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CLEANLINESS METER AND ITS APPLICATION
TO SOLVENT CLEANING

R. C. Marsh and F. W. Oswalt

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IN NUCLEAR SCIENCE ABSTRACTS

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by

R. C. Marsh and F. W. Oswalt

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ABSTRACT

This paper describes the development and application of a new instrument which will measure the soluble residue content of volatile cleaning solvents. The instrument, called the Solvent Purity Meter, is capable of giving an instantaneous and continuous read-out of residues as low as one part per million and may be used either for batch sampling or as an on-line continuous monitor for recirculating cleaning systems.

Typical laboratory studies using the meter, as well as results from in-process cleanliness testing applications on component production lines, are discussed. Evaluations of the instrument, to date, have shown it to be a useful production line monitor for testing the purity of cleaning solvents, as an indirect measure of component cleanliness, and for pointing out errors or problem areas when troubleshooting component cleaning problems.

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CLEANLINESS METER AND ITS APPLICATION TO SOLVENT CLEANING

Introduction

The evaluation of the effectiveness of a production cleaning process is a particularly difficult task. Methods to detect small amounts of contamination are time-consuming and inaccurate and attempts to assess the effectiveness of a particular process are usually only qualitative. It is often said that cleaning is an art, not a science. This is due, to a large extent, to the difficulty in obtaining significant numbers to describe the "cleanliness" of a part or the relative effectiveness of a cleaning solvent or process.

It is not the purpose of this paper to discuss the relative merits of the many techniques now in use but rather to describe the design and development of a new automatic nonvolatile residue tester. The test for nonvolatile residue (NVR) in solvents and its usefulness for solvent extraction analysis is not new; however, standard techniques are laborious and subject to many errors, thus limiting the usefulness of the test. The possibility of measuring NVR automatically and rapidly opens up a widespread usage of the technique for evaluating many areas of the cleaning process. The only requirement is that the soils of interest be soluble in the test solvents which are used. These various applications and their limitations will be discussed in some detail later in this paper.

Development of the Solvent Purity Meter

Salkowski and Werle^{*} first described an instrument of this type which they called a Nonvolatile Residue Nephelometer. The instrument measured the residue of manufacturing oils and greases in trichlorethylene by first aerosolizing the solvent and then observing the aerosol residue remaining after evaporation of the solvent with a dark-field optical system. The Sandia Solvent Purity Meter utilizes this basic concept in a versatile multipurpose instrument suitable for a wide range of applications. A major goal in the design of the Sandia instrument was that it be simple to operate and noncritical, suitable for production process control application.

Design Characteristics

The basic features of the instrument are illustrated in Figure 1. The major components are a solvent sampling system, an all-glass nebulizer, a drying tube, various air meters and gages, and a light-scattering photometer.

^{*}"Nonvolatile Residue Nephelometer," Salkowski and Werle, presented at the International Symposium on Surface Contamination, Gatlinburg, Tennessee, June 8, 1964.

Figure 1. Basic Features of Solvent Purity Meter

Sampling System

The sampling system provides for three modes of sampling. A prime consideration is that the system be completely noncontaminating to the solvent. All parts which physically contact the solvent are either glass, stainless steel or Teflon. The test solvent may be introduced into the nebulizer reservoir by one of three methods (illustrated in Figures 2, 3, and 4): (1) by pouring directly from a laboratory bottle or beaker, (2) by pumping through a flexible tube by means of a syringe hand pump, or (3) continuously circulating through the reservoir from a pressurized solvent source. Any one of the three modes of sampling can be selected by a single-function selector valve, a 90 degree bore, four-way stop cock.

When the sample is poured directly into the meter, as little as 10 cc can be used to make an NVR determination. When sampling from a large drum or other container by means of the hand pump, any length of sampling tube may be used. Sample volume will depend only on the volume of the sampling tube plus approximately 10 cc to fill the nebulizer reservoir. The hand pump apparatus is simply a 50 cc syringe and a two-way stainless steel check valve adapted from a standard BBL Automatic Pipetting Machine.* When the sample is continuously circulated through the meter, the flow rate is approximately

* Baltimore Biological Laboratories, Baltimore, Maryland.

