

# Evaluation of Post-Solder Flux Removal

*Various post-solder cleaning methods are evaluated for ionic residues in a series of experiments using a dynamic conductivity monitor*

BY J. BROUS

**ABSTRACT.** In electrical and electronic applications, the functional life of an assembly may be severely limited by the presence of ionic contaminants remaining after soldering which can originate from a multitude of sources. The development of a new instrument, the dynamic conductivity monitor (DCM) has facilitated the quantitative measurement of ionic contamination levels on electronic circuits and components. Using this as a tool, some variables of post-solder cleaning were evaluated in terms of their effect on final ionic cleanliness of the circuits. Cleaning effectiveness of different solvent cleaners, cleaning processes and procedures were measured. The DCM measurement process can therefore be a valuable tool for development and improvement of cleaning processes as well as a quality control inspection device for cleanliness monitoring.

## Introduction

One of the most important factors in the reliability of electronic circuits is their electrical stability with time. The presence of ionic contamination on insulating surfaces can seriously affect the electrical parameters, particularly in the presence of moisture. Additionally, ions can cause corrosion of metallic conductors.

In the manufacture of electronic circuit assemblies, highly corrosive ionic materials are generally used at one or more steps of the process. Circuit

board materials are plated, etched, handled by operators in assembly, coated with corrosive or potentially corrosive fluxes and finally soldered. Of course, there is cleaning at various steps along the way — for example after plating, etching and soldering — but each step, nevertheless, represents a potential source of ionic contamination which may be carried over on surfaces of the finished circuit. It is therefore most important that a thorough deionization of the surfaces be achieved after soldering. This is probably the last opportunity to remove ionic contamination accumulating on the circuit in the manufacturing processes.

There is a need to evaluate this important post-solder cleaning oper-

ation to determine the effectiveness of ionic residue removal. A procedure for measurement of residual ions after plating was described by T. F. Egan (Ref. 1) of Bell Laboratories. In this method, the increase in conductivity of a static volume of water is measured after extracting the water-soluble ions from the surfaces of the object being measured. After a fixed period of bathing of the sample, the conductivity of the water is compared with the initial water conductivity prior to sample immersion.

Modifications of this method were adapted by H. E. Phillips (Ref. 2), Freon Products Lab., DuPont Co. and J. W. Dennis Jr. (Ref. 3), Martin Marietta Aerospace Division, to indicate the presence of ionic residues on

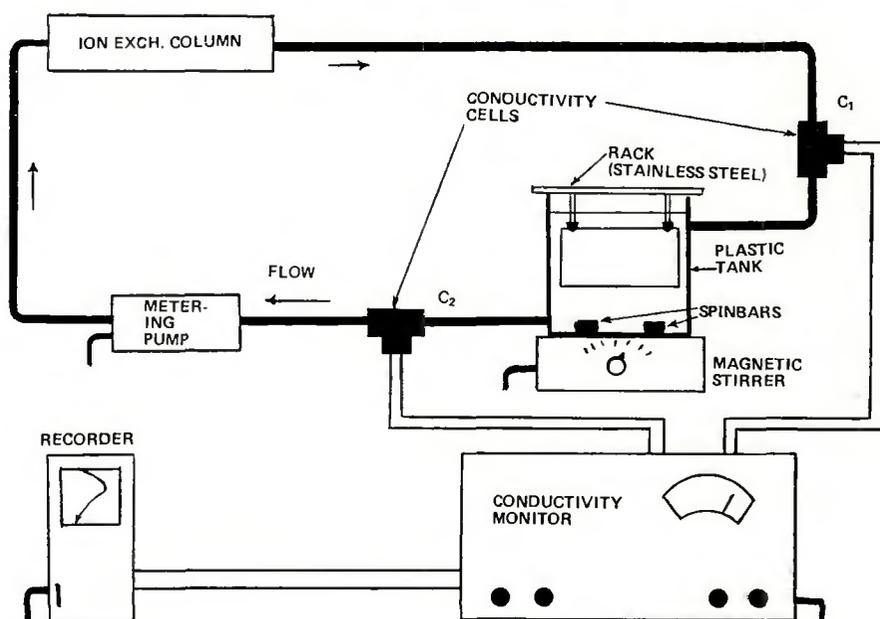


Fig. 1 — Schematic diagram of the Dynamic Conductivity Monitor (DCM)

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printed circuits after final defluxing following soldering. W. T. Hobson and R. J. DeNoon (Ref. 4) of the Naval Avionics Facility, Indianapolis, Ind., further modified the original technique of Egan using mixtures of water and alcohol to extract the residual ions from printed wiring assemblies. This greatly enhanced the ability to thoroughly extract all of the ions for measurement — especially in cases where rosin fluxes had been employed. Using a pure water extraction medium only, residual traces of rosin or other nonpolar residues, if present, can effectively mask the removal of water soluble ions.

