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Post Solder Semi-Aqueous Cleaning Handbook

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Developed by the Semi-Aqueous Cleaning Task Group (5-31b) of the
Cleaning and Coating Committee (5-30) of IPC

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Users of this standard are encouraged to participate in the
development of future revisions.

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Acknowledgment

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Post Solder Semi-Aqueous Cleaning Handbook

1 INTRODUCTION

1.1 Scope This handbook addresses semi-aqueous cleaning of electrical/electronic assemblies, parts and application tools after soldering. Semi-aqueous cleaning or hydrocarbon surfactant cleaning is a process in which flux residues and other contaminants are removed from printed wiring assemblies by first washing the assemblies in an organic solvent and then rinsing the organic solvent from the assemblies with water. The other parts and application tools are similarly cleaned using this same process or the emulsion variant of it. The semi-aqueous process specifically does not include cleaning with aqueous solutions of saponifiers or detergents.

1.2 Purpose The purpose is to provide a complete picture of the semi-aqueous cleaning process so that potential users will better understand the technology, and so seasoned users can operate their semi-aqueous cleaning processes properly. The document has been developed for those involved in printed wiring assembly (PWA) operations. It is intended to describe all aspects of semi-aqueous cleaning including cleaning mechanism, cleaning agents, cleanliness testing, environmental management, safety, equipment, and process control.

Two different types of semi-aqueous cleaning agents are described in this handbook. The first employs a water-insoluble organic solvent (Type I), while the second uses a water-soluble organic solvent (Type II). After the wash stage, the PWAs in either process are rinsed in pure water and then dried. The document includes information about composition, physical and chemical properties, waste treatment and compatibility. This document revision includes only commercially available cleaning agents.

1.3 Introduction Printed wiring assemblies that are not clean may fail because of corrosion or other failure mechanisms caused by residues from the assembly soldering process and other parts of the manufacturing process. For this reason, many electronics assemblers clean the assemblies after soldering. While not all assemblies are cleaned, most used in high reliability applications are cleaned. The semi-aqueous process was successfully introduced for surface mount cleaning applications to replace the ozone-depleting solvents (1,1,2-trichlorotrifluoroethane (CFC-113) and 1,1,1-trichloroethane (TCA)) that were being phased out.

The cleaning options now available to users include no-clean technology, non-ozone depleting solvent cleaning (IPC-SC-60A), semi-aqueous cleaning (this handbook), or aqueous cleaning (IPC-AC-62A). The semi-aqueous clean-

ing alternative combines solvent cleaning with an aqueous rinsing process. It has the advantage of effectively removing both water-soluble and water-insoluble contaminants. Semi-aqueous cleaning was developed to offer effective, environmentally preferred removal of rosin and synthetic activated (SA) flux and paste residues. Certain international authorities, such as the United Nations Environment Programme (UNEP) Solvent Technical Options Committee (STOC) to the Montreal Protocol, use the preferred synonymous term "hydrocarbon-surfactant" (HCS, HC/S or HC-S).

As electronic assemblies get more and more complicated, cleanliness is becoming increasingly important. For maximum cleanliness there are many factors that must be taken into consideration. Assemblies must be designed for cleaning, assembly fluxing and soldering processes must be controlled, cleaning machines must be effective, and fluxes/pastes must be compatible with the cleaning process.

1.4 Terms and Definitions

Semi-Aqueous Cleaning – A process in which flux residues and other contaminants are removed from printed wiring assemblies by first washing the assemblies in an organic solvent and then rinsing the organic solvent from the assemblies with water. Type I uses a water-insoluble organic solvent, while Type II uses a water-soluble organic solvent. The semi-aqueous process specifically does not include cleaning with aqueous solutions of saponifiers or detergents. The reader should see IPC-AC-62, Post Solder Aqueous Cleaning Handbook, for a discussion of aqueous cleaning.

