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An American National Standard

Standard Test Method for NONVOLATILE RESIDUE OF VOLATILE CLEANING SOLVENTS USING THE SOLVENT PURITY METER[1]

This Standard is issued under the fixed designation F 324; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the determination of nonvolatile residue of volatile cleaning solvents using a solvent purity meter.

2. Applicable Document

E 300, Recommended Practice for Sampling Industrial Chemicals[2]

3. Summary of Method

3.1 A method is described for measuring the nonvolatile residue of cleaning solvents using a solvent purity meter. The residue is concentrated in aerosol form by evaporation of the more volatile solvents. The volume of the concentrated aerosol is passed by a forward light scattering photometer. Experimentally devised curves relating photometer output to nonvolatile residue concentration are used to obtain parts per million of nonvolatile residue content of the cleaning solvents.

4. Apparatus

4.1 The solvent purity meter consists of a sampler, JM-2000A photometer, and interconnecting hoses (Fig. 1). Figures 2 and 3 illustrate the sampler-and-meter unit and the photometer individually.[3]

4.1.1 *Sample Source*—Figure 3 provides a graphic representation of three different methods of sampling by the solvent purity meter. Of the three methods, the pour sample method offers least opportunity for concentration of the sample by the system.

4.1.2 *Sampler and Meter Unit*—This unit reduces the sample to a fine spray and introduces the atomized sample into a drying tube where most of the solvent is evaporated. The remaining air-suspension of solvent and residue droplets is drawn to the input of the photometer. The meter of the unit is utilized to provide a visual readout of the photometer measurement.

4.1.3 *Photometer*—The photometer measures the concentration of the residual matter in the sample and provides a readout by means of a meter with a linear scale. If the recorder of the sampler-meter unit is being used, the readout is also furnished to the recorder. The photometer provides readings with an accuracy of 1 ppm. The value of the meter reading is translated into terms of nonvolatile residue per million parts, by volume, through use of a curve established during calibration for the particular solvent concerned.

5. Sampling

5.1 Solvents to be analyzed for nonvolatiles should be sampled in accordance with Recommended Practice E 300.

6. Preparation of Apparatus

6.1 *Preliminary Operation*—With the solvent purity meter connected to a 100-V, a-c, 60-Hz, power source, adjust the tester for proper operation.

6.1.1 Adjustment of NEBULIZER REGULATOR valve for indication of 5 psi (34 kPa) on NEBULIZER PRESSURE gage assures

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[1] This method is under the jurisdiction of ASTM Committee F-7 on Aerospace Industry Methods.

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[2] *Annual Book of ASTM Standards*, Parts 29 and 30.

[3] A satisfactory source of this equipment is Jet Instruments, Suite 1717, First National Bank Bldg, East Albuquerque, N. M. 87108.

proper air pressure for operation of the nebulizer.

6.1.2 Adjustment of DRIER VOLUME valve for indication of 25 (approximately 3 psi (21 kPa)) on DRIER VOLUME gage provides correct volume of airflow to drying tube.

6.1.3 A recorder (which is not part of the solvent purity meter, though sometimes housed therein) may be used as desired to provide a record of solvent purity meter indications.

6.1.4 Adjustment of photometer METER ZERO control for 0 indication on CONCENTRATION meter "bucks out" the dark current of the photomultiplier.

6.1.5 Adjustment of two red controls of dual flowmeters allows proper flow of air into the optical chamber, that is, approximately 26 litres/min of sampling air and 3 litres/min of clean air. (Flowmeters are located in room-air-intake filter line and in photometer air outlet lines as indicated in Fig. 4.) Therefore, airflow through the optical chamber consists of the sample surrounded by a sheath of filtered room air. This sheath keeps out most of the sample air suspension from distributing through the photomultiplier assembly, since it may contain impurities which would deposit on component parts.

6.1.6 By swinging the photomultiplier assembly off-axis, the photomultiplier tube receives light directly from the lamp in the light-source housing through a ground glass lens. The luminosity represents a reference 100 % light transmission to the photomultiplier tube. Adjustment of the meter gain control CONCENTRATION meter (Position 1) for full-scale deflection, corresponds to 100 % light reception. The RECORDER SENSITIVITY control is used to adjust the full-scale setting of the recorder so that the recorder tracks linearly with the CONCENTRATION meter. When the photomultiplier assembly is rotated to the on-axis position, the photomultiplier tube receives negligible light and the CONCENTRATION meter indicates 0.

6.2 *Purity Test:*

6.2.1 *For Continuous Monitoring of Recirculating Systems:*

6.2.1.1 Set the controls of the cleaning unit (not part of the solvent purity meter) to pump the fluid sample through the SOLVENT IN stem of the V4 valve (which is in the OPEN position), out the bottom stem of the V4 valve and into the nebulizer.

6.2.1.2 Open V1 and V2 to allow excess fluid to bleed off and return to the cleaning unit.

6.2.1.3 Make adjustments to the V1 and V4 valves to provide maximum flow of fluid from the cleaner while limiting the level of fluid in the nebulizer reservoir to a position approximately 25 mm (½ in.) below the lip of the atomizing tube. If the fluid is permitted to rise much higher than 25 mm below the lip of the atomizing tube, the unit will flood and the liquid sample will pass into the drying tube, nullifying test indications. If fluid falls much below the 25-mm level specified, too little sample may be available for atomization, and once again, test results are nullified.

