

# CARBONATION AND BOTTLING DEBUGGED

R.H. Molony RSCCAC. MNZIFST

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## Introduction

I first became interested in how Beverage Carbonation and Bottling actually worked in the early 1970s. A small winery I was working for had a new carbonation and bottling plant installed for its carbonated wines. After commissioning the plant could not operate faster than about 30% of its rated capacity due to foaming. This was blamed by the equipment supplier on properties of the product. However no amount of prefiltration protein removal etc from the product produced any significant resolution of the problem.

Realising that there might have been something else causing the problem I went right back to the basic molecular chemistry and physical chemistry of the process. The result of this were the theoretical models outlined in chapters (2) and (3). Using this theory I examined the existing plant process and had plant modifications made. After the process modifications the plant was able to operate continuously at approximately 120% of its original rated capacity. Benefits included lower CO<sub>2</sub> usage, less product loss, and a reduction in staff numbers. Unfortunately the staff reduction included members of my first wife's family.

This book looks first at a basic theory behind carbon dioxide solutions and their behaviour. Following this are applications of this theory to the practical aspects of carbonation and bottling of beverages.

Finally is a trouble shooting list of problems that can occur in a processing situation.

## CONTENTS

### THE SCIENCE

Chapter (1) The Carbonation of soda water and finished product.

Chapter (2) The Chemistry of solution of carbon dioxide.- General theory of solution of gases in liquids- Rates of carbonation - Boundary layer effects and surfactants.

Chapter (3) The chemistry of carbon dioxide release from carbonated liquids. Decomposition of combined carbon dioxide in solution.- Gas loss through the gas-liquid interface- Nucleation- Action of motes – Factors increasing the stability of carbonated liquids.

Chapter (4) The effects of non carbon dioxide gas on carbonation.- Non carbon dioxide gas in the gas phase - Sources of non carbon dioxide gas – Removal of dissolved gases from the feed liquid.

### THE MACHINES

Chapter (5) Tank carbonation and batch carbonation.

General operating procedure for tank carbonation. - Non carbon dioxide gas and tank carbonation - other factors - Product carbonation by dry ice.

Chapter (6) In line carbon dioxide injection.

In line sparging

Chapter (7) The vessel carbonator

The spray carbonator - The tower carbonator- Factors influencing carbonation in the vessel carbonator – Stabilisation of the carbon dioxide solution obtained in the vessel carbonator - The Carbo-cooler - Gas exchange in the carbonator vessel - The carbonator efficiency - Change of carbonation level.

Chapter (8) Filling machines and filling cycles for carbonated beverages -The snift filling machine - The full counter pressure filling machine - Clearing of the product - The gas tube purge - Effect of bottle contour on filling.

#### MISCELLANEOUS PRACTICAL TOPICS.

Non carbon dioxide gas in carbon dioxide storage systems.

Flavour removal from commercial carbon dioxide.

Carbon dioxide usage.

Pressure drops in the carbon dioxide line.

Pressure drops in the carbonated beverage line.

Moisture damage to bottles.

Special factors associated with the use of PET bottles.

Purging the carbonator vessel.

Leak testing the carbonation equipment.

#### FINDING THE PROBLEM

Where do we start

A collection of filling and carbonation problems.

## THE CARBONATION of SODA WATER and FINISHED PRODUCT.

Traditionally soft drinks were manufactured by filling soda water and syrup separately into a bottle, capping the bottle, and finally mixing the contents of the bottle. The more common procedure today is to mix the water and syrup together and carbonate the premixed product. In addition products such as fruit juices and wines are now sometimes carbonated directly.

Differences between soda water and finished product occur during carbonation and filling, In general finished product is more difficult to carbonate and fill than soda water. Product mix is generally strongly buffered to a low pH. This in turn raises the equilibrium partial pressure of carbon dioxide in product mix as compared to a similar volume dissolved in soda water. Product also contains sugars, alcohols, salts, etc. These by simple dilution of the water molecules present lower the water content of the solution. Also sugars and salts in solution due to hydration effects remove a large number of water molecules that would otherwise be available for combination with carbon dioxide. Any polar molecule or ion is capable of doing this. In product the overall number of water molecules available for the hydration of carbon dioxide is considerably reduced. This may in part be offset by the combination of carbon dioxide with other compounds other than water in the solution. All these effects change the ratio of the concentrations of carbon dioxide in the gas and liquid phases.

Dynamic effects also occur. Due to the lower availability of water molecules for combination with carbon dioxide, the rate of combination with carbon dioxide is lower. Secondly the presence of large solvated molecules would be expected to obstruct the movement of carbon dioxide molecules. The effect of this is to reduce the frequency of collisions between carbon dioxide molecules and available water molecules. The overall is a lower rate of stabilisation of carbon dioxide solution in product as compared to soda water under the same conditions.

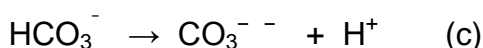
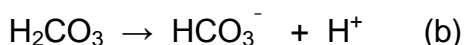
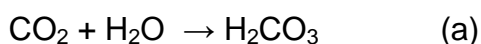
At bottling and capping viscosity effects become noticeable. High brix products and also some natural product require a longer filling time due principally to the longer time required for the product to clear before pressure release. (ie. a longer time is required for small gas bubbles to settle out). Colloids and other materials in some products may also cause nucleation necessitating a higher degree of product stabilisation prior to bottling. Overall the carbonation of product mix is more difficult than the simple carbonation of water.

## Chapter (2)

### The CHEMISTRY of SOLUTION of CARBON DIOXIDE.

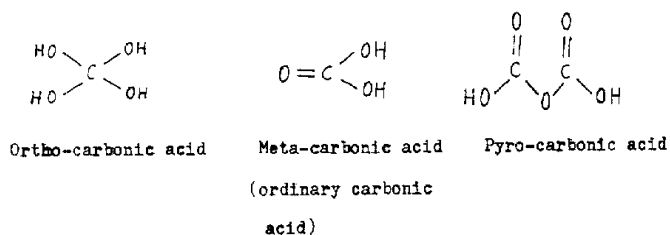
An aqueous solution of carbon dioxide is not a simple solution but rather a complex mixture of compounds in solution. These compounds are also in equilibrium with one another. The traditional picture of carbon dioxide dissolving in water to produce carbonic (actually meta-carbonic) acid which then dissociates is only part of the picture.

The traditional equations for the solution of carbon dioxide are as follows;



That the traditional chemical equations do not show the whole picture is shown by the well known increase in stability of a supersaturated of carbon dioxide after standing for a short time.

A number of other compounds of carbon dioxide and water are possible. Including hydrogen bonded carbon dioxide, ortho-carbonic acid and pyrocarbonic acid.



Although ortho-carbonic acid, pyro-carbonic acid and their salts have not been isolated (probably due to their instability) there is no reason to suggest that ortho and pyro-carbonic acids do not exist in dilute solution. These compounds may well contribute to the non meta-carbonic bound carbon dioxide.

In both wine and fruit juice a large number of compounds other than water occur which bind carbon dioxide. Both wines and fruit juices contain a wide range of proteins that are potentially able to reversibly bind carbon dioxide.

Much smaller organic molecules may contribute to carbon dioxide binding. For example an extremely unstable compound of diethyl ether and carbon dioxide is known. this compound decomposes at 50° C in the pure state. Many compounds similar to this are possible particularly in wine. A number of carbonic acid ethanol esters are known to occur in wine some of them are extremely labile.

#### A GENERAL THEORY OF SOLUTION OF GASES IN LIQUIDS.

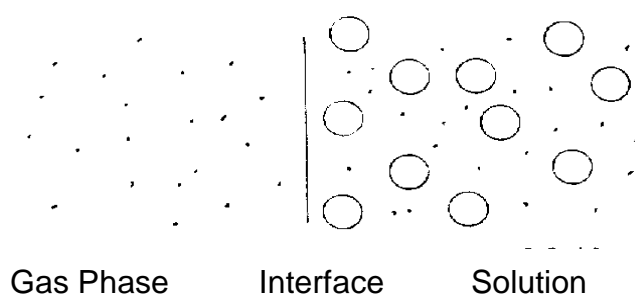
This theory considers an ideal gas in solution and looks at the way in which real gases in solution deviate from this model. The ideal unbound gas of the theory does not exist. All gases interact to some extent with the solvent molecules.

Consider a gas liquid Interface with the gas molecules being small in size with respect to the solvent “molecules” (the individual solvent molecules in this theory can be linked together in chains by hydrogen bonding to produce large solvent “molecules” cf (the structure of water). In the simplest case with no molecular interactions the concentration of gas in the free space of the liquid is equal to the concentration of gas in the gas phase under equilibrium conditions.

i.e.

$$GAS_{Liq\ phase} = \{ Total\ Vol - Vol\ Solvent\ mols \} \times GAS_{gas\ phase}$$

Higher gas solubility's than this indicate solvent-gas bonding.



A gas solution with no solvent gas interactions.

The simplest bonding is found in the inert gases where charges are induced by distortion of the electron cloud resulting in a dipole.

If helium is completely unbound in solution (some bonding would be expected in solution) an aqueous solution of nitrogen would contain 50%



bound nitrogen and an aqueous solution of oxide would contain 98% bound carbon dioxide.

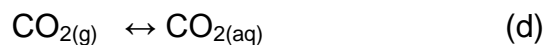
If the ratio of bound to unbound gas remains constant Henry's law concerning the solubility of gases in solution is obeyed. At high gas concentrations in solution many of the binding sites in the solution are occupied and deviations from Henry's law are observed.

Solvent molecules tend to align themselves at the interface of the gas and liquid phases. As a result the free paths through the interface tend to be narrower than those in the body of the liquid. The interface tends to act as a membrane between the two phases.

#### RATES of CARBONATION

As carbonation is primarily a dynamic process the rate of getting the gas into solution is of greater importance than the final equilibrium state. In fact the final gas content when using a vessel carbonator is controlled by the rate of gas absorption.

Carbonation can be considered as a series of reversible operations of which the forward operations constitute carbonation. The first operation is the passage of molecular carbon dioxide through the gas-liquid interface.



The rate of this first reaction is proportional to the partial pressure differential of the gas between each side of the gas-liquid interface, the interfacial area, and the velocity of the carbon dioxide molecules.

$$\begin{aligned} \text{i.e. Rate} &\propto P_{\text{CO}_{2(g)}} - P_{\text{CO}_{2(aq)}} \\ &\propto \Delta P_{\text{CO}_2} \end{aligned} \quad (ii)$$

$$\begin{aligned} -dp/dt &= K_1 P \\ dt &= K_2 \frac{1}{p} dp \end{aligned} \quad (iii)$$

On integration this becomes

$$\begin{aligned} t &= K_2 \text{Log } P + c \\ \Delta t &= K_2 (\text{Log } P_2 - \text{Log } P_1) \end{aligned} \quad (iv)$$

$\Delta t$  = Elapsed time

$P_2$  = Final partial pressure differential

$P_1$  = Initial partial pressure differential

$K_2$  = A constant

As the rate of collision of carbon dioxide molecules with the interface controls the speed of the operation.

$$\text{Rate} = K_3 A V$$

A = The interfacial area

V = Molecular velocity of the CO<sub>2</sub> Molecule

$$\text{As } KE = \frac{1}{2} M V^2$$

$$\text{Rate} = K_4 A T$$

K<sub>3</sub> and K<sub>4</sub> are rate constants the value of which is dependent on the individual system.

T is the absolute temperature on the individual system in absolute temperature.

The rate of passage of carbon dioxide molecules through the gas-liquid interface is increased by ;

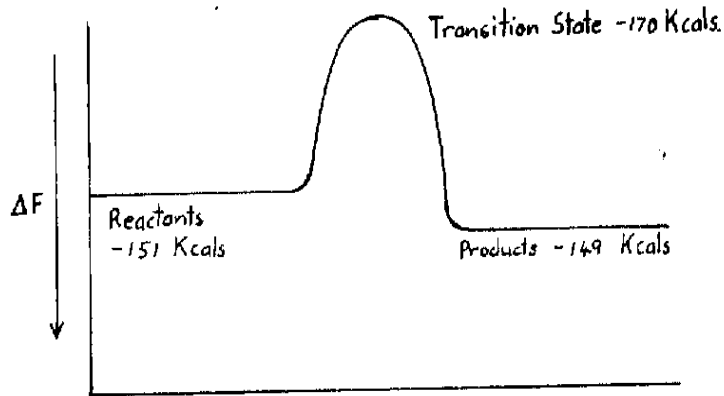
- (1) Higher carbon dioxide pressure
- (2) Larger liquid surface area
- (3) Increased temperature. This effect is limited by the increase in equilibrium partial pressure of carbon dioxide solutions with increasing temperature.

The second process is the reaction of uncombined carbon dioxide in the liquid phase with components of that liquid phase ( principally water) to produce the compounds discussed earlier.



This rate is dependent on the collision of uncombined carbon dioxide molecules with other components in the solution. The reaction is therefore at least first order with respect to uncombined carbon dioxide however the overall reaction will probably be first order. One of the reactants is the water solvent.

Carbonic acid formation has an activation energy of formation of approximately 19 kcals/mole



Energy diagram for the reaction  $\text{H}_2\text{O} + \text{CO}_2 = \text{H}_2\text{CO}_3$

The rate of carbonic acid formation at room temperature is measurably slow obeying the equation.

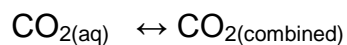
$$d[\text{CO}_2] / dt = K_{\text{CO}_2} [\text{CO}_2]$$

Where  $K_{\text{CO}_2} = 0.03 \text{ sec}^{-1}$

TIME TAKEN FOR 95% COMBINATION OF CARBON DIOXIDE AND WATER.

Temp°C	Time
0	19
5	10
10	5
15	3
20	1¾
25	1
30	½

The reactions with other components in the liquid phase are likely to show a similar relationship. The rate of the reaction



therefore shows a very marked dependence on both temperature and the concentration of uncombined carbon dioxide. Overall the extent of the reaction of carbon dioxide in the gas phase to the final combined state is dependent on the partial pressure of carbon dioxide in the gas phase, the temperature, the gas-liquid interracial area and the carbon dioxide liquid contact time.

#### BOUNDARY LAYER EFFECTS

Boundary layer effects can occur on both sides of the gas-liquid interface and have a significant effect on the gas transfer rate.

When the gas phase consists of pure carbon dioxide (with no non carbon dioxide gas of low solubility) no boundary layer effect occurs on the gas phase side of the interface. With a gas mixture of carbon dioxide and nitrogen the gas layer adjacent to the interface becomes depleted in carbon dioxide. Carbon dioxide must then diffuse through both the gas-liquid interface and the depletion layer. This slows down the absorption of carbon dioxide by the liquid.

Adjacent to the interface on the liquid side a concentration gradient of dissolved carbon dioxide tends to occur. The highest concentration of carbon dioxide occurs nearest to the interface. This reduces the carbon dioxide partial pressure across the interface and consequently the rate of carbon dioxide transfer. Agitation and turbulence reduce the boundary layer effects. In the absence of high turbulence where density inversions occur the salt fingering effect may help to keep the gradient thickness to a minimum. Liquid side concentration gradients would probably have their greatest effect in tank carbonation and in the absorption of gases from the filter bowl headspace.