Alcohol, Non-Linear – Non-Linear alcohols are derived naturally from spent rice hulls, sugar cane or corncobs. When referring to this alcohol as non-linear, the term denotes that this compound has a ring structure. Such organic compounds have low molecular weights and contain both an alcohol group and an ether linkage. The alcohol and ether groups enhance this compound's effectiveness in removing polar soils and provide hydrogen bonding sites which allows the compound to be completely soluble in water.

Alcohol, Synthetic – The synthetic alcohol derived for semi-aqueous electronic cleaning processes has both the ether group and the alcoholic hydroxyl group in its molecular structure which forms intramolecular and intermolecular hydrogen bonds and exhibits hydrophobic (water-insoluble) and hydrophilic (water-loving) properties. This molecule has low toxicity, low odor and a flash point ranging from 66 - 77°C [150 - 170°F].

BOD – Biological Oxygen Demand, the amount of oxygen required for the biochemical degradation of organic material under controlled test conditions.

Carbon Adsorption – A technique to remove impurities from liquid or gas streams by adsorbing the contaminants in the stream on activated carbon.

CFC – Chlorofluorocarbons, a class of compounds composed entirely of carbon, chlorine, and fluorine atoms.

COD – Chemical Oxygen Demand. The amount of oxygen required to oxidize organic materials to carbon dioxide and water, reported in mg of oxygen/liter of tested solution.

Combustible – This term is applied to liquid materials that have flash points greater than 38°C [100°F] by the closed cup method.

Decanting – The process separating two immiscible liquids by drawing a top layer of liquid material off of another layer of liquid without disturbing the lower liquid layer.

Dragout – Liquid cleaning agent or water which adheres to circuit assemblies or transport devices and which is carried from one section of a cleaning machine into another section of the machine.

Emulsion – A mixture of two or more immiscible liquids, such as oil and water, which is stable and uniform under conditions of use in the cleaning process.

Esters – A class of organic compounds formed by the reaction of alcohols with organic acids.

Ketones – A class of organic compounds in which the carbonyl group is attached to two alkyl groups. Ketones are used as solvents, and some common examples are acetone and methyl ethyl ketone.

Hydrocarbon – An organic compound containing only hydrogen and carbon.

Hydrocarbon-Surfactant – A hydrocarbon which exhibits hydrophobic (water-insoluble) and hydrophilic (water-loving) properties. It alters the wettability of surfaces and hence, is useful as a cleaning agent.

Hydrocarbon-Surfactant (HCS, HC/S, HC-S) Cleaning – An alternative nomenclature, synonymous with semi-aqueous cleaning, preferred by some international authorities.

Ozone Depleting Chemicals – Chemicals capable of reacting with and destroying ozone in the stratosphere.

Propylene Glycol Ether – Propylene Glycol Ethers are oxygenated hydrocarbons that provide formulation flexibility due to the wide range of chemical structures available. Selection of the structures that fit the evaporation rate, sol-

vency characteristic, flash point and water solubility desired is available. For semi-aqueous cleaning products two chemical structures have been selected which have low vapor pressures, flash points ranging from 74 - 110°C [165 - 230°F] and are completely water-soluble. Glycol ethers in general have good solvency for both polar and non-polar soils, which allow them to be good candidates for electronic cleaning processes. The propylene-series glycol ethers offer lower toxicity than the ethylene-series products.

Water Softening – A water treatment process used to remove calcium and magnesium salts from water.

Terpene – An unsaturated hydrocarbon based on the isoprene unit with the general formula $C_{10}H_{16}$.

Volatile Organic Compounds (VOC) – Regulated compounds containing carbon that have measurable vapor pressures.

2 APPLICABLE DOCUMENTS

The following documents, of the issue in effect on the date of contract agreement, form a part of this handbook to the extent provided herein.

