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LEAK DETECTION IN HERMETICALLY SEALED DEVICES

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16. Abstract

A method for detecting leaks in a hermetically sealed device has been developed. The method comprises the introduction of a low-boiling halogenated liquid into the device through the leaks, and detecting the vaporizing liquid as it escapes from the leaks by the use of a halogen detector. The leaks may thus be pinpointed, and the leak rate determined.

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LEAK DETECTION IN HERMETICALLY SEALED DEVICES

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SUMMARY

Many methods have been developed for the detection of leaks in hermetically sealed devices. Some will determine quantitatively the rate at which the device as a whole is leaking. The method described here can pinpoint each leak and determine its leak rate.

A low-boiling fluorocarbon liquid is introduced through the leaks by immersion of the heated, evacuated device in the cooled liquid. The escaping vapor is detected by an instrument which responds to the presence of the halogen upon being drawn between a heated, positively charge platinum emitter within a cylindrical collector electrode by an increase in the interelectrode conductance proportioned to the amount of halogen gas present. The range of detection is from greater than 1×10^{-5} standard cc/sec to approximately 1×10^{-9} standard cc/sec.

INTRODUCTION

Sensitive devices are frequently packaged in metal containers which are hermetically sealed in order to protect them from a variety of contaminants present in their operating environments. This practice is particularly common in the electronics industry where many devices such as transistors, integrated circuits, relays, etc., are protectively sealed in small metal or metal and ceramic enclosures. After having been sealed, the hermeticity of these cans must be tested to insure that the enclosed components will remain isolated from the environment.

Leak sizes are empirically defined as either gross or fine, with a gross leak permitting passage of more than 1×10^{-5} standard cc/sec of air and a fine leak, between 1×10^{-5} and 1×10^{-8} standard cc/sec. Various methods are now used to test hermetically sealed enclosures, and, frequently, different methods are required depending upon the sizes of the leaks being detected (refs. 1,2).

A practice even now in use for detection of gross leaks consists of immersing the device in a bath of ethylene glycol or glycerine heated to $150°C$ and watching for bubbles of gas escaping from the heated can. This method has been found to be highly unreliable, and so new methods have been sought. Three of these are worth mentioning. In one method, the device is immersed in

Freon 113 after one hour in a vacuum, and held for 3 hours in this liquid at a pressure of 90 psi. It is then immersed in fluid FC-75 at 70°C and subsequently fluid FC-43 at 125°C and observed for bubbles of Freon 113 vapor escaping (ref. 3).

In the second method, the devices are held in a vacuum and then immersed under pressure in a bath of FC-75 fluorocarbon liquid. A change in weight of the device indicates pickup of the fluorocarbon liquid through a leak (ref. 4).

A third method which is under development at ERC uses the standard helium leak detector, but is significantly modified by the use of a sample container having a controlled orifice. The range covers gross to fine leaks, and the particular advantage is that leaks that are intermediate between gross and fine leaks can be detected reliably (ref. 5).

The two principal methods now in general use for the detection of fine leaks involve the pressurization of the devices with helium or radioactive Krypton 85. In the former, the devices are pressurized at 3 to 5 atmospheres of helium for periods of from 1 to 20 hours. Leaks are then detected by a modified mass spectrometer. The Krypton 85 procedure also requires "bombing" for an extended period of time, followed by detection of the radioactive Krypton 85 by means of a scintillation counter. By modification of procedures, each method may be used to detect gross leaks. However, in the case of the helium test, there is the problem of such rapid loss of helium from a large leak (>> 10^{-5} cc/sec) that not enough remains to be detected, and the device may be passed as a good unit. For gross leak use with the Krypton 85, mineral oil or fluorocarbon liquid must be introduced into the device first. The gas dissolves in the oil and then gives off slowly enough to be detected.

Other procedures are now under development that show promise of fast, reliable leak detection over the entire range of gross and fine leaks.