SOLVENT PURITY METER

Figure 2. Open Pour Setup

SOLVENT PURITY METER

Figure 3. Syringe Pump Setup

SOLVENT PURITY METER

Figure 4. Continuous Circulation Setup

one liter per minute. From this flow only a small sample of about 100 to 200 cc actually flows through the nebulizer. Flow through the nebulizer is from the bottom overflowing the top to maintain a constant solvent level. For sampling lines of less than 10 feet in length, the response time of the meter is around 5 to 10 seconds.

A single sampling system and nebulizer has been used since it was found that normally only two to three flushes of the system with clean solvent is sufficient to remove any residual contamination left behind from previous samples. To use the instrument, a zero reading is always checked with the pure solvent immediately before an NVR determination is made to verify that the system is clean.

Nebulizer

The nebulizer used in the instrument has the atomizing characteristics of the standard No. 40 DeVilbliss atomizer, as far as the jet is concerned. The complete nebulizer is specially made by our scientific glass-blowing shop and includes the control valves (stop cocks) necessary to hold the sample in the reservoir during a measurement and to control the solvent flow when used in the continuous circulation sampling mode. The complete nebulizer and the drying tube are shown in Figure 5.

Drying Tube

The drying tube is simply a corrugated glass tube with two air inputs arranged so as to thoroughly mix two gases. The tube is used to mix the solvent vapor and droplet suspension with clean air to evaporate the major portion of the solvent from the droplets formed by the nebulizer. The tube is operated at

Figure 5. Special Design Nebulizer and Drier Tube

a slight overpressure to isolate the air in the tube from the surrounding environment. A gas overflow outlet is provided to vent the extra air that is not pulled through the photometer system.

The special nebulizer and drying tube are shown in Figure 5.

Air Handling System

The air required to operate the nebulizer and to mix with the solvent droplets in the drying tube is supplied from an external pressurized source. Standard compressed building air is adequate. The instrument contains the necessary air regulators, pressure gage and valves to meter the air to the nebulizer and drier systems. A pressure gage is provided for the input regulated air line and for the nebulizer pressure. A flow gage is used to measure the volume of air to the drying tube. The instrument also contains an in-line high-efficiency filter to remove any particulate contamination from the air supply.

Light Scattering Photometer

The photometer used is a commercial light-scattering photometer* which is used unmodified in any way and is calibrated with the filter slide in the number 2 position. Using this instrument, residues from 1 to approximately 1000 parts per million are readable on a single meter scale.

The complete instrument is shown in Figure 6.

To summarize the operation of the Solvent Purity Meter, the test solvent is introduced into the nebulizer reservoir where it is dispersed into a fine aerosol spray. Filtered air is then mixed with the solvent droplets, causing rapid evaporation of the solvent from the droplet. The resulting solvent-residue aerosol is then sampled with the photometer which responds in relation to the mass-concentration of the aerosol. The photometer reading is converted to parts per million of nonvolatile residue by empirically established calibration curves such as shown in Figure 7.

Calibration

The Solvent Purity Meter is calibrated by preparing standard solutions of contaminated solvents of known ratios. Calibration is normally established in terms of parts per million by volume. The standard solutions are prepared by adding 50 microliters of soluble oils to 500 milliliters of solvent, giving a solution of 100 ppm. This solution is then further diluted to obtain lower concentrations. This technique requires only an accurate microliter syringe and a laboratory graduate. A calibration in parts per million by weight can be similarly established, using a laboratory microbalance.

Figure 8 shows the calibration curves for a number of solvents. It can be seen that each solvent produces its own characteristic curve. Different contaminants in a single solvent, however, produce similar curves, as shown in Figure 8. Thus, except where high accuracy is required, a single curve may be drawn for each solvent.

* Aerosol Dust and Smoke Photometer, Model JM2000, Phoenix Precision Instrument Company, Philadelphia, Pennsylvania.

Figure 6. Solvent Purity Meter

Figure 7. Typical Curve Nonvolatile Residue Versus Photometer Reading

Figure 8. Calibration Curves for Various Solvents

It should be pointed out that if one solvent is contaminated with another solvent, erroneous results can be obtained, particularly in the case where an azeotrope is formed.

Experiments designed to determine the effects of the various parameters of the instrument itself on the accuracy of calibration have been encouraging. In general, the changes in the solvent occur rapidly after aerosolization. The solvent residue droplets are then quite stable and the readings are unaffected by small differences in the instrument hardware. Table I shows the sensitivity of the readings to solvent temperature, Table II the sensitivity to the drying air temperature and relative humidity, Table III the effect of varying the pressure on the atomizer, and Table IV the effect of the drier length. It is concluded from these data that the errors introduced by these variables are small and that the small changes one would expect under normal conditions would produce no significant errors.