2.1 IPC¹

IPC-B-36 Benchmark Test Printed Wiring Assembly

IPC-SC-60 Post Solder Solvent Cleaning Handbook

IPC-AC-62 Post Solder Aqueous Cleaning Handbook

IPC-CH-65 Guidelines for Cleaning of Printed Boards and Assemblies

IPC-TR-580 Cleaning and Cleanliness Testing Program Phase 1 Test Results

IPC-TM-650 Test Methods Manual²

2.3.25 Detection and Measurement of Ionizable Surface Contaminants by Resistivity of Solvent Extract

2.3.27 Cleanliness Test - Residual Rosin

2.3.28 Ionic Analysis of Circuit Boards, Ion Chromatography Method

2.3.38 Surface Organic Contaminant Detection Test

2.3.39 Surface Organic Contaminant Identification Test (Infrared Analytical Method)

2.6.3.3 Surface Insulation Resistance, Fluxes

2.6.9.1 Test to Determine Sensitivity of Electronic Assemblies to Ultrasonic Energy

2.6.9.2 Test to Determine Sensitivity of Electronic Components to Ultrasonic Energy

1. www.ipc.org

2. Current and revised IPC Test Methods are available through IPC-TM-650 subscription and on the IPC Web site (www.ipc.org/html/testmethods.htm).

2.2 Joint Industries Standards³

J-STD-001 Requirements for Soldered Electrical and Electronic Assemblies

J-STD-004 Requirements for Soldering Fluxes

3 ELECTRONIC MANUFACTURING RESIDUES

3.1 Introduction This section is concerned with the types of residues which accumulate during the printed circuit board fabrication and during the assembly processes; especially as they apply to post solder cleaning. Ideally, contaminants from all the steps prior to the soldering process would be removed by cleaning steps used during the printed circuit board and component manufacturing processes. If this were the case, these “soils” would not affect post soldering cleaning. In fact, such upstream cleaning cannot be always assumed. The “incoming cleanliness” of all components and printed circuit boards should be checked. It is important to know if any residues from these pre-soldering steps exist so that the most effective post soldering cleaning technology can be chosen. There are three basic types of soils: polar/ionic, non-polar/non-ionic and particulate. Each of these responds differently to cleaning agents, and this response must be considered to achieve successful cleaning.

3.2 Type of Residues

3.2.1 Polar/Ionic Polar/ionic contaminants can originate from nearly every step in the fabrication and assembly processes. Their distinguishing characteristic is that they are wholly or partially soluble and ionizable in water. Examples are plating salts, some ingredients of fingerprints, surfactants, flux activators, decomposition products, and water-soluble fluxes.

3.2.2 Non-Polar/Non-Ionic Residues that are both non-ionic and non-polar are usually insoluble in water. They generally are soluble in some organic solvents such as hydrocarbons, chlorocarbons, various fluorocarbons, alcohols, and other organic solvents. The most commonly encountered non-polar residues include rosin flux, metal salts of rosin and metal oxides. However, some polar, non-ionic materials, such as glycols and some surfactants, are water soluble, but they will neither hydrolyze nor cause an increase in the electrical conductivity of water. These materials generally cannot be detected by ionic contamination testing.

3.2.3 Particulates Particulates are small, solid particles. They can originate from a wide variety of sources including drilling, mechanical handling, packaging, etc. Debris

from drilling and machining steps, dust, lint, skin fragments, hair, solder balls, etc. are examples of particulate soils. Particulates rarely dissolve in cleaning agents. The cleaning fluids displace them from the surface of the printed wiring assemblies. Since the particulate removal process is physical, agitation of the cleaning agent and rinse agents during cleaning is a very important factor. The concentration of particulates suspended in cleaning agents must be kept low to avoid re-deposition. Filtration is an effective tool to remove particulates from cleaning agents. Solvent and equipment manufacturers can supply specific information about filter types and sizes.

3.3 Chemistry of Residues The primary residue to be removed in post soldering cleaning is flux residue, which is generally a mixture of ionic, non-ionic and particulate soils. There are different types of flux residue that must be considered when implementing a cleaning technology. A summary of cleaning options for different flux chemistries is illustrated in Figure 3-1.

