

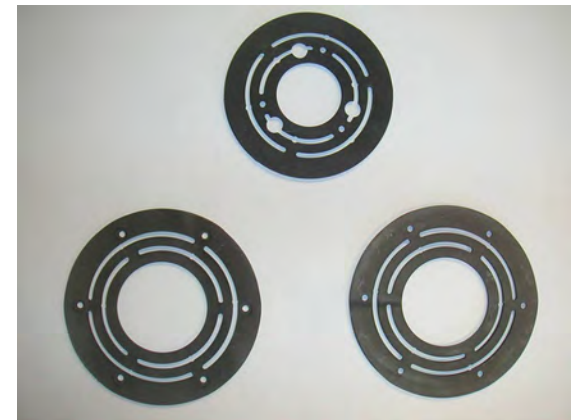


Laboratory Performance Testing & Implementation

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Samples from Clients

- Contaminant being removed
 - Request 200-500ml
 - Identify hardest contaminant to remove first
 - Check other contaminants after for effectiveness
- Parts
 - Three parts for each identified potential alternative per test
 - Start with coupons first to identify cleaners that are effective first



Developing a Test Plan

- Things to consider:
 - Cleaning method
 - Current equipment vs new equipment considerations
 - Vapor Degreaser → Ultrasonics
 - Rinse and dry step?
 - Chemistry compatibility with substrate/equipment
 - Vendor information
 - Non-client testing
 - Time
 - Refer back to Test Request Form

Initial Testing: Immersion Cleaning

- Pick 8-10 cleaners
 - CleanerSolutions Database or Online Search
 - EHS Evaluation using P2OASys
- Use Coupons
 - Use substrates that are the same as parts
- Immersion Testing
 - Evaluating the chemistry performance
 - Temperature
 - Concentration
 - Time
 - Doesn't work only or only showed partial removal?
 - Tweak parameters that seem appropriate



Next Step: Incorporating Equipment

- Pick 6-8 cleaners that worked best
- Choose appropriate equipment or method for the process:
 - Ultrasonics
 - Lab uses 40 kHz Branson 3800 & 80 kHz FB11201
 - Immersion Tank
 - Spray Wash
(High and Low Pressure)
 - Vapor Degreasing
 - Manual Wipe
 - Vacuum Cycling Nucleation (VCN)



Vacuum Cycling Nucleation (VCN)

- New equipment the lab is researching
 - Aqueous specific
 - RIT in NY has solvent version
 - Great for tiny orifices, piping, and very complex geometries
 - Trying out a variety of contaminants and parts
 - Compare to other equipment currently out there



Aqueous Vacuum Cycling Nucleation (VCN) System

	Ultrasonics	VCN
Internal Cleaning	No	Yes
Damage to Delicate Parts	Yes	No
Load Dependent	Yes	No
Uniform Treatment	No	Yes
Targets Contaminant	No	Yes
Chemical Delivery to Surface	No	Yes
Fluid Boundary Layer	Yes	No
Effectiveness With Solvents	Low	High
Concentrations Required	Hi	Low
Effective Rinse Method	No	Yes
Temperature Dependence	Hi	Low

