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OIL RECLAMATION USING MOLECULAR SIEVES (U)

R. DeMonbrun
I. G. Speas
W. E. Weathersby

RELEASED FOR ANNOUNCEMENT
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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

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Nuclear Division

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Contract W-7405-eng-26
With the US Atomic Energy Commission

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ABSTRACT

A study has been made of the reuse of various types of organic oils and tri-aryl phosphate ester fluids used in large volume at the Oak Ridge Y-12 Plant. It is concluded that Molecular Sieve desiccant affords an economical means of obtaining highly purified stocks for reuse. A simple regenerative equipment arrangement has proved to be suitable for the purification of a wide range of lubricating, hydraulic, and insulating fluids.

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SUMMARY

Molecular Sieve reclamation systems have proven to be simple to operate and economical for the purification of various lubricating, hydraulic, and insulating fluids. The essential components of the system are shown in Figures 1 and 2.

An experimental reclamation test facility has been in operation for three years. Typical field applications are listed in Table 1.

Figure 1. MOLECULAR SIEVE RECLAMATION SYSTEM.

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Figure 2. EXPERIMENTAL TEST UNIT FOR OIL RECLAMATION STUDIES AT THE OAK RIDGE Y-12 PLANT.

Table 1
TYPICAL FIELD RECLAMATION SYSTEMS

Application	Type Fluid	Old Reclamation System	Molecular Sieve System Installation Costs (\$)	Cost Savings/Year ⁽¹⁾ (\$)
7500-Ton Hydraulic Press (1964)	Tri-Aryl Phosphate Ester	Flash Vaporization, Clay Filter	6000 ⁽²⁾	16,000 ⁽³⁾
Insulating Oil (1965)	Naphthenic Mineral Oil	Flash Vaporization	475 ⁽⁴⁾	1,250 ⁽⁵⁾
Mechanical Vacuum Pump Oil (1966)	Paraffinnic Mineral Oil	Flash Vaporization, Clay Filter	1200 ⁽⁴⁾	4,000 ⁽⁶⁾
Isostatic Press (1965)	Paraffinnic Mineral Oil	Flash Vaporization, Clay Filter	4700 ⁽²⁾	4,000 ⁽⁷⁾
BONUS Nuclear Reactor (1965)	Tri-Aryl Phosphate Ester	None	3500 ⁽⁸⁾	(see Page 40)
Portable Oil Reclamation System (1963)	All Fluids	None	2000 ⁽⁴⁾	10,500 ⁽⁹⁾

(1) Molecular Sieve over old method.

(2) Desiccant adsorber regenerated at site.

(3) Savings in replacement lubricating fluid.

(4) Desiccant adsorber regenerated at central station.

(5) Savings in utility costs.

(6) Savings in operation and maintenance labor and oil.

(7) Savings in operation and maintenance labor.

(8) Desiccant replaced with new material upon depletion.

(9) Savings in lubricating, insulating, and hydraulic fluids.

Other comments:

1. Sources of Contamination - The influx of water from leaks in oil coolers and equipment seals and by condensation from air in oil storage reservoirs, the presence of oxygen, high-temperature service, and inadequate cleanliness control are principal mechanisms for contamination.
2. Reliability - Plant equipment requiring lubricating, hydraulic, and insulating fluids has proven significantly more reliable after institution of a purification program utilizing the Molecular Sieve reclamation system. As an example, in servo-controlled hydraulic systems the fluid must be maintained ultra clean. The reclamation unit has fulfilled this requirement.
3. Design Parameters - Reclamation-system design parameters should assist others in sizing adsorbers to suit their particular requirements.
4. Testing and Analysis - Conventional oil testing has been shown to be a valuable tool in recognizing composition changes and in detecting the presence of contaminants. The most common tests used in the Y-12 Plant are water content, neutralization number, dielectric strength, interfacial tension, infrared spectrophotometer, and corrosion product contamination.

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INTRODUCTION

Costly shutdowns of plant equipment due to contaminants in hydraulic fluids and the high costs of replacing dirty oils and tri-aryl phosphate ester fluids led to a study to determine an efficient method of fluid reclamation. A Molecular Sieve adsorber experimental unit was constructed to evaluate the purification ability of this desiccant. This study resulted in the development of a practical and economical oil reclamation system consisting of mechanical filtration and Molecular Sieve dehydration and reduction of acidity.