According to J-STD-004, fluxes used in soldering operations are categorized according to their chemistry into rosin based (RO), resin based (RE), organic (OR), and inorganic (IN) fluxes. They are further classified according to their activity levels. Fluxes may come in the following forms: liquid flux, paste flux, solder-paste, flux-cored wire, and flux-cored preforms. Each type removes oxides and promotes wetting by the solder. Fluxes are activated by heat before and during the actual soldering step.

All semi-aqueous cleaning agents effectively clean rosin fluxes and high activity non-rosin based synthetic activated (SA) fluxes. Not all formulations were specifically designed to remove water-soluble organic flux. However, some formulations will remove water-soluble flux in the wash stage, and all processes will remove water-soluble fluxes in the rinsing stages.

3.3.1 Residues from Rosin and Resin Fluxes Rosin fluxes are based on rosin that is isolated from pine trees. Rosin is a mixture of mild organic acids and esters like abietic acid and abietic acid esters. It is soluble in some organic solvents including alcohols, chlorocarbons, hydrochlorofluorocarbons, hydrofluorocarbons, hydrofluoroethers, ketones, and hydrocarbons. (The various milder fluorocarbons are often formulated with more aggressive solvents such as *trans*-1,2-dichloroethylene to achieve the needed cleaning power.) Non-emulsion type or “fully formulated Type I” semi-aqueous cleaning agents can be safely heated with properly designed equipment and controls to a temperature that begins to break down the crystalline form of rosin residues. The cleaning process then removes these softened or partially liquid residues quickly and easily.

