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A Survey on the Use of Supercritical Carbon Dioxide as a Cleaning Solvent

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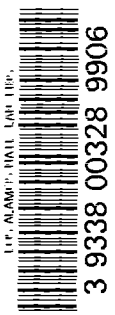

1.0 ABSTRACT

Because the physiochemical properties of supercritical carbon dioxide make it ideally suited for removing commonly encountered contaminants found in the cleaning of a wide variety of components and assemblies, an overall survey was conducted using a small scale supercritical fluid extraction system to investigate removal efficiencies of a wide variety of compounds from an assortment of surfaces using supercritical carbon dioxide. Data is presented demonstrating the successful removal of numerous oils, fluids, adhesives, and chemical compounds from a wide variety of surfaces with supercritical carbon dioxide. In total, the removal of 145 compounds from

some 49 different substrates was investigated. It was found that to a first approximation, cleaning with supercritical CO_2 appears to be contaminant dependent while being surface independent, with an 85-95% removal rate for a wide variety of the compounds investigated.

2.0 INTRODUCTION

Many industrial facilities currently using chlorocarbons and chlorofluorocarbons (CFCs) for the cleaning of a variety of items are facing a difficult situation because of the U.S. amendments to the Montreal Protocol (1987) banning the use of CFCs at the end of 1995. For this reason, these companies must implement economical replacement technologies for cleaning applications. Of course, any solvent cleaning replacement technology must take into account the type of items being cleaned, the contaminant to be removed from these items, and the final cleanliness level that the items must possess. Alternate technologies such as aqueous and semi-aqueous based systems are currently being implemented. While these systems have advantages over CFC cleaning methods, these systems suffer from disadvantages that may not be desirable to many cleaning operations. In the case of aqueous systems, disadvantages include long drying times and flash rusting in addition to many parts not being amenable to water cleansing. In addition, water treatment costs may also be prohibitive. Many semi-aqueous cleaning systems employ toxic terpenes or CFC replacements, and it is only a matter of time before these compounds face regulation. A final alternative technology involves the use of supercritical fluids, which have been used in food, fragrance, and petroleum processes for years, for the extraction of many common compounds.

Ultimately, most cleaning specifications are based on the amount of specific or characteristic contaminants remaining on the surface being cleaned. Common contaminants can include machining oils and greases, hydraulic and damping fluids, adhesives, waxes, human contamination, and particulates. In addition, a whole host of other chemical contaminants from a variety of sources may soil a surface. Therefore, any CFC replacement solvent under consideration should be able to remove any of these commonly encountered soils to specified levels from a variety of surfaces, including printed circuit boards, plastics, metals, rubbers, composites, and glasses. For the purposes of this paper, precision cleaning will be addressed as opposed to bulk cleaning. This precision cleaning level can be defined as an organic contaminant level of less than 10 micrograms of contaminant per square centimeter.[2] This 10 $\mu g/cm^2$ level of cleanliness is either very desirable or required by the function of parts such as metal devices, machined parts, electronic assemblies, optical and laser components, precision mechanical parts, and computer parts.[1]

While supercritical carbon dioxide may be an excellent cleaning solvent for many organic contaminants, many substances requiring removal in cleaning operations, inorganic or ionic contaminants, for example, are insoluble in carbon dioxide. In addition, many items requiring cleaning are intolerant of pressures associated with supercritical CO_2. For cleaning considerations, it should be noted that supercritical CO_2 is best suited for the removal of organic compounds with mid-to-low volatilities.[1] These types of compounds are often encountered as contaminants in precision cleaning, and it is on these compounds that our experimental studies were focused. Since the goal for most precision cleaning levels is less than 1 $\mu g/cm^2$ for most soils[3], the 49 substrate materials used in this survey were initially contaminated with 2 $\mu g/cm^2$ of the 145 contaminants investigated. It is the removal of this amount of material to below the desired 1 $\mu g/cm^2$ contamination level for this survey to determine the general applicability of supercritical fluid cleaning technology.

2.0 EXPERIMENTAL SECTION

The small scale supercritical CO_2 cleaning survey was undertaken to investigate the removal efficiency of a wide variety of contaminants and compounds from a wide assortment of substrates which could be encountered in a cleaning situation. The survey investigated the removal of six human based organic contaminants, five adhesives, seven different hydrocarbons, waxes, high molecular weight compounds, and thirteen different machining oils, fluids, and lubricants, including water miscible types, from fifteen different metal, nineteen polymeric, five rubber, five cable, three glass, and two fabric substrates. The different contaminants and substrates investigated are summarized in **Tables 1-15**. In addition, the removal of 114 different miscellaneous chemical compounds including polycyclic aromatic hydrocarbons (PAHs), amines, substituted phenols, substituted benzenes, phosphates, acids, and acid esters from 340 stainless steel, electrolytic grade copper sheet, glass fiber filled epoxy board, borosilicate glass, and cast magnesium. All of the different chemicals investigated in the survey are listed in **Tables 16-20**.

The contaminant materials were applied as a dilute solutions to 0.5 in. by 2 in. (12.9 cm^2) coupons made from the different substrate materials using a manual pipettor. The contaminant solutions were applied in such a manner so that the entire surfaces of the coupons were coated with 2 $\mu g/cm^2$ of each contaminant compound. While it is noted that a contamination level of 2 $\mu g/cm^2$ is below the precision clean standard of 10 $\mu g/cm^2$, 2 $\mu g/cm^2$ of contamination was visible in many cases and was

