 30 minutes till the temperatures drop then turn off the equipment.

12- Turn off the gases.

RESULTS

Using a standard chart determine the gases position in the final chart. Determine the mole percentage of each gas from the relative area of its peak. Report the composition of the natural gas sample in mole %. Make a simplified plot showing the relative peak time and area of each component.

METHOD 13

REF: Regs. 8-1-206 8- 5-101
 8-6-206 8-18-111
 10-9-301 10- 9-302

**DETERMINATION OF THE REID VAPOR PRESSURE OF
PETROLEUM PRODUCTS****1) PRINCIPLE**

The method provides for air partial saturation of petroleum products with a Reid vapor pressure below 26 lb using a Reid Vapor Pressure Bomb. The gasoline chamber portion of the vapor pressure apparatus is filled with a chilled sample and connected to the air chamber. The apparatus is immersed in a constant temperature bath ($100 \pm 0.2^{\circ}\text{F}$) and shaken periodically, until equilibrium is reached.

2) APPARATUS

2.1 Refrigerator or Freezer

2.2 Reid Vapor Pressure Bomb. This unit is equipped with a 4 in., 0 to 15 psig gauge (Fig. III-6).

2.3 Constant Temperature Water Bath ($100 \pm 0.2^{\circ}\text{F}$)

3) ANALYTICAL PROCEDURE

3.1 Handling of Sample.

3.1.1 The size of the sample container used in taking the sample shall not be less than 1 quart nor more than 1 gallon. The container shall be a solvent can, with a 1-3/4 in. lid opening, and filled to approximately 90% capacity.

3.1.2 In all cases after a sample has been taken, the container shall not be opened unless it has been cooled to $< 32^{\circ}\text{F}$.

- 3.1.3** Samples shall be put in ice as soon as taken, and transferred to the laboratory for immediate storage in the refrigerator, and stored there until the tests have been completed. Samples in leaky containers shall not be considered for tests but shall be discarded and new samples taken.
- 3.2 Preparation for Test.**
- 3.2.1 Air Saturation of Sample in Sample Container.** Remove the sample container from the refrigerator, unseal it examine it for its liquid content, which shall be approximately 90 percent of the container capacity. After the liquid content has been assured, reseal the container, shake vigorously and return it to the refrigerator.
- 3.2.2 Preparation of the Gasoline Chamber.** Place the open gasoline chamber and the sample transfer connection into the refrigerator for sufficient time to allow the chamber and connection to reach temperature (32°F to 40°F).
- 3.2.3 Preparation of Air Chamber (100°F Procedure).** After rinsing and purging the air chamber, connect the gauge to the air chamber. Immerse the air chamber to at least 1 in. above its top in the water bath maintained at $100 \pm 0.2^\circ\text{F}$ for not less than 10 minutes just before coupling it to the gasoline chamber. Do not remove the air chamber from the bath until the gasoline chamber has been filled as described in Section 3.3.
- 3.3 Sample Transfer.** With all in readiness, remove the chilled sample from the refrigerator, uncap it and insert the chilled transfer connection and air tube (Fig. III-5). Then place the empty chilled gasoline chamber over the sample delivery tube of the transfer connection. Invert the entire system rapidly so that the gasoline chamber is finally in an upright position with the delivery tube extending to within 1/4" of the bottom of the gasoline chamber. Fill the gasoline chamber to overflowing. Lightly tap the gasoline chamber against the work bench to insure that the sample is free of air bubbles. If any sample is displaced, refill the chamber to overflowing.
- 3.3.1 Assembly of Apparatus.** Without delay and as quickly as possible, attach the air chamber to the gasoline chamber.
- 3.3.2 Introduction of Apparatus to Bath.** Turn the assembled vapor pressure apparatus upside down to allow the sample in the gasoline chamber to run into the air chamber and shake vigorously in a direction parallel to the length of the apparatus. Immerse the assembled apparatus into the bath, maintained at $100 \pm 0.2^\circ\text{F}$, in an inclined position so that the connection of the gasoline and air chamber is below the water level and may be observed closely for leaks. If no

leaks are observed, immerse the apparatus to at least 1" above the top of the air chamber. Observe the apparatus for leaks throughout the test. If a leak is detected, discard the test.

