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ENVIRONMENTALLY FRIENDLY CLEANERS FOR REMOVING TAR AND ASPHALT FROM TACTICAL AND TRANSPORTATION VEHICLES

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ENVIRONMENTALLY FRIENDLY CLEANERS FOR REMOVING TAR AND ASPHALT FROM TACTICAL AND TRANSPORTATION VEHICLES

1. Purpose.

a. This Public Works Technical Bulleting (PWTB) identifies and provides recommendations for the selection of commercially available, environmentally friendly cleaners for removing road tar and asphalt from Army ground vehicles.

b. All PWTBs are available electronically at the National Institute of Building Sciences' Whole Building Design Guide (WBDG) webpage, which is accessible through the following link:

http://www.wbdg.org/ccb/browse_cat.php?o=31&c=215

2. Applicability.

a. This PWTB applies to all U.S. Army public works activities and facilities having ground vehicle surface cleaning activities.

b. Note that the contents of this report are not to be used for advertising, publication, or promotional purposes. Citation of trade names does not constitute an official endorsement or approval of the use of such commercial products. All product names and trademarks cited are the property of their respective owners. The findings of this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

3. References.

a. Army Regulation (AR) 200-1, *Environmental Protection and Enhancement*, 28 August 2007.

b. Solid Waste Disposal Act, 42 U.S.C. 6901-6992k, as amended by Public Law (PL) 107-377, 31 December 2002, commonly known as the Resource Conservation and Recovery Act (RCRA).

c. Hazardous and Solid Waste Amendments (HSWA) to RCRA, PL 98-616, 98 Stat. 3221, 8 November 1984.

d. ASTM Standard D6361/D6361M–98(2010)e1, *Standard Guide for Selecting Cleaning Agents and Processes*, 01 June 2010.

e. Department of Defense, Military Performance Specification, MIL-PRF-680B, *Solvent Selection Guide*, 26 October 2006.

4. Discussion.

a. AR 200-1 requires that Army installations comply with Federal environmental regulations, including air emission restrictions established by the Clean Air Act.

b. The Resource Conservation and Recovery Act requires the US Environmental Protection Agency (USEPA) to promulgate regulations regarding the storage, processing, and disposal of solid and hazardous wastes. In 1984 the Act was augmented by the Hazardous and Solid Waste Amendments, which included provisions to encourage the recycling and reuse of hazardous wastes.

c. The Waste Minimization and Pollution Prevention (WMPP) Program was established by Congress to demonstrate promising off-the-shelf environmental technologies at Army installations. Funding for the WMPP program ended in fiscal year 2005 (FY05). During the 12-year tenure of this program, many commercially available, environmentally friendly cleaners were evaluated and demonstrated in the laboratory.

d. ASTM Standard D6361/D6361M–98(2010)e1 is a general guide that is used in developing cleaning requirements for manufacturing, maintenance, or overhaul specifications. This guide was designed to be application specific for individual cleaning tasks, and to assure the design engineer that cleaning agents and processes selected by the industrial or manufacturing engineer will be compatible with both part materials and with subsequent processes. Industrial or manufacturing engineers use the guide to customize their selection of cleaning products based on: the materials of the part being cleaned; the cleanliness required for the subsequent processes; and environmental, cost, and health and safety concerns.

e. MIL-PRF-680B is a US Department of Defense (DoD) standard used to describe products that meet specific performance and manufacturing standards for equipment and chemicals.

f. US Army vehicle maintenance facilities make every effort to implement surface cleaners that are environmentally friendly. In other words: (a) cleaners that are free of hazardous air pollutants (HAPs), (b) cleaners that contain the minimum possible amount of volatile organic compounds (VOCs), and (c) cleaners that eliminate greenhouse gas emissions. Also, Army vehicle maintenance facilities have a great interest in reducing the overall energy costs associated with surface cleaning operations.

g. Appendix A contains an evaluation of environmentally friendly cleaners for removing tar and asphalt from tactical and transportation vehicles.

h. Appendix B contains a list of technical references cited in Appendix A and other resources on the subject of this PWTB.

i. Appendix C contains photographs of laboratory experiments performed to evaluate candidate cleaners for recommendation, as described in Appendix A.

j. Appendix D contains a list of abbreviations used.

5. Points of Contact.

a. Headquarters, U.S. Army Corps of Engineers (HQUSACE) is the proponent for this document. The point of contact (POC) at HQUSACE is Mr. Malcolm E. McLeod, CEMP-CEP, 202-761-5696, or e-mail: Malcolm.E.Mcleod@usace.army.mil.

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For JAMES C. DALTON, P.E., SES
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Directorate of Civil Works

Appendix A

EVALUATION OF ENVIRONMENTALLY FRIENDLY CLEANERS FOR REMOVING TAR AND ASPHALT FROM TACTICAL AND TRANSPORTATION VEHICLES

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Introduction

Background

Every Army vehicle receives regular maintenance. Ground vehicles that come into the maintenance facility (especially bitumen spreaders) commonly have asphalt ("road tar") on their surfaces. Before a vehicle is washed or painted, the tar must be removed. The current method for removing bitumen from the vehicles involves the use of a high-pressure (40,000 psi) water jet, followed by manually scraping any residual asphalt. Any residual asphalt is manually scraped ("spot cleaned") using such commercial formulations as Biopro and Teskol (from Inland Technologies). The vehicle is visually inspected and sometimes subjected to a water-break test to evaluate the success of the initial tar removal step. Additionally, when bitumen spreaders are cleaned, their nozzle manifolds must also be serviced. Clogged nozzles that cannot be cleaned are replaced. Such mechanical and manual methods of vehicle cleaning and servicing are very expensive and inefficient.

Old Cleaning Process

Before using the water jet to remove residual tar from vehicles, the Army relied on PD-680 type solvents and diesel fuel to clean surfaces coated with tar and asphalt.[*] These highly refined aliphatic hydrocarbon compounds have been phased out because they contain large amount HAPs and VOCs that have negative environmental impacts. High levels of HAPs and VOCs are toxic, flammable, can contribute to certain health problems, and are considered hazardous waste (Crockett 2007; Trivedi et al. 2007).

[*] The information regarding current practice to remove tar was obtained from the US Army Tank Automotive Research, Development, and Engineering Center (TARDEC).

It is also difficult to dispose of the waste from these cleaning processes, which may contain chemicals that must be treated before entering wastewater drains.

There are three types of PD-680 solvents, which differ only in their flash points, from Type I (which has the lowest flash point) to Type III (which has the highest). In general, the higher the flash point, the safer the cleaner will be (Crockett 2007). Conversely, the higher the flash point, the lower the overall solvency. In other words, environmentally safer solvents (with relatively high flash points) may not do such a good job. Types I and II do not meet Federal law because of their low flash point and high amount of VOC emissions (Rhee et al. 1999). Nevertheless, many users prefer Types I and II solvents because they are effective degreasers and are non-corrosive. Users are often reluctant to switch to safer substitutes, which leave residue and can corrode metal.

Recent Technological Advances

Most traditional commercial solvents for removing asphalt have been petroleum distillates, or chlorinated solvents, or a combination of the two (Kulkarni et al. 2003). However, recent formulations of tar and asphalt removers use biological extracts and environmentally benign materials (Baird et al. 2009). Several vendors have recently proposed more environmentally friendly ("green") cleaners to replace PD-680 type cleaners. Many of these cleaners are characterized by a high flash point, no HAPs, and little to no VOCs. Vendors also claim that these environmentally friendly cleaners are equal to or better than PD-680 type solvents.

The use of such environmentally acceptable solvents to remove residual tar from ground vehicles may offer a more efficient and cost-effective alternative. The Army is leading an effort to develop and demonstrate pollution prevention technologies to reduce hazardous air pollutants and other volatile organic emissions at surface cleaning and painting operations at DoD facilities. This effort focuses on evaluation of solvents for removal of tar from ground vehicle surfaces.

Selection of Commercially-Available Cleaners

Literature Search

A literature survey of commercially available solvents was done to identify commercially available solvents suitable for tar removal that were free from hazardous chemicals (i.e., that were safe for users), and that produced waste that could be disposed of simply. The Material Safety Data Sheets (MSDSs) and properties of each solvent were reviewed. Table A-1 lists the products found, their manufacturers, stated applications, and chemical compositions.

Table A-1. Commercial Tar Removing Solvents.