3. www.ipc.org

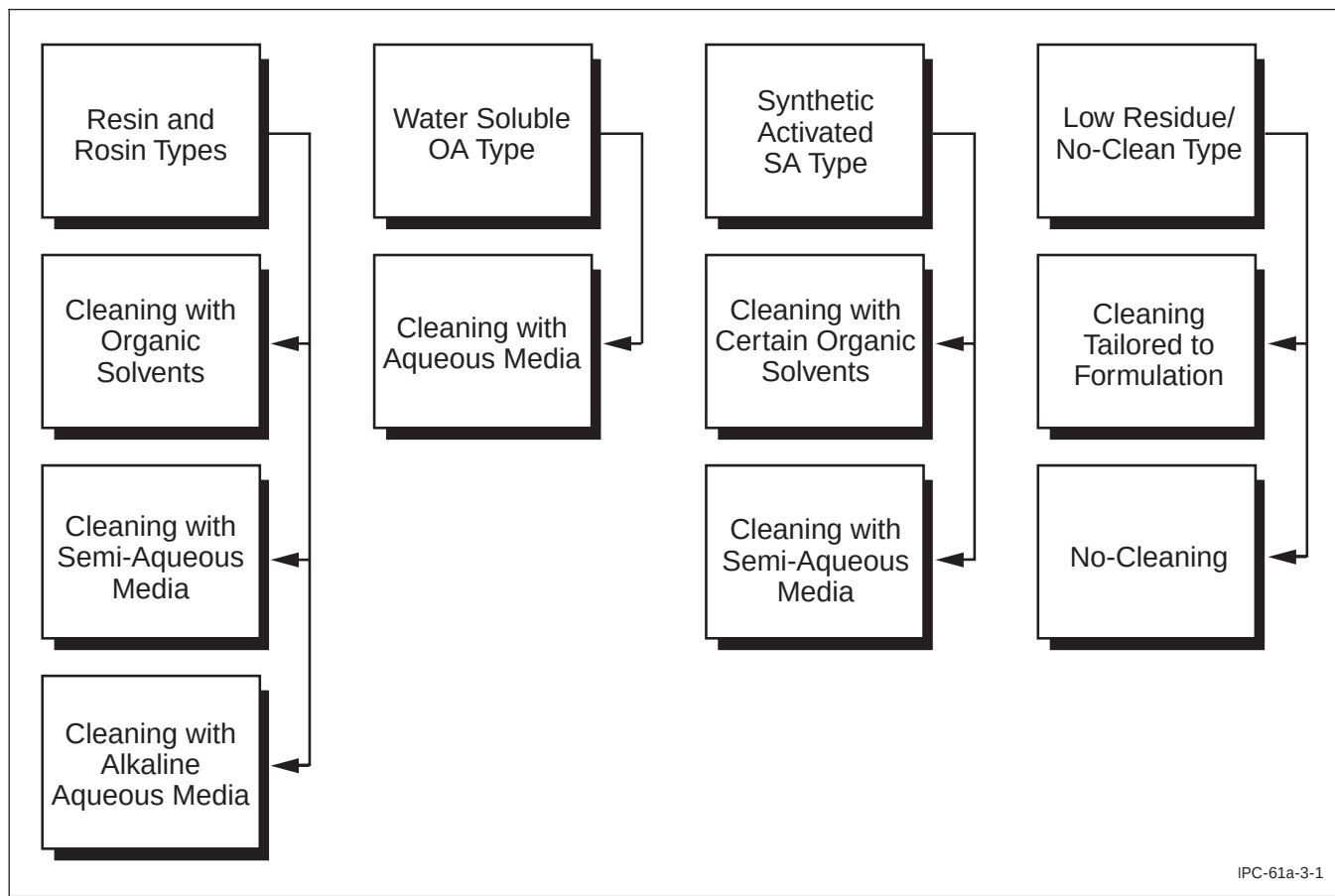


Figure 3-1 Cleaning Options for Different Flux Chemistries

The acid and ester groups in rosin will react with base to form soap-like materials that can be dispersed in water. This process is called saponification. Rosin fluxes can be activated or made more aggressive by the addition of activators, which are usually amine hydrohalides or organic acids. By their very nature, the activators can cause corrosion to circuit elements if they are allowed to remain on the printed wiring assemblies after soldering. Users must be aware that not all rosin molecules have the acid or ester groups needed for saponification. If these groups are not present, the unreacted material is referred to as Non-Saponifiable Rosin or NSR. Levels of NSR in typical rosin range from 4-7%.

Some of the soils that remain on printed wiring assemblies after soldering are by-products of chemical reactions of the rosin such as polymerization, pyrolysis, and combination with metal salts that can occur during the soldering process. In cases where this is severe, a white or tan residue, referred to as “white residue” or “white deposits,” is sometimes observed. It is only visible after the cleaning step or after exposure to water or humidity because the rest of the soldering residues have been cleaned from the surface to expose its presence. Because of its various chemical forms, white residue is an extremely difficult material to remove. Its presence usually signals that the soldering process is outside its control limits, the soldering flux or paste

contains white residue precursors, an incompatibility exists between soldering materials and cleaning agents, or contamination of either printing wiring boards or components. In some cases these residues are a by-product of incompletely cured UV curable liquid solder masks. According to J-STD-004, these fluxes are generally found in the L or M level activity category.

3.3.2 Residues from Water-Soluble Organic Fluxes

Water-soluble (OA) organic fluxes consist of water-soluble organic acids such as citric acid or organic hydrohalides and surfactants. Their residues are generally poorly soluble in hydrocarbons and other non-oxygenated organic derivatives. Water-soluble flux formulations vary greatly. They do not have a common element like the rosin in rosin fluxes. The closest material that could be said to take the place of rosin would be a high molecular weight poly-glycol, chosen to simplify removal of the residue by water. One common characteristic of most OA fluxes is that they are extremely reactive. This allows good soldering yields with heavily oxidized components and boards. Because of this reactivity, the residues after soldering must be completely and quickly removed to avoid long-term corrosion, SIR noise and other problems. As is the case for rosin fluxes, abusive soldering conditions can alter the chemical structures of organic acid flux residues making them more difficult to remove.

in water. For this reason, some users add various products, such as neutralizers, chelating agents, or detergents to water to enhance its cleaning ability for water-soluble fluxes. According to J-STD-004, these fluxes are generally found in the H level activity category.