3.3.3 Measurement of Vapor Pressure. After the assembled vapor pressure apparatus has been immersed in the bath for 5 minutes, tap the pressure gauge lightly and record the reading. Withdraw the apparatus from the bath, invert it, shake it vigorously and immediately place back in the bath. At intervals of about 10 minutes, repeat this agitation and gauge observation at least five times, until the last two gauge readings are constant. These operations normally require 60 to 90 minutes. Read the final gauge pressure to the nearest 0.05 psig for gauges with intermediate graduations of 0.1 psig and to the nearest 0.1 psig for gauges with graduation of 0.2 to 0.5 psig. The value obtained is the Reid Vapor Pressure of the sample under test.

3.3.4 A nomograph (Figs. III-7 and III-8) is used to convert Reid Vapor Pressure to true vapor pressure (See Ref. 4.2).

4) REFERENCE

- 4.1 Vapor Pressure of Petroleum Products (Reid Method) ASTM designation D-323-72, Book 23 ASTM Petroleum Products (1977).
- 4.2 Nomograph of Petroleum Products Vapor Pressure. Air Pollution Engineering Manual, 2nd Ed., AP40, 635-636.

EXPERIMENT NO. 3

Determination of Percent Reserve of Gas

INTRODUCTION

Natural gas is the naturally occurring hydrocarbon gas predominantly composed of methane. It is found in underground traps formed by structural and stratigraphic features called as reservoir. The prospect of accumulation of natural gas depends on coexistence of several factors in the subsurface which includes

- a) source rock,
- b) reservoir rock,
- c) trap,
- d) seal or cap rock etc.

Once a reservoir is discovered, it is necessary to estimate the reserve of it. Reserve is the quantity of gas that is recoverable from the underground gas deposits. In many places natural gas and crude oil are found coexisting because their origin and accumulation processes are similar in many accounts.

Reserves are estimated volumes of crude oils, condensate, natural gas, natural gas liquids and associated marketable substances anticipated to be commercially recoverable and marketable from a given date forward, under existing economic conditions, by established operating practice, and under current government regulations.

The process of estimating oil and gas reserves for a producing field continues throughout the life of the field. There is always uncertainty in making such estimates. The level of uncertainty is affected by the following factors:

- a) Reservoir type
- b) Source of reservoir energy,
- c) Quantity and quality of the geological, engineering, and geophysical data,
- d) Assumptions adopted when making the estimate,
- e) Available technology, and
- f) Experience and knowledge of the evaluator.

Due to the degree of uncertainty is inherent in the assessment the gas reserves are 'estimated' rather than 'measured'. Reserves are defined in different categories on the basis of uncertainty such as:

- A) Proved Reserve: up to 10% uncertainty
- b) Probable Reserve: 10 to 50% uncertainty.
- c) Possible Reserve: uncertainty level is as high as 90% .

CONVENTIONAL METHODS

Different methods can be used to estimate the reserve depending on the maturity of the reservoir. The commonly used methods are:

- a) Volumetric Method: at early stages.
- b) Material Balance Method: when production data is available.

Volumetric Method

In the early stages of development, reserves estimates are restricted to the volumetric calculations. The volumetric method entails determining the physical size of the reservoir, the pore volume within the rock matrix, and the fluid content within the void space. This provides an estimate of the hydrocarbons-in-place, from which ultimate recovery can be estimated by using an appropriate recovery factor. For a gas reservoir:

$$STOIIP = \frac{7758 Ah\phi(1 - S_{wc})}{B_{oi}}$$

Where:

GIIP = Gas initially in place (SCF)

VR = Reservoir volume (acre-ft)

ϕ = Porosity (fraction)

SWi = Interstitial water saturation (fraction)

Bgi = Initial gas formation volume factor (res bbl/SCF)

R.F = Recovery factor (fraction)

Volume

The reservoir volume is obtained from geologic and fluid pressure analysis data. The geologist provides contour maps of the top and base of the reservoir, as shown in Figure: 1.

