THE APPLICATION OF STEAM QUALITY TEST LIMITS

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Steam quality testing, once the sole preserve of the British National Health Service is being adopted within the pharmaceutical industry on an increasing scale. The perceived need for such tests varies from company to company and country to country. The purpose of this paper is to relate the impact of poor quality steam to the pharmaceutical sterilization processes and consider the validity of the test frequencies and limits generally applied.

Background

It is widely accepted that the original source for the test limits came from the British National Health Service with the first references appearing in HTM 10[1] (subsequently superseded by HTM 2010[2]). In addition to their adoption by European standards, the same limits may be found in ISO 11134[3]. At the outset, the limits were established pragmatically and further information may be found in The Derivation of United Kingdom Physical Steam Quality Test Limits[4].

While HTM 2010, ISO 11134 and EN 285[5] contain the same limits, no clear guidance is provided on the location of the sample points or interpretation of the results. In the case of both dryness value and superheat tests this is an important factor. Guidance is provided later in the paper of the assumptions made, but not stated in these standards, which will allow results to be correctly interpreted for specific sample point locations.

Application

Steam quality test limits should only be applied to the porous load or equipment sterilization process and not terminal bottled fluid sterilization or steam/sterilization in place applications. Explanations for this approach will be found later in the paper.

Introduction

Steam for sterilization requires a number of attributes in order to be an effective sterilant. Steam provides the moisture that allows the coagulation of cell wall proteins and supplies the energy that heats the components and maintains their temperature, the combination of temperature and moisture resulting in sterilization. The higher the temperature, the shorter the sterilization time required. Where steam comes into intimate contact with components that are either medical devices or that will come into contact with parenteral products, it should not add chemical or endotoxin contamination.

This paper will concentrate on the engineering aspects and intends to provide the reader with an appreciation of the potentially complex nature of what is often assumed to be a simple heating process.

Dryness Fraction/Value

The dryness fraction of steam is the measure of the moisture carried within steam. A measured value of 0 denotes 100% water and the value of 1 represents dry saturated steam, that is to say steam as a vapour having no entrained water. Therefore steam with a dryness fraction of 0.95 will be a mixture of 95% dry saturated steam and 5% water.

The dryness fraction of steam is inextricably linked with the latent heat that it possesses. Steam having an energy level equal to 50% of the latent heat for its saturation pressure will have a dryness fraction of 0.5 indicating a 50:50 water/steam mixture. Therefore, only when steam has its full quotient of latent heat will it be dry saturated and have a dryness fraction of 1.

Measurement of dryness value

By using simple calorimetry, the energy that steam possesses at a particular pressure may be measured and therefore its dryness assessed. When we use the methods described in EN 285 to measure the latent heat there is a significant error present. The steam sample is taken from the centre of the sterilizer steam supply pipe and as such takes no account of moisture present either as a film on the pipe wall or any condensate at the bottom of the steam pipe. As a result the calculated latent heat and therefore dryness fraction will not be accurate but be an approximation. It is for this reason that the calculations quote results in terms of dryness values and not dryness fractions. EN 285 states - *The test method described should be regarded not as measuring the true content of moisture in the steam, but as a method by which the provision of acceptable quality steam can be demonstrated.*

While more accurate results may be obtained, the linkage between these and the pragmatically established limits will have been lost, while at the same time increasing the scale, complexity and therefore the disruptive aspects of the test.

The impact of wet steam

At the end of a sterilization cycle, packaging materials should be sufficiently dry to maintain their sterile barrier properties (EN 554 - A.2.5). The correct steam quality combined with proper loading and packing techniques should ensure that dry loads are consistently achieved.

Because the heating process within a sterilizer inevitably results in condensation being generated, it is easily assumed that variations in the dryness value of the steam to be inconsequential. This indicates a failure to appreciate the fundamentals of the process and in particular misunderstands how the drying process works.

