Lesker Tech *"...vacuum science is our business."*

Volume 4, Issue 2 **March 2005**

Pumping Up Your Applications

Part 3B

Introduction

[Remember, all cautions given during the Introduction to Lesker Tech Vol 3 Iss 3A apply to Iss 3B too.]

During the proofing of this issue, I found *Primer on Spontaneous Heating and Pyrophoricity* (from the U.S. D.O.E) dealing with the dangers of flammable materials. Coal features prominently but there is information on many metals too. There are also 'Accident Case Studies' related to spontaneous fires and explosions involving metals—a few including fatalities. While most are not vacuum related, they are a strong reminder of *unintended consequences'* universal nature.

If your application involves finely divided metals or thin film deposition of metals that might, unintentionally, end up finely divided, I urge you to read the relevant pages of: www.eh.doe.gov/techstds/standard/hdbk1081/hbk1081.html#ZZ0

OK, So What Pump? (continued)

H. Flammable Gases — Hydrogen

Use extreme caution when pumping H_2! Its long 'explosive composition' range (4% vol to 75% vol in air) and its 'minimum ignition energy' (one tenth that of a gasoline/air mix) make it dangerous *stuff* to fool with. Select pumps with explosion proof motors and always use an inert fluid such as Fomblin®.

Diluting the pump's exhaust gas with N_2 or Ar is a must. You'd like to get the H_2 concentration much less than 4% when it finally exhausts into the atmosphere. Obviously, duct the exhaust out of the room/building, preferably to a place where lightning can't strike. And, I wouldn't use plastic tubing for the duct. I haven't heard anecdotes supporting my paranoia, but I'd worry about electrostatic discharges.

My suggested pump list for pumping H_2 is in **Table 8**.

Pump Rating Scale (Repeat)

No.	Meaning
1	OK (in most cases) *if. . .*
2	Possibly OK to so-so *if. . .*
3	Poor choice
4	Won't work and/or dangerous

Table 8 - Pumping Hydrogen

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 2, *3* • Hybrid turbos: 1 • Cryos: 4 • Getters: 1
Rough	• Diaphragm: 2 • Rotary vane, rotary piston: 1, *inert* • Screw, roots, claw: 3 • Scroll, dry piston: 2

I. Flammable Gases — Hydrocarbons

Treat methane, acetylene, ethylene, ethane, etc. at the pump's exhaust as if they are hydrogen and you won't go too far wrong. But note acetylene's (C_2H_2) explosive range in air (2.5% vol to 82% vol) is even greater than hydrogen's.

Once while browsing, I found a site offering vacuum mechanical pumps specifically for acetylene. Of course, now I need the URL I can't find it.

My suggestion for pumping hydrocarbons is in **Table 9**.

Table 9 - Pumping Hydrocarbons

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 1, *mag-lev* or *bleed* • Hybrid turbos: 1 *mag-lev* or *bleed* • Cryos: 1* • Getters: 4
Rough	• Diaphragm: 1, *corrosion* • Rotary vane, rotary piston: 1, *inert* • Screw, roots, claw: 3 • Scroll, dry piston: 2

* *Dangerous during regeneration?*

J. Flammable Gases — Spontaneously Combustible

When brought into contact with air at a transfer pump's exhaust, diborane (B_2H_6), arsine (AsH_3), phosphine (PH_3), and probably stibnine (SbH_3) spontaneously catch fire!

Clearly to avoid adding fuel to that fire, an oil-sealed pump, must be filled with Fomblin® or other inert fluid. But any type of fire at the pump's exhaust is hardly "good vacuum practice." So, these gases **must** be handled in a proper exhaust gas abatement system.

Nothing I've read claims these gases react with metals and, for gaskets, Viton®, Kalrez®, and Teflon® are OK. That is, the pump choice is not restricted as long as it has the right fluids, lubricant, gaskets, and seals. However, any reaction will give solid particles making oil filtration a very good idea.

My suggestions for pumping spontaneously combustible gases are in **Table 10**.