### Dynamic Conductivity Monitor (DCM)

A new procedure for quantitatively measuring the extractable ionic content of circuit assemblies was developed in the Research Laboratory of Alpha Metals. This method measures the ionic conductivity of a continuously circulating rinse solution rather than a static volume into which the ions are dissolved. Figure 1 is a schematic representation of this system.

In operation, an alcohol-water mixture is pumped continuously in a closed loop. The residual ions are stripped from a circuit assembly in a small plastic tank. The conductivity of the liquid flowing into or out of this tank can be monitored. Before flowing into the tank, the solution is passed through a mixed-bed ion exchange column which removes all ionic content, then through a conductivity cell,  $C_1$ . The solution emerging from the tank flows through another conductivity cell,  $C_2$ , and then through a controlled metering pump. The conductivity of either the input or output liquid of the tank can be monitored by the conductivity monitor. This is a continuously monitoring device which provides a dc voltage output which is linearly related to the conductivity. The voltage is fed to a chart recorder which displays the time function of conductivity. The cells,  $C_1$  and  $C_2$ , are temperature compensated to avoid reading variations due to temperature change.

With the system filled, the water-alcohol mixture recirculating through the ion exchange column will be purged of all ionic content. With no sample in the tank, a condition will be established in which conductivity values at  $C_1$  and  $C_2$  will be equal. A 1:1 mixture of isopropanol and water, is usually at a conductivity reading of 0.05 micromhos/cm (equivalent to 20 megohms cm). With the introduction of a contaminated sample, the conductivity of the tank output solution rises rapidly along with a corresponding indication on the conductivity

monitor and recorder. The test procedure is run until the conductivity returns to the level of the input solution. At that point, it can be concluded that no further ionic material is available for removal from the sample.

The entire amount of ionic material removed from the sample can be related to the integrated conductivity readings over the period of time required to dissolve the material and purge it through the tank, tubing and conductivity cell  $C_2$  as follows:

At any instant,  $t$ , the number of moles,  $n_t$ , of ionic material within the conductivity cell is  $n_t = V_c C_t$  where  $C_t$  is the concentration of ions and  $V_c$  is the cell volume which is constant. Over an infinite amount of time, the total number of moles of ions,  $N$ , passing through the cell will be:

$$N = \int_0^{\infty} n_t dt = V_c \int_0^{\infty} C_t dt$$

Since we are dealing with very high dilutions ( $<10^{-4}$  N), we can assume complete ionization for our purpose, therefore:

$$\text{Conductivity} = L = k C$$

(Assuming one salt to be present. Of course, different ionic salts with different ionic mobilities will give different conductivities for a given concentration.)

$$N = k V_c \int_0^{\infty} L_t dt$$

Since the meter and recorder responses are linear with respect to  $L$ , the area under the conductivity-time curve which is charted on the recorder is a function of the total amount of ions removed from the surface of the boards.

The system can be calibrated by making similar measurements after injecting a known amount of a salt, such as sodium chloride, and obtaining the integrated response of the meter. The conductivity can then be

represented, as is often done for water impurity measurements, as the conductivity of sodium chloride solutions. While not accurate in terms of actual concentrations of the unknown ions, this method enables us to represent ionic levels in a situation where many unknown ion types may be present in different proportions. This function is more meaningful than actual concentration measurements because it normalizes concentrations of all ions of various mobilities to a conductivity function which is more directly related to the surface electrical leakages on a circuit board.