In all of these methods for leak detection, the result is given in terms of the presence or absence of a leak in the device as a whole. Although a leak rate may be determined, there is no indication as to the location of the leak. There are several good reasons for knowing precisely where the leak is coming from in a given device. For example, if the sealing equipment is not seating properly, one side or a corner of the cover may be open. If there is insufficient heat used in the sealer, there may be several leaks. If the leaks can be pinpointed, more meaningful corrective action can be taken. If a leak is found in an expensive, hybrid relay, or other relatively large device, the source may be located and resealing accomplished.

This report will now describe a procedure for pinpointing leaks in hermetically sealed enclosures and determining the leak rate at each point from a gross leak rate up to approximately a rate of 1×10^{-9} standard cc/sec.

Basically, the method involves the introduction of a low-boiling fluorocarbon liquid into the sealed container through any leak, and the detection of the resultant vapor escaping from the leak by means of a halogen leak detector.

HALOGEN LEAK DETECTOR

A General Electric Company control unit with an H-5P pro-portioning probe is used as the detector (Figure 1). Calibration of this instrument is made using a General Electric Standard Leak, LS-20, calibrated for 1 to 10×10^{-7} standard cc/sec. An internal pump in the control unit draws air and a leaking halogen or gaseous halogen-containing compound through the probe head. The air-gas mixture is drawn between a heated, positively charged platinum emitter within a cylindrical collector electrode. The detector responds to the presence of halogen vapor with an increase in interelectrode conductance proportional to the amount of halogen gas present, which results in greater positive ion current. This emission current is amplified and the amplifier output causes the leak-indicator meter to go upscale and to sound an audible alarm. The control unit has a sensitivity selector switch with ten positions, permitting detection at 10^{-6} to 10^{-9} standard cc/sec on two scales of 0-3 and 0-10 divisions as well as two low uncalibrated positions.

The probe is located at the end of a hose that should be not greater than one meter long. It is modified by attaching a sawed-off section of a large hypodermic needle with Tygon tubing to the tip (Figure 1).

HALOGEN-CONTAINING LIQUIDS

In selecting a halogen-containing fluid, several criteria were used. A low-boiling liquid was desired both for easy removal from the surface of the package and for ease of volatilization after introduction into the package. A low surface tension was required to facilitate penetration into the smallest orifices. Chemical inertness was naturally a requirement because of the possibility of some of the liquid remaining inside a can. Finally, the liquid had to be non-toxic and cause no allergic reactions.

At the start of this investigation, the liquid that seemed to meet these criteria was trichlorofluoromethane (Freon 11, Genetron 11). The boiling point is 23.8°C, the surface tension is 18.7 dynes/cm, and the toxicity is very low. The only concern
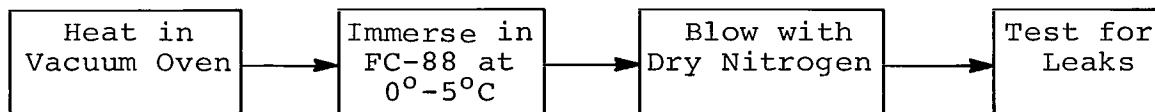
Figure 1.- Control unit

was potential hydrolysis in contact with certain metals and the possibility of corrosion by hydrolysis products. However, this liquid was very useful in proving the feasibility of this procedure.

During the course of this work, the 3M Company marketed a new, completely fluorinated liquid, FC-88. The major usage of this liquid was directed toward cooling and thermal stabilization of electronic packages and systems containing microcircuits. The boiling point is $32^{\circ}C$ and surface tension is 13 dynes/cm. The liquid is so inert chemically that it is permitted to be in direct contact with the devices at all times. In addition, the literature claims that FC-88 is "essentially nontoxic". FC-88 subsequently proved to be superior to the trichlorofluoromethane in producing stable and consistant readings.

PROCEDURE

The basic procedure involved is shown in the following flow chart:

Heat in Vacuum Oven	→	Immerse in FC-88 at $0^{\circ}-5^{\circ}C$	→	Blow with Dry Nitrogen	→	Test for Leaks

Vacuum Oven Heating

The device is heated to $50-150^{\circ}C$ in a vacuum oven at a pressure of 10 Torr or less for approximately one hour. The heat and vacuum treatment will create a partial vacuum in the device in preparation for the next step of back-filling. It is preferable to use the highest temperature that the device can tolerate.

