

Waste Vacuum Pump Oil Sample Preparation & Analysis

James Thomson



What is the function of the oil in a vacuum pump?

There are a wide variety of oils or lubricants that can be used for mechanical vacuum pumps. The first order of business is to understand what the oils do.

Firstly thing is they lubricate the pump. Because the tolerances in the pump are pretty tight it is important for the mating surfaces to receive proper lubrication. The correct viscosity for the speed and size of the pump is important.

Secondly the oil cools the pump. The majority of vacuum pumps are air cooled so the oil is the "heat exchanger" that carries the heat from the pump itself to the oil sump of the pump where the heat is dissipated.

The third and most important consideration is as a vacuum seal internal to the pump. The oil involved is intimately involved with creating and maintaining vacuum pressures in the pump and the system.

What types of vacuum oil are available?

- Hydrocarbon (mineral oil)
- Hydrocarbon (synthetic blend)
- Perfluoropolyether
- Silicone
- Polyphenyl ether
- Synthetic ester



Special vacuum pump oil is designed for use in universal and other brands of vacuum pumps, including rotary vane and piston types. This oil is specially compounded from highly refined paraffinic base oils and a balanced additive package including anti-wear, anti-oxidant, rust inhibitor, demulsifier and foam inhibitor additives. It possesses outstanding oxidation stability and demulsibility, especially in systems employing oil recovery. The low pour point ensures reliable operation in all weather conditions.

What additives are in vacuum oils?

Vacuum oils may contain any ordinary additives for lubricating oil, such as:-

- Anti-wear additive
- Extreme pressure additive
- Antioxidants
- Precipitation inhibitors
- Rust inhibitors
- Viscosity index improvers, etc.,

for the purpose of further improving the capabilities of the vacuum pump oil.



What additives are in vacuum oils?

Anti-wear additive

- Zinc dialkyldithiophosphate
- Molybdenum compounds

Extreme pressure additive

- Zinc dialkyldithiophosphate

Anti-oxidants

- Phenolic antioxidants (e.g. 2,6-di-tert-butylphenol)
- Amine-type antioxidants (e.g. dioctylphenylamine)
- Sulfur-type antioxidants (e.g. thioesters)
- Phosphorus-type antioxidants

What additives are in vacuum oils?

Precipitation inhibitor

- Nonionic surfactants
 - Polyethylene glycol
 - Polypropylene glycol

Rust inhibitor

- Alkenylsuccinic acid monooleates
- Polyamides
- Barium sulfonate
- Benzotriazole derivatives

Viscosity index improver

- Polymethyl methacrylates
- Polyisobutylenes

What other contaminants can be found to waste vacuum oil?

2-propanone
2-methyl-1,3-butadiene
1-hexene
butanal
2-butanone
hexane
acetic acid
1,3-hexadien-5-yne
1,5-hexadien-3-yne
benzene
2-pentanone
1,2-dimethyl-cyclopentane
heptane
1-heptene
pentanal
methyl-cyclohexane
propanoic acid
4-methyl-2-pentanone
toluene
1-octene
heptanoic acid
1-ethyl-2,3-dimethyl-benzene
2-nonanone
undecane
nonanal
decamethyl-cyclopentasiloxane

2-hexanone
cyclopentanone
octane
hexanal
tetrachloro-ethene
hexamethyl-cyclotrisiloxane
butanoic acid
3-methyl-cyclopentanone
1,3-dimethyl-benzene
ethyl-benzene
xylene
1,2-dimethyl-benzene
4-methyl-hexanal
2-heptanone
1,3-dimethyl-benzene
styrene
cyclohexanone
nonane
heptanal
pentanoic acid
alpha-pinene
6-methyl-2-heptanone

2-propanone
dihydro-5-methyl-2(3H)-furanone
1-ethyl-2-methyl-benzene
1-ethyl-4-methyl-benzene
benzaldehyde
1,3,5-trimethyl-benzene
octamethyl-cyclotetrasiloxane
2-octanol
1-decene
1,2,4-trimethyl-benzene
hexanoic acid
decane
octanal
d-cymene
1,2,3-trimethyl-benzene
hexanoic acid
limonene
1-methyl-3-propyl-benzene
1-ethyl-3,5-dimethyl-benzene
1,2,4,5-tetramethyl-benzene
5-ethyl-dihydro-2(3H)-furanone
1-phenyl-ethanone

And too many others to mention !!!!!

How does the oil become contaminated?

Tritium Handling facilities often have oil lubricated vacuum pumps in active portions of tritium systems. The interaction of tritium with the oil can lead to the formation of various classes of tritiated species, both volatile and non-volatile. These tritiated species can cause problems in terms of handling, dosimetry and ultimate waste disposal.

How does the oil become contaminated?

Radioactive oil waste produced in nuclear power plants consists of lubricating oils from primary heat transport pumps, hydraulic fluids from fuelling machines, and turbine oils. These are normally low level wastes containing only relatively small quantities of beta/gamma emitting radionuclides. They can become contaminated with volatile radionuclides or by contact with radioactive materials, but low levels of activation products may develop in power plant lubricants due to exposure to neutrons.