K. Flammable Gases — Oxidants

Yes, I know oxygen isn't flammable but read on and then we'll talk about it.

There are many vacuum processes that require pumping O_2 and some semiconductor processes where ozone (O_3) is used. I always think O_3 is like O_2 only much worse.

I read somewhere that materials which burn in 21% O_2 (normal air), burn violently in 25% O_2 atmosphere. Earth's flora and fauna wouldn't exist if air had ended up with 25% O_2. But skip hypotheticals and read these gory examples:

• Every O_2 cylinder regulator carries a strong warning against greasing its threads. My brother works for a large UK compressed gas manufacturer. Despite this company's huge education campaigns, greased regulator threads on O_2 cylinders account for ~10 fires/explosions per year.

• Since compressed O_2 is common in gas welding shops, welders frequently use it to blow metal shavings from their clothes. . . well, you wouldn't use acetylene would you? Trouble is, oil or grease patches on the guy's clothes catch fire! There aren't decent statistics on these events since, my brother believes, if the guy doesn't end up in hospital, such incidents are largely unreported.

• In my youth I worked with a 'lab clown'. His favorite trick was: dunk a small cotton-fiber ball in liquid O_2; drain; dump it on the concrete floor; and explode it by dropping a lighted match on it. Yes, very cold, finely divided cotton surrounded by ~100% O_2 at atmospheric pressure doesn't burn, it explodes!

Table 10 - Pumping Spont. Combustible

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 1, *bleed* • Hybrid turbos: 1 *bleed* • Cryos: 1*
Rough	• Diaphragm: 1, *corrosion* • Rotary vane, rotary piston: 1, *inert particle , acid* • Screw, roots, claw: 1, *semicon* • Scroll, dry piston: 2-3

* *Dangerous during regeneration?*

And here's the kicker—at any rough transfer pump's exhaust valve—when pumping pure O_2 from the chamber, no matter what the chamber's pressure, the gas exiting the exhaust valve is 100% O_2 at atmospheric pressure. Using a pump fluid that can burn is playing "Marco Polo" in a swimming pool full of crocodiles.

And what about lubricated bearings in dry pumps, or greased O-rings, or maybe even the O-rings themselves? Oh yeah! Ignore their influence when pumping O_2 and you're an easy gotcha-ee.

- Buna-N gaskets and O-rings, with an ignition source, will probably burn like old tires. Wait! With 100% O_2 make that old tires on steroids.
- Compatibility charts say Viton® is OK.
- Using a hydrocarbon grease (Apiezon L, M, N, etc) is challenging the fire gods. Thrust me, they win.
- Fomblin® grease should be OK.

In earlier *Lesker Techs* I noted dry rough pumps are not all equal. Some have bearings shielded from the vacuum volume while others have bearings removed from the vacuum volume. When pumping O_2, the latter group are preferred.

Table 11 - Pumping Oxygen and Oxidants

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 1, *mag-lev or bleed* • Hybrid turbos: 1 *mag-lev or bleed* • Cryos: 1*
Rough	• Diaphragm: 1, *corrosion*** • Rotary vane, rotary piston: 1, *inert* • Screw, roots, claw: 1, *semicon**** • Scroll: 1*** • Piston Dry: 1

* *Dangerous during regeneration?*
** *All-Teflon® construction*
*** *External bearings if possible*

When pumping O_2 or other oxidants, choose your pumps, fluids, lubricants, bearing locations, and gaskets as if your life depended on it. In the whole system, make certain you avoid anything that is even so-so flammable in air because in 100% O_2, you might be talking instant rocket fuel. With all those cautions in mind, see my suggestions in **Table 11** and remember, this is serious *stuff*.

L. Flammable Gases — Oddballs

Rocket engine fuels like hydrazine (N_2H_4) and dimethyl hydrazine ($(CH_3)_2N_2H_2$), organic reagents like methylamine (CH_3NH_2) and dimethylamine ($(CH_3)_2NH$), oddballs like hydrogen selenide (H_2Se), and many others, are flammable, toxic gases. Actually N_2H_4 is hypergolic, which (I've just learned) means it ignites on contact with an oxidant. It needs no ignition source!

