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HIGH VOLTAGE LEAK DETECTION (HVLD)

Introduction

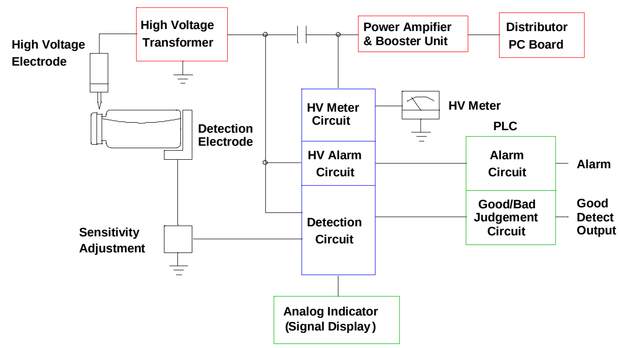
It is understood that no matter how carefully a product has been prepared, it would still be unsafe for human use if the container has a pinhole, a crack or a defective seal. Containers are commonly kept in an environment of varying temperature. When it is warm, the solution or product may expand and may leak from the container through a pinhole, a crack or defective seal, and when it is cold the product can shrink back into the container bringing with it such contaminants as bacteria from the exterior of the container, thus contaminating the product.

There are a variety of methods widely used in the detection of pinholes, cracks and defective seals in containers. These tests range from vacuum or pressure decay tests, to submerging the entire container into a dye solution under pressure and visually inspecting the container for the presents of dye or more advanced vision systems to inspect for certain defects in the container. Many of these tests are destructive causing the loss of good product and packaging components.

The High Voltage Leak Detection (HVLD) system is totally free from these shortcomings and has additional advantages for ensuring seal integrity. The most important advantages of the HVLD system is a non-destructive test method coupled with the assurance there is absolutely no secondary contamination to the product. Because of the detection principle, the HVLD system is capable of detecting pinholes not consistently found with other methods of inspection. Since the HVLD system is using the product in the container as a conductive path, small pinholes which would normally be closed by the product will be detected. This enables a more consistent and efficient way of detecting leaks while reducing the possibility of false rejection.

The HVLD system is designed for continuous 100 percent in-line inspection. There is no special chambers or pumps necessary so the integrity of the chamber seal is not an issue. The material handling is designed to accommodate the container being inspected at the required line speeds. Because the high voltage and machine parameters are adjustable, a variety of products and containers can be inspected on the same equipment with minimal change over time.

1. High Voltage System Block Diagram



The high frequency high voltage is generated in the control section which includes the Distributor PC Board, Power Amplifier and High Voltage Transformer. The high voltage is applied directly to the specific areas of the container through the Inspection Electrode. The current flow through the container and product is collected at the detection electrode. The current volume is regulated by the Sensitivity Potentiometer. The setting of the Sensitivity Adjustment determines the signal amplitude to the detection circuit. At the Detection Circuit, the signal is converted to a DC voltage whereby the judgment of good or bad is determined. The converted signal is then displayed on the Analog Indicator or Digital Signal display.

The PLC is used to activate alarm signals and other output signals for general operation.

2. **Principle of Inspection**

2-1 The High Voltage Leak Detection (HVLD) is based on the principle of a high voltage spark-test system and is capable of handling any product as long as the container is made of electrically insulated material such as glass, rubber, plastic or plastic film, and contains an electrically conductive solution. It can also be used for glass or plastic containers with an aluminum cap as long as an insulating material such as a rubber stopper or liner between the product and the aluminum cap is present.

