Lipophilic Super-Absorbent Swelling Gels as Cleaners for Use on Weapons Systems and Platforms

WP- 1761

Final Report

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14. ABSTRACT

The objective of this feasibility research is to develop an environmentally friendly disruptive technology for cleaning metal and non-metal surfaces for use on weapons systems and platforms. Lipophilic super-absorbent swelling gels are synthesized to absorb oil, grease, and particulates from metal surfaces. An octadecylacrylate-co-ethylene glycol dimethacrylate (ODA-co-EGDMA) polyelectrolyte, and poly(stearylacrylate-co-ethyleneglycol dimethacrylate) (SA-co-EGDMA) gels were synthesized, and characterized. Gel cleaning tests were conducted following ASTM test methods and MIL-PRF-680B specifications using metal coupons and field samples from the Navy. The cleaning ability of the gels was also compared with trichloroethylene (TCE). The gel cleaning was comparable to TCE, however, requires twice the time. The gels can be recycled for up to 10 cycles. In conclusion, a recyclable superabsorbent polymer gel cleaner and a processes are developed. Gel cleaning will result in drastic reduction of volatile organic compounds (VOCs) and hazardous air pollutants (HAPs) release, without increase in overall cleaning quality and costs.

15. SUBJECT TERMS
lipophilic polyelectrolyte gel, alkylstyrene copolymer, Stearylacrylate-co-ethylene glycol dimethacryale neutral gel (NG-18), Octadecylacrylate-co-ethylene glycol dimethacrylate polyelectrolyte gel (EG-18), surface & gel cleaning, VOCs & HAPs reduction

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List of Acronyms

AIBN	azobisisobutylonitrile
ASTM	American Society for Testing and Materials
DMSO	Dimethylsulfoxide
DoD	Department of Defense
EG-18	Octadecylacrylate-co-ethylene glycol dimethacrylate tetraalkylammonium tetraphenylborate polyelectrolyte gel
EGDMA	Ethylene glycol dimethacrylate
FTIR	Fourier transform infrared
HAPs	Hazardous air pollutants
MIBK	Methylisobutylketone
MSDS	Material Data Safety Sheet
NG-18	Stearylacrylate-co-ethylene glycol dimethacryale neutral gel
NIPAM	N-isopropylacrylamide
ODA	Octadecylacryalte
SA	Stearylacrylate
SPOTA	Sustainable Painting Operation for the Total Army
TCE	Trichloroethylene
TFPB^-	Tetraphenylborate
THF	Tetrahydrofuran
VOCs	Volatile organic compounds

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Abstract

Increasingly stringent environmental regulations on volatile organic compounds (VOCs) and hazardous air pollutants (HAPs) demand the development of disruptive technologies for cleaning weapons systems and platforms. Currently employed techniques such as vapor degreasing, solvent, aqueous, or blast cleaning processes suffer from shortcomings in environmental friendliness, personnel health and safety, cleaning efficiency, cost-effectiveness, management of contaminated cleaning media, or in maintaining the integrity of equipment material surfaces. We propose to use novel lipophilic super-absorbent swelling gels as a disruptive solid state cleaning technology that will facilitate the Department of Defense (DoD) in overcoming limitations of currently employed cleaning techniques. Lipophilic super-absorbent swelling gels have been developed that will not only absorb the oil and grease from these machine parts, but will also act as an automated sweeper due to the self-generating mechanical force of the gel. An octadecylacrylate-*co*-ethylene glycol dimethacrylate (ODA-*co*-EGDMA) tetraalkylammonium tetraphenylborate lipophilic polyelectrolyte gel (EG-18) and poly(stearylacrylate-*co*-ethyleneglycol dimethacrylate) (SA-co-EGDMA) neutral gel (NG_18) were evaluated for swelling and oil sorption capacity. The results were compared with a commercially available alkylstyrene copolymer (imbiber beads). For each gel, the swelling degree and oil absorption capacity were quantitatively investigated at 0-60°C using a variety of polar and nonpolar solvents. The cross-linking of the polymers was studied using infrared spectroscopy, and the compression strength was determined. The cleaning tests were performed on metal coupons using ASTM G122-96(2008) methods. Cleaning tests were also performed on field samples obtained from a Naval cleaning facility. NG-18 and EG-18 gels removed particulate contaminants and absorbed oils and grease on metal and non-metal surfaces without causing abrasion. The gels are also recyclable. The cleaning ability of the gels was compared with the standard solvent cleaner trichloroethylene (TCE) following ASTM G122-96(2008) test methods and MIL-PRF-680B procedure with MIL-PRF-10924 test grease. Polymer gel cleaners exhibited analogues extent and rate of cleaning as the TCE. In conclusion, the recyclable superabsorbent polymer cleaners developed in this research will allow drastic reduction in the use of VOC containing solvents and HAP release.

Objective

The overall objective of the proposed research was to develop an environmentally benign disruptive technology for cleaning metal and non-metal surfaces. The ability of two super absorbent polymer gel systems for removing oil, grease and particulates from metal and plastic surfaces was initially evaluated. Upon successful proof of the surface cleaning ability of these gel systems, further research will focus on improving the gel performance by design and synthesis of additional polymer gel systems. Further research will address the post-cleaning gel removal method, the use of non-fluorinated compounds in gel synthesis, and an evaluation of toxicity and environmental fate-and-effects of the gels. The proposed cleaner is in solid form and is VOC-exempt, HAP-free, non-toxic, non-corrosive, non-ozone depleting, recyclable, and selfgenerates the energy necessary for the cleaning function, thereby affording a new cost-effective, environmentally friendly cleaning technology. We hypothesized that lipophilic super-absorbent swelling gels would, upon contacting oil and grease on the metal and non-metal surfaces, exert enough mechanical forces by swelling to remove particulate matters, oil, and grease on the material surfaces simultaneously. Also, that the super-absorbent gels would exhibit low friction behaviors and therefore not stick to, or cause damage on the surface of metal and nonmetal materials. Existing solvent and blast cleaning technologies pose environmental concerns both during (VOC production from organic solvents and HAP production from forced-air blast cleaning processes) and after (disposal of waste streams for solvent cleaning; cleanup of blasted contaminants for forced air cleaning) the cleaning operation. In addition, these techniques require on-site equipment such as the soaking bath and air compressor, and often necessitate operation in a confined, well-ventilated space. Current limitations stated above call for a portable cleaning technology that will not pose environmental or health threats during or after the cleaning operations. The overall study aimed to utilize intricate designing of lipophilic super-absorbent swelling gels through careful selection of polymer backbone and ionic components, and the cross linking density for improved cleaning ability of the lipophilic swelling gels. After successful proof-of-concept, a follow-on project will be proposed to address other issues including the method for removing the gels after swelling, the use of non-fluorinated compounds in gel synthesis, and an evaluation of toxicity and environmental fate-and-effects of the gels.

Background

Environmentally benign VOC-exempt, and HAP-free surface cleaning technology, will support ongoing DoD programs such as the Sustainable Painting Operations for the Total Army (SPOTA). Technology developed in this research will result in dramatic overall reductions of VOCs and HAPs emissions from DoD surface cleaning operations. Polyelectrolyte gels are ionic polymer networks composed of charged polymer chains and freely mobile counter-ions. Polyelectrolyte super-absorbent wet-swelling hydrogels are known to undergo a dramatic but reversible volume change by absorbing large quantities of water. The polyelectrolyte hydrogels swell in water because of (1) osmotic pressure induced by freely mobile counter-ions within the polyelectrolyte, (2) increased entropy arising from the solvation of polymer ions and counter-ions, (3) electrostatic repulsion between the oppositely charged ions within the polyelectrolyte gel, and (4) stretching of polymer chains between crosslinks caused by the increase in entropy associated with mixing polymer with solvent [1, 2]. Polyelectrolyte hydrogels have found a wide range of applications in diapers, inks and display devices, separation media, and clean up of aqueous spills. Polyelectrolyte hydrogels are particularly useful for a wide range of environmental applications, because expansion and contraction of the gels can be engineered to be triggered by small changes in environmental parameters such as temperature, pH, and ionic strength. However, until recently, reports on gels that will swell by absorbing large quantities of nonpolar organic solvents were nearly nonexistent. In nonpolar solvents, most polyelectrolyte gels collapse, because the oppositely charged ions within the gel form ion pairs that then aggregate, rather than becoming solvated.

In 2007, Sada (a research collaborator on this project) and colleagues [3] reported, for the first time, a novel class of lipophilic polyelectrolyte gels bearing positively charged repeating units (substituted tetraalkylammonium with long alkyl chains) and negatively charged counter-ions (substituted tetraphenylborate; $TFPB^-$) that swell dramatically but reversibly by absorbing organic solvents having various polarities ($\varepsilon = 1.9$-46; the lower the dielectric constant (ε), the less polar the solvent). Superior swelling ability in nonpolar solvents (illustrated in Figure 1, (a)-(b) is enabled by making both the polymer chains and the counter-ions lipophilic, preventing counter-ions from forming ion pairs, thereby enabling the solvation of ionic gel components in solvents. Lipophilic polyelectrolyte gel presented in Figure 1 (a)-(b) is hereby termed EG-18 and will serve as a candidate cleaner in this proposal. Figure 1 (c)-(d) illustrates swelling behavior of NG-18, a neutral analogue of EG-18 that does not contain the ionic tetraalkylammonium tetraphenylborate unit. As shown in Figure 1, neutral gel NG-18 swells to a much lesser extent than the ionic EG-18 gel. Neutral polymer gels swell in organic solvents because of the stretching of polymer chains between crosslinks caused by the increase in entropy associated with mixing polymer with solvent. Additional swelling mechanisms of polyelectrolyte gels such as the solvation of ionic groups do not exist in neutral gels. Therefore, neutral gels may be of limited use as cleaners compared to polyelectrolyte gels, but can be used as a measure of swelling capacity arising from the compatibility of the polymer chains with solvents alone.

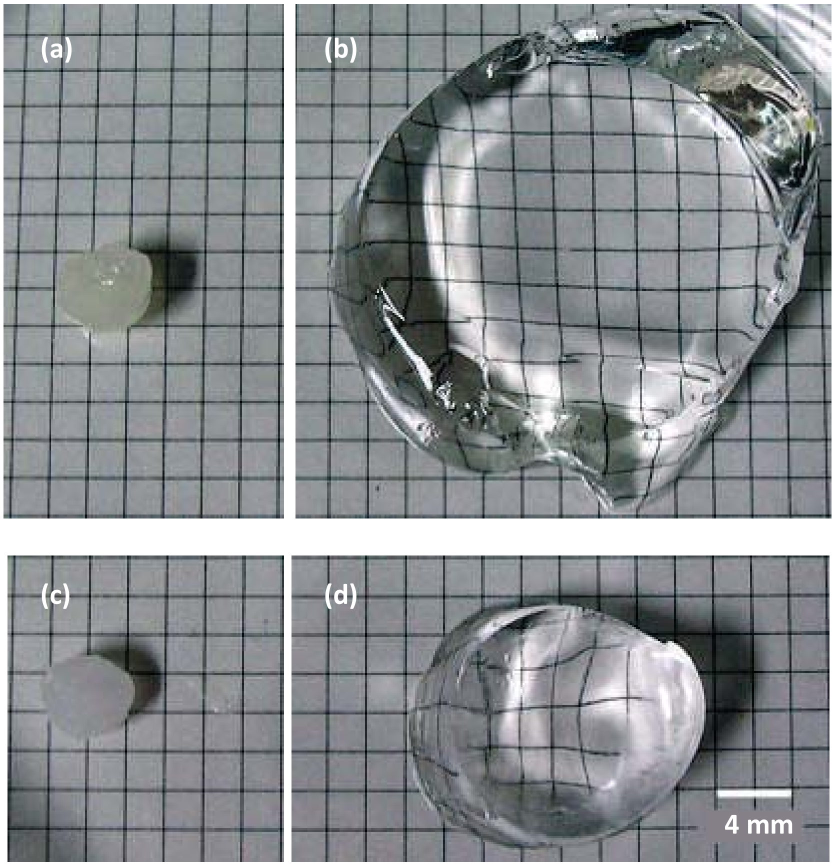
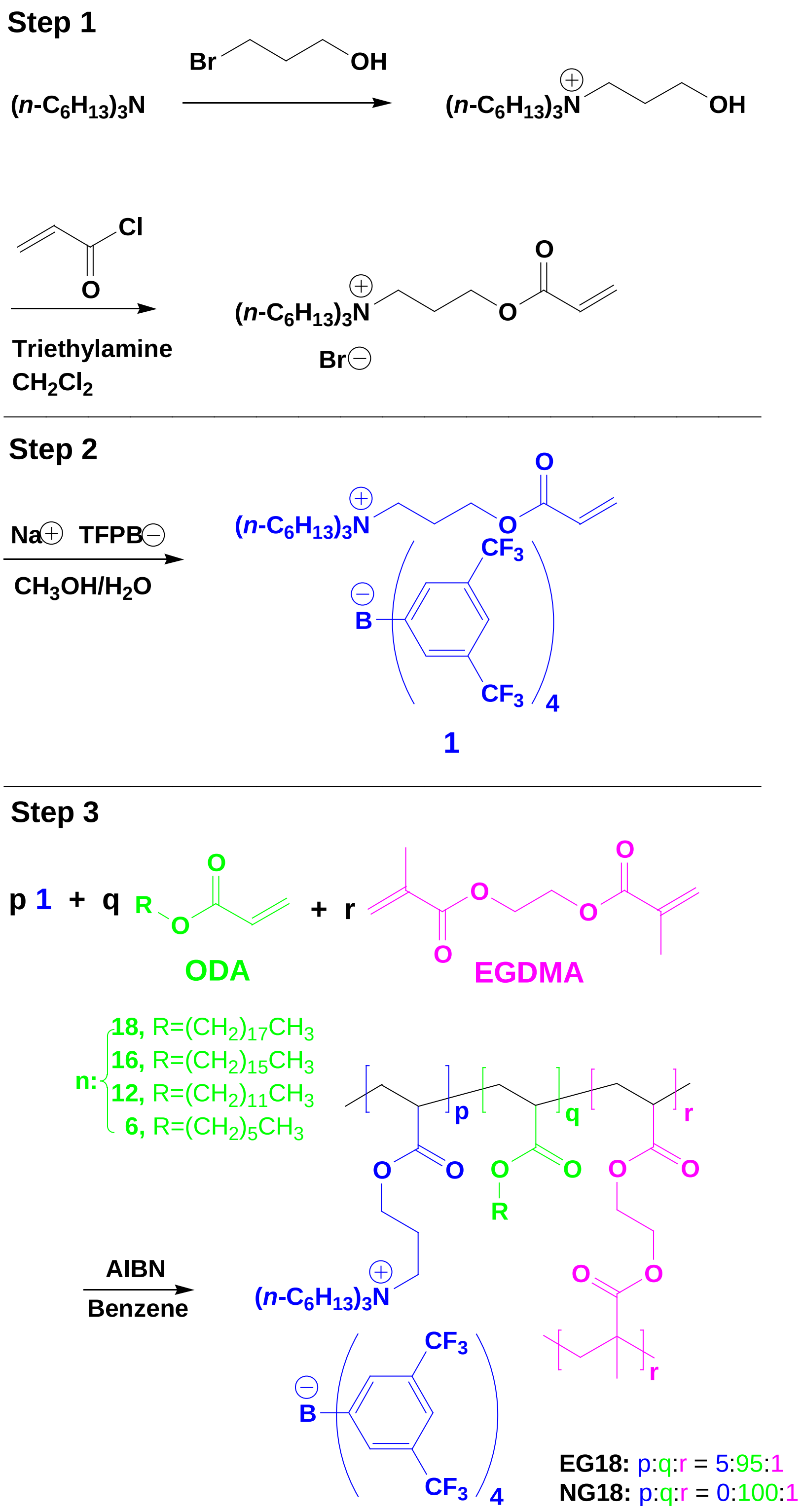


Figure 1. (a) Dry lipophilic polyelectrolyte gel (EG-18), (b) EG-18 gel swollen in tetrahydrofuran (THF) ($\varepsilon = 7.6$), (c) Dry neutral analogue (NG-18), (d) NG-18 gel swollen in THF. Figures are in scale with one another. Figure adapted from Reference [3].

Figure 2 presents pathways for preparing the lipophilic polyelectrolyte swelling gel (EG-18) whose swelling behavior in organic solvent was presented in Figure 1 (top). As shown in Figure 2, tetraalkylammonium tetraphenylborate polyelectrolyte gel (EG-18) can be prepared via the following steps:



Synthesis of quaternary alkylammonium halide salt.

Reaction of the product from Step 1 with substituted tetraphenylborate ($TFPB^-$; weakly coordinating lipophilic anion) to form lipophilic ionic acrylate monomer.

Copolymerization of the product from Step 2 with the polymer backbone octadecyl acrylate (ODA) using azobisisobutyronitrile (AIBN) as the initiator and ethylene glycol dimethacrylate (EGDMA) as the cross linker.

Figure 2. Preparation of candidate lipophilic tetraalkylammonium tetraphenylborate polyelectrolyte gel (EG-18) and its neutral analogue (NG-18). Figure adapted from Reference [3]

As illustrated in Figure 2, the ratio of ionic unit tetraalkylammonium tetraphenylborate (p), polymer backbone ODA (q), to cross linker EGDMA (r) for EG-18 is kept at p:q:r = 5:95:1 for EG-18 to maintain low content of ionic groups [4]. Ionic group content must be kept low to avoid aggregation of ionic groups. Neutral analogue NG-18 can be prepared by simply excluding the ionic tetraalkylammonium tetraphenylborate unit (p) for the feed ratio of p:q:r = 0:100:1 (Figure 2).