Figure 2A shows a typical recorded curve obtained by injecting a quantity of a sodium chloride solution directly into the tank. This curve shows a rapid build-up of conductivity followed by an exponential decline as the salt is purged through the system. A measurement of the area under this curve can then be related to the known amount of salt which it represents and used to determine the ionic content of an unknown sample. Figure 3 shows the linear calibration curve obtained by inject-

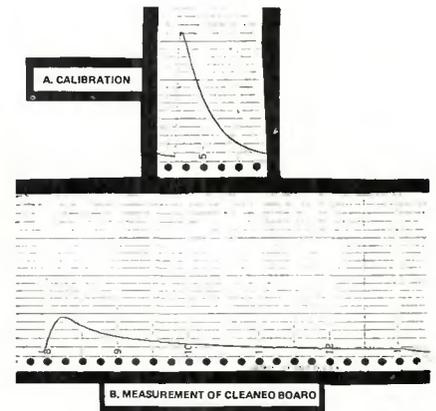


Fig. 2 — DCM traces. (A) Calibration with NaCl; (B) measurement of a cleaned board

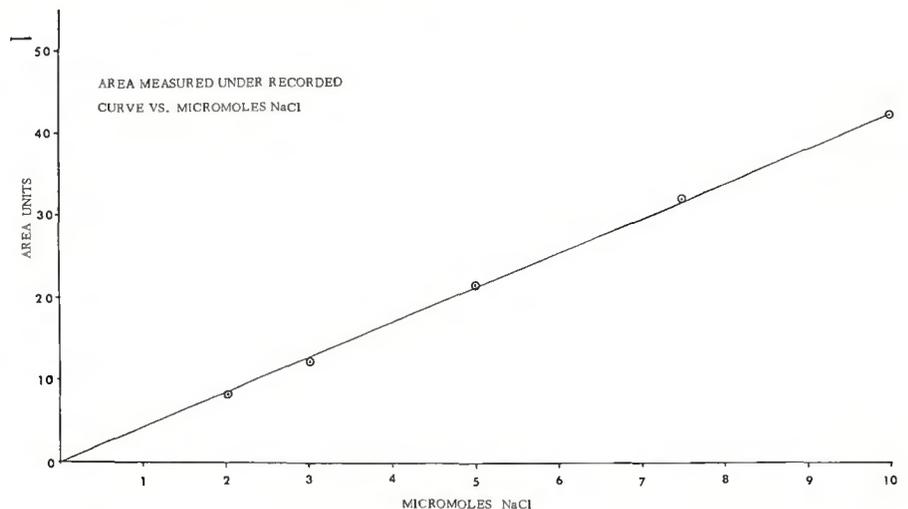


Fig. 3 — Calibration curve obtained by injecting various amounts of 0.1 N NaCl solution into the tank



Fig. 4 — Portable DCM system

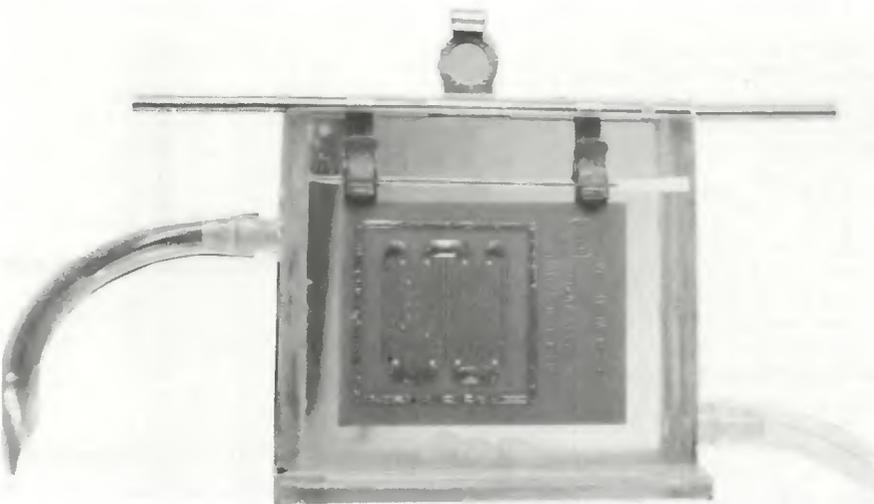


Fig. 5 — A board suspended in the plastic tank for measurement of ionic contamination

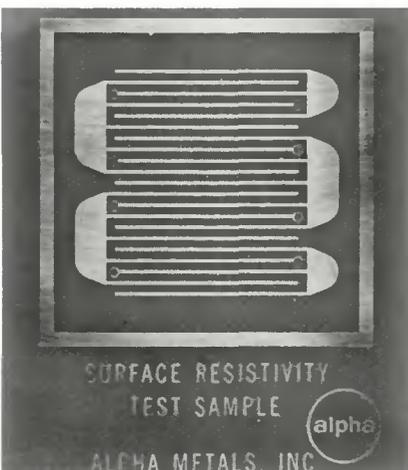


Fig. 6 — Typical board used as a test sample

ing various amounts of a 0.1 N NaCl solution into the tank.