Immersion in Liquid

The device is removed from the oven as soon as the vacuum has been broken, and immediately immersed in the FC-88 fluid which is held at $0-5^{\circ}C$. It remains in the liquid for at least 1 hour. It may be left here for any longer period of time provided the container is covered to prevent condensation of water vapor and contamination by dust.

Blowing with Nitrogen

When ready for testing, the device is removed from the FC-88 liquid and rotated in a rapid stream of dry nitrogen for at least 30 seconds. This will remove any adhering liquid that might

contribute to a high background, and should be done in a hood so as not to contaminate the area.

Testing for Leaks

The detector should be set up in a well-ventilated area free from contamination by any halogen-containing vapors, for example, trichloroethylene, carbon tetrachloride, chloroform and Freons and Genetrons. The pressure cans of cleaners, lubricants, etc., can leak significant amounts of halogen-containing propellant. Tobacco smoke is also a bad contaminant.

The detector is first calibrated using the standard leak as directed. The background is then determined by setting the detecting range at sequentially higher settings to determine stability at each range. If the needle does not remain steady, at least at 1×10^{-8} standard cc/sec, the environmental contamination is too high.

Gross leaks are so easily detected that it is recommended that the devices be first scanned rapidly using the low, uncalibrated settings. If the hot emitter of the detector is exposed to too high a level of halogen, it becomes desensitized and a few minutes delay results until the instrument recovers. Furthermore, the life of this element is shortened.

A higher range is then selected and the needle probe is moved around the sealed areas of the device, pausing only long enough at each pin, or at distances apart of approximately 5 millimeters to see if the instrument registers (Figure 2). With a probe hose of 1 meter, response time is not more than 3 seconds.

DISCUSSION

One of the first considerations in the use of this procedure was the accuracy and reproducibility of the measurements. The detector is calibrated for the use of Freon 12, dichlorodifluoromethane. Tables are available for the use of a multiplication factor when other halogen gases are used. There were no factors available for FC-88, so comparison tests were made on the same devices for FC-88 vs. Freon 11 vs. standard helium mass spectrometer equipment. It must be kept in mind that running any leak test on a device can open the leak further, or reduce or close it by introducing a solid contaminant. Comparison of results on the same devices by different procedures can sometimes lead to erroneous conclusions.

Table I compares the test results of the same group of 14-lead flatpacks, 0.25 in. x 0.25 in., with ceramic covers. Although many leaks were found in each unit, numbers given refer only to the largest detected. On the average, multiplying the FC-88

Figure 2.- Needle probe

values by 1.4 brings them in line with Freon 11 results.

TABLE I

COMPARISON OF RESULTS USING HALOGEN LEAK TESTER
AND TWO HALOGEN-CONTAINING GASES

Unit No	FC-88 (std. cc/sec)	F-11 (std. cc/sec)
003	7×10^{-7}	1×10^{-6}
005	8×10^{-7}	1×10^{-6}
007	3×10^{-7}	6×10^{-6}
023	8×10^{-7}	9×10^{-7}
1	1×10^{-6}	1×10^{-6}
2	7×10^{-7}	1×10^{-6}
3	1×10^{-6}	1×10^{-6}
4	8×10^{-7}	1×10^{-6}
5	6×10^{-7}	1×10^{-6}
6	7×10^{-7}	1×10^{-6}

Table II compares another group of similar devices when tested using FC-88 and the halogen leak detector vs. a helium leak tester. The results show excellent correlation. They further indicate that no multiplying factor is necessary to compare these results with those of the helium leak tester. The latter was calibrated using a standard leak.

