

Aug. 31, 1965

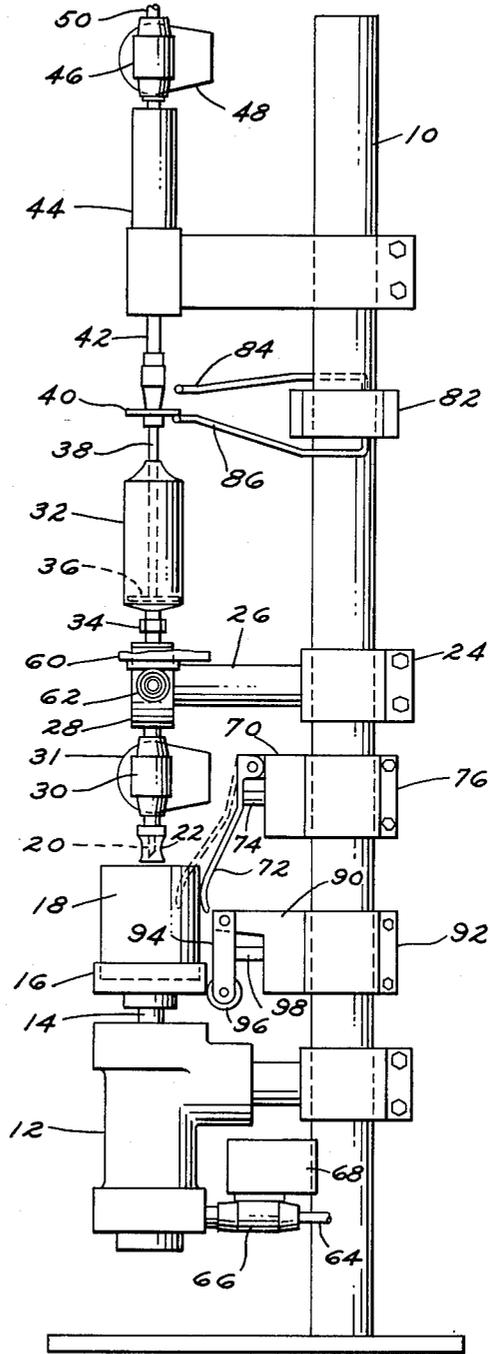
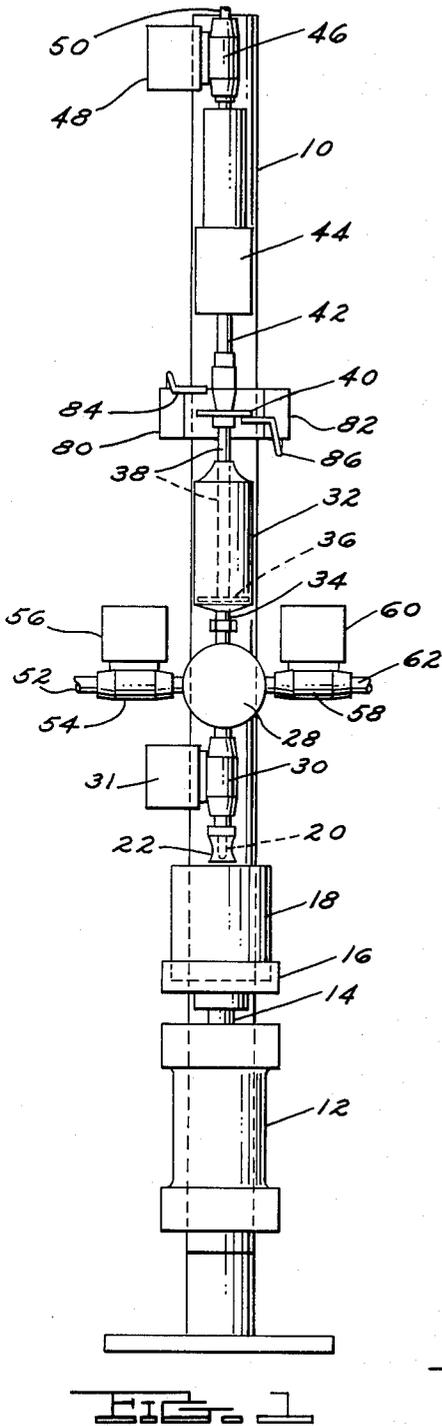
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3,203,248