Surfactants when present can produce molecular layers at the gas-liquid interface. These molecular layers are able to markedly retard the gas transfer rate through the interface. In the vessel carbonator this shows up as a higher than expected carbonator overpressure to produce the required carbonation. The effect is usually product specific. Clouding agents based on oil emulsions contain surfactants as stabilisers. These surfactants commonly

retard the rate of carbon dioxide solution. If the effect is excessive, problems may be reduced by reformulating the product. In some cases, however, the removal or reduction of the concentration of an objectionable component may not be an option. eg. Gingerol, the major flavouring component in ginger, is a surfactant.

It is possible to identify potential problems during product formulation. In many cases, there is a general correlation between the product surface tension and the rate of gas absorption in the carbonator. Surface tension can be simply and easily measured by the Du Nuoy ring pull balance. Accidental contamination by cleaning agents containing surfactants, may on occasions produce a non-product specific carbonation difficulty. Simple surface tension measurements can assist in the identification of this problem.

## THE CHEMISTRY OF CARBON DIOXIDE RELEASE FROM CARBONATED LIQUIDS

The release of carbon dioxide from carbonated liquids is essentially the reversal of the processes taking place during carbonation. One exception is that gas may not only pass through the gas-liquid interface but that bubble initiation may occur within the body of the liquid.

### DECOMPOSITION OF COMBINED CARBON DIOXIDE IN SOLUTION.

Carbon dioxide in the gas phase is only produced from the unbound portion of carbon dioxide in the solution. For the gas in the combined state chemical bonds must be broken to provide uncombined carbon dioxide. This carbon dioxide can then be released as a gas. As the greater proportion of carbon dioxide in solution is in the combined state, the breakdown of combined carbon dioxide is of considerable importance in controlling the rate of gas release.

The frequency with which covalent and hydrogen bonds are ruptured is dependent on the amplitude of bond stretching. This means that the rate of breakdown of combined carbon dioxide is highly temperature dependent. Catalysis may also be of importance in the decomposition of combined carbon dioxide. The catalysts may be chemically active surfaces or possibly metal compounds in the solution. Iron and copper compounds are known to have caused trouble in this respect in beer. The presence of proteins is particularly important if there is a possibility that the enzyme carbonic anhydrase may be present. In this case the destruction of proteins by heat or proteases could assist stabilisation.

### GAS LOSS THROUGH THE GAS-LIQUID INTERFACE

The major loss of gas from solution occurs by diffusion through the gas liquid interface.

The rate of diffusion through the gas-liquid interface is proportional to both the

partial pressure differential across the interface and the velocity of the carbon dioxide molecules.

$$\begin{aligned} \text{ie. Rate} &\propto \text{PCO}_2(\text{aq}) - \text{PCO}_2(\text{g}) \\ \text{also Rate} &\propto \text{Velocity of the CO}_2 \text{ molecules} \\ &\propto \sqrt{T} \end{aligned}$$

## NUCLEATION:

Nucleation is important in the loss of gas from a supersaturated solution due in a large part to the formation of new gas-liquid interfaces. The gushing of product when the pressure is released after filling is primarily a result of nucleation. Nucleation may occur at the surface of the bottle, in the liquid, or on motes in the solution. After nucleation the bubbles rise growing due to absorption of gas from the solution.

For a bubble to be produced a certain number of molecules must collide to produce a stable inhomogeneity or bubble. Below a critical size the molecules will tend to disperse back into the liquid. Above this size the bubble will grow. For water at room temperature the critical inhomogeneity radius is about 10 angstrom (10<sup>-7</sup>) (after Aphel) This figure refers to water under low pressure a similar but not necessarily the same figure will apply to a supersaturated solution of carbon dioxide at atmospheric pressure.

The probability that any molecule or gas will be at a particular point in the liquid is proportional to the concentration of gas and their velocity. From this the probability that n molecules will collide at a given point is given by the equation.

$$\text{Frequency} = K [\text{gas}]^n V^n \quad (\text{iii})$$

From This

$$\text{Nucleation Frequency} = K [\text{gas}]^n T^{n/2} M^{-n/2} \quad (\text{iv})$$

Where K= a constant

[gas] = Conc of uncombined gas in soln.

T = Temperature absolute

M = Molecular wt of gas molecules

N = No of molecules required for a stable inhomogeneity

The number of molecules required to form a stable inhomogeneity is dependent on the applied pressure.

This equation is for homogenous nucleation however there is a similar equation for nucleation by motes.

For nucleation by motes (heterogeneous nucleation) an extra term is introduced by the concentration of active motes.

### Heterogeneous Nucleation

Where  $K =$  a constant Nucleation Frequency  $= K m [\text{gas}]^n T^{n/2} M^{-n/2}$  (v)

$m =$  The concentration of active motes

$[\text{gas}] =$  Conc of uncombined gas in soln.

$T =$  Temperature absolute

$M =$  Molecular wt of gas molecules

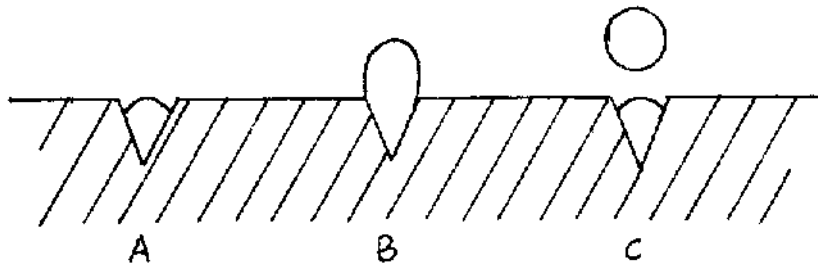
$N =$  No of molecules required for a stable inhomogeneity

Nucleation caused by motes is generally more important than homogenous nucleation. The constants  $K$  and the number of gas molecules required to form a stable inhomogeneity are different for homogenous and heterogeneous nucleation.

### ACTION of MOTES

One theory of the action of motes suggests that small bubbles of inert gas (any gas of low solubility) are trapped in crevices on the surface of either motes or the surface of the bottle. These bubbles then grow by the absorption of gas according to eqns. (i) and (ii) until the buoyancy of the bubble overcomes the surface tension of the liquid and a portion of the bubble is released.





Stages in the nucleation of gas bubbles on a mote.

This explains quite well the efficiency of the common practice of wetting bottles before bottling to help prevent bubble formation and gushing. Sometimes stearates etc. (as a coating applied to the outside of the bottle to prevent scuffing) contaminate the inside of the bottle. When this occurs the bottle surface is not properly wetted out and gushing of the bottle usually occurs.

On the old syruper type filling machines if syrups containing fine air bubbles are used similar foaming occurs after filling. Typically the bubbles may be seen rising from the surface of the syrup in the bottom of the bottle.

Another theory of nucleation on motes suggests that an active surface absorbs a multilayer of gas. This then acts similarly to a gas liquid interface which then grows by gas absorption from the solution releasing bubbles. Both these mechanism are possible and both my contribute to heterogeneous nucleation

Sound vibrations may induce homogenous nucleation. Strong sound vibrations or shock wares produce in the body of the liquid a series of alternate compressions and rarefactions. In the rarefactions the number of gas molecules required to form a stable inhomogeneity is reduced hence an increase in nucleation frequency occurs. An example of this is the gushing produced when an open bottle of carbonated beverage is tapped or shaken. The short persistence of product instability after shaking is the basis of the practical joke where the victim is given a vigorously shaken but unopened container of a carbonated beverage.

Carbon dioxide release in the product is probably caused by the rarefactions. This released carbon dioxide is then immediately redissolved in the product. This dissolution and resolution process causes a short term increase in the amount of poorly bound carbon dioxide in solution hence the instability. Dispersion of fine bubbles of non carbon dioxide headspace gas may also be a contributing factor.

Due to higher molecular velocity and the number of molecules required to form a stable inhomogeneity the nucleation frequency has a high degree of temperature dependence. This combined with the increase in bond fission with increasing temperature explains the instability of supersaturated solutions if at higher temperatures.

## FACTORS INCREASING THE STABILITY OF CARBONATED LIQUIDS

- (1) Low temperatures slow the decomposition of combined carbon dioxide and inhibit bubble formation.
- (2) Fine filtration to remove suspended solids which may catalyse the decomposition of combined carbon dioxide or nucleate bubble formation.
- (3) Protein inactivation or removal to destroy carbonic anhydrase if present.
- (4) Efficient wetting of the bottle surface to remove air bubbles in crevices and pits.
- (5) Minimisation of vibrations in the liquid after pressure release.
- (6) As large proportion of the carbon dioxide in the combined state as possible.

## THE EFFECTS OF NON-CARBON DIOXIDE GAS ON CARBONATION

Non carbon dioxide gas has two types of effects on carbonation. One due to its presence in the gas phase during carbonation the other due to its presence in the liquid phase during and after carbonation.

### NON-CARBON DIOXIDE GAS IN THE GAS PHASE

The presence of non-carbon dioxide gas in the gas phase has the principal effect of diluting the carbon dioxide in the gas phase.

As the rate of gas absorption through the gas-liquid interface is proportional to the carbon dioxide partial pressure differential across the interface ( eqn. (ii) Chap 2) the absorption of carbon dioxide is markedly slowed down.

Eqn. (ii) Ch 2 Then becomes

$$-dp/dt = K_1 [(P_t - P_a) - P_{CO_2(aq)}]$$

where  $dp/dt$  = Rate of gas transfer through the interface.

$P_t$  = Total pressure in the gas phase.

$P_a$  = Partial Pressure of non CO<sub>2</sub> gas.

$P_{CO_2(aq)}$  = CO<sub>2</sub> Partial pressure in the liquid phase

The rate of carbon dioxide absorption is even more adversely affected if adequate agitation does not occur. A depletion layer of gas adjacent to the interface may form and further retard the rate of gas absorption.

When appreciable non-carbon dioxide gas is present, at equilibrium the carbon dioxide content of the liquid phase is lower than would be expected from the total gas pressure of the system.

## NON-CARBON DIOXIDE GAS IN THE LIQUID PHASE.

Apart from any problems caused by non-carbon dioxide gas in the gas phase this gas can dissolve in the liquid phase and cause problems later. Non-carbon dioxide gas in solution can have a marked effect on nucleation after pressure release at bottling. Mixed gas nucleation may occur due to the collision of both carbon dioxide and non-carbon dioxide gas components of the solution.

For nucleation of a single gas solution: Eqn. (iv) Ch 3.

$$\text{Nucleation frequency} = K [\text{gas}]^n T^{n/2} M^{n/2}$$

As molecules of more than one gas may collide and produce nucleation.

$$\text{Nucleation} = K[\text{CO}_2]^{na} T^{na} M_{\text{CO}_2}^{-na/2} [\text{B}]^{nb} T^{nb/2} M_b^{-nb/2}$$

Where  $K = A$  constant

$[\text{CO}_2]$  = Concentration of uncombined carbon dioxide

$[\text{B}]$  = Concentration of uncombined non- $\text{CO}_2$  gas.

$M_{\text{CO}_2}$  = Molecular wt of carbon dioxide

$M_b$  = Molecular wt of non-carbon dioxide gas.

$T$  = Temperature absolute

$na$  = No of molecules of carbon dioxide at the nucleation point.

$nb$  = No of molecules of non-carbon dioxide gas at the nucleation point.

Such that  $na + nb = n$  the total number of gas molecules required to form a critical inhomogeneity.

Points to note.

- (1) Only gas in the uncombined state can contribute to nucleation. Therefore at the same total gas content solutions of gases with a low combined gas

content have a much higher tendency to induce nucleation.

% Uncombined gas when dissolved in water at  
20°C (Assuming that helium is 100% uncombined)

Gas	%Uncombined
Helium	100
Hydrogen	47
Nitrogen	50
Oxygen	26
CO <sub>2</sub>	0.87
Air	43

The importance of air contamination in causing gushing and gas loss can hardly be underestimated. This is primarily due to the nitrogen content.

- (2) With increasing degrees of carbonation lower non-carbon dioxide gas levels become effective in the induction of nucleation.
- (3) A marked increase in nucleation frequency occurs with increasing temperature.
- (4) Total uncombined gas is important in causing nucleation.  
All dissolved unbound gas contributes to the formation of a stable inhomogeneity.
- (5) Gases of low molecular weight have a much tendency to induce nucleation due to their molecular velocity.

All these effects are multiplied many times due to the magnitude of n

### SOURCES of NON CARBON DIOXIDE GAS.

- (i) Air drawn in through leaking pump glands.
- (2) Air or nitrogen dissolved in the feed liquid being carbonated.  
Nitrogen can be produced from yeast by autolysis during the production of wine by the Charmat process.
- (3) Contamination of the carbon dioxide used for carbonation

During carbonation gas exchange can take place both to and from the liquid phase.

## REMOVAL OF DISSOLVED GASES FROM THE FEED LIQUID

(1) Sparging with carbon dioxide gas before carbonation.

(2) Mechanical deaeration with a vacuum deaerator.

The common practice of adding an oxygen scavenger while removing oxygen has no effect on the dissolved nitrogen, the major objectionable gas. Oxygen scavengers may be added for other reasons.

## Chapter (5)

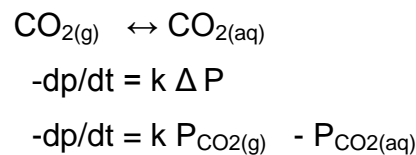
### TANK CARBONATION and BATCH CARBONATION

The tank carbonation process is the most commonly used method of carbonation used in the wine industry. Due to the prolonged contact time the carbon dioxide solution obtained is the most stable of any process.

The basic process consists of injecting carbon dioxide into product contained in a pressure vessel with agitation until the desired carbon dioxide level is obtained.

Due to the small surface area to volume relationship in the tank carbonation process, the rate controlling step is the passage of carbon dioxide through the gas-liquid interface.

The rate of carbonation is controlled by the equation



From this the carbonation rate can be greatly increased by the injection of carbon dioxide into the pressure vessel under a large over pressure. For example where a final equilibrium pressure of 30 psi is required the tank could be pressurised to 60 psi. with carbon dioxide and the required weight of carbon dioxide is injected in. This reduces the time taken for gas absorption. The time taken for gas absorption can be calculated from the equation

$$t = k \log \left[ \frac{P_{\text{tank}} - P_{\text{final}}}{P_{\text{tank}} - P_{\text{initial}}} \right]$$

where t = Time taken to reach the final pressure of carbon dioxide in solution.