TABLE II

Unit No	FC-88 (std. cc/sec)	He Leak Tester (std. cc/sec)
8128	4×10^{-8}	4.2×10^{-8}
7900	2×10^{-8}	2.5×10^{-8}
8133	8×10^{-8}	1.6×10^{-7}
7894	1×10^{-8}	1.8×10^{-8}
1	5.5×10^{-8}	5×10^{-8}
3	1×10^{-8}*	3×10^{-10}
A	5×10^{-9}**	5×10^{-10}
B	3×10^{-7}	1.3×10^{-7}
C	1×10^{-8}*	2×10^{-10}
D	2×10^{-6}	3.4×10^{-8}
E	6×10^{-8}	3.8×10^{-10}
F	6×10^{-8}	6.6×10^{-8}
G	1.5×10^{-8}*	2.2×10^{-10}
H	5×10^{-8}	5.2×10^{-8}
I	6×10^{-9}**	3.4×10^{-10}
J	6×10^{-8}	5×10^{-8}
K	3.5×10^{-8}	6×10^{-8}
L	8.5×10^{-8}	5×10^{-8}

*Highest sensitivity for apparatus before filter change.
**Highest sensitivity after filter change.

A major problem in using some leak detection methods has been the rapid loss of detector gas from gross leaks, leaving some of these undetectable by the time the device was inserted in the apparatus. In a series of experiments, devices with different leak rates were checked over a period of time to get an idea of the duration of detectable leaks. Figure 3 shows measurements made on a 0.22 in. x 0.22 in. x 0.050 in. 14-lead flat-pack. This was a gross leaker with six detectable leaks. The decay rates of four of these are given. The fluid in this case was Freon 11. It can be seen that plenty of time is available for measurement.

In Figure 4, using FC-88 the largest initial leak was 3.5 x 10^{-7} cc per second, and it took over fifteen minutes for the leaks to decay to an easily detectable 1 x 10^{-8} cc per second.

Two identical 1 in. by 1 in. ceramic-covered 32-lead hybrids were examined both by this procedure and by the standard helium leak test. The first gave no indication of leaks by either procedure. The other showed a gross leak by both methods. The halogen leak detector method was still indicating several leaks far greater than 1 x 10^{-5} cc per second 5-1/2 hours later. Moreover, the principal leak area was identified as a large opening at one corner of the device.

The following rules should be followed for good measurements:

(1) The precise leak point or areas are pinpointed.

(2) A controlled environment of fresh air must be available.

(3) Smoking should not be permitted in the area, and all halogen-containing gases and liquids must be kept away, including aerosol bombs.

(4) The FC-88 fluid can be continuously filtered to remove particles that might clog leaks.

(5) For good stabilization, the air filter of the detector must be changed periodically.

In summary, the advantages of this halogen leak detector procedure are as follows:

(1) Operation is in air. No pressurization is required.

(2) Quantitative leak rates are measurable both for a total system or for each one of multiple leaks.

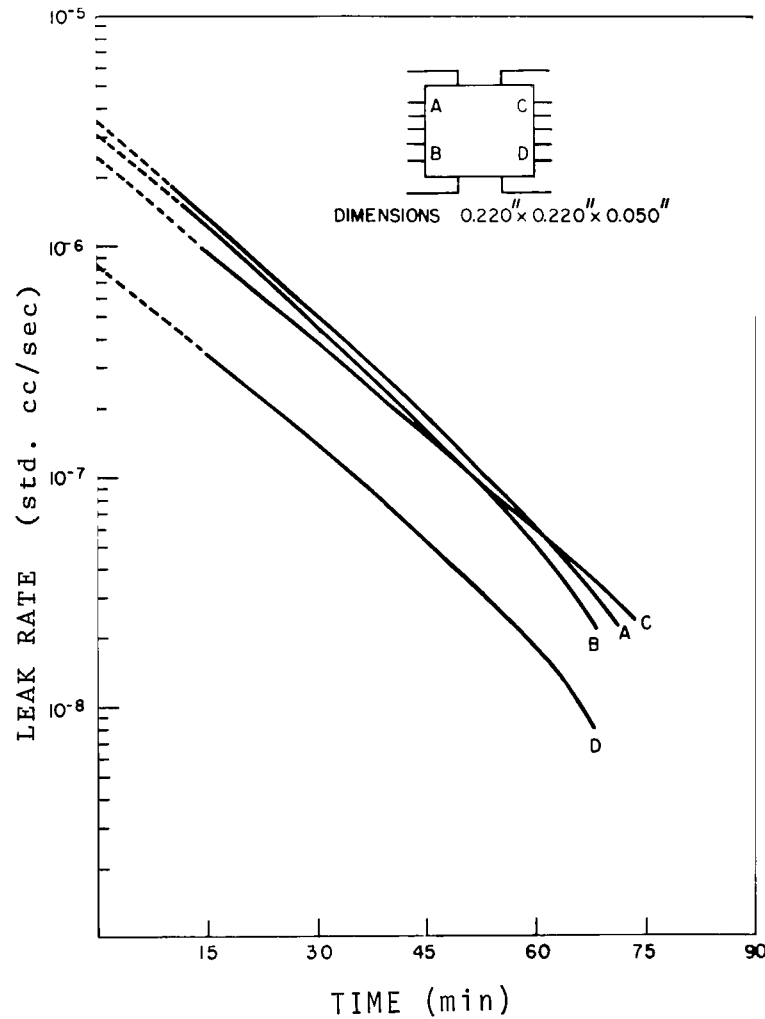(3) The detector is relatively inexpensive and easy to use.