The drying of loads

When we heat a component with steam, it condenses and gives up its latent heat. If all the water associated with this heating process were to somehow remain on the component,

when the chamber is under drying vacuum conditions the boiling point is reduced (to 32.9^{o} C at 50 mBarsA). A reduction in pressure from 3 BarA to 50 mBar (shown as (a) in Figure 1) will result in the water on the component having an excess of sensible heat at the lower pressure of 561 – 138 = 423 kJ/kg. This excess energy is sufficient to cause only some 15% of the water to be turned to steam. The balance of the energy required to evaporate the remaining 85% of the condensate must come from the only available source, the component. That is to say that the energy used to heat a component is also used to dry it.

FIGURE 1

Should surplus condensate settle on the component either as a result of water entrained in steam or by condensate dripping from another component, it may not dry. In practice, as steam condenses, much of the water generated will drain by gravity (provided the load is correctly loaded and configured) and reduce the need for the latent heat contribution from the component. In this example any component having more than 15% of the condensate present at the end of the sterilizing stage that is needed to heat it, will be much slower to dry. The drying time will be dependent upon the location of the condensate, its surface contact area and the specific heat of the component. That is to say large quantities of condensate in contact with a small surface area of a component will be slow to evaporate and that insulators will dry more slowly than good conductors of heat.

This brief explanation serves to explain why if a wet component is loaded into a sterilizer, at the end of the process it will remain wet. A reduction in pressure alone is insufficient to dry components.

It will be seen that the impact of wet steam will depend upon specific components and their loading methods. The dryness values quoted in EN 285 are > 0.9 for porous loads and > 0.95 for metal loads. The higher quality requirement being for loads typically used in the pharmaceutical industry.

Water as an insulator

In addition to the risk of wet loads, water can act as an insulator and prevent good heat transfer to component surfaces causing locations that are slow to heat. A static film of water 1-mm thick is equivalent to a layer of copper 500 - 600 mm thick. Obviously the effects of turbulence and convection reduce the impact, but in any event standing water should be avoided by correct loading techniques.

Sample point location

It should be noted that in EN 285 the measurement point for the dryness value test is not defined. If we use the combination of HTM 2010 and standard UK hospital design practices for guidance, it will be seen that the sample point is assumed to be located as shown in Figure 2. This assumes a further 2 Bar pressure drop will occur after the sample

point, before the steam enters the chamber and that a steam separator will be fitted to the sterilizer. Given that a pressure drop will tend to improve the dryness of steam, these design aspects must be taken into consideration when conducting dryness value tests and interpreting the results. The ability to maintain the limits in the standards need not be a guarantee of dry loads and should be used as guidance and corrected if the conditions at the sample point differ from that shown in Figure 2.

FIGURE 2

Pragmatically, if loads are not wet, the steam has a sufficiently high dryness value, though ideally, the steam should be in a dry saturated condition when it enters the sterilizer as to present the load with the absolute minimum entrained water possible and the maximum amount of available energy. While the reduced energy content of wet steam will have an impact on the heating effect, this is likely to be minimal.

Where a clean steam generator is utilised having a well engineered separator and is controlled to prevent moisture and therefore endotoxin carryover, under all demand conditions, it will produce steam that is dry saturated. Its condition can only deteriorate within the steam distribution system as a result of heat loss causing condensation (unless excessive pressure drops are present, which will tend to dry the steam). Good pipeline design, insulation and trapping practices combined with the use of steam separators should ensure the condition of steam is maintained at the point of use.

Component design has a large part to play in the elimination of wet loads as components must be capable of being drained and this is a fundamental aspect of the purchasing/validation process for a sterilizer. Components should be assessed to ensure that they are capable of self-draining, if not, they should be modified.