Figure 3 illustrates the impact of crosslinked polyacrylate polymer backbones on the swelling degree of ionic (EGn) and neutral (NGn) gels. Gels presented in Figure 3 possess polyacrylate backbones with alkyl chain lengths ranging from n=18 (R=$(CH_2)_{17}CH_3$), 16 (R=$(CH_2)_{15}CH_3$), 12 (R=$(CH_2)_{11}CH_3$), to 6 ((R=$(CH_2)_5CH_3$); see ODA structures in Figure 2). Swelling degrees (Q in wt/wt) in Figure 3 were quantitatively determined by soaking a selected gel in an appropriate solvent for a fixed time using the following equation:

$$Q = \frac{W_{wet} - W_{dry}}{W_{dry}} \qquad \text{Eq. 1}$$

where W_{dry} and W_{wet} are the weights of the dry and swollen gels, respectively.

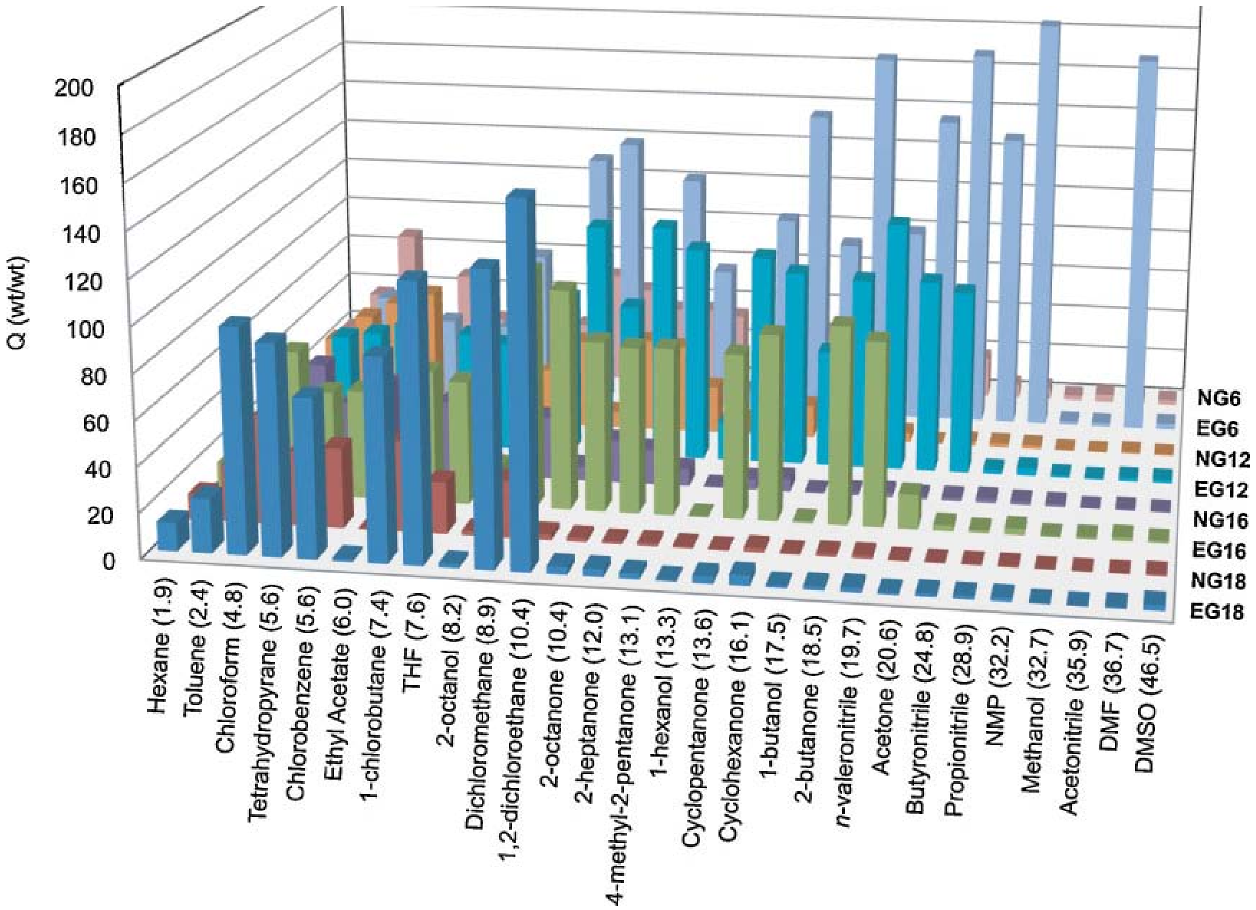


Figure 3. Swelling degree (Q) of lipophilic polyelectrolyte gels (EGn where n represents alkyl chain length of polyacrylate polymer backbone, as shown in Figure 2) and neutral analogues (NGn) in organic solvents (in increasing order of polarity from left to right). Figure adapted from Reference [4].

Figure 3 demonstrates that maximum absorbency of the polyelectrolyte gel (EGn) shifts toward solvents with lower polarity as alkyl chain length (i.e., lipophilicity) of polyacrylate backbone increases from n=6, 12, 16, to 18. That is, EG-18 exhibits maximum swelling by absorbing large quantities of organic solvents with dielectric constants between 3 and 10 (Figure 3). On the other hand, EG6 shows maximum absorbency for much more polar solvents (ε = 16-32). As shown in Figure 3, for a given alkyl chain length (n), ionic gel (EGn) swells to a much greater degree than the neutral analogue (NGn), as demonstrated visually for EG-18 and NG-18 in Figure 1. As shown in Figure 3, in solvents having dielectric constants below 3, comparable degrees of swelling are observed for ionic and neutral gels. In such nonpolar solvents, dissociation of ions within EGn is suppressed and ionic groups are tightly bound as ion pairs. As a result, the swelling of EGn results only from stretching of polymer chains between crosslinks caused by the increase in entropy associated with mixing polymer with solvent.

The following conclusions can be made from the review of recent and ongoing studies on polyelectrolyte and neutral lipophilic swelling gels provided above:

(1) Swelling degree and absorbency of lipophilic polyelectrolyte gels are much greater than their neutral analogues.

(2) Increased lipophilicity of both polymer backbones and ionic groups results in greater swelling capacity and absorbency in solvents having low dielectric constants.

A promising candidate is the lipophilic super-absorbent gel that will swell by absorbing nonpolar organic solvents (e.g., hydrocarbon oils, VOCs) several hundred times their dry weight. This study aims to utilize intricate designing of lipophilic super-absorbent swelling gels through careful selection of polymer backbone and ionic components, and the cross linking density. Designed swelling gels will function as the cleaner of the metal and non-metal surfaces by (1) absorption of oil and grease and (2) removal of particulate contaminants by self-generated mechanical forces obtained from swelling in (1). After the cleaning operation, cleaning media can be safely collected and recycled or used in fuel blends.

In order to develop a new cleaning technology based on lipophilic super-absorbent swelling gels for the removal of oil, grease and particulate matter from metal and non-metal surfaces, specific tasks are formulated to maximize the cleaning efficiency of lipophilic gels by (1) testing cleaning ability of candidates EG-18 and NG-18 gels and (2) designing lipophilic gels with improved cleaning ability by appropriate selection of lipophilic polymer backbone, weakly coordinating anions, and enhancement of mechanical strength. Elimination of fluorinated compounds in the gel synthesis was the focus of this and subsequent phases of this research.

TECHNICAL APPROACH

Our research has identified two potential gels, a lipophilic polyelectrolyte (EG-18) gel and a neutral (NG-18) polymer gel for surface cleaning applications. These gels were evaluated during this initial phase of the study following the ASTM G122-96(2008) and MIL-PRF-680B protocols. Representative contaminants of oil, grease and particulate materials were selected. The proposed scope of the full project was to elucidate the chemical and physical mechanisms in the removal of oil, grease, and particulate contaminants from metal and non-metal surfaces by lipophilic super-absorbent swelling gels. Research outcomes will facilitate the development of environmentally compliant, economically feasible cleaners for a wide range of DoD applications, providing a promising alternative to traditional vapor degreasing, solvent, aqueous, or blast cleaning processes. EG-18 gel studies are not included in this report as these were reported by Sada and co workers [3].

Materials and Methods

All chemical reagents were used as received. Stearylacrylate (SA), ethylene glycol dimethacrylate (EGDMA), benzene, azobisisobutylonitrile (AIBN), methanol, ethanol, diethylether, and carbon tetrachloride were purchased from Sigma-Aldrich (Milwaukee, WI).

Tetrahydrofuran (THF), isopropanol, acetonitrile, and dichloromethane were obtained from Acros Organics (Morris Plains, NJ). Dimethylsulfoxide (DMSO) and 1-octanol were supplied from Alfa Aesar (Ward Hill, MA). Chloroform and cyclohexane were distributed from VWR International (West Chester, PA). Acetone, methylisobutylketone (MIBK), toluene, and *n*-hexane were supplied from Fisher Scientific (Pittsburgh, PA). An Instron 5900 Electromechanical System was used for the compression of the gels. A Jasco FT/IR-4100 Fourier Transform Infrared Spectrometer was used for infrared spectroscopy.

Synthesis

A typical protocol for the synthesis of NG-18 gel is as follows: 10.0 g (30.8 mmol) of SA (monomer) and 61 mg (0.31 mmol, the case of x=1) of EGDMA (crosslinker) as initiator were placed in a vial tube and dissolved in 2 mL of benzene by heating at 50 °C. Oxygen in the solution was excluded by bubbling nitrogen gas for 45 min then 101 mg (0.62 mmol) of AIBN was added. The vial tube was sealed tightly and heated at 65 °C for 24 h for polymerization. Gels with low crosslinking densities were prepared in a similar way by reducing the feed ratios of EGDMA. The synthesized gels were washed by swelling in hexane repeatedly, air-dried for 2 days, and dried in vacuum overnight. SA-*co*-EGDMA with two crosslinker ratios were prepared by radical copolymerization, which are represented as NG-18-x% (x= 1 or 0.5; x denotes the mole ratio of crosslinker to SA.

A typical protocol for the synthesis of EG-18 gel is as follows: 125 mg (0.1 mmol) of $TFPB^-$ and 617 mg (1.9 mmol) of ODA, 3.96 mg (0.02 mmol) of EGDMA, and 6.57 mg (0.04 mmol) of AIBN were placed in a capillary of 7.0 mm in diameter and dissolved in benzene adjusted to 1.0 mL. The solution was degassed and polymerized by heating at 60°C for 24 h. The feed ratio was adjusted to $TFPB^-$:ODA:EGDMA = 5:95:1. Gels with low crosslinking densities were prepared in a similar manner by reducing the feed ratios of EGDMA. The formed gels were washed by swelling in benzene for 10 h, and then air-dried at room temperature. The sample was cut into cylinders of about 1.0 mm in length, and the cylinders were dried *in vacuo* at 40°C.

Synthesis of EG-18 gel was performed by Sada and co workers [3] at Kyushu University, Japan.

Characterization

The Fourier transform infrared (FTIR) spectra were obtained using Jasco FT/IR-4100. Compression strength was measured with Instron 5900 electromechanical system and the compression speed was 0.25 mm/min. UV-vis spectra were colleceted using a ThermoSpectronic Aquamate 100 UV-vis Spectrometer.

Swelling Studies

Swelling behavior of NG-18 gels was determined with the following solvents of various polarities at 25±1 °C using 5 mL vials: water, DMSO, methanol, ethanol, isopropanol, 1-octanol, acetone, MIBK, acetonitrile, THF, diethylether, dichloromethane, chloroform, carbon tetrachloride, benzene, toluene, hexane, and cyclohexane. The mass of each empty vials was recorded and then a specified amount of dried gel was added to each vial. The vials were weighed and the amount of dried gel was noted (W_{dry}). The vials were then filled with a solvent and allowed to equilibrate for 24, 48 and 72 hours. The excess solvent was removed from the vials and the gels were weighed again (W_{wet}). The amount of solvent absorbed by the gels was obtained from the difference in weights. The swelling degree (Q) was defined by Equation 1.

Temperature dependence on swelling degree of NG-18-1% gel was measured in the above solvents at 20, 40, 60, (60→) 0, and (25→) 0 °C. Here, (60→) 0 °C indicates that the sample was heated at 60 °C to achieve the equilibrium once and then cooled to 0°C. This was performed to investigate the record of the heating and cooling process. Due to low boiling points, dichloromethane and diethylether were not used at 40°C, similarly acetone was not used at 60°C. Likewise, DMSO and cyclohexane were not used at 0 °C due to high freezing points. These values were indicated as Q = 0. To understand the kinetics of swelling behavior, the above procedure was followed with several vials and the amount of solvent absorbed was determined at different time intervals.

Critical Temperature Studies

Critical temperatures were determined by UV-vis spectroscopy. Swollen NG-18-1% gel in THF was placed in a temperature controlled quartz cell, which was monitored with thermocouples (OMEGA DP462). The transmittance at 700 nm was measured as a function of temperature by changing temperature at 0.1 °C/min. While the swollen gel was transparent, the collapsed gel was opaque. The values of critical temperature in the heating and cooling process were obtained from a plot of transmittance versus temperature.

Cyclic Temperature Change Test

Cyclic temperature changes of both swelling degree and transmittance were performed to ascertain the reversibility of the gel. In the swelling test, a piece of NG-18-1% gel was first placed in THF for 48 h at 25 °C. Excess THF was removed from the vial, weighed, and the swelling degree was calculated. Then the vial was filled with THF again, placed at 0 °C for 24 h, and swelling degree was measured by the same procedure. This cycling was repeated five times in total. In the transmittance study, THF swollen gels were placed in a temperature controlled quartz cell. The transmittance at 700 nm was measured at 25 °C and 0 °C alternatively. Each step took about 30 min, and the procedure was repeated for a total of five cycles. The gels achieved equilibrium values at each step in both swelling and transmittance test.

Compression Strength

A piece of dried NG-18-1% and 0.5% gels was swelled by adding excess toluene or THF. After eliminating extra solvent, compression strength was measured at a compression speed of 0.5 mm/min.

Oil Absorption

Stainless steel metal coupons were washed by acetone and methanol, and dried *in vacuo* for 72 hours. The coupons were soaked in SAE-30 oil and allowed to drip excess oil for 30 minutes. Half of these coupons were also sprayed with alumina powder. The contaminated coupons were immersed in NG-18 gels or toluene for 30 minutes. The percent of oil absorbed was then measured by comparing the weight of each coupon before and after immersion.

Analogous procedure was followed for field samples obtained from a Naval cleaning facility.

Grease Cleaning

Metal coupons were prepared according to MIL-PRF-680B (Appendix A), and uniformly coated with MIL-PRF-10924 grease. Beakers with the NG-18 gels and trichloroethylene were placed into an ultrasonic cleaner. The test was started with a timer. The coupons were observed until all grease was visibly removed from the metal coupon, and the time was recorded in minutes. If a portion of grease remained on the metal coupon after 100 minutes, the test was immediately terminated with the testing time being recorded as 100 minutes. The cleaning power was determined by the equation

$$\text{Solvent cleaning power}\,\% = \left(\frac{100-A}{100}\right)\times 100 \qquad \text{Eq. (2)}$$

where A is the average cleaning time in minutes of the three tested runs [18].

Results and Discussion

Characterization of NG-18 Gels

FT-IR spectra of SA monomer and NG-18-1%, -0.5% are provided in Figure 4. Compared to the spectrum of stearylacrylate monomer, NG-18 gels showed the disappearance of peaks in four regions. Each peak was identified as follows: 1634 cm^{-1} is C=C bond vibration, 1410 cm^{-1} is C-H of C=CH$_2$ in-plane scissoring, 1297 cm^{-1} is C-H of C=CH in-plane vibration, and 997 and 893 cm^{-1} are C=CH out-plane vibration. The disappearance of these peaks of the vinyl group indicates that NG-18 gels include a little non-reacted SA. Figure 5 shows the compression strength of swollen NG-18-1% gel in THF. The first breaking point is the stress of 0.371 MPa and the fracture strain of the gel is λ=67 %. The NG-18-1% gel could withstand a similar degree of compression as reported by single network poly acrylamide gel prepared by Gong et al. [11].

