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THE DETERMINATION OF TRACE RESIDUES
ON METAL SURFACES

by

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THE DOW CHEMICAL COMPANY

ROCKY FLATS DIVISION

GOLDEN, COLORADO

U.S. ATOMIC ENERGY COMMISSION CONTRACT AT (29-1)-1106

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THE DETERMINATION OF TRACE RESIDUES
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ABSTRACT

Analytical methods have been developed to characterize or identify residues removed from metal surfaces. The results are quantitative for several compounds; such as, certain hydrocarbons, inorganic chlorides, water, and amine hydrochlorides. Solvents are used directly as surface rinsing agents or to rinse cold traps used on high vacuum cleaning systems. A method of determining water in halogenated solvents in the near infrared region is also reported. A unique method of drying the reference solvent using a water-solvent equilibrium is described.

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INTRODUCTION

Trace quantities of organic residues, inorganic chlorides, or water on a metal surface are undesirable in many applications, particularly if the surface is radioactive or subject to high energy radiation. Radiolysis of surface contaminants can produce deleterious gases corrosive to the metal surface^{(1),(2),(3)}.

The methods are more informative than current residual gas analysis techniques to determine how clean or the quantity of residue on a surface^{(4),(5),(6)}. Direct infrared reflectance measurements for macro quantities of organics are available. However, the direct method lacks sensitivity and does not provide the necessary quantitative data.

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- (1) S. C. Lind, Radiation Chemistry of Gases, (Reinhold Publishing Corp., New York, N.Y., 1961).
 - (2) F. A. Bovey, The Effects of Ionizing Radiation on Natural and Synthetic High Polymers, (Interscience Publishers Inc., 1958) pp 37-47, pp 143-145.
 - (3) A. O. Allen, The Radiation Chemistry of Water and Aqueous Solutions, (D. Van Norstrand Co., 1961).
 - (4) J. H. Bloom, C. E. Ludington, and R. L. Phipps, "The Omegatron vs. a Sector-Type Mass Spectrometer for Residual Gas Studies", *Nuovo cimento, Suppl 1* (2), (1964), pp 442-451.
 - (5) J. R. Bailey, "Residual Gas (Mass) Spectra in High and Very High Vacuum Systems", *Nuovo cimento, Suppl 1* (2), (1963) pp 494-519.
 - (6) H. G. Heide, "Composition of Residual Gas in a Electron Microscope", *Z Angew. Phys.* 17 (2), (1964), pp 70-72.

SUMMARY

Methods have been developed to evaluate metal degreasing processes, handling and assembly operations, storage containers and conditions, and the efficiency of vacuum baking as a cleaning method for various components. These methods, based on solvent rinse techniques, are not absolute but provide a convenient means of measuring the relative cleanness of one metal surface as compared to another surface exposed to a different environment.

Metal surfaces, varying from films to those of large immovable objects, including stainless steel, mild steel, Monel, silver, gold, copper, brass, aluminum, uranium, beryllium and plutonium have been rinsed with solvents for surface and occasionally imbedded contaminants. Conventional analysis of the soluble residues were made using infrared, emission, x-ray, or other spectroscopic techniques.

When 40 milliliters of carbon tetrachloride was used to rinse a 1000-square centimeter surface, the smallest quantity of a straight chain aliphatic hydrocarbon measurable was found to be 30 micrograms. If water was used as a solvent on the same sized surface, as little as 1.5 micrograms of chloride could be measured in 15 milliliters of water. The chloride method used was the ferric ammonium-mercuric thiocyanate colorimetric method⁽⁷⁾. When acetone was used as a solvent, it was possible to measure 40 micrograms of diisopropylamine hydrochloride rinsed from a metal surface.

Methods are described which use anhydrous carbon tetrachloride to determine the water content of a gaseous system. Curves are

(7) J. G. Bergmann, J. Sanik, Jr., "Determination of Trace Amounts of Chlorine in Naptha", Anal Chem 29, (1957), p. 241.

presented which show that chlorinated solvents exhibit an equilibrium with the moisture in an ambient gas, i.e., the water content of the halogenated solvent depends upon the humidity of the system.

EXPERIMENTAL

Apparatus

Perkin Elmer Model 137 Infracord

Perkin Elmer Model 21

Perkin Elmer Model 137G Infracord

Emission Spectrograph

X-ray Diffraction Unit

Carbon-Hydrogen Analyzer

Alpha and Gamma Spectrometers

Vapor Pressure Osmometer

Vacuum Drying Oven - Modified to serve as a filter air
room temperature evaporator.