My recommendation is—don't buy a pump until you've thoroughly researched all aspects of the vapor in question and the application. Talk to people who've successfully pumped these vapors before. Come to think of it, to talk to people who unsuccessfully pumped these vapors, you'd have to hold a seance.

M. Solvent Vapors

In very small amounts and in high vacuum situations, the presence of solvent vapors is usually ignored. But large quantities heading for the rough pump are a royal PIT. . .

There are two main approaches:
- Condense the vapor ahead of the pump.
- Let the vapor go through the pump.

If the **gas** is close to 100% solvent vapor and if the condenser is well-designed, it's possible to condense a considerable fraction of the *stuff* leaving the chamber. Condensers are less successful if the vapor concentration is, say, 5% in a non-condensible carrier gas. Some fraction of vapor is 'carried' through the condenser without hitting a condensing surface. Industrial vacuum equipment companies have a bunch of different condensers designed to avoid this. However, it's best to accept Murphy's Law and assume some fraction of the solvent will reach the pumps.

But a reasonable question is, OK, so what happens if (modest quantities) of solvent vapor enter a mechanical pump?

- Mechanical compression will likely cause a liquefaction/volatilization cycle, giving rotten inlet pressures.
- Solvent vapors dissolve in the pump fluid, screw with the fluid's lubricity, and ruin the bearings and 'wiped' surfaces.
- If the solvent is flammable it forms an explosive mixture at the exhaust port.

- If an oil-sealed pump uses a flammable oil, its vapor adds to the explosive force if the solvent ignites. Any vapor explosion may cause the bulk oil to catch fire.
- Vent solvents into the atmosphere (in the U.S.) and you'll be surprised how fast the EPA gets on your case about VOC emissions.

To alleviate or avoid (some) of these problems:

- Run the gas ballast and oil-case bleed wide open.
- For flammable solvents, use an inert ballast gas.
- For flammable solvents, use inert pump oil [Fomblin®].
- Maintain high ballast and bleed flows to dilute the vapor below its explosion limits when it hits the atmosphere.
- Install a proper exhaust gas abatement device.

If a liquid ring pump suits your pressure requirements, the pump's cold water often reduces the solvent's vapor pressure, dramatically increasing the rated pumping speed. Of course, the water is contaminated with solvent. But that's OK if the solvent **is** water or is something that can be recovered from water.

There are pumps called 'once-through-oil' (OTO) types. These are rotary vane mechanical pumps designed, in part, to handle vapors. The pump has a large oil reservoir but at any one time, only a small oil quantity is exposed to the vacuum volume. The exposed oil is then dumped into the collection sump for later disposal or reclaim.

The suggested pumps for solvents vapors are the same as those used for monomeric vapors. See **Table 12** for selection guidance.

Table 12 - Pumping Solvents Vapors

Vacuum	Pump Selection
High	• Diffusion: 1, *inert* • Turbos: 1, mag-lev or *bleed* • Cryos: 1, *regen* • Getters: 4
Rough	• Diaphragm: 1, *corrosion* • Rotary vane: 1, *inert, ballast, bleed* • Screw, roots, claw: 2, *ballast* • Scroll, dry piston: 2, *ballast* • OTO Rotary vane: 2, *ballast*
Coarse	• Liquid Ring: 1 • Steam ejectors: 1

N. Monomeric Vapors

For those unfamiliar with the word, a monomer is a material that, with a little help, turns into a polymer or plastic. And a hot pump might be just the help a monomer needs to polymerize. Look on a monomeric vapor as a solvent vapor...with an attitude.

Read section **M** and accept that monomeric vapors are probably all flammable even if the final plastic is non-flammable.

Again, given the right pressure requirements, liquid ring pumps are a good choice. For other vacuum levels, my suggestions are in **Table 12**.