required to provide a reasonable detector signal for proper quantitation of the contaminant removal results. Once the application solvent had evaporated to dryness, a contaminated coupon was placed in a 10 ml extraction or cleaning vessel in a Suprex SFE/50 supercritical fluid extractor (Suprex Corp., Pittsburgh, PA). All contaminated coupons were cleaned or extracted dynamically, meaning that there was continuous solvent flow through the cell for each survey. The extractions were conducted using SFC/SFE grade CO_2 (with siphon tube and 1500 psi He head space, Scott Specialty Gases, Inc., Longmont, CO) at 300 atm and 45 °C for 15 min. with a flow rate of 2.8 ml/min. After flowing through the extraction cell, the supercritical CO_2 containing dissolved contaminant was depressurized directly into the inlet of a Hewlett Packard (HP) 5971 gas chromatograph equipped with an HP 5972 series mass selective detector (GC-MS). The GC-MS was operated in the split mode with a split ratio of 150 to 1. The GC column was a 60 m x 0.25 mm i.d. DB-5 (5% crosslinked Ph-Me silicone) column programmed from 30 to 275 °C with a temperature ramp of 7 °C/min. Chromatographic peak areas and subsequent corresponding concentrations of the extracted compounds were calculated from the total ion chromatograms by the HP software. The concentrations obtained using this method were then compared to the initial concentrations of contaminant placed on the substrate coupons and prepared as percent of original material removed from the substrates. The extraction surveys were run in triplicate which yielded an overall average 7 % relative standard deviation for all of the compounds investigated.

3.0 RESULTS AND DISCUSSION

Of the wide variety of contaminants and compounds investigated in the small scale supercritical CO_2 cleaning survey, of particular importance are compounds

associated with human based contamination which is often a significant component of organic contamination found in many cleaning operations, especially those involved in precision cleaning. Human based contaminants can be found in sweat, fingerprints, and other human soils and can contain hundreds of different chemical compounds. Generally, the major constituents of this type of organic contamination are made up of fatty acids and oils found in the skin. For this study, representatives of the chemical classes found in skin lipids were used and consisted of squalene, triglycerol, diglycerol, cholesterol, and palmitylpalmitate. In addition to skin oils, fingerprints tend to be commonly encountered contaminants on parts, components, and assemblies. In order to investigate the removal of fingerprints from surfaces, a fingerprint surrogate consisting of a mixture of skin lipids was prepared based upon previous work.[4-6] The components of the surrogate fingerprints consisted of 30% triolein, 25% oleic acid, 25% cotyl palmitate, 15% squalene, 2.5% cholesterol, and 2.5% cholesterol oleate (components obtained from the Aldrich Chemical Company, WI). While salts are certainly components of fingerprints, these compounds were not added to the mixture since they were incompatible with the experimental detection system. In any event, the surrogate used for this study was assumed to behave in an analogous manner to actual fingerprints.

The results for the removal of human based organic contamination from the 49 different substrates investigated are summarized in **Tables 1-3**. The results presented in **Table 1** summarize the removal of squalene, triglycerol, diglycerol, cholesterol, palmitylpalmitate, and synthetic fingerprints from 15 metal and 3 glass surfaces. These results show near quantitative removal of synthetic fingerprints, squalene, and palmitylpalmitate from most all of the metal and glass surfaces. However, using the test conditions as described, the cast metals, cast aluminum, magnesium, and iron, showed lower extraction efficiencies. For example, cast magnesium had a synthetic fingerprint removal rate of 56 %, while stainless steel 306 had a removal rate of 97 %. The low removal rate from the cast metals is believed to be due to the porosity of the substrate

surface. Because of their high diffusivities and low viscosities, supercritical fluids are inherently capable of penetrating porous surfaces and removing contaminants, and increased removal rates from the cast metals were easily accomplished through parametric changes. For example, longer extraction times of 30 to 45 min. resulted in quantitative removal of the synthetic fingerprints from the cast magnesium surface. The removal rates of the glycerols and cholesterol were lower than the other human contaminants due to their lower solubilities in supercritical CO_2.[7] The removal of these compounds can be improved with a longer extraction time as in the case of the cast metals or through the use of a static extraction step where the substrate is immersed in supercritical CO_2 with no flow through the cell and then followed by a dynamic extraction.

The results of the removal of the skin lipids from the 19 polymeric materials used in this survey are summarized in **Table 2**. These results compare similarly with those observed for the removal of the lipids from the 3 glass surfaces shown in **Table 1**. Again, near quantitative removal of synthetic fingerprints, squalene, and palmitylpalmitate was observed with the same lower removal efficiencies for the glycerols and cholesterol. In general, the same results were observed for the removal of these compounds from the 5 rubber, 5 cable, and 2 fabric substrates as seen in **Table 3**. Palmitylpalmitate was not as effectively removed from the rubber surfaces, probably due to surface interactions with the acid moiety of the compound. While the fabric samples can be thought of as porous substrates, contaminant removal efficiencies from these surfaces were much higher than the cast metals because unlike the metals, supercritical CO_2 can flow through the fabrics thus limiting surface interactions between contaminant and substrate.

The results for the removal of common machining oils and fluids from the selected substrates are summarized in **Tables 4-6**. Oil removal rates from the 34 smooth surfaces investigated, metals, glasses, and plastics, were near quantitative as

seen from **Tables 4** and **5**. The overall removal rates of the oils and fluids from all of these surfaces were quite good, averaging from about 90 to 97 %. Of particular note, as seen in **Table 4**, is that supercritical CO_2 was quite effective in the removal of the various oils and fluids from all smooth metal surfaces, removing, for example, from about 89 to 99% of the Tapmatic® cutting fluid. These results show the applicability of supercritical CO_2 cleaning to machined and precision metal parts and components. Again, however, observed cleaning efficiencies using the described conditions were not as high for the porous metal substrates. On the other hand, quantitative removal of the investigated machining oils and fluids from the rubber, fabric, and cable substrates listed in **Table 6** was observed to be near quantitative, averaging from about 85 to 99 %. The two compounds that did not extract well from any of the 49 surfaces and not included in the aforementioned average removal rates were Molykote lubricant and silicone oil. Since Molykote consists primarily of inorganic particulate matter in a high molecular weight grease, it was expected to have low removal efficiencies with supercritical CO_2. Silicone oil was also not as efficiently removed as the other contaminants due to low solubility or to fractionation of the oil with the higher molecular weight, less soluble components remaining on the surface.