$P_{\text{tank}}$  = Tank head pressure

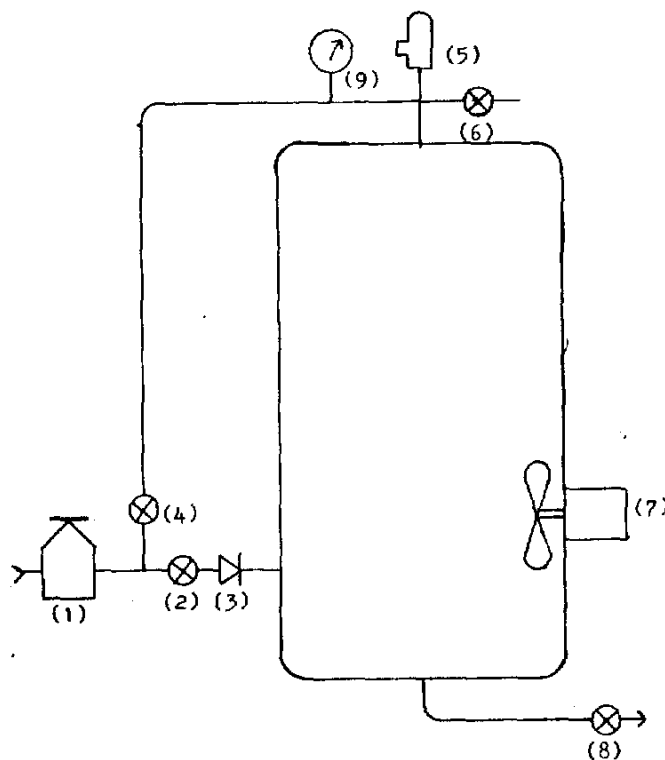
$P_{\text{injital}}$  = Initial partial pressure of CO<sub>2</sub> in solution

$P_{\text{final}}$  = Final partial pressure of CO<sub>2</sub> in solution

k = A constant.

The constant is dependent on the individual system and is controlled by a variety of factors. As the reaction rate is controlled by the area of the gas-liquid interface any method of increasing this area will increase the carbonation rate. A common method of increasing the interracial area is by the injection of the carbon dioxide as fine bubbles into the bottom of the tank. Injection of the gas into the bottom of the tank results in a longer bubble life which increases the apparent surface area.

The temperature effects on the rate of gas absorption are small as the rate is proportional to the square root of the absolute temperature.



### CARBONATION TANK AND ASSOCIATED EQUIPMENT.

- (1.) Carbon Dioxide inlet regulator
- (2.) Inlet Flow control valve
- (3.) Gas non return valve
- (4.) Gas counter pressure supply valve
- (5.) Safety Valve
- (6.) Tank headspace bleed valve.
- (7.) Tank stirrer and motor.
- (8) Product outlet valve
- (9) Pressure gauge



## OPERATING PROCEDURE FOR THE CARBONATION TANK.

- (1) Open the tank headspace bleed valve and the product outlet valve.
- (2) Pump the product in through product outlet valve. The tank should not be filled completely. About 5% of the tank should be left as headspace.
- (3) Close off both the headspace bleed valve and the product outlet valve.
- (4) Open the gas counterpressure supply valve and pressurise the tank to about 4 atmospheres.
- (5) Close the gas counter pressure valve and open the headspace bleed valve to release all the head space gas.
- (6) Repeat steps (4) and (5) at least twice more.
- (7) Close off the headspace bleed valve.
- (8) Start the tank stirrer and slowly inject the required amount of carbon dioxide in by opening the gas inlet flow control valve the supply regulator should be set at least one atmosphere above the final equilibrium pressure. If for any reason the tank pressure rises too high close off the inlet flow control valve until the excess carbon dioxide is absorbed.
- (9) When the required amount of gas has been absorbed shut off the inlet flow control valve.
- (10) Agitate the tank until the tank pressure does not fall any more turn off the stirrer and allow the tank to stand for sometime.
- (11) When the tank pressure does not fall any more open the head space bleed valve and slowly bleed off the headspace gas.
- (12) As soon as the headspace gas is bled off close the bleed valve.
- (13) Repressurise the head space by opening the gas counter pressure supply valve.
- (14) Connect the filling plant to the product outlet valve and open the product valve and the gas counter pressure supply valve. During the filling a head pressure of at least half an atmosphere above the equilibrium partial pressure of carbon dioxide in the solution should be maintained above the product.

## NON-CARBON DIOXIDE GAS AND TANK CARBONATION

The effects of non-carbon dioxide gas in the tank head space (is the reduction of the partial pressure of carbon dioxide in the head space and the absorption of non carbon dioxide gas by the product.) can be minimized by flushing the headspace with carbon dioxide prior to carbonation.

Dissolved non-carbon dioxide gas can readily be removed by bleeding of the headspace gas after carbonation and repressurising the tank with pure carbon dioxide prior to bottling. As only uncombined gas is lost through nucleation and bubble growth gases which are poorly combined are preferentially lost. Little carbon dioxide is lost. This is because at least 98% of the carbon dioxide in solution is in the bound state where as nitrogen in solution only is about 50% bound.

## OTHER FACTORS

Stirring the tank during gas absorption is very important as density and concentration gradients may be produced. These density gradients could be induced by either temperature or differences in density. Possible density differences may occur due to varying amounts of carbon dioxide in solution. The overall effect would be variations in carbonation levels as the product is drawn off from the tank for bottling.

The tank carbonation process is relatively simple and comparatively foolproof. The process however has the disadvantage that large numbers of expensive pressure tanks are required for even a comparatively low production. The "charmat" process is a special case of tank carbonation. Carbon dioxide is produced endogenously by fermentation instead of being introduced from the outside. In addition to carbon dioxide the yeast can produce other substances. The claimed superior flavour of the charmat process wine is supposedly due to flavour compounds produced during pressure fermentation. In addition nitrogen gas can be produced during yeast autolysis. Amino acids and other nitrogen compounds can become deaminated during autolysis. This nitrogen can cause foaming troubles during the filling operations.

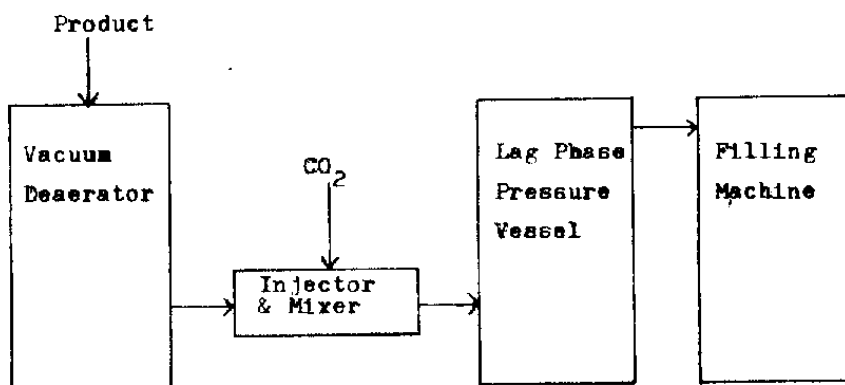
## PRODUCT CARBONATION BY DRY ICE.

Drinks may be carbonated by adding a pellet of dry ice to the bottle immediately before sealing. The carbon dioxide gas produced by the dry ice dissolves in the product producing & carbonated beverage. Automatic pelleting machines were available at one time. These machines not only made the dry ice pellet but added it to the bottles passing on the line prior to capping. A standard  $\frac{3}{4}$  litre bottle requires a six gram pellet to give a carbonation of four volumes. Strong bottles are required as high pressures can be produced during the gas absorption.

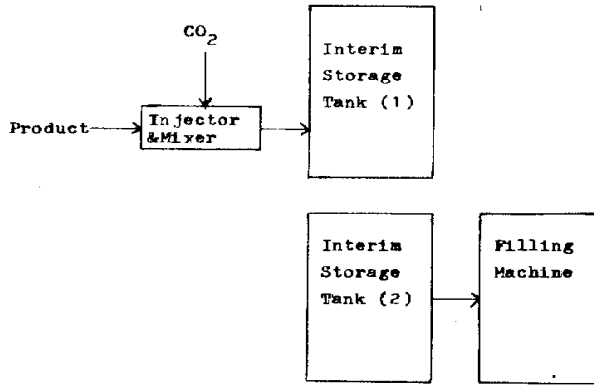
### INLINE CARBON DIOXIDE INJECTION

Carbonation can be carried out by directly metering carbon dioxide into a product line. Traditionally the gas is injected in through a sparge head into a pressurised product line giving a very fine gas dispersion. This is usually followed by some type of an in line mixer. Due to the rapid gas absorption the gas initially in solution is largely uncombined. This results in an extremely unstable solution which is difficult to bottle unless the solution is held for a period of time to stabilise.

The older procedure uses batch storage of the carbonated beverage, in a pressure tank, prior to bottling. As with tank carbonation non carbon dioxide gas can be removed by venting the headspace of the interim storage tank. When this is carried out the product does not require initial deaeration. The stability of the carbon dioxide solution is comparable with that obtained by tank carbonation.



. The basic in line injection system of carbonation.



Inline injection with interim storage system

Continuous in line carbonation with direct injection is also possible. Non carbon dioxide gas must be removed by deaeration before injection to prevent foaming. Also an inline lag phase must also be introduced to help stabilize the carbon dioxide solution. In its simplest form the lag phase consists of a pressure vessel in which the product enters at the bottom and leaves at the top.

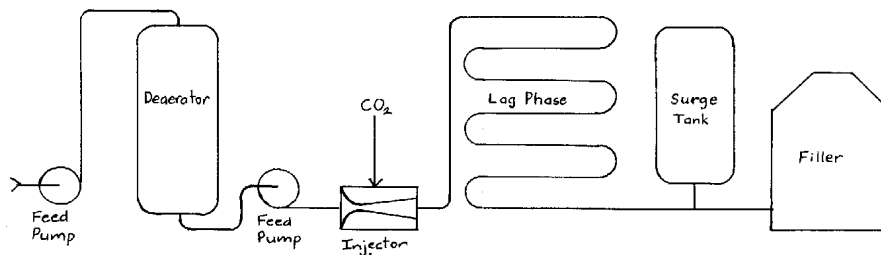
In line carbon dioxide systems lend themselves to accurate carbonation control. New technical developments have enabled in line carbon dioxide injection systems to become more important. Two basic types of carbon dioxide control systems are possible. In the feed back system the finished carbonation is measured and then used to control the carbon dioxide injection rate. In the feed forward system the product flow rate is metered. This measurement is then used to control the amount of carbon dioxide injection. In actual practice a combination of the two systems is often used.

In addition to improved carbonation controls a number of features have been incorporated in the newer in line injection systems. These include;

- (1.) Deaeration of warm product. Better air removal is possible when the product is warm.
- (2.) Cooling of the carbonated product after carbon dioxide injection and during the lag phase. A reduction in the required lag time and improved stabilisation is obtained.
- (3.) Injector design improvements provide better initial gas dispersion. These include venturi nozzle types, which

can handle large gas volumes and improved in line gas dispersion and in line mixers.

- (4.) The use of a supply pump to provide the system over pressure. Better over pressure control on the system helps to prevent destabilisation of the gas solution.
- (5.) The surge or buffer tank is used to compensate for flow variations in the system. Inside the surge tank is a gas cushion which helps to stabilise the system pressure.

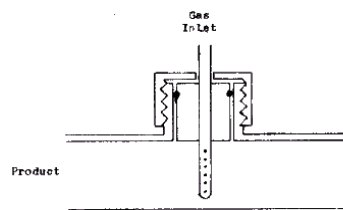


Typical layout of an advanced injection carbonation system

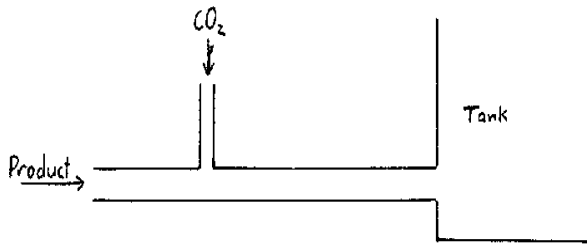
## INLINE SPARGING

Another application of in line injection is sparging to remove gases and volatile materials in the solution. This process is sometimes used to remove non carbon dioxide from solution prior to carbonation.

In the simplest case carbon dioxide is introduced into the product line. The product is usually discharged into a container of some type which is not under pressure. Non-carbon dioxide gas etc is carried out of the solution as the gas separates from the solution. This in the process used by Mojonier in some of their carbonators. ( the "reflux system")

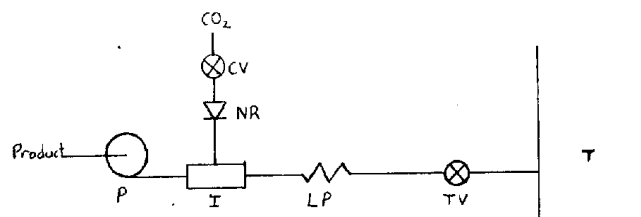


A typical inline gas sparger unit.



Simple carbon dioxide injection.

More efficient use of the sparging gas is obtained if the gas is injected into the product line underpressure and after a short time the pressure is released. Initially the sparging gas dissolves in the liquid underpressure. When the pressure is released the sparging gas precipitates out of solution. Due to the gas precipitation the sparging gas is more efficiently used than with sparging at a single pressure.

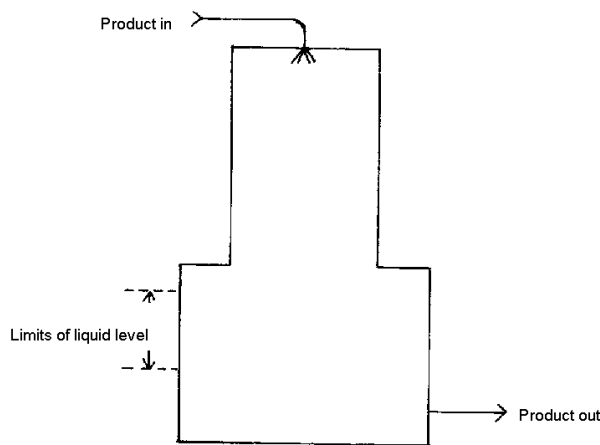


Typical in line sparger layout with back pressure application.

P Product feed, I gas sparger unit, LP length of pipe (this length controls the contact time), TV throttle valve (this valve controls the pressure in the sparging unit) T receiving tank, NR non return valve, CV control valve (to control the carbon dioxide flow rate).

## THE VESSEL CARBONATOR

The basic vessel carbonator consists of a vessel filled with carbon dioxide under pressure. Product is pumped into the top of the vessel where it passes through the carbon dioxide atmosphere and is collected in the bottom of the vessel. Various devices are used to slow down the passage of the product through the vessel atmosphere and also to increase the product-carbon dioxide interfacial area.