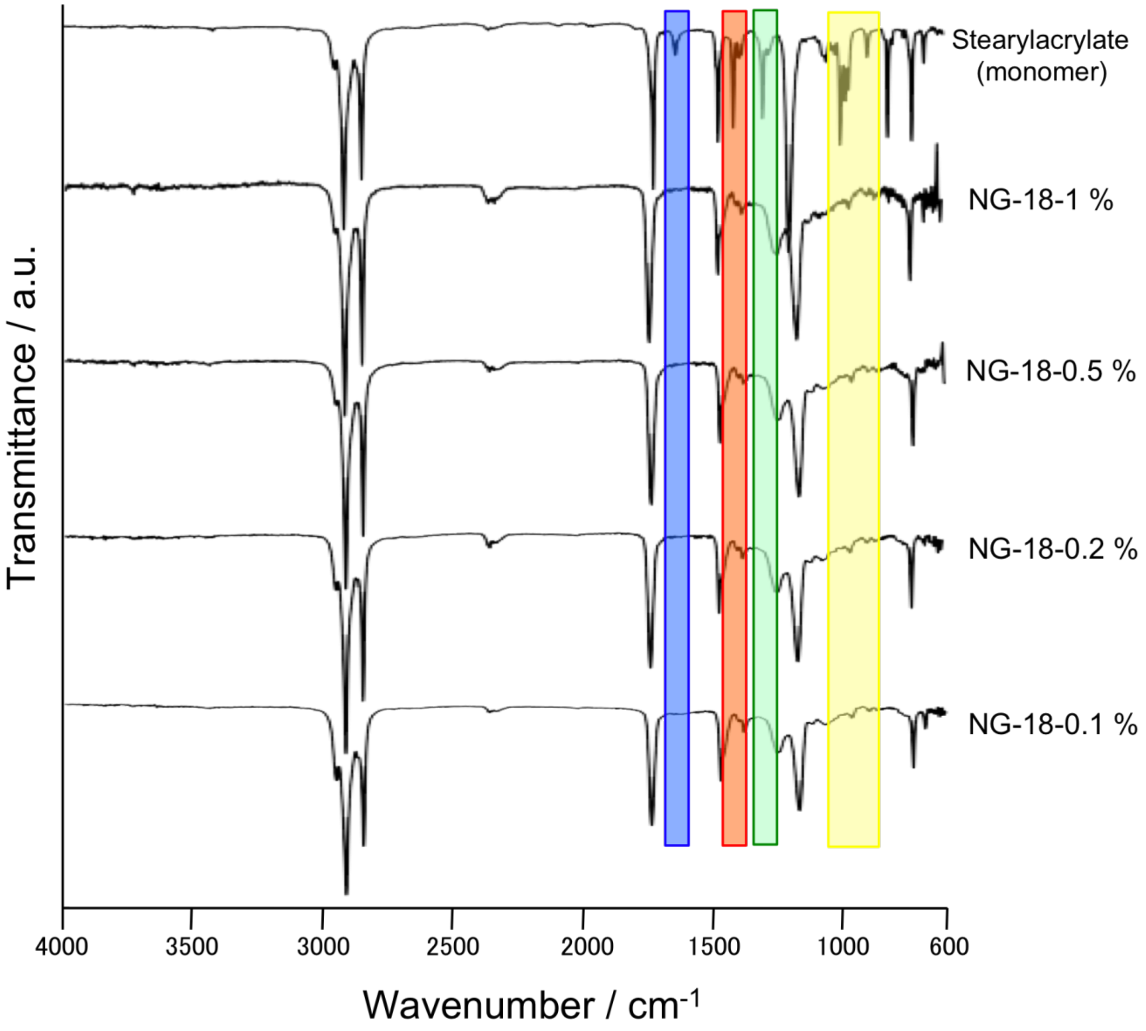


Figure 4. FTIR spectra of stearylacrylate

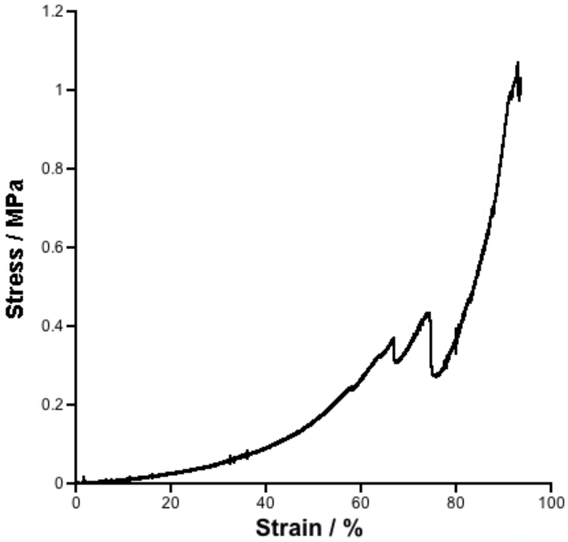


Figure 5. Compression strength of swollen NG-18-1%, THF has a breaking point around 0.371 MPa.

Swelling Behavior

The swelling behavior of NG-18 gels (NG-18-1% and -0.5%) in solvents with various polarities from cyclohexane to water at 25 °C over the time periods of 24, 48 and 72 hours were investigated (Figure 6). The swelling degree increased with increasing polarity from cyclohexane, and the maximum value was observed in chloroform. On the other hand, the gels collapsed in the more polar solvents (dielectric constant $\varepsilon > 10$). Particularly, NG-18 gels absorbed large amounts of chlorinated solvents such as chloroform (Q=35 on NG-18-1%) and carbon tetrachloride (Q=36). Moreover, NG-18 swelled in a moderate amount of solvents such as ether (diethylether: Q=12, THF: Q=17), aromatic compounds (benzene: Q=21, toluene: Q=22), and aliphatic reagents (hexane: Q=14, cyclohexane: Q=20). In more polar solvents, such as water, DMSO, alcohols (methanol, ethanol, isopropanol, and 1-octanol), ketones (acetone and MIBK), and acetonitrile, NG-18 didn't swell at all (Q<1). Also, enhancing swelling ability by reducing the cross linker density was attempted. Reducing the feed ratio of the crosslinker to the monomer from 1 mol% to 0.1 mol% enhanced the swelling ratio. However, the gels less than 0.2 mol% crosslinker density were too soft to separate excess solvent and swelling degree could not be accurately measured. NG-18-0.5% indicated the same tendency as NG-18-1% and had a higher swelling degree than NG-18-1%. These swelling behaviors of NG-18 gels essentially depend on the compatibility of the polymer chain with the media. NG-18 didn't allow penetration of the highly polar molecules into the polymer networks, while non-polar solvents were absorbed.

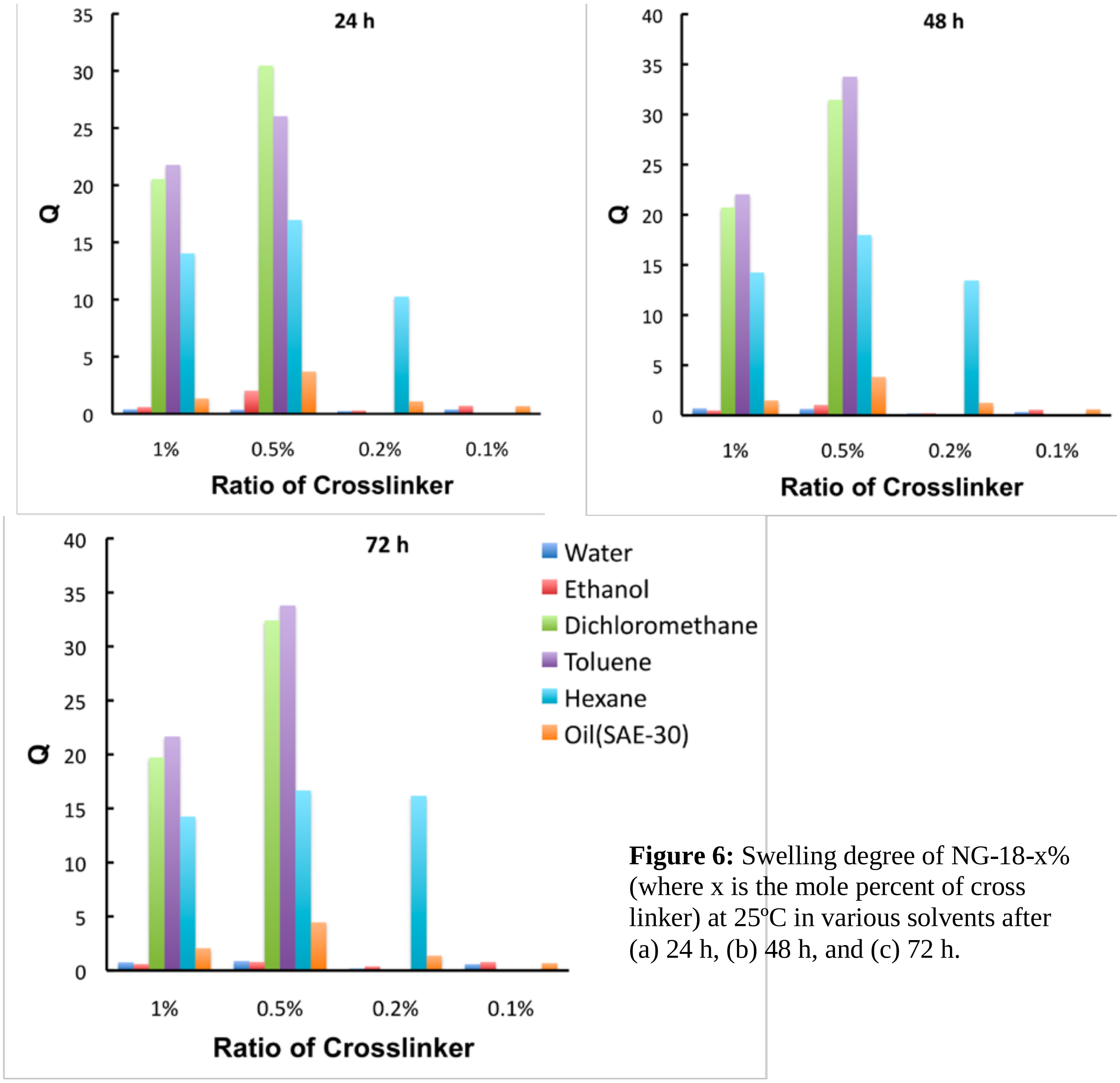


Figure 6: Swelling degree of NG-18-x% (where x is the mole percent of cross linker) at 25°C in various solvents after (a) 24 h, (b) 48 h, and (c) 72 h.

Subsequently, temperature dependence on the swelling degree with NG-18-1% gel was examined in Figure 7. The temperature was changed from 20 °C to 40, 60, and 0 °C successively, and the variety of solvents is the same as ones used in swelling degree test at 25 °C. Also, the sample cooled from 25 °C to 0 °C was studied to investigate the influence of thermal temperature changes during the heating process for swelling behavior, which is represented as (25→) 0 °C. The comparison between (60→) 0 °C and (25→) 0 °C was summarized in Figure 8.

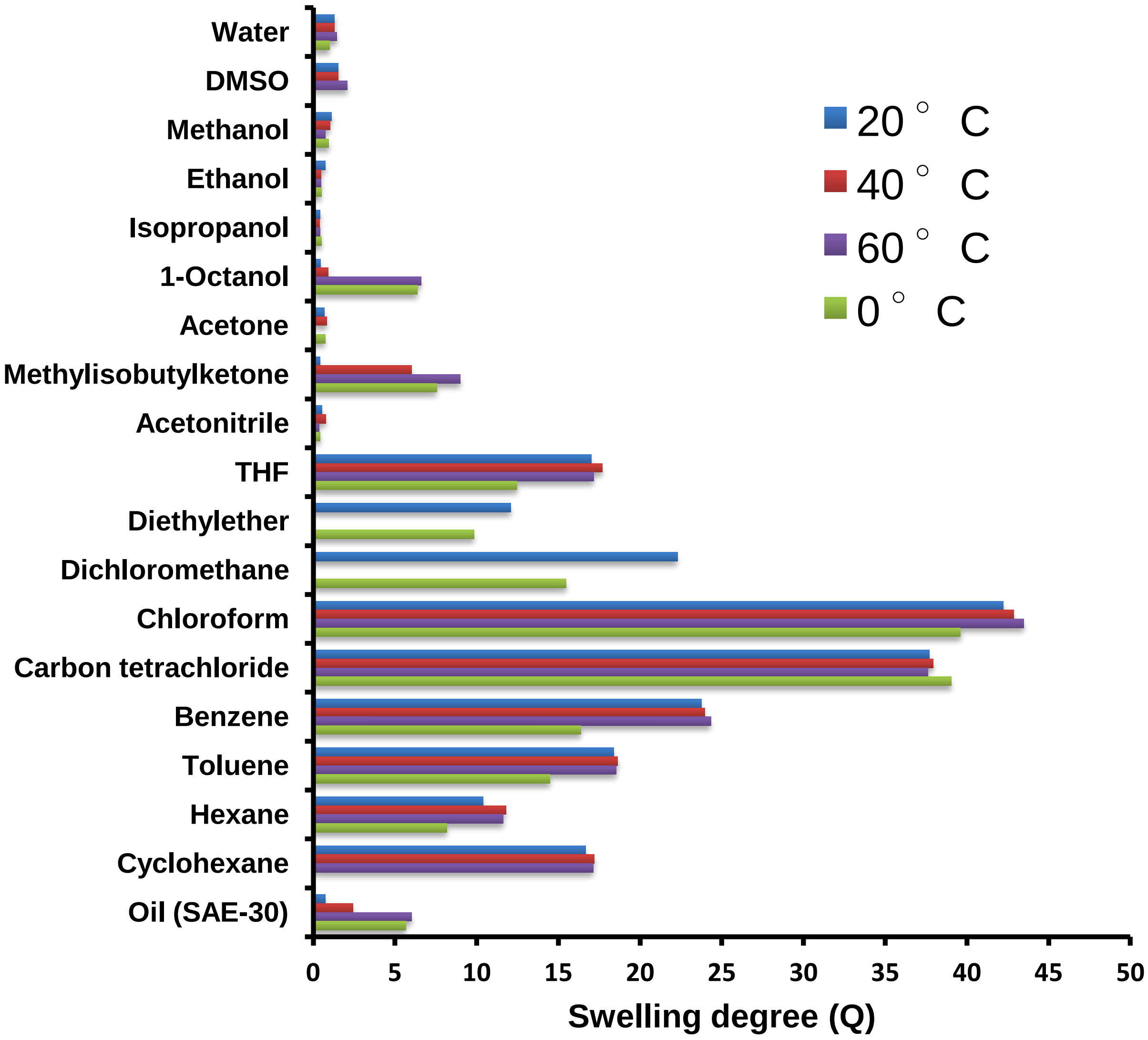


Figure 7. Temperature dependence of swelling degree (Q) of NG-18-1% in various solvents.

The results in Figure 7 were categorized as follows: 1) In the following solvents swelling degree did not change in both heating and cooling processes: water, DMSO, methanol, ethanol, isopropanol, acetone, acetonitrile, carbon tetrachloride, and cyclohexane. 2) Whereas the Q value didn't change by heating a maximum of 25 wt% of the following solvents was dislodged from the gel in cooling process: THF, diethylether, dichloromethane, chloroform, benzene, toluene, and hexane. 3) The swelling degree increased by heating, but was almost the same by cooling in the solvents 1-octanol and MIBK. The second category is especially remarkable because it showed the changes of swelling degree and the color change from transparent to opaque by cooling to 0 °C. These transition behaviors are attributable to crystallization of long-alkyl chain among stearylacrylate. It is expected that this ability can be utilized to develop a VOC recycling system composed of both uptake and ejection. On the other hand, the transitions in the third category were irreversible as shown in Figure 8.

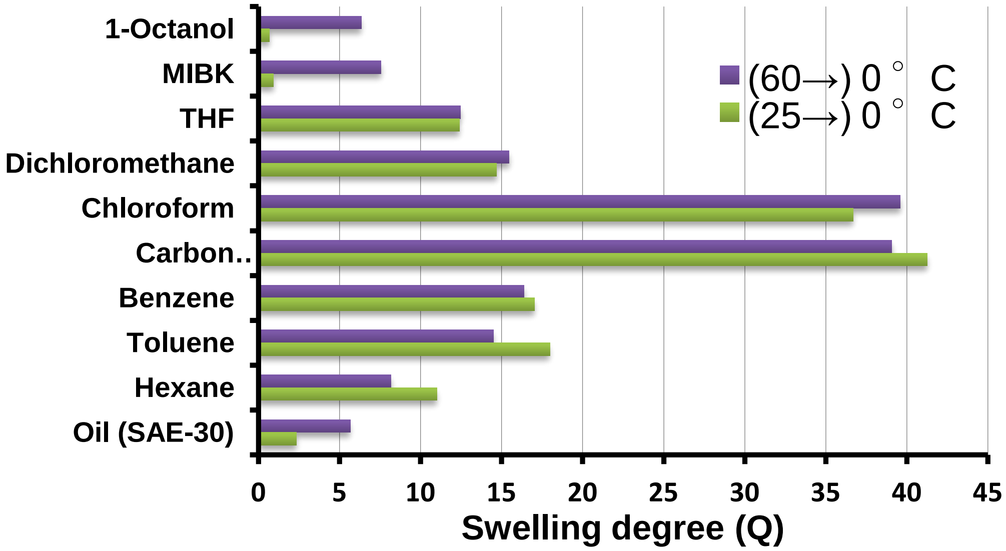


Figure 8. Thermal response of NG-18 gels

Additionally, in order to determine the time dependence on the swelling degree of NG-18 gels in THF, the swelling ratio was determined as a function of time. Figure 9 shows the time required for each gel to reach the equilibrium swelling degree. A cubic dry gel (NG-18-1%, -0.5%), 5 mm on a side, was placed in a vial with excess THF at 25 °C.

The kinetics of the swelling behavior was examined by fitting the data to Lagergren pseudo-first and pseudo second order kinetic equations [20-22]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad \text{Eq. (3)} \qquad \frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad \text{Eq. (4)}$$

The values of the first and second order rate constants obtained through the linearization of equation 2 and 3 are included in Table 1, along with the values of the regression coefficient, R^2, which describes the correlation between graphed points with one being the best possible correlation and zero being the worst. A second order kinetic equation better describes the swelling behavior of NG-18 gels because the regression coefficient is closer to one.