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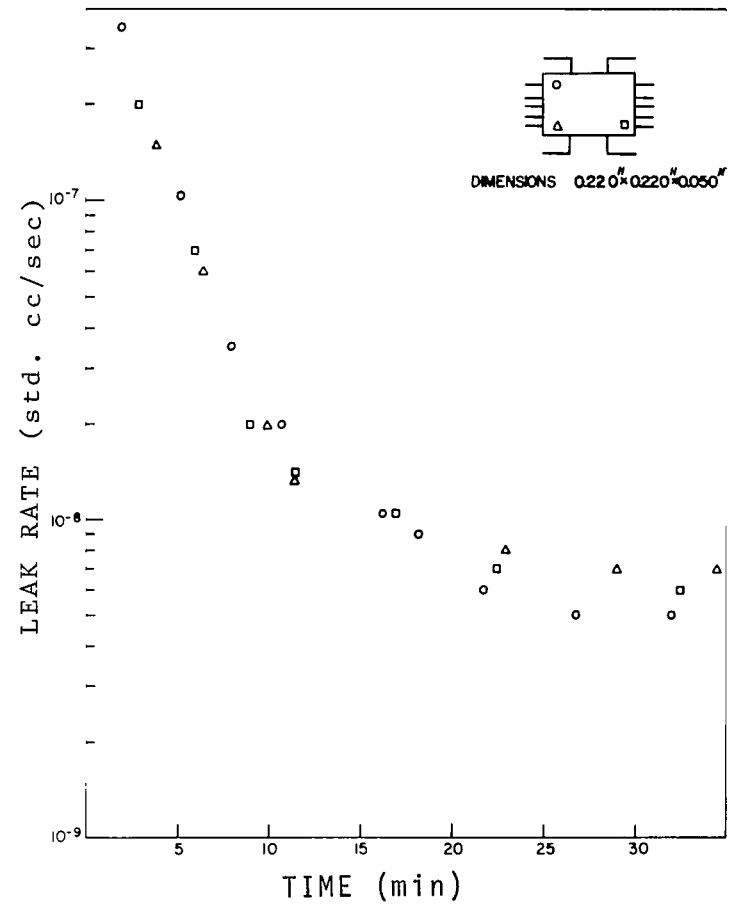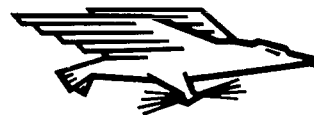Figure 3.- Leak rates vs. time for an integrated circuit; Fluid: Freon 11

Figure 4.- Leak rate vs. time for an integrated circuit; Fluid: FC-88

(4) The detection is apecific for halogen compounds (and tobacco smoke).

(5) There is plenty of time for detection of gross leaks.

(6) There is no restriction as to the size of the hermetically sealed device that can be tested.

(7) This method could also lead itself to an automated detection system by suitable design.

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