Reagent Grade acetone, carbon tetrachloride, carbon disulfide,
and water.

Miscellaneous hypodermic needles, Teflon-ware, cold traps,
heat lamps, pellet presses.

Direct Rinsing of Metal Surfaces

The problem of analytically rinsing a metal surface for a residual organic compound can be resolved into two major propositions:

1. The solvent must be capable of dissolving the contaminants.
2. The volume of the rinsing solvent must be as small as possible.

The analytical method can then be extended to its limit by choice of cells and differential techniques using spectrophotometric methods. For example, a small diameter tube can be rinsed with a few microliters of solvent which can be directly transferred into a small volume infrared cell. A large object requires several milliliters of rinsing solvent with transfer to a corresponding larger volume cell for maximum sensitivity.

Several solvents may be used successively on a single surface. Generally, a non-polar type is followed by a polar-type solvent. Each solvent rinse is examined separately for residues.

Solvents

All solvents are contained in glass reagent bottles (dark glass) except distilled water, which is stored in polyethylene. Each bottle of solvent must be checked for spectral purity at wavelengths for specific functional groups. The residue from 100 ml of solvent, after evaporation at room temperature using filtered air, must be less than 0.1 mg⁽⁸⁾. Any residue from this test is dissolved in 20 microliters of fresh solvent and added to 300 mg of potassium bromide. The excess solvent is evaporated at the solvent's boiling point. The potassium bromide is pressed to a pellet whose infrared spectrum is taken. This spectrum must be the same as that found for a pure potassium bromide pellet in order for the solvent to be considered residue free.

Experience has shown that each bottle of solvent must be checked since bottles of the same lot may contain small traces of organic contaminants (generally a plasticizer or a silicone grease). Reagent grade solvents are used exclusively. Inhibited solvents

(8) An evaporator oven can be made by altering a cylindrical vacuum oven. A heat sink is provided by 2-inch thick aluminum with holes drilled for nesting the beakers. A constant supply of filtered air flows into each beaker. At an oven temperature of 40-50°C, the cooling of the solvent tends to keep the residue at about 30°C.

should be avoided. The solvent should be selected for clear windows in the infrared region, at frequencies which are capable of confirming the presence of a specific compound or functional group.

Cells and Infrared Sodium Chloride Plates

Commercial cavity infrared cells (Barnes Engineering Company, Wilton, Connecticut) are used for some determinations. Other cells have been made in the laboratory. A convenient cell of any reasonable path length can be constructed from 25-mm diameter glass tubing. Suitable windows are then glued to each end with an epoxy resin. A standard glass stopper is provided for filling.

The outer surfaces of the cells are kept free of radioactive contamination by careful transfers and frequent wiping with solvents and absorbent tissue. The salt plates are easily decontaminated by rinsing in 95% alcohol and repolishing on a felt cloth, using 600 grit aluminum oxide. The plates are polished with an alcohol slurry of Barnsite compound (finely powdered rare earth oxides).

Silica cells are used in the near infrared region. Pyrex cells are used in the visible spectral region.

Special Equipment

Delicate parts and accessories can be rinsed without risk of damage by using epoxy resin coated tongs and tweezers. Glass syringes are used almost exclusively for liquid transfers. Needles of over 4-foot length are available in Teflon or stainless steel. The use of those needles is desirable to rinse

the interior of small tubes which may be twisted into shapes not readily rinsed by conventional methods. Teflon gaskets, beakers, "O" rings, etc., are not subject to solvent attack and are suitable for this type work.

Cold traps for vacuum systems can be made either from metal or glass. All valves used in such a system must be either Monel or stainless steel bellows type with a Kel-F seat (The M. W. Kellogg Company - Trifluorochloroethylene). Stainless steel ball-type Teflon-seal valves are also available which will hold a vacuum at 10^{-6} Torr. (Both valves are available through Hoke Incorporated). Valves with rubber gaskets (including Viton compounds) must be avoided since solvents readily remove plasticizer from the organic materials. Valves with copper bellows are undesirable, since the copper is subject to attack by amine salts and HCl vapors.