No	Company	Cleaner	*BP	*VP mmHg	*VOC g/L	*FP	Major Chemicals	Performance	Cost	Residuals/ Byproducts	Waste Disposal	Toxic	First Aid
1	Beaver Research 3700 W. Kilgore Rd. Portage, MI 49002 Toll Free: 800.544.0133 Phone: 269.382.0133 Fax: 269.382.0214 sales@beaverresearch.com	57 A	360410 °F	30 @ Rm Temp		145 °F *TCC Tester	Diethanolamine (D-60) solvent naphtha medium aliphatic	Rinses freely & completely.		CO & CO_2	Incinerate according to federal, state, and local regs.	No toxic chemicals according to reporting requirements, Section 313.40 CFR Part 372	Standard (see footnote for details)
2	Biochem systems 3511 N. Ohio Wichita, KS 67219 TEL: (316) 838-4739 (800) 777-7870 FAX: (316) 681-2168 http://www.biochemsys.com/	Bio T Max	334 °F	<2	780	130°F *PMCC Tester	D-limonene	Wipe clean or rinse with water. Can be diluted. Hand wipe, ultrasonic tank, dip tank, conveyorized spray system, pressure sprayers	$25.65 per gal	CO	Biodegradable	Non-toxic; no chlorinated solvents & petroleum distillates	Standard
3	BioSystems, Inc. PO 464 Fort Collins, CO 80522-0464 (800) 224-4605 info@biosystemsinc.com	BioPro	347 °F	2		>122°F CC Tester	D-limonene Nonionic surfactant	Insoluble in water			100% biodegradable	Non-toxic; no aerosol; no CFCs	Eyes: flush with water; Skin: wash with soap & water.
4	Chemco Corporation 5731 Manchester Ave. St. Louis, MO 63110 1-800-854-4236 Fax: 314-647-1850 info@ChemcoCorp.com	Tarva Sol	349 °F	25 1.4	N/A	125 °F *TCC Tester	D-limonene	Spray on, wipe off, can be diluted with water	5-gal pail = $21.60 gal	CO & CO_2	Biodegradable		Eyes: flush with water for 15 min. See physician if irritation persists
5	Citrus Depot 800-424-8045 www.citrusdepot.net/	Citrus King	>310 °F			115°F PMCC	Terpenes	Spray on or brush on	5 gal pail = $129.00	Biodegrad-able		Non-toxic	Skin: flush with water for 15 min. See physician if irritation persists. Skin: wash with soap & water; Inhalation: remove to fresh air; if symptoms persist, seek medical attention; Ingestion: give milk magnesia, glass water, or milk. Seek physician help.

*BP = boiling point *COC = Cleveland open cup tester *FP = flash point *PMCC = Pensky-Martens closed cup tester

*TCC = Tag (Tagliabue) closed cup tester *VOC = volatile organic compound *VP = vapor pressure

Standard first aid response would include the following steps, as applicable. INHALATION: Remove affected person to fresh air; provide oxygen if breathing is difficult; if affected person is not breathing, administer CPR and seek emergency medical attention. SKIN: Remove contaminated clothing; wash affected area with soap and water; launder contaminated clothing before reuse; if irritation persists, seek medical attention. EYES: Check for and remove contact lenses. Flush eyes with clear running water for 15 minutes while holding eyelids open; if irritation persists, seek medical attention. INGESTION: DO NOT induce vomiting; if vomiting occurs spontaneously, keep head below hips to prevent aspiration of liquid into lungs; seek immediate medical attention. Vomiting may be induced only under the supervision of a physician.

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No	Company	Cleaner	*BP	*VP mmHg	*VOC g/L	*FP	Major Chemicals	Performance	Cost	Residuals/ Byproducts	Waste Disposal	Toxic	First Aid
6	Cleanline Products, Inc. PO Box 625 Canton, TX 75103 1-888-536-5185 FAX: 903-567-4600 info@clineproducts.com	Citrus Blast	<300 °F	As water		128°F *COC Tester	Isoparaffins		32 oz, 128 oz, 55 gal drums	From combustion: smoke, CO_2, unknown organic compounds	Biodegradable organics		Eyes: flush with flowing water for 15 min; Ingestion: give 2 glasses of water & summon medical attention.
7	Coastwide Labs www.coastwidelabs.com 1-800-775-3289	Orange Waterless	212 °F	NA		<160 °F *COC Tester	Nonionic surfactant Beta-Pinene Citrus distillate	Dissolves, no scrubbing, wipe away suspended particles	12-1 qt case; 4-1 gal case; 55 gal drum		BiodegradableNo phosphates or Petroleum products	Non toxic as far as known to Coastwide	Eyes: immediately flush eyes with water for 15 min., lifting eyelids occasionally. Get medical attention Skin: immediately wash skin with soap & water. Remove contaminated clothing & shoes. Wash before reuse. Inhale: remove to fresh air. Get medical attention. Give artificial respiration if not breathing Ingestion: do not induce vomiting. Get immediate medical attention.
8	Cogent Environmental Solutions 13 Adrian Ave, Mansfield, Ontario, Canada L0N 1M0 (705) 434-4489 FAX: (705) 434-9675 cogentenvironment@ecogent.ca	ECO-gent	N/A			none	2-Hydroxypropanoic acid Alkyl polyglycoside Glucopyranose, oligomeric, decyl octyl glycosides	Apply undiluted; allow to penetrate; agitate with cloth or sponge; rinse	1 gal, 5 gal, 55 gal	May include & not limited to oxides of carbon	Review fed, state, local regs before disposal	No pesticides or preservatives	Eye: flush with flowing water for 15 mins Skin: flush with water. Wash with soap & water. Obtain medical attention. Inhale: move to fresh air. Ingestion: do not induce vomiting.
9	Continental Research, Corp. PO Box 15204 St. Louis, MO 63110 1-800-325-4869	Full Force	352 °F	2		155 °F TCC	1,8-P-Menthadiene Propane/n-Butane Carbon Dioxide Nonionic Surfactant	High pressure flushing spray	Aerosol 1 doz. cans=$166			Contains no chlorinated solvents or petroleum distillates	Eye: flush with water for 15 min. Skin: rinse with soap & water Ingestion: give small glass of milk or water. Do not induce vomiting. Inhalation: Remove victim to fresh air.
10	Continental Research, Corp. PO Box 15204 St. Louis, MO 63110 1-800-325-4869	Tuff Stuff	348 °F	1.0	>350 °F	115 °F TCC	Poly-(oxy-1,2-ethanediyl)-alpha-undecyl-omega Alcohols, C12-13, ethoxylated carbon dioxide		Aerosol 1 doz. cans = $191; 6.8 gal - $50/gal; 55 gal - $39/gal			Oral LD50 (rat) = >5000-15000 mg/kg Dermal LD50 (rabbit) = >2000 <equal to 20000 mg/kg	Standard

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No	Company	Cleaner	*BP	*VP mmHg	*VOC g/L	*FP	Major Chemicals	Performance	Cost	Residuals/ Byproducts	Waste Disposal	Toxic	First Aid
11	Continental Research, Corp. PO Box 15204 St. Louis, MO 63110 1-800-325-4869	Rid-O-Grease	348 °F	1.0		115 °F TCC	Citrus solvent		Aerosol or bulk		Biodegradable		Eye: flush with plenty of water for at least 15 min. Skin: flush with cold water and wash with soap & water; Ingestion: give milk or water; Inhalation: remove to fresh air
12	Delco Cleaning Systems of Fort Worth 2513 Warfield Street, Fort Worth TX 76106-7554 Phone: 800-433-2113 Fax: 817-625-2059 delco@dcs1.com http://www.dcs1.com	R-109 Delco Red Truck Wash Powder		Spec. Gray 7.84 lb/gal			Sodium Metasilicate, Penta Glycol Ether EB	Cold pressure washers, hot high pressure washers, and steam cleaners	50 lb $141; 100 lb $260; 500 lb $1,170; 1000 lb $1,600; Mix 0.5 lb/gal		See local authorities for restrictions on disposal for chemical waste		Eye: flush with plenty of water for at least 15 min. Lifting eyelids, remove contacts if possible. Call physician. Skin: flush with cold water and wash with soap & water. Ingestion: rinse mouth & drink 2 large glasses of water, do not induce vomiting.
13	Delta Foremost Chemical Corporation 3915 Air Park St. Memphis, TN 38118 (901) 363-4340	Citri-Kote	176 °C	VP not establ.	None	115 °F TAG	Natural solvent	Insoluble in water	55 gal drums			No VOCs or HAPs. No CFCs or other ozone depleting substances	Eye: flush with flowing water for 15 min. Skin: wash with copious amount of water & soap. Ingestion: call physician immediately
14	Delta Foremost	Citri-Zip	176 °C	VP not established		100 °F – 115 °F TAG, cc	Natural solvent	Emulsifier			Dispose according to local rules		Standard
15	Dysol 2901 Shamrock Ave. Fort Worth, TX 76107 (817) 335-1826 info@dysol.com	DS-104	257 °F 125 °C	3.5 mm Hg at 20 °C		105 °F 40.6 °C		Hand wipes, dip tank, ultrasonic	55 gal drum $1,281.16 (GSA Schedule NSN 7930-01-367-0985)			Low toxicity; no ODS or HAPS; Propylene glycol monoethyl ether acetate: 8532 mg/kg (oral rat); N-Butyl acetate: 14000 mg/kg (oral rat)	Standard
16	EaCo Chem. Inc. 765 Commerce Ave Newcastle, PA 16101 1-800-313-8505 Fax: (724) 656-0757 info@eacochem.com	C-Tar Melt	N/A	VP N/A; Spec. Gray 0.85		>125 °F TCC	Petroleum Hydrocarbon Ethylene glycol n-butyl ether	Can remove many materials in 15 min. Heavy layers overnight soak, Pressure washer rinsing with at least 1500 psi for best results.	EC 016-55g $1005.00 EC 016-5g $106.90	Hazardous decompositi on not known	Biodegradable. Prevent materials from entering sewers, storm drains, waterways	Non hazardous liquid	Eye: flood with water 15 min. Skin: wash off with soap & water Ingestion: drink lots of water; seek immediate medical attention. Inhaled: move to fresh air.