6.2.1.4 Return the unatomized portion of the sample to the cleaner, through open valve V1 and the sample-return hose.

6.2.2 *For Pour-Sample Analysis*—Pour at least 25 cm^3 of sample to be tested into the syringe. With V1 and V2 closed, operate the syringe to pump approximately 20 cm^3 of solvent into the nebulizer. Subsequent to test, open V1 to permit the unatomized portion of sample to drain out.

6.2.3 *For Hand-Pump Sample Analysis*—Use the syringe pump of the solvent purity meter to pump solvent to the SOLVENT IN stem of the V4 valve (which is in the OPEN position) and through the check valve into the syringe. With V1 and V2 closed, pump approximately 20 cm^3 of solvent into the nebulizer. Subsequent to test, open V1 to permit the unatomized portion of sample to drain out.

6.2.4 With the reservoir of the nebulizer filled to the proper level, feed compressed air (see AIR INPUT, Fig. 4) to the nebulizer and to the drying tube of the sampler and recorder unit. The nebulizer reduces the sample to a fine spray and introduces it to the drying tube where, if the sample is pure enough, it is transformed by evaporation into an almost completely gaseous state. Sample flow from the drying tube is greater in volume than that which the pump in the photometer can draw. Excess sample spills out the gas overflow, keeping the mixture at approximately ambient pressure for accurate sampling rates.

6.2.5 The sample moves into the highly illuminated optical chamber, where light is

excluded from the photomultiplier tube by a cone of darkness, as shown in Fig. 4.

6.2.6 If the sample is extremely pure, the solvent will be in an almost pure gaseous state, with negligible refractive or reflective qualities. Most contaminating residues are of relatively low vapor pressure and a small percentage of such residues changes the vapor pressure of the mixture quite significantly. Such impurities in the sample will retard vaporization in the drying tube.

6.2.7 Droplets, therefore, reach the optical chamber where light strikes them and reflects or refracts into the cone of darkness and passes on to the photomultiplier tube as indicated in Fig. 4. Excitation of the photomultiplier tube produces an electrical signal which is proportional to the amount of light it receives. The rate at which small droplets in the atomized sample are evaporated is inversely proportional to the amount of low-volatile material in the sample.

6.2.8 The signal from the photomultiplier tube is amplified and transmitted to the CONCENTRATION meter which provides a direct indication relative to the purity of the sample. If the recorder is being used, the recorder also indicates the degree of impurity. The value of the indication is translated into impurities in parts per million by volume, through use of a graph established during calibration of the tester.

6.2.9 Calibration of the meter is accomplished by measuring known solutions of typical contaminants of low volatility dissolved in the solvent of interest. From these known solutions, a calibration curve of residue content in parts per million by volume versus the indication of the CONCENTRATION meter can be established.

7. Calibration

7.1 *Calibration Interval*—The tester must have been calibrated at some time during the 6-month period preceding use, for the particular solvent in question.

7.2 *Invalidation*—Calibration is invalidated by shipment, removal from use and subsequent storage, expiration of prescribed calibration interval, replacement of glassware of sampler and recorder, or by maintenance of the photometer. Calibration is also required upon introduction of a new type of cleaning solvent to be tested.

7.3 *Sequence*—All calibration procedures must be performed in paragraph sequence.

7.4 *General Information:*

7.4.1 For calibration, introduce several samples consisting of various ratios of the applicable clean solvent diluted with a known volume of nonvolatile contaminant into the solvent purity meter. Plot readings that result from each of these samples on semi-log paper to obtain a characteristic calibration curve. Then use the curve during test operations to convert any given photometer indication to impurities in parts per million for that particular solvent. Figure 5 is typical for trifluorotrichloroethane.

7.4.2 For making the curve, take readings of dilutions of 1, 2, 5, 10, 20, 40, 60, 80, and 100 ppm by volume. The ideal contaminant would be the one most likely to be confronted in the cleaning process. However, as contaminants are usually of similar volatility when compared with the solvent, lightweight machine oil, liquid solder flux, or similar material will provide results that will form a good general curve.

7.4.3 To circumvent the need for measuring extremely small amounts of contaminant several times, make a master mix of 100 ppm and obtain the lesser ratios by diluting the clean solvent with the master mix. This technique minimizes human error during measurement.

7.5 *Calibration Procedures:*

7.5.1 Prepare the solvent purity meter in accordance with procedures in 8.1 through 8.1.9.7, except omit procedures in paragraph 8.1.8.11 or 8.1.9.7, depending on whether the recorder is used or not.

7.5.2 Clean all glassware of calibration equipment, using the solvent of the type concerned.

7.5.3 Fill the two 1-litre flasks with clean solvent from the same source.

7.5.4 Measure 500 ml of clean solvent from either 1-litre flask into a 500-ml graduate. Using a 50-microlitre syringe, add 50 microlitres of contaminant to the solvent in the 500-ml graduate. This provides a master mix of 100 ppm.

7.5.5 To facilitate handling, pour solvent oil mixture from the 500-ml graduate into a 250-ml beaker. For the same purpose, pour clean solvent from either 1-litre flask into the