Typical Procedure for Determining Surface Contamination Using Carbon Tetrachloride and Acetone Rinsing

1. Rinse the transfer syringe, rinse container and tongs with the same quantity of carbon tetrachloride as will be used to rinse the part. Transfer into an infrared cell and check the absorbance at 3.4 (C-H) and 5.8 (C=O) microns region. If no absorbance is noted, transfer the solvent to a clean tared beaker. This will serve as the "equipment-blank" (the process must be repeated until no C-H, or C=O can be detected). The quantity of solvent depends upon the size of the object one is rinsing.
2. Transfer the metal object into the rinse container and add sufficient solvent to adequately and repeatedly rinse the surface. Grasp the piece with the epoxy-coated tongs and wet the entire surface several times

by swirling the solvent and rotating the part in the solvent. Transfer the solvent into the infrared cell and check the absorbance for C-H at 3.4 microns and C=O at 5.8 microns. Repeat the process on the same piece until blank absorbance values are obtained on the carbon tetrachloride after rinsing the part. The total C-H or C=O can be reported as milligrams of an oil or a carbonyl-type compound. The rinses are then composited, added to a clean tared beaker and evaporated at low temperature (about 30°C) along with the "equipment blank" solvent from paragraph 1 on preceeding page. See "Solvent" section for details. The equipment blank and part residues weights are obtained by weighing the respective beakers.

3. The residue obtained from the carbon tetrachloride rinse is then identified by any available method. For oily-type organics, infra-red analysis is an excellent method and is done by placing the residue directly on a salt window to utilize the greatest flux of the infrared beam. Using scale expansion, as little as 0.1 mg of a residue can be characterized or identified. After identification, the residue can be rinsed from the window, dissolved in a suitable solvent, and quantitative results obtained. Radioactive contaminated samples can be handled in the same manner without serious decontamination problems. If the quantity of residue is large (10 mg or more), carbon, hydrogen, nitrogen analyses can be made, or molecular weights can be determined using a vapor pressure osmometer. If metallic residues are present, micro emission spectrographic methods are used. If microscopy examination indicates a crystalline compound, a few crystals can

be ground and examined in a capillary tube by x-ray diffraction. If the residue contains radioactive materials, these can be dissolved in acids or fused with suitable fluxes for subsequent solution. The radioactive elements can be identified by alpha and gamma spectrometry.

4. The same part can then be rinsed in a polar solvent-acetone in this case. Equipment blanks are included to validate the results. Two acetone rinses are usually sufficient to remove all residue. After evaporation of the acetone, the residue is weighed. Identification of the residue is by the previously mentioned techniques. The KBr infrared pellet technique is excellent, since certain compounds (i.e., amine salts, nitrates, carbonates and nitrides) can be determined quantitatively as well as qualitatively. Cellulose lint and talc can also be identified using the KBr pellet technique.

DISCUSSION

Background

The solvent approach to determine the cleanness of metal surface was used at Rocky Flats Plant for several reasons:

1. Metal surfaces can be contaminated with a known amount of a substance and the recovery determined experimentally.
2. Infrared techniques had already been extended to the ppm range for oily and ester-type compounds. Calculations indicated very small significant quantities could be detected on a surface.
3. Experiments proved that a residue weighing as little as 0.1 mg could be identified by infrared or x-ray diffraction methods.

Solvent rinse methods have been applied directly to metal surfaces, and with slight modification to the rinsing of cold traps from various gas and/or air streams and vacuum systems.

Precautions have to be observed to prevent erroneous results. Solvents must be carefully checked. Containers must have glass, metal or Teflon seals. Paint fumes and other organic vapors can contaminate solvents even when far removed from the rinsing operations. The solvents used for analytical rinsing must be checked for such contamination before and after the rinsing steps. The same precautions must be taken for equipment used in the rinsing steps.

Determination of CH on Aluminum, Uranium and Plutonium Surface

Typical data on rinses from three types of metal (Al, Pu, U) for hydrocarbons are shown in Table 1. The values were obtained using a 10-cm NaCl window cell after each surface was rinsed with 40 ml of carbon tetrachloride. The results are considered valid since the pre- and post-blanks returned to the detection limit (<0.03 mg). The organic contamination is reported as milligrams of C-H hydrocarbon using Shell Vitrea as a reference (C 20-21 straight chain alkane--Shell Petroleum Company). The 3.4-micron C-H stretching band was used for the determination. The standards for the absorbance curves were obtained by adding known quantities of Shell Vitrea to reagent grade carbon tetrachloride. The absorbances were then determined by infrared measurement of the standards in a 10-cm cell with NaCl windows. The average recovery for this method was 100%, based on rinses of surfaces contaminated with known quantities of the oil, using an oil-carbon tetrachloride mixture sprayed on the surface with a hand atomizer (see Table 2).