Table 1. Lagergren first and second order rate constants (k_1 and k_2) for swelling of the NG-18 gels

	First order		Second order	
Sample	k_1 min^{-1}	R^2	k_2,g g^{-1} min^{-1}	R^2
NG-18-1%	4.38x10^{-3}	0.971	5.55x10^{-4}	0.998
NG-18-0.5%	5.30x10^{-3}	0.985	5.72x10^{-4}	0.999

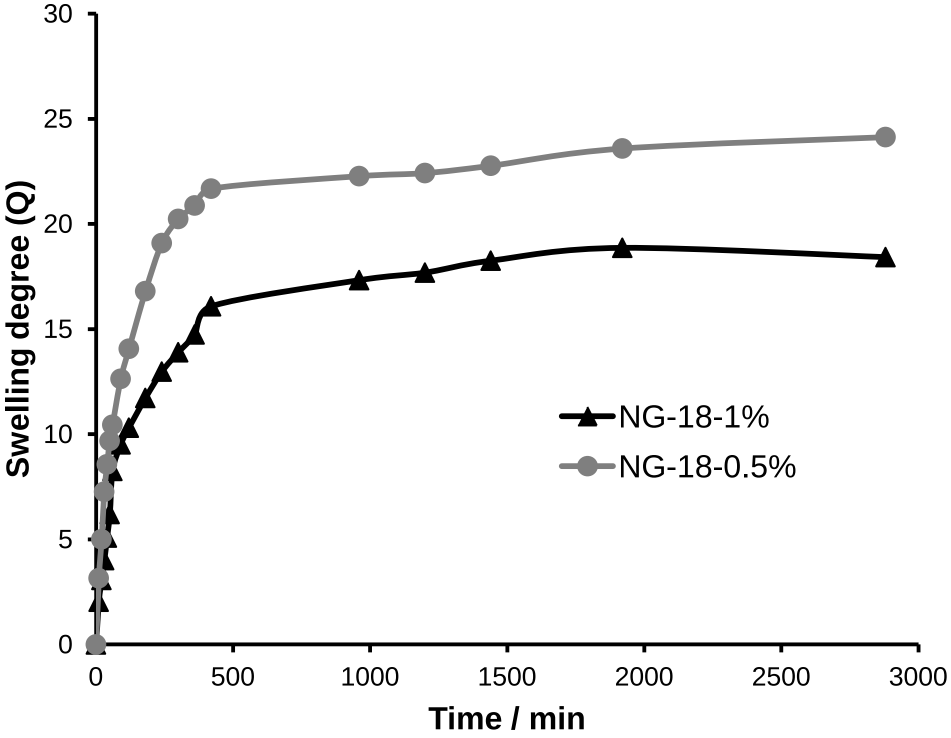


Figure 9. Swelling degree changes of NG-18 with time in THF

Critical Solution Temperature

Critical solution temperature was determined for swollen NG-18-1% in THF. Figure 10 shows the result obtained at 700 nm. The swollen gel is relatively transparent, while the collapsed gel is opaque. Thus, the transmittance values sharply change when the gel is collapsed. The transmittance values were plotted against temperature to obtain approximate critical temperature in both the heating and cooling processes. Transition temperature results in 6.6 °C in the cooling process and 12.4 °C in the heating process. This hysteresis was due to supercooling phenomenon on the cooling process. This transition process is different from NIPAM (N-isopropylacrylamide) in water, depending on the crystallization of long alkyl chain among stearylacrylate unit.

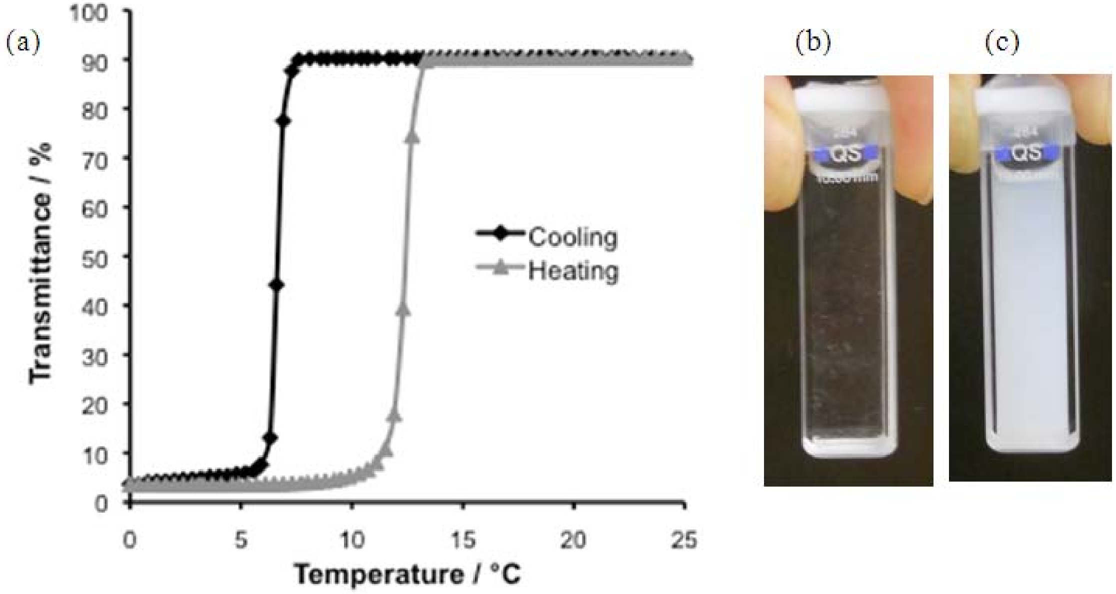


Figure 10. (a) Temperature dependence of transmittance at 700 nm of NG-18-1% gel swelled in THF. (b) Photograph of transparent state at 25 °C. (c) Photograph of opaque state at 0 °C.

Thermal Cycling of NG-18-1% gels

The cyclic swelling degree and transmittance studies were performed in order to investigate the reversibility and reproducibility of the swelling behavior. The procedures were followed for five cycles for NG-18-1% gels and the results are shown in Figure 11. The gel appears to be stable and retains its transition characteristics even after five cycles. In other words, this transition is reversible and non-destructive for the gel network. The change of transmittance is very fast and the color change occurs in less than 30 min, but the change of swelling degree was slow, taking more than 24 h. This means that the color change is part of the swelling process but the color change is not equivalent to the change of swelling degree.

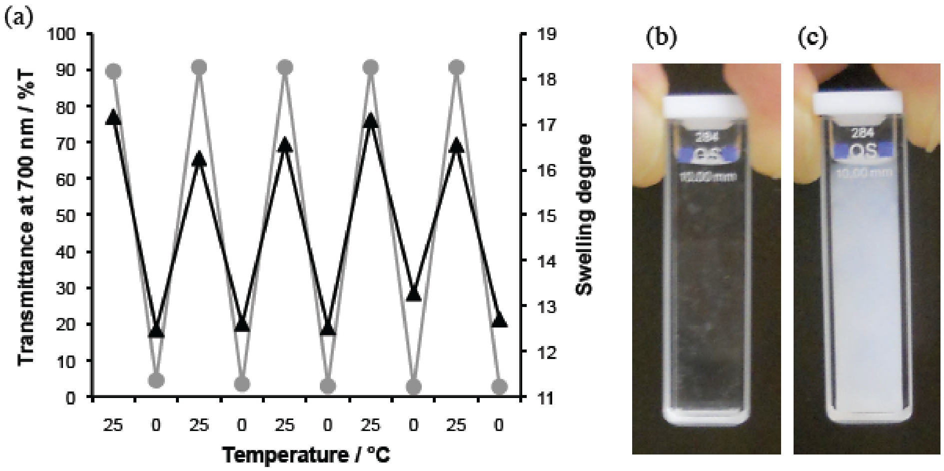


Figure 11. (a) Results of thermal cycling (25 °C →0 °C) test for NG-18 gels: changes of (circle) transmittance at 700 nm, (triangle) swelling degree in THF. (b) Photograph of transparent state at 9th step. (c) Photograph of opaque state at 10th step.

The cyclic swelling degree was also preformed with the solvent cyclohexane. The procedures were follow for 3 cycles for NG-18-1% gels and the results are shown in Figure 12. The gel is stable after three cycles. Cyclohexane is more environmentally benign than THF (Appendix B), and shows a similar swelling degree (Q=16 for cyclohexane and Q=17 for THF). Further testing of additional cycles will be carried out during the subsequent phases of this research.

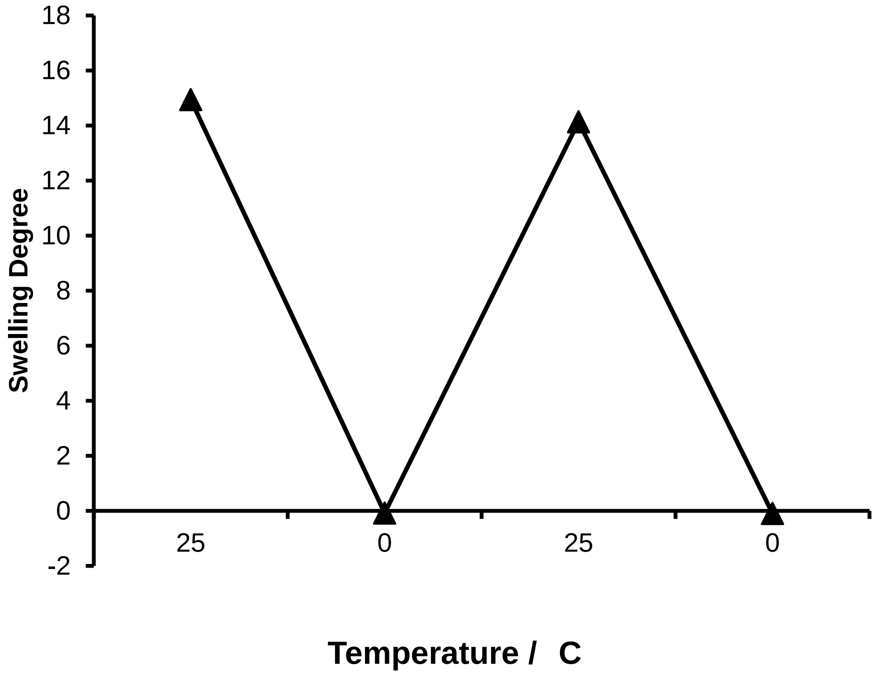


Figure 12. Results of thermal cycling of (25°C → 0°C) NG-18-1% gel swollen in cyclohexane

Compression Studies

The first breaking point is 0.167 N in NG-18-1% and about 1N in NG-18-0.5%. Compared to EG-18, NG-18 gels are much stronger (Figure 13).

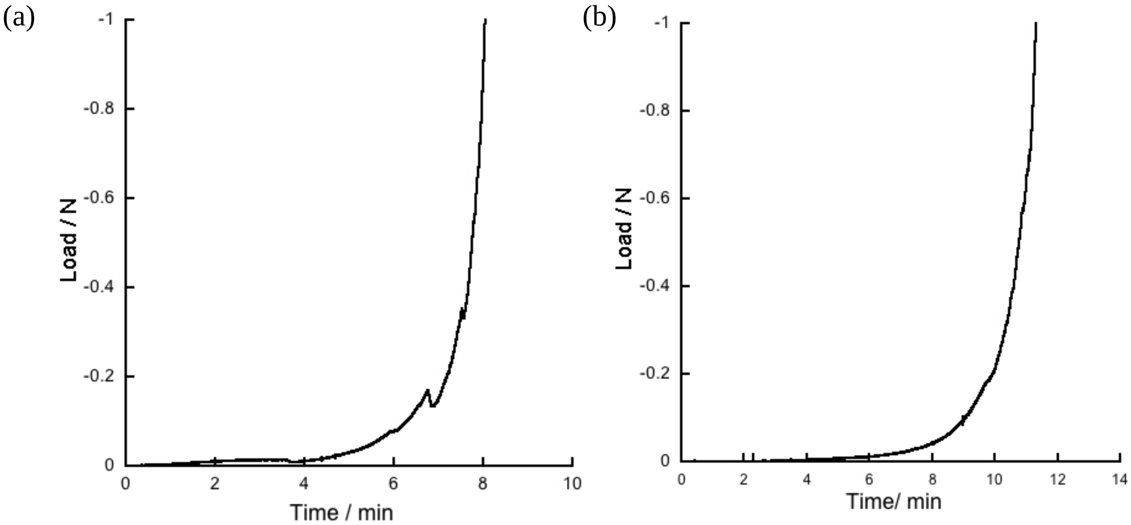


Figure 13. Compression strength of swollen (a) NG-18-1% and (b) NG-18-0.5% in toluene.

Oil Absorption

Oil absorption was tested with both NG-18 gels in toluene and in THF. The results of the tests with NG-18-X with swollen in THF are shown in Figures 14-15. It was determined that NG-18-1% in THF was the best performer in this category because of its high swelling degree, good recyclability, the high oil and alumina absorption properties. THF is also a relatively low- or non-toxic and environmentally friendly solvent. NG-18-0.5% gel was not able to clean all of the alumina powder and oil. Along with metal coupons, NG-18 gels were tested on painted coupons and stainless steel parts with bolts, the results of which are shown in the appendix C. The painted coupons did not show any signs of peeling, and the bolts and flat coupons were cleaned almost as well as using solvent by itself.

Temperature did change the swelling degree of NG-18-1% in 1-Octanol, methylisobutylketone, and SAE-30 oil when heating from 20 °C to 40 or 60 °C (Figure 7). Even if they were cooled to 0 °C, however, they did not collapse.

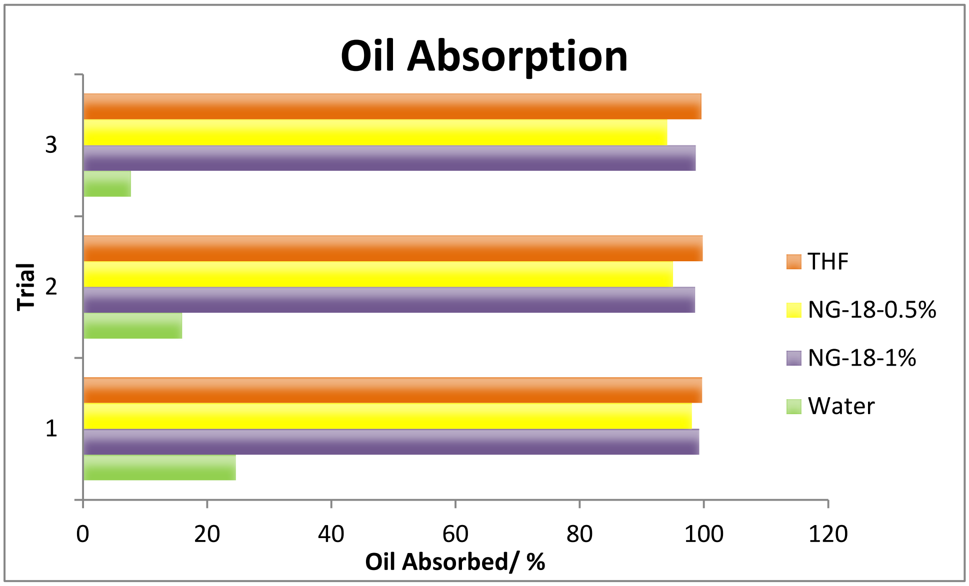


Figure 14. Oil absorption properties of NG-18 gels.

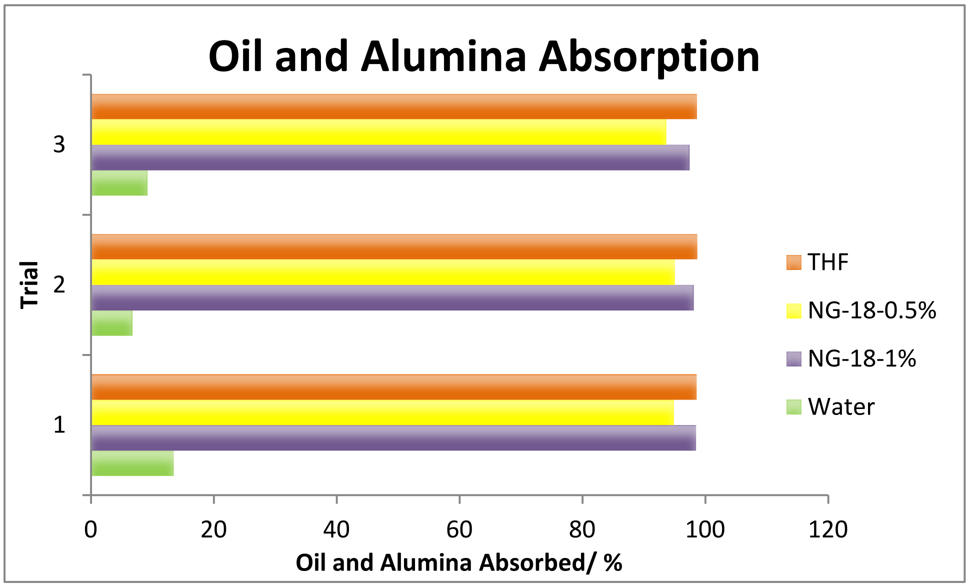


Figure 15. Metal surface cleaning properties of NG-18 gels.

In addition to testing performed on metal coupons, field samples obtained from Portsmouth Naval Shipyard, Portsmouth, NH. These parts include threaded sail adjustment screws, hadraulic valave stems, and miscellaneous nuts and washers. Some of these tested parts are shown in Figures 16-17. The samples consisted of large bolts and nuts approximately four to six inches long. Rusted matertials can be cleaned of grease and oil, but not of rust, because rusting is a chemical process and not a physical process. Rusted parts in general are discarded.