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DISCUSSION OF THE STUDY

BACKGROUND

The high costs of replacing contaminated hydraulic fluid in presses, measuring machines, and machining equipment, and insulating oil in transformers prompted a study to determine an efficient method of reclaiming the used fluids. In addition, equipment malfunctions which were caused by contaminants in the hydraulic fluid emphasized the need for improved cleanliness control to prevent costly shutdowns and increased maintenance costs. The following discussion is presented to assist the operator of equipment to better evaluate his own particular problems of oil reclamation.

TYPES OF OIL CONTAMINANTS

Lubricating, insulating, and hydraulic oils become contaminated even under normal service conditions. The more harmful types of contaminants are:

1. Water, which may be condensed from atmospheric air inside oil reservoirs. Condensation may be accelerated when the oil level in a reservoir is frequently changing thereby drawing in moisture-laden outside air. Oil cooler and equipment seal leakage may also contribute to moisture contamination.
2. Oxidation products or sludge, which result from the breakdown of the oil when exposed to higher temperatures. These products may be soluble or insoluble in the oil. The soluble products which are formed corrode certain metals and increase oil viscosity; the insoluble products make systems sluggish or inoperative by reducing clearances and plugging valves, lines, filters, and other system components. Most of the oxidation products are soluble in water. The water becomes acidic and will act as a catalyst in the presence of metals such as iron and copper to form metallic soaps, which in turn accelerates oxidation.
3. Polymerization products, which cause oil thickening. At normal or high temperatures, unsaturated oil molecules can join to form heavier molecules which may be soluble or insoluble in the oil.
4. Abrasives such as rust, weld spatter, dust, core sand, and metal particles from the wear of machine parts.

METHODS OF OIL RECLAMATION USED IN INDUSTRY

Several methods of cleaning oil are used in industry. The type oil, degree of cleanliness, and cleaning cost are the prime considerations for selecting the purification

mode. Of primary interest is the fact that these cleaning processes were developed for use with petroleum oils and not the relatively newer fire-resistant fluids. Tri-aryl phosphate ester fluids require different treatment, primarily because they have a higher specific gravity than petroleum oils. In addition, water in the fluid can hydrolyze the ester yielding an organic acid form.

The acid will corrode metal components of a system yielding fine abrasive products. This condition is particularly undesirable in servo valve systems where the particles can plug valve orifices and erode valve metering surfaces. Excessive leakage at the high-pressure drop through scored valves can result in overheating the hydraulic fluid causing its partial thermal decomposition and a buildup of sludge in the valves. A brief discussion of these purification methods and their suitability for tri-aryl phosphate ester fluids is presented in the sections that follow.

Gravity Purification

This method provides the simplest treatment but does not give complete purification. In addition, considerable time is required for the heavier contaminants to separate from the oil in settling tanks. Settling is not adaptable to tri-aryl phosphate ester fluids because there is not enough difference in weight between the contaminants and the fluid. Furthermore, water which has a lower specific gravity than the fluid, will migrate to the top and soluble contaminants will remain in the fluid.

Centrifuging

A centrifuge is a mechanical device for accelerating the separation of two or more phases of a fluid when a difference in density exists between them. This method is widely used to remove suspended contaminants and water from petroleum oils. Water washing in combination with centrifuging can be effective in removing soluble oxidation products which are more soluble in water than oil. However, this process is not suitable for inhibited oils because the water removes the wear and oxidation-preventive additives. As with gravity settling, purification of tri-aryl phosphate ester fluids by centrifuging is more difficult because the fluid is more dense than water. With petroleum oil, the heavier particles and water are separated to the outside and removed by centrifugal force.

Mechanical Filtration

Numerous types of mechanical filters are available.⁽¹⁾ Selection is usually dependent on fineness of filtration required, pressure drops, and absorption capacity. Absorption filters are very effective for the removal of solid contaminants; however, they do not remove water and soluble oxidation and polymerization products.

Adsorption Filtration

Fuller's earth, activated alumina, silica gel, charcoal, and activated bauxite are the more common oil adsorbents. In addition to mechanically filtering solid con-

taminants, these adsorbents reduce the amounts of water and soluble oxidation products. Unfortunately, they also remove oil additives.