Other common contaminants associated with a machining environment can include water miscible machining fluids and surfactants. For this reason, the removal efficiencies of select compounds from these classes of fluids were also investigated. For the survey, the removal efficiencies of TRIM® SOL, Cimcool, Cimtap, which are water miscible machining fluids, and the nonionic surfactant Triton X-100 from the 49 substrates were studied. The results of this set of experiments are summarized in **Tables 7-9**. Surprisingly, these water soluble materials had fairly high removal rates, generally averaging above 80 % removal from all but the porous metal substrates using the specified conditions. This example suggests that while an aqueous cleaning process might under consideration as a cleaning system replacement, supercritical CO_2

may be a viable cleaning option in cases where components are not ideally suited to aqueous immersion.

Due to the physiochemical properties of a supercritical fluid, cleaning with supercritical CO_2 has a potential advantage over other cleaning technologies due to its ability to rapidly clean completely assembled components systems. In many instances, assembled components that are in need of cleaning contain adhesives, epoxies, and/or sealants. While in some cases it may be desirable to remove these substances from a surface, in other cases it may be desirable to clean the surface and leave these substances intact. With both of these strategies in mind, a selection of adhesives, epoxies, and sealants were applied to each of the 49 substrates and extracted with supercritical CO_2. The results from this portion of the cleaning survey are summarized in **Tables 10-12**. As seen from these tables, it is clear that supercritical CO_2 was ineffective in removing the various adhesives from any of the substrates. For the specified conditions, from about 23 to 52 % of the RTV-732 and 3110 Silastic Adhesive-Sealant and the Loctite® 242 Threadlocker were removed from the surfaces. While, parametric variations such as longer extraction times, increased temperatures and/or pressures, or the inclusion of a static extraction step may increase removal rates, it is unlikely that these compounds would be quantitatively removed from the surfaces, thus precluding effective cleaning with supercritical CO_2. On the other hand, removal rates of less than 10 % were observed for Devcon F-Fast Setting Epoxy and Eastman 910 Super Glue. This low removal rate could conceivably correspond to the extraction of residual solvents from the adhesives, thus demonstrating relative inertness to CO_2 exposure. Therefore, it is conceivable that components assembled with these or similar products could be cleaned with supercritical CO_2 without damage to the adhesive bonds.

The results from the last set of contaminants surveyed for removal efficiencies from the 49 different substrates are listed in **Tables 13-15**. These contaminants are

representative of larger classes of contaminants which may be encountered in cleaning operations. For example, hexadecane and tetracontane can be found in kerosene and diesel. Waxes, such as paraffin wax, are used as lubricants and mold releases. Carbowax and Microwax are chromatographic stationary phases, but they are forms of polyethylene glycol which is also a lubricant. Finally, methyl silicone gum and other methyl silicone resins are often used in protective coatings. The lower molecular weight materials, hexadecane, tetracontane, and paraffin, had fairly high removal rates, generally averaging above 80 % removal from all but the porous metal substrates using the specified conditions. On the other hand, the high molecular weight waxes had fairly low removal efficiencies in the 13 - 39 % range. This was to be expected since supercritical fluid extraction using CO_2 is known not to do well in dissolving high molecular weight compounds. Again, however, the removal of these compounds could probably be improved with a longer extraction time or through the use of a static extraction step followed by dynamic extraction, but it is unlikely that CO_2 cleaning alone would quantitatively remove such high molecular weight contaminants.

Because a wide combination of chemical contaminants from a variety of sources may soil a surface, the cleaning survey also included removal studies of 114 different organic chemicals from a variety of classes of compounds. These compounds include PAHs, amines, substituted phenols, substituted benzenes, phosphates, acids, acid esters, as well as an assortment of miscellaneous compounds. Using the aforementioned cleaning or extraction conditions, the removal of the compounds listed in Tables 16-20 from 5 surface representatives from the larger, previously investigated group was investigated. The surfaces that were contaminated consisted of coupons made from 340 stainless steel, electrolytic grade copper sheet, glass fiber filled epoxy board, borosilicate glass, and cast magnesium

The results for the removal of PAHs from the 5 surface substrates are summarized in Table 16. In general, the 23 PAHs listed in the table averaged removal

rates around 90 % from the smooth surfaces and over 80 % for the porous cast magnesium surface. In contrast, supercritical fluid extraction studies using CO_2 for the removal of PAHs from soils for environmental applications have shown relatively poor removal efficiencies for many of the compounds listed in the table often requiring the addition of secondary solvents to the CO_2.[8] However, it appears that from the results on the removal of the PAHs shown in **Table 16**, surface contamination is much easier to extract and remove than interstitial or sorbed contamination as in the case of soils where a wide range of contaminant-substrate interactions are possible. Since surface interactions with the contaminants are expected to be minimal with the stainless steel, epoxy board, copper sheet, and borosilicate glass, the observed high removal rates were intuitively expected. While surface interactions may not be a dominant controlling factor in PAH removal, the chemical nature of individual PAHs control removal efficiencies. In this case, as substitution increased for various PAHs, removal efficiencies decreased. For example, pyrene had a removal rate of 97 % from the glass surface whereas indeno(1,2,3-CD)pyrene had only an 86 % removal rate.