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17	EcoLink Coporate Headquarters 2177-A Flintstone Drive Tucker, GA 30084 800-886-8240 800-886-8240 info@ecolink.com	Electron 296	349 °F	0.3 @ 68 °F	810	147 °F *TCC Tester	Citrus terpene severely hydro-treated light distillates	Fully evapo-rative, leaves no residue	55 gal $1,181.72 5 gal $101.04	May form CO and CO_2	Not haz. waste prod.; dispose according to regs.	Non hazardous EPA & OSHA definitions. Acute toxicity: oral toxicity (mice)-LD50 5.6-6.6 g/kg; Skin toxicity: absorption (rabbits)- LD50 500 mg/kg	Ingestion: seek medical attention immediately. If individual is drowsy or unconscious, do not give anything by mouth; place individual on left side with head down. Contact medical facility or poison control center for advice on whether to induce vomiting. Inhalation: remove to fresh air. If breathing is difficult, give oxygen. Keep person warm & quiet. Seek medical attention. Eye: irrigate immediately with water for at least 15 min. Get medical attention if irritation persists. Skin: wash with soap & water. Thoroughly clean contaminated clothes & shoes before reuse.
18	Environmental Green Products (541) 301-2632	Ecoprocote SoyGreen 6000	> 600 °F	Not determined	<5-3.42%	> 200 °F	Trade secret		1 gal $44.95; 5 gal $214.65		Biodegradable		Eye: do not rub, flush with water, get medical attention. Skin: wash thoroughly with soap & water, wash clothing before reuse. Ingestion: get medical attention. Inhalation: remove to fresh air.
19	Inland Technology, Inc. 401 East 27th St. Tacoma, WA 98421 800-552-3100 inland@inlandtech.com	Teksol EP	310 °F	(mm Hg/70 °F) <10 mm Hg	Percentage by volume = 100	112 °F PMCC	Hydrotreated heavy Naphtha/C10-C11 Paraffinic hydrocarbons D-Limonene	Insoluble in water	5 gal $232.84	Haz decomp products: oxides of carbon and hydrocarbons	Contact fed, state, or local environmental regulatory agencies		Eye: flush with water at least 15 min; call physician if irritation continues. Skin: remove contaminated clothing and wash with soap & water. Inhalation: remove from exposed area and call physician. Ingestion: do not induce vomiting & call physician immediately.
20	Inland Technology, Inc.	Break-through	370 °F	<2 mm Hg at 25 °C	percentage by volume = 100	150 °F PMCC	Naphthol Spirits (C12-C13 Hydrocarbons)	Insoluble in water	5 g $149.11; 55 gal $1,053.63; 15 gal $383.32	Hazardous decomp products: oxides of carbon & hydrocarbons	Contact Fed, state of local environmental regulatory agencies		Eye: flush with water at least 15 min.; call physician if irritation continues. Skin: remove contaminated clothing and wash with soap & water. Inhalation: remove from exposed area and call physician. Ingestion: do not induce vomiting & call physician immediately.
21	ITW Rocol North America 3624 West Lake Avenue, Glenview, IL 60026 800-452-5823	Daraclean 282GF	212 °F (approx)	18 mm Hg at 20 °C	Not estab	None to BP (212 °F)	Potassium hydroxide 1-5% by wt.	Aqueous alkaline cleaner	5 g 55g	Dissolves in water and biodegradables	Dispose according to federal, state, and local laws and 40 CFR	Potassium hydroxide 1-5% by wt	Eye: rinse under upper & lower eyelids with water. Skin: wash off with water; use soothing lotion Inhalation: remove to fresh air. Ingestion: if conscious, give 2 glasses water; call physician or poison center.

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22	Kleen All Plus (800) 537-9545 info@kleenallplus.com	#141 Vehicle Wash	212 °F	N/A			Dipropylene Glycol Methyl Ether Anhydrous Sodium Hydroxide		55 gal drum $399 plus freight. Military $325/drum	No byproducts	Review fed, state, local regs before disposal		Eye & Skin: flush with water. Ingestion: get immediate medical attention.
23	Momar 1830 Ellsworth Industrial Dr. Atlanta, GA 30318	Agri-Sol	300-320 °F	1.6	non-volatile	250 °F	Methyl Ester Soybean oil	Dissolves asphalt/tar on contact. Spray 1 gal on 100 sq ft without diluting. Allow to soak 3-5 min to penetrate & dissolve. Hose down using water under pressure. For stubborn areas, scrub or scour pad to rinse.	55 gal drum = $33.75/gal 35 gal drum = $34.05/gal 20 gal drum = $34.40/gal 5 gal metal pail $35.50/gal 4-1 gal case = $37.40/gal	CO, CO_2	Biodegradable, biobased emulsifiable	No chlorinated solvents, HAPs, or CFCs	Eyes: flush with water for 15 min.; if irritation persists, call physician. Skin: apply moisturizing lotion; if irritation persists, call physician. Ingestion: do not induce vomiting.
24	Momar 1830 Ellsworth Industrial Dr. Atlanta, GA 30318	Vega-Sol	340 °F - 372 °F	Not estab.	Low % Volatile By vol. >40	>145 °F	Ethyl lactate Methyl Ester Soybean oil		55 gal drum = $50.85/gal 35 gal drum = $51.15/gal 20 gal drum = $51.50/gal 5 gal metal pail $52.60/gal 4-1 gal case = $54.50/g		Biodegradable, biobased, emulsifiable	No chlorinated solvents, HAPs, CFC's, ODC's	Eyes: flush with water for 15 min.; if irritation persists, call physician. Skin: apply moisturizing lotion; if irritation persists, call physician. Ingestion: do not induce vomiting.

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25	Orange Products, LLC 5403 Bosher Lake Dr McLeansville, NC 27301 Toll Free: (877) ORANGE2 Telephone: (336) 698-0000 Fax: (336) 272-0064 Terry@Orangeproduct.com	Orange Oily Slicker Asphalt Remover and Release Agent	310°F	1 mm at 25 °C		151 °F (ASTM D-93 PMCC)	Trade secret		1 g/$70.95 55/$1,480.00				Eyes: flush with water for 15 mins. If irritation persist call physician. Skin: Apply moisturizing lotion, if irritation persists, call physician. Ingestion: Do not induce vomiting. Drink large volume of water. Inhalation: Mist should be avoided
26	Orange Products, LLC www.orangeproduct.com	Citrus Soy Gold	N.D.			173 °F (TCC)	Soy methyl ester D-limonene Surfactant Soap Amine		$72.59 for 1 gal; $1580.00 for 55 gal.				Standard
27	Ostrem Chemical Co. 2310-80 Ave Edmonton AB T6P 1N2 (780) 440-191	T-300 Tar Remover	320 °F	N/A		114.8 °F *TCC Tester	Petroleum distillates Ethylene Glycol Monobutyl-Ether	Apply full strength with pressure sprayer or brush. Allow 5 min contact time, rinse with steam or hot water.	205 L drum $1,167.70 (Canadian) 20 L Pail $139.83 (Canadian)	Haz. combustion products: fumes, smoke, CO & sulfur oxides (in case of incomplete combustion).	Treat as petroleum solvent. Dispose according to local regs.		Standard
28	PCI of America, Inc. 2701 Tower Oaks Blvd., Suite 300 Rockville, MD 20852 301-468-1700	Hurrisafe 9030	212 °F 178 °C	14.2 mm Hg 77 °F 25 °C	SCSQMD method Concen: 47 g/l Dil 2:1 with water 15 g/l	None TCC	Aqueous based	Water soluble. Use as a presoak prior to steam cleaning. Use with high-pressure steam cleaners and pressure washers.		Non-hazardous, non-flammable, non-toxic, no VOCs, no terpenes.	Dispose according to local, state, & federal regs. Readily biodegradable.		Eyes: flush with water for 15 min; if irritation persists, call physician. Skin: apply moisturizing lotion; if irritation persists, call physician. Ingestion: do not induce vomiting. Inhalation: remove to fresh air.

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29	Petroferm, Inc. 2416 Lynndale Road Fernandina Beach, FL 32034 904-261-8286 FAX: 904-261-6994	Axarel 32	430 °F - 563 °F	<0.1 68 °F		205 °F *PMCC Tester	Mixed aliphatic hydro-carbons Diisobutyl dibasic acid ester mixture diisobutyl glutarate diisobutyl adipate diisobutyl succinate Alkyloxy polyethylene oxyethanol		$44.92/g		Waste: treat or incinerate used material in compliance with all applicable government regulations.	Diisobutyl dibasic acid Esters: LD50/oral/rat=16426 mg/kg LC50/inhalation/4hrs/rat=>31.9 Mg/L	Standard
30	Petroferm, Inc.	Bioact 105	>305 °F (>152 °C) (780 mm Hg)	<2 mm Hg (20 °C)		105°F (41C) ASTM D93-85 PMCC	Isoparaffinic hydrocarbon (C9-12 isoalkanes) 1-Propoxy-2-propanol 1-Methyl-4-(1-methyl-ethenyl)-cyclohexene		55g drum (365 lb) $2.55/lb; 5 gal cans (33 lb) $4.24/lb	None other than normal products of combustion	Waste: treat or incinerate in compliance with all applicable government regulations.	Isoparaffinic hydrocarbon (c9-12 isoalkane): LD50/oral/rat = > 10000 mg/kg LD50/dermal/ rabbit = > 3200 mg/kg; Inhalation: LC50 = 20 mg/L1-Propoxy-2-propanol:LD50/oral/rat= 3250 mg/Kg LD50/dermal/rabbit = 3560 mg/kg 1-methyl-4-(1-methylethenyl)-cyclohexene: LD50/oral/rat = > 5000 mg/kg LD50/dermal/rabbit= > 5000 mg/Kg	Standard
31	Petroferm, Inc.	Bioact 121	340 °F – 372 °F	<2		117 °F PMCC	1-Methyl-4-(methylethenyl) - cyclohexene	Hand wipe; commercially available equipment; evaporates					Eyes: flush with water for 15 min; if irritation persists, call physician. Skin: apply moisturizing lotion; if irritation persists, call physician. Ingestion: do not induce vomiting. Inhalation: remove to fresh air.