Coalescence Separation

Separation of free water from petroleum oil by coalescence has been effective with certain lubricants.⁽²⁾ Coalescence is the process through which minute water droplets come together and form larger droplets which are separated from the oil. Special Fiberglas elements have been developed which have coalescence and filtration properties. The suitability of these elements with tri-aryl phosphate ester fluids is not known by the authors.

PROBLEMS ENCOUNTERED WITH SYSTEMS IN THE Y-12 PLANT

Certain problems have been encountered with high-pressure (3000 psig) systems using the fire-resistant phosphate ester fluids. Moisture and acidity accumulate and cause corrosion of the system and sludging of the fluids. These sludge and corrosion products collect in such places as the low-velocity areas and around pump seals. Finite particles of iron and copper have produced worn places in valves and pumps. Other materials such as molybdenum disulfide and carbon have entered the fluid reservoirs by improperly designed air vents.

Laboratory analyses of oil from mechanical vacuum pump systems have indicated very significant accumulations of free and dissolved water (~1.5%) which result in high acidity. Sludge deposits as much as three inches in depth have been found in the oil reservoirs of pumps. Pump maintenance costs have been excessive and sometimes a complete replacement of pumps was required. In some systems, the oil has been changed as much as three times before the desired vacuum was obtained.

Excessive water content, resulting in a low dielectric strength, has been found in the transformer-type oils that are used in electrical gear and coolant systems.

Approximately 80 percent of the commercially designed oil reclamation systems in the Plant have been removed from service because of high maintenance and operating costs plus the fact that the majority of the oil problems still persist. This situation resulted in a complete investigation of the many types of oil systems in an effort to design better methods of reclamation and to improve system operation performances.

Specifications for the more conventional oils which are used in the Y-12 Plant are shown in Appendix A.

EXPERIMENTAL TEST FACILITY

The following desirable capabilities of an oil reclamation system were considered in the Experimental Test Facility investigations:

1. The system must be economically feasible.
2. The oil reclamation system must be suitable for use on any lubricating oil (SAE 5 to SAE 30) and the phosphate ester fire-retardant fluids.
3. The system must provide the ultimate in purification including removal of free and dissolved water, reduction in acidity, and improvement of clarity by removal of such insoluble particles as carbon and dirt.
4. The adsorption system used to remove contaminants must have a long service life and be a regenerative material.
5. The system should not remove the chemical additives that are blended with certain fluids to improve performance characteristics. (This report does not include experimental data to verify whether or not chemical additives are removed from oil when passing through a Molecular Sieve bed; however, experiments by others indicate they are not removed.)⁽³⁾

The Experimental Test Facility consists of three 50-gallon stainless steel tanks, a gear pump, two cartridge-type prefilters, a rotameter, an adsorber column, and a cartridge-type after filter (see Figure 2, Page 7 and Figure 3, Page 17). One of the stainless steel tanks is wrapped with heat tape, insulated, and thermostatically controlled. The adsorber column contains 1.15 cubic feet or approximately 50 pounds of Type 4A, 8 x 12 mesh, Molecular Sieve beads.

The heat-traced tank was used in the tri-aryl phosphate ester experiments to improve the viscosity of these fluids for pumping. Hydrocarbon oil experiments were conducted at room temperatures. Refining of hydrocarbon oils and tri-aryl phosphate ester fluids with the Molecular Sieve adsorber was performed on an equilibrium basis; that is, the flow of the lubricant from the reservoir was recirculated through the Molecular Sieve adsorber several times until the adsorber reached equilibrium with the adsorbant in the fluid. The circulation of the total contents of the reservoir through the sieve adsorber is referred to as one cycle. The more passes or cycles through the sieves, the more the lubricant contaminant level is reduced to reach the equilibrium capacity of the sieve adsorber.