Sterilizer jackets at reduced temperatures

Operating a sterilizer with its jacket at a colder temperature than the sterilizing temperature is often cited as good practice in the USA to prevent/eliminate the threat of superheat. While superheat will be dealt with later in this paper, it is self evident that this will result in extraneous water dripping onto the load, having the same effect as wet steam. Given that wet loads may be a greater threat to sterility than superheat (where metal loads are concerned), this practice is not recommended.

Dryness value and steam/sterilize in place

Where steam is used in steam/sterilize in place applications, the dryness value (and any superheat) will have little or no impact on the efficacy of the process. Pipes and/or vessels will often not be insulated and large quantities of condensate will be generated in any event. Unlike a variety of components offered for processing within a sterilizer, process vessels and pipework will have been designed specifically to self-drain. Provided the combination of moisture and temperature are present, sterilization will occur. This is

not to suggest that well designed and engineered steam systems are not required for such systems, but that point of use testing is not necessary.

Superheat

Superheated steam is steam that is at an elevated temperature for its saturation pressure. It cannot be generated at source by a conventional clean steam generator, as energy would have to be applied to the steam once it was in a dry saturated condition. Superheated steam is usually generated as the result of pressure drops through either pressure reducing valves or orifices. The impact of the pressure drop is to modify the pressure of the steam while its energy content remains the same. The excess of energy for the pressure present will result in any excess moisture turning to steam. If the steam is already dry saturated or if excess energy is still present after turning what moisture is present to steam, an increase in temperature will be evident. Once steam is dry saturated only a small amount of energy is required to create high temperatures as will be seen in Figure 1. Table 1 below provides some examples of the effects of a pressure reduction from 5 BarA on the dryness fraction.

TABLE 1

Risks of superheat

The risk to the sterilization process of superheated steam is that the steam will not condense and provide moisture until the steam temperature has reduced to the saturation temperature. Until this occurs, the steam will act as hot air and at the temperatures present will have little or no sterilizing effect. The excessive temperatures generated can result in damage to both components and packaging.

The rate at which superheat will decay is dependant upon the nature of the load and will be present for longer where loads have a low heat capacity. The worst case condition usually experienced is where small quantities of batch documents are processed and will have a greater impact where short sterilizing times are utilised. While the pharmaceutical industry generally uses sterilization cycles at 121^{o} C for greater than 15 minutes, hospitals generally use 134^{o} C/3 minute cycles, where there is a very short time for the heat exchange and therefore loss of superheat to occur. The risk cause by superheat is therefore greater under these conditions and can pose a very real threat.

Superheat & Water

It is often assumed that superheat and the presence of condensate are mutually exclusive. Indeed, in the description above it is indicated that the impact of surplus energy is to evaporate excess moisture. What must be considered however is that the process takes a finite time and it is perfectly possible for the two conditions to co-exist. In this respect, the concept may be considered to be similar to the presence of ice at room temperature.

While our thermodynamic tables indicate that ice cannot exist under these conditions, it plainly does until the heat exchange process is completed.

Pressure drop ratio

To reduce the impact of excessive pressure drops EN 285 indicates the need to have pressure drops not exceeding a ratio of 2:1. Another important aspect is the length of pipe between pressure drops, which will allow superheat to decay through the evaporation of excess moisture and conduction through the pipe wall. In practice, the pressure drop ratio may be exceeded, provided the drop occurs sufficiently far away from the sterilizer.

Sample point location

The superheat test in EN 285 is designed to measure the condition of the steam between the 4 and 2 BarG pressure drops as shown in Figure 1. It is an inferential test and seeks to establish if the result will cause more or less than 5^{o} C of superheat in the chamber, following a separator and a further pressure drop of 2 Bar. Once more, this important assumption is not stated in EN 285 and as with the dryness value test, care needs to be taken with the location of the sample point and the interpretation of the results obtained.