TABLE I
Solvent Temperature Effects

<u>Temperature (F)</u>	<u>SPM reading</u>
100	2.15
50	2.10
30	2.10

TABLE II
Drying Air Effects

<u>RH</u>	<u>Temperature</u>	<u>SPM reading</u>
20 - 25%	70°	2.10
20 - 25%	120°	2.15
90 - 95%	70°	2.10
Bottled dry nitrogen		2.10

TABLE III
Atomizer Pressure Effects

<u>PSI</u>	<u>SPM reading</u>
2	3.35
4	3.25
6	3.25
8	3.30
10	3.30

TABLE IV
Drying Tube Length Effects

<u>Inches from atomizer output</u>	<u>SPM reading</u>
0	3.90
2	3.60
4	3.40
6	3.40
8	3.35
12	3.35
36	3.40

Application

The ease and simplicity with which dissolved residues can be determined in a variety of solvents make the Solvent Purity Meter useful for a wide variety of applications. The meter was originally developed as a production line tool for monitoring the purity of Freon TF solvent as used in a recirculating cleaning system. These systems are utilized for final cleaning and flushing of electromechanical components which are highly sensitive to small amounts of organic materials. Because the solvent in the recirculating system is reused over and over, it is necessary that the purity of the solvent be continuously monitored.

Many factors can contribute to the residue content of the solvent in such systems, such as the number of parts cleaned per unit time, the amount of contamination on the parts, and the ever-present possibility of a major accident or error, such as a worker cleaning a dirty tool or retrieving a lost part with the bare hand. The meter has been successfully used in conjunction with these systems. A major advantage is that a complete strip-chart record of the condition of the solvent during cleaning cycles is readily obtained.

A number of other applications have also shown the meter to be invaluable both for establishing the cleaning process parameters and for process control usage. A few examples should serve to illustrate this wide potential.

Example 1: The purification rate or recovery time of a particular recirculating cleaning system can be quickly established by contaminating the solvent in the system and then monitoring the solvent as it re-purifies. Also, the number of parts which may be cleaned per unit of time without exceeding the purification rate of the system can be easily established. Figure 9 shows a typical trace obtained from such an experiment.

Figure 9. Clean Down Trace from Recirculating Cleaner

Example 2: The optimum time cycle for ultrasonic cleaning can be determined by measuring the amount of contamination removed from a test part per unit time. It can be easily seen when no more contamination is being removed that further ultrasonic cleaning is accomplishing little if any good. It is also possible to quickly determine if different solvents are more effective for removing certain contaminants.

A typical experiment is shown in Table V.

Example 3: An indirect measurement of piece-part cleanliness can be made by measuring the increase in reading of a known pure solvent in which the part has been immersed or cleaned. This method has the advantage that the test in no way affects the part and can be made without disrupting the normal production cleaning sequence of the parts. For high-reliability component requirements this becomes extremely important. The disadvantage is that the test solvent must remove the contaminant in order for it to be detected.

TABLE V
Comparison of Solvents Experiment

Procedure

1. Dip test parts in No. 1544 Kester Solder Flux.
2. Place in oven and bake for 3 hours at 165°C.
3. Ultrasonically clean for 3 minutes in Freon TF.
4. Take SPM reading on beaker of clean trichlorethylene.
5. Ultrasonically clean part for 3 minutes in trichlorethylene.
6. Take SPM reading on beaker of trichlorethylene.

Results

Beaker reading before cleaning (trichlorethylene) 0.20.

Beaker reading after cleaning (trichlorethylene) 0.75.

Example 4: Maintaining the cleanliness of cleaning beakers, handling tools and storage containers is a constant production problem. The cleanliness of such containers is easily established by simply rinsing the container with a suitable solvent and noting any increase in reading.

Example 5: Certification of fresh drums or other stock of solvents can be rapidly done with the Solvent Purity Meter. The meter also has been used in connection with small Freon reclaiming stills to check the purity of the reclaimed solvent.

Example 6: The instrument has proved to be extremely useful as an educational tool for training production personnel. It is a simple matter to show a worker the effect of placing a fingerprint inside a cleaning beaker or grasping a clean part with the bare fingers.