<http://vacuumprocessingsystems.net/>



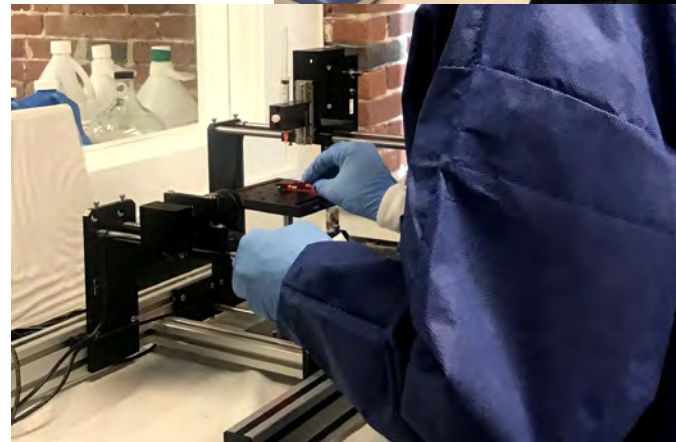
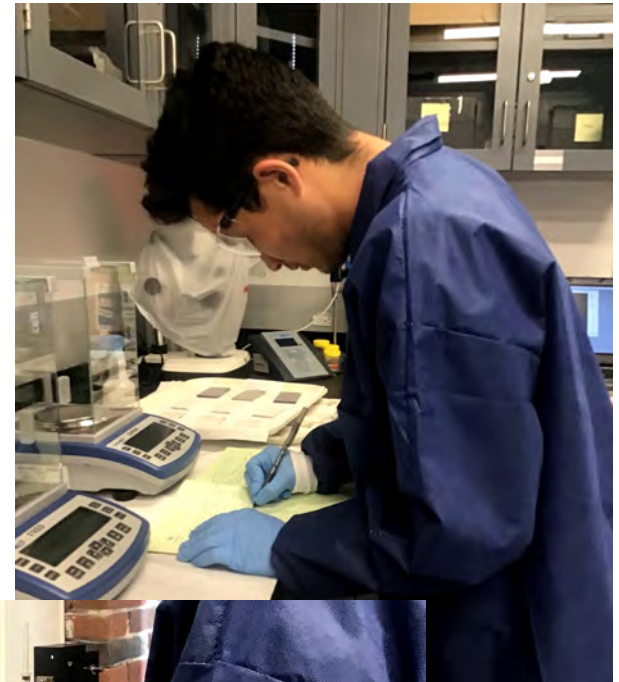
Testing with Parts

- Pick 3-4 cleaners that worked best
- Test on contaminated parts
 - Use temperature and concentration identified
 - Use equipment identified
- Compare to provided clean parts as a baseline of cleanliness

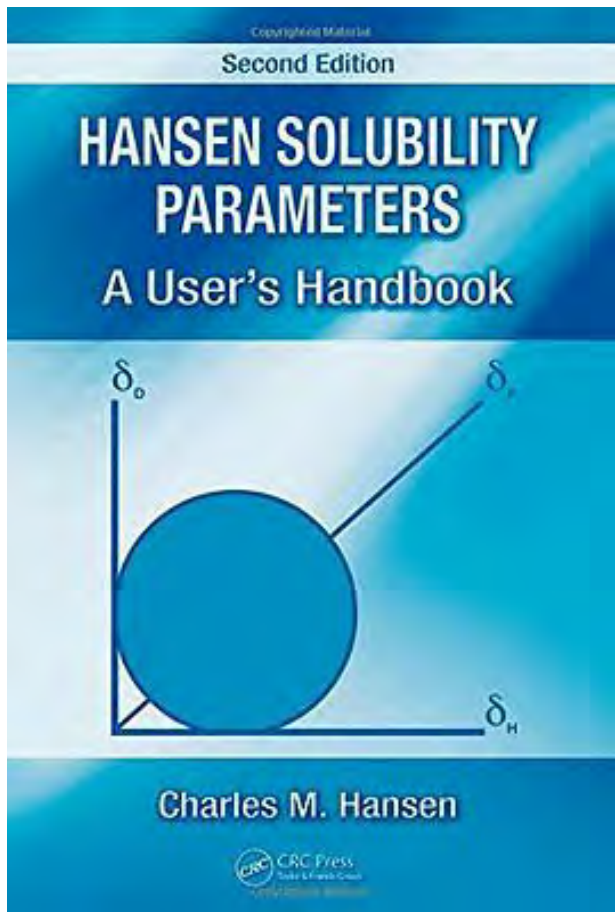


Determining cleanliness

- Most companies only need visual verification
- Quantitative data for a company's customers:
 - Gravimetric
 - Contact angle
- May need more verification testing
 - Military standards



So None of the Market Cleaners
Worked... Now What?