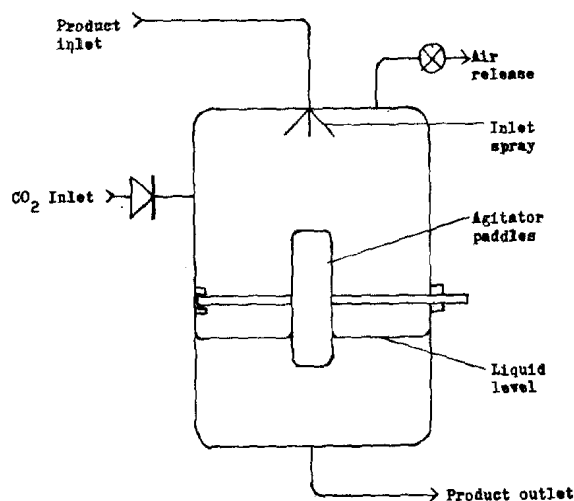


## THE SPRAY CARBONATOR

The simple spray carbonator consists of a vessel into the top of which product is pumped in the form of a fine spray. Gas absorption takes place as the spray falls through the carbonator atmosphere. Usually paddles are provided which provide more mixing of the product and the carbonator atmosphere. This gives more gas absorption. The base of the carbonator vessel provides some hold up time to stabilise the carbon dioxide solution.

In the spray carbonator only a single gas liquid partition occurs. This is important when the removal of non-carbon dioxide gas is considered as it influences the amount of carbon dioxide purge or snift required to remove the non-carbon dioxide gas.





A Typical Spray Carbonator

## THE TOWER CARBONATOR

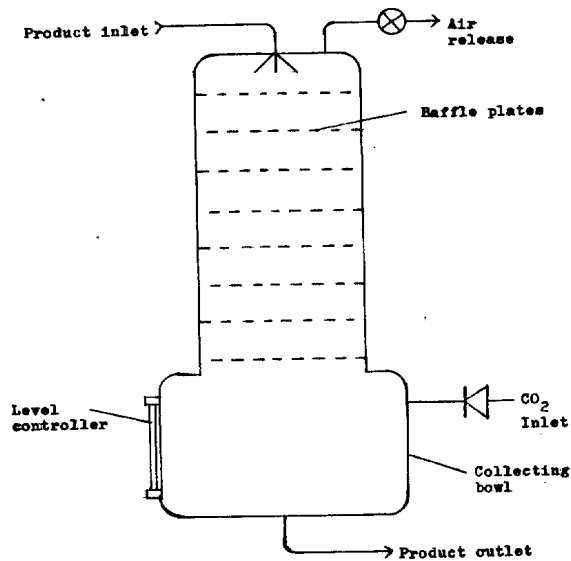
The tower carbonator consists of a baffle plate column, which is filled with carbon dioxide under pressure. Product is sprayed into the top of the column and trickles down over the baffle plates providing a large surface area for gas absorption.

The factors controlling the rate of carbonation are the same as for the vessel carbonator generally. However more effective control of the non carbon dioxide gas is possible than with the spray carbonator. When the gas flow in the column is counter current to the liquid (as when the carbon dioxide is introduced into the bottom of the column and venting occurs from the top of the column) multiple partitioning occurs between the gas and liquid phases. When the column is vented from the top an increasing concentration of non-carbon dioxide gas occurs going up the column. With a multiplate column extremely efficient air removal is possible with a comparatively low carbon dioxide loss as compared to the spray carbonator.

The stability of the carbon dioxide solution produced is largely dependent on the holdup time at the bottom of the carbonator.

If oil is present in the feed liquid it can prevent proper wetting of the baffles.

The available surface area for absorption is then reduced causing a reduction in the level of carbonation.



A Typical tower carbonator

#### FACTORS INFLUENCING CARBONATION IN THE VESSEL CARBONATOR.

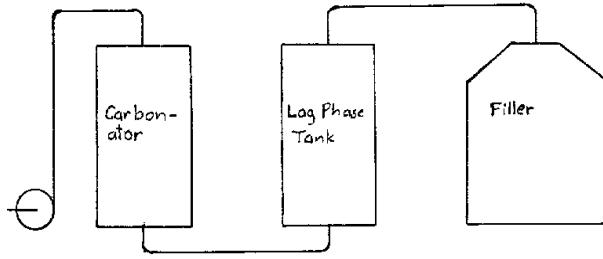
- (1) The rate of gas absorption is proportional to area of the gas liquid interface. A large gas liquid interface is produced in the spray carbonator by the fine spray and also by the action of paddles which are partially submerged in the liquid. In the tower carbonator this effect is obtained by the use of the plates which spread the liquid out into thin layers.
- (2) The rate of carbon dioxide absorption is proportional to the partial pressure across the gas-liquid interface. Any noncarbon dioxide gas reduces the rate of gas absorption by reducing the carbon dioxide partial pressure in the gas phase.
- (3) Temperature effects. Higher temperatures increase the equilibrium partial pressure of carbon dioxide in the liquid phase. The partial pressure difference between the phases is therefore reduced. However this effect may be partially offset by an increase in the diffusion rate of carbon dioxide. Reduction in product viscosity with increasing temperature may have an effect in some cases.

## STABILISATION OF THE CARBON DIOXIDE SOLUTION OBTAINED IN THE VESSEL CARBONATOR.

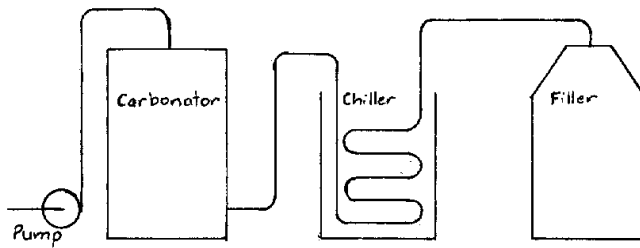
Due to the large surface area to volume relationship in the vessel carbonator rapid gas absorption takes place. However rapid combination of the absorbed gas does not take place. Provision of some means to stabilise the carbon dioxide solution is required. Introduction of a lag phase of none type is the obvious solution. At 3°C a minimum contact time of about ten mins is required for stable bottling. This can vary with the type of product and the plant.

### METHODS of STABILISATION of the SOLUTION

- (1) Most plant manufacturers overcome the stabilisation problem by constructing their carbonators with a large reservoir capacity at the base of the vessel.
- (2) Most of the older carbonators (which have been designed for soda water) do not have enough hold up time to give a stable carbon dioxide solution when used for product mixes . If one of these carbonators is used for product mix an unstable carbon dioxide solution which causes filling problems is produced. The easiest way to correct this is to put a pressure vessel in between the carbonator and filler.
- (3) Another approach is to reduce the lag time required for stabilisation of the carbon dioxide solution. An increase in the rate of solution and the rate of combination may be achieved by carbonating the product warm and chilling prior to bottling. This provides an effective means of reducing the lag time and eliminating the need for an extra pressure vessel.

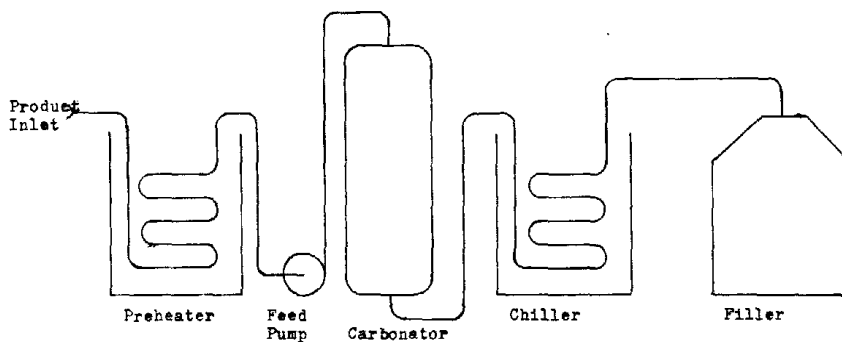


Arrangement for carbonating and packaging product with an intermediate lag phase tank.



Arrangement for carbonating and packaging product with an intermediate lag phase tank.

In one factory bottling carbonated fruit juice the above arrangement worked well during the summer months. During the first winter after the installation of the plant It was found that the ambient temperatures were too low for adequate stabilisation. This was corrected by the Installation of a precarbonation warmer and the foaming during filling stopped.



A full stabilization system using temperature manipulation.

An important point to note is that the heat exchangers must be capable of maintaining accurate temperature control or variations in carbonation will

occur. In the above plant the heat exchangers were tank coils in thermostatically controlled tanks.

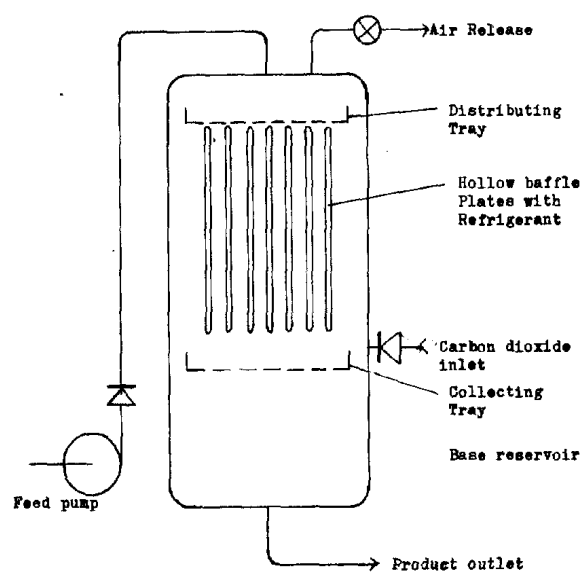
## THE CARBO-COOLER

The carbo-cooler is basically a type of tower carbonator in which both carbonation and cooling take place in the carbonator. Product enters the vessel by a distributor and flows down over a series of hollow baffle plates. Freon or ammonia is passed through the plates. Both cooling and gas absorption takes place as the liquid flows in a thin layer over the surface of the plates. Finally the product is collected in the bottom of the vessel where it is drawn off.

The carbocooler tends to give better combination of the carbon dioxide than the simple vessel carbonator. A large portion of the gas absorption and combination occurs at a higher temperature.

Additional stabilisation occurs in the bottom of the carbocooler vessel. Overall the degree of stabilisation is less than that produced by tank carbonation but more than the simple vessel carbonator.

The presence of baffles also tends to encourage a counter current gas flow. This helps to give multiple partitioning between the phases and consequently gives good air removal from the product.



A classical "Mojonier" type Carbocooler

## GAS EXCHANGE IN THE VESSEL CARBONATOR

When a liquid containing non carbon dioxide gas is introduced into the carbonator vessel not only does carbon dioxide pass into solution but non carbon dioxide passes out of solution and into the gas phase of the carbonator vessel The result is that non-carbon dioxide gas tends to accumulate in the vessel atmosphere.

Gas passes out of the liquid phase into the gas phase according to the equation.

$$t = k \log (P_L - P_2)/(P_L - P_1)$$

Where  $t$  = Time

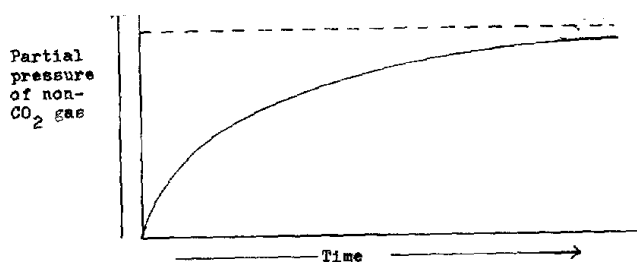
$P_L$  = Partial pressure of non-carbon dioxide in the feed liquid

$P_1$  = Initial partial pressure of non-carbon dioxide in the gas phase

$P_2$  = Partial pressure of non-carbon dioxide gas in the gas phase at time  $t$

As under normal circumstances the initial non-carbon dioxide in the gas phase is negligible.

$$t = k \log (P_L - P_2)/(P_L)$$



Accumulation of air in the carbonator atmosphere.

Accumulation of non-carbon dioxide in the carbonator atmosphere results in a reduction in the concentration of carbon dioxide with a consequent reduction in the degree of carbonation in the finished product. Dissolved non-carbon dioxide gas in the finished product also increases bottling problems. Some type of vent system must be incorporated in the carbonator vessel if the feed

liquid is not subject to vacuum deaeration.

The carbonator air release system usually operates automatically releasing a small amount of the carbonator vessel atmosphere. This maintains the non-carbon dioxide gas at a low constant level.

Points to note about carbonator vessel venting.

- (1) The non-carbon dioxide gas is only lowered in concentration and not completely removed.
- (2) The amount of non-carbon dioxide gas removed depends both on the type of carbonator and the amount of the vessel atmosphere removed. In particular the number of gas-liquid partitions taking places in the carbonator are important.
- (3) Vessel venting will not cope with large amounts of non-carbon dioxide gas in the feed liquid.

## THE CARBONATOR EFFICIENCY

The gas absorption in the carbonator vessel during its operation is largely controlled by the rate of passage of gas through the gas-liquid interface. This can be represented by the equation.

$$\Delta t = k_2 (\log P_2 - \log P_1) \quad (i)$$

Where t = The elapsed time

$P_2$  = Final pressure differential

$P_1$  = Initial pressure differential

$k_2$  = Constant

$$\Delta t = k_2 \log (P_g - P_2) / (P_g - P_1) \quad (ii)$$

$P_g$  = Partial pressure of non-carbon dioxide in the feed liquid

$P_1$  = Initial partial pressure of non-carbon dioxide in the liquid phase

$P_2$  = Final pressure of non-carbon dioxide gas in the liquid phase.

In any particular carbonator  $t$  and  $K_2$  are constant also  $P_1$  is normally zero.

From this 
$$K_c = (P_g - P_2) / P_g \quad (iii)$$

Where  $k_c$  is the normal operating constant for any particular carbonator.

The pressures are absolute pressures and not gauge pressures.

Also  $P_{ii}$  is derived from the determined  $CO_2$  of the carbonator output and is corrected to the average carbonator temperature.

So that

$$k_c = (P_a - P_v) / (P_a + 100)$$

Where  $P_a$  = The carbonator vessel pressure gauge reading in  
KPa

$P_v$  = The pressure of  $CO_2$  in the product corrected to  
the average carbonator temperature.

Note; This assumes the noncarbon dioxide gas in the vessel atmosphere is close to zero.

From the equation it can be seen that the lower  $k_c$  the better the gas absorption is. The carbonator constant can be easily calculated from the normal operation and any increase from the normal value would tend to indicate a malfunction. Changes in dissolved solids in the product being processed cause apparent changes in the carbonator efficiency. Increased sugar levels typically decrease the carbonator efficiency. see Chap 1

## CHANGE of CARBONATOR OUTPUT CARBONATION LEVEL

From Eqn (iii) 
$$k_c = (P_g - P_1) / P_g$$

As  $k$  is constant

$$(P_{g1} - P_{L1}) / P_{g1} = (P_{g2} - P_{L2}) / P_{g2}$$

$$P_{L1}/P_{g1} = P_{L2}/P_{g2}$$

At constant temperature and sugar levels as an approximation the amount of gas in solution is proportional to the partial pressure of  $CO_2$ .