TABLE 1

PARTS RINSED FOR C-H WITH CCl_4 Results reported as mg of Shell Vitrea (C_{20} - C_{22} Alkane)

Type Metal	Blank	Rinse No. 1	Rinse No. 2	Rinse No. 3	Total
Pu	<0.03	0.10	0.05	0.03	0.18
Al	<0.03	0.11	0.07	<0.03	0.18
U	<0.03	0.24	0.09	<0.03	0.33

TABLE 2

RECOVERY OF A KNOWN QUANTITY OF RESIDUE FROM VARIOUS SURFACES

Type Spike	Surface	Weight Added	Recovered	% Recovered
C-H*	Plutonium	1.5 mg	1.6 mg	107
C-H	Plutonium	1.5	1.5	100
C-H	Plutonium	1.0	0.96	96
C-H	Plutonium	1.0	1.02	102
C-H	Plutonium	1.0	0.97	97
DIA·HCl**	Uranium	0.14	0.12	86
DIA·HCl	Uranium	0.12	0.07	56
DIA·HCl***	Glass	0.16	0.15	94
DIA·HCl	Glass	0.12	0.15	125
Cl-****	Plutonium	0.42	0.43	102
Cl-	Plutonium	0.42	0.48	114
710 Silicone Oil	Extracted 150 gm Pu with CS_2	0.55	0.42	76

*Shell Vitrea (C_{20} - C_{22} Alkane) added, diluted with CCl_4 and hand atomizer.

**Diisopropylamine hydrochloride added diluted with acetone and hand atomizer.

***Added diluted with acetone with a syringe.

****Added as dilute HCl with a glass syringe.

Determination of Silicone Oils from Metal Surface

Carbon disulfide is used as an analytical solvent for silicone oils. The techniques are the same as used with carbon tetrachloride. Care must be taken because of the fire hazard. The limit of detection when using 20 ml of solvent on a surface is 0.1 mg. See Table 3. The method had been used to determine sub-surface contamination on plutonium of silicone from a heat treating bath. Using a 100-gram sample and extracting the dissolved metal (HCl) with carbon disulfide, a detection limit of 1 ppm of silicon oil (Dow Corning 550 or 710) can be obtained (naturally, the surface oil must be completely removed prior to dissolution and extraction). A 76% recovery (Table 2) was made on a synthetic spike in 150 grams of dissolved plutonium metal with 0.6 mg of the Dow Corning Silicon 710 oil.

Determination of Chlorides on Metal Surfaces

Water was found to be the only suitable solvent for inorganic chloride (Cl^-). Ethyl alcohol, acetone, or acetone containing three percent water would not remove chloride quantitatively from a plutonium surface. Since water attacks some metal surfaces, destroying their integrity, the test may be destructive. Again, blanks are run at the beginning and finish of a part rinse or series of parts. The Cl^- content of the equipment blank must be the same as found in the reagent water. The Cl^- content of the rinse water is determined by the standard ferric ammonium sulfate-mercuric thiocyanate method(7). A typical series of data for Cl^- on plutonium surfaces is shown in Table 4. Forty-ml of water was used for the rinse. The recovery using this technique following a standardized hydrochloric acid spike was 108% as shown in Table 2.