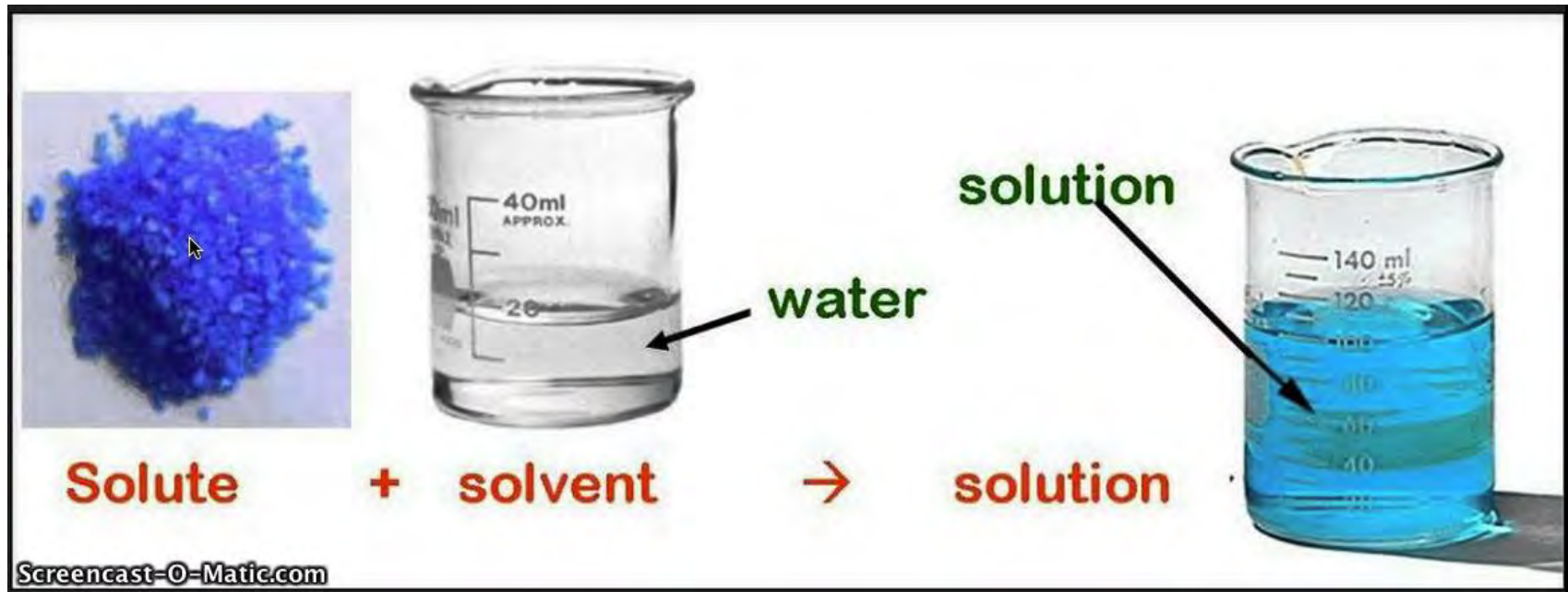


Hansen Solubility Parameters Theory

<https://hansen-solubility.com/>

Solute, Solvent, and a Solution

Solute + Solvent = Solution



- The solid solute is the substance being dissolved (e.g. a polymer)
- The liquid solvent dissolves the solute. Both substances must be similar to dissolve
- When the solute is dissolved a solution is created

