Pressure Changes in Bottles during Sterilization by Autoclaving

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Abstract □ A method is described to measure changes in pressure inside a bottle during autoclaving without disturbing the integrity of the seal. Experiments showed that, in a perfectly sealed bottle, the pressure rose to a maximum of 58.2 psi absolute. If the rubber liner was unable to maintain the seal, air leaked slowly from the bottle both during the heating-up and the early sterilization period.

Keyphrases □ Sterilization, autoclaving—pressure changes in bottles, method for measurement □ Bottles, sealed—pressure changes during sterilization by autoclaving, method for measurement □ Seals, bottles—effects of sterilization by autoclaving, pressure changes in bottles measured

The use of screw-capped bottles as containers for parenteral fluids and noninjectable water has recently been criticized (1). The protection against microbial contamination offered by such systems to sterile fluids has been shown to be inadequate (2). One serious fault with many rubber seals is their inability to prevent air from escaping from inside the bottles during autoclaving (3, 4). The presence of air above the liquid contents of the bottle ensures that the pressure within that bottle will be greater than the pressure in the autoclave during sterilization.

The objective of this study was to measure changes in pressure inside a typical screw-capped bottle during sterilization when the container remained hermetically sealed and also when an imperfect bottle seal allowed air to escape. The standard 1-liter noninjectable water bottle[1] was used as the test system. A bottle was modified so that pressure changes inside such a container could be measured during autoclaving. The highest pressure readings obtained were compared with calculated maximum values.

THEORETICAL

The theoretical maximum bottle pressure during autoclaving at 121° is calculated as follows. The pressure within a bottle is the sum of the partial pressures of air and steam. The pressure of steam at elevated temperatures is known, and the component due to expansion of the air in the bottle may be calculated according to the ideal gas equation where (PV/T) = a constant. Then V (the head-space volume of the bottle) is reduced at elevated temperatures by the expansion of water in the bottle. By comparing the density of water at 20° (0.9983 g/cm^3) and 121° (0.9421 g/cm^3) (5), the increase in volume over this range is:

$$\left(\frac{0.9981}{0.9421} - 1\right)10^2 = 5.97\% \qquad \text{(Eq. 1)}$$

Therefore, the expansion of 1 liter of water will reduce the head-space volume in a bottle at 121° by 59.7 ml. Conversely, the expansion of the glass will result in a potential increase in volume, estimated to be about 1.0 ml, for the 1-liter soda-lime glass bottle[1] (linear coefficient of expansion of glass = 9.8×10^{-6}).

Other factors to be considered include the air content of the water placed in the bottle. If water at 20° is air saturated at a pressure of 1 atm, it contains 18.7 ml air/liter (water for injection rapidly becomes resaturated after distillation). At 121°, virtually all of this air would be expelled, adding to the pressure produced in the head space. Also, a small volume of the air in the bottle head space will be displaced by water vapor, equal to 4.4 ml at 20° (saturation vapor pressure of water at 20° is 175 mm Hg). Therefore, under these conditions, in a perfectly sealed noninjectable water bottle[1], the internal pressure (IP) at 121° will be (assuming standard atmospheric pressure conditions):

$$\text{IP} = \left[\frac{14.7\{(220.0 + 18.7) - 4.4\}394}{293(221.0 - 59.7)}\right] + 14.7 + 15.0 = 58.4 \text{ psi (absolute)} \qquad \text{(Eq. 2)}$$

EXPERIMENTAL

A hole, 1 cm in diameter, was drilled into the base of a 1-liter noninjectable water bottle. Into this was fixed a rubber tire valve casing, which was connected *via* a length of stainless steel reinforced polytetrafluoroethylene flexible tubing to an instrumentation port in the roof of the autoclave chamber. A pressure transducer[2] was sealed into the same instrument port on the outside of the autoclave chamber. Within the bottle, a length of glass tubing extended from the tire valve to the air space (Fig. 1).

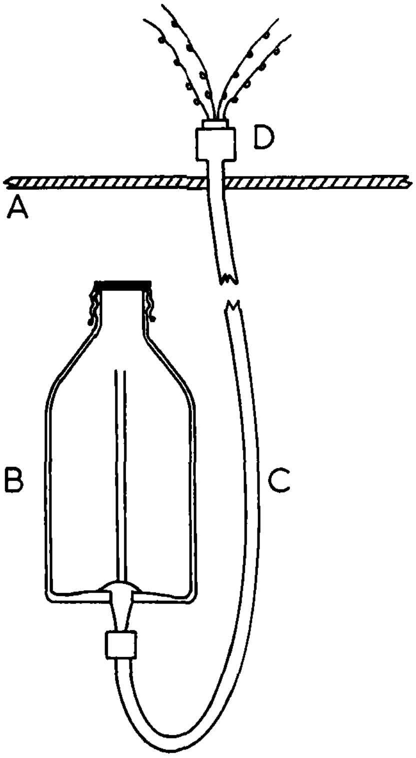

Figure 1—*Apparatus for measuring pressure changes inside the noninjectable water bottle during autoclaving. Key: A, roof of autoclave chamber; B, noninjectable water bottle; C, pressure measurement pipe; and D, pressure transducer.*

[1] Supplied by the U.K. Department of Health and Social Security.