The small load test

Even where the pipework design and steam quality measurements are satisfactory, superheat may still be evident in the chamber. This is typically measured during the course of a small load test where a thermocouple is placed 50 mm above a standard linen test pack. The test requirement for superheat is that the temperature measured by this thermocouple at the start of the sterilizing stage should not exceed the control temperature by more than 5^{o} C and should reduce to less than 2^{o} C in one minute. Given that the small load test directly measures the condition in the chamber and is easily carried out at frequencies greater than that recommended for the steam quality tests, its use is suggested by the author as an effective alternative. Most out of specification results tend to be marginal failures, though very occasionally persistent superheat will be encountered. This may be the result of large pressure drops from local steam generators and is usually evident to a greater extent on small chambers. While usually only detected by thermocouples within the chamber, it is possible that the extent of superheat is sufficient to affect the chamber temperature sensor. Under these conditions, the results are often misinterpreted as a temperature control problem.

Overheat

Where superheat is measured even where the pressure drop ratios and superheat tests are satisfactory, the cause may be due to a phenomenon known as overheat and is due to the velocity of the steam entering the chamber. The process may be considered similar water hammer. In the case of steam, the flow is brought to an abrupt halt as it enters a comparatively small chamber. Instead of the mechanical action with the water pipe, the

effect of the kinetic energy is to increase the temperature. Provided the pipework design and steam quality entering the chamber is satisfactory, the phenomenon indicates a design problem with the steam port sizing. The calculation for overheat may be simplified to: -
Overheat = $V^2/2C_p$

Where V is the velocity of the steam in m/s and C_p is the specific heat at constant pressure (@ 1900 at 2 BarG)

It will be seen that a steam velocity of 200 m/s will result in an excess of temperature of 10.5^o C.

Overheat is not seen to pose the same threat to the process as superheat, as it automatically reduces with the steam flow, as the load is heated to the sterilizing temperature. As with genuine superheat and exothermic superheat, detailed below, the phenomena can result in damage to components and packaging. Devices and baffles fitted to the steam entry ports can effectively reduce the steam velocity and sometimes eliminate overheat.

Exothermic superheat and gassing

Another cause of the superheat phenomena is due to an exothermic reaction and occurs where condensing steam rehydrates previously dehydrated materials with a resulting increase in temperature. This phenomenon is evident where paper products are processed, wipes, sponges, mop heads etc. or filters that have been dried in a cabinet. It is probable that the products themselves will be sterilized, as the phenomenon can only exist when they have been rehydrated.

A similar phenomenon known as gassing can cause elevated temperatures to be recorded due to the reaction between steam and certain adhesives, linen which has been laundered with certain detergents/conditioners, filters that have traces of IPA on them etc. EN 285 requires of packaging material - .. *in the case of wrapped goods and porous loads where a saturated steam environment is required, they should not generate gases which could restrict the removal of air and penetration of steam.*

Non Condensable Gases

Non-condensable gases are gases liberated by steam when it condenses. The source of such gases is usually from the steam generator feedwater and the impact of such gases is that they modify the steam from being pure water vapour to a mixture of steam and gas and are therefore an unwanted contaminant.

If we consider the heating of a single component by steam it will be seen that as steam condenses on the item its volume will reduce by a factor to 1/841 of the original value (for steam at 122^o C). This rapid reduction in volume causes a low-pressure area, which in turn is refilled with more steam. Until the product is heated, this process will continue.

It will be seen that the flow of steam is always towards the component. As any gases are liberated at the point of condensation, it will be seen that any gases present in the steam will be forced by the flow of steam towards the product. If the component is hollow or porous, any liberated gases will be forced to the centre. There is little difference in the specific mass of air and steam at the sterilization temperature and therefor no real gravitational effect to cause the air to leave the component. Air is approximately 12,000 times more resistant to heat transfer than copper and whether present as a film or a pocket, may prevent direct steam contact or insulate the component. Such conditions are identical to inadequate air removal, where very small quantities of air remain.