3.3.3 Residues from Synthetic Activated Fluxes High activity non-rosin based Synthetic Activated (SA) fluxes were formulated to overcome the natural variability of rosin fluxes, to activate heavily oxidized metal surfaces, to eliminate “white residues” and to maximize the cleaning with the stabilized CFC-113/methanol azeotrope. They do not contain rosin, and their residues are easily soluble in certain organic solvents. The residues are quite reactive and should be removed completely after soldering to avoid corrosion failures. SA flux residue, which does not become hard like rosin, should be removed within one hour of soldering to prevent corrosion. According to J-STD-004, these fluxes are generally found in the H level activity category.

3.3.4 Residues from Low Residue/No-Clean Fluxes Low residue fluxes are designed not to be cleaned. However, some manufacturers of electronic assemblies may choose to or be required to remove such residues, which can be quite difficult. Low residue fluxes vary widely in their ability to be removed by semi-aqueous cleaning agents or any other cleaning process. Contact the appropriate flux or cleaning agent suppliers for guidance. According to J-STD-004, these fluxes are generally found in the L level activity category.

3.4 Amount and Location of Residues In addition to the type of residue, the amount of residue and the location of the residue are important. For surface mount assemblies that are reflow soldered, the amount of flux residue will be low; but the residue will be concentrated around solder joints and under components. The spaces between components and the printed circuit boards are often small and cleaning can be difficult. This is particularly true for surface mount assemblies. For wave soldered assemblies, larger amounts of flux residue will be distributed over the entire surface of the assembly, and the liquid flux will flow into tight spaces under components. Liquid flux can also flow through vias and under large components where it can be trapped. Users should be aware that flux residue can consist of reacted flux, un-reacted flux and condensed flux vapors.

4 SEMI-AQUEOUS CLEANING AGENTS

4.1 Introduction Semi-aqueous cleaning refers to those processes in which printed wiring assemblies are first cleaned in an organic solvent and then rinsed with water.

When cleaning electronic assemblies, the objective is to solvate the soils from the surface of the assembly or component. Once the wash step is completed, the parts go through a series of rinse steps to remove the solvent with dissolved soils (temporary soluble solder mask, non-ionic, and some particulate soils) and any water-soluble (ionic) residues. The parts are dried free of water and typically have contamination levels that are undetectable.

There is a broad array of semi-aqueous cleaning agent choices. Each uses organic solvent mixtures but with different chemical structures. Most are a combination of low vapor pressure solvents, surfactants and, if needed, inhibitors. They are designed to remove both polar (flux, ionic salts) and non-polar (light oils, fingerprints, dust) soils. Bath life can be two months to one year due to their stable structures and tendency to hold high levels of soil. These semi-aqueous solvents can be heated to enhance their cleaning efficiency. The maximum use temperatures depend on the solvent's flash point. A good “rule of thumb” is to keep the use temperature 17°C [30°F] below the solvent's flash point.

Semi-aqueous cleaning agents are designed to have good compatibility with most components used in electronic assembly. The process is robust and offers an excellent cleaning alternative. Semi-aqueous cleaning agents have been formulated to achieve high solvency for soldering flux residues and can clean effectively even when they contain a high concentration of flux residue. Some common issues are solvent entrapment, part marking degradation, seal, gasket and elastomer compatibility, and solvent consumption due to evaporative and drag-out losses. Careful attention to equipment design and cleaning agent choice can minimize these problems.

The reader should be advised that semi-aqueous cleaning agents are combustible and are volatile organic compounds (VOCs). Cleaning equipment should be designed specifically for the semi-aqueous cleaning agent and corresponding cleaning process.

Appendix A provides a listing of suppliers of semi-aqueous cleaning agents known at time of publication.⁴

4.2 Properties of Semi-Aqueous Cleaning Agents There are two categories of semi-aqueous cleaning agents - water-insoluble (Type I) and water-soluble (Type II). Table 4-1 and Table 4-2 list the physical properties of semi-aqueous cleaning agents that have been commercially available for at least six months, are presently in commercial use and are well supported by their suppliers.