Conclusions

The instrument described in this paper has become a valuable tool for monitoring and evaluating several phases of production cleaning processes. The minimum sensitivity of 1 to 10 parts per million, depending upon the solvent used, is adequate for this usage. This limitation is due almost entirely to the commercial photometer which is used. Salkowski and Werle used a special design high-sensitivity photometer and reported a minimum measurable contamination level of 0.1 ppm in trichlorethylene.

For measuring soluble residues the instrument is an excellent tool. It should be pointed out, however, that insolubles and particulates which do not affect the vapor pressure of the solvent are sensed only in concentrations of several orders of magnitude higher.

APPENDIX A
OPERATION AND CALIBRATION PROCEDURES FOR SOLVENT PURITY METER

Section 1
General Information

The Solvent Purity Meter (SPM) shown in Figure 6 measures the nonvolatile residue content of volatile cleaning solvents. Residues are detected by measuring the light-scattering characteristics of the aerosolized solvent droplets with an aerosol photometer. The relative residue content is read out on the meter on the light-scattering photometer.

Calibration of the meter is accomplished by measuring known solutions of typical low volatile contaminants dissolved into the solvent of interest. From the known solutions a calibration curve of residue content of the solvent in parts per million versus the photometer reading can be established.

The SPM measures very low concentrations of contamination; therefore, extreme care must always be exercised to prevent contamination of handling containers and the SPM sampling system from creating false readings. Good practice is to always take a reading on pure solvent before making a residue determination to assure that the glassware is clean.

Section 2
Sampling Methods

The sample of solvent to be tested may be introduced into the SPM by three different methods as shown in Figure 1.

1. The SPM may be connected to a continuous source of solvent to give a continuous reading of residue content. Solvent source must have enough pressure to keep the solvent flowing through the SPM.
2. The solvent sample may be pumped into the SPM by means of a syringe hand pump. The sample thus pumped into the SPM may be retained in the nebulizer while the reading is taken to give a residue content on a batch basis.
3. The solvent sample may be poured from a beaker or flask into the syringe with the plunger removed. This is the most direct method of introducing the solvent into the SPM and provides the minimum opportunity for contaminating the sample by the sampling system.

Section 3
Preparation for Use

Facilities Necessary

1. POWER. Power requirements of the Solvent Purity Meter are as follows:
SINCLAIR-PHOENIX PHOTOMETER requires 115 v AC, 60 cps, from a source capable of supplying 7 amp.
SAMPLER AND RECORDER unit requires a compressed air source capable of delivering 1 cfm at 25 psi for extended periods. In addition, electrical power is required for whatever recorder is used with the Solvent Purity Meter.
2. SPACE REQUIREMENTS. The overall dimensions of the Solvent Purity Meter are height, 42 inches; width, 26 inches; and depth, 22 inches.

Assembly and Connections

1. Unpackage and assemble tester in accordance with Figure 6.
2. Connect 6 foot long, 3/8 inch diameter length of clear PVC tubing between sample output of the sampler and recorder and photometer input.

Section 4
Operating Instructions, Equipment Setup

General

This section provides procedures for measuring the residue content of volatile cleaning solvents.

Preliminary Procedures

1. On photometer front panel, operate METER SWITCH to OFF.
2. On photometer front panel, operate RECORDER, HIGH VOLTAGE, MAIN POWER, PUMP, and LAMP switches to the OFF (down) position.
3. Connect power cable of photometer to AC power source and operate MAIN POWER switch to ON. MAIN POWER lamp of photometer lights. Note the time for future reference, to provide 10 minute warmup prior to performing procedures in paragraph 10.
4. Connect input air hose between compressed air source capable of delivering 25 psi for extended periods and AIR INPUT fitting on panel of sampler and recorder.
5. Rotate INPUT REGULATOR of sampler and recorder fully clockwise and then back it off three turns. Adjust external compressed air source to obtain indication of 20 psi on gage of compressed air source.