Organic amines constitute a wide class of compounds ranging from solvents such as aniline and pyridine to familiar chemicals such as nicotine. Altogether, a selection of 23 organic amines, most of them aromatic compounds, was investigated in the cleaning survey. The results of the removal efficiencies of organic amines from stainless steel, copper sheet, epoxy board, borosilicate glass, and cast magnesium are summarized in **Table 17**. In this case, removal efficiencies were entirely compound dependent, based predominantly on contaminant solubilities in supercritical CO_2. For example, compounds such as N-nitrosodimethylamine, which is soluble in water, and N-nitrosophenylaniline had low removal efficiencies ranging from 30 to 40 % from the smooth surfaces and only 21 % removal from cast magnesium. On the other hand, 2-nitroaniline and 4-nitroaniline, which are soluble in polar organic solvents had over 97 % and 90 % removal rates, respectively. All in all, the organic amines had a general

average removal rate near 80 % which still shows fairly effective supercritical CO_2 cleaning.

In general phenols are polar organic compounds primarily soluble in polar organic solvents, and in some cases, water. For this reason, it was expected that removal rates for these types of compounds from the 5 substrates in the chemical removal survey would be rather low. In fact, for most of the 19 substituted phenols surveyed, the removal rates averaged near 60 %, and these results are summarized in **Table 18**. However, several of the substituted phenols, the cresols, for example, had very effective removal rates, averaging above 90 % removal using supercritical CO_2 at 40 °C and 350 atm despite the fact that CO_2 is a nonpolar solvent. It is possible that the high removal rates of these compounds could be attributed to the fact that they are liquids at 40 °C, thus facilitating extraction due to faster kinetics. This implies that higher removal efficiencies for the other phenols could be accomplished through higher temperature extractions. Also summarized in **Table 18** are the removals of substituted benzenes from the same surfaces. Again, in this case the chemical nature of individual compounds was the controlling factor governing removal efficiencies. On average, these compounds had removal rates around 85 %, again showing fairly effective supercritical CO_2 cleaning.

Summarized in **Table 19** are the results for the removal of organic phosphates, acids and acid esters. Only one organic acid, benzoic acid, was investigated. Since this compound is soluble in water, it was expected to have a low removal efficiency from all 5 of the surfaces. This was indeed the case with an average removal of 42 % from the smooth surfaces and 35 % from cast magnesium. Once an organic acid is esterified, it is generally less polar than the precursor, thus increasing lipophilicity. The acid esters investigated in this survey were all phthalates, and they averaged around 90 % removal efficiencies. The three organic phosphates listed in the table show average removal efficiencies around 77 % for the smooth surfaces. While trimethylphosphate is

water soluble, it had the highest removal efficiency of the phosphates once again suggesting that supercritical CO_2 cleaning may be an aqueous cleaning alternative is some limited instances.

Finally, **Table 20** lists the removal efficiencies of another 29 miscellaneous chemical compounds from stainless steel, copper sheet, epoxy board, borosilicate glass, and cast magnesium. Again, the important observation is that surface interactions appear not to be controlling compound removals as the smooth surfaces tend to have the same removal efficiencies. The chemical nature of individual compounds controls the removal efficiencies.

4.0 CONCLUSION

While supercritical CO_2 is not an absolute or drop-in solution to all cleaning problems, it is noted for its solvation of organic compounds having mid-to-low volatilities, and these types of compounds are common contaminants requiring removal to precision clean levels. Based upon the survey results presented in this chapter for the removal of 145 different compounds from 49 surfaces, it was shown that to a first approximation, cleaning with supercritical CO_2 is contaminant dependent and surface independent. Furthermore, in the case of PAHs, it was shown that surface contamination was much easier to extract and remove than interstitial or sorbed contamination. In addition, it was shown that supercritical CO_2 is also capable of removing many compounds traditionally removed by aqueous cleaning, thus expanding the scope of cleaning applicability. Therefore, besides the effectiveness of cleaning with CO_2, the economics of the entire cleaning process may direct the use of CO_2 in cleaning applications where other replacement technologies are under consideration as well as processes other than precision cleaning. Finally, the use of supercritical CO_2 as

a cleaning solvent can reduce the overall use of organic solvents in manufacturing processes.

5.0 ACKNOWLEDGMENTS

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Table 1. Percent Removal of Human Based Organic Conlamination from Metal and Glass Substrates using Supercritical CO_2.

Substrate	Fingerprints	Squalene	Triglycerol	Diglycerol	Cholesterol	Palmityl palmitate
Metals						
Machined Aluminum	94	88	79	60	52	87
Cast Aluminum	81	78	81	62	41	74
Foil Aluminum	89	88	75	62	53	84
Machined Magnesium	94	89	79	58	51	76
Cast Magnesium	56	71	77	54	41	71
Cast Iron	48	65	75	58	38	70
Stainless Steel 340	94	94	82	65	51	89
Stainless Steel 316	95	97	83	64	52	85
Stainless Steel 306	97	92	81	68	53	84
Silver	96	91	88	66	52	88
Gold	94	90	89	61	51	87
Tin	98	92	87	66	51	84
Copper	95	90	84	62	54	85
Copper clad wire board	98	92	85	67	52	88
Brass	94	99	88	59	49	88
Glasses						
Borosilicate Slide	94	87	78	64	54	85
Fused Silica Plate	95	84	82	65	55	84
Sapphire Flat	99	87	78	62	51	85

Table 2. Percent Removal of Human Based Organic Contamination from Polymeric Substrates using Supercritical CO_2.

Polymer Sheet	Fingerprints	Squalene	Triglycerol	Diglycerol	Cholesterol	Palmityl palmitate
HDPE	94	86	75	65	54	86
Polyethylene	91	88	76	63	52	81
Nylon 66	92	87	79	62	52	79
Kevlar	91	89	78	61	53	88
Polypropylene	94	88	74	65	51	84
PVC	94	78	75	67	48	85
Teflon	97	84	78	65	52	87
Epoxy PC Board	96	86	79	64	47	85
Polyimide	94	89	86	62	51	87
Polystyrene	93	89	84	68	52	84
Polymethylmethracrylate	95	89	82	64	54	81
Polyisobutylene	93	87	85	69	55	89
Polytetrafluoroethylene	95	89	81	66	56	85
Polycarbonate	96	90	85	59	52	86
Polyvinylidene	91	89	85	65	59	78
Vinylchloride-acrylonitrile	94	86	86	64	48	89
Polyacrylonitrile	98	86	84	68	52	85
Polyvinyl Alcohol	95	84	81	62	51	85
Polyacrylate	91	88	85	62	54	82

Table 3. Percent Removal of Human Based Organic Contamination from Rubber, Coaxial Cable, and Fabric Substrates using Supercritical CO_2.