APPARATUS AND METHOD FOR SAMPLING HEADSPACE GAS OF CANS

Filed April 19, 1963

3 Sheets-Sheet 1



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3 Sheets-Sheet 2

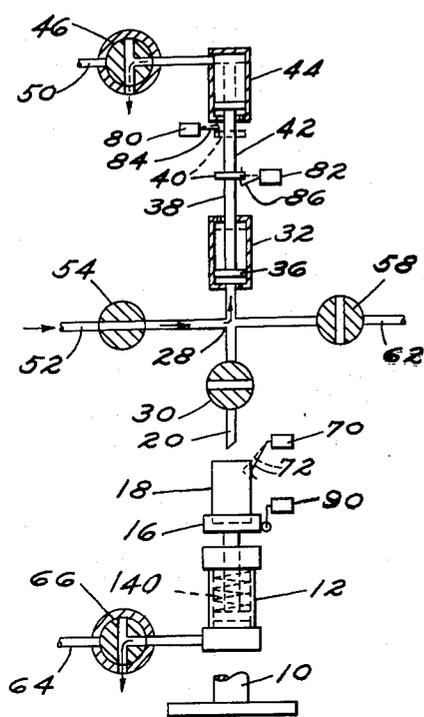


FIG. 3

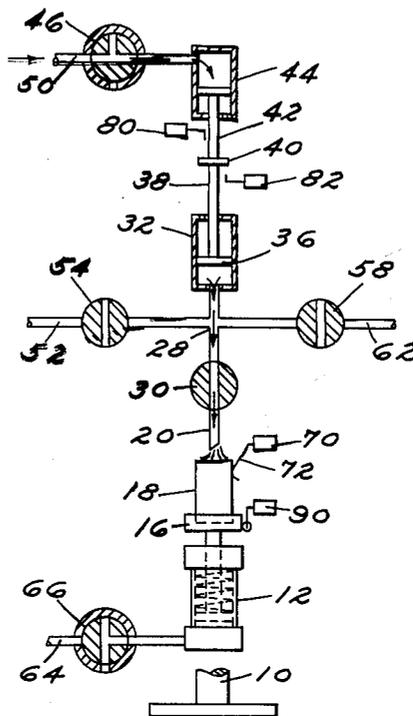


FIG. 4

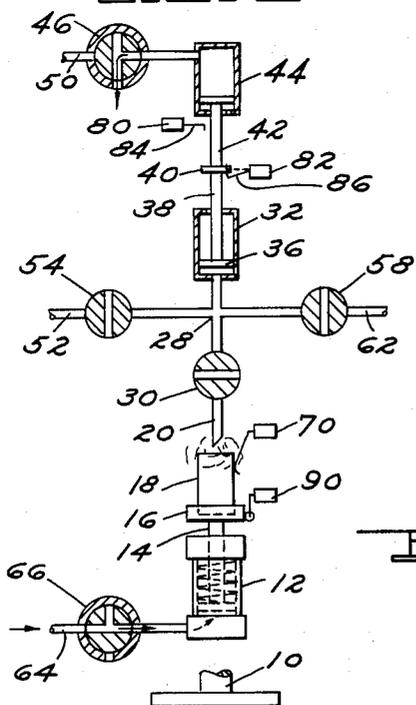


FIG. 5

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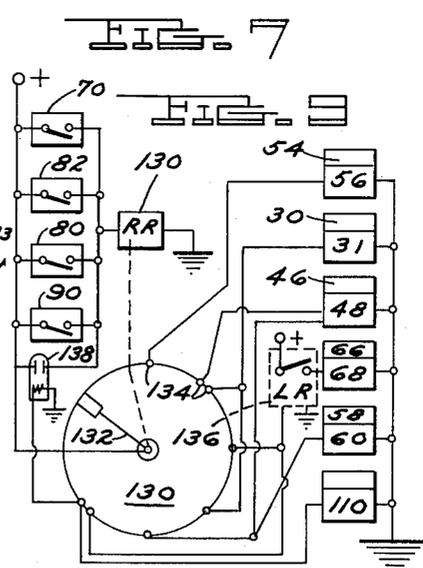
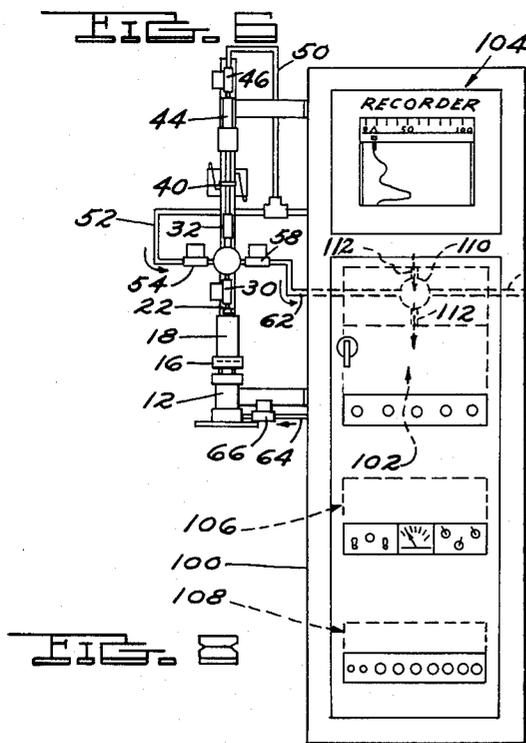
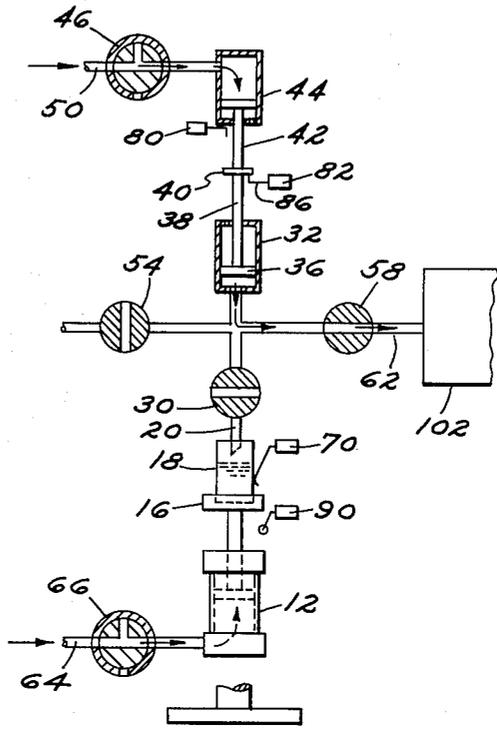
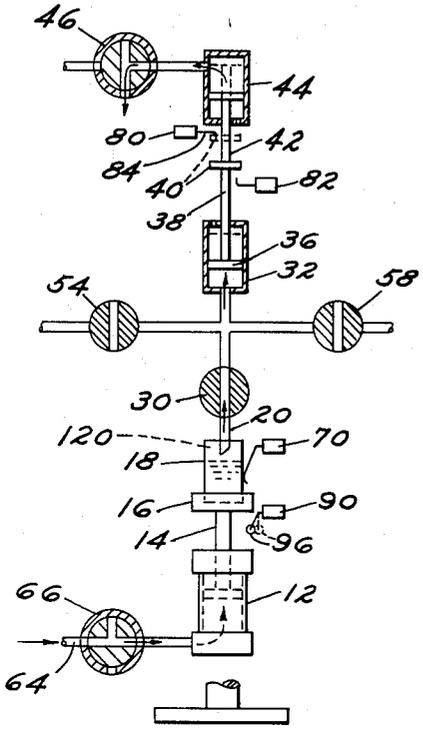
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APPARATUS AND METHOD FOR SAMPLING HEADSPACE GAS OF CANS