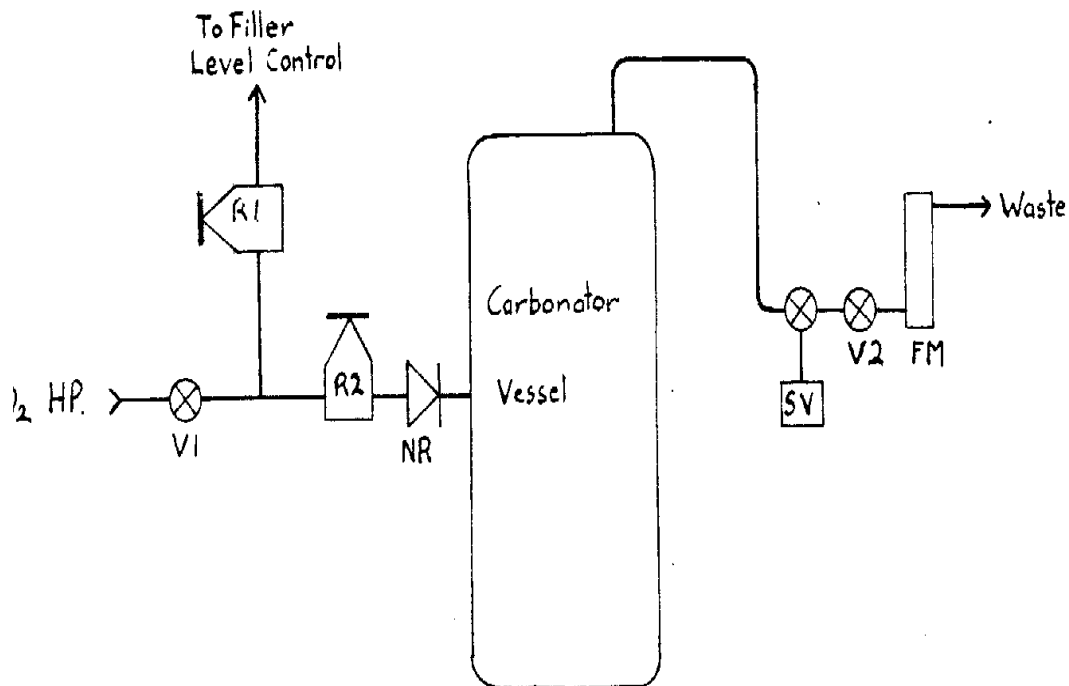


$$V_{L1}/V_{L2} = P_{g1}/P_{g2}$$

$V_L$  = Carbonation volumes

$P_g$  = Carbonator pressure absolute

(Gauge pressure plus one atmosphere pressure)



Gas Piping Layout for a typical vessel carbonator.

(V1) Inlet gas stop valve

(V2) Vessel purge flow control needle valve.

(R1) Filler bowl level control supply regulator

(R2) Carbonator vessel pressure control regulator.

(FM) Vessel purge flowmeter.

(SV) Vessel purge solenoid operating valve (linked to vessel supply pump control)

(NR) Vessel inlet nonreturn valve

## Chapter (8)

### FILLING MACHINES AND FILLING CYCLES FOR CARBONATED BEVERAGES

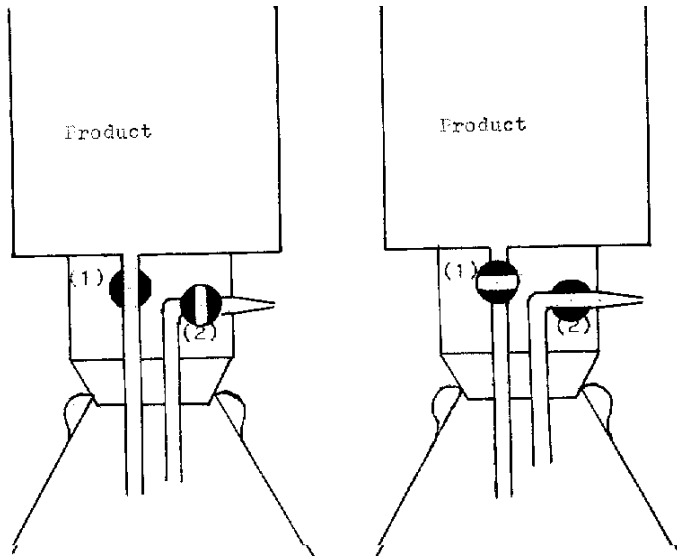
Due to the presence of carbon dioxide in a supersaturated solution, carbonated beverages must be filled under pressure. If they are not filled in pressure filling machines foaming and loss of both product and carbonation occurs.

During the filling operation the bottle must be filled to the required level without significant loss of product secondly the filling the pressure must be released in order that the bottle may be sealed. Two basic types of filling machine are used, the "Snift" filler and the full counter pressure machine. The snift type of machine is seldom used except on small plants now days. It has been largely superseded by the counter pressure machine that gives quieter filling and higher filler operating speeds.

#### THE SNIFT FILLING MACHINE

The filling head has two valves the product valve and the pressure release or snift valve. The original filling machines had only one valve, the product valve. The bottles were manually snifted by the operator releasing the neck of the bottle from the filling machine rubber very slightly. This allowed some of the gas to escape so that filling could continue. The snift may be either intermittent or continuous.

In both cases the snift was restricted so that the pressure drop in the bottle was not violent enough to cause serious foaming.

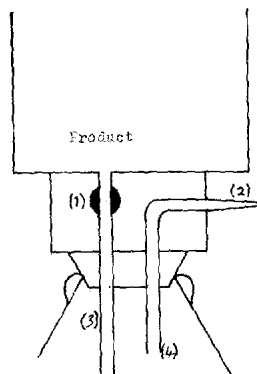


A Filling valve (1) open  
 Snift valve (2) closed  
 Product is forced into  
 The bottle under  
 Pressure.

B Filling valve (1) closed  
 Snift valve (2) open  
 pressure in the bottle is  
 Pressure in the bottle is  
 Released.

### VALVE SEQUENCE FOR THE INTERMITTENT SNIFT FILLING MACHINE

This sequence is repeated two or three times until the bottle is filled. When the bottle is filled position B is held to depressurise the bottle and the bottle is removed from the machine.



### THE CONTINUOUS SNIFT MACHINE

I

The product valve (1) is opened and the bottle fills until the bottom of the air release tube is submerged. Excess product then flows out of the snift restrictor

(2) The flow rate of product into the bottle is controlled by the release of gas through the snift orifice. When the bottle is filled the product valve is closed and the excess pressure is allowed to vent from the snift restrictor before the bottle is removed from the machine.

There is a variation on the continuous snift machine where the bottle is prepressurised with gas prior to opening the product valve. The prepressurisation version gives a performance similar to the full counter pressure machine but with a much higher gas wastage. The author uses a small pressure tank followed by a head prepressurization filler for the preparation of samples. The system provides accurate carbonation control and stable filling.

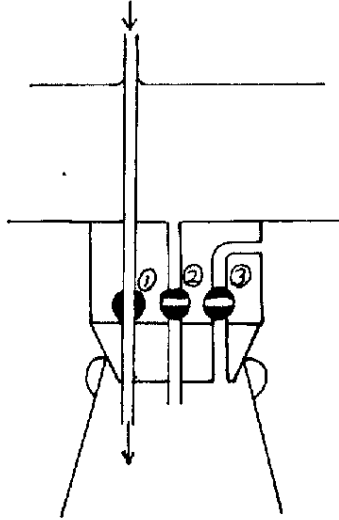
## THE FULL COUNTERPRESSURE FILLING MACHINE.

Most modern filling machines are of the full counterpressure type. The filling head has three separate valves. The gas valve or counterpressure valve (9), the filling or product valve (8), and the snift or pressure release valve (10). In operation the counter pressure valve is opened first. This connects the filler bowl headspace to the bottle head space and the bottle is pressurised by filler headspace gas. When the bottle is pressurised the product valve opens and product flows into the bottle until the end of the air tube is submerged when the bottle stops filling. Finally the filling valve and air valve are both closed. The snift valve is then opened to release pressure from the bottle prior to its removal from the filler. The speed of the pressure release is usually controlled by an orifice or some type of restriction to prevent violent pressure release and foaming.

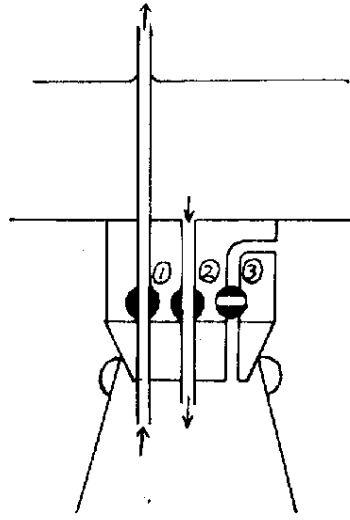
The older type of counter pressure filling machines for example the CEM 28 and 48 used a plate valve which included all the valve functions. The counterpressure and gas valve functions on this type of valve have separate channels. On the newer type of valve the counterpressure function and the gas valve function are combined on the one valve by timing of its opening. Eg. The Meyer valve the CEM Uniblend valve etc.

The modern counterpressure filler is capable of a fill cycle time of approximately six seconds for a 300 ml bottle.

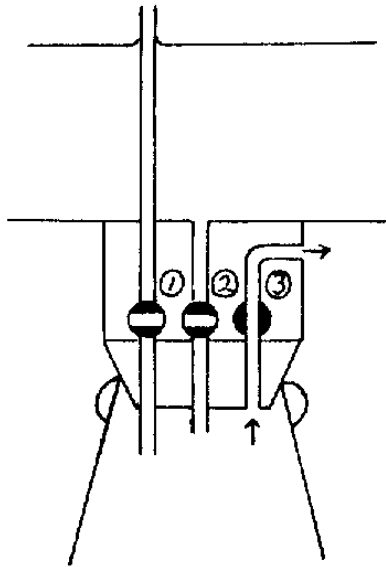
### FILLING SEQUENCE FOR A STANDARD COUNTER PRESSURE FILLER



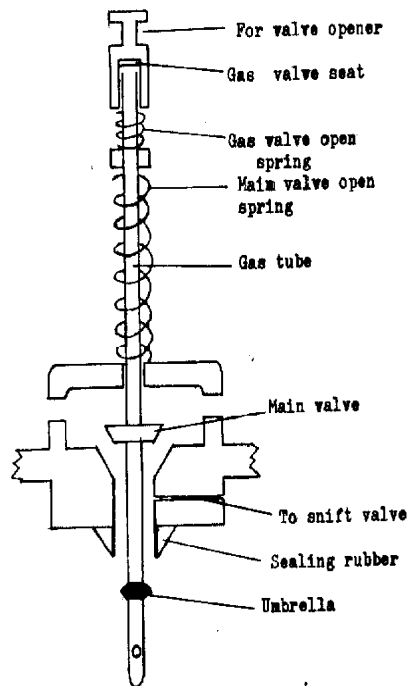
The gas valve is open  
The other valves are closed  
Bottle is being pressurized



The gas and product valves  
are open. The snift valve  
is shut. The bottle is being  
Filled with product



The Snift valve is open.  
The other valves are closed  
The pressure in the bottle is  
being released



A TYPICAL MODERN FILLING VALVE showing arrangement of components and valve opening springs.

## OPERATION SEQUENCE FOR THE MODERN FILLING VALVE.

Most modern filling valves are variations on the same theme eg. the new "Meyer", the CEM "Uniblend" etc. Most have a concentric arrangement as shown, in the diagram.

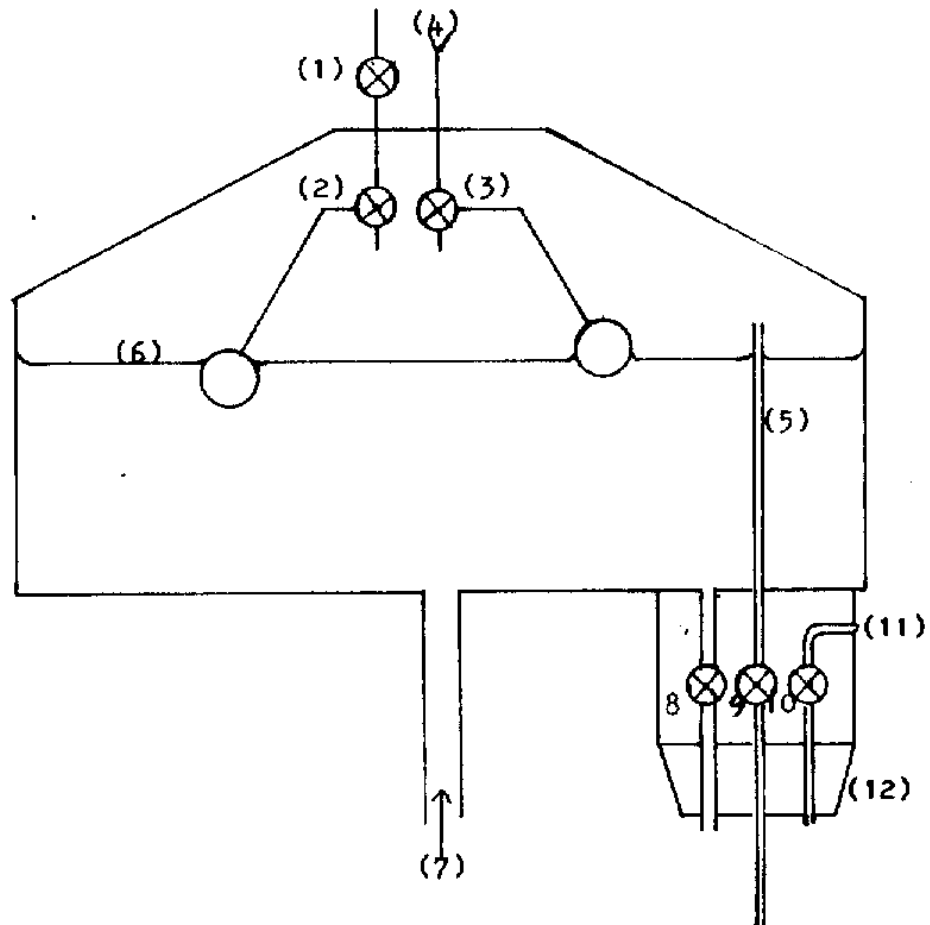
### SEQUENCE

- (1) Valve Closed; The gas valve and the main valve are held closed by the filler bowl pressure against the pressure of their opening springs.
- (2) Pressurisation of the bottle; The valve opener lifts the gas valve off its seat. Filler headspace gas can flow into the bottle.
- (3) When the pressure in the bottle is nearly equal to that in the filler bowl, the main valve is lifted off its seat by the opening spring. Product starts to flow into the bottle.
- (3a) Leaking bottle on the filling head. Pressure in the bottle is insufficient to allow the main valve to open.
- (4) The valve opener shifts to the mid position.

The bottle keeps filling as the gas valve is held open by its opening spring.

- (4a) Valve opener shifts to the mid position. The gas valve is not held open any more and is forced shut by the pressure in the filler bowl.
- (5) When the vent holes in the gas tube are covered the gas tube floods and the bottle stops filling.
- (6) The valve opener is pushed down closing both the gas valve and the main valve.
- (7) The bottle is snifted by opening the snift valve and the bottle is removed from the filler.