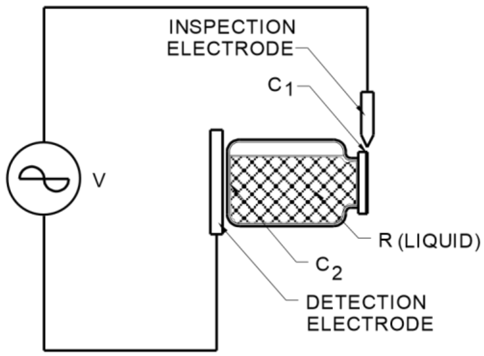


Fig. 1

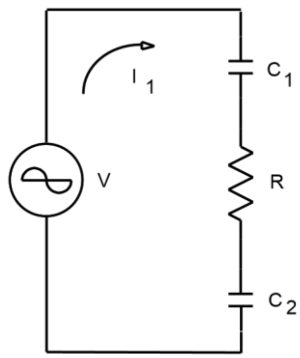


Fig. 2

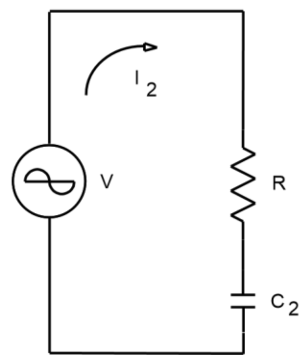


Fig. 3

V = High voltage source
R = Electric resistance of product
C_1 = Volume of static electricity between the inspection electrode and the product
C2 = Volume of static electricity between the ground electrode and the product
I1 = Electric current which is produced when the container is sealed
I2 = Electric current which is produced when the container seal is defective

High frequency high voltage (V) is applied to a specific area of the hermetically sealed container made of a non-conductive material. The high voltage is generated in the control section and boosted up in the high voltage transformer and applied to the inspection electrode. If the container should have a leak at the seal area, a discharge current will flow through the pinhole, crack or defective seal into the container. A signal is sent to the inspection circuit through the detection electrode. Detecting the change in this current volume enables the presence of a defect to be recognized.

2-2 Fig. 1 is the electrical equivalent circuit of Fig. 2 and Fig. 3. In Fig. 2, the electrical circuit describes a good container while Fig. 3 describes the circuit of a defective container. It should be noted that C1 and C2 work as electrostatic capacitors whereby making the container and the cap liner an insulator while R is the specific resistance of the contents of the container.

The formula for a good container is as follows:

$$I1 = \frac{V}{R + \frac{1}{2\pi f C1} + \frac{1}{2\pi f C2}}$$

When a leak is present, there will be an electrical discharge into the container. The formula for a defective container is as follows:

$$I2 = \frac{V}{R + \frac{1}{2\pi f C2}}$$

f = Frequency of high voltage

C_1 = Volume of static electricity between the inspection electrode and the product $C2$ = Volume of static electricity between the ground electrode and the product

2-3 With a constant voltage being applied to the container, a defective container will have a larger electric current volume (I2) than a container with no leak present (I1). The difference of the electric current volume determines whether the seal is defective.

$$I = I2 - I1$$

The picture illustrates a defective vial with a crack present in the shoulder area. The high voltage being applied is seeking the least resistive path to the product. In this case the high voltage sparks directly to the crack in the shoulder area of the vial. The increase in the current volume through the product determines the whether the container is defective. If a crack, missing stopper or other defect is present under the aluminum cap, the spark would contact the aluminum cap directly.

Defective Vial, Shoulder Crack

The electrical conductivity (R) of every product will vary due to the chemical makeup. The high voltage value (V) must be set so that there is no damage to the insulating materials of the container (C1).

3. Detection Performance Capability

3-1. The HVLD system is extremely sensitive and detects pinholes and cracks of such small size that it is not humanly possible to bore a hole which is undetectable by this equipment. Therefore, to test the capability of this equipment, samples with the smallest holes were selected from among the containers found defective by other leak detection methods. An electron microscopic was used to examine the samples revealing pinholes as small as 0.5 micron in diameter. The HVLD system is capable of detecting holes of that size consistently. Note, however, that the electron microscope actually views the hole at its surface and, in the depth of the hole, it can be much narrower as shown in Fig. 4. Because the detection takes place at the narrowest point of the pinhole, the HVLD system can detect holes considerably smaller than 0.5 micron in diameter.

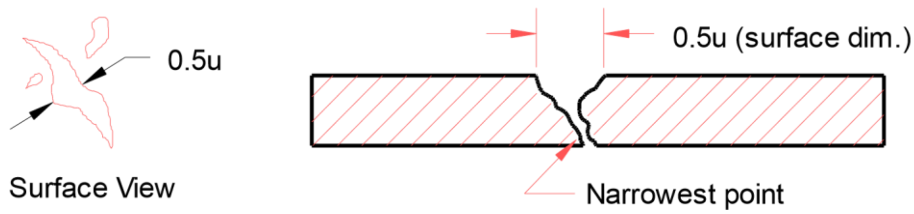


Fig. 4

3-2. Absolutely No Secondary Contamination

The effects in which the HVLD system may have on containers free of any pinholes, cracks or related factors are considered to be the following:

a. Time: Approximately 20msec (when 200 containers are processed per minute).

b. High tension electric field.

c. Corona discharge current.

d. Light purple rays due to corona discharge.

Defective containers with pinholes, cracks or other defects will be subjected to the following forces:

f. Minor discharge current.

g. Light rays of purple or orange in color due to discharge current.