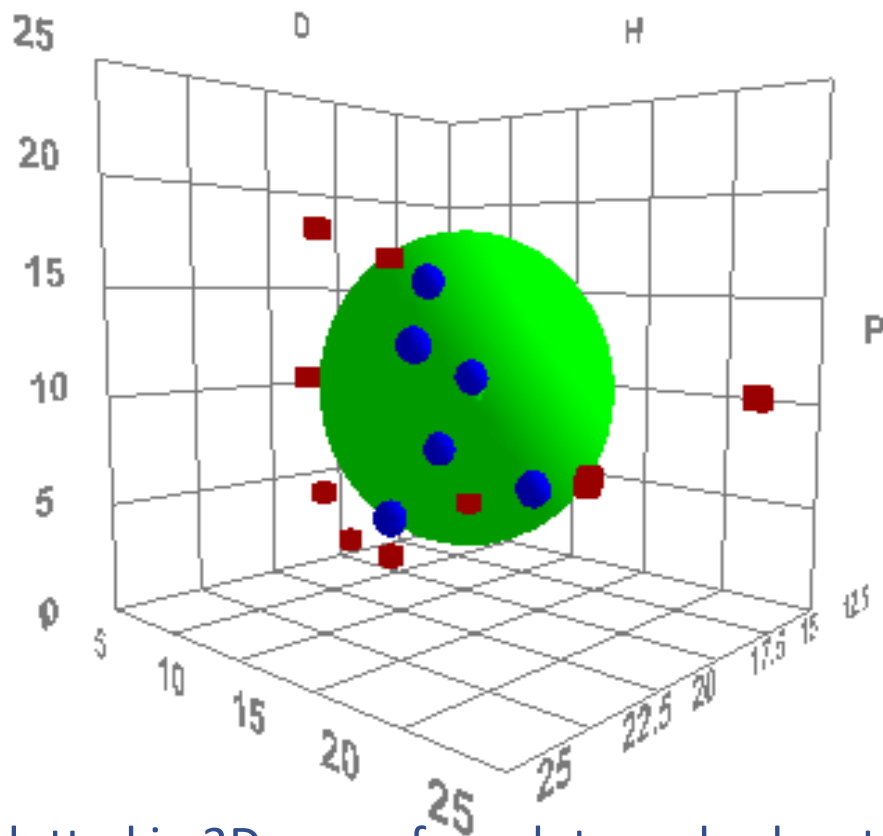
Solubility

- Hansen Solubility Parameters (HSP) theory is based on the relationship between energies of the solute and solvent
- “Like Dissolves Like” is the fundamental concept

HSP and Inter-molecular Forces

1. Dispersion force (also called London Force)
2. Polar force (also called dipole-dipole force)
3. Hydrogen bonding force
4. Ionic Force (not included in HSP theory – mostly used for aqueous applications)

Strength of forces: hydrogen bonding > polar > dispersion



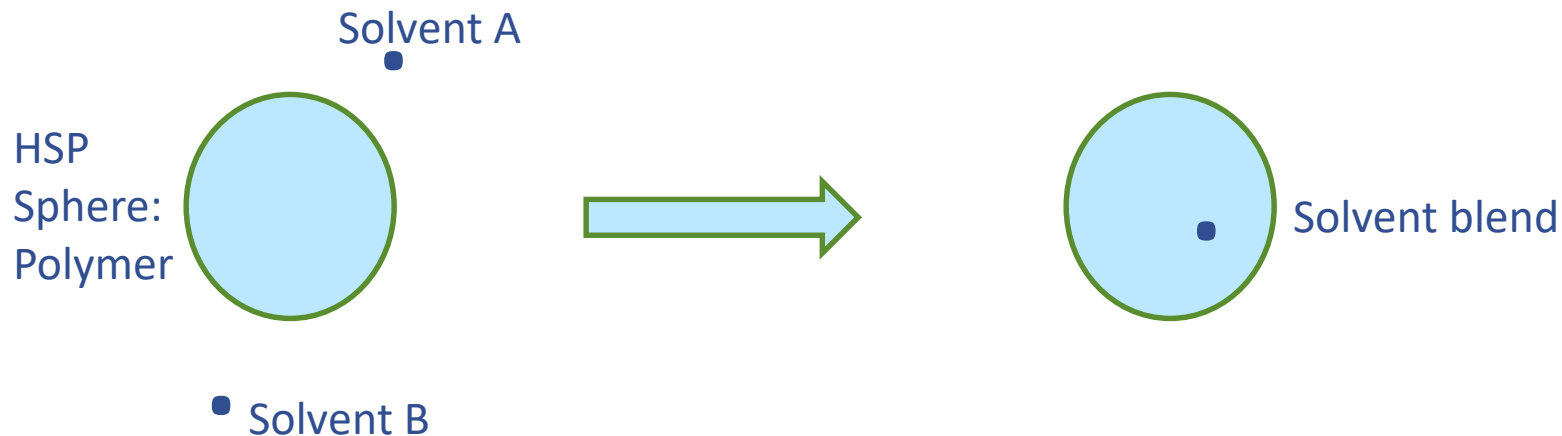
δD , δP , and δH are plotted in 3D space for solute and solvents.