Some of the newer bottling machines have introduced a gas purge into the filling cycle. The bottle is flushed with carbon dioxide prior to the Initial bottle pressurisation. The primary purpose of this is to reduce air pickup during bottling. A similar system has been used for many years in the wine industry for bottle sterilisation. In this case sulphur dioxide gas with gas flushing is used for bottle sterilisation.



Diagrammatic arrangement of a full counterpressure filler, one filling valve only shown.

- (1) Vessel air release outlet valve.
- (2) Vessel air release float valve.
- (3) Vessel counter pressure gas inlet float valve.
- (4) Counter pressure gas inlet.
- (5) Filler valve air tube
- (6) Liquid level in filler bowl.
- (7) Product inlet to filler bowl.
- (8) Filling valve; product valve.
- (9) Filling valve; gas valve.
- (10) Filling valve; snift valve.
- (11) Snift valve outlet orifice.
- (12) Filling valve sealing rubber.



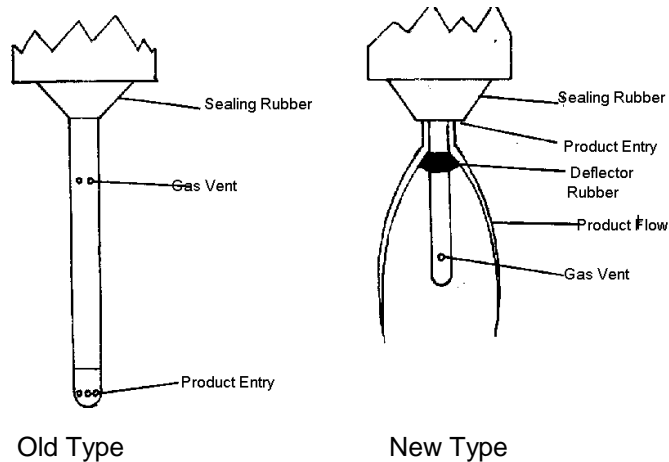
The liquid level in the filler bowl of the counter pressure filler is critical. If the level is too low there may be insufficient head at high filling speeds with the result that low filling may occur. If the bowl level is too high flooding of the air tubes occurs and very low filling or no filling occurs.

Control of the liquid level is carried out by two float controlled valves. The vessel (bowl) air release valve (a) opens if the liquid level falls too low and releases filler bowl headspace gas. During filling air and gas displaced from the bottles accumulates in the filler bowl and must be released regularly. The counter pressure gas inlet valve opens if the liquid level is too high. The counter pressure gas supply is connected to a carbon dioxide gas supply which is at higher pressure than the filler contents. Occasionally compressed air is used for the counter pressure supply this is undesirable due to the problems caused by air being dissolved in the product. This occurs if the filler is stopped for a period of time. The liquid in the filler bowl then has time to absorb air from the headspace gas.

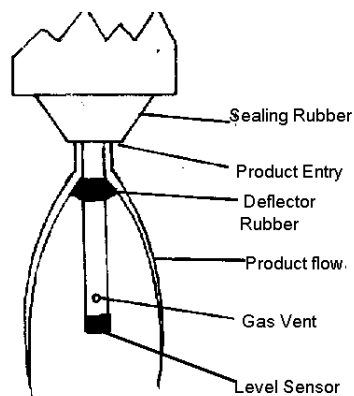
In the counter pressure filling machine filling is stopped by flooding of the gas tube. Liquid coming into the bottle must be kept clear of the vent holes on the gas tubes until the bottle is filled.

In the older type the product entry is below the vent holes. A variation on the old type of filling tube is the long filling tube which is used on many can filling machines. The product is discharged near the bottom of the container by a filling tube which extends well down into the container. This has the advantage that air entrainment is reduced.

On the newer type the product entry is above the vent holes. The product is prevented from coming into contact with the vent holes by a deflector rubber (umbrella) on the gas tube above the vent holes.



Comparison of filling tube arrangements on older and new type fillers.



Newer Type variation with electronic level control

## CLEARING OF THE PRODUCT

During the time that the product is entering the bottle many small gas bubbles are entrained in the product. These bubbles if not allowed to settle out will cause violent foaming of the product when the bottle is released from the filler. Clearing or settling out of these gas bubbles takes place in two phases. First as soon as the gas vents on the filler vent tube are submerged product stops flowing into the bottle. The turbulence due to filling stops and the gas bubbles start to settle out.

The most important clearing takes place during sniffting of the bottle. As the pressure is slowly released during sniffting the small gas bubbles rapidly grow due to the reduction in pressure. These enlarged gas bubbles settle out very

rapidly and the product quickly clears.

If the pressure release is too rapid the gas bubbles grow too much before they have settled and product is blown out of the bottle.

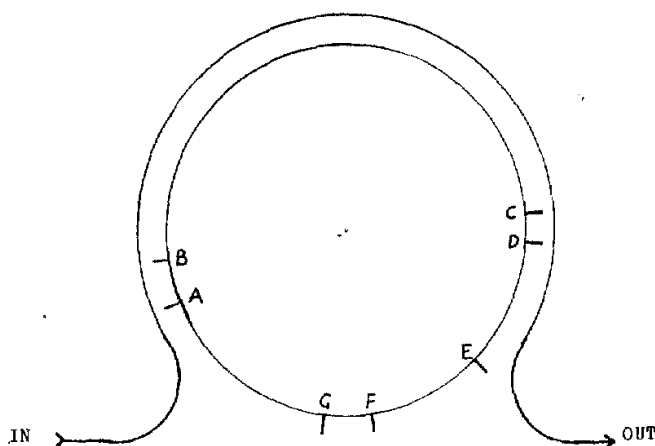
With insufficient sniffing the product may not clear properly and gushing occurs when the bottle is released from the sealing rubber.

## THE GAS TUBE PURGE.

To pressurise the bottle initially on a counter pressure filler the gas valve is opened. A powerful jet of gas enters the bottle charging it with headspace gas. If there is any product in the air tube it is ejected violently striking the bottle wall. Due to the impact a film of product containing fine gas bubbles is deposited on the bottle wall. These fine gas bubbles can then initiate fobbing in the product at pressure release.

Some machines have an optional air tube purge. This consists of a valve opener and closer close together at a point on the machine prior to the bottle entry. In operation the gas valve briefly opens and any product left in the tube is blown out. The system appears to be most useful at high carbonation levels and high filling pressures.

A recent innovation is the electronic level control. This is operated by a sensor on the air tube below the vent hole. As the air tube does not become flooded there is little product to be ejected and the problem is minimized.



Typical Positioning of Valve Functions on a Modern Counter pressure Filler

(A) Valve opener. (B) Neutral position cam. (C) Valve closer. (D) to (E) Snift operating bar. (G) to (F) Gas tube purge.

## PRESSURE DROPS IN THE CARBON DIOXIDE LINE.

Flow in a pipe line will only occur in response to a pressure drop. If the pressure at the high pressure end of a pipe is kept constant the pressure at the down stream end is dependent on the flow rate of fluid through the pipe. With high flow rates the pressure at the down stream end is reduced. This is the situation that occurs with the typical carbonation system.

The vessel carbonator is supplied with carbon dioxide at constant pressure by a regulator. The draw off rate for carbon dioxide is not constant with the result that varying pressure drops occur in the gas line between the regulator and the carbonator vessel, Variations in the carbonator vessel pressure occur. As the amount of carbonation is dependent on the pressure of carbon dioxide in the vessel variations in carbonation occur.

In order to reduce these variations in the amount of carbonation the supply line between the gas regulator and the carbonator vessel should be as short as possible and also have the largest bore practical. By these means the vessel pressure variations can be kept low enough that they are not objectionable.

The problems of pressure drop in the line between the regulator and carbonator can be eliminated if a compensated regulator is used. With the compensated regulator a sensing line is taken from the carbonator vessel to the regulator. This line is used for control only and another line is used to supply the gas. Today nearly all the large vessel carbonation systems use a flow compensated carbon dioxide supply system.

Electronic pressure sensors can also be used to control the carbon dioxide inlet valve. Provided that the sensor is connected directly to the carbonator vessel this provides flow compensation.

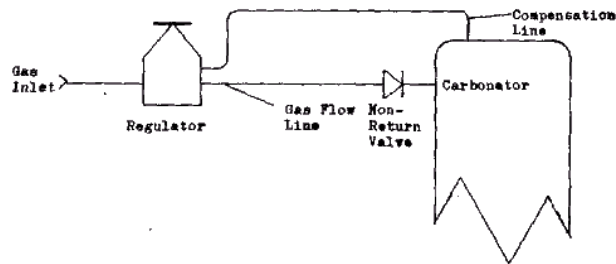
The flow rate of gas that a regulator can supply is dependent on the size of the orifice in the valve of the regulator and on the pressure drop across the regulator.

If the pressure on the high pressure side of the regulator is too low (the pressure drop across the regulator is too low) variations in vessel pressure and carbonation can occur. Typical causes of this problem are;

Low pressure in the gas storage system,

Blockages in the supply lines,

Valves not fully open, etc.



Piping diagram for a flow compensated gas supply system

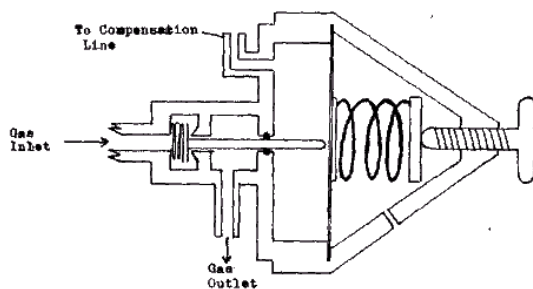


Diagram for a simple flow compensated regulator

## PRESSURE DROPS in the CARBONATED PRODUCT LINE

For flow to occur in a piping system a pressure drop must occur. The greater the restriction the greater the pressure drop required to produce a given flow rate.

In addition flow past partial obstructions may cause a localised pressure drop by the Bernoulli effect.

In a carbonated beverage line if the applied pressure at any point is less than the equilibrium pressure of the carbon dioxide in solution bubble nucleation and growth tends to occur, (see c2;6).

Even if the bubbles are redissolved the distribution of bound to unbound carbon dioxide in solution is changed. This will contribute to a less stable carbon dioxide solution which has a greater tendency to foam during filling.

As the degree of carbonation in the carbonator is controlled by the rate of carbon dioxide absorption the solution never reaches saturation.

The result of this is that the product leaving the carbonator is under a greater applied pressure than that due to the partial pressure of the carbon dioxide in solution. Generally unless substantial restrictions occur in the product line the applied pressure does not drop below the equilibrium partial pressure of the carbon dioxide in solution. (see 02;4)

Some carbonation systems ( eg the Mojonnier "carbotrol" ) incorporate a precarbonation carbon dioxide injection into the product line. These systems enable carbonation to be carried out at a lower vessel pressure. It is possible with these systems for the beverage carbon dioxide pressure to approach the carbonator vessel pressure. In this case if care is not taken the system overpressure may be reduced to the extent that foaming problems occur during filling.

In practice an overpressure of about seven psi. (0.5 bar), above the

equilibrium pressure of carbon dioxide in solution, through the system will usually prevent pressure drop induced foaming during filling.

Attention to pipe sizing will help to prevent pressure drop problems. For carbonated Beverage lines between the carbonator and filler the American Bottlers of Carbonated Beverages in 1959 suggested the following pipe sizes; 1 inch for a maximum of 30 bottles per min. (equivalent to 10L /min)

1½ inch for a maximum of 100 bottles per min. (equivalent to 30L /min)

2 inch for over 100 bottles per mm. . (equivalent to 10L /min)

These figures were for soda water and not for product mix.

For the modern practice of carbonating product mix larger pipes should possibly be used.

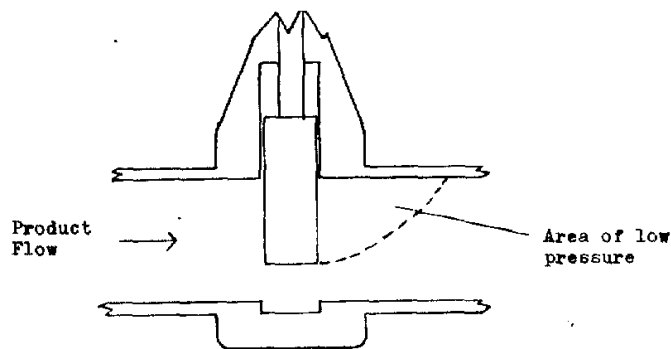


Diagram showing the region of low pressure on a partially closed gate valve.

## MOISTURE DAMAGE to GLASS BOTTLES

Water attacks new glass surfaces dissolving out alkalis and leaving behind a relatively inert layer of silica. Normally this has no significant effect and only conditions the glass surface.

When only small amounts of moisture are present, as for example condensation an alkali solution is produced. On subsequent evaporation this solution becomes concentrated and will attack the glass causing microscopic pitting. If these pits are not completely wetted out and air displaced bubble growth and foaming may occur after pressure release on the filler, See chap 3

Attacked glass typically has dendritic crystals of sodium carbonate on the surface inside the bottle.

On the bottling machine "gushing" often occurs without a visible trail of bubbles rising from a point. When a trail of bubbles does occur it rapidly dies out due to the loss of non carbon dioxide gas from the pit. Used bottles are not very susceptible to this type of damage. The "conditioning" of the glass surface results in an alkali deficient barrier layer which retards the further solution of alkali from the glass.

Obviously this problem can be prevented by not storing new bottles for long periods of time under moist conditions.

Sodium carbonate deposits caused by moisture damage and deposits of stearate (used to prevent scuffing) inside the bottle may be confused. Also during filling stearate may cause foaming similar to pitting. This is because the stearate tends to produce a hydrophobic surface, which inhibits adequate wetting out of the bottle surface. In order to distinguish between the two deposits the following test is useful;

(1) Cut the bottle open.

(2) Add a small drop of 5N hydrochloric acid. Sodium carbonate gives a momentary effervescence and dissolves completely. Stearate may or may not effervesce but gives a thin white film which floats on the surface of the acid.



## SPECIAL FACTORS ASSOCIATED WITH THE USE OF PET. BOTTLES

Today many carbonated beverages are packed in plastic bottles. These are usually PET. (polyethylene terephthalate) although a number of laminates have been proposed. These laminates include PET/EVOH/PET. EVOH is an ethylene vinyl alcohol copolymer. The properties which make PET bottles different to both metal cans and glass bottles are chemical permeability, physical strength (compressive), and surface chemistry.

Glass bottles and metal cans are impermeable to all chemicals, which do not physically destroy their structure. Many plastics including PET have a very marked chemical permeability. This chemical permeability is usually due to a specific phenomenon, that of "activated diffusion". " Activated diffusion" has a series of steps. Initially the diffusing component dissolves in the bottle wall material, the dissolved component then migrates through the bottle wall where dissolution occurs.