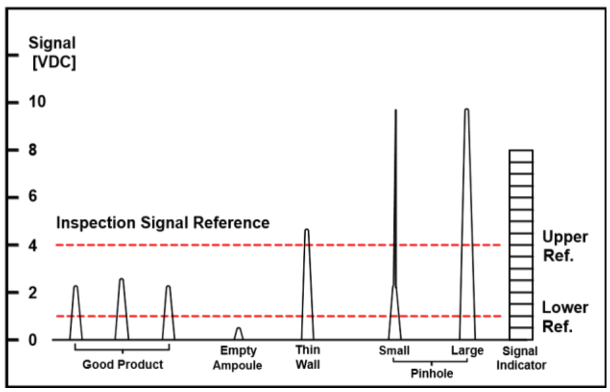
The effects are limited to those of electric current passing through the solution and those of light rays. Absorbance test, pH test, and composition analyses of the processed by HVLD system have revealed no harmful effects to the product from the high voltage being applied.

3-3. High-Speed Test on a Production Line is Possible (automatic operation)

Since an instantaneous, purely electrical test is sufficient for the leak detection, this method can be incorporated into the production line. The factor limiting the processing speed of the actual testing equipment is the physical size of the container being tested. The HVLD can be manufactured to operate at speeds equivalent to most packaging lines. Unlike the conventional leak detection methods of inspection, fully automatic operation is possible with use of this equipment.

4. Inspection Signal Reference Chart

The inspection signal values are adjusted by the amount of high voltage applied to the container and the sensitivity setting. These preset values are determined during the validation process. Although inspection signals generated may vary slightly from container to container, they will usually stay within the inspection signal range.

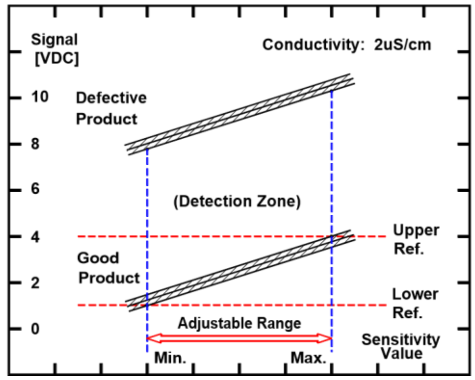


Good container signal values should be between the 1VDC and 4VDC range. An empty container will show little or no signal value therefore determined as faulty (not exceeding 1VDC Lower Reference value). As the material thickness in the container changes (thin wall), the inspection value may slightly exceed the Upper Reference value (4VDC). The container may not have a defect, but the inspection signal value is higher due to the higher electrical current flow through the thinner material. Small and large pinholes normally do not show a significant difference in signal values. Due to the strength of the high voltage volume, the larger pinhole will cause a wider or longer signal as shown in the chart. The inspection signal value is typically displayed on analog LED array or a digital display.

The inspection signal values will vary depending on the consistency of the wall thickness of the container, the conductivity of the product and high voltage and sensitivity settings. The high voltage and sensitivity settings should be determined for every product through the validation process. As the high voltage or sensitivity is increased or decreased, the inspection signal will also increase or decrease in value. These two parameters play an important role in determining inspection performance. A sensitivity value too low may result in not detecting a faulty product, whereby a sensitivity value too high may result in a high false rejection rate.

5. Sensitivity Comparison Chart

The data shown in the Sensitivity Comparison Chart was collected by inspecting the same good container and same faulty container five (5) times at increasing sensitivity values. The high voltage was set at 18KV and *not changed*. As shown on the chart, the Adjustable Range for the Sensitivity value was between 400 and 600. The sensitivity value is an arbitrary number set on a dial of a ten turn potentiometer used within the system for adjusting the input voltage to the detection circuit. The dial potentiometer values ranging from 0 to 999. As the sensitivity value (input value) is changed, the detection signal is also changed.



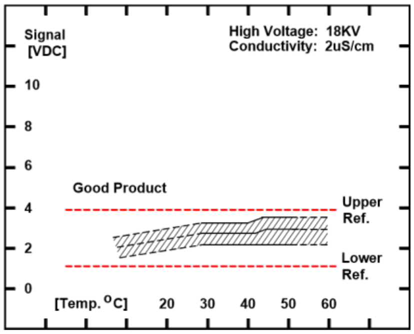
From the graph you can see as the sensitivity value is increased, so is the inspection signal value. At the minimum sensitivity setting of 400, the inspection signal is at the Lower Reference value. This would cause the good container to be rejected for a low signal value (under the Lower Ref.). This is considered a “false rejection” and the sensitivity value should be raised. At the maximum value of 600, the inspection signal value will exceed the Upper Reference value (4VDC) and will be rejected. This is also considered a “false reject”. The ideal setting would be somewhere between these two values. Please note, these containers where repeatedly tested five (5) times, each time the container was touched by a human hand with no significant changes in the inspection signal values. Therefore it is not necessary to wear gloves or special garments in a production environment.