TABLE 3
THE DETERMINATION OF TRACE RESIDUES ON METAL SURFACES

Rinse Solvent	Compound or Functional Group Desired	Peak Microns	Window	Thick-ness	Make	Capacity (ml)	Detection Limit
Acetone	DIA.HCl***	4.03, 7.19	KBr Pellet	300-mg			0.04-mg
Acetone	Water	1.92	Silica	10-mm	Beckman	3.5	0.02%
Acetone	Water	1.92	Silica	20-mm	Beckman	6	0.005%
CS ₂	(C-X) Trichloroethylene	10.7	NaCl	5-mm	CIC*	0.5	0.002%
CS ₂	(C-X) CCl ₄	12.7	NaCl	5-mm	CIC	0.5	0.001%
CS ₂	(C-X) Freon TF	8.6	NaCl	12-mm	RF**	4.0	0.0005%
CS ₂	(C-H) Hydrocarbon Oil	3.4	NaCl	0.1mm	CIC	0.02	0.03%
CS ₂	(CH ₃ -Si) Silicone 550 Oil	7.9	NaCl	12-mm	RF	4	0.0005%
CS ₂	(CH ₃ -Si) Silicone 710 Oil	7.9	NaCl	12-mm	RF	4	0.0008%
CS ₂	(C-H) Hydrocarbon	3.4	NaCl	12-mm	RF	4	0.001%
CCl ₄	(C-H) Hydrocarbon Oil	3.4	NaCl	5-mm	CIC	0.5	0.002%
CCl ₄	(C=O) Dioctylphthalate	5.8	NaCl	5-mm	CIC	0.5	0.002%
CCl ₄	Water	2.6	NaCl	5-cm	RF	20	0.0003%
CCl ₄	(C-H) Hydrocarbon Oil	3.4	NaCl	10-cm	RF	40	0.0001%
CCl ₄	(C=O) Dioctylphthalate	5.8	NaCl	10-cm	RF	40	0.0001%
Freon TF	(C-H) Hydrocarbon Oil	3.4	NaCl	12-mm	RF	4	0.0005%
Water	Cl ⁻ (Inorganic)	0.460	Silica	10-mm	Beckman	3.5	0.00001%

*Connecticut Instrument Corporation, Wilton, Connecticut. (Barnes Engineering)

**Cells made at Rocky Flats Plant, Golden, Colorado.

***Diisopropylamine Hydrochloride.

TABLE 4

PLUTONIUM PARTS RINSED WITH 40-ml WATER FOR CHLORIDE (mg Cl⁻)

Sample	Equipment Blank	Rinse No. 1	Rinse No. 2	Total Cl ⁻
1	0.02	0.48	0.16	0.62
2	0.02	0.33	0.10	0.41
3	0.01	0.27	0.13	0.39
4	0.03	0.28	0.11	0.36
5	0.02	0.42	0.13	0.53

Determination of Diisopropylamine Hydrochloride (DIA·HCl)

Certain commercial grades of trichlorethylene used for ultrasonic or vapor degreasing operations contain the inhibitor diisopropylamine. Hydrogen chloride is produced by the decomposition of trichloroethylene and reacts in either vapor or liquid phases to form the salt-diisopropylamine hydrochloride (DIA·HCl). When trichloroethylene is used as a solvent for metal degreasing, there is good probability that the surface is contaminated with the amine salt during the cleaning process. Acetone rinsing of such surfaces is used to remove the amine salt for subsequent identification and quantitative measurement. Analytical results for this determination of DIA·HCl on plutonium surfaces is presented in Table 5.

The method used follows: A blank was run on the rinse equipment prior to rinsing the parts. Then, each part was rinsed two times and each rinse composited. An equipment blank was then run after the part rinsing. The quantities were determined by infrared measurement at 4.03 microns of a KBr pellet (300 mg)

TABLE 5

ACETONE RINSE OF PLUTONIUM SURFACES FOR
DIISOPROPYLAMINE HYDROCHLORIDE (DIA·HCl)

Sample	DIA·HCl (mg)
Equipment Blank	<0.04
# 1	0.24
# 2	0.36
# 3	0.40
# 4	0.32
Equipment Blank	<0.04

containing the evaporated residues. The working curve was prepared by measuring the absorbance at 4.03 and 7.19 microns of 300-mg KBr pellets containing known quantities of the DIA·HCl salts.

The percentage recovery of the acetone rinse and infrared method was checked by applying the technique to glass and uranium surfaces which had been sprayed with known amounts of the amine salt dissolved in acetone. Based on these tests, the data of Table 2 are apparently biased 30% low. No correction was applied since there is a chance that some of the DIA·HCl was lost in the spraying step. The amine salt is capable of forming an aerosol because of its small crystal size.

Determination of Water in Acetone

Figure 1 shows an analytical curve for the determination of water in acetone using the 1.92-micron combination OH band.

Figure 1
Water in Acetone

The sample is compared in a 10-mm silica or Pyrex cell against a matched reference cell containing anhydrous acetone. The dry acetone is obtained by contacting the acetone with Linde 4A molecular sieves. (Linde Company, Division of Union Carbide).