To what extent are non-condensable gases an issue? The limit in EN285 is 3.5% and is expressed in terms of ml of gas collected per 100 ml of condensate. This is often misinterpreted to be 3.5% by volume. In practice the percentage by volume is 3.5ml/169.4 l = 0.002066% (20.66 ml per kg of steam). When homogeneously mixed with steam, such levels are unlikely to have any impact, as the molecules are so widely dispersed as not to prevent the sterilization of simple surfaces. This would be the case for a fluid sterilization process, for example.

While of apparently minimal consequence, such quantities of gases are capable of having an impact on the equipment sterilization process. If one calculates the residual air remaining after an effective air removal stage, as shown below in Figure 3, it will be seen that an air removal stage having 6 pulses between 200 and 2000 mBarA, will have some 20 ml of residual air per 1 m^3 of chamber volume. If we fill a 1 m^3 chamber with steam it will require 1000/846.1 = 1.18 kg of steam. If the steam has the amount of non-condensable gases allowed by the limit we shall add a further 24.4 ml of gases to the to the process, effectively doubling the desired value. At twice or three times the limit, where some impact is often evident on Bowie Dick test, it will be seen that the effectiveness of the air removal stage will be considerably reduced. While the volume of the limit is very small, it is put into context by the amount of residual air at the end of the air removal stage. The calculation ignores any cumulative effect of the gases liberated during the pulsing and the real impact is likely to be even greater than the simple calculation assumes.

The use of simple calculations and the modification of an air removal stage will allow cause and effect to be easily demonstrated using thermocouples and equilibration time, chemical indicators and biological indicators, all of which will be adversely affected.

FIGURE 3

The design of the component, its mass and the amount of non-condensable gases in the steam will determine the extent of any problem and the limit in EN 285/HTM 2010 should be easily satisfied by well designed and installed generation plant. As with both the dryness value and superheat tests EN 285 applies a caveat - *The test method described should be regarded not as measuring the exact level of non-condensable gases but a method by which the provision of acceptable quality steam can be demonstrated.* Unlike

the former tests, the location of the sample point with respect upstream and downstream pressures will not have an impact on the non-condensable gas test result.

Air vents

While air vents are particularly successful in removing large quantities of air when systems are started up, they are less effective in dealing with air that tends to be homogeneously mixed with the steam. Air will collect and may be removed by vents under no/low flow conditions, but when travelling at speeds of 25 m/s (90 km/hour) or faster, gases are more likely to be carried to the point of use than removed.

Non-condensable gases may be evident either as continuous levels or intermittently and this will be the result of feed pump operation etc.

Clean steam versus utility steam generators

While the perception in the industry is that we design and construct high quality steam generation and distribution systems, this aspect is often restricted to the measures we take to eliminate chemical contamination and endotoxins. In fact the presence of high levels of such gases in clean steam systems is more likely than in plant or utility steam systems for the following reasons: -

1. Feedwater for clean steam generators is rarely heated and the solubility of gas in water reduces with increased temperature. While the water may be heated as an energy conservation measure immediately before it enters the generator, no means of eliminating the gas will be evident on most standard systems. By contrast, a well designed utility steam system uses a large proportion of condensate return for its feedwater which will naturally be hot and is often heated, where necessary, to maintain temperatures in excess of 80^{o} C.

2. The gases are liberated at the point steam condenses. Where this occurs in a steam main, the low mass gases will be carried with the flow of steam and need not necessarily leave the system by means of steam traps. The condensate return is therefore essentially deaerated and in utility steam will be reused to feed the boiler.

3. The impact of feedwater systems incorporating numerous stages, each with storage and recirculation systems result in water delivered to the clean steam generator being excessively aerated. This does not occur to the same extent with simpler systems on utility steam. Even where pre-heaters are used on clean steam systems, bubble size, convection currents and direction of water flow can all combine to prevent gases easily and quickly leaving the water.

4. Utility steam systems utilise chemicals such as hydrazine to scavenge oxygen from the water in order to minimise corrosion. Where corrosion is a major issue, as in the

case of high-pressure systems, water make up may be mechanically degassed and the feedwater deaerated.