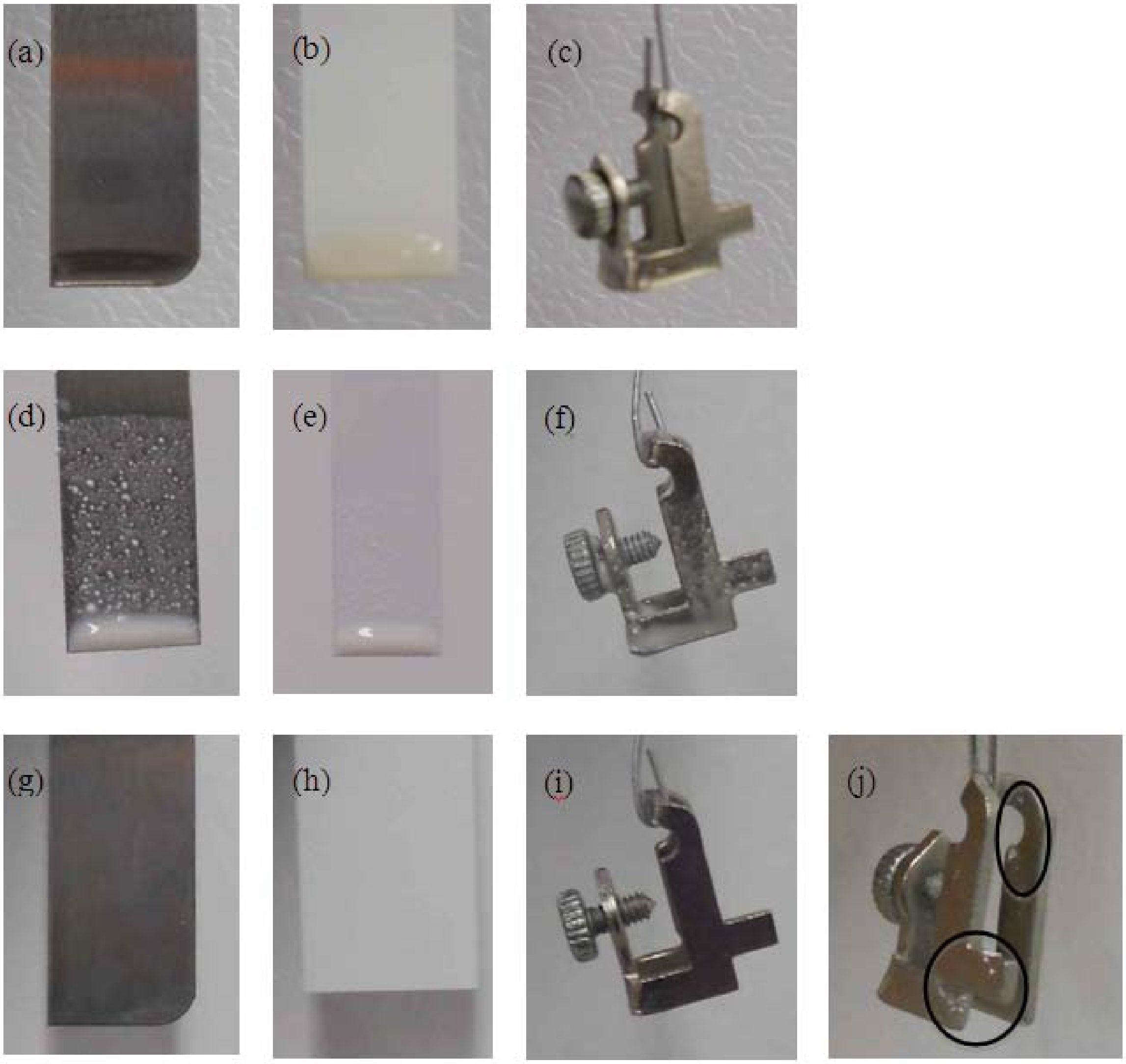


Figure 16. Photographs of metal coupons (a)-(c) soaked in SAE-30 oil, (d)-(f) soaked in the mixture of SAE-30 oil and alumina powder, (g)-(i) immersed in NG-18-1% gel, and (j) immersed in NG-18-0.5% gel (circles shows that some alumina remaining on the surface).

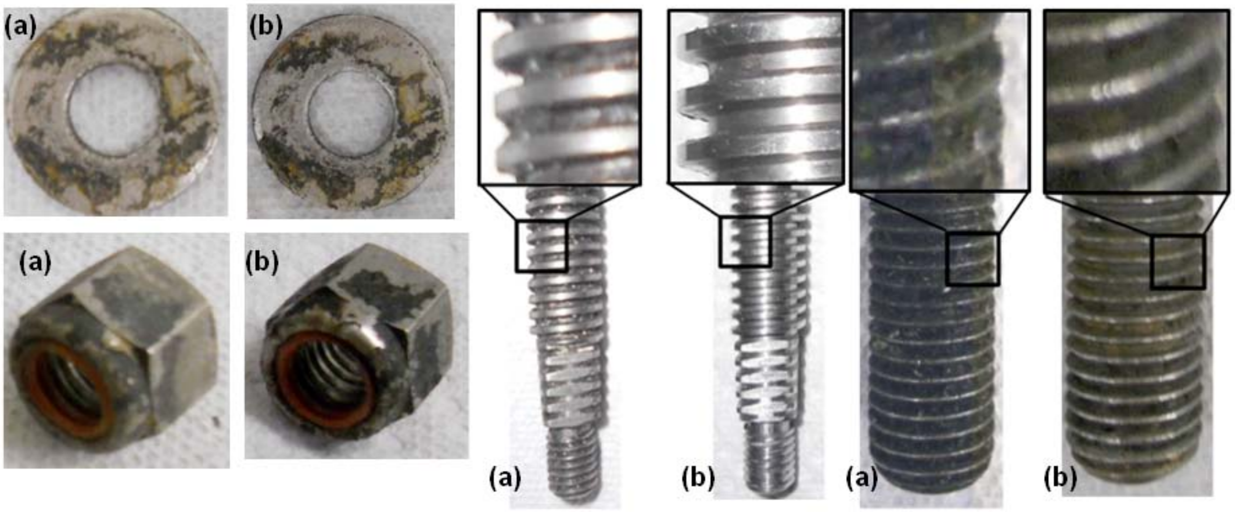


Figure 17. Photographs of soiled metal parts cleaning tests (a) before and (b) after cleaning with NG-18-1%gel swollen in THF.

Grease Cleaning

The grease absorption capabilities of NG-18 gels were comparable to trichloroethylene all showing greater than 99% absorption capabilities. As shown in Figures 18-19, NG-18-0.5% outperformed the TCE in two of three trials. NG-18-1% showed a greater cleaning power than NG-18-0.5% but had less grease absorption. This could be prevented further rinsing with ethanol immediately after cleaning if necessary.

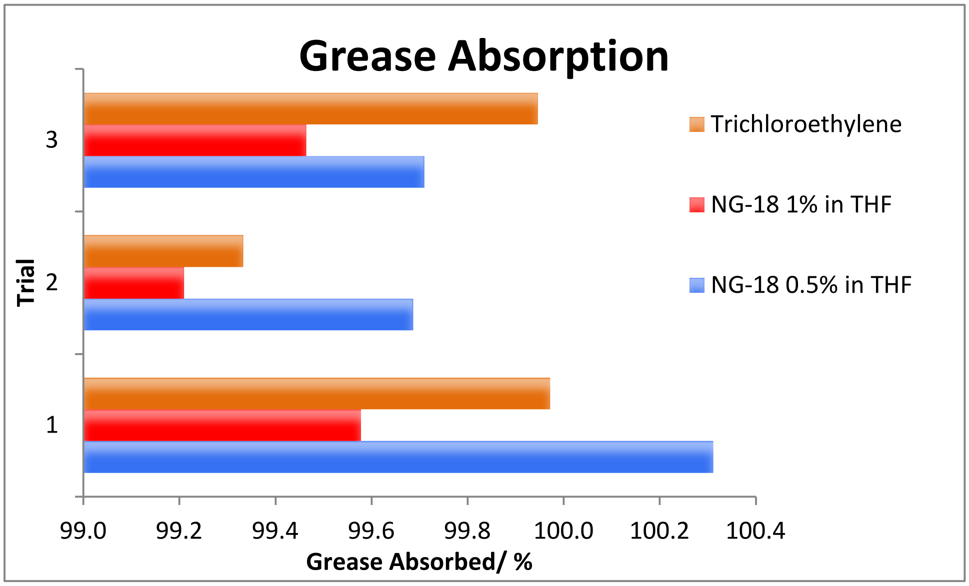


Figure 18. Grease absorption properties of NG-18 gels

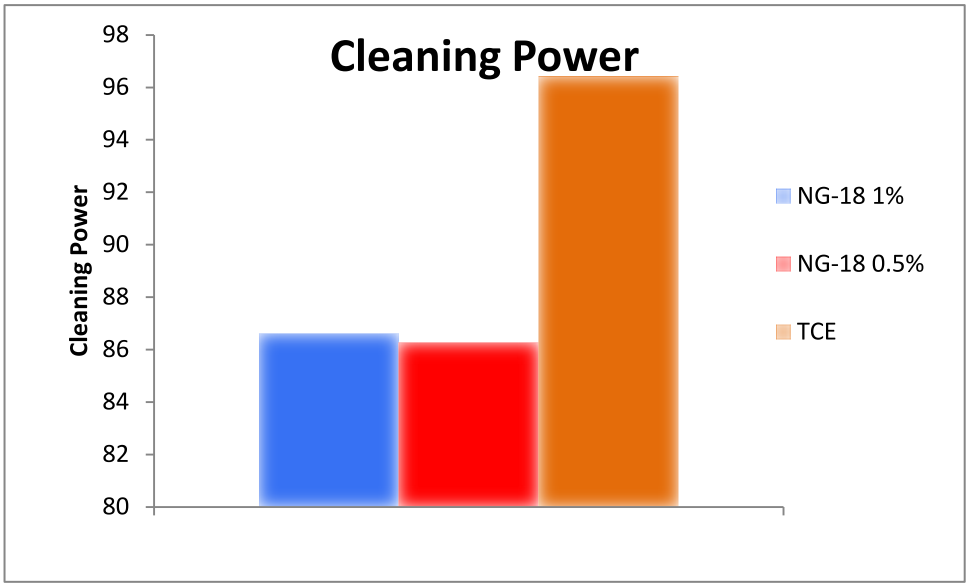


Figure 19. Grease Cleaning power of NG-18 gels

Recyclability of NG-18 Gels:

Figure 20. Cyclic surface cleaning process with swollen NG-18-1% gel in THF

The recyclability of NG-18 1% gel for surface cleaning is illustrated in Figure 20. This process is pending patent application [19]. The recyclability was tested by five consecutive oil absorption-desorption processes (Figures 21-22). The swelling degree of the gel in each step was not measured because the initial weight of the gel can change due to addition of oil and particulates. Instead, the amount of absorbed oil squeezed out by the collapsed gel was weighed in each step. The weight of the particulates and THF were accounted for by filtration, and evaporation, respectively.

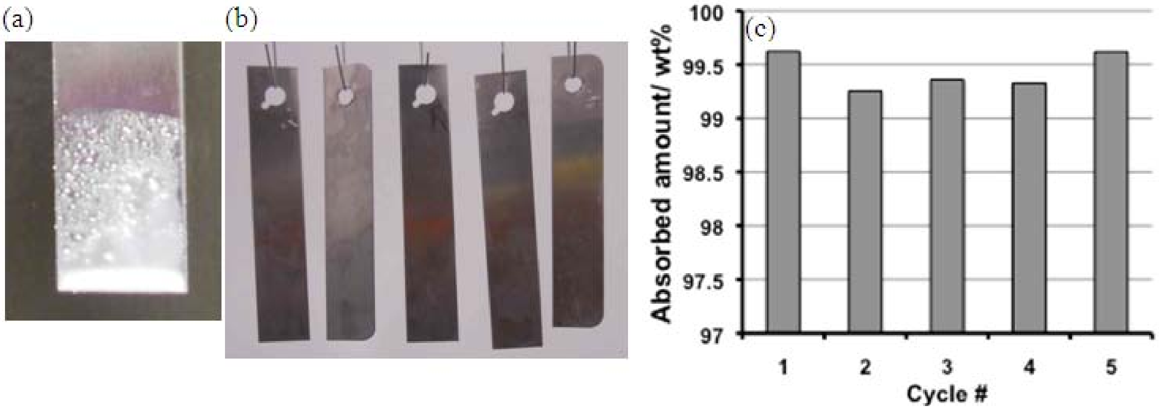


Figure 21. Photographs of metal coupons (a) before and (b) after immersing the NG-18-1% gel: from left side, cycle1-5. (c) Absorbed amount in each cycle.

The cleaning property of NG-18-1% gel was maintained at >99 wt% even after five cleaning cycles. By cooling, a solution containing particulate, THF, and oil was removed from the gel. The particles were removed by filtration, and 10-25% THF could be evaporated, recollected and recycled.

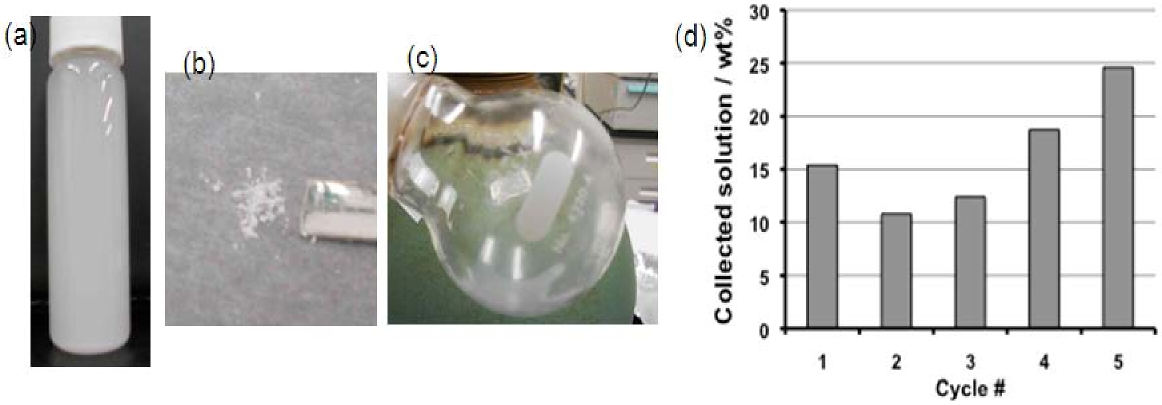


Figure 22. Photographs of (a) collected solution, (b) filtrated residual particulates, and (c) residual oil after evaporation. (d) Ratio of collected solution amount for the weight of gel on each cycle.

Cost Assessment

Cost Reporting

The objective of this project was to evaluate and demonstrate oil sorbent gels as replacements for MIL-PRF-680 solvents for use on weapons systems and platforms. Implementing these processes would require minimal capital investment, as most facilities already have cleaning tanks on hand. The only substantial investment would be in production of the NG-18-x gel and solvent use. Existing equipment would not need to be replaced.

The cost of production was analyzed based on 10’x4’x4’ (3400 L) cleaning tank filled to various degrees, assuming no solvent loss between cleanings, and a 250 day work year accounting for holidays and weekends.

For comparison of costs of solvent cleaning versus the gel cleaning system, the following assumptions were made.

1) The solvent could be used till it is saturated with oil, grease and dirt. The solvent would be used for three cycles before the solvent is replaced.
2) The gel system is used for eight solvent cycles.

The cost analysis is for comparing the two cleaning approaches. With an optimized system the absolute costs for each may be different. Tables 2-5 show the amounts of chemicals used and overall costs of each cleaning system. From this analysis it was determined that the cost of the gel system is less than that of the current cleaning system.

Cost Analysis

Table 2. Chemical cost and amounts needed to make one kilogram of dry NG-18 gel

				NG-18-1%		**NG-18-0.5%**	
Chemical	**Price per unit ($)**	**Amount per unit**	**Number of Units required**	**Amount Needed**	**Cost ($)**	**Amount Needed**	**Cost ($)**
Benzene	67.00	1 L	1	236.1 mL	67.00	225.28 mL	67.00
SA	89.20	1 kg	2	1.1806 kg	178.40	1.2642 kg	178.40
EGDMA	33.80	100 g	1	7.2 g	33.80	3.86 g	33.80
AIBN	30.60	25 g	1	11.9 g	30.60	12.8 g	30.60
Hexane	349.20	208.198 L (55 gal)	1	30 L	349.20	30 L	349.20
				Total Cost	**659.00**	**Total Cost**	**659.00**

Table 3. Chemical amounts of NG-18 and trichloroethylene needed to fill a 3400 L cleaning tank to various levels

NG-18-1%

	Amount needed per fill height					
Chemical	100%	75%	67%	50%	33%	25%
Benzene	5.352 L	4.014 L	3.568 L	2.676 L	1.784 L	1.338 L
SA	27.154 kg	20.365 kg	18.103 kg	13.577 kg	9.051 kg	6.788 kg
EGDMA	165.600 g	124.200 g	110.400 g	82.800 g	55.200 g	41.400 g
AIBN	273.700 g	205.275 g	182.467 g	136.850 g	91.233 g	68.425 g
Hexane	690.000 L	517.500 L	460.000 L	345.000 L	230.000 L	172.500 L
THF	2300.000 L	1725.000 L	1533.333 L	1150.000 L	766.667 L	575.000 L
TCE	3400.000 L	2550.000 L	2278.000 L	1700.000 L	1122.000 L	850.000 L

NG-18-0.5%

	Amount needed per fill height					
Chemical	100%	75%	67%	50%	33%	25%
Benzene	5.730 L	4.298 L	3.820 L	2.865 L	1.910 L	1.433 L
SA	29.035 kg	21.776 kg	19.357 kg	14.518 kg	9.678 kg	7.259 kg
EGDMA	88.780 g	66.585 g	59.187 g	44.390 g	29.593 g	22.195 g
AIBN	294.400 g	220.800 g	196.267 g	147.200 g	98.133 g	73.600 g
Hexane	690.000 L	517.500 L	460.000 L	345.000 L	230.000 L	172.500 L
THF	2300.000 L	1725.000 L	1533.333 L	1150.000 L	766.667 L	575.000 L
TCE	3400.000 L	2550.000 L	2278.000 L	1700.000 L	1122.000 L	850.000 L

Cost Comparison

Table 4. Cost per batch of NG-18 gels compared to trichloroethylene when cleaning tank is filled to various levels

Cleaning material	NG-18-0.5%	NG-18-1%	TCE	Tank Level
Cost of Purchase Per Batch ($)	29,987.16	29,810.55	26,860.45	100.00%
	22,490.37	22,357.91	20,145.34	75.00%
	19,991.44	19,873.70	17,996.50	66.67%
	14,993.58	14,905.27	13,430.22	50.00%
	9,995.72	9,936.85	8,863.95	33.33%
	7,496.79	7,452.64	6,715.11	25.00%

Table 5. Cost over time of NG-18 gels compared to trichloroethylene when cleaning tank is filled to various levels assuming no depreciation and one use per day.