6. Slowly adjust INPUT REGULATOR of sampler and recorder clockwise until INPUT PRESSURE gage indicates 10 psi.
7. Rotate NEBULIZER PRESSURE control of sampler and recorder until NEBULIZER PRESSURE gage sampler and recorder indicates 5.
8. Rotate black DRIER VOLUME control until indicating ball centers on 25 marking of DRIER VOLUME flow gage.
9. Readjust INPUT REGULATOR, NEBULIZER PRESSURE, and DRIER VOLUME controls to overcome interaction, until INPUT PRESSURE gage indicates 10 psi, NEBULIZER PRESSURE gage indicates 5, and DRIER VOLUME flow gage indicates 25.
10. Verify that at least 10 minutes have elapsed from the time noted in paragraph 2.
11. On photometer, operate RECORDER, HIGH VOLTAGE, PUMP and LAMP switches to up position. LAMP, PUMP, HIGH VOLTAGE, and RECORDER lamps light.
12. On photometer, operate METER SWITCH to +.
13. If it is desired to use the recorder, perform procedures in paragraph 13.1 through 13.11; otherwise, proceed to paragraph 14.

Use With Recorder

- 13.1 Connect power cord of recorder to RECORDER receptacle on rear of photometer. Connect recorder signal cable to output jacks (negative to black and positive to red) on rear of photometer and operate switch adjacent to output jacks to ON.
- 13.2 On photometer front panel, rotate METER ZERO control to obtain 0 indication on AEROSOL CONCENTRATION meter. Recorder also indicates 0.
- 13.3 Loosen three knurled nuts securing rotatable light trap tube flange. Rotate photomultiplier assembly to the off-axis position (counterclockwise when looking from photomultiplier end of photometer), and temporarily clamp it in the off-axis position by tightening one of the knurled nuts. Withdraw the filter slide from position 1 (fully inserted) to position 2, as indicated when filter slide is felt to disengage and then reengage (first detent out from fully inserted position locates position 2).
- 13.4 Adjust two red controls at top of dual flow meters to balance the indications of the two flow meters while obtaining indications as near the yellow line on each flow meter as possible.
- 13.5 Rotate RELATIVE RECORDER SENSITIVITY control on front panel of photometer fully counterclockwise to 0.

- 13.6 Adjust black knob (gain) on rear of photometer for full-scale indication on photometer AEROSOL CONCENTRATION meter.
- 13.7 Adjust recorder for full-scale indication. Be sure recorder tracks linearly with AEROSOL CONCENTRATION meter.
- 13.8 Repeat procedures in paragraphs 13.6 and 13.7 as many times as necessary to allow for interaction of controls.
- 13.9 Loosen the single tightened knurled nut clamping the rotatable light trap tube flange. Rotate the photomultiplier assembly to the on-axis position (fully clockwise, when looking from photomultiplier end of photometer). Tighten the three knurled nuts securely.
- 13.10 Check that AEROSOL CONCENTRATION meter indicates 0 (recorder also indicates 0). If indication is not 0, repeat procedures in paragraphs 13.2 through 13.10.
- 13.11 Proceed to Section 5 which provides operating procedures for sample determination.

Use Without Recorder

14. Perform procedures in paragraphs 14.1 through 14.7 if recorder is not to be used.
 - 14.1 ON photometer front panel, rotate METER ZERO control to obtain 0 indication on AEROSOL CONCENTRATION meter.
 - 14.2 Loosen three knurled nuts securing rotatable light trap tube flange. Rotate photomultiplier assembly to the off-axis position (full counterclockwise when looking from photomultiplier end of photometer), and temporarily clamp it in the off-axis position by tightening one of the knurled nuts. Withdraw the filter slide from position 1 (fully inserted) to position 2, as indicated when filter slide is felt to disengage and then reengage detent (first detent out from fully inserted position locates position 2).
 - 14.3 Adjust two red controls at top of dual flow meters to balance the indications of the two flow meters while obtaining indications as near the yellow line on each flow meter as possible.
 - 14.4 On front panel of photometer, rotate SENSITIVITY controls (COARSE ADJUST and FINE ADJUST) to obtain full-scale indication on AEROSOL CONCENTRATION meter.
 - 14.5 Loosen the single tightened knurled nut clamping the rotatable light trap tube flange. Rotate photomultiplier assembly to the on-axis position (full clockwise, when looking from photomultiplier end of photometer). Tighten the three knurled nuts securely.
 - 14.6 Check that AEROSOL CONCENTRATION meter indicates 0. If it does not, repeat procedures in paragraphs 14.1 through 14.6.