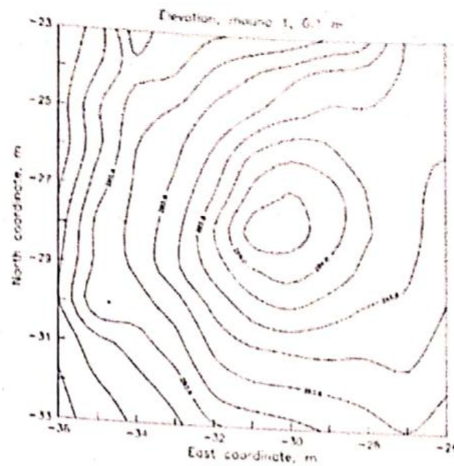


Figure 1: Contour Map

Such maps have contour lines drawn for every 20 feet, or so, the elevation can be determined by determining the gas water contact (GOC).

Porosity

Effective porosity of a sample is defined as the following ratio: Bulk volume Interconnected pore volume Effective Porosity Effective porosity of the reservoir rock can be determined from study of core analysis. The porosity of clean sandstones may be calculated from electric logs.

Interstitial water saturation, SWi

Water saturation is defined as the following ratio:

Water Saturation = Volume of water present in pore spaces/ volume of total pore spaces.

Interstitial or connate water saturation may be determined from electric logging information or by laboratory determinations run on cores by restored state, evaporation mercury injection, or centrifuge methods.

Formation volume factor ,Bg_i

Gas formation volume factor is the ratio of volume of gas at the reservoir condition to that of gas at the standard condition. Mathematically:

$$B_{gi} = \frac{V_{\text{reservoir condition}}}{V_{\text{base condition}}} = \frac{P_b T_i Z_i}{5.615 P_i T_b Z_b}$$

Where:

Bgi= Initial gas formation volume factor (res bbl/SCF)

- pb= Base pressure (psia)
- pi= Initial reservoir pressure (psia)
- Tb= Temperature at base conditions (OR)
- Ti= Temperature at initial conditions (OR)
- Zb= Gas deviation factor at base conditions
- Zi= Gas deviation factor at initial reservoir conditions.

Recovery factor (R.F)

Recovery factor is a number between zero and unity representing the fraction of recoverable oil. It depends on the following factors:

- a) Current economic circumstances.
- b) Environmental and ecological considerations.
- c) Governmental regulations as well as politics.
- d) Physics of the reservoir-fluid system.

Finally, GIP can be obtained by using all the parameters in the initial equation. Although volumetric method is the only method available at the early stage of the reservoir, the result might be harmed by erroneous determination of the reservoir area. Reservoir boundary is subject to large errors. Again, this method cannot provide the EUR (Expected Ultimate Recovery).

Material Balance Method

Material balance methods provide a simple, but effective alternative to volumetric methods for estimating not only original gas in place, but also gas reserve at any stage of reservoir depletion. Material balance equation is simply a statement of conservation of mass, or

(Original hydrocarbon mass)- (Produced hydrocarbon mass)=(Remaining hydrocarbon mass)

The general material balance equation reduces to the conventional gas material balance equation as:

$$\begin{aligned}
 & \boxed{N_p [B_o + (R_p - R_s) B_g] + W_p B_w} = N \boxed{E_o + E_{fw} + m E_g} + W_e + W_i B_w + G_i B_g \\
 & \uparrow \qquad \qquad \qquad \uparrow \qquad \qquad \qquad \uparrow \qquad \qquad \qquad \uparrow \qquad \qquad \qquad \uparrow \\
 & \text{Total Cumulative Production} \qquad \qquad \text{Total Expansion} \qquad \text{Aquifer Influx} \qquad \text{Water Injection} \qquad \text{Gas Injection}
 \end{aligned}
 \tag{3}$$

Without Water Influx

If there is no external energy from other sources, such as aquifer is applied, the reservoir is considered as completely enclosed. If $W_p=0$; $W_e=0$ and HCPV=constant, then the equation (3) reduces to:

$$P/Z = [P_i/Z_i - P_i/Z_i(G_p/G)] \dots\dots(4)$$

This equation is applicable for:

1. Constant reservoir temperature
2. No phase change in the reservoir
3. No water influx
4. No rock compaction
5. No connate water expansion.

From equation (4) a plot of p/Z vs. G_p can be found.