The sieve adsorber is regenerated by washing the sieves with low-pressure (10 - 15 psig) steam. First, the steam is directed up through the sieves (opposite direction of normal flow) to wash out the heavy sludge material. As soon as the condensate is observed to be free of heavy sludge deposits, the steam flow is reversed. Steam flow is maintained until the condensate is observed to be clear and free of oil. The steam flow and time required for steam cleaning will depend upon the cleanliness of the oil processed. Then oil-free air having a dew point of -75°F (measured at atmospheric pressure) is heated electrically to 350°F and directed up through the sieve adsorber (opposite direction of normal flow). When the effluent air temperature

Figure 3. COMPONENTS OF THE EXPERIMENTAL TEST UNIT FOR THE OIL RECLAMATION STUDIES.

reaches a constant value (300°F for this adsorber), it is maintained at this level for 30 minutes. Theoretically, this treatment reduces the residual moisture in the adsorber to 0.8 - 1.0 percent by weight. Higher temperatures (600°F maximum allowable) would further reduce this residual moisture if desired. After heating with hot air, the adsorber is isolated and allowed to cool to ambient temperature. Air pressure is maintained while cooling to eliminate the possibility of inleakage from the atmosphere.

RESULTS OF EXPERIMENTS

Tri-Aryl Phosphate Ester Fire-Resistant Fluids

The first group of experiments performed with the tri-aryl phosphate ester fluids was conducted on actual fluids removed from systems that were encountering operative difficulties. The fluid was heated to 110°F (viscosity - 580 SUS at 100°F) and the sieve adsorber operated on an equilibrium basis. Four samples of this fluid were evaluated to determine what contaminants could be removed. Since the fluid contained excessive amounts of sludge, experiments were conducted to determine if the regeneration cycle after reclaiming each sample would remove the sludge and not limit the sieve capacity. Figure 4 shows a comparison of the sieve material removed from the adsorber compared to new material. Table 2 shows the data obtained from the first three samples of this fluid.

A fourth test was made to determine the cycles required to reduce the water and acid contents of the fluid to acceptable levels (water content $< 100\text{ ppm}$ and neutralization number $< 0.05\text{ mg KOH/gm}$). The fluid had an initial water content of 600 ppm and a neutralization number of 0.082 mg KOH/gm . These data are plotted in Figure 5.

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Figure 4. MOLECULAR SIEVE DESICCANTS.

Table 2
LABORATORY ANALYSES OF PHOSPHATE ESTER FLUID

Number of Cycles	Initial			100			200	600
Sample Number	1	2	3	1	2	3	3	3
Water Content (ppm)	860	400	800	91	30	-	20	20
Neutralization Number (mg KOH/gm)	0.0890	0.140	0.167	0.062	0.030	-	0.103	0.068
Interfacial Tension (dynes/cm)	23.1	18.7	21.8	24.0	23.8	-	22.2	23.4

Figure 5. PURIFICATION PROPERTIES OF MOLECULAR SIEVES USING RECIRCULATED PHOSPHATE ESTER FLUID.

Analyzing the data in Table 2 and Figure 5, the total water content was reduced rapidly; however, the removal of acidity required more contact time. The fluid clarity was greatly improved since the mechanical ten and one-micron filters and the adsorber all removed sludge deposits.

The second set of experiments was in reality a recovery of 445 gallons of tri-aryl phosphate ester fluid that had been released into a recovery pit by rupture of hydraulic press high-pressure line. The fluid contained such contaminants as dirt, floor sweepings and water. The heavy sludge was allowed to settle in the tanks and the excess free water was removed by siphoning. The purpose of these tests was to determine the amount of water loading on the sieve adsorber, to refine the fluid at least to the water content and clarity of new fluid, and to return the hydraulic press to operation. Table 3 summarizes the data obtained while recovering the fluid. Figure 6 illustrates the clarity of the phosphate ester fluid before and after refining.