Filed April 19, 1963

3 Sheets-Sheet 3



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3,203,248
**APPARATUS AND METHOD FOR SAMPLING
 HEADSPACE GAS OF CANS**

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 Filed Apr. 19, 1963, Ser. No. 274,157
 11 Claims. (Cl. 73—421.5)

This invention relates to inspection of sealed containers wherein a sample of gas is obtained from the headspace of the container and qualitatively and/or quantitatively analyzed, and more particularly to improvements in methods and apparatus for sampling the headspace gas present in a sealed container and analyzing it for air content.

The analysis of headspace gas in a sealed container is of importance because of the detrimental effect which high concentration of certain components, such as air or oxygen, in the headspace gas may have on the shelf-life and stability of the product in the container. This is true of many packaged products in various industries, particularly in the food and brewing industries. For example, the harmful effects of oxygen on packaged beer have been recognized for many years; see "A New Bottling Technic for Experimental Studies of Beer" by P. F. Gibbs et al., pp. 130-148 of the American Society of Brewing Chemists Proceedings (1950). "Off-tastes" and physical hazes can often be attributed in part to oxygen inclusion, and the rate at which these off-tastes and hazes are formed is somewhat proportional to the oxygen content. Thus, an elevated oxygen content shortens shelf-life.

Considerable effort is devoted toward keeping the air and/or oxygen inclusion at a minimum during processing. Continuous or bulk segments of the product can be maintained under control by gas analysis at various stages of lagering and sufficient time is available for corrective action to be taken before the product is finally approved for packaging.

Modern packaging machines attain speeds as high as 1000 individual units (containers) per minute. Air or oxygen inclusion at this packaging stage in the process is also undesirable. In contrast to the lagering stage mentioned above, time is of essence in learning of the air inclusion because the packages are being filled at a 1000 units/minute rate and no corrective action can be taken on the sealed containers.

Existing analytical procedures and sampling techniques are ill-adapted for rapid, efficient and economical production quality control. Headspace gas samples are taken manually at relatively infrequent intervals and are subjected to a time consuming analysis in order to detect high air conditions in the packaged beer. This is usually accomplished by warming up the package, piercing the container and shaking out all of the gas into an absorption burette filled with caustic. The gas is composed mainly of CO₂ which is soluble in the caustic. The volume of the gas not dissolved in the caustic is measured and reported as air.

Gas chromatography has also been used to measure the oxygen content of headspace gas under conditions where sufficient time has elapsed for the headspace gas to reach a state of equilibrium with the dissolved gas, and this has been shown to be related to the air in the whole package; see "Determination of the Oxygen Content of Air in Beer by Gas-Solid Chromatography" and "Effect of Ascorbic Acid on the Oxygen Content of Beer as Determined by Gas Chromatography" by J. L. Bethune et al., pp. 170-175 of the Journal Inst. Brew. (1958) and pp. 62-65 of the American Society of Brewing Chemists Proceedings (1958) respectively.

An object of the present invention is to provide an automated sampling device for obtaining samples of head-

space gas from sealed packages for quality control analysis of the packaged product at a rate commensurate with high-speed packaging equipment.

Another object of the present invention is to provide an improved method of operating sampling equipment for obtaining a sample of gas from the headspace of a sealed package which reduces contamination of the sample and insures speed and accuracy of operation for effective quality control of modern high-speed packaging equipment.

Yet another object is to provide an improved inspection method wherein a sealed container is sampled at a predetermined time during the packaging operation to obtain a more accurate estimate of atmospheric pick-up during filling to thereby improve packaging quality control.

A further object is to provide an improved apparatus adapted to receive packages selected manually or mechanically from a production line at any given time interval, rapidly extract a relatively large, statistically reliable sample of gas from the headspace of the package and transfer a portion of it to a gas sampling apparatus for analysis.

Still another object is to provide an improved device for sumping headspace gas which is relatively simple and economical in construction and operation.

A more particular object of the invention is to provide a complete mechanized unit for sampling the headspace in packaged beer, analyzing the headspace gas content by gas chromatography and recording the results with the speed and accuracy required for effective control of high-speed packaging equipment.

Other objects, advantages and features of the present invention will become apparent from the following description taken in conjunction with the accompanying drawings wherein:

FIG. 1 is a front elevational view of a gas sampling device of the invention, somewhat simplified in order to facilitate understanding.

FIG. 2 is a side elevational view of the device of FIG. 1.