O. Particles

First, three factoids about particles:

- They are process by-products.
- Gravity affects them.
- Some are pyrophoric.

Process by-products implies process pressures in the transitional or continuum flow regime. That is, the 'billions and billions' of gas molecules hitting the particle at any instant have enough momentum to transport micro-sized particles to the pumps. In these flow regimes, your choices are:

- Protect pumps with upstream filters
- Use pumps largely unaffected by particles

If the particles are formed in the molecular flow regime, for example, overspray from a thin film deposition process, then avoid gravity's effect—don't mount pumps on the chamber's bottom surface. But notice, during pumpdown the chamber **must** go through continuum-transitional regimes before reaching molecular flow. So, if you open the mechanical pumps full bore to the chamber, don't be surprised if dust devils form on the chamber floor, carrying particles into the pumps. Make gas removal slow enough to 'ease' through the higher pressure regions without turbulent flow starting.

As for pyrophorics—ever heard of flour mill and grain silo explosions? According to a Kansas State University webpage, there are ~15 grain/flour explosions a year. Yes, it's unlikely your 1m^3 chamber (when vented) contains enough suspended flour dust to bake a small scone (the air-flour ratio that explodes) but you know about pyrophoric materials, right? . . . those finely divided materials that catch fire on contact with air?

If you don't know, read the sidebar and the DOE's website given in the introduction. And note, it's not impossible for a deposition process's overspray to be flaky enough to ignite when you vent the chamber with air.

Suggested pumps for handling particles are in **Table 13**.

Table 13 - Pumping Particles

Vacuum	Pump Selection
High	Diffusion: 1 Turbos: 3-4 Cryos: 2
Rough	Diaphragm: 3-4
	Rotary vane: 3-4 Rotary piston: 2 Screw, roots, claw, scroll: 2, *semicon* Dry piston: 4
Coarse	Liquid Ring: 1 Steam ejectors: 1

Pyrophoric Metals

In 1969 the U.S. Naval Ammunition Depot published *Handbook of Selected Properties of Air- and Water-reactive Materials.*

Fire Science and Technology Inc. assembled an impressive index of pyrophoric materials for that handbook, from which I've chosen the elements:

aluminum, barium, beryllium, bismuth, boron, cadmium, calcium, cerium, cesium, chromium, cobalt, copper, europium, hafnium, iron, lead, lithium, magnesium, manganese, nickel, phosphorus, plutonium, potassium, rubidium, silicon, silver, sodium, strontium, sulfur, thorium, tin, tungsten, uranium, zinc, and zirconium.

And even this may be short since other lists include:

hafnium, iridium, palladium, platinum, tantalum, and titanium.

P. Droplets & Bulk Liquids

The combination of bulk liquids and high vacuum conditions are sufficiently uncommon that I'm ignoring them. (If you have such an application see me privately after class.)

Any process involving rough vacuum such as, solvent evaporation, solvent extraction, de-gassing liquids, or vacuum distillation, may lead to aerosols, foams, mists, droplets, slugs, and bulk liquids entering the pump. And while some pumps accept this without a murmur, others blow up. It's important to distinguish between those behaviors.

Vacuum pumps depending on mechanical compression—rotary vane, rotary piston, reciprocating piston, scroll, roots, etc.—react poorly when liquids enter their inlets. Liquids are incompressible and attempts to prove that untrue lead to expensive pump repair (if you're lucky) or pump replacement (if you're not).

By contrast, slop a bucket of glop into a steam ejector or liquid ring pump and who cares? The pump soldiers on without a hiccup.

My suggestions, such as they are, are in **Table 14**.

Table 14 - Pumping Liquids (accidentally)

Vacuum	Pump Selection
Rough	Diaphragm:1* Rotary vane, piston: 4 Regular screw, roots, claw, scroll: 4 Dry piston: 4
Coarse	Liquid ring: 1 Steam ejectors: 1

* *providing you use the right type*

While coarse pumps do well, the performance of rough pumps are less than staggering. This is sufficiently limiting that folks find ways to use 4-rated pumps in this application. The main approach is to install an upstream *knock-out pot*. The KOP is basically a large vacuum volume with inlet and outlet tubes mounted on the top surface with baffles between them. That allows the incoming liquid to drop to the pot's bottom without heading to the pumps.