Around the solute center point is a Green sphere of solubility, with a unique radius.

- Blue Dots: solvents that will dissolve the solute (inside or on surface of the green sphere)
- Red Cubes: solvents that will not dissolve the solute (outside of the green sphere)

Solvent Blends - Example

Solvent	Volume %	D	P	H
Solvent A	100	12	4	21
Solvent B	100	18	11	3
Solvent Blend	50:50	15	7.5	12



Factors that Affect HSP Performance

- Molar Volume (or Molecular Weight Effect)
 - Important when dealing with polymers
- Temperature Effect
 - Working on refining process for identifying alternatives for drop-in alternatives for vapor degreasing applications
- Viscosity Effect
 - Can act as a brake and reduce ability to mix
- Time Effect
 - Transport properties can impact rate of equilibrium
 - Affect heat, mass, and momentum transfer

The HSPiP Solvent Test

- Come up with a test plan
 - Review Test Request Form
 - Consider how chemical is being used and replicate
- Created a set of 24-30 solvents to develop sphere from scores
 - Currently working on a list of safer solvents/solvent blends to replace current set of solvents for test



Hansen Solubility Parameters in Practice (HSPiP) Software

File Dist. Diff. Adh./Visc. ForceFit Teas HPLC IGC GC Temp. Evap. FindMols Grid DPC SMILES Help HSPiP Master Data

No.	Solvent	δD	δP	δH	Score	RED	MVol	CAS	SMILES
1	Acetaldehyde	14.7	12.5	7.9			56.5	75-07-0	CC([H])=O
2	Acetaldoxime	16.3	4	20.2			61.2	107-29-9	C/C=N/O
3	Acetamide	17.3	18.7	19.3			59	60-35-5	CC(N)=O
4	Acetanilide	20.6	14.4	13.5			131	103-84-4	CC(=O)N...
5	Acetic Acid	14.5	8	13.5			57.6	64-19-7	CC(O)=O
6	Acetic Anhydride	16	11.7	10.2			95	108-24-7	CC(OC(C)...
7	Acetone	15.5	10.4	7			73.8	67-64-1	CC(C)=O
8	Acetonecyanhydrin	16.6	12.2	15.5			91.8	75-86-5	CC(C)(O)C...
9	Acetonemethyloxime	14.7	4.6	4.6			96.7	3376-35-0	CC(=NOC)C
10	Acetonitrile	15.3	18	6.1			52.9	75-05-8	CC#N
11	Acetophenone	18.8	9	4			117.4	98-86-2	CC(C1=C...
12	Acetoxime	16.3	3.7	10.9			80.2	127-06-0	CC(=NO)C
765	1-Acetoxy-1,3-Butadiene	16.1	4.4	8.3			118.4	1515-76-0	CC(=O)O...
1116	Acetyl Salicylic Acid	19	6.9	9.7			149.4	50-78-2	CC(=O)O...
785	2-Acetyl Thiophene	19.1	12.2	9.3			108	88-15-3	CC(C1=C...