FIGS. 3, 4, 5, 6 and 7 are simplified semi-schematic illustrations of the sampling device as viewed in FIG. 1 respectively illustrating the steps of the method of the invention as well as the operating sequence of the device through a sampling cycle.

FIG. 8 is a front elevational view of a complete mechanized sampling and analyzing unit of the invention incorporating the sampling device of FIG. 1 in an improved co-operative arrangement with a gas chromatograph and associated recording, control and power supply equipment.

FIG. 9 is a simplified schematic diagram of an electrical control circuit for the gas sampling and analyzing apparatus of the invention.

Referring in more detail to FIGS. 1 and 2, there is shown by way of example a presently preferred embodiment of a gas sampling device of the invention. The device includes an upright supporting column 10 to which is attached a pneumatic cylinder 12 containing a piston-actuated plunger 14 for raising and lowering a container supporting platform 16 mounted on the upper end of plunger 14. Platform 16 is recessed for receiving and supporting the particular shape and size of the package to be sampled, herein schematically illustrated as a can of beer 18 which may be manually placed on platform 16. It is to be understood that platform 16 may be adapted to operate in conjunction with a suitable timed conveyor (not shown) adapted to select packaged beer from a production line at any given time interval and present them one at a time to platform 16 when it is in its lowermost, loading position of FIGS. 1 and 2.

A hollow piercing needle 20 encased in a rubber seal-

ing gasket 22 is supported in fixed position directly above and closely spaced from the top of container 18. The supporting structure for needle 20 comprises a clamp bracket 24 (FIG. 2), a horizontal tube 26 secured at one end to bracket 24 and having a fluid conduit junction box 28 secured to the other end thereof, and a two-way valve 30 coupled at its upper end to chamber 28 and having the piercing needle assembly secured to its lower end. Piercing head 20-22 may be of known design, such as the bottle cap piercer sold by Zahm & Nagel Co., Inc., of Buffalo, N.Y. Needle 20 is made of hardened steel and is pointed for puncturing the top of can 18 or the cap on a bottle. Gasket 22 completely encircles needle 20 and extends therebelow so that it is axially compressed by the top of the container before the container top is punctured by needle 20 in order to provide a sealed connection between the headspace of can 18 and the hollow interior of needle 20.

A sampling cylinder 32 is supported in a fixed vertical position above junction box 28 by a free flow inlet and outlet fitting 34 connected at its lower end with junction box 28 at its upper end with the bottom of the cylinder. A plunger reciprocates vertically with a gas tight fit in cylinder 32 and comprises a piston 36 secured to the lower end of a vertical piston rod 38. Rod 38 extends through the top of cylinder 32 and carries a cross arm 40 at its upper end which is also secured to the lower end of a plunger 42 of a pneumatic cylinder and piston assembly 44 of conventional construction. The upper end of cylinder 44 supports a three-way inlet-exhaust valve 46 which is operated by a solenoid 48 for admitting motive pressure fluid from a supply line 50 to cylinder 44 and for exhausting the same therefrom.

Purging gas is supplied to junction 28 via a line 52 (FIG. 1) and a two-way valve 54 controlled by a solenoid 56. Another two-way valve 58 and associated operating solenoid 60 are connected to the opposite side of junction 28 and control the flow of gas therefrom through a discharge line 62 which leads to a suitable gas analyzing device, preferably a gas chromatograph as described in more detail hereinafter. Sampling valve 30 is likewise controlled by a solenoid 31.

The container-elevating cylinder 12 is supplied with motive fluid via a line 64 (FIG. 2) which is connected to the lower end of the cylinder via a three-way valve 66 controlled by a solenoid 68.

The control mechanism for the gas sampling device of the invention includes four microswitches of known design. One microswitch 70 (FIG. 2) has a pivoted arm 72 normally biased outwardly therefrom to the broken line position of FIG. 2 by a spring-pressed switch button 74. Placing container 18 on platform 16 moves arm 72 to the solid line position of FIG. 2 and thereby depresses button 74 to actuate switch 70. The position of switch arm 72 may be adjusted for the particular container by moving a switch clamp 76 along column 10.

The second and third microswitches 80 and 82 (FIG. 1) are adjustably mounted on opposite sides of column 10 and adjacent the path of travel of cross arm 40. Vertically pivotable and adjustable switch arms 84 and 86 extend respectively from switches 80 and 82 so as to project into the path of travel of cross arm 40. Arm 86 is normally positioned so that it is struck by cross arm 40 at the lower limit of travel thereof, e.g., when piston 36 bottoms in cylinder 32. Arm 84 is normally positioned so that cross arm 40 strikes it at the upper limit of travel of piston 36. Microswitches 80 and 82 are thus adapted to serve as limit switches for piston travel and are actuated by vertical reciprocation of the piston.

The fourth microswitch 90 (FIG. 2) is secured by a clamp 92 to column 10 adjacent the path of travel of platform 16. Switch 90 supports an arm 94 which pivots at its upper end on switch 90 and carries a roller 96 journalled on its lower end. Microswitch 90 is positioned such that when platform 16 is in its loading position roller 96 rides against the rim of the platform to thereby main-

tain a switch actuating plunger 98 of switch 90 depressed against the pressure of its spring. When platform 16 is elevated by plunger 14, roller 96 rides off the lower edge of the rim of platform 16, permitting arm 94 to be swung outwardly by plunger 98, thereby actuating switch 90.