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32	Petroferm, Inc.	Bioact 120	340 °F – 372 °F	< 2		117 °F PMCC	1-methyl-4-(methylethenyl)-cyclohexene Poly(oxy-1,2-ethanediyl), alpha,-(4-onylphenyl),alpha,-(4-onylphenyl)-omega-hydroxy-	Water rinse	55 gal drum (390 lb)= $3.50/lb; 5 gal cans (35 lb)= $4.84/lb			1-Methylethenyl)-cyclohexene: LD50/oral/rat = > 5000 mg/kg LD50/dermal/rabbit = > 5000 mg/kg Poly(oxy-1,2-ethanrdiyl), alpha. –(4-onylphenyl)-omega.-hydroxy: LD50/oral/rat = 1300 mg/kg LD50/dermal/rabbit 1.8 ml/kg to 4.,400 mg/kg	Standard
33	Petroferm, Inc.	Bioact MSO	340 °F – 372 °F	< 2		117 °F PMCC	1-methyl-4-(methylethenyl)-cyclohexene		55 gal drum (380 lb)= $3.21/lb; 5 gal cans (35 lb) = $4.91/lb				Eyes: flush with water for 15 min; if irritation persists, call physician. Skin: apply moisturizing lotion; if irritation persists, call physician. Ingestion: do not induce vomiting. Inhalation: remove to fresh air.
34	Plush 801-377-5874	Dissolve Away							32 oz. $19.95 plus S&H				
35	Schaeffer Mfg Co. Eau Galle, Wisconsin 715-283-4031 4molyoil@wwt.net	#739 Citrol II		PSIG @ 70 °F: Max 50			Monocyclic terpene	Spray on or apply by brush. For best results, let set for 5 min. Rinse with H_2O.	55 gal drum = $1,437.15; 12 cans /case = $89.86	CO2	Biodegradable Citrol II waste treatable by standard POWTPs. Not considered a primary pollutant.	All natural, organic citrus based solvents	Standard
36	Selden Research Ltd Staden Business Park Staden Lane, Buxton Derbyshire, SK17 QRZ Tel : 01298 26226 sales@selden.co.uk	Tar N Glue				116°F	1,2,4-Trimethylbenzene Alcohol Ethoxylate Solvent, Light aromatic Naptha (Petroleum) Xylene-ortho	Apply soft cloth & rub until residue removed. Wipe all areas with wet sponge.			Dispose waste & residues in accordance with local authority requirements.	Vapor can be hazardous if inhaled.	Standard

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37	Sentinel Products, Inc. 51 NE 77th Ave Mpls, MN 55432 800-373-0633 Fax: 763-571-1819	Sentinel 700	370 °F – 518 °F	Negligible		146°F	Refined petroleum solvents Ethylene Glycol Monobutyl Ether	Apply to surface. Agitate or soak for 4-8 min. Rinse with water under pressure.		Thermal decomposition in presence or air may yield CO and/or CO_2.		All natural, no hazardous chlorinated or flammable solvents. Non caustic & non corrosive.	Standard
38	Simple Green 15922 Pacific Coast Hwy, Huntington Harbour, CA 92649 800-228-0709	Extreme Simple Green Aircraft & Precision Cleaner	210 °F	20.7 mm Hg	8.00% (80 g/L) EPA Method 24 2.04% SCAQMD Method 313	None (ASTM D-93)	Proprietary ethoxylated fatty alcohol <5% by wt; Proprietary fatty alkyl cocoamide < 5% by wt; Propylene glycol < 1% by wt; Propylene glycol butyl ether < 5% by wt; Triethanolamine <1 1%		55 gal = $494.00; 275 kg tote = $2,696.00		Biodegradable	Non-hazardous, VOC compliant	Eye: flush with plenty of water. After 5 min remove contact lenses. Flush another 10 min moving eyelids out of way. If irritation continues, call physician. Ingestion: drink plenty of water.
39	Smart Washer ChemFree, Corp. 8 Meca Way, Norcross, GA 30093 Tel: (770) 564-5580 Fax: (770) 564-5533 www.chemfree.com	Ozzy Juice SW-6 (for the Smart Washer System)	210 °F 99 ° C	<5 mm Hg	< 18g/L	None >200 °F COC		(For the Smart Washer System)			Biodegradable	No phosphates, formaldehydes, biocides, or solvents. Non-toxic, non-hazardous, non-caustic	Eyes: rinse if necessary. Skin: wash with soap & water. Ingestion: seek medical attention if necessary. Inhalation: move to fresh air.
40	SOYsolv 6154 N CR 33 OH 44883 800-231-4274 Fax: 419-992-4595 sales@soysolv.com	SOYSolv	420°F	<1	<50g/L		Mixed Fatty & Methyl Esters: Linoleic Oleic Palmitic Linolenic Stearic Palmitoleic Erui		32oz spray $10.72		Biodegradable	Non toxic	Standard
41	SOYsolv	SOYSolv II	420°F	0	<50g/L		Mixed Fatty Acid Methyl Esters		32oz spray $12.80	Thermal decomposition CO & CO2 from burning	Biodegradable	Non toxic	Oral: call physician. Ventilation procedure: local, mechanical, special

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42	SOYsolv	SOYSolv II Plus	292°F	0.9 @ 68°F			Ethyl Lactate Methyl Soyate			Decomposes to H_2O & CO_2 completely combusted.	Biodegradable	Non toxic	Standard
43	SSPENVIRO Safety Short Production Environmental Division 950 Gemini, Suite 1 Houston, TX 77058 1-800-458-2236 Fax: 281-956-1000 www.sspenviro.com	GoldSolv	> 200 °F	< 5@ 70 °F	0	212°F	Organic ingredients	Apply with sprayers, brushes, rollers. Rinse with water.	Case (6-1 gal jugs) $125.00; 5 gal pail $102.50; 30 gal drum $573.95; 55 gal drum $997.50	No decompose-tion products	Biodegradable	Eye contact : irritant Skin Contact : LD50 (rats/24hr) >2000mg/kg Inhalation: LC50 (rats/4hrs) >5400mg/kg Ingestion: LD50 >4090 mg/kg (rat) (RTECS 1985-86)	Standard
44	Stoner, Inc. 1070 Robert Fulton Hwy, Quarryville, PA 17566 800-227-5538 www.stonersolutions.com	Bug & Tarmi-nator	Not applic.	35.00 psig @ 70 °F			Petroleum distillates Halogenated hydrocarbon Petroleum hydrocarbon Glycol ether Aliphatic hydrocarbon Dimethyl carbinol Citrus distillates		12 can case = $40.87 (Free S&H)	Burning can produce: CO, CO_2, aldehydes, various hydro-carbons, decomposed by very high temps; may produce hydro-fluoric acid and carbonyl fluoride	According to regulations.		Eyes: flush with water for 15 min; contact physician if irritation persists Skin: wash with water for 15 min; contact physician if symptoms persist. Wash clothing before reuse. Ingestion: don't induce vomiting; give 8 ounces water to drink; contact physician. Inhalation: remove to fresh air; seek medical attention.
45	United Labs Canadian Headquarters United Laboratories of Canada 214 Dolomite Drive Toronto, ON M3J 2N2 (800) 323-2594 sales@unitedlabsinc.ca	United 399	347 °F	~1.0 @ 77 °F	7 lb/ gal	115-125°F *TCC Tester	d-1,8(9)-p-menthadiene	Don't allow to dry on surface. Hose off with water.	Liquid 5 L Liquid 20 L; Liquid 200 L	When ignited produces CO and CO_2.	Accumulate run-off into oil/water separator.	No petroleum distillates, acids, or caustics	Standard

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46	Walter Surface Technol. J. Walter Inc. 810 Day Hill Road Windsor, CT 06095 (800) 522-0321 Fax: (860) 560-7300	Bio Clean	257 °F		900 g/L	113 °F	Orange terpenes Ethyl Lactate		$44.75/g	Carbon oxides	Dispose according to local, state, and federal regulations.		Standard
47	Walter Surface Technol. J. Walter Inc. 810 Day Hill Road Windsor, CT 06095 (800) 522-0321 Fax: (860) 560-7300	X-Force (L-74E)	>500°F			266 °F			Bottle 5 L $67.91	CO	Biodegradable	Non-hazardous, Non-toxic	Eye: rinse thoroughly for 15 min. Skin: rinse with water until removed. Inhalation: N/A. Ingestion: do not induce vomiting; take approximately 5-10 g of edible oil & eut. animal charcoal.