Permeability is dependent on the type of plastic and also the degree of crystallinity in a given type of plastic. Crystalline plastics are more impermeable than amorphous plastics. During the blowing of PET bottles molecular alignment occurs resulting in pseudocrystallinity. The actual permeability factor for PET bottles is between that of amorphous material and crystalline material. The laminates have a lower permeability than PET. PET/EVOH/PET is reputed to have a gas permeability one third that of PET. Due to acceptable carbon dioxide losses the shelf life of a 1.25 L soft drink could be as low as twelve weeks at 25° C.

Acceptable carbon dioxide losses mean that the shelf life for smaller bottles would be even shorter.

Carbon dioxide losses from PET bottles can be partially compensated for by a higher initial carbonation. Many bottlers set a carbonation standard for PET bottles 0.3 volumes above the standard for a similar product packed in glass. Against this is the permeability of PET to oxygen. Oxygen can permeate the

bottle wall and cause flavour damage to the product inside. Citrus products are particularly susceptible to oxidation flavour changes.

#### GAS PERMEABILITY OF PET.

Gas	Permeability	
Crystalline PET	CO <sub>2</sub>	0.17
	O <sub>2</sub>	0.035
Amorphous PET	CO <sub>2</sub>	0.3
	O <sub>2</sub>	0.059

Permeability factor units

$$= [\text{cm}^3 (\text{stp})]\text{cm.cm}^2 \text{sec}^{-1} (\text{cm. Hg})^{-1} \times 10^{-11}$$

Oxygen Transfer Coeff of 1.5 L Soft Drink Bottles

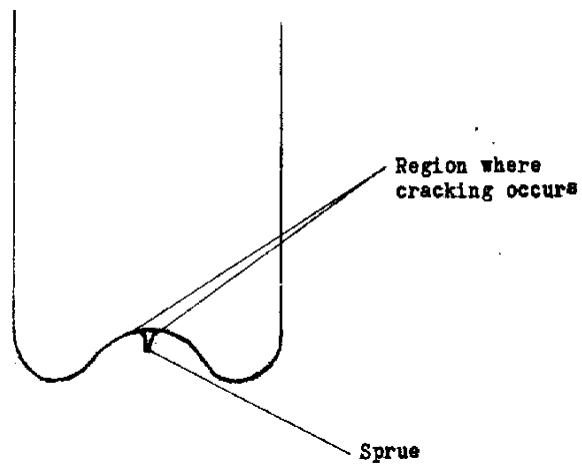
Plain PET vs Laminates

PET	0.25 mls/bottle.day @ 25°C
PET/HXDX6/PET	0.06
PET/EVOH/PET	0.08

While the surface of PET is not as polar as glass the degree of wetting is sufficient and bubble nucleation is not a problem. Occasionally bottles are found where bubble nucleation occurs over large areas of the bottle surface when pressure is released after filling. PET is readily wet by oil. If the air used for bottle blowing is not completely oil free occasional droplets of oil may enter the bottles. These droplets on contacting the bottle surface rapidly spread making the PET very hydrophobic. This hydrophobic surface causes bubble nucleation on pressure release.

The effect is very similar to that caused by the polyethylene coating sometimes applied to one way bottles.

PET bottles are susceptible to crack formation around the sprue. (injection point). These cracks can trap air, which in turn can nucleate bubble formation and fobbing after filling.



Region in PET Bottles where Cracking and Gas Nucleation tend to occur

## CARBON DIOXIDE USAGE.

During carbonation and filling: carbon dioxide in excess of that dissolved in the product is used. The bottle headspace, the carbonator vessel purge (air release) and filler bowl counter pressure supply in addition all contribute to the total carbon dioxide usage.

All these usages are usually proportional to the amount of product bottled. A theoretical usage may be calculated and compared to the actual usage. The deviances found may then be used for trouble shooting the plant

Add CO<sub>2</sub> delivery problem at PNG

### CALCULATION

One gram of carbon dioxide has a volume of 509 mls under standard conditions.

The number of volumes of carbon dioxide under standard conditions which dissolve in one volume of product is the "carbonation volumes" One "volume" is equal to a carbon dioxide concentration of approximately 1.96 grams of carbon dioxide per litre of product.

Dissolved CO<sub>2</sub> (gms) = Product volume (mls) x "volumes" x 1/509

The approximate amount of carbon dioxide in the bottle headspace at room temperature (15°C).

$$\text{Wt CO}_2 = \text{Vol of headspace} \times \text{"volumes"} \times 1/509$$

The carbonator vessel purge is usually linked to the carbonator feed pump so that the amount of vessel purge is proportional to the volume of product carbonated. Carbonator manufacturers usually provide data on the feed pump capacities and many carbonators have in addition a flow gauge on the vessel purge. This gauge is normally used to adjust the setting on the vessel purge, and can be used to calculate the gas loss. The purged gas is mostly

carbon dioxide and normally contains only a small percentage of air. Provided that the filler bowl level controls are working correctly the gas usage through the counter pressure line is minimal. Usually the usage calculation only includes the dissolved carbon dioxide, the bottle headspace gas and the carbonator vessel purge.

#### PARTIAL LIST of PROBLEMS CAUSING HIGH CARBON DIOXIDE USAGE.

This applies primarily to a vessel carbonator and counter pressure filler system.

- (1) Actual delivery of carbon dioxide not equal to invoiced delivery.
- (2) Leaking gas lines or carbonator vessel.
- (3) Supply regulator main valve leaking causing overpressure in the carbonator vessel. This in turn causes the overpressure stabilising valve (where present) to release gas.
- (4) Precarbonation carbon dioxide injection (eg Mojonnier carbotrol) too high causing vessel overpressure and gas release by the stabilising valve.
- (5) Pressure stabilising valve leaking.
- (6) Filler bowl level control valves out of adjustment or leaking. Both gas inlet and outlet valves may cause the problem.
- (7) Leaking bottle sealing rubber. (may cause filling problems as well.)
- (8) Excessive breakages on the filler caused by a faulty batch of bottles.
- (9) Setting of carbonator vessel purge too high.

Monitoring carbon dioxide usages and establishing the normal usage variance is an important tool for trouble shooting the bottling plant operation.

Any variance outside the established norm should be investigated.

For example;

On one occasion the author was confronted by the company accountant about a sudden increase in carbon dioxide usage in excess the normal variance. Nothing could be found in the plant so further investigation was carried out.

The carbon dioxide purchased from the supplier was transported as liquid in a tanker truck. The truck was weighed prior to delivery and after delivery to calculate the amount of carbon dioxide delivered. The weigh bridge close to the bottling plant had broken down and the tanker operator was using another weigh bridge several miles away. The supposed increase in carbon dioxide usage was actually the weight of fuel used by the tanker truck between the two weighings.

## LEAK TESTING THE CARBONATION SYSTEM.

Air can be drawn into the carbonation system against a high pressure differential by the venturi effect. It is important that any leaks in the high pressure parts of the carbonation system are eliminated in addition to those in the suction side of the carbonator pump.

There are a number of methods of finding air leaks the most effective of these is the freon tracer method, if the materials and equipment are available.

### THE FREON TRACER PROCEDURE.

The carbonator and plant are drained of water and are closed off. The system is then pressurised first with freon 12 to few psi (approx 0.1 – 0.2 bar) gauge pressure then with carbon dioxide to the working pressure. Leaks may be detected with a refrigerant leak detecting torch. In particular all pipe joints and welds should be carefully checked. After the test all the pressure should be bled off and the system thoroughly purged before being put back into service. Freon 12 is an ozone depleting substance its use for this type of test is now no longer appropriate. Ethyl chloride does not have the same restriction and could be used as a tracer addition to the gas used to pressurise the system.

### THE DYE LEAK TESTING PROCEDURE.

The carbonator discharge is disconnected and a white bucket is placed under the carbonator outlet. The carbon dioxide inlet valve is opened slightly and water is pumped through the carbonator by the carbonator pump. A strong solution of dye (containing no undissolved particles) is painted around each joint in turn. Suitable dyes are Fluorescein or Rhodamine WT. A few minutes must be allowed after each dye application to allow the dye from any leak to appear in the bucket. The author prefers fluorescein as very low levels can be detected in the dark using a UV light. This procedure is particularly useful for detecting feed pump problems (usually a leaking pump gland).

## THE SOAP LEAK TESTING PROCEDURE.

The old method of leak testing with soap solution is still useful. The system is drained of water and pressurised with carbon dioxide. A soap or detergent solution is painted over all suspect areas. Leaks large enough to cause trouble usually show up immediately.

The best test solution is a 1% solution of soap or detergent containing 10% glycerine the rest of the solution being water. Traditionally a 1% solution of sodium bromo-oleate was used. An effective more modern agent is a dilute solution of sodium laureth sulphate, the most common readily available source is womens shampoo.



## FLAVOUR REMOVAL FROM COMMERCIAL CARBON DIOXIDE.

Carbon dioxide used for beverage carbonation must be free from off tastes and aromas. Many impurities can not be readily separated from carbon dioxide by the normal liquefaction process.

For example carbon dioxide prepared by burning oil may contain traces of sulphur dioxide, nitric oxides, and unburned hydrocarbons. On the other hand carbon dioxide prepared as a fermentation by-product may contain hydrogen sulphide and a variety of volatile organic aroma compounds. Traditionally carbon dioxide is cleaned up using a scrubber containing a solution of sodium carbonate and potassium permanganate. The colour of the solution provides a good indication of its condition. An exhausted scrubber solution contains suspended brown manganese dioxide. If the manganese dioxide is allowed to settle an active solution still contains purple permanganate. If no purple permanganate is present the scrubber solution is exhausted and must be replaced. This scrubber mix can remove most of the likely impurities. Carbon dioxide is usually separated from the cleaned gas stream by an absorption-desorption process using an organic amine (usually monoethanolamine ).

Finally as an additional safe guard the carbon dioxide is usually passed through an activated carbon filter followed by a particle filter of some type to prevent particulate matter being carried through and fouling the gas regulation system.

The gas manufacturer should adequately scrub all food grade carbon dioxide prior to liquefaction or compression. Most bottlers however use a backup system of some kind in the carbon dioxide line immediately prior to the carbonator supply regulator. This helps to remove any residual flavour, which may be present in the gas and helps to prevent losses caused by the accidental connection of a "bad" cylinder of gas.

Typically the system would consist of a trap followed by a particle filter, a carbon filter, and finally another particle filter.

Activated carbon absorbs many volatile chemicals. Carbon filters continue to

absorb material until they reach the loading capacity for the particular carbon and substance being absorbed. When the carbon reaches this loading the material which was previously being absorbed suddenly starts to pass through. This is known as breakthrough behavior. Absorption on the carbon is in most cases reversible and previously absorbed materials can be displaced by materials that are more strongly absorbed.

Considerable care must be taken with carbon filters to prevent them getting hot. Activated carbon when heated starts to desorb material which has been previously absorbed. Sudden introduction of hot gas (for example from a gas preheater) may cause the carbon to dump all the materials previously absorbed back into the gas stream.

It is advisable to have any gas heater some distance prior to the filter plant.

Positioning the filter plants as shown in the supply system diagrams have the advantage that the supply regulators are protected from any extraneous matter (corrosion products etc ) which may be carried with the gas flow.

Secondly placing the supply filters remote from the gas heating system allows any excess heat to be dissipated.

Commercial food grade carbon dioxide as supplied should require no treatment other than these simple filters.

#### SOME OF THE IMPURITIES THAT HAVE BEEN FOUND IN COMMERCIAL CARBON DIOXIDE

##### Group (1)

Contaminants which are soluble in liquid or Supercritical carbon dioxide.

These components are carried in the gas stream as either gases or precipitated materials.

Nitric Oxides NO, N <sub>2</sub> O	Produced during combustion. Normally removed by permanganate scrubbers.
Organic Nitrate and Nitro Comps	Produced by the reaction of nitric oxides with ethanolamine in the purifier.

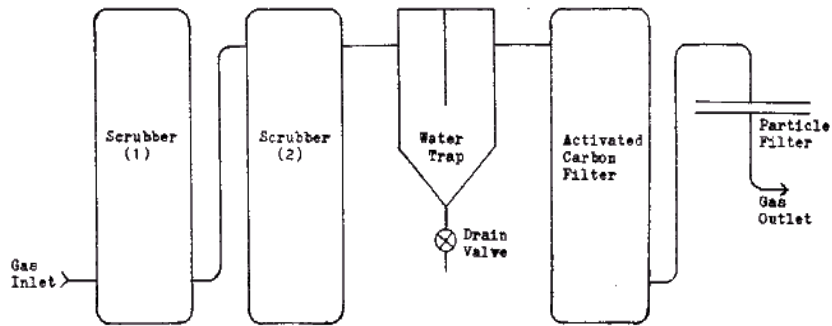
Sulphur Dioxide	Produced by burning high sulphur fuel Normally removed by permanganate scrubbers.
Monoethanolamine	Carry over from the purifiers
Hydrogen sulphide	Component of fermentation gas. Normally removed by permanganate scrubbers.
Variety of organic aroma compounds Including Ethanethiol Acetaldehyde etc	Normal component of fermentation gas Aroma may be beery or winey. Normally removed by a combination of permanganate scrubbers and activated carbon filtration
Oil	Possibly derived from partially burnt fuel. Most probable source is carryover from the gas compressor.
Water	Carry over from purifiers. May precipitate in gas lines as ice which can cause problems.
Arsine	Toxic but detectable by its strong aroma at an extremely low level. Produced by acid or moisture attack on metals containing arsenic impurity.
Phosphine	Produced by acid or moisture attack on metals containing phosphorus impurity.

#### Group (2)

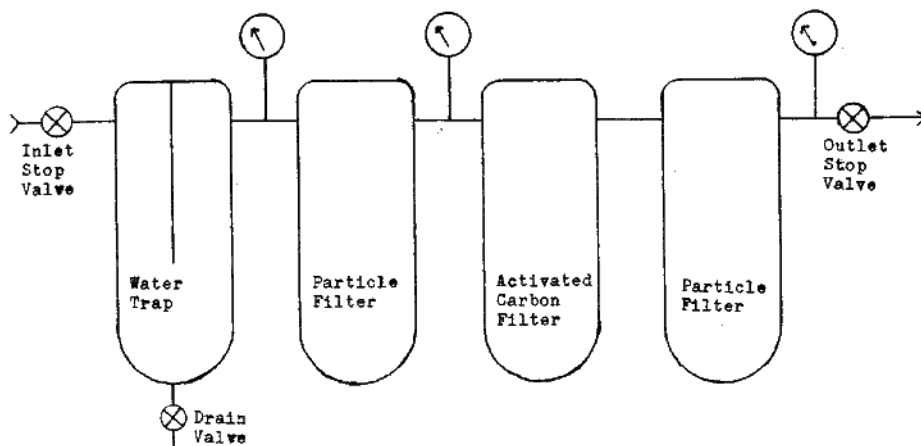
Particulates insoluble in liquid or supercritical carbon dioxide.

Usually carried in the gas stream as fine particles.

