SAND2013-6390C

Sandia National Laboratories

Exceptional service in the national interest

Critical Cleaning Agents for Di-2-ethylhexyl Sebacate

Mya Hartley and Kim Archuleta

Abstract: It is required that Di-2-ethylhexyl Sebacate oil, also commonly known as Dioctyl Sebacate oil, be thoroughly removed from certain metals, in this case stainless steel parts with narrow, enclosed spaces. Dioctyl Sebacate oil is a synthetic oil with a low compressibility ideally used for high pressure calibrations. The current method to remove the Dioctyl Sebacate from stainless steel parts with narrow, enclosed spaces is a labor-intensive, multi-step process, including a detergent clean, a deionized (DI) water rinse, and several solvent rinses, to achieve a nonvolatile residue (NVR) of ≤ 0.04 mg per 50 mL rinse effluent. This study was done to determine a superior detergent/solvent cleaning method for the oil to reduce cleaning time and/or the amount of detergent/solvent used.

Figure 1. Skeletal formula for Dioctyl Sebacate oil.

Approach: For this study, solvents and detergents were chosen based on detergent/solvent availability, human hazard, and their ability to remove the soil of interest, Dioctyl Sebacate oil, based on predictions using ***Hansen*** solubility parameters. Solvents with similar ***Hildebrand*** solubility parameter values to that of the Dioctyl Sebacate oil were chosen for Hansen solubility comparison analysis.

Hildebrand solubility parameter:

$$\delta = \sqrt{c} = [\frac{\Delta H - RT}{V_m}]^{1/2}$$

c - cohesive energy density
H - heat of vaporization
R - gas constant
T - temperature
V_m - molar volume

Hansen solubility parameters (3):

δ_d - dispersion force
δ_p - polarity
δ_h - hydrogen bonding

Hansen/Hildebrand correlation:

$$\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$$

R_o, shown in Figure 2, is a given solvent's radius of interaction. R_A, not shown in Figure 2, is another radial dimension, found empirically, that takes into account a solvent's distance from another solvent in 3-dimensional Hansen space.

$$R_A = \sqrt{4 \cdot (\delta_{d,2} - \delta_{d,1})^2 + (\delta_{p,2} - \delta_{p,1})^2 + (\delta_{h,2} - \delta_{h,1})^2}$$

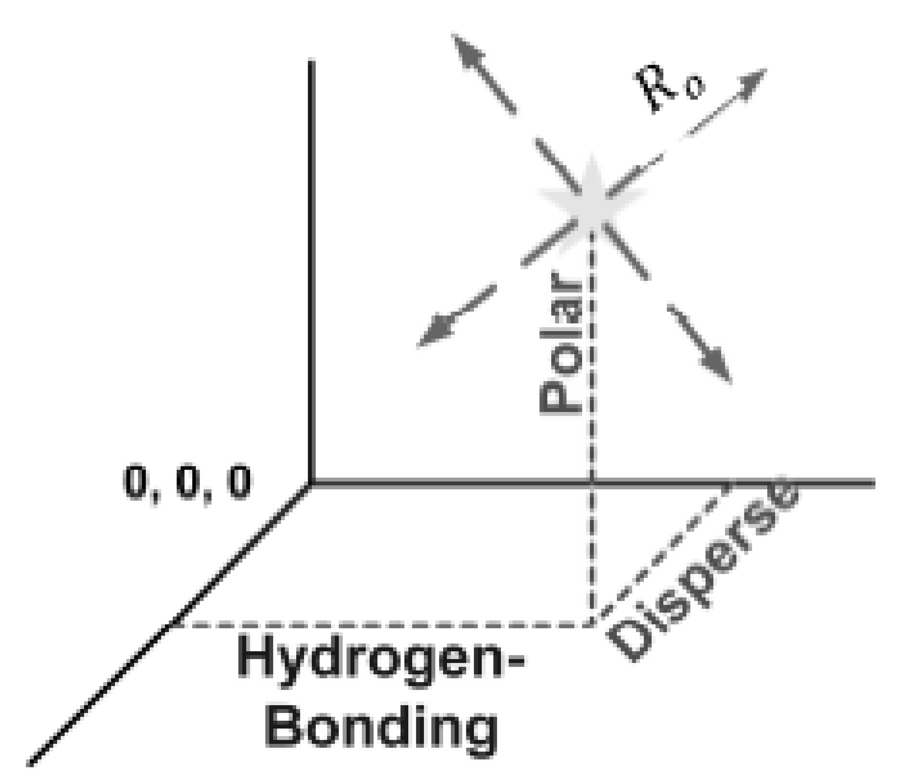

Figure 2. Hansen solubility parameters (3) of a particular solvent in 3-dimensional space; where R_o is a radius of interaction.