Water-insoluble cleaning agents consist of two major families. The first family is based on terpene hydrocarbons derived from natural sources such as citrus fruit and pine

4. This list is not meant to be comprehensive, nor are any of the suppliers/products endorsed by IPC. The list is a reflection of available industry recognized suppliers/products. Other suppliers/products may be available that are not listed and new suppliers/products may be developed in the future.

trees. The second family of water-insoluble semi-aqueous cleaning agents is based on petroleum hydrocarbons. When a water-insoluble solvent is used in the wash step, the aqueous rinsing process first forms an emulsion, then displaces the emulsion droplets from the rinsed surfaces. The properties of water-insoluble (Type I) cleaning agents can be found in Table 4-1.

Water-soluble cleaning agents utilize a different rinsing mechanism. The solvent is dissolved from the surface of the printed circuit assemblies with water. In this case, a different wastewater treatment strategy should be employed. The properties of water-soluble (Type II) cleaning agents can be found in Table 4-2.

Semi-aqueous products are not continually distilled as in vapor degreasers. Instead they are used in either batch or in-line equipment in which parts are washed in the semi-aqueous organic cleaning solvents, rinsed in a series of aqueous rinses, and then dried. The fact that the neat (non-water-diluted) semi-aqueous cleaning agents are combustible, greatly influences the processing techniques. However, all these processes have been run safely for many years. These techniques and equipment will be described in later sections of this handbook.

4.3 Compatibility of Cleaning Agents The compatibility of semi-aqueous cleaning agents with plastics, elastomers,

Table 4-1 Physical Properties of Commercially Available Water-Insoluble (Type I) Semi-Aqueous Cleaning Agents

Property	Petroform Axarel® 32	Petroform Bioact® EC-7™	Petroform Bioact® EC-7R™	Exxon Actrel ED*	Petroform Bioact® EC-Ultra®	Petroform Axarel® 36	Petroform Bioact® EC-15	Dr. O.K. Wack Chemie/ Zestion Corp. Zestron® VD	Dr. O.K. Wack Chemie/ Zestion Corp. Zestron® SD-100
B.P., °C	221 - 295	**	**	184 - 226	260	221 - 295	170 - 185	170 - 175	150 - 178
Flash Pt., Closed Cup, °C	>93	51	51	80	141	>93	>121	62	42
Auto Ignition, °C	>220	**	**	**	**	>220	**	165	>200
National Fire Protection Classification	IIIB	II	II	**	IIIB	IIIB	IIIB	IIIA	II
Aromatic Content	Nil	None	None	**	None	Nil	None	None	None
Viscosity, cp 25°C	2.8	0.8	0.8	2.0	6	2.8	5 m Pas	2.3	1.71
Surface Tension, dyne/cm 25°C	27	27	27	**	28	27	**	27	24.4
Sp. Gr. 25°C	0.84	0.84	0.84	**	0.87	0.87	0.085 - 0.087	0.88	0.76
Ozone Depletion	None	None	None	None	None	None	None	None	None
Odor	Almost None	Orange	Orange	**	Almost None	Hydro- carbon	Almost None	Mild	Almost None
Volatile Organic Compound	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Vapor Pressure, mm Hg 25°C	<0.1	1.6	1.5	0.533	0.003	<0.1	<0.01	0.75	3.45
Evaporation Rate, Butyl Acetate=1	<0.1	<1	<1	**	<1	<0.1	<0.1	0.07	0.28
Vapor Density (air = 1)	6.8	>1	>1	0.09	>1	6.7	>1	>1	2.7
pH of 5% Dispersion or Solution	5-6	5-7	5-7	**	4-7	5-6	9-11	6-7	7 (10 g/l)
Solution in Water	<0.1 wt. %	Insoluble	100 ppm	Negligible	**	<0.1 wt. %	Insoluble	Insoluble	<15 wt. %

*Distribution by Safety Kleen

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