Substrate	Fingerprints	Squalene	Triglycerol	Diglycerol	Cholesterol	Palmityl palmitate
Rubber Sheet						
Buna	97	95	80	52	42	75
Viton	94	93	80	51	41	74
Butyl	97	83	74	45	45	75
Silicone	94	94	74	51	41	71
Neoprene	95	88	75	55	40	70
Coaxial Cable						
UI-1354	95	84	81	61	59	81
RG-71B/U	93	85	81	60	56	84
RG-174/U	96	85	82	61	52	82
RG-58C	93	88	83	64	58	84
RG-223	92	86	84	65	57	81
Fabric Sheet						
Wool	99	89	85	62	55	88
Cotton	93	89	82	61	56	84

Table 4. Percent Removal of Machining Oils and Lubricants from Metal and Glass Substrates using Supercritical CO_2.

Substrate	3-in-One® Lubricating Oil	SAE 30W	Mineral Oil	Molykote Lubricant	DC 200 Silcone Oil	Convoil® Synthetic Pump Oil	Tapmatic® Cutting Fluid	Regal® 648 Cutting Fluid	Threadcut® Cutting Fluid
Metals									
Machined Aluminum	86	94	87	21	85	96	97	90	79
Cast Aluminum	75	88	71	25	71	90	95	81	80
Foil Aluminum	88	89	88	26	81	95	98	95	81
Machined Magnesium	79	91	89	24	86	95	97	89	82
Cast Magnesium	75	80	70	27	71	89	99	79	88
Cast Iron	64	75	65	25	60	78	96	65	89
Stainless Steel 340	98	91	98	27	88	98	95	94	90
Stainless Steel 316	96	92	94	29	84	97	94	92	92
Stainless Steel 306	97	93	95	24	84	96	98	91	87
Silver	96	94	91	25	86	89	99	97	83
Gold	97	94	93	27	81	92	97	94	90
Tin	91	97	91	21	80	95	89	89	92
Copper	96	91	93	22	79	96	96	94	90
Copper clad wire board	98	96	88	28	86	97	97	98	98
Brass	99	95	89	25	89	98	89	97	95
Glasses									
Borosilicate Silde	89	91	88	21	85	97	98	94	89
Fused Silica Plate	91	92	85	23	84	98	97	94	87
Sapphire Flat	95	94	89	24	81	97	96	92	86

Table 5. Percent Removal of Machining Oils and Lubricants from Polymeric Materials using Supercritical CO_2.

Polymer Sheet	3-in-One® Lubricating Oil	SAE 30W	Mineral Oil	Molykote Lubricant	DC 200 Silcone Oil	Convoil® Synthetic Pump Oil	Tapmatic® Cutting Fluid	Regal® 648 Cutting Fluid	Threadcut® Cutting Fluid
HDPE	94	86	86	23	80	97	97	90	90
Polyethylene	94	87	84	24	81	98	98	89	92
Nylon 66	97	88	87	22	75	95	99	84	93
Kevlar	86	84	88	25	74	96	96	90	91
Polypropylene	87	86	81	27	79	94	97	90	88
PVC	89	81	86	21	75	96	89	85	87
Teflon	90	87	91	30	88	97	99	94	90
Polyacrylate	97	95	89	21	89	94	95	97	96
Polyimide	94	96	89	24	89	94	94	96	95
Polystyrene	89	96	90	25	90	97	98	97	96
Polymethylmethracrylate	90	98	92	28	97	95	97	96	94
Polyisobutylene	92	90	92	29	88	94	95	92	98
Polytetrafluoroethylene	96	99	95	26	92	96	94	99	97
Polycarbonate	97	98	99	23	90	97	96	98	98
Polyvinylidene	96	99	92	25	89	95	98	89	95
Vinylchloride-acrylonitrile	95	98	90	22	89	95	95	97	95
Polyacrylonitrile	94	96	93	23	85	94	99	92	96
Polyvinyl Alcohol	93	98	90	26	90	96	99	96	99
Epoxy PC Board	93	94	89	29	83	98	98	94	96

Table 6. Percent Removal of Machining Oils and Lubricants from Rubber, Coaxial Cable and Fabric Substrates using Supercritical CO_2.

Substrate	3-in-One® Lubricating Oil	SAE 30W	Mineral Oil	Molykote Lubricant	DC 200 Silcone Oil	Convoil® Synthetic Pump Oil	Tapmatic® Cutting Fluid	Regal® 648 Cutting Fluid	Threadcut® Cutting Fluid
Rubber Sheet									
Buna	85	90	80	26	74	96	85	89	98
Viton	86	90	82	24	73	95	84	89	97
Butyl	93	95	85	25	84	90	91	86	95
Silicone	95	98	88	27	81	89	90	87	94
Neoprene	87	94	89	28	80	88	88	88	96
Coaxial Cable									
UI-1354	94	93	87	29	87	94	94	93	98
RG-71B/U	95	94	88	28	86	97	95	95	94
RG-174/U	96	93	87	27	88	92	96	92	97
RG-58C	95	92	86	26	86	91	95	91	98
RG-223	93	94	83	25	87	95	98	93	94
Fabric Sheet									
Wool	97	99	99	24	93	89	94	98	95
Cotton	98	99	98	26	95	88	95	98	97

Table 7. Percent Removal of Water Miscible Machining Fluids and Nonionic Surfactant from Metal and Glass Substrates using Supercritical CO_2.