Table 3
RESULTS OF THE RECLAMATION TESTS FOR THE HYDRAULIC
PRESS PHOSPHATE ESTER HYDRAULIC FLUID

Sample Number	Quantity (gallons)	Number of Cycles	Initial Water Content (ppm)	Initial Color	Final Water Content (ppm)	Final Color
1	85	15	1600	Brown-Green	59	Clear Green
2	100	20	1820	Brown-Green	600	Clear Green
3	50	8	1960	Brown-Green	1300	Milky Green
4	50	20	1210	Brown-Green	960	Clear Green
Molecular Sieves Regenerated						
5	50	20	2140	Brown-Green	490	Clear Green
6	50	20	810	Brown-Green	320	Clear Green
7	60	20	3610	Almost Black	180	Clear Green

Samples 3 and 4 indicate that the sieves had almost reached their equilibrium capacity for water. The adsorber was then isolated and regenerated and Samples 5, 6, and 7 processed. The water capacity of the sieve adsorber for the first four samples was calculated to be 5.3% or 5.3 lbs water per 100 lbs sieve. The water capacity of the sieve adsorber for the last three samples calculated to be 7.4% or 7.4 lbs water per 100 lbs sieve. These data indicate how easily the unit can be used to recover an expensive fluid that would have normally been replaced with new fluid had the facilities not been available.

If time had not been limited, the fluid could have been purified to a very low water content as shown by the first set of experiments.

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Figure 6. RECLAIMED PHOSPHATE ESTER FLUID.

Transformer Oil - Naphthenic Base

A sample of oil from a 5000-gallon storage tank containing used oil from several transformers was tested to determine if the sieve adsorber would improve the fluid so that it could be reused. Table 4 summarizes the results of this test in comparison with new oil.

Table 4
RESULTS OF RECLAMATION TESTS ON TRANSFORMER OIL
(Sample from a 5000-Gallon Storage Tank)

Number of Cycles →	Initial	300	800	1600	New Oil
Water Content (ppm)	31	-	24	11	1
Neutralization Number (mg KOH/gm)	0.115	-	0.021	0.021	0.02
Interfacial Tension (dynes/cm)	23.5	-	30.7	31.8	42.8
Dielectric Strength (kv)	27	35	35	35	35

Although the oil tested had some discoloration after refining, the adsorber did indicate removal of acidity and water from the fluid. It was possible to return the 5000 gallons of transformer oil to the plant oil system after refining with a sieve adsorber.

Two other samples of used transformer oil which had been removed from operating systems were tested. The results are shown in Table 5.

Table 5
RESULTS OF RECLAMATION TESTS OF TRANSFORMER OIL
(Samples from Operating Systems)

	Sample 1		Sample 2	
	Initial	After 10 Cycles	Initial	After 10 Cycles
Water Content (ppm)	12	9	30	20
Neutralization Number (mg KOH/gm)	0.054	0.028	0.086	0.036
Interfacial Tension (dynes/cm)	23.7	28.7	24.0	36.7

The 1200 gallons of transformer oil involved in these tests were reclaimed and returned to the plant system. The color of this oil was returned to its original lemon-yellow color.

Straight Mineral-Base Oil

Samples of this paraffinic-base mineral oil were removed from a hydraulic system normally operating at 30,000 psig. This oil was slightly discolored and was prefiltered with ten and one-micron filters before passing through the sieve adsorber. Table 6 lists the results of this test.

These data indicated that the sieve adsorber removed acidity and water from the mineral-base oil. This oil had been constantly refined with three flash vaporization-clay filtration units; however, improvement was still obtained with the sieve adsorber.

Typical Water Adsorption Curves

A two-inch-diameter by 42-inch-long Pyrex tube containing 2.9 lbs of Type 4A sieves, a centrifugal pump, a rotameter, and two 50-gallon tanks were the components used for determining water adsorption curves (Figure 7).

TI Temperature Indicator

Figure 7. TEST SYSTEM FOR DETERMINING THE EFFECTS OF A VARIABLE FLUID MOISTURE CONTENT ON A MOLECULAR SIEVE ADSORBER.

Table 6
RESULTS OF RECLAMATION TESTS ON
MINERAL-BASE HYDRAULIC OIL

	Sample 1		Sample 2		
	Initial	After 20 Cycles	Initial	After 12 Cycles	After 72 Cycles
Water Content (ppm)	14	8	17	17	17
Viscosity (SUS at 100° F)	88	87.6	87	87.9	86.9
Neutralization Number (mg KOH/gm)	0.202	0.127	0.109	0.061	0.028
Interfacial Tension (dynes/cm)	23.8	31.6	22.5	25.0	32.3
Flash Point (° F)	345	-	358	355	358

The sieve adsorber was regenerated with clean air (-75° F dew point measured at atmospheric pressure) at 400° F in the direction opposite to the normal flow.