When the same test is performed with a defective container (laser pinhole), the results are similar. As the sensitivity value is increased, the inspection signal value is also increased. As shown, the inspection signal is well above the Upper Reference value. This is considered the Detection Zone.

It is important during the validation process that the sensitivity value not be set so high that good products are falsely rejected, but it is as important to determine a sensitivity value high enough that when a defective container is inspected, the inspection signal will exceed the Upper Reference value. These parameter settings are the most important settings for consistent inspection performance.

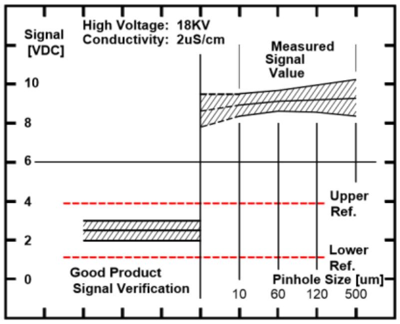
6. Temperature Comparison Chart

As shown in the Temperature Comparison Chart, the temperature of the container and liquid can change the inspection signal value. It is normally a slight change but may have to be considered during the initial determination of high voltage and sensitivity values. The temperature presents more of a factor with BFS containers than other container types. Because of this, it is necessary to determine when the inspection will take place before determining the inspection parameters.



7. Pinhole Signal Comparison

Part of the validation process will be to determine the detectablility of various size pinholes, cracks and seal defects. The Pinhole Signal Comparison Chart shows inspection signal values at a preset high voltage and sensitivity value of both good and faulty products. The inspection of good products will first provide the inspection signal information necessary to ensure that the high voltage and sensitivity values are within the Good Product range.

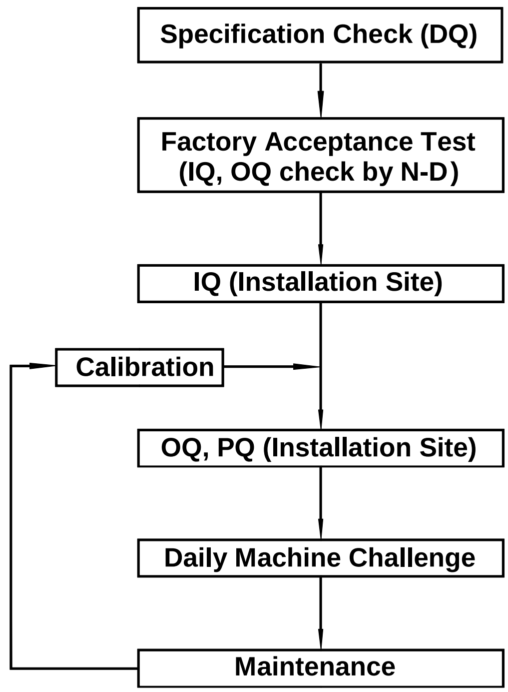


This is the Good Product Signal Verification. Containers with known defects inspected at these same parameter are then recorded. The various defects or pinholes at known sizes should produce an inspection signal value above the Upper Reference value (4VDC). As shown, the inspection signal values recorded indicate that the detectablility of defects due to pinhole size is only slightly different. Therefore, the high voltage system can not be used as a quantitative method of inspection for specific pinhole sizes. The high voltage inspection signal values are relatively the same for both a 10um holes verses a 500um hole. The high voltage system is saturated at approximately 9VDC ~10VDC.

This test procedure should be performed for every product. The difference in container material types and product conductivity will change the inspection signal values. Not all product configurations have the same dielectric values.