5. Where sterilizers are supplied with utility steam, the distribution systems tend to be much larger and have plant demands much greater than from sterilizers. This results in large quantities of steam being used for space, water and process heating which ensures that the systems are continually being purged of non-condensable gases.

Steam/sterilize in place systems

Unless active air removal systems are utilised in steam/sterilize in place systems of an effectiveness equivalent to an equipment sterilizer, non-condensable gases are unlikely to have a measurable impact on the process. That is to say that the amount of residual air following free steaming, will probably exceed the amount of non-condensable gases present in steam by several orders of difference.

Other requirements

EN 285 requires that fluctuations in the supply pressure to the sterilizer should not exceed +/- 10% and that the rate of pressure change in the chamber should not exceed 10 Bar per minute, the latter requirement being to prevent damage to packaging.

Conclusions

It will have been seen that wet steam, superheated steam or non-condensable gases all have the potential to adversely affect the sterilization of equipment loads in the pharmaceutical industry. The extent to which the process is affected will be dependent on the extent of the problem and the nature of the load. Good practice indicates that we should be aware of the condition of the steam we use for the sterilization of equipment or porous loads, to both confirm that the design requirements of our steam raising plant have been satisfied and to assist with troubleshooting. While routine testing at an annual frequency at the point of use is indicated by HTM 2010, the period between tests may be inadequate to detect any transient or seasonal problems that exist. Furthermore, it is implicit in HTM 2010 that daily Bowie Dick tests are conducted and equipment is fitted with air detectors, both of which may under certain conditions detect non-condensable gases.

A suitable test frequency for the pharmaceutical industry will be determined a number of aspects: -

1. Extensive testing of the steam generator when initially validated. This will allow the demand conditions, under which poor quality steam can be produced, to be known in advance. Such conditions may then be avoided using appropriate controls.

2. A good understanding of the distribution system and how it performs under different demand conditions may allow sampling at certain locations to provide valid data for

more than one sterilizer. If the pressure drops in a system are known and monitored, superheat from this cause can be avoided.

3. The use of supporting data from Bowie Dick tests and the use of air detectors.

4. The use of a testing regime that will detect the presence of superheat (small load test or data from empty chamber mapping).

5. Procedures that provide high levels of assurance that wet loads will be detected by operators.

6. If a deaerator is fitted, a temperature logger or recorder can provide assurance that the feedwater temperature is maintained at a minimum temperature and can continuously monitor its performance. This could reduce the need for non-condensable gas tests.

The key to minimising the impact of testing is knowledge of both the cause and effects of poor steam quality. Unless a system has been designed to avoid the problems described, a more extensive testing regime is indicated. Properly designed and constructed steam generation plant and distribution systems should have no difficulties in meeting the pragmatically established limits.