Test 1 was made on new transformer oil that was "spiked" with water to a range found in many operating systems; Test 2 was made on transformer oil that had been "spiked" with water to a range 1.75 times greater than Test 1. Figure 8 is a plot of the adsorption curve for transformer oil with an influent water content of 134 ppm by weight. The flow velocity in this test was maintained at 0.5 foot per minute and the oil was passed once downward through the adsorber and into the second 50-gallon tank. Figure 9 shows the adsorption curve for transformer oil with an influent water content of 235 ppm by weight. The flow velocity was maintained at 0.5 foot per minute and the oil flow was passed once upward through the adsorber and into the second 50-gallon tank.

The following conclusions were made from these tests:

1. The effluent water content of the transformer oil was reduced to approximately 20 ppm by weight by a single pass through the Molecular Sieve bed even though the influent water content was varied by a factor of 1.75 and the flow was reversed to observe any bed movement at this velocity. Test 1 reduced the water content from 134 ppm to approximately 20 ppm (85% reduction) and Test 2 reduced the water content from 235 ppm to approximately 20 ppm (93% reduction). Since the length of the bed, the flow velocity, and the oil temperature were the same in both tests, and the only variables were change in the direction of flow and the influent water content, the results of these tests indicate that the mass transfer zone in oil drying is affected by the viscosity of the fluid. Theoretically, should this zone be a small portion of the desiccant, the effluent water content should have approached the one-ppm level. The mass transfer zone of

Figure 8. TRANSFORMER OIL WATER ADSORPTION CURVE. (Test 1; Downward Flow Direction)

Figure 9. TRANSFORMER OIL WATER ADSORPTION CURVE. (Test 2; Upward Flow Direction)

an adsorber is that quantity of the desiccant where the adsorbent concentration in the fluid being dried is decreasing to the equilibrium value of the desiccant at the outlet of the bed.

2. The oil flow passing upward through the bed actually raised the bed two inches with a velocity of 0.5 foot per minute. This action demonstrates the importance of installing screens at both the inlet and outlet of the adsorber. Also, to keep the sieve material in a packed condition, the oil flow should be downward through the adsorber.

Test 3 was conducted on tri-aryl phosphate ester fluid that was "spiked" with water to a level found in many operating systems. Figure 10 presents the adsorption curve for the phosphate ester fluid with an influent water content of 1626 ppm by weight. The flow velocity was maintained at 0.5 foot per minute and the flow was downward through the adsorber and into the second 50-gallon tank. The moisture content of the phosphate ester was reduced from 1626 ppm to an average of 600 ppm (63% reduction).

Summarizing these data, more efficient results can be obtained from the Molecular Sieve adsorber for oil purification when it is operated on an equilibrium basis (passing the oil through the sieves several times until the adsorber reached equilibrium).

Figure 10. TRI-ARYL PHOSPHATE ESTER WATER ADSORPTION CURVE. (Test 3)

with the adsorbant in the fluid). It is evident that the mass transfer zone is very long due to the viscosity of the fluid. In air or gas drying, the various molecules are very active and become intermixed several times while passing through the adsorber thus exposing the adsorbant to the sieves many times. In oil drying, the water solubilized in the oil does not become intermixed due to the viscosity of the fluid; therefore, it would require a tremendously long unit in order to have the effective mass transfer zone to adsorb all contaminants in a single pass. However, the data illustrated in Figure 5 show that for this particular test, only eight passes through the adsorber were required to reduce the moisture content from 600 to 100 ppm.

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FIELD UNITS IN SERVICE

7500-TON PRESS FLUID RECLAMATION SYSTEM

The tri-aryl phosphate ester fluid reclamation system for the 7500-ton hydraulic press is shown in Figures 11 and 12. This system was designed to operate on a side-stream basis and continuously process the 7700 gallons of phosphate ester fluid in the main reservoir of the press. Fluid from the main reservoir is pumped at a rate of ten gallons per minute through a rotameter, a ten-micron filter, a Molecular Sieve adsorber, a one-micron filter, a second rotameter (at the press console), and returned to the main reservoir. The return line to the reservoir is installed so that the outlet is below the minimum low level of the phosphate ester fluid to prevent entrainment of moisture-laden air and oxygen into the fluid. The rotameter at the press console is used by the press operator to monitor the fluid flow through the reclamation system which is installed in the basement.