Referring to FIG. 8, the above-described gas sampling device of the invention is shown attached to one side of a cabinet 100 which contains all of the necessary equipment to provide a completely mechanized headspace gas sampling and analyzing unit in accordance with the invention. This equipment includes: a gas chromatograph 102 of known design, such as that identified by the trademark Aerograph, Model No. A-90, made by Wilkens Instrument & Research, Inc.; a one millivolt potentiometric recorder 104, such as that identified by the trademark Elektronik, Model No. 15307856-01-05-0-000-790-07009, made by Brown Instruments Division of Minneapolis-Honeywell, Philadelphia, Pennsylvania; a power supply and electrical bridge unit 106 for the gas chromatograph 102; and a control unit 108 for the entire apparatus.

Cabinet 100 also houses a container (not shown) of compressed gas, such as helium, to provide a source of supply of carrier gas for chromatograph 102. This supply of carrier gas is also connected via supply line 52 to the gas sampling device to thereby serve as a source of purging gas. Supply lines 50 and 64 leading to pneumatic cylinders 44 and 12 respectively may either be connected to this source of carrier gas (as shown in FIG. 8) or to a separate supply of compressed air, whichever is more convenient and economical for the particular installation. The sample gas discharge line 62 is connected to a solenoid-operated valve 110 disposed within chromatograph 102. Valve 110 is adapted to trap about one-half cubic centimeter of the thirty-odd cubic centimeters of sample headspace gas supplied thereto by the gas sampling device via line 62 as the gas is flowing through the valve. Valve 110 is also connected to the intake line 112 supplying carrier gas to the chromatographic column (not shown) of chromatograph 102 so that after valve 110 is actuated to trap the one-half cc. of sample gas, this trapped portion of the total sample is diverted from sampling line 62, 113 to line 112 and carried through the chromatographic column by the incoming carrier gas in the direction shown by the broken line arrows in FIG. 8. The untrapped portion is vented to atmosphere via a vent line 113. Valve 110 may comprise a conventional six-way petroleum gas sampling valve, such as that made by Wilkens Instrument & Research, Inc.

One suitable control circuit for the sampling device of the invention is shown in simplified schematic form in FIG. 9 and centers around an industrial-type ratcheting relay 130. Relay 130 is stimulated by an impulse from any one of the four microswitches 70, 80, 82 and 90. Each impulse causes the rotatable conductor 132 of the relay to index clockwise (as viewed in FIG. 9) from one contacting position to the next in the usual manner. Conductor 132 thus supplies energizing current to the particular solenoid or solenoids 56, 31, 48, 68, 60 and 110 connected to the contact or contacts 134 in registry with the conductor at any given time. There are six contact positions and an off position, and suitable interconnections are provided at each position to obtain the sequence of operation described hereinafter. A conventional latching relay 136 is used in the circuit to hold the container in the pierced condition of FIGS. 6 and 7 for more than one operation. A conventional time delay tube 138 is used to hold an injector (not shown) of the gas chromatograph in the circuit long enough to allow all of the sample gas to be driven from the gas sampling valve 110.

Operation

Referring to FIG. 3 the sequence of operation is started when a capped or closed container, such as a bottle or can of beer to be sampled is placed on platform 16. This moves arm 72 to the solid line position of FIGS. 1 and 3 which trips microswitch 70, thereby energizing

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solenoid 56 which opens valve 54, valves 30 and 58 being closed at this time. Pure carrier gas at line pressure is thus admitted from line 52 via junction chamber 28 and fitting 34 into cylinder 32, forcing piston 36 and rod 38 upwardly until the sampling cylinder is filled with carrier gas at line pressure (broken line position, FIG. 3). Upward movement of plunger 38 is opposed only by gravity and atmospheric pressure since valve 46 at this time connects cylinder 44 above plunger 42 to atmosphere.

When the sampling cylinder is full, cross arm 40 strikes arm 84 (FIG. 3), tripping microswitch 80 which closes valve 54 and opens valves 46 and 30 (FIG. 4). With valve 46 open, gas under pressure from line 50 is admitted into cylinder 44 to force plungers 42, 38 and piston 36 downwardly, thereby forcing the pure carrier gas out of cylinder 32, down through valve 30 and needle 20 and out over the top or crown of the package to be sampled.

The above-described cylinder-filling and cylinder-emptying steps thus purge the sampling cylinder 32, junction 28 and associated valve connections and needle 20 of contaminating gases, a metered amount of carrier gas serving as the purging gas. Also, the carrier gas issuing from needle 20 momentarily displaces atmosphere in the space between needle 20 and the top of container 18 just prior to and during the puncturing step described hereinafter, thereby forming a protective gas envelope which temporarily is free of contaminating atmosphere.

When all of the carrier gas has been forced from cylinder 32 by piston 36 being driven to its bottomed position (FIG. 5), cross arm 40 strikes arm 86 of microswitch 82. This de-energizes solenoids 48 and 31, thereby opening cylinder 44 to exhaust via valve 46, and closing valve 30. This also energizes solenoid 68 which opens valve 66, thereby admitting compressed air to cylinder 12 to force platform 16 and hence can 18 upwardly until needle 20 pierces the top of container 18 and projects into the headspace 120 thereof above the liquid level in the container (FIG. 6).

During this upward movement of the container, the rubber sealing gasket 22 (FIGS. 1 and 2) engages the top of container 18 and is compressed axially so that by the time the needle punctures the top of the container, the gasket is in firm sealing engagement therewith. Due to the protective carrier gas envelope (FIG. 5) displacing atmosphere in the vicinity of needle 20, little or no atmospheric contamination occurs as the sealed connection is made between needle 20 and the container headspace.