Donor/Acceptor Genetic Algorithm Hide Unused
 MVol Correction Show Selected Save as hsd
 Sphere Rad. Chk ESC Alert DC-Rad 4.0 Limit

Search_Text: Font Size: Medium Info: Both only

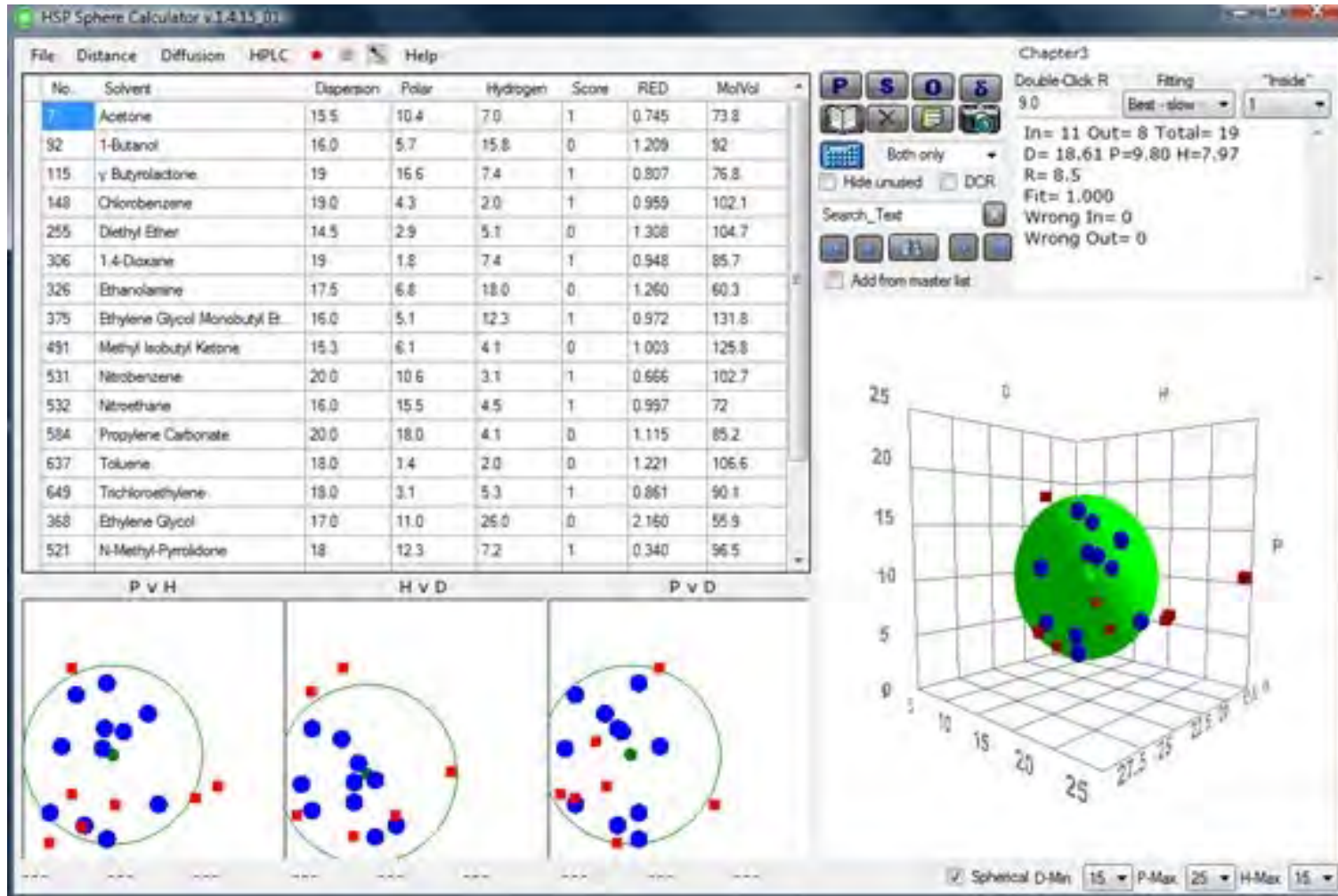
Show Master Dataset

Total Compounds = 1235

$\delta P \text{ v } \delta H$ $\delta H \text{ v } \delta D$ $\delta P \text{ v } \delta D$