Cleaning method	NG-18-0.5%	NG-18-1%	TCE	Tank level
Reuse times before cleaning	3	3	3	
1 year cost ($)	2,213,784.63	2,211,944.90	2,238,370.76	100%
2 year cost ($)	4,427,569.27	4,423,889.79	4,476,741.52	
10 year cost ($)	22,137,846.33	22,119,448.97	22,383,707.62	
1 year cost ($)	1,660,338.47	1,658,958.67	1,678,778.07	75%
2 year cost ($)	3,320,676.95	3,317,917.34	3,357,556.14	
10 year cost ($)	16,603,384.75	16,589,586.72	16,787,780.71	
1 year cost ($)	1,475,856.42	1,474,629.93	1,499,708.41	67%
2 year cost ($)	2,951,712.84	2,949,259.86	2,999,416.82	
10 year cost ($)	14,758,564.22	14,746,299.31	14,997,084.10	
1 year cost ($)	1,106,892.32	1,105,972.45	1,119,185.38	50%
2 year cost ($)	2,213,784.63	2,211,944.90	2,238,370.76	
10 year cost ($)	11,068,923.16	11,059,724.48	11,191,853.81	
1 year cost ($)	737,928.21	737,314.97	738,662.35	33%
2 year cost ($)	1,475,856.42	1,474,629.93	1,477,324.70	
10 year cost ($)	7,379,282.11	7,373,149.66	7,386,623.51	
1 year cost ($)	553,446.16	552,986.22	559,592.69	25%
2 year cost ($)	1,106,892.32	1,105,972.45	1,119,185.38	
10 year cost ($)	5,534,461.58	5,529,862.24	5,595,926.90	

Toxicity

Both THF and TCE are considered highly toxic in liquid form. THF is not used in liquid form in this experiment. Instead it is used in gel form, which will reduce exposure, and limit the health risks. Cyclohexane or other solvents may be used to address any toxic or environmental concerns.

Conclusions

In this study, we first demonstrated the synthesis and characterization of poly(SA-*co*-EGDMA) (NG-18) gels. The swelling characteristics of the gels were studied as a function of the solvent polarity and temperature, and the kinetics of swelling were also examined. Volume transition via crystallization of the long alkyl chain was investigated by transmittance at 700 nm light with controlling temperature. Moreover, the reversibility and reproducibility of the transition were studied by both swelling and transmittance with cyclic temperature change. These properties suggested the utility of NG-18 gels as recyclable VOCs absorbent materials.

The gel system uses THF for swelling, however, cyclohexane or other benign solvents with similar swelling properties may also be used as a swelling agent. In particular cyclohexane is a good possibility with swelling ratio (Q) of 20. Other solvents such as toluene or other non-polar solvents may also be used. We have not tested several other potential solvents during this phase of the research.

Based on the preliminary cost assessment the gel cleaning process appears to be costing a similar amount. The gel cleaning process has the advantage of avoiding emissions of hazardous air pollutants (HAPs) and volatile organic compounds (VOCs).

Further research to improve the neutral gel systems with increased swelling properties and recyclability are recommended. Optimization of the gel synthesis, cleaning process and kinetics at room temperature is recommended.

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APPENDICES

Appendix A: Miltary Proformance Specification for Degreasing Solvent, MIL-PRF-680B

NOT MEASUREMENT SENSITIVE

MIL-PRF-680B
26 October 2006
SUPERSEDING
MIL-PRF-680A
25 December 2003

PERFORMANCE SPECIFICATION

DEGREASING SOLVENT

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers degreasing solvent that consists of four types of petroleum distillates. The different types are referred to as “Stoddard solvent”, “141 degrees Fahrenheit (°F) (60.6 degrees Celsius (°C)) solvent”, “200°F (93.3°C) solvent”, and “141°F d-limonene blended solvent”. They are used for degreasing of machine parts in equipment maintenance.

1.2 Classification. Degreasing solvents are of the following types (see 6.2).

Type I	- Low flash point (Stoddard solvent) (Military Symbol SD-1)
Type II	- High flash point (Military Symbol SD-2)
Type III	- Very high flash point (200°F) (Military Symbol SD-3)
Type IV	- High flash point with citrus odor (Military Symbol SD-4)

1.2.1 NATO classification:

Type I	- S-752
Type II	- S-753
Type III	- S-760
Type IV	- None

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, and 5 of this specification. This section does not include documents cited in other sections of this

Beneficial comments, suggestions, or questions on this document should be addressed to U.S. Army Tank-automotive and Armaments Command, 6501 E. 11 Mile Road, Warren, MI 48397-5000 or emailed to standardization@tacom.army.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at http://assist.daps.dla.mil.

AMSC N/A FSC 6850

specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirement documents cited in sections 3, 4, and 5 of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

SPECIFICATION

DEPARTMENT OF DEFENSE

MIL-PRF-10924	- Grease, Automotive and Artillery.

(Unless otherwise indicated, copies of the above specifications, standards, and handbooks are available from the Document Automation and Production Service, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094 or at http://assist.daps.dla.mil/).

2.2.2 Other Government documents, drawings and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

ENVIRONMENTAL PROTECTION AGENCY (EPA)

EPA Method 420.1	- Methods for Chemical Analysis of Water and Wastes Total Recoverable in Water and Solids.
EPA Method 3585	- Method for Waste Dilution for Volatile Organics.
EPA Method 8260B	- Method for Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS).

(Application for copies should be addressed to the US EPA Headquarters, 401 M Street SW, mail code 3204, Washington, DC 20460 or at http://www.epa.gov).

2.3 Non-Government publications. The following document forms a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

AMERICAN SOCIETY FOR QUALITY (ASQ)

Z1.4	- Sampling Procedures and Tables for Inspection by Attributes (DoD adopted).

(Application for copies should be addressed to the American Society for Quality, P.O. Box 3005, Milwaukee, WI 53201-3005 or at http://www.asq.org).

ASTM INTERNATIONAL

D 56 - Standard Test Method for Flash Point by Tag Closed Cup Tester
D 86 - Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure
D 130 - Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test
D 156 - Standard Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
D 235 - Standard Test Method for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry-Cleaning Solvent)
D 847 - Standard Test Method for Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons
D 1133 - Standard Test Method for Kauri-Butanol Value of Hydrocarbon Solvents
D 1296 - Standard Test Method for Odor of Volatile Solvents and Diluents
D 1298 - Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D 1353 - Standard Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products
D 2879 - Standard Test Method for Vapor Pressure-Temperature Relationship and Initial Decomposition Temperature of Liquids by Isoteniscope
D 3257 - Standard Test Method for Aromatics in Mineral Spirits by Gas Chromatography
D 4057 - Standard Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 - Standard Practice for Automatic Sampling of Petroleum and Petroleum Products
F 483 - Standard Test Method for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals
F 945 - Standard Test Method for Stress-Corrosion of Titanium Alloys by Aircraft Engine Cleaning Materials
F 1110 - Standard Test Method for Sandwich Corrosion Test

(Application for copies should be addressed to ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or at http://www.astm.org).

SOCIETY OF AUTOMOTIVE ENGINEERS (SAE)

AMS-QQ-A-250 - Aluminum and Aluminum Alloy, Plate and Sheet (DoD adopted).

AMS 2470 - Anodic Treatment of Aluminum Alloys Chrome Acid Process (DoD adopted).
AMS M 3171 - Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion on (DoD adopted).
AMS 4377 - Magnesium Alloy, Sheet and Plate 3.0A1-1.0Zn-0.20Mn (AZ31B-H24) Cold Rolled, Partially Annealed
AMS 4911 - Titanium Alloy Sheet, Strip, and Plate, 6AI-4V Annealed
AMS 5046 - Carbon Steel Sheet, Strip, and Plate, (SAE 1020 and 1025) Annealed

(Application for copies should be addressed to the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001 or at http://www.sae.org).

2.4 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification takes precedence. Nothing in this specification, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Qualification. Solvents furnished under this specification shall be products that are authorized by the qualifying activity for listing on applicable qualified products list before contract award (see 4.2 and 6.3). Any change in the formulation of a qualified product will necessitate its requalification.

3.2 Materials. The hydrocarbon solvent shall be a virgin grade or recycled solvent derived from petroleum distillates, fraction from reclaiming and re-refining processes, or a mixture of these fractions. The d-limonene and hydrocarbon blended solvent is permitted as a Type IV solvent. The resultant solvent must be produced in such a manner as is necessary to meet the specified requirements.

3.3 Appearance. The solvent shall be clear and free from suspended matter and undissolved water when observed at a temperature between 60.1 and 78.1°F (15.6 and 25.6 °C).

3.4 Toxicity. The solvent shall have no adverse effects on human health when used as intended (see 6.1 and 6.6).

3.5 Carcinogenicity. The solvent shall contain no chemicals listed as carcinogens (see 6.7). Any carcinogenic components in the solvent in a concentration of 0.1 percent (%) or greater by weight (wt) or volume (vol) will be regarded as the presence of a carcinogen in the solvent.

3.6 Hazardous Air Pollutants. The solvent shall contain no chemicals listed as Hazardous Air Pollutants (HAPs) (see 6.7). Any HAP components in the solvent in a concentration of 1.0 percent (%) or greater by weight (wt) or volume (vol) will be regarded as the presence of a HAP in the solvent. The product containing less than 1 % of HAP shall be considered as a HAP free solvent. For carcinogenic HAPs see 3.5.

3.7 Physical and chemical property requirements. The solvents shall conform to the physical and chemical requirements in Table I when tested as specified in Table II.

TABLE I. Degreasing solvent properties.

Characteristics	Type I	Type II	Type III	Type IV
Flash point (pt), °C (°F)	38-60	61–92	93-116	61–92
	(100-140)	(141-198)	(200-241)	(141-198)
Distillation, °C:				
Initial boiling pt., minimum (min)	149	177	220	171
Dry point, °C, maximum (max)	208	212	300	240
Kauri-Butanol value	27 to 45	27 to 45	27 to 45	27 to 45
Aromatic content, vol %, max	1	1	1	1
Total phenol content, parts per million (ppm), max	0.5	0.5	0.5	0.5
Dichlorobenzene, milligrams per liter (mg/L), max	0.5	0.5	0.5	0.5
Benzene, mg/L, max	0.5	0.5	0.5	0.5
Tetrachloroethylene, mg/L, max	0.7	0.7	0.7	0.7
Trichloroethylene, mg/L, max	0.5	0.5	0.5	0.5
Apparent specific gravity, 60/60°F	0.754 to 0.820	0.754 to 0.820	0.754 to 0.840	0.754 to 0.820
Total chlorine content (ppm), Max	100	100	100	100
Non-volatile residue, (mg/100 mL), max	8	8	8	8
Color, min	25	25	25	25
Odor 1/	Low & non-residual	Low & non-residual	Low & non-residual	Citrus & non-residual
Corrosion, copper, max 2/	1b	1b	1b	1b
Sandwich corrosion, max	1	1	1	1
Total immersion corrosion 3/	Pass	Pass	Pass	Pass
Titanium stress corrosion	No cracking	No cracking	No cracking	No cracking
Acidity	Neutral	Neutral	Neutral	Neutral

Doctor test	Negative	Negative	Negative	Negative
Vapor pressure, millimeters of Mercury (mm Hg) @ 20ºC, max	7.0	2.0	0.4	2.0
Soil cleaning test, %, min	85	85	85	88

1/ Samples of MIL-PRF-680A, Type III, having satisfactory low odor characteristics shall be used as reference standards.

2/ Test for three hours at 100 °C (212ºF).

3/ See 4.4.2

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

a. Qualification inspection (see 4.2)
b. Conformance inspection (see 4.3).

4.1.1 Inspection conditions. Unless otherwise specified, all inspections shall be performed in accordance the test conditions specified in 4.2.

4.2 Qualification inspection. The qualification inspection shall consist of all tests specified herein. Failure of any test shall be cause for rejection.

4.3 Conformance inspection. Conformance inspection shall consist of a sample for tests (see 4.3.2), samples for examination of filled containers (see 4.3.3), and the tests specified in Table II except for sandwich corrosion, total immersion corrosion, and titanium stress corrosion.

4.3.1 Lot. A lot shall consist of solvents from one batch or tank offered for delivery at one time. If material cannot be identified by batch or lot, a lot shall consist of not more than 10 000 gallons offered for delivery at one time (see 6.2).

4.3.2 Sampling for tests. Sampling of a lot for test purposes shall be in accordance with ASTM D 4057 or D 4177.

4.3.3 Sample for examination of filled containers. A random sample of filled unit containers and a sample of shipping containers fully prepared for delivery shall be selected from each lot of solvent in accordance with ASQ Z1.4.

4.4 Methods of inspection.

4.4.1 Tests. Tests shall be performed in accordance with the applicable methods specified in Table II and 4.4.2 through 4.4.3 to determine conformance with the requirements specified in 3.6.

TABLE II. Test methods for inspections.

Characteristic	ASTM methods	Other methods
Flash point	D 56	
Distillation	D 86	
Kauri-Butanol value	D 1133	
Aromatic content	D 3257	
Apparent specific gravity	D 1298	
Color	D 156	
Odor	D 1296	
Non-volatile residue	D 1353	
Copper corrosion	D 130	
Sandwich corrosion	F 1110	
Total immersion corrosion	F 483	
Titanium stress corrosion	F 945	
Acidity	D 847	
Doctor test	D 235	
Vapor pressure	D 2879	
Total phenol content		EPA 420.1
Total dichlorobenzene content		EPA 3585 and 8260B
Total benzene content		EPA 3585, 8260B
Total trichloroethylene		EPA 3585, 8260B
Total tetrachloroethylene		EPA 3585, 8260B
Soil cleaning test		APPENDIX A

4.4.2 Total immersion corrosion. The solvents shall not show any indication of staining, etching, pitting, or localized, or cause weight change to an average of three (3) test panels greater than that shown in Table III.

TABLE III. Alloy and maximum average weight loss.

Alloy	Average of 3 panels wt. loss, max milligrams per square centimeter ($mg/cm^2/168$ hrs)
Magnesium (AZ 31B-H24) AMS 4377 surface treated in accordance with AMS-M-3171, Type III	0.50
Aluminum, AMS-QQ-A-250, T3 surface, treatment in accordance with AMS 2470	0.15
Titanium, AMS 4911, 6AL-4V	0.10
Steel, AMS 5046, Grade 1020	0.25

4.4.3 Soil cleaning test. The test shall be conducted according to the solvent soil test method described in the appendix. The soil used in this evaluation is MIL-PRF-10924 grease. The acceptable limit for each type of solvent is defined in Table I.

4.4.4 Total chlorine content. The total chlorine content of the solvent shall be determined by a gas chromatographic method, microcoulometric or by the use of a portable test kit for the quantitative analysis of chlorine (see 6.6). Nonconformance to Table I shall constitute failure of this test.

5. PACKAGING

5.1 Packaging. For acquisition purpose, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity with the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contracting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. These solvents are hydrocarbon-based solvents and are used as degreasers and cleaners for painted or unpainted metal parts. However, the compatibility between existing paints and solvents should be verified prior to use. It is recommended that these solvents should be used with an appropriate recirculated parts washer. No other commercial specification is available to cover these military applications.

6.1.1 Type I. Type I (Stoddard solvent) is intended for use where a low odor solvent with fast drying characteristics is desired. This solvent is currently defined as a flammable material due to its low flash point. It is recommended that the alternative solvents for Type I are Types II, III and IV. When Type I solvent is used indoors, ventilation should be sufficient to prevent the accumulation of vapors above allowable limits.

6.1.2 Type II. Type II (low odor with high flash point solvent) is intended for use where a solvent with a higher flash point is desired. It is recommended over Type I for safety and regulatory reasons. When Type II solvent is used indoors, ventilation should be sufficient to prevent the accumulation of vapors above allowable exposure limits.

6.1.3 Type III. Type III (low odor with very high flash point solvent) is intended to be used where confined atmospheric conditions require a cleaner that conforms to the Federal Government's directives for reduced hazardous materials.

6.1.4 Type IV. Type IV (citrus odor with high flash point) is intended for use where a solvent with a high flash point and strong solvency is desired. This solvent is formulated with petroleum based hydrocarbon solvents and d-limonene additive. When Type IV solvent is used indoors, ventilation should be sufficient to prevent the accumulation of vapors above allowable exposure limits.

6.2 Acquisition requirements. Acquisition documents must specify the following:

a. Title, number and date of this specification.
b. Type and quantity (see 1.2. and 4.3.1).
c. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.2.1 and 2.3).
d. Packaging requirements (see 5.1)

6.3 Qualification. With respect to products requiring qualification, awards will be made only for products which are at the time of award of contract, qualified for inclusion in Qualified Product List QPL No. 680 whether or not such products have actually been so listed by that date. The attention of the contractors is called to these requirements, and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or purchase orders for the products covered by this specification.

6.3.1 Specification preparing activity. Information and instructions regarding qualification inspection under this specification may be obtained from the Department of the Army, TACOM-TARDEC, ATTN: AMSRD-TAR-E/268, 6501 E. 11 Mile Road, Warren, MI 48397-5000.

6.4 International standardization. Certain provisions of this specification are the subject of international standardization agreement (NATO STANAG 1135, ANNEX C). When amendment, revision, or cancellation of this specification is proposed which would affect or violate the international agreement concerned, the preparing activity will take appropriate reconciliation action through international channels, including departmental standardization offices, if required.

6.5 Disposal actions. Disposal of this product should be in accordance with local, state and Federal regulations. Care should be taken to avoid mixing used MIL-PRF-680 with other waste materials, especially those containing halogenated solvents. However, these solvents can be recycled instead of disposal.

6.6 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets (MSDSs) prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313; and 29 CFR 1910.1200 requires that the MSDS for each hazardous chemical used in an operation must be readily available to personnel using the material. Contracting officers will identify the activities requiring copies of the MSDS (see 3.4 and 4.4.4).