14.7 Proceed to Section 5 which provides operating procedures for sample determination.

Section 5

Operating Instructions, Sample Determination

Procedures for Continuous Monitoring of Recirculating Systems

1. On front panel of sampler and recorder, rotate frosted end of SELECTOR VALVE to CONT. PUMP position. Be sure pump of cleaner unit (not part of Solvent Purity Meter) is on.
2. On front panel of sampler and recorder, rotate V1 and V2 valves to open position (knob aligned with tube).
3. Connect 3/8 inch diameter amber PVA sample return hose between bottom stem of nebulizer and cleaner.
4. Connect 1/4 inch diameter amber PVA sample input hose between SOLVENT IN stem of SELECTOR VALVE and pump output of cleaner.
5. Slowly operate controls of cleaner to pump solvent to Solvent Purity Meter. Watch the level of fluid in the reservoir of the nebulizer and adjust pump controls of cleaner so that the fluid stays beneath the lip of the atomizer tube itself, but not lower than 1/4-inch below the lip.
6. Adjust SELECTOR VALVE and V1 valve of sampler and recorder so that fluid level in nebulizer remains within approximately 1/4-inch of, but below, lip of nebulizer tube, while, at the same time, some fluid returns through V1. (This permits greater flow of fluid from cleaner, which affords better response time of the monitoring system.)
7. AEROSOL CONCENTRATION meter, and recorder if being used, indicate the mass concentration of impurities at any given time. Refer to calibration curve for the particular solvent being used.
8. To end monitoring, perform the following procedures: (1) Turn off input and then drain valves of cleaner, (2) Disconnect sample return hose from cleaner, (3) Disconnect sample-input hose from cleaner.

Procedures for Pour-Sample Analysis

1. On front panel of sampler and recorder, rotate frosted end of SELECTOR VALVE to OPEN VALVE position.
2. On front panel of sampler and recorder, close V2 valve and open V1 valve.
3. Connect sample-return hose between bottom stem of nebulizer and suitable drain vessel or outlet.

4. Extract plunger from syringe completely and pour approximately 20 cc of clean solvent of the type being used into the syringe. Reinsert the plunger with sufficient force to flush the solvent through the system, to clean tubing, valves, etc. of any residue.
5. Extract plunger from syringe again and pour approximately 20 cc of clean solvent of the type being used into the syringe. Close V1 valve. Reinsert plunger to pump approximately 10 cc of solvent into the nebulizer. Check indication of AEROSOL CONCENTRATION meter against calibration curve for the solvent, to insure cleanliness. Repeat procedures in paragraphs 4 and 5 as necessary to obtain indication which verifies system is clean.
6. Open V1 valve, pump remainder of solvent from system and allow system to drain.
7. Extract plunger from syringe and pour sample to be tested into the syringe (at least 15 cc). Close V1 valve, reinsert plunger and pump approximately 10 cc of solvent into the nebulizer.
8. Check indication of AEROSOL CONCENTRATION meter and refer to calibration curve for the solvent being tested to determine impurities in parts per million.
9. Open V1 valve and pump remainder of sample from syringe and allow system to drain.
10. Extract plunger from syringe again and pour approximately 20 cc of clean solvent of the type being used into the syringe. Close V1 valve. Reinsert plunger to pump approximately 10 cc of solvent into the nebulizer. Check indication of AEROSOL CONCENTRATION meter against calibration curve for the solvent, to insure cleanliness. Repeat procedures in paragraphs 4 and 5, as necessary, to obtain indication which verifies system is clean.
11. Open V1 valve, pump remainder of solvent from system and allow system to drain.

Procedures for Hand-Pump-Sample Analysis

1. On front panel of sampler and recorder, rotate frosted end of SELECTOR VALVE to SYRINGE PUMP position.
2. Close V2 valve and open V1 valve.
3. Connect 1/4 inch diameter sample-input hose between SOLVENT IN stem of SELECTOR VALVE and vessel containing pure solvent of type to be used.
4. Connect 3/8 inch diameter sample-return hose between bottom stem of nebulizer and suitable drain vessel or outlet.
5. Operate plunger of syringe to pump 20 to 30 cc of fluid through system to clean tubing, valves, etc. of any residue.
6. Close V1 valve. Pump approximately 10 cc of fluid into nebulizer.