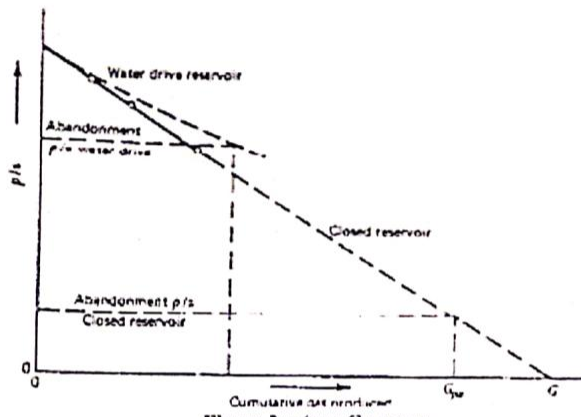


Figure 2: p/z vs G_p curve

Figure 2: p/z vs G_p curve

With water influx

If there is a water drive in the reservoir, then the pore volume is reduced by the amount equal to the volume of encroaching water. Now, considering water influx, equation (3) becomes:

$$GB_g = (G - G_p)B_g + W_e - W_p B_w \dots(5)$$

Now, using production data we can use equation (4) or (5) to determine the amount of gas remaining $(G - G_p)$ in the reservoir.

Material balance is a good tool to measure the reserve. But, for material balance it is needed to shut down the reservoir to determine the parameters. Thus, the data is as available as production data and the well manager is unwilling to do it frequently as it is not economically feasible.

EXPERIMENT 4

SPECIFIC GRAVITY DETERMINATION

Purpose:

This lab is performed to determine the specific gravity of soil by using a pycnometer. Specific gravity is the ratio of the mass of unit volume of soil at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature.

Standard Reference:

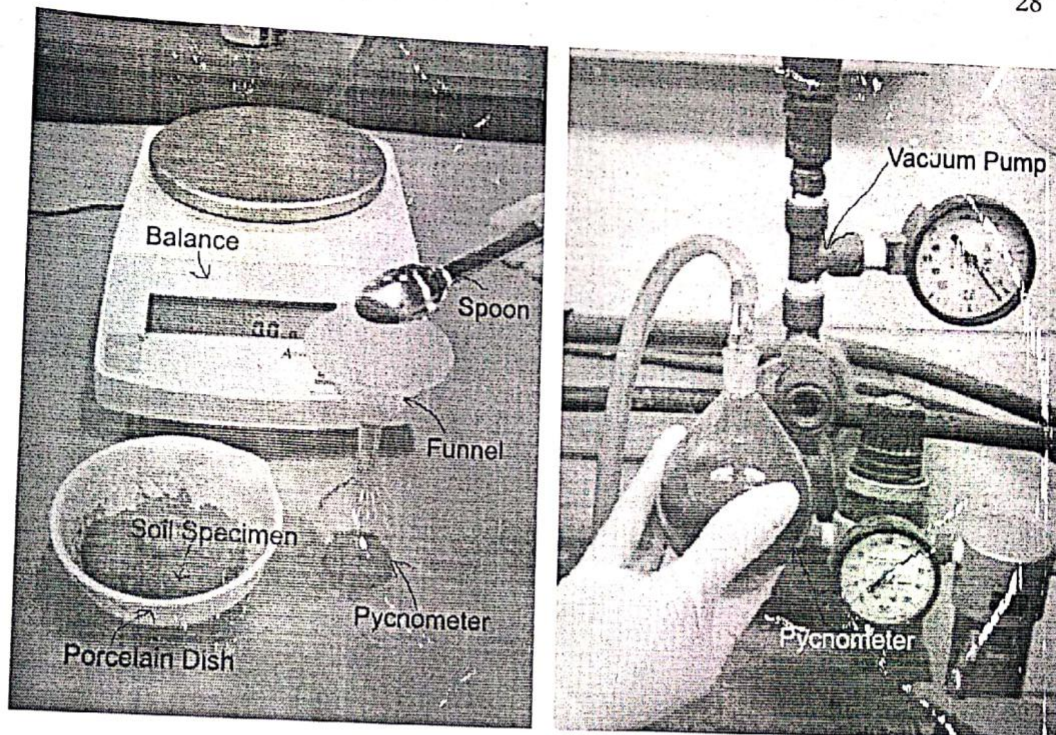
ASTM D 854-00 – Standard Test for Specific Gravity of Soil Solids by Water Pycnometer.

Significance:

The specific gravity of a soil is used in the phase relationship of air, water, and solids in a given volume of the soil.