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Basic Requirements for Green Cleaners

The green solvent that works best must fill three basic criteria:

1. Cleaning Effectiveness. This is a quantitative measurement of how much tar or asphalt the solvent can remove. Simply stated, the best solvent removes the most tar or asphalt.

2. Corrosiveness. This refers to the solvent's ability to damage or rust the metal. Very corrosive solvents can cause surface rust immediately after cleaning. Checking the pH of the solvent can give an indication of its corrosiveness. If the pH is either too high or too low, it can easily corrode the metal, rendering it useless.

3. Cost. Army maintenance facilities use large quantities of solvents to clean vehicles and other equipment, and have a great interest in minimizing solvent costs. Overall cost can be reduced by the ability to recycle and reuse the solvent after its initial use.

Weapons System-Specific Criteria

Trivedi et al. (2004) presented an excellent review of general cleaner requirements and green cleaning alternatives for weapon systems and machine parts for the DoD. Trivedi et al. have evaluated select cleaners in a laboratory environment, and listed the following requirements for weapon-systems cleaners.

1. Contaminant Removal. The primary requirement is that the cleaner must be able to remove contaminants, such as grease, oil, sand/dirt, carbon, copper fouling, etc.

2. Solvent Residue. The solvent must be able to dry without leaving a film of nonvolatile residue that may corrode or create toxicity problems. (This is a measure of "solvent purity.")

3. Material Compatibility. The solvent must not degrade or accelerate surface corrosion of different materials used in the weapon system. The degreasing aqueous solution should have a pH between 2.5 and 11.0.

4. Solvent Stability. The cleaner should have low VOCs. The cleaner stability affects process cost and operating hazards.

5. Flammability. Flammability is an important issue with non-aqueous cleaners. The concentrated product should not be ignitable, i.e., the flash point for the compound should be above 140 °F (60 °C).

6. Toxicity. The concentrated cleaning compound shall have no adverse affects on health of personnel or the environment.

7. Biodegradability. The organic ingredient should be readily biodegradable. The manufacturer must provide recycling and disposal instructions, which must comply with applicable local, state, and Federal regulations.

8. Cost Effectiveness. The replacement green solvent should be cost effective both in terms of procurement and waste disposal.

9. Ozone Depletion. The product should not contain any ozone depleting substances. An ozone depleting substance is any compound with ozone depletion potential greater than 0.01. For example, the ozone depletion potential of CFC 11 is 0.1. Note that this general guidance was adapted with some modifications while developing this PWTB with recommendations for solvents and methods to remove tar and asphalt from metal surfaces prior to painting and as part of general maintenance.

Vehicle-Specific Criteria

A survey of DoD facilities (Rhee et al. 1995) listed some desired general properties of cleaning solvents and provided specific guidance to identify cleaners for application to surfaces of tactical and transport vehicles. Those criteria are listed below.

1. The solvent must effectively remove tar and asphalt, and must be fast-drying (~20-30 min).

2. The solvent must have low VOCs.

3. The solvent must have low toxicity.

4. The solvent must have high flash point.

5. The solvent must have low flammability.

6. The solvent must be recyclable.

7. The solvent’s residues must be biodegradable and easily treatable along with regular wastewater streams.

8. The solvent must be compatible with the material(s) being cleaned. The use of the solvent should not lead to corrosion or erosion. If possible, the cleaner should provide a corrosion protection layer.

9. The cost of the solvent and the solvent requirement should be minimal.

Experimental Protocols for Laboratory Evaluation

A literature search of laboratory standard testing protocols for tar removal from metal surfaces was conducted on multiple databases.[2] The following section summarizes the most pertinent results. Tar removal experiments were designed and conducted using three selected commercial solvents, which were tested on metal coupons simulating the metal surfaces of military tactical and transport vehicles.

Literature Review

Kulkarni et al. (2003) found a variety of environmentally friendly and safe asphalt-removing solvents available in the market. However, they noted there is no quantitative standardized procedure to compare the efficacy of these solvents. Their goal was to develop a standardized procedure that would yield quantitative and repeatable results.

After evaluating various alternatives like metal and glass plates, ceramic tiles, and aluminum foils, the aluminum dish was found most suitable for the study. Test results obtained for solvent comparison were found to be consistent and repeatable, with the coefficient of variation for asphalt removed less than 10% for most solvents. Furthermore, this study provides an outline for cost-effective analysis of solvents used in relation to diesel fuel, and the procedure is able to rank solvents quantitatively.

Sacco (2004) has studied the blending of two plant-derived solvents to clean asphalt from trucks, shovels, and other equipment used to handle paving operations. One of the solvents was ethyl lactate, made from ethanol and lactic acid made by fermenting corn sugars. The other solvent was methyl soyate, a

[2] Databases included Scopus; Academic Search Premier [Ebsco]; Academic Onefile [Gale]; and Web of Science, including Social Sciences, Medicine, Humanities, and Engineering.

mixture of methyl esters of the fatty acids found in triglycerides from soybean oil. The new solvent, “Agri-Solve,” was found to clean without leaving a residue and proved to perform better than diesel fuel and several other solvents currently used.

Bryant and Cannon (1996) found that a substitute, 3% hydrogen peroxide (H_2O_2), effectively cleans tenacious residues off glass surfaces. They evaluated the solvent both at moderately elevated pH conditions and iron-based catalysts. Results revealed that 100% of an asphalt residue could be removed from glass surfaces within 105 min when it was submerged in a 3% H_2O_2 solution at pH 9.5 and ambient temperature. Furthermore, the asphalt residue could be completely removed within 45-60 min if the H_2O_2 solution also included 10^{-3} M ferric chloride ($FeCl_3$).

Lahib (2003) also found that 3% H_2O_2 in water effectively removed residues from glass surfaces. To simulate industrial cleaning conditions, asphalt was employed as a representative surrogate for tough-to-clean residues. Asphalt cleaning was dramatically enhanced by mild heating: whereas 3% H_2O_2 at pH 9.5 and 23 °C removed 100% of a fresh asphalt residue within 60 min., heating to 53 °C achieved full removal within 2 min. As asphalt became aged or dried by exposure to air, longer cleaning durations were required. Nevertheless, all of the asphalt could still be removed with 3% H_2O_2 at pH 9.5 and 70 °C within 2–60 min., even after the asphalt had dried onto glass for a week. H_2O_2 removed asphalt even when visible light was not present. When the H_2O_2 was excluded, a pH 9.5 bath at 70 °C removed only a small fraction of this asphalt, if any.

The IceMaster process (Kipp 2007) has penetrated many areas of industry where coatings must be gently removed from surfaces. In the IceMaster process a mixed stream of dry ice particles and compressed air is emitted from a nozzle on to the surface being cleaned. The strong refrigeration effect of the dry ice embrittles materials such as oils, waxes, greases, paints, and bitumens. The coating cracks and the dry ice particles convert to carbon dioxide gas and evaporate. The surfaces themselves being cleaned are not attacked or embrittled by the cold. Therefore, it is not necessary to remove seals and rubber parts when using IceMaster process. After cleaning, only coating residues must be removed. To supply the handheld IceMaster device, a carbon dioxide flask with feed pipe or tank and a high performance compressor are needed. The compressed air need is small at a rate of 0.75-8.00 m^3/min. (depending on facility size). IceMaster can run at 4.5 bar, is almost maintenance-free, and is simple to use.

Kopparal et al. (2005) developed a countercurrent continuous washing apparatus for tar removal under ultrasonic irradiation. Tar was dissolved in dimethylformamide (DMF) and sand was soaked into the resulting tar solution to prepare samples of tar-contaminated sand. Tar contents in DMF were determined by a UV-spectrophotometer from absorbance at 336.5 nm. The removal rate of tar content from this tar-contaminated sand was measured in two different conditions, one under the condition of mechanical stirring and the other with ultrasonically induced agitation. The removal rate was described in terms of a first order reaction equation, which makes it possible to calculate the residue fraction in continuous washing at a steady state. Comparison of tar removal with mechanical stirring and ultrasonically induced agitation has demonstrated that the ultrasound is more effective than the simple mechanical stirring. The basic mechanism of tar removal is, for both removal procedures, peeling of a tar-covered layer on the sand surface and the particles produced under the ultrasonic field are much finer than for the case of mechanical agitation.

Sheldon (2005) found that a non-toxic, non-hazardous, environmentally safe composition provides an effective, fast-acting cleaning solution for removal of tar, oils, asphalt and other bituminous materials from industrial equipment surfaces. The composition is a mixture of a carrier monocyclic monoterpene and a nonionic surfactant such as an alkylphenol ethoxylate. The mixture is applied directly to surfaces to be cleaned, and rinsed with water in the absence of mechanical intervention.

Zaki and Troxler (2005) found that water-soluble solvent compositions removed petroleum residue from a substrate. Compositions included:

- from about 10% to about 60% by weight of an aromatic ester
- from about 30% to about 60% by weight of an aliphatic ester
- from 0% to about 15% by weight of a co-solvent
- from 0% to about 20% of one of a cyclic terpene and a terpenoid
- from 0% to about 1% by weight of an odor-masking agent
- from 0% to about 20% by weight of a nonionic surfactant.