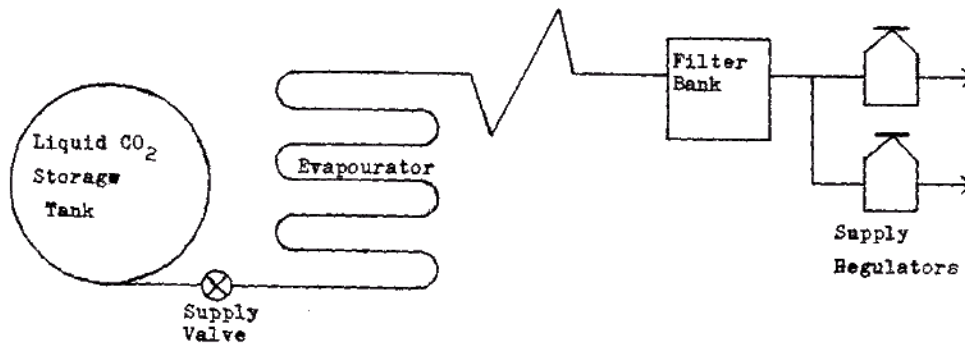
Copper Nitrates	Produced by the attack by nitric oxides on Copper pipe particularly in the presence of moisture.
Manganese Dioxide	Carry over from the permanganate scrubbers.
Ferric Hydroxides	Corrosion products from the gas line
Yeast.	A normal aerosol component of fermenter gas. Unlikely to pass through a normal gas scrubber system.



Full Carbon Dioxide Purification using scrubbers and filters, Scrubber (1)  
 Contains permanganate solution  
 Scrubber (2) Contains sodium carbonate solution  
 or  
 Scrubber (1) Contains water scrubber  
 Scrubber (2) Contains Sodium carbonate and potassium per manganate solution.



Typical Filter Bank for a Carbonated Beverage Line



Installation point for filter bank

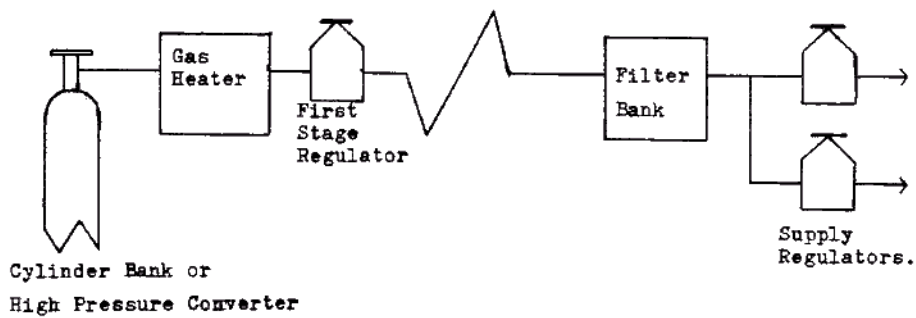


Diagram showing the location of the filter bank an gas heater in a high pressure gas supply system. The first stage regulator and piping run prior to the filter bank helps to facilitate the dissipation of any excess heat prior to the carbon filter.

# FILLING AND CARBONATION PROBLEMS

## WHERE TO START LOOKING

When the technologist is called to examine a bottling problem passive observation of the operation is the first step. This is then followed by a series of simple checks.

## TYPICAL QUESTIONS TO BE ANSWERED BY OBSERVATION ARE DURING NORMAL OPERATION;

What is the problem.

Is product fobbing

When the bottle is released is the fobbing gentle or explosive from the filling rubber. Does the fobbing cease after the initial burst or does it slowly continue.

Are the bubbles isolated or are they produced as a stream from a point.

Where is this point on the bottle wall or floating in the product.

Are there distinct patches on the bottle wall covered by bubbles after the initial fob.

Are fill heights correct.

Are all filling heads affected.

If only some bottles are affected is it the same filling heads all the time.

Is there a pattern or sequence shown in which filling heads are affected.

Are there any sealing rubber leaks.

Observe the in bottle product flow pattern during fill is it correct. Use protective face gear and other equipment while doing this as safety covers will be open.

Is the filler bowl liquid level correct.

What is pressure difference between the carbonation pressure in a closed bottle and the filler bowl after allowing for any temperature difference.

Are all valves in the product line between the carbonator and filler fully open.

Is the carbonator vessel vent valve operating.

Check the empty bottles prior to the line for any irregularities.

Is the machine turning smoothly without any shuddering.

Are the bottles moving smoothly after pressure release and before closing without any sudden impacts or irregular movements. Particularly look at transfer off lift platforms. With faster machines a variable speed strobe light should be considered an essential trouble shooting tool.

## FINISHED PRODUCT OBSERVATIONS

Have the carbonation tests made during the production run remained steady or are they slowly changing.

Has the temperature of product in the bottle after immediately filling changed during the production run.

Is there an abnormal difference between the initial pressure in the bottle and the post snift pressure.

Does the bottle open normally without violent fobbing.

RUN THE MACHINE FULL OF PRODUCT WITH PRESSURE BUT WITH OUT BOTTLES.

During the routine CIP is a good time to routinely make these observations

.

Does the gas and/or product squirt violently of the gas vent holes in the filler stem during the prepressurisation phase and stop at the end of this phase.

Is there any product or gas leak from either the product valve or the gas vent holes after the end of the prepressurisation phase.

WITH THE MACHINE STILL TURNING CLOSE THE PRODUCT INLET VALVE TO THE FILLER.

Initially only product and gas will be vented from the gas vent holes.

When the pressure in the filler bowl has dropped sufficiently all the main product valves should open.

At this stage only after the valve closer and the before the start of the prepressurisation stage should the product valves be closed. Note some machines have a gas tube purge between these two points. If this is operating the main valve will open briefly then.

Do all valves show a full product flow.

After the product valves have opened this is a good time to observe the product flow pattern.

When bottling problems occur do not assume that only one problem is present. There may be two or more problems occurring together with one obscuring another.

On one occasion the author was asked to check a plant on which some of the bottles were fobbing badly as they left the machine. The fobbing was head specific. On close examination it was found that all the heads not fobbing had leaking sealing rubbers. Further examination showed that the carbonator vessel vent had been turned off supposedly to save gas. The carbonated beverage was highly unstable due to the high air content but this was being compensated for by a prolonged snift due to the leaking sealing rubbers. The initial replacement of leaking rubbers of course resulted in all bottles fobbing. Setting the carbonator vessel vent correctly enabled correct filling of all bottles



at a lower carbonator vessel pressure. Variations of this scenario are not uncommon.

In any production operation simple observation is the key to reducing problems.

An essential if not critical part of technologists job is the FACTORY WALK ABOUT. During this the technologist simply walks around all the production areas LOOKING, LISTENING and SMELLING for anything abnormal or out of the ordinary. Anything abnormal should be checked out regardless of its initially apparent significance or insignificance . A good technologist does not sit down in the lab between tests etc he or she is on the factory floor observing.

## TABLE of CARBONATION and BOTTLING PROBLEMS

- (1) Leaking product valve on filler  
Symptoms; Bottles on the leaking valve foam as the bottle is released from the sealing rubber.
- (2) Leaking air valve on the filler.  
Symptoms as for (1).
- (3) Blocked snift valve  
Symptoms as for (1) & (2)  
Usually caused by a blockage in the snift orifice or valve passage.  
(particularly a problem after bottling products containing fruit pulp).  
Occasionally may be caused by a worn actuating pin or wear on the snift bar.
- (4) Leaking Snift Valve.  
Symptoms; The bottle tends to overfill. With a high over fill the bottle may spurt as it is released from the sealing rubber.  
With a poppet type valve it is usually caused by a cut valve rubber or a broken valve spring.  
On a plate valve it is usually caused by a worn leather or bad valve adjustment.
- (5) Leaking Bottle sealing rubber  
Symptoms; As for (4)  
Usually caused by cuts on the sealing rubber.
- (6) Bottles Dry Inside, in particular glass bottles.  
Symptoms; Odd bottles foam for a short time after being released from the sealing rubber.  
Any filling head may be affected.  
Usually found after a line stoppage when bottles have had a chance to dry out after rinsing.
- (7). Stearate inside the bottles , glass bottles.  
Symptoms; Bottles foam for up to 2minutes after being released from the sealing rubber. The patch of stearate is usually visible because of the bubbles forming on it .
- (8) Traces of oil inside the bottles PET particularly.

Symptoms; As for (7).

A particular problem with PET bottles if the air used for the final blowing is contaminated with oil. Oil droplets wet the surface of the PET and tend to spread readily over the bottle surface.

(9) Dirty bottles.

Symptoms; Bottles foam after release from the sealing rubber. Bubbles continue to rise in a stream from points on the dirt.

(10) Etched Bottles, glass only.

Symptoms ; Odd bottles foam on being released from the sealing rubber and bubbles may continue in a stream from a point on bottle surface with the stream gradually dying out .

Found only on new glass bottles. Before rinsing the affected bottles show soda ash crystal dendrites on the inside surface of the bottle.

(11) Cracking around the sprue on PET bottles.

Symptoms; as for (10).

The stream or streams of bubbles rise from the region inside the bottle adjacent to the sprue.

(12). Dicalite or filter aid in the bottling syrup.

Symptoms; All bottles foam when released from the sealing rubber.

Foaming tends to continue for a short time after pressure release.

Sometimes the bubbles are produced in a stream originating from a point floating freely in the beverage. Usually caused by a perforated filter pad or insufficient circulation of the syrup or product through the filter pad prior to the final filtration run.

(13). Flooded Filler Bowl.

Symptoms; All bottles come off the filler only partially filled.

Caused by the filler bowl being flooded above the level of the air valves.

This may in turn be caused by;

- (a) No gas counter pressure supply.
- (b) Leaking filler valves.
- (c) Leaking filler bowl vent valve
- (d) Jamming level control float.
- (e) Incorrect setting of float control.

- (f) Jamming counter pressure gas supply valve.
- (14) Insufficient product in the filler bowl.  
Symptoms; Many or all bottles come off the filler partially filled.  
Filler bowl has full pressure inside.  
This may be caused by;
- (a) Filler bowl vent valve not open.
  - (b) Level control float jamming.
  - (c) Incorrect float setting.
- (15) Deflector rubber (umbrella) missing or damaged.  
Symptoms ; One bottle only partially fills.
- (16) Air accumulation in carbonator vessel.  
Symptoms; (a) Carbonation level lower than expected for the machine settings. Plant pressure readings are correct.  
(b) With higher air levels gushing occurs as the bottles come off the sealing rubbers. The foaming immediately dies down. All valves are affected.  
Causes; (a) Carbon dioxide storage vessel headspace gas being used. Non carbon dioxide gas accumulates in the storage vessel head space gas.  
(b) Carbonator vessel vent valve not working.  
(c) Air leaking in through the carbonator pump gland.  
This can occur despite a higher pressure inside the pump.
- (17) Carbonator discharge valve not fully open or other restriction in the carbonated beverage line.  
Symptoms; Bottles foam as they come off the filler and the product has low carbonation.  
Caused by a restriction in the carbonated product line resulting in a localised pressure drop. Check the filler bowl pressure. This should at all times be 10 to 15 psi (60 to 100 KPa) higher than the equilibrium partial pressure of carbon dioxide anywhere in the system.
- (18) Yeast in the product.  
Symptoms; All bottles foam as they come off the filler. The foam persists for some time after the bottle comes off the filler.
- (19) Misalignment of lift platforms and bottle runners.

Symptoms; Particularly on high speed machines bottles from one or more heads foam after leaving the filler. The foaming dies down immediately. Occasionally in severe cases a bottle may jump out of the star wheel.

(20) Valve operating lever loose.

Symptoms ; Intermittent foaming from the affected head.

(21) Valve operating lever jamming.

Symptoms; Intermittent foaming from one or more heads. The foaming is not persistent. Particularly on high speed machines the faulty valve does not always foam. Instead due to valve closer bounce heads following the affected head foam. A common pattern is that the first and third head following the jamming valve foam. Other patterns may occur.

(22) Valve closer incorrectly positioned.

Symptoms; Bottles from some or all of the filling heads foam. A repeating pattern of low fills tends to occur. Valve closer bounce may also occur see (21).

(23) Valve opener not operating correctly.

Symptoms; Some or all valves are not filled at all a repeating pattern of no fills may occur.

(24) Snift bar incorrectly positioned.

Symptoms; Some or all of the bottles foam as they come off the filler . A repeating pattern of low fills may occur.

(25) Refrigeration failure; Depending on the carbonation plant either low carbonation or foaming may occur. The carbo-cooler typically gives low carbonation without foaming.

(26) Oil in the carbon dioxide or product.

Symptoms; (a) Product tends to foam when coming off the filler. The foaming tends to persist for sometime. After the product settles down there is usually an oily ring or mark around the inside of the bottle neck. Citrus oils if not properly dissolved can cause foaming.

(b) On carbonators which have baffles or plates traces of oil may prevent full wetting of the plates. Low

carbonation then results.

(27) Surfactants present in product.

Symptoms; Carbonation is lower than expected for the machine settings. Product does not normally gush or foam. Effect may be product type dependent and consistent from batch to batch as is normally found with products containing oil emulsions. Or it may occur unexpectedly due to contamination with cleaning agents for example.

(28) Air in syrup.

Symptoms; Syrupe Machines. Bottles foam when leaving the filler. Bubbles of gas may be seen rising from the surface of the syrup in the bottom of the bottle.

(29) Too much precarbonation injection.

Symptoms; Bottles foam as they come of the filler. The foam dies down immediately. Carbon dioxide usage is high. The partial pressure of the carbonated beverage approaches the pressure in the filler bowl. Can occur with the Mojonier "carbotrol" system and almost any carbon dioxide injection system

(30) Excessive snift.

Symptoms; Bottles after being filled lose an excessive amount of beverage during snifting and may either come off the filling machine quietly or may foam badly depending on the severity of the snift.

Usually caused by either an enlarged snift orifice or where the amount of snift is adjustable, by incorrect adjustment.

(31) Filling time too short.

(a) Not sufficient time for the product to clear.

Symptoms; The bottles are filled but spurt violently when released from the sealing rubber. There is insufficient time for finely dispersed gas bubbles to settle out.

(b) Not sufficient time for the bottle to fill.

Symptoms; Part filling of the bottles occurs. Typically this happens if the filler speed is not reset after changing bottle

sizes from a small to a larger bottle.

(32) Filler Bowl Headspace gas dissolved in the product.

Symptoms; Bottles gush when released from the bottle sealing rubber.

All filling heads affected.

Causes; (a) Filler stopped for a length of time allowing headspace gas to accumulate in the product.

(b) Excessive agitation eg. too much stopping and starting of the filler causing an excessive amount of filler bowl headspace gas to be dissolved in the product.

The filler bowl headspace contains air derived from the bottles being filled.

(33) Low carbonator vessel pressure, pressure does not rapidly change .

Symptoms; Low carbonation no foaming.

Causes ; (a) Blockage in gas supply line.

(b) Incorrect regulator setting.

(c) Regulator failure.

(34) Sudden fall in carbonator pressure.

Symptoms; Low carbonation and foaming as the bottles are released from the sealing rubber.

Causes ; (a) Carbon dioxide has run out.

(b) Sudden Regulator failure or line blockage.