Equipment:

Pycnometer, Balance, Vacuum pump, Funnel, Spoon.



Test Procedure:

- (1) Determine and record the weight of the empty clean and dry pycnometer, W_p .
- (2) Place 125g of a dry soil sample (passed through the sieve No. 10) in the pycnometer. Determine and record the weight of the pycnometer containing the dry soil, W_{PS} .
- (3) Add distilled water to fill about half to three-fourth of the pycnometer. Soak the sample for 10 minutes.
- (4) Apply a partial vacuum to the contents for 10 minutes longer, to remove the entrapped air.

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- (5) Stop the vacuum and carefully remove the vacuum line from pycnometer.
- (6) Fill the pycnometer with distilled (water to the mark), clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and contents, W_B .
- (7) Empty the pycnometer and clean it. Then fill it with distilled water only (to the mark). Clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and distilled water, W_A .
- (8) Empty the pycnometer and clean it.

Data Analysis:

Calculate the specific gravity of the soil solids using the following formula:

$$\text{Specific Gravity, } G_s = \frac{W_0}{W_0 + (W_A - W_B)}$$

Where:

W_0 = weight of sample of oven-dry soil, $g = W_{PS} - W_P$

W_A = weight of pycnometer filled with water

W_B = weight of pycnometer filled with water and soil

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EXAMPLE DATA

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SPECIFIC GRAVITY DETERMINATION DATA SHEET

Date Tested: September 10, 2002

Tested By: CEMM315 Class, Group A

Project Name: CEMM315 Lab

Sample Number: B-1, SS-1, 2'-3.5'

Sample Description: Gray silty clay

Specimen number	1	2
Pycnometer bottle number	96	37
W_p = Mass of empty, clean pycnometer (grams)	37.40	54.51
W_{ps} = Mass of empty pycnometer + dry soil (grams)	63.49	74.07
W_b = Mass of pycnometer + dry soil + water (grams)	153.61	165.76
W_a = Mass of pycnometer + water (grams)	137.37	153.70
Specific gravity (G_s)	2.65	2.61

Example Calculation: $W_p = 37.40$ g, $W_{ps} = 63.49$ g, $W_b = 153.61$ g,

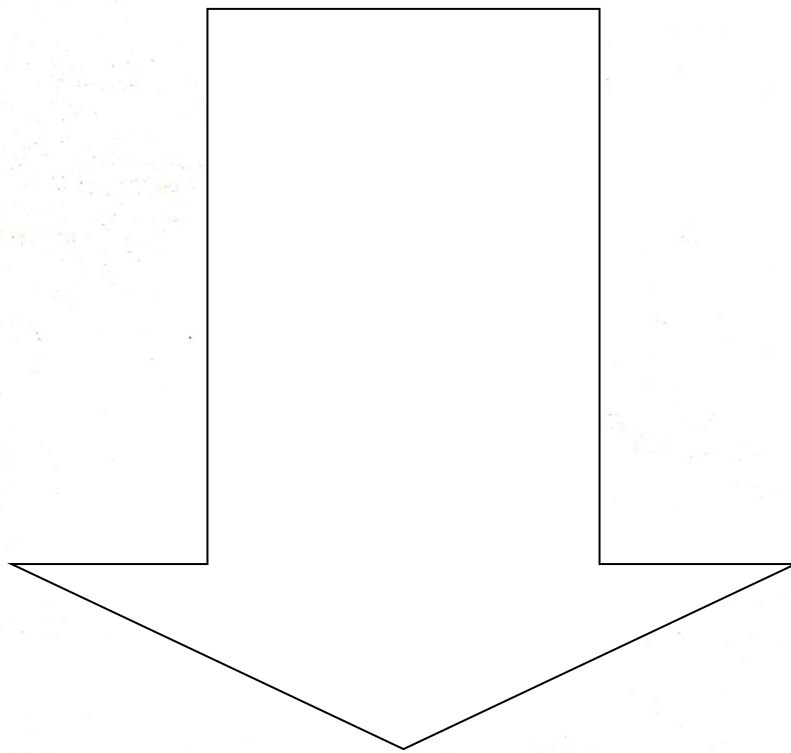
$$W_a = 137.37 \text{ g}$$

$$W_o = 63.49 - 37.40 = 26.09 \text{ g}$$

$$G_s = \frac{26.09}{26.09 + (137.37 - 153.61)} = 2.65$$

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SPECIFIC GRAVITY DETERMINATION DATA SHEET

Date Tested:

Tested By:

Project Name:

Sample Number:

Sample Description:

Specimen number	1	2
Pycnometer bottle number		
W_P = Mass of empty, clean pycnometer (grams)		
W_{PS} = Mass of empty pycnometer + dry soil (grams)		
W_B = Mass of pycnometer + dry soil + water (grams)		
W_A = Mass of pycnometer + water (grams)		
Specific Gravity (G_s)		

Calculations:

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EXPERIMENT NO. 5: Detecting Carbon Dioxide

Introduction:

The compound carbon dioxide (CO₂) is composed of the elements carbon and oxygen. The atmosphere contains about 1% carbon dioxide. Plants, through the process of *photosynthesis*, take CO₂ and water (H₂O) and produce sugar (C₆H₁₂O₆). Animal including human cells break sugar down to CO₂. This process is known as *respiration*. The CO₂ produced in respiration is carried away from the cells by the blood to the *capillaries* in the lungs. Gas exchange occurs between the capillaries and the *alveoli sacs* in the lungs. Finally CO₂ is exhaled when the *diaphragm muscle* moves up.

The presents of CO₂ can be detected by using the indicator solution bromothymol blue. BTB turns from blue to green to yellow when CO₂ gas is bubbled through it.

Purpose

The purpose of this investigation is to test exhaled breath, air, and the product of the reaction between baking soda and vinegar for the presence of carbon dioxide.

Materials

Per group: 4 small flasks, 1 large flask, bromothymol blue solution, 4 straws, air compressor, balloons filled with air, empty balloons, baking soda and vinegar, film canister, prongs, rinse bucket

Procedure

1. Put 25 ml of bromothymol blue solution into a flask. Gently blow through the straw into the solution. Continue blowing until the solution changes color. Record the color change in the table below.
2. Put a fresh 25 ml of BTB solution into the flask. Insert a straw into the mouth of the balloon containing air. Secure with your finger so that no air escapes around the outside of the straw. Insert the tip into the BTB and slowly release the air so that it bubbles through the solution. Record the color change in the table below.
3. Put a fresh 25 ml of BTB solution into the flask. Put 25 grams of baking soda into a larger clean dry flask. Fill a film can with vinegar. Use prongs to place the can into the flask without spilling it. Place a balloon over the mouth of the flask. Tilt the flask so that the vinegar spills into the baking soda. Collect the gas in the balloon and bubble it through the BTB as you did in step 2. Record the color change in the table below.

Data: CO₂ test BTB

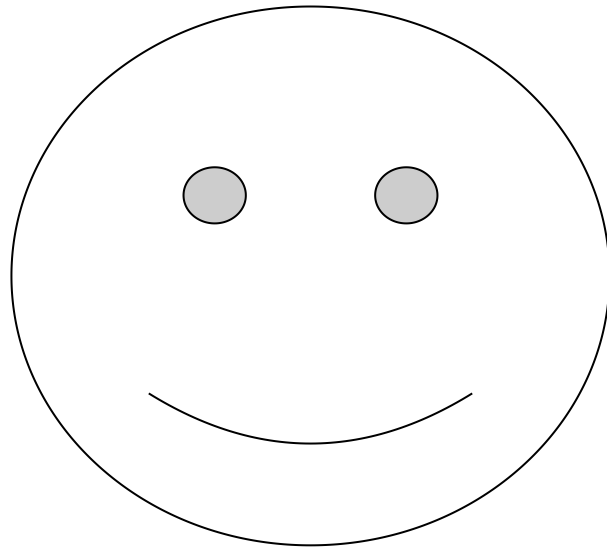
GAS	ORIGINAL COLOR	FINAL COLOR	CO ₂ presence + or -
Exhaled breath			
Air			
Vinegar + baking soda product			

Analysis:

1. Explain the result of the breath test.
2. Explain the result of the air test.
3. Explain the result of the chemical reaction between baking soda and vinegar test.
4. Which test showed the most CO₂?

Conclusion:

Write a paragraph explaining why CO₂ is in exhaled breath.



THE - END