The upward motion of platform 16 during the container piercing step moves platform 16 out of engagement with roller 96 (FIG. 6), allowing arm 94 to be swung away from switch 90 by switch button 98 and thus tripping microswitch 90. This re-opens valve 30 and allows the gas in the container headspace 120, which in the case of a standard 12 ounce can of beer is under normal packaging pressure of about 30 p.s.i., to flow upwardly into sampling cylinder 32, valves 54 and 58 still being closed. At this pressure and at packaging room temperatures the volume of gas in the headspace of a 12 ounce bottle or can of beer is sufficient to force piston 36 upwardly again until cylinder 32 is full of a predetermined metered amount of headspace gas, for example 30 cubic centimeters. When the upper limit of piston 36 is set at a cylinder volume of 30 cubic centimeters, a drop in the headspace pressure almost to atmospheric pressure can be obtained by employing a properly dimensioned and lightweight piston-plunger assembly 36-42 and a sensitive microswitch 80.

When cross arm 40 strikes arm 84 at the end of the second cylinder-filling stroke of piston 36 (broken lines, FIG. 6), microswitch 80 is again tripped and valve 30 is re-closed, thereby isolating cylinder 32 from container 18. This also causes valves 46 and 58 to open (FIG. 7). With valve 46 open, plungers 42, 38 and piston 36 are again forced downwardly, thereby forcing the 30 cc. of

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sample gas out of cylinder 32, through valve 58 and line 62 into the gas chromatograph 102.

When all of the sample has been forced out of cylinder 32, cross arm 40 strikes arm 86 which trips microswitch 82, thereby closing valves 58, 46 and 66 which opens cylinders 44 and 12 to atmosphere (FIG. 3). With cylinder 12 opened to exhaust, a return spring 140 in cylinder 12 acts on plunger 14 to disengage piercing needle 20 from the container and return platform 16 to the loading-unloading position of FIG. 3.

The last-mentioned actuation of microswitch 82 also operates solenoid valve 110 (FIG. 8) so as to inject a small portion (approximately ½ cc.) of the total sample (about 30 cc.) into the gas chromatographic column via line 112 for analysis. After a predetermined time delay, for example three seconds, during which time the trapped aliquot of the sample gas is thoroughly driven from the small lumen of the valve 110, time delay tube 138 (FIG. 9) energizes ratcheting relay 130 to close down all circuits and return the apparatus to home position ready for another sample.

The entire sampling operation is completed in a minimum of time, on the order of five seconds, and is accurately controlled in a simple manner by the two aforementioned mechanical motions and associated microswitches and circuit.

From the foregoing description it will now be apparent that the gas sampling device of the invention, both alone (FIGS. 1 and 2) and in combination with the gas analyzing apparatus of the invention (FIG. 8), as well as the manipulative method of the invention illustrated by the manner in which the improved mechanism is operated in FIGS. 3-7, provides an improved apparatus and method for rapidly, accurately and reliably obtaining a sample of the headspace gas from a closed container and analyzing the same.

Assuming that the apparatus of the invention is positioned relatively close to a production line, a visual read-off is provided on the chart paper of recorder 104 within approximately thirty seconds from the time the sample container is taken from the production line. As compared to known prior art techniques, this constitutes a reduction in the time lag between malfunction and shutdown which at normal brewery production rates substantially reduces the number of defectively packaged containers. Thus, the gas sampling and analyzing apparatus of the invention provides an important step towards complete automation of beverage packaging control with the attendant savings in production cost as well as improvement in product quality.

In accordance with the illustrated method and apparatus of the invention, the carrier gas conventionally required as a background or reference gas for chromatograph 102 is also employed as a purging gas and motive fluid to operate the sampling device. This simplifies the apparatus by elimination of extra supply lines and sources of supply. Since gas chromatograph 102 requires only ½ cc. of sample from the headspace to provide an accurate and reliable analysis of the oxygen or other component content of the headspace gas, a relatively small pressurized volume of headspace gas in a closed container of beer may be used to lift piston 36 to fill cylinder 32 with the sample. This in turn eliminates the need to shake the container to develop additional pressure in order to obtain enough sample, a messy and unreliable procedure common to prior art techniques.

It is also to be noted that the headspace gas is well mixed as it is released from the headspace pressure and expands into cylinder 32. Moreover, due to the rapidity of operation, the sample is obtained and valve 30 re-closed before any appreciable amount of carbon dioxide or other gas dissolved in the beer can effervesce into the headspace in response to the pressure drop produced therein by release of the sample gas to cylinder 32. Hence the composite sample obtained in the sampling cylinder

represents a true average of the gaseous content of the container headspace. Then valve 110 traps about $\frac{1}{60}$ of the total sample taken, preferably the last portion of the sample being forced via line 62 through valve 110. This results in improved reliability from a statistical standpoint.

Another advantage of the apparatus and method of the invention is the utilization of first the purging (carrier) gas and then the sample gas to actuate the piston 36 on the successive cylinder-filling strokes of its four stroke cycle. This simplifies the electrical controls and mechanical apparatus required while also metering the purging gas. The micro-switches associated with container 18 and the two moving parts, e.g., platform 16 and piston-plunger 36, 38, provide a simple and accurate system for controlling the complete cycle of events once a container has been placed on the platform.

It is to be understood that the sampling apparatus of FIGS. 1 and 2 is not limited to use with a gas chromatograph; if desired, line 62 may be connected to a gas absorption burette or other device for analysis of the headspace gas in a known manner. Also, some gas other than helium, such as carbon dioxide, may be used as the purging gas.