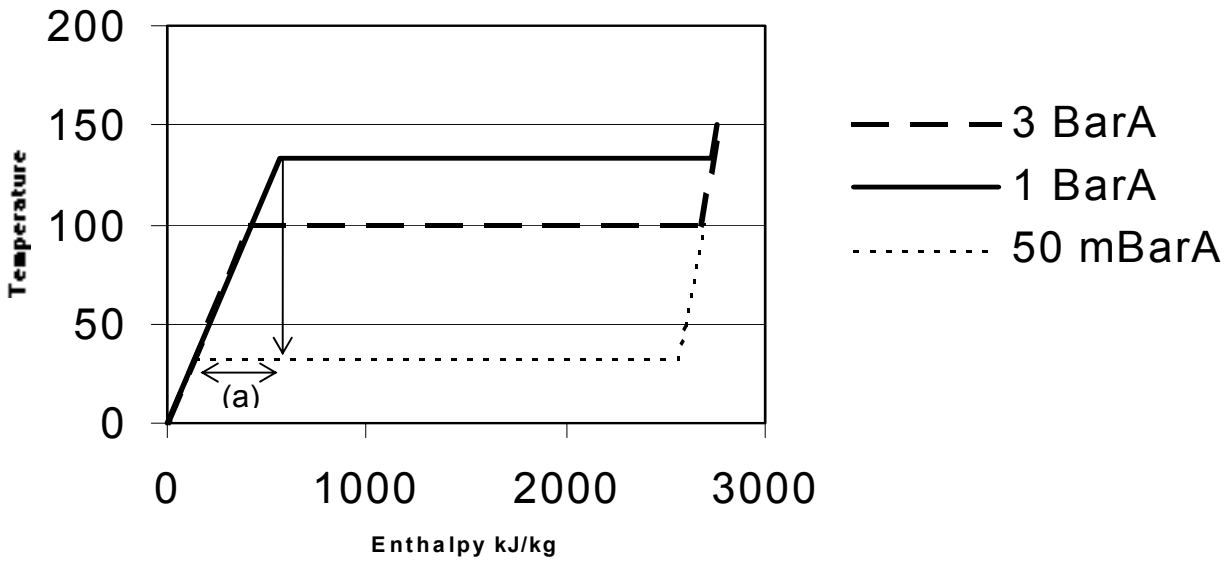
While the standard test methodologies would seem to be crude, they provide a linkage with pragmatically determined limits. Alternative methods are acceptable within EN 285, provided that they have been calibrated against the standard and should be used as and when companies feel necessary.

Care is needed with determining the location of sampling points and the interpretation of results for both dryness value and superheat tests. The EN 285 methodology makes a number of assumptions regarding the steam pressure at the sample point and within the chamber, but these are not stated.

References

1. Health Technical Memorandum 10 Sterilization (1980)
2. Health Technical Memorandum 2010 Sterilization (1994) – Part 3 Validation and Verification, Section 9 Steam quality tests.
3. ISO 11134: 1994 Sterilization of health care products -- Requirements for validation and routine control - Industrial moist heat sterilization.
4. Keith Shuttleworth. The Derivation of United Kingdom Physical Steam Quality Test Limits. PDA Letter - December 1999.
5. EN 285 Sterilization – Steam Sterilizers – Large Sterilizers (1996) - Section 24 Steam quality tests.