Figure 2B shows a typical curve obtained with a board which had been fluxed with an activated rosin flux, soldered and cleaned. A comparison of the curve with that of Fig. 2A shows a more gradual build-up of conductivity to the peak value and a more gradual decline to the base level. This indicates that there is a finite rate of extraction of residual contaminants from the boards which brings the ionic material into solution over a period of time.

Figure 4 shows the entire system built to fit into a small suitcase. Figure 5 shows a board being measured in the plastic tank. It was found that the use of a magnetic stirrer with several

small Teflon coated magnetic spin bars, greatly improved the smoothness and uniformity of the curves obtained but had no effect on the integrated conductivity readings. It is used, therefore, to obtain curves which are smooth and easily interpreted.

### Evaluation of Cleaning Variables

The ability to measure amounts of residual ionic soils, offers a useful tool to study the parameters of the cleaning operation in terms of their effects on final cleanliness. In the work reported in this paper, the cleaning effectiveness of different solvent and chemical flux cleaners as well as the cleaning capabilities of different cleaning processes were studied. An attempt was made to select typical processing conditions using most of the commercially important solvent cleaners and processes.

### Boards

The test samples used were epoxy-fiberglass (G-10) boards with dimensions of 2.75 X 3.5 X 0.0625 in. (Fig. 6). These boards, which regularly have been used for surface conductivity tests, had a total surface area of 20 in.<sup>2</sup> (129 cm<sup>2</sup>).

Prior to fluxing and soldering, all boards were thoroughly cleaned in a sequence of solvent rinses as follows:

1. Perchloroethylene
2. Isopropanol (two rinses)
3. Deionized water
4. Acetone (electronic grade)
5. Air dry

Each rinse was one minute. This pre-cleaning was used to eliminate the random variabilities of the previous history of plating, etching and handling operations of each board, reducing them all to a uniformly clean level. A check in the DCM system of boards cleaned in this manner, showed a complete absence of ionic material.

### Soldering

In order to reproduce actual conditions encountered in an industrial soldering process, a large Electrovert wave soldering system was used. The boards were wave fluxed with Alpha 711-35 which is a type RA (activated rosin) flux. The solder used was 60/40 Sn/Pb alloy maintained at 500 F. Preheaters were set at 450 F. The transport rate was 3 ft/min.

### Cleaning Materials and Processes

The soldered, uncleaned boards constituted the test vehicle for the study of processing and material variables. Four different cleaning procedures were evaluated:

1. Room Temperature Solvent Cleaning with Agitation — Boards

were immersed, with agitation, for two minutes in a solvent cleaner at room temperature. This was repeated in fresh solvent and then air dried.

**2. Vapor Cleaning and Distillate** — In a small, one gallon capacity, two compartment vapor degreaser, boards were held in hot vapor for one minute followed by a one minute immersion in the distillate. This was then repeated twice, followed by a slow removal of the cleaned board through the vapors.

**3. Vapor Cleaning with Immersion in Sump, Distillate and Vapor** — This is the same as the previous procedure except that the first immersion was directly into the boiling liquid of the sump. The other two dips were as above. The materials in the sump for this and the prior procedure were initially contaminated by adding 3% by volume of solid, activated rosin flux.

**4. Water Soluble Chemical Rosin Cleaning** — An attempt was made to simulate in laboratory glass beakers, the water soluble rosin stripping method. Industrially, this is performed in large spray cleaning installations of dishwashing equipment. The object of this evaluation was to estimate the capabilities of this relatively new saponification method for rosin flux stripping rather than to run a comparison of specific, commercially available proprietary formulations or of large cleaning installations.

A beaker containing a 10% by volume solution of a proprietary commercial formulation was maintained at 160 F ± 5 F. The boards were vigorously agitated in the hot cleaner for three minutes. This was followed by a brief rinse under tap water followed by agitated immersion for one minute in hot (160 F), then cold, deionized water. The boards were air dried.