The present invention also contemplates an inspection method preferably employing the above-described apparatus and manipulative method in a given sequence related to given steps in the packaging operation to obtain improved quality control results. Taking a conventional beer bottling process as an example, the empty bottles are fed to the usual filling machine where they are rapidly and continuously filled with liquid beer under an atmospheric cover. Then due to the turbulence of filling, plus the added effect of vibrators and/or impact devices associated with the filler, the CO_2 dissolved in the liquid beer is released therefrom and foams up to drive the air from the headspace just prior to capping of the filled bottle in the capping machine. At this point, random or periodic inspection is performed in accordance with the present invention by taking the sample sealed package, e.g., the selected filled and capped bottle of beer, from the line immediately after capping and maintaining it in a relatively quiescent condition until the headspace of the package is sampled for analysis in the previously described manner.

One important aspect of this inspection method resides in the ability to perform the sampling step within a given short period of time after the sealing operation. This time period is determined by the rate of transfer of gas from the headspace to the liquid (or solid) product in the container, the sampling step being performed before an equilibrium condition can occur between the gaseous phase headspace and liquid (or solid) phase product in the sealed container. For example, the headspace of packaged beer is sampled as soon as possible after sealing, preferably within a period of a minute or two but no longer than about fifteen minutes under ordinary bottling room conditions. The sample of gas obtained from the headspace is then analyzed to determine the relative amounts of gaseous constituents thereof to provide an indication of the extent to which such constituents have been introduced in the filling and sealing operation.

Inspecting sealed packages of beer in the above manner results in a much more accurate estimate of air pick-up during filling and a more rapid indication of the occurrence of a malfunction in the filling equipment, both of which contribute to a significant improvement in quality control and reduced scrappage rate. By employing the above-described gas sampling and analyzing unit, the sample may be obtained with the requisite rapidity from the headspace, that is, while much of the air picked up at the filling-capping equipment is still in the headspace rather than after it has dissolved in the beer to produce interfacial equilibrium. Since the gross air con-

tent of the headspace immediately after sealing substantially exceeds that existing after equilibrium, it will represent a larger percentage of the raw sample and hence provide more accurate test results for a given sampling test error. Also the reduction in the elapsed time between sealing and testing reduces the effect of environmental variables in the test procedure and hence provides improved reliability.

We claim:

1. Apparatus for sampling gas present in the headspace of a closed container comprising means for piercing the container to establish communication with the headspace thereof, a cylinder, a piston movable in said cylinder, a conduit connecting said piercing means with said cylinder on one side of said piston, a valve operable for opening and closing said connection, means for admitting purging gas to the cylinder during movement of the piston away from said conduit to a first position and while said valve is closed, means for moving the piston from said first position towards said conduit to a second position, means for opening said valve during said movement of the piston from the first to second positions to thereby expel the purging gas from the cylinder via said piercing means, means for causing relative movement of said container and piercing means to effect said piercing and means controlling said piston moving means for effective reciprocation of the piston a second time for admitting the headspace gas via said piercing means into the cylinder and for expelling it from the cylinder to a sample receiver.

2. Apparatus for sampling gas present in the headspace of a closed container comprising means for piercing the container to establish communication with the headspace thereof, means for causing relative movement between the container and said piercing means to effect said piercing, a cylinder, a piston movable in said cylinder, a conduit connecting said piercing means with said cylinder on one side of said piston, a valve operable for opening and closing said connection, means for admitting purging gas to the cylinder during movement of the piston away from said conduit to a first position and while said valve is closed, means actuated by movement of the piston to said first position to cause movement of the piston in the opposite direction towards said conduit to a second position and to open said valve for expelling the purging gas from the cylinder via said piercing means, and means actuated by movement of said piston to said second position to cause said relative movement between said container supporting and piercing means to effect said piercing to thereby admit headspace gas to said cylinder.

3. The combination set forth in claim 2 wherein said piston is adapted to be moved from said second to said first position by the force exerted thereon by the purging gas and headspace gas admitted to said cylinder.

4. The combination set forth in claim 2 including a sample receiver connected to said conduit and wherein said means actuated by movement of said piston to said first position is adapted, after said headspace gas is admitted to said cylinder, to close said valve and initiate movement of said piston towards said conduit to expel headspace gas from the cylinder to said sample receiver.

5. The combination set forth in claim 4 wherein said sample receiver comprises a gas chromatograph, a carrier gas circuit for said chromatograph including means for supplying carrier gas under pressure and means operably connecting said cylinder in said circuit such that the carrier gas serves as said purging gas and said cylinder meters the amount of carrier gas utilized for purging.

6. Apparatus for sampling headspace gas in a closed container comprising:

- (1) a support,
- (2) a piercing needle fixed on said support,
- (3) a platform movably mounted on said support and adapted for supporting the container with the top thereof spaced below said needle,

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- (4) means for raising said platform to move the container into pierced engagement with said needle,
 (5) a piston and cylinder unit mounted on said support and having a port communicating with said needle,
 (6) means for moving said piston between positions remote from and adjacent said port,
 (7) means for supplying a purging gas to said cylinder and for conducting gas expelled from said cylinder by movement of said piston,
 (8) valve means for controlling the cylinder-needle communication, the supply of purging gas to and the expulsion of gas from said cylinder, and
 (9) control means associated with said piston and said platform for actuating said platform raising means, said piston moving means and said valve means in the following operating sequence:

(a) move said piston to its remote position to fill the cylinder with purging gas, shut off the supply of purging gas and initiate movement of the piston to expel the purging gas from the cylinder through said port and needle,

(b) move said piston to the adjacent position to empty the cylinder of purging gas, cause said platform to move the container into pierced engagement with said needle and permit headspace gas from the container to flow via the needle into the cylinder, and

(c) move the piston to its remote position to fill the cylinder with headspace gas, close needle-cylinder communication, connect the cylinder solely to said conducting means and move the piston towards its adjacent position to expel the headspace gas therefrom to the conducting means.