Wire Frame D-Min: 12.5 P&H-Max: 25

HSPiP Software with Sphere



Database of Safer Solvents (DoSS)

- “Safer” chemicals
 - Avoiding solvents that are
 - Carcinogens
 - Reproductive/ developmental toxicants
 - Mutagens
 - Neurotoxicants
 - Endocrine disruptors
 - Halogenated solvents
 - Still refining list of chemicals for different endpoints
 - Application
 - Choose solvent appropriate environment
 - Cost
 - Bulk costs aren’t reliable; may be cheaper

The screenshot shows the DoSS Optimization Guide interface. It includes a search bar for solvent databases with fields for Cas Number and Name. Below the search bar are several sections of criteria, each with a dropdown menu and a search button:

- EHS Information:** Glove Type
- General EHS:** NFPA Health Hazard Rating, NFPA Fire Hazard Rating, GHS Flammability Limit
- Oral Exposure:** GHS Oral Acute Toxicity Limit
- Dermal Exposure:** GHS Dermal Acute Toxicity Limit, GHS Skin Corrosion/Irritation Limit, GHS Target Organ-Single-Skin Limit
- Ocular Exposure:** GHS Eye Damage/Irritation Limit
- Inhalation Exposure:** GHS Inhalation Acute Toxicity Limit, GHS Target Organ-Single-Respiratory Limit, GHS Aspiration Toxicity Limit
- Aquatic Toxicity:** GHS Acute Aquatic Toxicity Limit, GHS Chronic Aquatic toxicity Limit

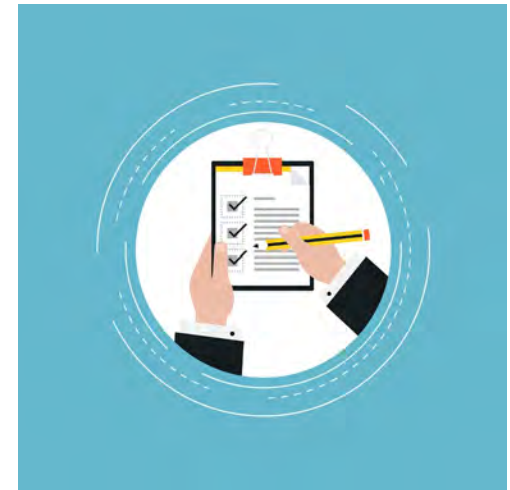
On the right side, there are sections for Solubility Theory (D, P, H) and Physical Properties (Molecular Weight, Molar Volume, Melting Point, Boiling Point, Viscosity, Flash Point, Density, Refractive Index, RER). A **Cost** section at the bottom includes fields for Bulk Price and Lab Price, with a note: "Pricing of solvent based on online vender information."

Examples of How We Are Using HSPiP

- Finding alternative to TCE to spot clean/immerse flux contaminated graphite molds
 - Company is currently throwing out molds
- Finding alternative to Toluene and NMP for immersion and spot cleaning to remove adhesive mixtures off stainless steel rollers
- Finding an alternative paint stripper for wood and metal to replace Methylene chloride

We Identified an Alternative!

- Implementation
 - Gather key informants and decision makers
 - Budget
 - Training
 - Connect with technical organization
 - MA – OTA
 - MN – MnTAP
 - Connect client to the vendor(s)
 - Chemistry
 - Equipment
- Evaluation
 - Are there issues with the new chemistry or equipment?
 - Feedback from workers



Questions?

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