Substrate	TRIM® SOL	Cimcool	Cimtap	Triton X-100
Metals				
Machined Aluminum	83	75	86	93
Cast Aluminum	75	61	80	87
Foil Aluminum	87	78	88	89
Machined Magnesium	84	76	86	89
Cast Magnesium	72	60	78	78
Cast Iron	70	51	67	84
Stainless Steel 340	94	78	87	98
Stainless Steel 316	90	77	86	97
Stainless Steel 306	88	75	88	98
Silver	78	79	84	99
Gold	86	74	89	97
Tin	90	75	88	94
Copper	85	78	87	93
Copper clad wire board	88	80	88	97
Brass	85	82	84	98
Glasses				
Borosilicate Silde	84	78	88	95
Fused Silica Plate	82	75	86	93
Sapphire Flat	81	74	84	89

Table 8. Percent Removal of Water Miscible Machining Fluids and Nonionic Surfactant from Polymer Substrates using Supercritical CO_2.

Polymer Sheet	TRIM® SOL	Cimcool	Cimtap	Triton X-100
HDPE	84	77	75	92
Polyethylene	83	79	78	93
Nylon 66	81	75	79	92
Kevlar	88	74	89	94
Polypropylene	87	74	88	95
PVC	82	79	84	91
Teflon	84	75	89	95
Epoxy PC Board	88	84	88	99
Polyimide	89	85	89	96
Polystyrene	86	83	85	97
Polymethylmethracrylate	84	81	85	95
Polyisobutylene	87	88	84	96
Polytetrafluoroethylene	90	85	88	98
Polycarbonate	90	85	89	98
Polyvinylidene	89	83	88	90
Vinylchloride-acrylonitrile	90	82	89	99
Polyacrylonitrile	86	83	85	99
Polyvinyl Alcohol	85	84	89	99
Polyacrylate	82	78	85	94

Table 9. Percent Removal of Water Miscible Machining Fluids and Nonionic Surfactant from Rubber, Coaxial Cable, and Fabric Substrates using Supercritical CO_2.

Substrate	TRIM® SOL	Cimcool	Cimtap	Triton X-100
Rubber Sheet				
Buna	81	76	56	87
Viton	82	78	72	89
Butyl	81	74	64	89
Silicone	90	72	58	88
Neoprene	89	75	59	87
Coaxial Cable				
UI-1354	79	76	82	96
RG-71B/U	79	77	81	97
RG-174/U	80	75	84	97
RG-58C	80	78	83	97
RG-223	81	80	85	98
Fabric Sheet				
Wool	89	86	89	97
Cotton	89	88	85	97

Table 10. Percent Removal of Adhesives and Sealants from Metal and Glass Substrates using Supercritical CO_2.

Substrate	RTV-3110 Silastic Adhesive-Sealant	RTV-732 Silastic Adhesive-Sealant	Devcon F- Fast Setting Epoxy	Loctite® 242 Threadlocker	Eastman 910 Super Glue
Metals					
Machined Aluminum	26	56	5	26	5
Cast Aluminum	46	35	4	35	4
Foil Aluminum	35	46	6	24	6
Machined Magnesium	46	37	3	27	7
Cast Magnesium	42	50	4	21	3
Cast Iron	35	46	5	28	4
Stainless Steel 340	45	42	4	24	5
Stainless Steel 316	28	45	7	23	2
Stainless Steel 306	41	47	6	24	3
Silver	36	50	5	27	5
Gold	27	45	6	24	4
Tin	34	39	3	22	3
Copper	27	47	4	24	6
Copper clad wire board	31	37	5	25	5
Brass	35	45	7	23	6
Glasses					
Borosilicate Silde	35	45	3	24	6
Fused Silica Plate	34	39	4	25	4
Sapphire Flat	36	44	2	22	3

Table 11. Percent Removal of Adhesives and Sealants from Polymeric Materials using Supercritical CO_2.

Substrate	RTV-3110 Silastic Adhesive-Sealant	RTV-732 Silastic Adhesive-Sealant	Devcon F- Fast Setting Epoxy	Loctite® 242 Threadlocker	Eastman 910 Super Glue
HDPE	30	45	2	25	4
Polyethylene	31	44	6	23	5
Nylon 66	34	46	4	22	3
Kevlar	45	38	5	25	5
Polypropylene	24	52	3	24	4
PVC	36	43	5	22	6
Teflon	40	46	4	26	5
Polyacrylate	32	46	4	21	4
Polyimide	36	48	8	24	5
Polystyrene	39	49	2	26	3
Polymethylmethracrylate	36	48	7	25	4
Polyisobutylene	35	47	5	25	5
Polytetrafluoroethylene	32	48	6	23	4
Polycarbonate	34	51	9	21	6
Polyvinylidene	36	49	5	23	4
Vinylchloride-acrylonitrile	31	47	4	26	5
Polyacrylonitrile	32	49	5	25	3
Polyvinyl Alcohol	36	52	8	24	6
Epoxy PC Board	29	45	6	31	4

Table 12. Percent Removal of Adhesives and Sealants from Rubber, Coaxial Cable, and Fabric Substrates using Supercritical CO_2.

Substrate	RTV-3110 Silastic Adhesive-Sealant	RTV-732 Silastic Adhesive-Sealant	Devcon F- Fast Setting Epoxy	Loctite® 242 Threadlocker	Eastman 910 Super Glue
Rubber Sheet					
Buna	25	34	5	24	3
Viton	26	27	4	25	4
Butyl	26	34	4	24	7
Silicone	25	32	5	20	3
Neoprene	24	28	8	20	4
Coaxial Cable					
UI-1354	34	46	6	26	5
RG-71B/U	31	44	8	25	4
RG-174/U	38	50	4	25	4
RG-58C	35	47	9	22	3
RG-223	34	45	5	20	6
Fabric Sheet					
Wool	29	45	8	28	6
Cotton	28	46	7	21	4

Table 13. Percent Removal of Hydrocarbons, Waxes, and High Molecular Weight Compounds from Metal and Glass Substrates using Supercritical CO_2.