But there are two downsides to KOPs: (a) aerosols may not be knocked out since, almost by definition, the aerosol droplets are 'carried' by the gas; (b) if the liquid has a high vapor pressure, the KOP does nothing about the solvent vapor problem.

Q. High Temperature Process

The *stuff* coming down the pipe is heat. For any vacuum application having a hot zone of 300°C or more, pump selection and pump location are critical. The killer is thermal radiation into the pump's mouth.

Contrary to popular opinion, unless large quantities of hot gas are flowing through the system, you needn't bother with the gas's heating effects. To illustrate that, let's look at two systems:

- Hot N_2 flowing into pump (20°C) at 60 sccm
- Disc heater 6" diam., from a pump (20°C) mounted on a 6" diam port

Shuffling through the numbers, very approximately, the watts dumped into the pump are shown in the table.

Heat Source	Temperature Gas/Heater	Approx. Watts
Gas	**820°C**	**1×10^{-3}**
Heater		**132**
Gas	**1020°C**	**1.3×10^{-3}**
Heater		**259**
Gas	**1220°C**	**1.5×10^{-3}**
Heater		**461**

In **Table 15** I've rated the pumps by the structure used to attach them to the chamber. Obviously, a short, straight pumping port gives the pump a much more direct 'look' at the hot zone than does a long radius elbow.

But as explained in *Lesker Tech Vol. 2, Iss. 4*, radiant heat transfer depends on many factors.

Where I boldly quote temperature limitations for a pump attached in a specified way, take those temperatures with a 40 lb sack of salt. At best I'm guessing.

Table 15 - Pumping from High Temperature Conditions

Vacuum	**Pump Selection**
High	• Diffusion (short, straight pp*): 1, *wc** cold cap, no LN2 trap, limit 350°C* • Diffusion (wc** long radius elbow pp*): *1, inert, w-c** cold cap, no LN2 trap, limit 1800°C* • Turbopumps (short, straight pp*): 1, *wc**, limit 300°C* • Turbopumps (wc** miter elbow pp*): 1, *wc**, limit 1000°C* • Cryopumps (short, straight pp*): 1, *limit <100°C* • Cryopumps (wc** miter elbow pp*): 1, *limit 500°C* • Cryopumps (wc** optical baffle, miter elbow pp*): 1, *limit 1200°C*
Rough	• All Types: 1 (*A realtor's dream-location means thermal radiation not a factor.*)
Coarse	Implies high mass flows: gas heat transfer may be a factor • Liquid Ring and Steam Ejector: 1 • Rotary vane or Piston: 2 - 3, *wc***

* *pp means pumping port*
** *wc means water-cooled*

And Finally

I hope I've convinced you that pump selection by application is serious business. I also hope you have enough ammo to avoid two classic approaches to pump selection:

- “Oh look, I found this pump in storage.”
- “I don't have the budget. Use this pump.”