The composition can also include higher concentrations of water. Furthermore, the method for removing petroleum residue from a substrate can allow the solvent to be recycled using a countercurrent separation column charged with compressed ammonia and/or carbon dioxide and a spinning band distillation column to separate the solvent from the petroleum residue.

Discussion of Literature and Experimental Protocol

From the review of the literature it appears that the best performing solvents all have appreciable ability to dissolve asphalt and asphalt compounds. Both terpene based compounds and vegetable oil esters appear to be especially favored due to their perceived environmental friendliness. Commercial biobased formulations such as Citrus King, BioAct MSO, Citri-Kote, and Citrus Zip claim excellent performances for removing tar and asphalt on surfaces. The inclusion of surfactants appears to aid the process. This may explain the differences in effectiveness along with other compounding differences for the widely different cleaning efficacies of a number of terpene based cleaners (Kulkarni et al. 2003). It appears that dioctylsulfosuccinate could be particularly useful (based on Phieffer et al. 2003).

The use of H_2O_2 is intriguing (Brant and Cannon 1996), but the results were obtained on glass surfaces. More study is needed to see whether such an approach will work on metal surfaces.

The physical approach of cryogenic blasting may also be particularly useful as no chemicals are involved and such processes have a history of use within the DoD.

Only two of the above cited papers (Kulkarni et al. 2003; Brant and Canon 1996) are of direct relevance to adoption of an experimental protocol to evaluate solvent effectiveness for removal of asphalt. The protocols discussed by Kulkarni et al. (2003) can be summarized as follows.

Protocol 1

Protocol 1 includes the following steps:

1. Number each aluminum dish and determine its weight. The dishes used are FISHERBRAND™ Aluminum Weighing Dishes (Fisher Scientific, Pittsburgh, PA). The catalog number is 08-732 and the capacity of each dish is 42 mL.

2. Apply 1.5 g of emulsified asphalt (CRS-2) into the standard aluminum dish, ensuring that asphalt emulsion fully covers the bottom surface area of the dish.

3. Heat the aluminum dish, with asphalt emulsion, for 24 hours at the temperature of 140°F. (60°C).

4. Remove the dish after 24 hr and cool to room temperature. Determine the weight of the dish and calculate the weight of residual asphalt.

5. Apply 0.5 g of solvent into the dish by dropper. Make sure that the asphalt remains completely submerged in the solvent for 5 min.

6. Let the dish drain for 5 min by putting it upside down.

7. Rinse the dish thoroughly for 5 min under running water.

8. Heat the dish at 140°F (60°C) for 15 hr to remove the traces of water completely.

9. Weigh the dish to calculate asphalt removed.

Strengths and Weakness of Protocol 1

Protocol 1 is clearly defined, easily carried out, and allows quantitative comparisons of the different solvents. However, it suffers from the restriction of using fixed substrate (aluminum). This raises the possibility that the results obtained with this test may not be applicable to other surfaces, especially painted surfaces. Another drawback in this method is that it measures the relative effectiveness of the *dissolution* powers of the solvent alone. In normal practice, additional forms of energy input may be present from activities such as wiping or spraying. Finally, a water rinsing step is also employed in this protocol. As explained in Greenway (2000), this step simulates the practice among asphalt paving workers of applying a cleaning solvent to the truck beds followed by water rinsing to minimize residual solvent. Apparently, the accumulation of excess residue results in poor-quality asphalt by leaching binders from the mix. However, this consideration may not be relevant for the present application—cleaning vehicles prior to rebuilding.

Protocol 2

Protocol 2 includes the following steps:

1. Preparation of Test Strips.

   a. The assay uses test strips of stainless steel with dimensions 1.5 x 2.0 x 1/32 in. Immersions in solvents were carried out by placing the strips in clamps and immersing two thirds of the total area of the strip. This provides a total uniform area of exposure of 2.0 sq in. (The 1/32-in. thickness of the strip was disregarded.) The strips were desiccated and weighed with the clamp assembly so that the strip itself would not be handled.

b. The asphalt used in these experiments was a standard commercially available material containing latex polymers called CRS28, manufactured by Patterson Oil Company, Sullivan, MO. Upon procurement, each batch was cured by heating in a conventional laboratory oven for 7 days at 200 °F (93 °C).

c. A bath of the cured latex polymer-containing SuperPave asphalt was heated to 175 °F -180 °F (79.5 °C–82 °C). The strips were immersed in the molten asphalt to provide 2.0 sq in. of exposure. Exposure time was 2-3 sec. The strips were cooled to room temperature, desiccated for 24 hr, and weighed. Each data point is the arithmetic average of 10 strips treated identically.

2. Assay

The strips were immersed in the test solvents so that the entire asphalt coated areas were exposed to the solvent. The strips were withdrawn from the solution after 60 sec and drained for 2 min. They were again immersed for 60 sec and withdrawn. The strips were allowed to dry at room temperature for 2 hr and desiccated overnight. Desiccations were performed in an ordinary bell jar in the presence of a standard commercial desiccant. The test strips were then reweighed. The data expressed in percentage by weight of removal was calculated by subtracting the weight of the treated strip from the weight of the untreated strip and dividing by the weight of the untreated strip.

Strengths and Weakness of Protocol 2

Protocol 2 is also a clearly defined protocol that allows replications and quantitative evaluations. The coating of the strips by immersion may lead to variations in the individual weight, but this variation can be minimized by simultaneous dip coating, temperature control, and withdrawal. The effect of such variations can also be accounted for by normalizing the residual amount with respect to the initial coat weight. This protocol also follows a more rigorous and realistic aging of the asphalt contaminants that are likely to adhere to military vehicles. Finally, the protocol allows flexibility in the choice of coupons. One drawback in this method is that it measures the relative effectiveness of the *dissolution* powers of the solvent alone. In normal practice, additional forms of energy input may be present from activities such as wiping or spraying. While this protocol does not explicitly include a water rinsing step, the source literature clearly indicates that such a step is usually carried out.

Experimental Procedure

Based on the literature review of the protocols presented in the previous section, this experimental study followed the modified protocol described below. These experiments were designed to facilitate ranking the performance of the cleaners. The cleaner evaluation involves estimating the solubility/dissolution power of the solvents, and cleaning ability using coupon studies. The coupon studies at different temperatures and times will provide information if any residues that remain on the surface and practical details useful for developing cleaning process. Experimental evaluation also includes cleaning complex parts such as chains obtained from the bitumen spreaders.

Materials

CRS-2 asphalt and stainless steel 2 x 3.5 x 0.02-in. test panels (#Q-D-46) were obtained from Emulsicoat, Inc. (Urbana, IL) and Q-Lab Corporation (Cleveland, OH), respectively. Bio T Max was acquired from Biochem Systems (Woodland, TX). BioAct 105, BioAct 120, BioAct 121, and BioAct MSO were all obtained from Petroferm Inc. (Gurnee, IL). Delta Foremost Chemical Corporation (Memphis, TN) supplied both Citri-Zip Asphalt Remover and Citri-Kote. DS-104 was acquired from Dysol (Fort Worth, TX). Full Force, Rid-O-Grease, and Tuff Stuff were obtained from Continental Research Corporation (St. Louis, MO). Orange Products LLC (McLeansville, NC) supplied Orange Oily Slicker Asphalt Remover and Release Agent as well as Citrus Soy Gold. Citrus King Asphalt Remover was acquired from Citrus Depot (St. Petersburg, FL). The isopropanol was purchased from Acros Organics (Thermo Fisher Scientific of Fairlawn, NJ).

Asphalt Solubility in Vials

Glass vials of about 10 mL volume were cleaned using isopropanol and then dried in an oven at 60 °C for 1 hr. The vials were then cooled to room temperature before being labeled and recording the weight. A pipette was used to transfer approximately 5 mL of asphalt into the vial. The asphalt was cured in a 60 °C oven overnight and then cooled to room temperature before weighing. There were 5 mL of each solvent added to marked vials, and the solvents were mixed using an oscillator for 15 min (See Appendix C: Figure C-1). The solvents were drained from the vials, after which they were allowed to drip dry (Figure C-2) for 30 min, and then put into the oven at 60 °C for 2 hr to evaporate the remaining solvent and re-solidify the remaining asphalt. The samples were cooled to room temperature and weighed.

Asphalt Removal on Metal Coupons

The 2 x 3.5 x 0.02-in. test coupons were cleaned with isopropanol and then dried in an oven at 60 °C for 1 hr. The coupons were cooled to room temperature before recording the weight. This procedure was also used for the control coupon. The ASTM G122-98: Method B (nonvolatile residue (NVR) sample, double side) was used to apply the contaminant, CRS-2 asphalt. The coated coupon was baked in the oven at 60 °C for 1 hr. After allowing the coupon to cool to room temperature, the test strip was weighed and then placed in a desiccator.