### Solvent Cleaners

The solvent cleaners included in this study were:

- 1,1,1, trichloroethane
- Perchloroethylene
- Perchloroethylene with 10% ethyleneglycol monoethyl ether
- Trichlorotrifluoroethane (FC-113)
- FC-113 with 4.5% ethanol (azeotrope)
- FC-113 with 49.5% methylene chloride (azeotrope)
- Tetrachlorodifluoroethane (FC112) with 28% isopropanol (azeotrope)
- FC-112 with 14.5% n-propanol (azeotrope)

With the exception of the solvents containing perchloroethylene, all of the materials were evaluated under the first three process procedures.

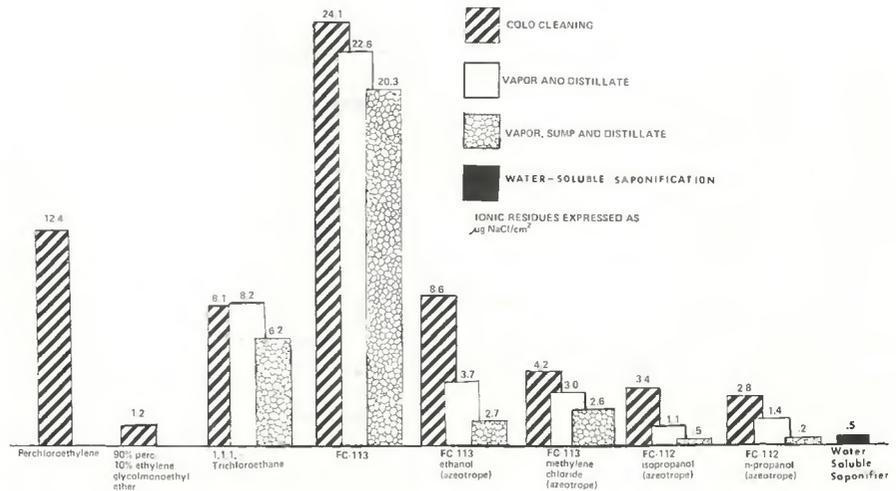


Fig. 7 — Relative amounts of ionizable residues left on PCBs after cleaning

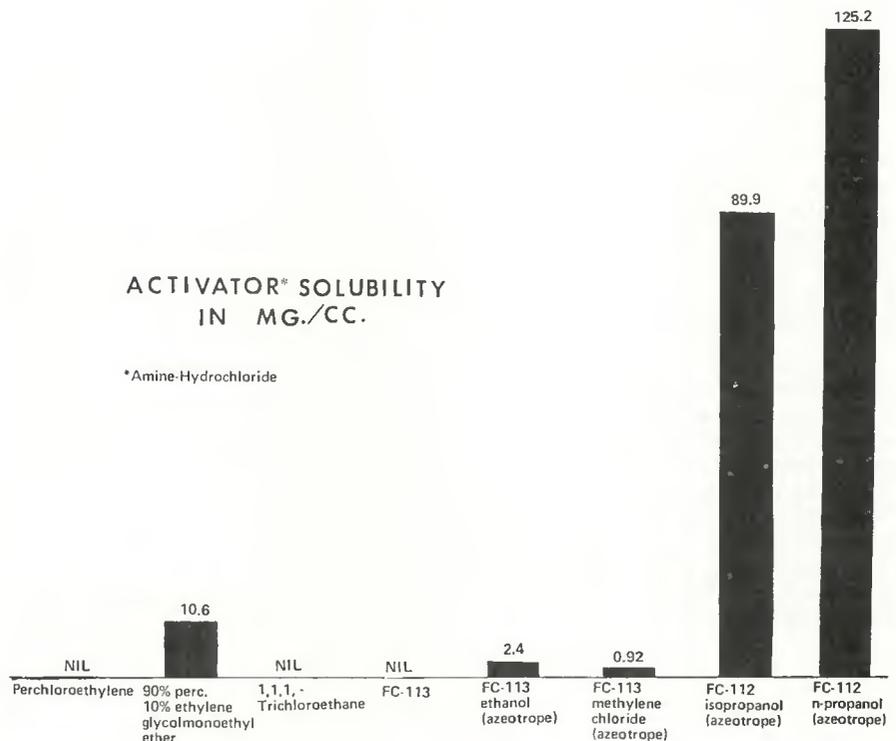


Fig. 8 — Activator solubility in milligrams per cubic centimeter in various solvent cleaners

Perchloroethylene-containing solvents were not evaluated under the hot vapor methods since these are usually precluded for perchloroethylene by the solvent aggressiveness toward plastics, marking inks, resists and circuit boards.