6.7 Definitions.

6.7.1 Carcinogens. The Occupational Safety and Health Administration (OSHA) definition of carcinogens are those chemicals / processes appearing in lists 1, 2A, and 2B of the International Agency for Research on Cancer (IARC) [1/]; substances known to be carcinogenic and occupational exposures associated with a technological process known to be carcinogenic by the National Toxicology Program (NTP) Report on Carcinogens (latest annual report) [2/]; and OSHA regulated carcinogens.

6.7.2. Hazardous Air Pollutant (HAP). HAP is defined as any substance listed under Section 112 of the Clean Air Act or its modifications. The text of the Clean Air Act, listed pollutants and modifications are kept by the Environmental Protection Agency (EPA) and are accessible through the website: http://www.epa.gov

[1/] Copies of these volumes may be found in medical libraries or through the World Health Organization, 1211 Geneva 27, Switzerland.

[2/] Copies may be obtained from the Public Health Service, National Toxicology Program, Public Information Office, P.O. Box 12233, MD B2-04, Research Triangle Park, NC 27709 or at http://ntp-server.niehs.nih.gov

6.8 Subject term (key word) listing.

d-limonene
d-limonene/hydrocarbon blended solvent (type IV)
Mineral Spirits
Naphtha
Petroleum Spirits (USA)
Stoddard Solvent (type I)
141 F Solvent (type II)
200 F Solvent (type II)

6.9 Changes from previous issues. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

SOLVENT CLEANING POWER BY U.S. ARMY SOIL TEST METHOD

A.1. SCOPE.

A.1.1 Scope. This test method is used for determining the cleaning power of solvents that have been subjected to soiled mechanical parts. This Appendix is a mandatory part of the specification. The information contained herein is intended for compliance only.

A.2 APPLICABLE DOCUMENTS

(This section is applicable to this appendix)

SPECIFICATION

STANDARD

FEDERAL

FED-STD-791C, Method 5308.7 - Corrosiveness and Oxidation Stability of Light Oils (Metal Squares).

A.3 Summary of method. A small amount of grease applied to a steel coupon is immersed in the test solvent and then placed in an ultrasonic cleaner maintained at 122ºF (50°C). The grease is dissolved by the solvent and removed from the steel coupon by the propagation of sonic sound. The solvent power is determined based on the time to completely remove grease from the coupon.

A.4 Significance and use. This test method measures the solvency of all types of cleaners and differentiates their cleaning powers.

A.5 Apparatus.

A.5.1 Ultrasonic Cleaner

A.5.2 Beaker, 12.2 cubic inches (200ml)

A.5.3 Test specimen assembly of steel metal square 0.032 inch (in.) thick by 0.98 in. square (0.081 centimeter (cm) thick by 2.5 cm square)) with holding wire (Figure 1). This specimen has been used in Federal Standard Test Method 791C Method 5308.7. (NOTE: Paper clips have been successfully used for holding metal square.)

(a) Metal square

(b) Test assembly

FIGURE A-1. Metal square dimensions and test assembly.

Custodians:	Preparing Activity:
Army - AT	Army - AT
Air Force - 68	
Navy – AS	(Project 6850-2006-011)

Review Activities:
Army - AV, EA, MD1, MI, MR, SM
Air Force - 03, 11, 50
Navy - MC, SH
DLA - GS, GS3, DP
DTRA - DS
GSA/GSS - 7FLE
MISC – MP

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at http://assist.daps.dla.mil.

SIGMA-ALDRICH sigma-aldrich.com

Material Safety Data Sheet

Version 4.1
Revision Date 02/13/2011
Print Date 05/24/2011

1. PRODUCT AND COMPANY IDENTIFICATION

Product name	: Tetrahydrofuran
Product Number	: 401757
Brand	: Sigma-Aldrich
Product Use	: For laboratory research purposes.
Supplier	: Sigma-Aldrich 3050 Spruce Street SAINT LOUIS MO 63103 USA
Manufacturer	: Sigma-Aldrich Corporation 3050 Spruce St. St. Louis, Missouri 63103 USA
Telephone	: +1 800-325-5832
Fax	: +1 800-325-5052
Emergency Phone # (For both supplier and manufacturer)	: (314) 776-6555
Preparation Information	: Sigma-Aldrich Corporation Product Safety - Americas Region 1-800-521-8956

2. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards
Flammable liquid, Target Organ Effect, Harmful by ingestion., Irritant, Carcinogen

Target Organs

Central nervous system, Liver, Kidney

Other hazards which do not result in classification
May form explosive peroxides.

GHS Classification
Flammable liquids (Category 2)
Acute toxicity, Oral (Category 4)
Acute toxicity, Dermal (Category 5)
Skin irritation (Category 3)
Serious eye damage (Category 1)
Specific target organ toxicity - single exposure (Category 3)

GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)

H225	Highly flammable liquid and vapour.
H302	Harmful if swallowed.
H313	May be harmful in contact with skin.
H316	Causes mild skin irritation.
H318	Causes serious eye damage.
H335 + H336	May cause respiratory irritation, and drowsiness or dizziness.

Precautionary statement(s)

P210	Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
P261	Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray.
P280	Wear protective gloves/ eye protection/ face protection.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

HMIS Classification

Health hazard:	2
Chronic Health Hazard:	*
Flammability:	3
Physical hazards:	3

NFPA Rating

Health hazard:	2
Fire:	3
Reactivity Hazard:	0

Potential Health Effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation. Vapours may cause drowsiness and dizziness.
Skin	Harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.
Ingestion	Harmful if swallowed.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms : THF

Formula : C4H8O
Molecular Weight : 72.11 g/mol

CAS-No.	EC-No.	Index-No.	Concentration
Tetrahydrofuran			
109-99-9	203-726-8	603-025-00-0	-

4. FIRST AID MEASURES

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

5. FIRE-FIGHTING MEASURES

Conditions of flammability
Flammable in the presence of a source of ignition when the temperature is above the flash point. Keep away from heat/sparks/open flame/hot surface. No smoking.

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for fire-fighters
Wear self contained breathing apparatus for fire fighting if necessary.

Hazardous combustion products
Hazardous decomposition products formed under fire conditions. - Carbon oxides

Further information
Use water spray to cool unopened containers.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Use personal protective equipment. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.

Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

Methods and materials for containment and cleaning up
Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13).

7. HANDLING AND STORAGE

Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment. Keep away from sources of ignition - No smoking. Take measures to prevent the build up of electrostatic charge.

Conditions for safe storage
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Components with workplace control parameters

Components	CAS-No.	Value	Control parameters	Basis
Tetrahydrofuran	109-99-9	TWA	50 ppm	USA. ACGIH Threshold Limit Values (TLV)
Remarks	Central Nervous System impairment Upper Respiratory Tract irritation Kidney damage Confirmed animal carcinogen with unknown relevance to humans: The agent is carcinogenic in experimental animals at a relatively high dose, by route(s) of administration, at site(s), of histologic type(s), or by mechanism(s) that may not be relevant to worker exposure. Available epidemiologic studies do not confirm an increased risk of cancer in exposed humans. Available evidence does not suggest that the agent is likely to cause cancer in humans except under uncommon or unlikely routes or levels of exposure. Danger of cutaneous absorption			
		STEL	100 ppm	USA. ACGIH Threshold Limit Values (TLV)
	Central Nervous System impairment Upper Respiratory Tract irritation Kidney damage Confirmed animal carcinogen with unknown relevance to humans: The agent is carcinogenic in experimental animals at a relatively high dose, by route(s) of administration, at site(s), of histologic type(s), or by mechanism(s) that may not be relevant to worker exposure. Available epidemiologic studies do not confirm an increased risk of cancer in exposed humans. Available evidence does not suggest that the agent is likely to cause cancer in humans except under uncommon or unlikely routes or levels of exposure. Danger of cutaneous absorption			
		TWA	200 ppm 590 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		STEL	250 ppm 735 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		TWA	200 ppm 590 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants

	The value in mg/m3 is approximate.			
		TWA	200 ppm 590 mg/m3	USA. NIOSH Recommended Exposure Limits
		ST	250 ppm 735 mg/m3	USA. NIOSH Recommended Exposure Limits

Personal protective equipment

Respiratory protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type AXBEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Eye protection

Tightly fitting safety goggles. Faceshield (8-inch minimum). Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin and body protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form	liquid, clear
Colour	colourless

Safety data

pH	no data available
Melting point/freezing point	-108.0 °C (-162.4 °F)
Boiling point	65.0 - 67.0 °C (149.0 - 152.6 °F)
Flash point	-17.0 °C (1.4 °F) - closed cup
Ignition temperature	321 °C (610 °F)
Autoignition temperature	321.0 °C (609.8 °F)
Lower explosion limit	1.8 %(V)
Upper explosion limit	11.8 %(V)
Vapour pressure	152.0 hPa (114.0 mmHg) at 15.0 °C (59.0 °F) 190.7 hPa (143.0 mmHg) at 20.0 °C (68.0 °F) 213.3 hPa (160.0 mmHg) at 25.0 °C (77.0 °F) 373.3 hPa (280.0 mmHg) at 38.0 °C (100.4 °F)
Density	0.89 g/cm3
Water solubility	soluble
Partition coefficient: n-octanol/water	log Pow: < 1

	The value in mg/m3 is approximate.			
		TWA	200 ppm 590 mg/m3	USA. NIOSH Recommended Exposure Limits
		ST	250 ppm 735 mg/m3	USA. NIOSH Recommended Exposure Limits

Personal protective equipment

Respiratory protection

Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type AXBEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Eye protection

Tightly fitting safety goggles. Faceshield (8-inch minimum). Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin and body protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form	liquid, clear
Colour	colourless

Safety data

pH	no data available
Melting point/freezing point	-108.0 °C (-162.4 °F)
Boiling point	65.0 - 67.0 °C (149.0 - 152.6 °F)
Flash point	-17.0 °C (1.4 °F) - closed cup
Ignition temperature	321 °C (610 °F)
Autoignition temperature	321.0 °C (609.8 °F)
Lower explosion limit	1.8 %(V)
Upper explosion limit	11.8 %(V)
Vapour pressure	152.0 hPa (114.0 mmHg) at 15.0 °C (59.0 °F) 190.7 hPa (143.0 mmHg) at 20.0 °C (68.0 °F) 213.3 hPa (160.0 mmHg) at 25.0 °C (77.0 °F) 373.3 hPa (280.0 mmHg) at 38.0 °C (100.4 °F)
Density	0.89 g/cm3
Water solubility	soluble
Partition coefficient: n-octanol/water	log Pow: < 1

Viscosity, kinematic	0.512 mm2/s at 25 °C (77 °F) 0.403 mm2/s at 50 °C (122 °F)
Relative vapour density	no data available
Odour	no data available
Odour Threshold	no data available
Evaporation rate	no data available

10. STABILITY AND REACTIVITY

Chemical stability

Stable under recommended storage conditions.

Possibility of hazardous reactions

Vapours may form explosive mixture with air.

Conditions to avoid

Heat, flames and sparks. Extremes of temperature and direct sunlight.

Materials to avoid

Oxidizing agents, Oxygen

Hazardous decomposition products

Hazardous decomposition products formed under fire conditions. - Carbon oxides
Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

Acute toxicity

Oral LD50

LD50 Oral - rat - 1,650 mg/kg

LD50 Oral - guinea pig - 2,300 mg/kg

Inhalation LC50

LC50 Inhalation - rat - 3 h - 21000 ppm
Remarks: Drowsiness Lungs, Thorax, or Respiration:Respiratory stimulation. Ingestion may cause gastrointestinal irritation, nausea, vomiting and diarrhoea.

Dermal LD50

LD50 Dermal - rat - > 2,000 mg/kg

Other information on acute toxicity

no data available

Skin corrosion/irritation

Skin - rabbit - Mild skin irritation - Draize Test

Serious eye damage/eye irritation

Eyes - rabbit - Risk of serious damage to eyes. - Draize Test

Respiratory or skin sensitization

mouse - Did not cause sensitization on laboratory animals.

Germ cell mutagenicity

In vivo tests did not show mutagenic effects

Carcinogenicity

IARC:	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Reproductive toxicity

No toxicity to reproduction

Teratogenicity

no data available

Specific target organ toxicity - single exposure (Globally Harmonized System)
Inhalation - May cause respiratory irritation.
May cause drowsiness or dizziness. - Nervous system

Specific target organ toxicity - repeated exposure (Globally Harmonized System)
The substance or mixture is not classified as specific target organ toxicant, repeated exposure.

Aspiration hazard
No aspiration toxicity classification

Potential health effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation. Vapours may cause drowsiness and dizziness.
Ingestion	Harmful if swallowed.
Skin	Harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.

Signs and Symptoms of Exposure
Central nervous system depression, Cough, chest pain, Difficulty in breathing, Exposure to high airborne concentrations can cause anesthetic effects., To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Synergistic effects
no data available

Additional Information
RTECS: LU5950000

12. ECOLOGICAL INFORMATION

Toxicity

Toxicity to fish	LC50 - Pimephales promelas (fathead minnow) - 2,160 mg/l - 96 h
Toxicity to algae	Growth inhibition NOEC - Algae - 3,700 mg/l - 192 h

Persistence and degradability
Expected to be biodegradable

Bioaccumulative potential
no data available

Mobility in soil
no data available

PBT and vPvB assessment
no data available

Other adverse effects

no data available

13. DISPOSAL CONSIDERATIONS

Product
Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)
UN number: 2056 Class: 3 Packing group: II
Proper shipping name: Tetrahydrofuran
Reportable Quantity (RQ): 1000 lbs
Marine pollutant: No
Poison Inhalation Hazard: No

IMDG
UN number: 2056 Class: 3 Packing group: II EMS-No: F-E, S-D
Proper shipping name: TETRAHYDROFURAN
Marine pollutant: No

IATA
UN number: 2056 Class: 3 Packing group: II
Proper shipping name: Tetrahydrofuran

15. REGULATORY INFORMATION

OSHA Hazards
Flammable liquid, Target Organ Effect, Harmful by ingestion., Irritant, Carcinogen

SARA 302 Components
SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Tetrahydrofuran	109-99-9	2007-03-01

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Tetrahydrofuran	109-99-9	2007-03-01

New Jersey Right To Know Components

	CAS-No.	Revision Date
Tetrahydrofuran	109-99-9	2007-03-01

California Prop. 65 Components
This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

16. OTHER INFORMATION

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Co., shall not be held liable for any damage resulting from handling or from contact with the above product. See reverse side of invoice or packing slip for additional terms and conditions of sale.

[redacted]

Material Safety Data Sheet

Version 3.0
Revision Date 08/20/2009
Print Date 06/15/2011

1. PRODUCT AND COMPANY IDENTIFICATION

Product name : Trichloroethylene

Product Number : 372145
Brand : Sigma-Aldrich

Company : Sigma-Aldrich
3050 Spruce Street
SAINT LOUIS MO 63103
USA
Telephone : +1 800-325-5832
Fax : +1 800-325-5052
Emergency Phone # : (314) 776-6555

2. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms : TCE
Trichloroethene

Formula : C_2HCl_3
Molecular Weight : 131.39 g/mol

CAS-No.	EC-No.	Index-No.	Concentration
Trichloroethylene			
79-01-6	201-167-4	602-027-00-9	-

3. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards
Irritant, Carcinogen

Target Organs

Liver, Central nervous system, Heart, Lungs

HMIS Classification
Health Hazard: 2
Chronic Health Hazard: *
Flammability: 0
Physical hazards: 1

NFPA Rating
Health Hazard: 2
Fire: 0
Reactivity Hazard: 0

Potential Health Effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation. Vapours may cause drowsiness and dizziness.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.
Ingestion	May be harmful if swallowed.

4. FIRST AID MEASURES

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing give artificial respiration Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

5. FIRE-FIGHTING MEASURES

Flammable properties

Flash point	no data available
Ignition temperature	410 °C (770 °F) -

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for fire-fighters
Wear self contained breathing apparatus for fire fighting if necessary.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Use personal protective equipment. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas.

Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

Methods for cleaning up
Soak up with inert absorbent material and dispose of as hazardous waste. Keep in suitable, closed containers for disposal.

7. HANDLING AND STORAGE

Handling
Avoid inhalation of vapour or mist.
Normal measures for preventive fire protection.

Storage
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Light sensitive. Handle and store under inert gas.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Components with workplace control parameters

Components	CAS-No.	Value	Control parameters	Update	Basis
Trichloroethylene	79-01-6	TWA	50 ppm 270 mg/m3	1989-01-19	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
Remarks	Skin notation				
		STEL	200 ppm 1,080 mg/m3	1989-01-19	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
	Skin notation				
		TWA	100 ppm	2007-01-01	USA. Occupational Exposure Limits (OSHA) - Table Z2
	Z37.19-1967				
		CEIL	200 ppm	2007-01-01	USA. Occupational Exposure Limits (OSHA) - Table Z2
	Z37.19-1967				
		Peak	300 ppm	2007-01-01	USA. Occupational Exposure Limits (OSHA) - Table Z2
	Z37.19-1967				
		TWA	10 ppm	2008-01-01	USA. ACGIH Threshold Limit Values (TLV)
	Central Nervous System impairment cognitive decrement Renal toxicity Suspected human carcinogen: Human data are accepted as adequate in quality but are conflicting or insufficient to classify the agent as a confirmed human carcinogen; OR, the agent is carcinogenic in experimental animals at dose(s), by route(s) of exposure, at site(s), of histologic type(s), or by mechanism(s) considered relevant to worker exposure. The A2 is used primatrily when there is limited evidence of carcinogenicity in humans and sufficient evidence of carcinogenitity in experimental animals with relevance to humans.				
		STEL	25 ppm	2008-01-01	USA. ACGIH Threshold Limit Values (TLV)
	Central Nervous System impairment cognitive decrement Renal toxicity Suspected human carcinogen: Human data are accepted as adequate in quality but are conflicting or insufficient to classify the agent as a confirmed human carcinogen; OR, the agent is carcinogenic in experimental animals at dose(s), by route(s) of exposure, at site(s), of histologic type(s), or by mechanism(s) considered relevant to worker exposure. The A2 is used primatrily when there is limited evidence of carcinogenicity in humans and				

	sufficient evidence of carcinogenitity in experimental animals with relevance to humans.