The cleaners were diluted according to the manufactures recommendations. The test coupons were immersed in solvent such that the entire asphalt coated area was exposed to the solvent. The coupons were removed from the cleaner after 60 sec and allowed to drip for 2 min. This was repeated 2 more times for a total of 3 solvent rinses. Afterward the coupons were rinsed with water and allowed to dry at room temperature over night. The spray solvents were held 6-8 in. away while applying. After applying over the entire contaminated area, the coupons were allowed to drip for 2 min. This was also repeated 2 more times for a total of 3 solvent applications, and then rinsed with water and allowed to dry overnight at room temperature. Weight of the dried coupons was acquired, and the cleaning effectiveness factor (CEF) was calculated using the following equation:

$$CEF = \frac{MX2 - MX3}{MX2 - MX1}$$

where:

MX2 is the weight of the coupon with the asphalt
MX1 is the weight of the coupon
MX3 is the weight of the coupon plus any residual asphalt after the solvent has been applied (See Appendix C: Figure C-3 through Figure C-14).

Asphalt Removal on Chains at Room Temperature

Chains covered in asphalt were acquired from bitumen spreaders that were waiting to be cleaned at Red River Army Depot, Texarkana, TX. These chains were originally at a length of approximately 1 ft. These were cut down to 3-in. pieces, which were more suitable for the test procedure. About 400 mL containers were used to hold 300 mL of one of the five solvents chosen for this experiment. The solvents Citri-Zip, Citrus Soy Gold, BioAct MSO, Bio T Max, and Citrus King were chosen based on how much asphalt was removed during the vial tests. The chains were weighed with a paperclip that was used to suspend the chain in the solvent. The chains were then immersed in the solvent with constant agitation using a magnetic stir bar. After 30 min, the chains were taken out and washed with water to obtain the amount of asphalt remaining on the chain. This was repeated every 30 min for a total of 2 hr. The chains were allowed to air dry overnight and were then re-weighed.

Asphalt Removal on Chains at 90 °F

Five additional 3-in. asphalt-covered chain pieces were weighed with paper clips. The solvents Citri-Zip, Citrus Soy Gold, BioAct MSO, Bio T Max, and Citrus King were again chosen for this experiment. Exactly 300 mL of solvent was placed in a 400 mL container, which was then placed in beaker filled with water acting as a water bath to keep a consistent temperature. A Corning Stirrer/Hot Plate and a Cole Parmer Stirrer/Hot Plate were used both as heating elements and as sources of agitation for the solvent. The solvent and water bath were heated to 90±5 °F for 30 min before introducing the chain to the solvent so that the solvent would reach equilibrium with the water at the desired temperature. After 30 min of having the chain in the solvent, the chain was taken out and washed. This was repeated every 30 min over a course of 2 hr. The chains were allowed to air dry overnight and then were weighed again with their respective paperclips (See Appendix C, Figure C-15 and Figure C-16).

Evaluation of Solvents

From the vial test, it was possible to narrow down solvents that would perform best based on how much asphalt was removed. Based on the percentage of asphalt dissolved, the data in Table A-2 indicated that the top 10 solvents are: Citri-Zip, BioAct 121, Citrus Soy Gold, Bio T Max, BioAct 105, Citrus King, BioAct MSO, BioAct 120, Full Force, and Tuff Stuff.

Table A-2. Removal of Asphalt from Vials.

Solvent	Bottle Wt. (g)	Bottle Wt. with Tar	Tar Wt. (g)	Bottle Wt. after 5 mL Solvent (g)	Amount Tar Removed (g)	% Tar Removed
Tuff Stuff	12.79	17.66	4.87	17.55	0.11	2.26%
Rid-O-Grease	12.69	17.29	4.60	17.20	0.09	1.96%
Full Force	12.70	17.33	4.63	17.19	0.14	3.02%
Citri-Zip	12.64	17.13	4.49	16.95	0.18	4.01%
DS-104	12.60	17.72	5.12	17.61	0.11	2.15%
BioAct 105	12.75	17.56	4.81	17.40	0.16	3.33%
BioAct 120	12.61	16.88	4.27	16.75	0.13	3.04%
BioAct 121	12.16	16.94	4.78	16.75	0.19	3.97%
BioAct MSO	12.20	17.30	5.10	17.14	0.16	3.14%
Bio T Max	12.61	17.52	4.91	17.35	0.17	3.46%
Orange Oil Slicker	12.68	17.56	4.88	17.45	0.11	2.25%
Citrus King	12.16	17.23	5.07	17.07	0.16	3.16%
Citrus Soy Gold	12.64	16.60	3.96	16.45	0.15	3.79%
Citri-Kote	12.69	17.01	4.32	16.93	0.08	1.85%

After reviewing the MSDS and product sheets of each of the top solvents along with the cost per gallon shown in Table A-3, five solvents were selected to undertake the next experiment, asphalt removal from chains.

Table A-3. Cost of Solvents.

Solvent Name	Cost per gallon*
Tuff Stuff	$166.00**
Rid-O-Grease	$56.00
Full Force	$39.00
Citri-Zip	$58.85
DS-104	$30.82
BioAct 105	$16.92
BioAct 120	$24.82
BioAct 121	$22.40
BioAct MSO	$22.18
Bio T Max	$25.65
Orange Oil Slicker	$26.91
Citrus King	$39.00
Citrus Soy Gold	$28.73
Citri-Kote	$36.50
*Price per gallon based on a 55-gal drum. ** Only sold in packs of a dozen aerosol cans. Net weight 15 oz per can. Costs were obtained during June-July 2010.	

Cleaners Chosen for Detailed Study

Based on the tar removal and cost information shown in Tables A-2 and A-3, five solvents were selected for further evaluation.

The cleaners chosen for detailed study were Citri-Zip, BioAct MSO, Citrus Soy Gold, Citrus King, and Bio T Max. Time of immersion and temperature of the solvent were chosen during this experiment using the experimental setup shown in Figure C-7 and Figure C-11 (Appendix C). A comparison of Table A-4 and Table A-5 reveals that the optimum temperature is at 90°°F, which generally cuts the immersion time by at least 60 min. It also shows that the maximum time that every solvent (except citrus soy gold) needs to work is 60 min. Therefore, the recommended time of immersion is between 30 and 60 min. Note that Figure C-15 and Figure C-16 show significant corrosion of the chain. Unfortunately, it could not be determined whether this corrosion occurred before the asphalt was accumulated, if it was caused by the solvents, or if the metal rusted during the period of time the asphalt was on the chain. Nevertheless, the metal coupon tests helped narrow down the cleaner selection process for further testing. Based on these results, and on cost, the three solvents chosen for field-testing were: Bio T Max, BioAct MSO and Citrus King.

Table A-4. Removal of asphalt from chains at 78 °F.

Solvent Name	Weight	Time submerged (min)	Amount of Solvent (mL)	Weight After	Tar Removed
Citri – Zip	54.31	120	300	47.49	6.82
Bio T Max	50.84	120	300	46.62	4.22
Citrus King	54.04	120	300	47.79	6.25
BioAct MSO	54.25	120	300	47.5	6.75
Citrus Soy Gold	54.35	120	300	47.97	6.38

Table A-5. Removal of asphalt from chains at 90 °F.

Solvent Name	Weight	Time submerged (min)	Amount of Solvent (mL)	Weight After	Tar Removed
Citri – Zip	54.95	60	300	48.86	6.09
Bio T Max	60.27	60	300	55.66	4.61
Citrus King	55.02	60	300	48.48	6.54
BioAct MSO	50.6	60	300	47.25	3.35
Citrus Soy Gold	51.31	120	300	47.75	3.56

Conclusions and Recommendations

This PWTB presented a simple approach to evaluate commercial tar/asphalt cleaners. Several ASTM test methods are also available for evaluation of grease and oil cleaner performance. The simple laboratory experiments described in this PWTB can be used to evaluate tar and asphalt cleaners.

This work also identified two broad categories of solvent blends that can remove asphalt from metal: (1) terpene-based solvents/esters, and (2) blends of aliphatic hydrocarbons and esters assisted by surfactants. Of the solvents reviewed, the five solvents listed in Table A-5 performed well in the laboratory. It is recommended that any of these five cleaners may be used for removing the tar/asphalt from surfaces:

1. Citri – Zip
2. Bio T Max
3. Citrus King
4. BioAct MSO
5. Citrus Soy Gold

Of these five tested solvents, Citrus King appears to combine both functionality and desirable environmental characteristics. The actual selection, however, would be based on the specific application (such as removing tar from a smooth surface or a part with complex geometry such as a chain) and mode of application (flexibility to heat the part, immersion cleaning or spray cleaning, and the time that could be allocated for cleaning).

Note that the feasibility of using these solvents for routine large scale cleaning must be demonstrated in the overall framework of economics, environment, and health. New cleaners are continuously being developed by commercial vendors. Follow-on studies shall be conducted within a constraining set of environmental and health criteria and a price point specific to field conditions. Nevertheless, the information in this PWTB should be sufficient to give operators enough information to select a cleaner for surface spot cleaning or immersion cleaning for tar and asphalt removal based on operational requirements, negotiated cost of solvents, and waste disposal requirements.

Appendix B

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Appendix C

PHOTOGRAPHS OF LABORATORY EXPERIMENTS

Vial Experiments

Figure C-1. Vials with tar and solvent on rotating shaker.

Figure C-2. Vials drained after shaking.

Coupon Experiments

Figure C-3. Asphalt-coated coupon.