RED - <u>R</u>elative <u>E</u>nergy <u>D</u>ifference:

$$RED = {R_A}/{R_o}$$

$0 < RED \ll 1$ – High Affinity
$RED \approx 1$ – Boundary Condition
$RED > 1$ – Decreased Affinity
As RED increases, affinity decreases

- Reagents with similar Hildebrand values to the Dioctyl Sebacate and common laboratory reagents were chosen and R_A-values were computed between each reagent and the Dioctyl Sebacate oil.
- Pairs with smaller R_A-values are predicted to be more compatible and the paired solvent more efficient.

Reagents chosen for testing, predicted from relative "best" to "worst" solvent for the Sebacate oil found by the computed R_A-values:

Best
Diethyl Ether
N-Methyl-2-pyrrolidone (NMP)
D-Limonene
Hexane
Ethanol
Worst

Reagents chosen for testing, Hansen and Hildebrand solubility parameters unknown:

Synergy 3000 CCS™

Brulin 815GD™*

*Brulin 815GD™ is a detergent

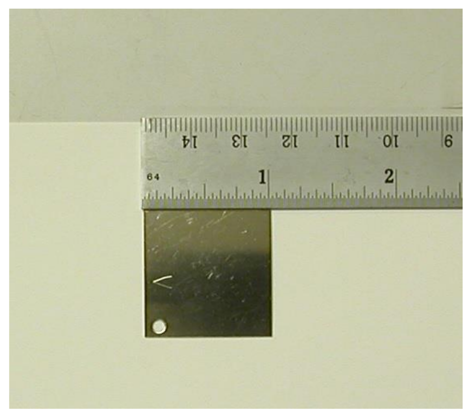

Figure 3. 1"x1" 304 SS Coupons utilized for testing.

Experimental Method: 1"x1" stainless steel (304SS) coupons were coated in the Sebacate oil at ambient temperature. In a class 1000 clean room, each cleaner was tested by applying varying cleaning/soak times (5 and 10 minutes) to each coupon and then a non-volatile residue (NVR)test was performed on each of cleaned coupons. Brulin was tested at two volume concentrations (5 and 15%) and both Brulin and NMP were tested at two different temperatures as well.

Results:

Agent	Temperature (°C)	Concentration (vol%)	Time (min)	Coupon #	BP (°C)	Rinse Agent	NVR (mg)
diethyl ether	25 (ambient)	100	5	21	34.6	Ethanol	0.00
			10	22			0.00
NMP			5	15	202-204	Water	0.02
			10	17			0.01
	60		5	16			0.00
			10	18			0.00
d-Limonene	50		5	13	176/178	IPA	0.02
			10	14			0.01
Hexane	25 (ambient)		5	19	69		0.01
			10	20			0.00
Ethanol			5	10	78.37	Ethanol	0.01
			10	12			0.01
Brulin	60	15	5	5	100		0.05
			10	7			0.06
		5	5	1			0.04
			10	3			0.05
	80	15	5	6			0.05
			10	8			0.06
		5	5	2			0.06
			10	4			0.02
Synergy CCS	25 (ambient)	100	5	9	assume near IPA	IPA/Water	0.04
			10	11			0.00

Table 1. Cleaning agents' variables tested and NVR results for each coupon.

Recommendations: The data indicates that diethyl ether, heated NMP, hexane, and ethanol are the best choices of the cleaners tested. Consideration of human hazard and other factors, hexane and ethanol were chosen as the best solvent suitable to remove the oil. Synergy CCS may also be a good candidate, though more tests need to be undertaken to confirm a longer time period would be required. Ambient temperature NMP, d-Limonene, and Brulin are only fair candidates. Brulin-cleaning may be taken out of the cleaning regimen with negligible effect. It should be noted that heating the metal parts is likely to reduce cleaning time as heating the oil will make the oil less viscous and easier to remove. Future work would be required to evaluate this.

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.
SAND No. 2011-XXXXP