How does the oil become contaminated?

The removal of tritium from heavy water in the heat transport system of pressurized heavy water reactors can produce intermediate level Tritiated oil waste through contamination of vacuum pump oil by gaseous tritium, which then becomes organically bound through isotopic exchange. This waste oil may have up to 1.8 TBq/Lt of Tritium.

What methods can be considered?

- **Direct addition**
- **Freeze drying**
- **Azeotropic distillation**
- **Oxidation**
- **Combustion**

What methods can be considered?

Direct addition

Direct addition of waste oil to a cocktail will be problematic due to colour quench and more importantly chemiluminescence.

The thermal degradation products found in waste oil can be extremely chemiluminescent and can generate count rates of >1,000,000 cpm that persist for months.

Colour quenching is also an issue and this restricts sample size to <0.1 ml.

Overall this is not a recommended method.

What methods can be considered?

Freeze drying

Freeze drying is a process where a sample is frozen and then exposed to high vacuum. The water contained in the sample goes from a solid state directly to a vapour (sublimation). If the vacuum is drawn through a cold trap, this vapour can be collected as pure water.

Due to the fact that Tritium can be present as OBT (Organically Bound Tritium) it is likely that not all the Tritium will be measured.

Overall this is not a recommended method.

What methods can be considered?

Azeotropic distillation

Azeotropic distillation provides a simple method to extract water, from a wide variety of media (e.g. honey, milk, vegetation, soil, fish), for tritium analysis using liquid scintillation counting. However, for the same reasons given under Freeze Drying it will not be possible to extract the OBT, only the free water.

Overall this is not a recommended method.

What methods can be considered?

Oxidation (1)

With the Perkin Elmer Sample Oxidiser it is possible to measure both Tritium and Carbon-14 in waste oil. However the maximum sample size is 100 μ l and Combustaid must be added to achieve soot-free combustion.

The only other drawback of this method is the initial capital outlay of £38K for the instrument.

Recommended method



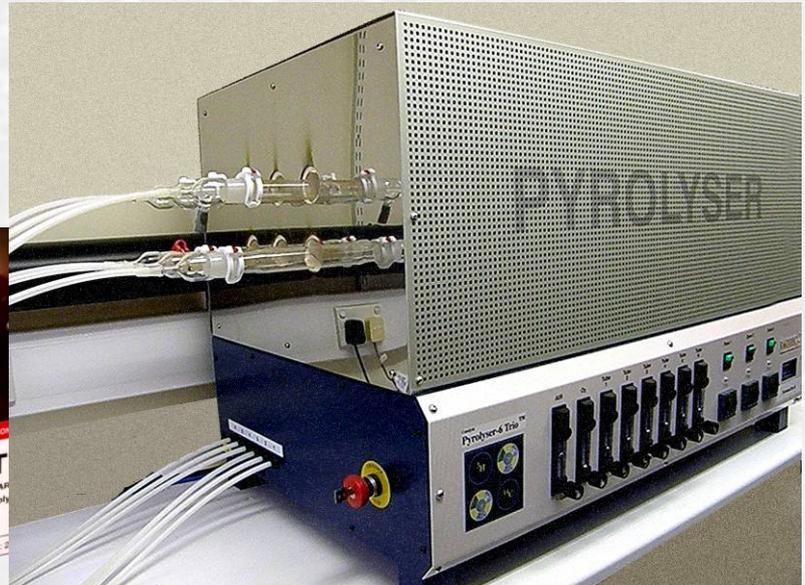
What methods can be considered?

Oxidation (2)

With the RADDEC Pyrolyser it is possible to measure both Tritium and Carbon-14 in waste oil. With this instrument the maximum sample size is 500 μ l and to achieve soot-free combustion the sample is absorbed on a bed of sand.

Capital outlay for this instrument is £20K making it more affordable.

Recommended method



What methods can be considered?

Combustion

It is possible to use a Parr Oxygen Bomb to combust up to 5 g waste oil. The sample is placed in a cup inside the bomb and then combusted after being pressurised with oxygen. “Combustion water” is formed and condenses inside the bomb. This is then removed for Tritium analysis. Requires passive distillation before analysis.

Capital outlay for this instrument is £2K to 3K making it an affordable option.



Recommended method

Is there another alternative?

Oil-free Vacuum pump

A wide range of oil-free vacuum pumps are available on the market to meet the needs of various applications, including those that require very strong suction.

Oil-free vacuum pumps eliminate the possibility of a spill that would create additional waste and require effort to clean.

Other savings include:-

- Cost of waste oil analysis
- Cost of waste oil disposal
- Cost of replacement oil
- Time spent replacing waste oil
- Time spent with associated paperwork

Conclusions and recommendation

If waste oil is generated then sample oxidation (combustion) is the only recommended method for Tritium and Carbon-14.

The RADDEC Pyrolyser in combination with CarbonTrap & CarbonCount, offers the most affordable solution to the analysis problem.