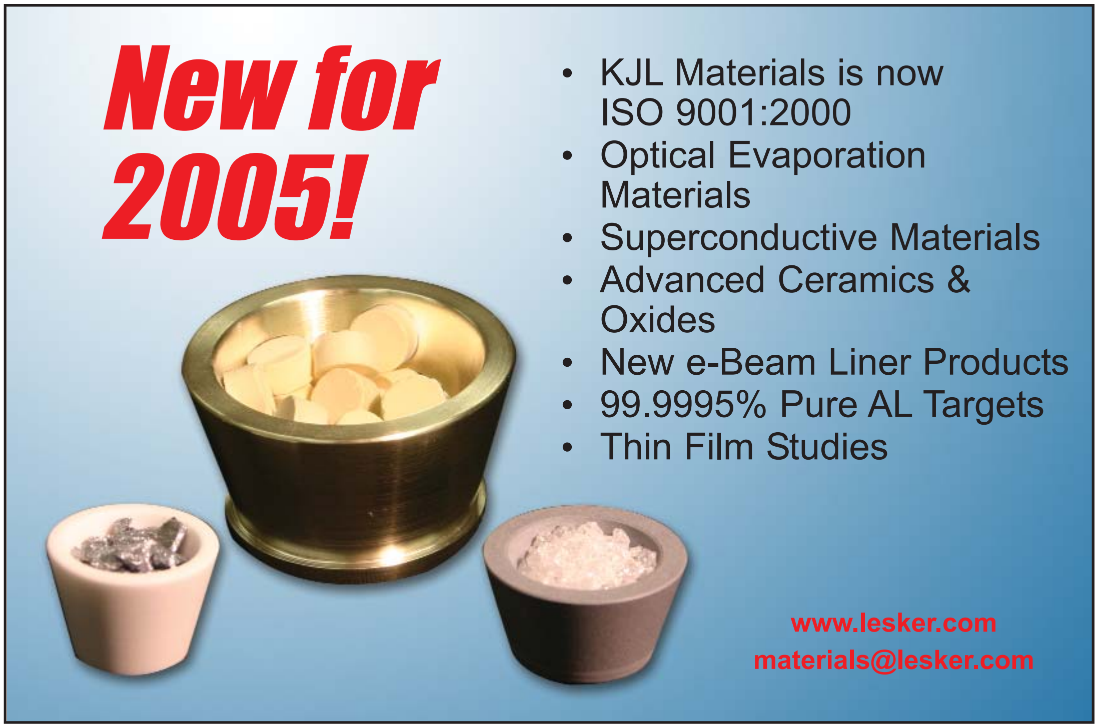
If your boss uses either approach, point out the risks associated with that particular application and suggest, politely, that he/she operate the system.

Above all, before selecting any pump, look at your application with all the cautious scepticism you'd exercise if invited to pet a rabid raccoon.

In either case, your health and welfare are at risk.

Registered Trademarks

Fomblin	Solvay Solexis
Viton	DuPont Dow Elastomer
Kalrez	DuPont Dow Elastomer
Levitra	Bayer AG and GlaxoSmithKline PLC
Cialis	Lilly ICOS
Viagra	Pfizer
Teflon	E.I. du Pont de Nemours



New for 2005!

- KJL Materials is now ISO 9001:2000
- Optical Evaporation Materials
- Superconductive Materials
- Advanced Ceramics & Oxides
- New e-Beam Liner Products
- 99.9995% Pure AL Targets
- Thin Film Studies

www.lesker.com
materials@lesker.com

Lesker On The Road

Date	Event	Location	Booth
Mar 15-17	Semicon China	Shanghai, China	4575
Mar 21-23	APS (Amer Physical Soc)	Los Angeles, CA	601
Mar 22-23	No CA AVS & ICMI	Santa Clara, CA	133
Mar 29-31	Spring MRS	San Fran., CA	507

LeskerTech@lesker.com
www.lesker.com/LeskerTech/subscribe.cfm

North American Headquarters
Ph: 412.387.9200
Fax: 412.384.2745
Sales (US):
1.800.245.1656
sales@lesker.com

West Coast Office
Ph: 925.449.0104
Fax: 925.449.5227
Sales (US):
1.800.245.1656
davidl@lesker.com

Canada Office
Ph: 416-588-2610
Fax: 416-588-2602
Sales (Canada):
1.800.465.2476
canada@lesker.com

Europe Headquarters
Ph: +44 1424-719101
Fax: +44 1424-421160
sales@leskerltd.com

Hungary Office
Ph: +36 1 383-5322
Fax: +36 1 383-4369
peters@hu.inter.net

If you believe a colleague would enjoy reading *Lesker Tech*, please feel free to forward this issue along with our compliments.

As always, your comments and suggestions are valued and welcomed.

Kurt J. Lesker Company