[2] Type ITQS, Kulite Semiconductor Ltd., Basingstoke, United Kingdom.

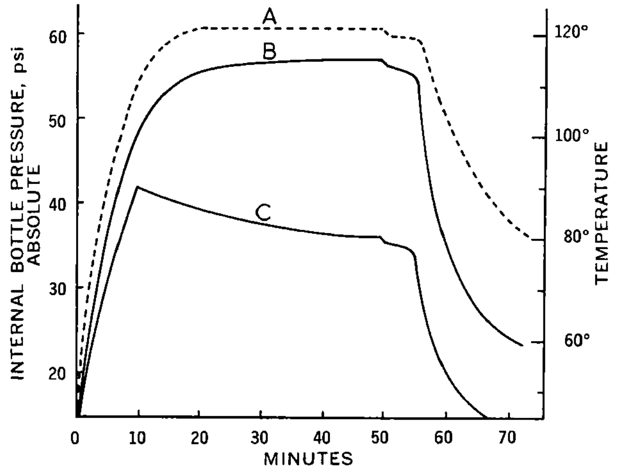

Figure 2—*Temperature and head-space pressure changes in noninjectable water bottles containing 1 liter of distilled water, sealed with flat rubber liners and autoclaved, at 121°. Key: A, changes in temperature; B, changes in pressure, perfectly sealed bottle; and C, changes in pressure, leaking bottle seal.*

Pressure changes were recorded on a potentiometric recorder; temperature measurements, also recorded, were made with a copper–constantan thermocouple in a second bottle placed alongside that adapted for pressure measurements in the autoclave chamber. The bottles were filled with 1 liter of distilled water at 20°. They were then autoclaved and spray cooled[3].

RESULTS AND DISCUSSION

Figure 2 shows temperature and pressure changes in a noninjectable water bottle, sealed with flat rubber liner and aluminum screw cap, autoclaved at 121°. Curve A shows the temperature profile during autoclaving within the bottle head space. Curve B shows the concomitant pressure changes in a well-sealed bottle. Allowing for the additional volume of air within the pressure measurement tubing and the volume of glass tubing in the bottle, the maximum theoretical pressure in the experimental system described in Fig. 1 would be 57.2 psi (absolute).

The pressure inside the bottle rose to a level close to the maximum theoretical value, attaining a pressure of 57.0 psi (absolute) at equilibrium (adjustments were made for day-to-day variations in atmospheric pressure). On cooling the bottle to 20° after autoclaving, the pressure was found to return to atmospheric pressure, indicating that no air had been leaked from the bottle head space during the sterilization procedure.

These findings suggest that the apparatus described is suitable for detecting pressure changes within glass containers during autoclaving. It is essential that the tubing employed to connect the bottle to the pressure detection device is temperature and pressure resistant. It is also important to ensure that the pressure transducer is resistant to steam and responds linearly over the range of temperature up to 121°.

Clearly, the closure of a bottle is placed under considerable strain during autoclaving. Where the seal was not maintained, the pressure increased until a point was reached beyond which it gradually fell again due to leakage of air from the bottle (curve C). On cooling to 20°, such bottles were always observed to contain a vacuum. Results, therefore, show that air may escape from the bottle during the heating-up phase of the sterilization cycle.

The air space increases rapidly in temperature, resulting in an initial rapid increase in pressure, followed by expansion and evaporation of water. If the seal is imperfect, then at some stage during the heating process, the rubber liner is lifted sufficiently from its seating on the rim of the bottle to allow air to escape. This leakage then continues slowly as the temperature of the bottle and its contents rise. This is probably aided by the expansion of the aluminum cap. As soon as the spray cooling commences, steam in the head space condenses and the rubber liner is sucked back onto the rim of the bottle to reform the seal. When cool, the bottle will consequently contain a vacuum. Results reported elsewhere (3) have shown that, in the majority of cases of the seal leaking, up to 90% of the air originally present is lost from the bottle head space.

[3] Getinge model SAR 450, Getinge A.G., Uppsala, Sweden.

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