7. Check indication of AEROSOL CONCENTRATION meter against calibration curve for the solvent, to insure cleanliness. Repeat procedures in paragraphs 1 through 7 as necessary to obtain indication which verifies system is clean.
8. Rotate selector valve to Cont. Pump position to allow sample-input hose to drain.
9. Open V1 and V2 valves, pump remaining sample from syringe and allow sample to drain into suitable receptacle.
10. Close V1 and V2 valves. Connect sample input hose to vessel containing solvent sample to be tested.
11. Rotate selector valve to syringe pump position.
12. Pump approximately 10 cc of fluid into nebulizer.
13. Check indication of AEROSOL CONCENTRATION meter against calibration curve for the solvent, to determine impurities in parts per million by volume. (Cleanliness of solvent used to clean component can be used to assist in determining the relative cleanliness of the component.)
14. Rotate selector valve to Cont. Pump position to allow sample-input hose to drain.
15. Open V1 and V2 valves, pump remaining sample from syringe and allow sample to drain into suitable receptacle.
16. Perform procedures in paragraphs 2 through 9 to flush system.

Post-Test Procedures

1. On photometer front panel, operate METER SWITCH to OFF.
2. On photometer front panel, sequentially operate the following switches to off (down) position: LAMP, PUMP, HIGH VOLTAGE, and MAIN POWER, and if applicable, RECORDER. All lighted lamps go out.
3. Turn off compressed air source. Disconnect compressed air source from Solvent Purity Meter, if desired.
4. Disconnect or withdraw sample return and sample input hoses from container or cleaner and loop hoses over photometer or disconnect them and store in desired location.

Section 6

Calibration Procedures

General

The basic idea of calibration of the Solvent Purity Meter is to dilute known clean solvent with a known amount of nonvolatile contaminant and to obtain photometer reading of various ratios of the two. These readings can be plotted on semilog paper to obtain a calibration curve. The curve, in turn, can be used to convert any photometer meter reading to ppm for that particular solvent.

To obtain a reasonable curve, dilutions of 1, 2, 5, 10, 20, 40, 60, 80, and 100 ppm are used. The ideal contaminant to be used to dilute the clean solvent would be the one most likely to be confronted in the cleaning process. However, as the contaminants are generally of similar volatility when compared with the solvent, lightweight machine oil, liquid solder flux, or something similar will give a good general curve.

To circumvent the need of measuring extremely small amounts of contaminant several times, it is recommended that a master mix of 100 ppm be made and that the lesser ratios be obtained by diluting the clean solvent with the master mix. This technique will reduce human error in measuring.

Equipment Necessary

- Supply of known clean solvent
- Supply of contaminant to be used
- Two each 1 liter flasks
- Two each milliliter beakers (250 ml)
- One each 500 ml graduate
- One each 50 microliter syringe
- One each 10 ml graduate
- Eight each 100 ml calibrated flasks (alternate 100 ml graduates)

Suggested Procedures

1. Prepare Solvent Purity Meter per Section 4.
2. Clean all glassware from the calibration kit with clean solvent.
3. Fill the two 1 liter flasks with clean solvent from the same source.
4. Measure 500 milliliters of clean solvent from either flask into the 500 milliliter graduate. Using a 50 microliter syringe, add 50 microliters of contaminant.
5. Clean solvent from one of the two 1 liter flasks may be poured into one of the 250 milliliter beakers to facilitate handling. Use the other 250 milliliter beaker for the solvent/oil mixture.

6. Using the 10 milliliter graduate, measure 1 milliliter of the mixture into the 100 milliliter flask.
7. Fill the flask to the 100 milliliter mark with clean solvent from either of the 1 liter flasks.
8. Pour 15 cc of the mixture in the 100 milliliter flask into the syringe on the Solvent Purity Meter. Obtain and record the photometer reading of the 1 ppm solution.
9. Flush the Solvent Purity Meter and the 100 milliliter flask with clean solvent.
10. Repeat steps 5 through 9, changing the ratio of solvent/oil solution from 1 to 2, 5, 10, 20, 40, 60, and 80 progressively. This will produce the ratio required i.e., 1, 2, 5, 10, 20, 40, 60, and 80 ppm.
11. Obtain and record the photometer reading of the mixture in the 500 milliliter graduate. This is the 100 ppm sample.
12. Plot the readings on semilog graph paper, with log being ppm and linear being photometer reading.
13. Draw curve from above plot. (Figure 7 shows a typical curve.)