Personal protective equipment

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection
Handle with gloves.

Eye protection
Safety glasses with side-shields conforming to EN166

Skin and body protection
Choose body protection according to the amount and concentration of the dangerous substance at the work place.

Hygiene measures
Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form	liquid, clear
Colour	colourless

Safety data

pH	no data available
Melting point	-84.8 °C (-120.6 °F) - lit.
Boiling point	86.7 °C (188.1 °F) - lit.
Flash point	no data available
Ignition temperature	410 °C (770 °F) -
Lower explosion limit	8 %(V)
Upper explosion limit	10.5 %(V)
Vapour pressure	81.3 hPa (61.0 mmHg) at 20.0 °C (68.0 °F)
Density	1.463 g/mL at 25 °C (77 °F)
Water solubility	no data available
Partition coefficient: n-octanol/water	log Pow: 2.29

10. STABILITY AND REACTIVITY

Storage stability
Stable under recommended storage conditions.

Materials to avoid
Oxidizing agents, Strong bases, Magnesium

Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Hydrogen chloride gas
Hazardous decomposition products formed under fire conditions. - Nature of decomposition products not known.

11. TOXICOLOGICAL INFORMATION

Acute toxicity

LD50 Oral - rat - 4,920 mg/kg

LC50 Inhalation - mouse - 4 h - 8450 ppm

LD50 Dermal - rabbit - > 20,000 mg/kg

Irritation and corrosion

Skin - rabbit - Severe skin irritation - 24 h

Eyes - rabbit - Eye irritation - 24 h

Sensitisation

no data available

Chronic exposure

This product is or contains a component that has been reported to be probably carcinogenic based on its IARC, OSHA, ACGIH, NTP, or EPA classification.

IARC:	2A - Group 2A: Probably carcinogenic to humans (Trichloroethylene)
NTP:	Reasonably anticipated to be a human carcinogen (Trichloroethylene)
OSHA:	No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Laboratory experiments have shown mutagenic effects.

Signs and Symptoms of Exposure

burning sensation, Cough, wheezing, laryngitis, Shortness of breath, Headache, Nausea, Vomiting, Exposure to and/or consumption of alcohol may increase toxic effects., Gastrointestinal disturbance, Kidney injury may occur., narcosis

Potential Health Effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation. Vapours may cause drowsiness and dizziness.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.
Ingestion	May be harmful if swallowed.
Target Organs	Liver, Central nervous system, Heart, Lungs,

Additional Information
RTECS: KX4550000

12. ECOLOGICAL INFORMATION

Elimination information (persistence and degradability)

Ecotoxicity effects

Toxicity to fish	LC50 - Pimephales promelas (fathead minnow) - 41.00 mg/l - 96 h

	LOEC - other fish - 11 mg/l - 10 d
	NOEC - Oryzias latipes - 40 mg/l - 10 d
Toxicity to daphnia and other aquatic invertebrates.	EC50 - Daphnia magna (Water flea) - 18.00 mg/l - 48 h
Toxicity to algae	IC50 - Pseudokirchneriella subcapitata (green algae) - 175.00 mg/l - 96 h

Further information on ecology

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.

Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

13. DISPOSAL CONSIDERATIONS

Product
Observe all federal, state, and local environmental regulations. Contact a licensed professional waste disposal service to dispose of this material. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging
Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)
UN-Number: 1710 Class: 6.1 Packing group: III
Proper shipping name: Trichloroethylene
Marine pollutant: No
Poison Inhalation Hazard: No

IMDG
UN-Number: 1710 Class: 6.1 Packing group: III EMS-No: F-A, S-A
Proper shipping name: TRICHLOROETHYLENE
Marine pollutant: No

IATA
UN-Number: 1710 Class: 6.1 Packing group: III
Proper shipping name: Trichloroethylene

15. REGULATORY INFORMATION

OSHA Hazards
Irritant, Carcinogen

DSL Status
All components of this product are on the Canadian DSL list.

SARA 302 Components
SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components

	CAS-No.	Revision Date
Trichloroethylene	79-01-6	2007-07-01

SARA 311/312 Hazards
Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Trichloroethylene	79-01-6	2007-07-01

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Trichloroethylene	79-01-6	2007-07-01

New Jersey Right To Know Components

	CAS-No.	Revision Date
Trichloroethylene	79-01-6	2007-07-01

California Prop. 65 Components

	CAS-No.	Revision Date
WARNING! This product contains a chemical known in the State of California to cause cancer. Trichloroethylene	79-01-6	2007-09-28

16. OTHER INFORMATION

Further information

Appendix C: NG-18 Gel Cleaning Pictures and Additional Data Tables

Within this appendix are extra pictures and tables showing the results of the NG-18 gel trials. The MSDS for Imbiber beads, TCE and THF are also included in the appendix

Figure 23. Photographs metal coupon immersed in gels: (left) NG-18-1%, (center) NG-18-0.5%, and (right) toluene

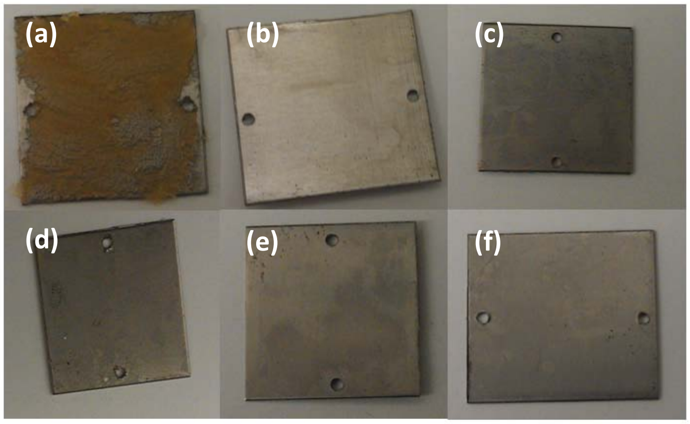

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Figure 24. Metal coupon before (b), and after (a) being contaminated with MIL-PRF-10924 grease. Metal coupons cleaned with NG-18-0.5% (c)-(d) for 13 minutes and 32 seconds, TCE for 5 minutes 12 seconds (e), and NG-18-1% for 12 minutes 58 seconds (f)

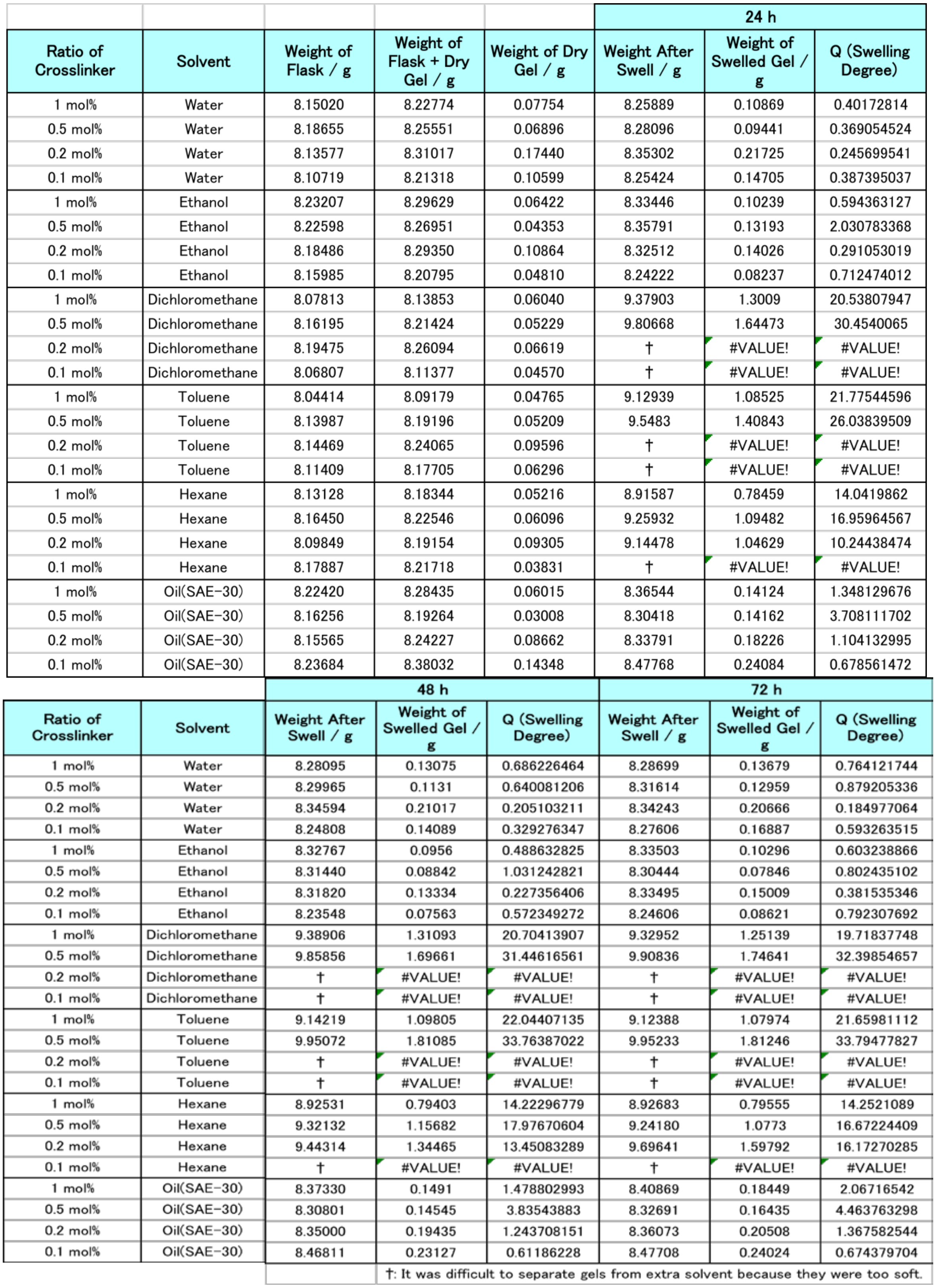
Table 6. Experimental grease absorption data of NG-18 gels

Coupon Weight (g)	Coupon with grease weight (g)	Grease weight (g)	Cleaning Product	Time (min)	Clean weight (g)	Amount cleaned (g)	Percent of grease removed
2.65958	3.01575	0.35617	NG-18-0.5%	13.87	2.65847	0.35728	100.312
2.67286	3.06526	0.3924	NG-18-0.5%	13.83	2.67409	0.39117	99.687
2.73117	3.18272	0.45155	NG-18-0.5%	13.53	2.73248	0.45024	99.710
2.57225	2.89459	0.32234	NG-18-1%	12.97	2.57361	0.32098	99.578
2.70775	3.17597	0.46822	NG-18-1%	13.58	2.71145	0.46452	99.210
2.99139	3.37955	0.38816	NG-18-1%	13.67	2.99347	0.38608	99.464
2.42991	2.82545	0.39554	TCE	5.20	2.43002	0.39543	99.972
2.52677	2.97063	0.44386	TCE	5.17	2.52973	0.44090	99.333
2.56630	2.99553	0.42923	TCE	5.10	2.56653	0.42900	99.946

Table 7. Cleaning power data of NG-18 gels

Cleaning Product	Average Percent removed (g)	Average time (min)	Cleaning Power
NG-18-1%	99.42	13.41	86.59
NG-18-0.5%	99.90	13.74	86.26
TCE	99.75	5.16	94.84

Table 8. Swelling data of NG-18-x% gel in various solvents for 24 h, 48 h, and 72 h.



					24 h		
Ratio of Crosslinker	Solvent	Weight of Flask / g	Weight of Flask + Dry Gel / g	Weight of Dry Gel / g	Weight After Swell / g	Weight of Swelled Gel / g	Q (Swelling Degree)
1 mol%	Water	8.15020	8.22774	0.07754	8.25889	0.10869	0.40172814
0.5 mol%	Water	8.18655	8.25551	0.06896	8.28096	0.09441	0.369054524
0.2 mol%	Water	8.13577	8.31017	0.17440	8.35302	0.21725	0.245699541
0.1 mol%	Water	8.10719	8.21318	0.10599	8.25424	0.14705	0.387395037
1 mol%	Ethanol	8.23207	8.29629	0.06422	8.33446	0.10239	0.594363127
0.5 mol%	Ethanol	8.22598	8.26951	0.04353	8.35791	0.13193	2.030783368
0.2 mol%	Ethanol	8.18486	8.29350	0.10864	8.32512	0.14026	0.291053019
0.1 mol%	Ethanol	8.15985	8.20795	0.04810	8.24222	0.08237	0.712474012
1 mol%	Dichloromethane	8.07813	8.13853	0.06040	9.37903	1.3009	20.53807947
0.5 mol%	Dichloromethane	8.16195	8.21424	0.05229	9.80668	1.64473	30.4540065
0.2 mol%	Dichloromethane	8.19475	8.26094	0.06619	†	#VALUE!	#VALUE!
0.1 mol%	Dichloromethane	8.06807	8.11377	0.04570	†	#VALUE!	#VALUE!
1 mol%	Toluene	8.04414	8.09179	0.04765	9.12939	1.08525	21.77544596
0.5 mol%	Toluene	8.13987	8.19196	0.05209	9.5483	1.40843	26.03839509
0.2 mol%	Toluene	8.14469	8.24065	0.09596	†	#VALUE!	#VALUE!
0.1 mol%	Toluene	8.11409	8.17705	0.06296	†	#VALUE!	#VALUE!
1 mol%	Hexane	8.13128	8.18344	0.05216	8.91587	0.78459	14.0419862
0.5 mol%	Hexane	8.16450	8.22546	0.06096	9.25932	1.09482	16.95964567
0.2 mol%	Hexane	8.09849	8.19154	0.09305	9.14478	1.04629	10.24438474
0.1 mol%	Hexane	8.17887	8.21718	0.03831	†	#VALUE!	#VALUE!
1 mol%	Oil(SAE−30)	8.22420	8.28435	0.06015	8.36544	0.14124	1.348129676
0.5 mol%	Oil(SAE−30)	8.16256	8.19264	0.03008	8.30418	0.14162	3.708111702
0.2 mol%	Oil(SAE−30)	8.15565	8.24227	0.08662	8.33791	0.18226	1.104132995
0.1 mol%	Oil(SAE−30)	8.23684	8.38032	0.14348	8.47768	0.24084	0.678561472

		48 h			72 h		
Ratio of Crosslinker	Solvent	Weight After Swell / g	Weight of Swelled Gel / g	Q (Swelling Degree)	Weight After Swell / g	Weight of Swelled Gel / g	Q (Swelling Degree)
1 mol%	Water	8.28095	0.13075	0.686226464	8.28699	0.13679	0.764121744
0.5 mol%	Water	8.29965	0.1131	0.640081206	8.31614	0.12959	0.879205336
0.2 mol%	Water	8.34594	0.21017	0.205103211	8.34243	0.20666	0.184977064
0.1 mol%	Water	8.24808	0.14089	0.329276347	8.27606	0.16887	0.593263515
1 mol%	Ethanol	8.32767	0.0956	0.488632825	8.33503	0.10296	0.603238866
0.5 mol%	Ethanol	8.31440	0.08842	1.031242821	8.30444	0.07846	0.802435102
0.2 mol%	Ethanol	8.31820	0.13334	0.227356406	8.33495	0.15009	0.381535346
0.1 mol%	Ethanol	8.23548	0.07563	0.572349272	8.24606	0.08621	0.792307692
1 mol%	Dichloromethane	9.38906	1.31093	20.70413907	9.32952	1.25139	19.71837748
0.5 mol%	Dichloromethane	9.85856	1.69661	31.44616561	9.90836	1.74641	32.39854657
0.2 mol%	Dichloromethane	†	#VALUE!	#VALUE!	†	#VALUE!	#VALUE!
0.1 mol%	Dichloromethane	†	#VALUE!	#VALUE!	†	#VALUE!	#VALUE!
1 mol%	Toluene	9.14219	1.09805	22.04407135	9.12388	1.07974	21.65981112
0.5 mol%	Toluene	9.95072	1.81085	33.76387022	9.95233	1.81246	33.79477827
0.2 mol%	Toluene	†	#VALUE!	#VALUE!	†	#VALUE!	#VALUE!
0.1 mol%	Toluene	†	#VALUE!	#VALUE!	†	#VALUE!	#VALUE!
1 mol%	Hexane	8.92531	0.79403	14.22296779	8.92683	0.79555	14.2521089
0.5 mol%	Hexane	9.32132	1.15682	17.97670604	9.24180	1.0773	16.67224409
0.2 mol%	Hexane	9.44314	1.34465	13.45083289	9.69641	1.59792	16.17270285
0.1 mol%	Hexane	†	#VALUE!	#VALUE!	†	#VALUE!	#VALUE!
1 mol%	Oil(SAE−30)	8.37330	0.1491	1.478802993	8.40869	0.18449	2.06716542
0.5 mol%	Oil(SAE−30)	8.30801	0.14545	3.83543883	8.32691	0.16435	4.463763298
0.2 mol%	Oil(SAE−30)	8.35000	0.19435	1.243708151	8.36073	0.20508	1.367582544
0.1 mol%	Oil(SAE−30)	8.46811	0.23127	0.61186228	8.47708	0.24024	0.674379704

†: It was difficult to separate gels from extra solvent because they were too soft.

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