Substrate	Hexadecane (C16)	Tetracontane (C40)	Paraffin Wax (C20-C60)	Carbowax (15M)	Microwax	Methyl Silicone Gum (SE-30)	Polyethylene Glycol (MW 2000)
Metals							
Machined Aluminum	95	87	78	35	24	14	28
Cast Aluminum	89	70	76	20	26	15	15
Foil Aluminum	94	84	77	35	25	15	26
Machined Magnesium	94	81	69	29	24	14	29
Cast Magnesium	85	74	79	22	25	15	16
Cast Iron	75	65	82	21	21	16	12
Stainless Steel 340	91	89	82	35	24	14	29
Stainless Steel 316	90	92	80	36	20	15	28
Stainless Steel 306	94	94	79	39	25	12	25
Silver	94	86	89	38	23	14	26
Gold	97	88	83	37	26	18	27
Tin	91	89	88	35	25	17	25
Copper	92	88	90	36	24	19	28
Copper clad wire board	98	88	85	36	24	14	27
Brass	98	88	85	32	21	13	24
Glasses							
Borosilicate Silde	98	89	78	36	28	18	25
Fused Silica Plate	95	88	79	39	29	18	26
Sapphire Flat	97	88	80	38	28	17	29

Table 14. Percent Removal of Hydrocarbons, Waxes, and High Molecular Weight Compounds from Polymeric Materials using Supercritical CO_2.

Polymer Sheet	Hexadecane (C16)	Tetracontane (C40)	Paraffin Wax (C20-C60)	Carbowax (15M)	Microwax	Methyl Silicone Gum (SE-30)	Polyethylene Glycol (MW 2000)
HDPE	91	87	85	36	21	14	29
Polyethylene	94	84	84	35	25	15	26
Nylon 66	97	85	87	32	23	18	24
Kevlar	94	86	88	36	26	14	26
Polypropylene	98	78	75	35	29	19	25
PVC	96	85	89	36	25	15	23
Teflon	92	88	84	39	24	16	25
Polyacrylate	98	86	88	37	25	16	23
Polyimide	97	88	81	33	29	15	25
Polystyrene	98	87	82	33	25	13	29
Polymethylmethracrylate	98	89	80	36	21	12	25
Polyisobutylene	99	89	78	35	24	15	24
Polytetrafluoroethylene	99	93	86	32	21	18	28
Polycarbonate	96	89	85	36	26	17	26
Polyvinylidene	98	87	89	36	21	12	26
Vinylchloride-acrylonitrile	97	86	88	35	25	13	23
Polyacrylonitrile	96	87	88	39	20	15	26
Polyvinyl Alcohol	99	87	89	38	21	18	25
Epoxy PC Board	96	87	90	37	23	15	26

Table 15. Percent Removal of Hydrocarbons, Waxes, and High Molecular Weight Compounds from Rubber, Coaxial Cable, and Fabric Substrates using Supercritical CO_2.

Substrate	Hexadecane (C16)	Tetracontane (C40)	Paraffin Wax (C20-C60)	Carbowax (15M)	Microwax	Methyl Silicone Gum (SE-30)	Polyethylene Glycol (MW 2000)
Rubber Sheet							
Buna	87	78	70	28	25	12	11
Viton	86	78	71	26	25	14	18
Butyl	97	88	65	21	23	19	11
Silicone	89	86	62	20	23	15	14
Neoprene	88	83	60	19	22	12	15
Coaxial Cable							
UI-1354	90	86	87	35	23	18	28
RG-71B/U	89	90	86	36	24	15	25
RG-174/U	91	82	89	39	21	19	25
RG-58C	90	84	84	38	25	15	26
RG-223	91	86	85	35	26	14	22
Fabric Sheet							
Wool	98	89	84	35	25	15	24
Cotton	98	89	86	39	25	16	24

Table 16. Percent Removal of Polyaromatic Hydrocarbons from Stainless Steel, Copper Sheet, Epoxy Board, and Cast Magnesium using Supercritical CO_2.

PAHs	Substrate				
	SS	Cu	Epoxy	Glass	Mg
Naphthalene	97	96	97	98	89
2-Chloronaphthalene	86	85	86	85	67
2-Methylnaphthalene	89	89	92	93	86
Acenaphthalene	96	97	96	97	90
Acenaphthene	95	97	96	94	84
5-Nitroacenaphthene	89	90	92	89	78
Fluorene	97	98	98	97	89
2-Acetylaminofluorene	78	76	77	75	68
Fluoranthene	97	98	98	98	89
Benzo(B)Fluoranthene	91	91	92	92	87
Benzo(K)Fluoranthene	90	90	89	90	81
Phenanthrene	89	90	92	93	81
Anthracene	97	98	96	95	90
Dibenzo(A,H)Anthracene	85	86	84	85	75
Pyrene	96	97	95	97	98
Benzo(A)Pyrene	95	95	96	97	90
Benzo(E)Pyrene	92	93	94	92	83
Dibenzo(A,E)Pyrene	89	90	88	89	78
Indeno(1,2,3-CD)Pyrene	88	87	89	86	88
Benzo(G,H,I)Perylene	78	79	80	80	65
Chrysene	93	93	92	94	88
Isodrin	88	86	87	85	78
Coronene	75	76	78	78	65

Table 17. Percent Removal of Organic Amines from Stainless Steel, Copper Sheet, Epoxy Board, and Cast Magnesium using Supercritical CO_2.