8. Validation Flow Chart

The Validation Flow Chart explains the required steps necessary for the validation of the Nikka Densok Pinhole Inspector. The validation starts with the machine design or Design Qualification (DQ). This would include machine specifications, number of inspection areas on the container, container and product evaluation. Once the machine is manufactured, the



1. Container Parameters
2. Speed Requirements
3. Product Evaluation

1. Machine Specification Check 2. Machine Performance Test
3. Inspection Parameter Setting

1. Specification Checklist
2. H. V. Setting
3. Sensitivity Setting
4. Inspection Signal Check
5. Rejection Check
6. Operation Function Check
7. Alarm Function Check

1. Performance Test (each product)

1. Daily Performance Test

Factory Acceptance Test procedure would occur. This would include the foundation for the Installation Qualification (IQ) and Operational Qualification (OQ). The Factory Acceptance Test would include machine specifications check, machine performance test and initial product inspection parameter setting.

Once at the installation site, the same IQ and OQ procedure used at the Factory Acceptance Test can be applied. The Performance Qualification (PQ) must then be performed on all products to be inspected on this piece of equipment. Some products can be grouped into certain conductivity groups or dielectric characteristics, but it is still necessary to validate every product separately.

A daily machine challenge may be necessary as proposed in a Standard Operating Procedure (SOP). Machine challenges may include creating defects in containers at specific inspection areas or having a machine challenge set of containers.

Any maintenance or calibration may result in requalifying or revalidation of the equipment. The same performance tests procedures will need to be performed.

9. Validation Procedure

A common validation procedure is to create a Validation Flow Chart for the inspection criteria. These tests may include a comparison test from another inspection process such as a dye test or batch challenge test. When creating the inspection flow chart, additional criteria such as a Detection Performance Test and False Rejection Test can be incorporated into the data.

10. Daily Machine Challenge Set

Machine Challenge Sets can be manufactured to challenge the inspection machine at each of the inspection zones. The container is duplicated in size and shape and a high tension resistor is placed within the container to simulate a defect at each of the inspection zones. Various resistance values determine a "good" or "faulty" container. This sample set is normally validated at specific inspection parameters and used on a daily basis to challenge the functions of the inspection equipment.

11. Comparison: Capabilities of Various Leak-Test Methods

Table 1. Comparison of Various Leak Tests

METHOD	LIMIT OF DETECTION	SAFETY OF PASSED SAMPLES	PROCESS OF INSPECTION
NIKKA DENSOK HighFrequency Spark Test	Detection of 0.5 micron holes has been confirmed. Its superiority over the crystallization method has been established, as all of the crystallized samples are detected by this method and no crystallization occurs among the samples passing this test. It is not affected by colors of the ampules.	Harmless. No adverse effect was found in chemical analyses (absorbance test, pH test and test for contents) following the inspection by this method.	Continuous Inspection, up to 18,000 ampules/hr; fully automatic, requiring no attention.
Crystallization	Detection of 0.5 micron holes has been confirmed. This method is superior to all others with the exception of the High-frequency, High-voltage Spark Test, as crystallization can occur due to migration of molecules. However, this method is workable only with crystallizable solutions and has the shortcoming of requiring very long periods of time for testing.	Harmless	Time-consuming (24 hours or more) Visual inspection
Autoclave	Limit of detection has not yet been determined. The leaktest using the dyeing method is performed concurrently with the sterilization process. This is more accurate than the vacuum-dye solution method.	Greater possibility of contamination of the contents.	Batch system, visual inspection

Radioactive Substance	Limit of detection has not yet been determined. Follows steps similar to those of the vacuum-dye solution method except that it uses a radioactive element of a short halflife in lieu of a dye. Superior to the vacuum-dye solution method.	Greater possibility of contamination of the contents.	Batch system, measured with measuring instruments
Vacuum-dye solution	Limit of detection is about 3 microns (in the case of 0.0015% methylene blue at 100 mmHg for 30 minutes). Mathematically, 2200 hours are required to detect a pinhole of 0.1 micron in diameter, and even for a hole of 0.36 micron the time required is 16 hours. A considerable number of defective ampules (including those contaminated by dye) are found among the samples passed by this test. It is not suitable for opaque ampules.	Contamination of the contents observed in some of the samples.	Batch system, visual inspection
Dyeing after autoclaving	Limit of detection has not yet been determined. Its accuracy is approximately the same as that of the vacuum dye solution method. Test is performed immediately after autoclaving.	Greater possibility of contamination of the contents.	Batch system, visual inspection

12. **Conclusion**

The HVLD system is a non-destructive, highly accurate and efficient way of inspecting for pinholes, cracks or defective seals in containers. The HVLD system is a validatible process and has been recognized by the FDA as a 100 percent in-line testing device for seal integrity. There is no threat of secondary contamination or degradation to the products being inspected, therefore the technology can be utilized in a variety of industries.