### Results

Distinct differences in cleanliness levels were found, varying with the materials and processes of cleaning. The residues remaining on the boards after the processes described above, are indicated in the bar graphs shown in Fig. 7. Each result shown is the average value of three measurements.

It was found that vapor cleaning generally resulted in an improvement of cleanliness over room temperature immersion cleaning. In every case, however, significant improvement in cleanliness was seen when the boards were first dipped in the contaminated boiling sump rather than the distillate only.

The most highly bipolar solvent blends left the boards in the cleanest condition, while the pure chlorocarbons or fluorocarbons left behind higher levels of the ionic flux activators. In cold cleaning, the most efficient flux removal was obtained with the 90% perchloroethylene-10% ethylene glycol monoethyl ether mixture with the FC-112 n-propanol

azeotrope close behind.

In the vapor cleaning methods, the FC-112 azeotropes show significant improvement over the other solvents tested and in comparison to themselves in cold cleaning.

Figure 8 shows a bar graph representation of the measured solubilities of an amine-hydrochloride which is commonly used as an activator for fluxes. Close examination of these values indicate, not surprisingly, that those solvents which can dissolve the greatest amounts of the amine salt, are those which leave behind the lowest levels of ionic residues on the boards.

The laboratory test of the commercial water-soluble rosin saponifier, indicated that this method can reduce ionic levels to those obtained by vapor cleaning with the most effective solvent cleaners.

### Additional Measurements and Results

Several brief experiments were conducted using the DCM system to explore other parameters which could have a bearing on ionic cleanliness of circuit boards.

### Ultrasonic Cleaning

When ultrasonics were used in conjunction with solvents at room temperature, the cleaned products were found to retain substantially the same amounts of ionic residues as their counterparts cleaned at room temperature with only mechanical agitation. It appeared, however, that the ultrasonic cavitation was very effective in assisting the removal of insoluble particulate matter.

### Airborne Ionic Contamination

Cleaned boards which were hung in room air for periods of from one day to two weeks, were found to pick up ionic contamination at a rate of about 0.3 micrograms NaCl/day. Cleaned boards which were stored for similar periods in clean plastic bags or in sealed jars with or without included moisture, did not indicate any increase of ionic content with time. This atmospheric parameter is, of course, variable with the location and most likely with factors such as climate, wind velocity and direction, and industrial and other activities in the area which could affect airborne content. These results indicate one of the most important benefits which can be expected from the use of conformal coating-protection of cleanliness of insulation surfaces.

### Fingerprints

Measurements were made of the salt contributed by individual fingerprints. This also is highly variable depending upon the individual and the many contributing factors affecting the salt content of his fingertips. Individual prints on boards contributed as much as 30 micrograms of NaCl as measured on the DCM. This, of course, is a more concentrated ionic deposit and not averaged over the whole board surface, making it potentially more damaging to the circuit function. It emphasizes the need to exercise precautionary measures to avoid bare-handed contact of circuits once final cleaning has been accomplished.

### Conclusions

The measurement capability of the DCM has enabled the following con-

clusions to be made:

1. Hot vapor cleaning reduced ionic levels below those obtained with room temperature solvent cleaning.

2. Immersion of the soldered circuits into the boiling sump of a vapor degreaser, despite very high sump contamination levels, resulted in the cleanest final product.

3. Those solvent blends which exhibited the highest degrees of bipolar character, as for example, their ability to dissolve significant amounts of the ionic activator salts, were also most effective in reducing ionic residues on the soldered boards.

4. The use of water-soluble rosin saponifiers can be an effective means of removing rosin fluxes and residues after soldering.

5. Ultrasonic cleaning does not reduce ionic residue levels to values lower than those otherwise obtained without ultrasonics.

6. Airborne dust particles can be a significant source of ionic contamination.

7. Hand contact with cleaned circuit assemblies can be a significant source of ionic contamination.

8. The DCM was demonstrated to be an effective instrument for the quantitative measurement of residual ionic levels.

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## WRC Bulletin

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Sept. 1973

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