Amines	Substrate				
	SS	Cu	Epoxy	Glass	Mg
Aniline	56	57	53	56	45
4-Chloroaniline	89	92	88	91	76
2-Nitroaniline	97	98	99	98	87
4-Nitroaniline	90	92	93	90	88
5-Chloro-2-Methylaniline	78	76	77	75	56
2-Methyl-5-Nitroaniline	67	68	78	68	76
2,4,5-Trimethylaniline	89	90	93	94	79
4,4'-Oxydianiline	78	79	78	77	67
Pyridine	89	90	91	90	78
O-Toluidine	87	85	86	87	67
O-Anisidine	87	87	87	88	78
5-Nitro-O-Anisidine	87	88	89	88	56
P-Phylenediamine	89	89	80	87	80
4-Chloro-1,2-Phenylenediamine	87	89	88	87	78
4-Chloro-1,3-Phenylenediamine	88	86	87	90	78
P-Cresidine	78	87	68	90	67
Methapynlene	90	90	91	90	78
Moca	56	54	55	56	40
N-Nitrosodimethylamine	34	36	30	41	21
N-Nitrosodi-N-Propylamine	84	82	75	89	78
N-Nitrosodiphenylamine	31	34	41	29	31
2-Picoline	87	89	86	77	67
Nicotine	78	78	77	76	56

Table 18. Percent Removal of Substituted Phenols and Benzenes from Stainless Steel, Copper Sheet, Epoxy Board, and Cast Magnesium using Supercritical CO_2.

Compound	Substrate				
	SS	Cu	Epoxy	Glass	Mg
Substituted Phenols					
Phenol	56	57	56	60	45
2-Chlorophenol	65	65	67	66	56
4-Chlorophenol	68	67	65	67	55
2,4-Dichlorophenol	56	57	68	67	54
2,4,6-Trichlorophenol	57	57	57	57	45
2,4,5-Trichlorophenol	56	57	55	54	45
Pentachlorophenol	45	46	47	43	35
2-Nitrophenol	56	67	65	64	68
4-Nitrophenol	56	56	54	57	45
2,4-Dinitrophenol	52	54	56	53	46
2-Methyl-4,6-Dinitrophenol	62	61	62	63	52
2-Methylphenol	67	68	66	66	56
4-Methylphenol	68	66	65	67	60
2,4-Dimethylphenol	67	68	68	67	56
Resorcinol	89	89	90	91	78
Thiophenol	87	87	89	88	79
O-Cresol	89	89	93	95	80
M-Cresol	91	92	93	92	89
P-Cresol	89	92	90	91	91
Substituted Benzenes					
1,3-Dichlorobenzene	78	78	79	76	68
1,4-Dichlorobenzene	79	79	76	78	56
1,2-Dichlorobenzene	79	79	78	76	60
1,2,4-Trichlorobenzene	87	86	88	87	68
Hexachlorobenzene	97	98	97	96	89
2,4-Diaminotoluene	78	68	78	72	50
Nitrobenzene	82	84	83	89	70
1,2-Dinitrobenzene	86	87	88	86	80
1,4-Dinitrobenzene	86	87	87	86	78
2,6-Dinitrotoluene	89	89	89	88	78
2,4-Dinitrotoluene	88	87	89	86	76

Table 19. Percent Removal of Various Organic Compounds from Stainless Steel, Copper Sheet, Epoxy Board, and Cast Magnesium using Supercritical CO_2.

Compound	Substrate				
	SS	Cu	Epoxy	Glass	Mg
Phosphates					
Trimethylphosphate	79	79	78	77	67
Tris(2,3-Dibromopropyl)Phosphate	76	75	77	76	70
Tri-P-Tolyl Phosphate	75	76	74	77	65
Acids					
Benzoic Acid	40	41	42	44	35
Acid Esters					
Dimethyl Phthalate	88	90	89	92	87
Di-N-Butyl Phthalate	82	84	80	79	74
Butylbenzyl Phthalate	91	9[illegible]	88	89	78
Bis(2-Ethylhexyl) Phthalate	98	97	96	98	85
Di-N-Octyl Phthalate	97	98	98	96	87

Table 20. Percent Removal of Miscellaneous Organic Compounds from Stainless Steel, Copper Sheel, Epoxy Board, and Cast Magnesium using Supercritical CO_2.

Compound	Substrate				
	SS	Cu	Epoxy	Glass	Mg
Bis(2-Chloroethyl) Ether	17	20	18	19	20
Bis(2-Chloroisopropyl) Ether	56	62	50	61	30
Bis(2-Chloroethoxyl)Methane	67	68	56	67	60
4-Chlorophenylphenyl Ether	78	76	80	84	73
4-Bromophenylphenyl Ether	70	75	78	76	79
Benzyl Alcohol	47	56	54	49	40
Dibenzofuran	97	96	97	98	89
Hexachloroethane	82	83	84	83	68
Hexachlorophene	89	90	91	91	80
Isophorone	90	89	91	90	77
Hexachlorobutadiene	89	90	92	92	78
Hexachlorocyclopentadiene	88	89	89	89	79
Azobenzene	99	98	98	97	90
Isosafrole	89	89	88	87	79
1,4-Naphthaquinone	87	86	87	88	59
Safrole	78	67	70	81	56
2-Aminoanthraquinone	67	68	65	67	60
4-Aminoazobenzene	88	87	88	89	78
3-Amino-9-Ethylcarbazole	76	75	76	77	67
Diethylsulfate	78	79	76	78	67
Hexamethylphosphoramide	89	85	86	87	65
Maleic Anhydride	67	68	67	65	45
Phthalic Anhydride	87	89	86	87	79
5,5-Diphenylhydantoin	68	67	68	66	60
4-Nitrobiphenyl	91	92	90	92	80
Propylthiouracil	67	87	58	89	60
Strychnine	78	77	77	76	56
Cholersterol Oleate	95	94	97	94	56
Mestranol	89	89	90	91	80