Figure 1
Enthalpy of Steam at Constant Pressure



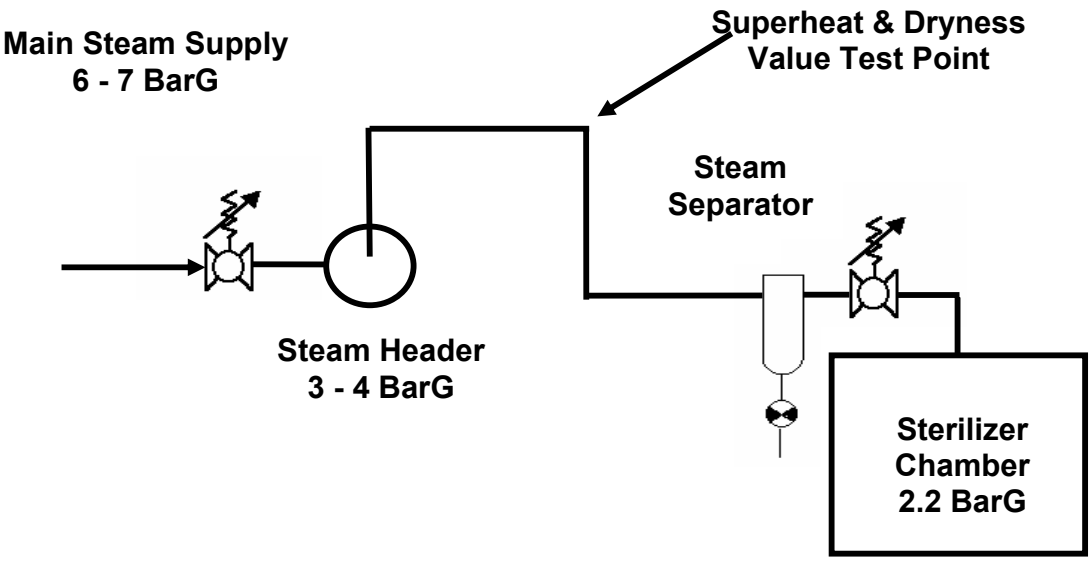
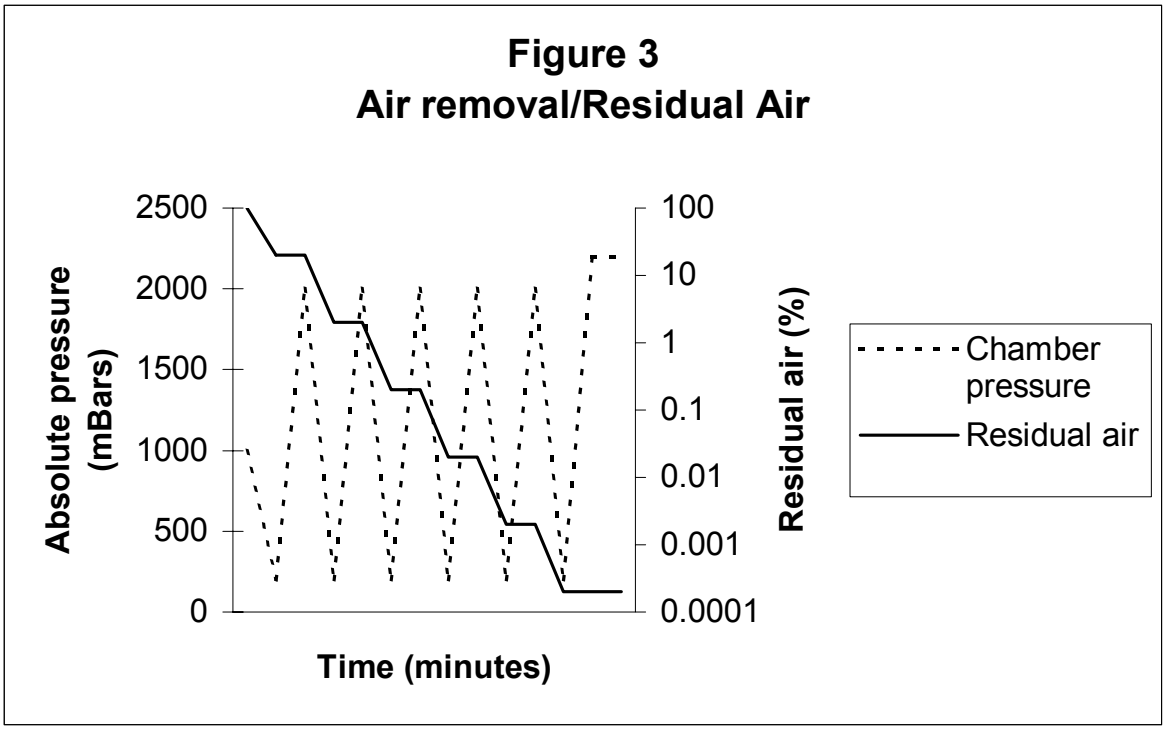


Figure 2
Implied steam supply pipework arrangement (HTM 2010)



Pressure/Dryness Fraction before pressure drop	Pressure/Dryness Fraction After pressure drop
5 BarA /0.95	3.2 BarA/0.96
5 Bar A/0.98	3.2 BarA/0.99
5 BarA/0.95	2.1 BarA/0.97
5 BarA/0.98	2.1 BarA/1.0
5 BarA/0.98	1.0 BarA/@ 10^{o} C of superheat

Table 1
Examples of pressure drops on dryness fraction