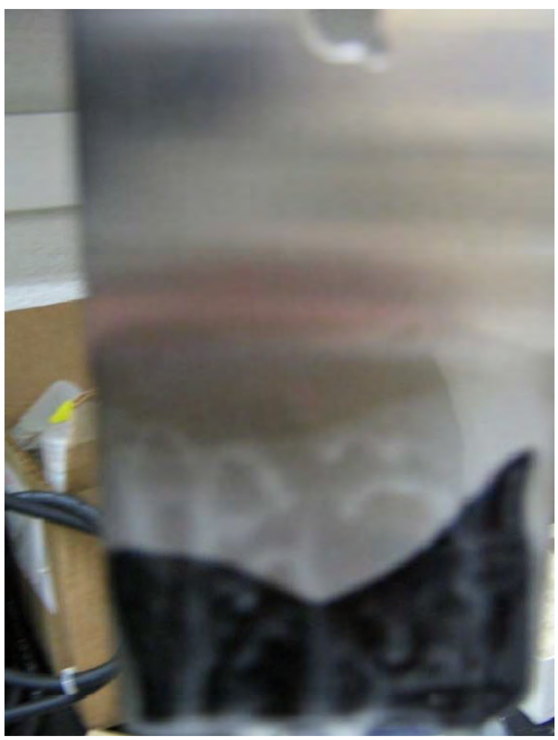

Figure C-4. Asphalt-coated coupon submerged in 400 mL Citrus King (15 min at 30-35°C).

Figure C-5. Asphalt-coated coupon submerged in 400 mL Citrus King (30 min at 30-35 °C).

Figure C-6. Asphalt-coated coupon submerged in 400 mL Citrus King (45 min at 30-35 °C).

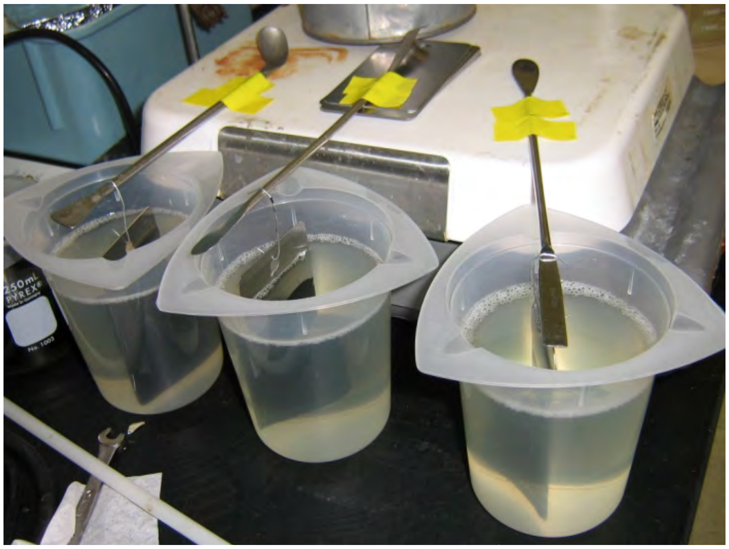

Figure C-7. Solvent Simple Green set-up at room temperature.

Figure C-8. Asphalt-coated coupon submerged in Simple Green for 30 min at room temperature.

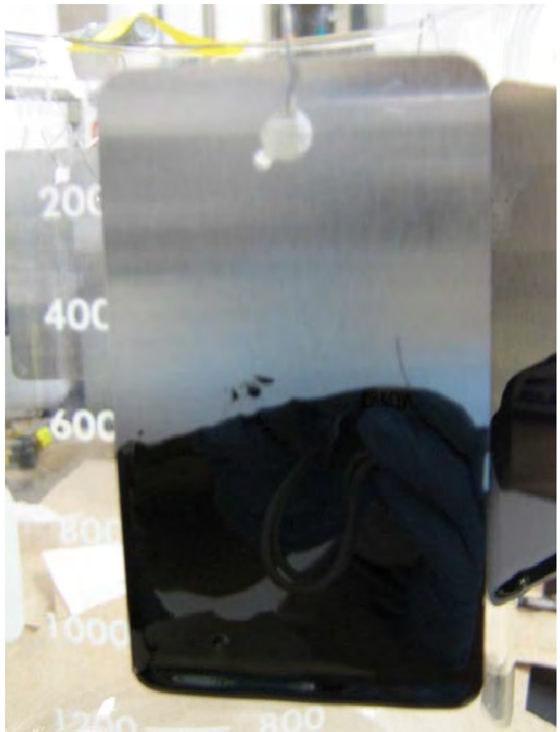

Figure C-9. Asphalt-coated coupon submerged in Simple Green for 60 min at room temperature.

Figure C-10. Asphalt-coated coupon submerged in Simple Green for 90 min at room temperature.

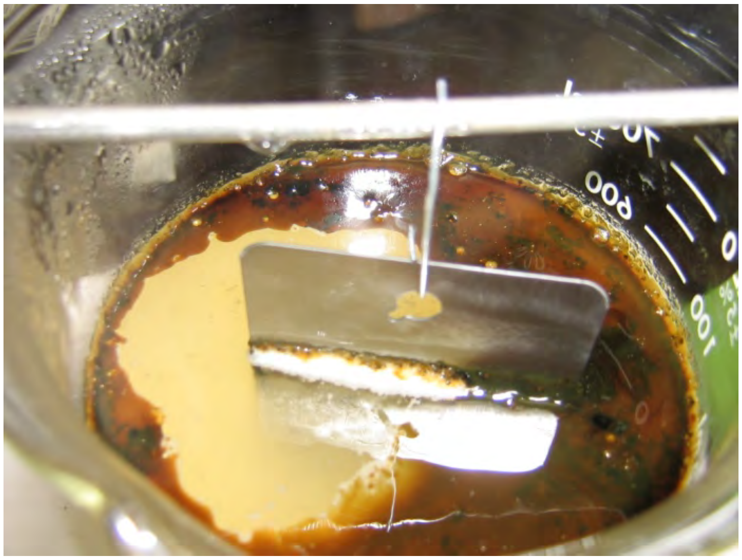

Figure C-11. Solvent Simple Green set-up at 80 °C.

Figure C-12. Asphalt-coated coupon submerged in Simple Green at 80 °C for 30 min.

Figure C-13. Asphalt-coated coupon submerged in Simple Green at 80 °C for 60 min.

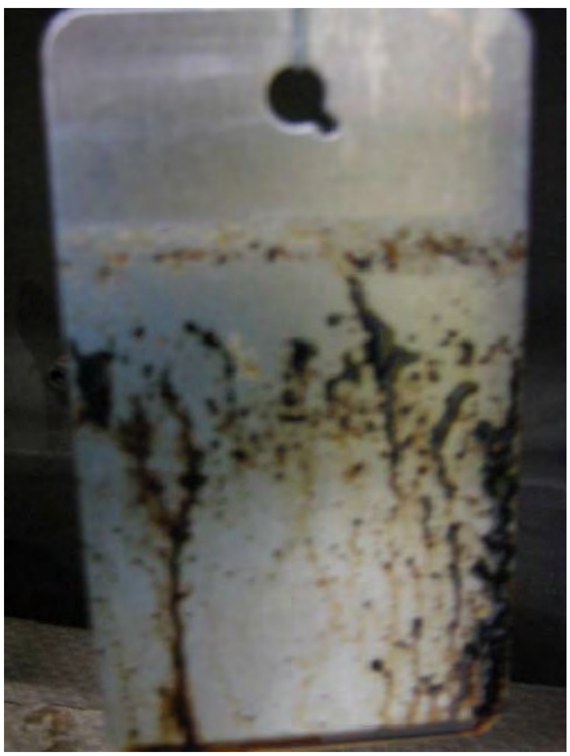

Figure C-14. Asphalt-coated coupon submerged in Simple Green at 80 °C for 90 min.

Chain Experiments

Figure C-15. Dirty chains before immersion in Citri-zip solvent.

Figure C-16. Chain after immersion in Citri-Zip for 90 min and after rinse.

Appendix D

ABBREVIATIONS

Abbreviation	**Spelled Out**
AR	Army Regulation
CECW	Directorate of Civil Works, U. S. Army Corps of Engineers
CEF	cleaning effectiveness factor
CEMP	Directorate of Military Programs, U. S. Army Corps of Engineers
CERL	Construction Engineering Research Laboratory
CFC	Chlorofluorocarbon
CFR	Code of the Federal Regulations
DA	Department of the Army
DMF	dimethylformamide
DPW	Directorate of Public Works
DoD	Department of Defense
ERDC	Engineer Research and Development Center
H_2O_2	hydrogen peroxide
HAP	hazardous air pollutant
HQUSACE	Headquarters, U.S. Army Corps of Engineers
HSWA	Hazardous and Solid Waste Amendments
MSDS	Material Safety Data Sheet
NVR	non-volatile residue
PL	public law
POC	point of contact
PWTB	Public Works Technical Bulletin
RCRA	Resource Conservation and Recovery Act
TARDEC	Tank Automotive Research, Development, and Engineering Center
USACE	US Army Corps of Engineers
USEPA	US Environmental Protection Agency
VOC	volatile organic compound
WBDG	Whole Building Design Guide
WMPP	Waste Minimization and Polution Prevention