7. Apparatus for obtaining a sample of headspace gas confined under pressure in a closed container comprising:

- (1) a hollow piercing member,
 (2) a support for the container adapted to position the headspace of the container adjacent said member,
 (3) a sample cylinder having an inlet communicating with said piercing member,
 (4) a piston reciprocable in said cylinder,
 (5) a valve operable to open and close communication between said piercing member and said cylinder inlet,
 (6) means for selectively supplying a purging gas under pressure to said cylinder for moving said piston away from said inlet and for conducting gas expelled therefrom by said piston while said valve is closed,
 (7) means for moving said piston towards said inlet to thereby expel the purging gas from the cylinder via said inlet and said piercing member while said valve is open to the space between said piercing member and the top of the container,
 (8) and means for moving the container and said piercing member relative to one another until said member pierces the container and projects into the headspace thereof to thereby admit headspace gas via the piercing member to said cylinder while said valve is open, said piston being moved away from said inlet in response to headspace gas filling the cylinder,

said piston moving means being operable to again move said piston to expel the headspace gas from the cylinder to said expelled gas conducting means while said valve is closed.

8. Apparatus for sampling and analyzing gas in the headspace of a closed container comprising a piercing needle, means for supporting the container with the headspace thereof adjacent said needle, means for moving the container support and needle relative to one another until said needle pierces the container and communicates with the headspace, a piston-cylinder unit communicating with said needle, means for supplying a carrier gas to the cylinder to effect movement of the piston on a cylinder-filling stroke, means for closing cylinder-needle communication

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when the cylinder is being filled with the carrier gas, means for actuating said piston on a cylinder-emptying stroke, means for shutting off said supply means and opening cylinder-needle communication during said cylinder-emptying stroke for expelling carrier gas from the cylinder through the needle, means for causing said piston-actuating means to move said piston on another cylinder-emptying stroke after the cylinder has been filled with gas from the container headspace, means for closing cylinder-needle communication during said last-mentioned stroke and a gas analyzing device operably connected to said cylinder for receiving and analyzing headspace gas expelled therefrom on said last-mentioned stroke.

9. Apparatus for sampling and analyzing gas in the headspace of a closed container comprising a piercing needle, means for supporting the container with the headspace thereof adjacent said needle, means for moving the container support and needle relative to one another until said needle pierces the container and communicates with the headspace, a piston-cylinder unit communicating with said needle, means for supplying a carrier gas to the cylinder to effect movement of the piston on a cylinder-filling stroke, means for closing cylinder-needle communication when the cylinder is being filled with the carrier gas, means for actuating said piston on a cylinder-emptying stroke, means for shutting off said supply means and opening cylinder-needle communication during said cylinder-emptying stroke so that carrier gas is expelled from the cylinder through the needle, means for causing said piston-actuating means to move said piston on another cylinder-emptying stroke after the cylinder has been filled with gas from the container headspace, means for closing cylinder-needle communication during said last-mentioned stroke, a gas outlet conduit connected to said cylinder for receiving headspace gas expelled therefrom on said last-mentioned stroke and means connected to said outlet conduit in the flow path of the headspace gas expelled from said cylinder for trapping a portion of the expelled gas during flow thereof through the outlet conduit.

10. A method of operating a gas sampling device for sampling gas confined under super-atmospheric pressure in the headspace of a closed container wherein the device has a piercing needle communicating via a control valve with a variable volume sampling chamber, comprising the steps of:

- (1) supporting the container with its headspace adjacent the needle,
 (2) introducing purging gas into the chamber while increasing the chamber volume so that the purging gas fills the same,
 (3) reducing chamber volume to expel the purging gas therefrom through the needle while the valve is open to thereby purge the device and envelop the space between the needle and container with the purging gas,
 (4) causing the needle to pierce the container and project into the headspace thereof while the space between the needle and container is enveloped with the purging gas,
 (5) permitting the chamber to increase in volume in response to headspace gas flowing thereinto via the needle while the valve is open, and
 (6) closing said valve to trap the headspace gas in the chamber for subsequent transfer to a gas analyzing device.

11. Apparatus for sampling gas confined in the headspace of a closed container including in combination a piercing needle, a variable volume sampling chamber communicating with said needle, means for controlling the volume of said chamber, valve means controlling communication between said chamber and needle, supply means for supplying purging gas into said chamber and cooperable with said chamber volume control means and said valve means to increase chamber volume as the purging gas fills the same and thereafter expel the purging gas

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from the chamber through the needle to thereby purge the chamber, the valve means and the needle, and means for moving said needle relative to the container synchronized with said chamber volume control means and said valve means for causing the expelled purging gas to envelop the piercing area of the container and while so enveloped causing the needle to pierce the container and project into the headspace thereof, said chamber volume control means thereupon being cooperable with said valve means and said supply means to increase chamber volume to fill the same with headspace gas flowing thereinto via the needle and to thereafter reduce chamber volume to thereby expel the headspace gas from the chamber for analysis.

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