This method is very useful in determining the quantity of water removed from a closed system by vacuum pumping. To accomplish this, it is only necessary to insert a cold trap immersed in trichloroethylene-dry ice into the system to freeze the water removed. The trap is then removed from the system, warmed and rinsed with anhydrous acetone. Water is then determined at 1.92 microns. The technique also is excellent for determining the water content of a gas. A known volume of gas is passed through a cold trap and the water frozen out. The trap is then warmed and rinsed with dry acetone and the quantity of water determined. This method is not affected by organic vapors which can affect instruments whose operation depends upon conductivity of a P_2O_5 film. Since the vibration of the water molecule is being measured, the method can serve as a reference for other water-measuring instruments. Table 6 presents data on the recovery of water injected into an air stream and recovered in a cold trap (1/4-inch U tube). The water was injected into a dry gas system which had a flow rate of 500 cc/minute. A gas chromatograph syringe with a rubber septum was used. The flow was maintained for 10 minutes at the 500 cc/minute rate. Also included in Table 6 is a recovery from the evacuation of a 4-liter volume which contained air with 45 mg of water vapor (45% R.H.). In this case, a stainless steel 50-cc volume down-delivery-double-walled trap was used. From these results, one can assume recoveries from an air or evacuation to be at least 96% of the actual quantity of water in the system.

TABLE 6

RECOVERY OF WATER FROM AN AIR STREAM OR EVACUATION

System Description	Added	Found	% Recovery
500 ml/min flow-water added through septum*	5.0 mg	5.0 mg	100
500 ml/min flow-water added through septum*	5.0 mg	4.6 mg	96
500 ml/min flow-water added through septum*	5.0 mg	5.0 mg	100
Evacuated 4-liter volume**	≈45.0 mg***	44.6 mg	≈100

*Simple 1/4-inch U tube trap.

**50-ml volume doubled-walled stainless steel down delivery trap.

***Calculated 4-liters air at 45% R.H. ≈ 45-mg H₂O. Tri-chloroethylene-dry ice mixture was used for trap coolant.

Determination of Water in Carbon Tetrachloride and Trichloroethylene

The absorbance curve for determining the water content of carbon tetrachloride is shown in Figure 2. The measurements were made in an 8-cm cell using air as the reference, and measuring the 2.6-micron peak (water fundamental OH vibration). Figure 3 is a curve for the determination of water in trichloroethylene using a 10-cm cell at 1.92-micron peak (differential analysis against air-dried trichloroethylene). During the early investigations of the water content of carbon tetrachloride, it was observed that the absorbance at 2.6 microns would become less as a freshly opened reagent bottle aged. The relative humidity was 8% at the time. Further investigations were made by subjecting carbon tetrachloride to air at various

Figure 2. Water in Carbon Tetrachloride

Figure 3. Water in Trichloroethylene

humidities. The results of this study are shown graphically in Figure 4. The curve (Figure 4) defines the relationship of the water content of carbon tetrachloride as a function of the humidity and shows that the water content of carbon tetrachloride varies from zero content to saturation in a linear mode. In later experiments it was found that trichloroethylene, Freon TF, straight chain hydrocarbon oils, and an olein-type oil have the same relationship to water. The plots of water concentration against humidity are not shown for these compounds. Chlorinated hydrocarbons can be dried by contact with anhydrous air. This method has been used to dry trichloroethylene for use as a reference liquid for the determination of water in trichloroethylene as previously described. The dried reference trichloroethylene is stored in the matched cell in a desiccator over a suitable desiccant.

Dry carbon tetrachloride can be injected into a closed system containing a gas. The water content (or humidity) of the gas can be determined by measuring the water content of the carbon tetrachloride by infrared comparison against the dry solvent (after equilibrium is reached).

The Determination of Other Compounds or Functional Groups

Freon TF can be used as a solvent for the determination of C-H hydrocarbon.

Carbon disulfide is an excellent solvent and has a clear window in the 9-12 micron region. Therefore, carbon disulfide is a good solvent for residues containing C-X linkages such as the Fluorlube-type oils (Fluocarbons). One may also rinse a part with carbon disulfide following a halogenated solvent to detect small quantities of residual solvent of the surface (see Table 3).

Figure 4
Water Content of Carbon Tetrachloride
at Various Humidities

Acetone will readily dissolve many metal nitrates. It is possible to rinse a uranium surface with acetone and determine quantitatively the amount of $\text{UO}_2(\text{NO}_3)_2$ removed from the surface. The nitrate is determined quantitatively in a KBr pellet.

Aluminum surfaces rinsed with alcohol, water and acetone show a large quantity of residue. So far the compounds have not been identified.

Table 3 shows the parameters, wave-lengths, cells, and detection limits on ten compounds, using 5 solvents which are used at Rocky Flats Plant.