Each filter contains six cotton cellulose spiral-wound replaceable cartridges, two inches in diameter by ten inches in length. The adsorber is 18 inches in diameter by 42 inches in length and contains 278 pounds of Type 4A Molecular Sieves, 8 by 12-mesh beads. The ten-gallons-per-minute fluid flow through the adsorber is equivalent to a fluid velocity of 0.825 foot per minute. The sieve adsorber is regenerated with instrument air (-75°F , measured at atmospheric pressure) at 450°F and with 15 psig steam at 235°F .

The contents of the main reservoir are circulated through the reclamation system in approximately 13 hours. This time is referred to as one cycle. When the reclamation system was initially installed, the phosphate ester fluid contained 600-ppm water by weight and had a neutralization number of 0.085 mg KOH/gm. The fluid was black in color and contained considerable amounts of sludge. After one week of operation, the water content was reduced to < 20 ppm and the neutralization number was reduced to 0.03 mg KOH/gm. After 22 days of operation, all filter cartridges were changed due to a high-pressure drop caused by excessive loading. The fluid clarity was greatly improved and no visible sludge was observed.

The Molecular Sieve system of reclaiming was selected over other systems as it offered the advantages of lower costs for installation, operation, and maintenance. This system was installed at a cost of \$6000. The operating cost for electric power has been \$70 per year. Regeneration of the Molecular Sieve adsorber has been at eight-month intervals. The regeneration procedure involves a full eight-hour shift for one press operator. The principal maintenance for the system has been in the changing of filter cartridges about four times per year, requiring three hours for all filters. For comparative purposes, another reclamation system, proposed for this press, was investigated. This facility involved centrifuging, heating the phosphate ester fluid to 165°F , flash vaporization, and clay filtration. The cost of this system was estimated at \$25,000 and required more operating and maintenance time.

118336(U)

Figure 11. RECLAMATION SYSTEM FOR TRI-ARYL PHOSPHATE ESTER FLUID USED IN THE 7500-TON HYDRAULIC PRESS.

The 7500-ton press had been a constant source of hydraulic problems causing faulty operation and downtime. Phosphate ester fluid valued at approximately \$16,000 had been used each year to flush out the system to keep the press operating. This press recently operated 1 1/2 years before a shutdown, which was due to pump failure. Very small quantities of the phosphate ester fluid have been added to the system. Any spilled fluid (usually due to packing leaks) has been returned to the dirty oil tank and processed by the reclamation system for reuse.

INSULATING OIL RECLAMATION SYSTEM

The insulating oil reclamation system consists of a Molecular Sieve adsorber and a rotameter (see Figures 13 and 14). This facility side streams a portion of the oil flow from the main circulating pump through the Molecular Sieve adsorber and back to the storage tanks.

Figure 12. COMPONENTS OF THE RECLAMATION SYSTEM FOR TRI-ARYL PHOSPHATE ESTER FLUID USED IN THE 7500-TON HYDRAULIC PRESS.

116166(U)

Figure 13. RECLAMATION SYSTEM FOR THE INSULATING OIL COOL-
ANT SYSTEM.

The adsorber is 8 inches in diameter and 30 inches long and contains 30 pounds of Type 4A Molecular Sieves (8 x 12-mesh beads). A 2.5-gallon-per-minute flow is maintained through the adsorber, equivalent to a flow velocity of 0.95 foot per minute. The 1000-gallon oil system passes one cycle through the adsorber in 66.6 hours.

This reclamation system replaced a flash vaporization system that caused a building fire. The flash vaporization system required that the oil be heated to 175° F and then sprayed over a vacuum chamber to remove moisture and other contaminants. The vacuum pump oil required changing every two days due to damage from the oil vapors from the reclamation system that entered the pumps. Floor space requirements for this system was 70 square feet.

PI Pressure Indicator

Figure 14. COMPONENTS OF THE RECLAMATION SYSTEM FOR THE INSULATING OIL COOLANT SYSTEM.

The Molecular Sieve adsorber does not require heating the oil nor the use of any vacuum pumps. In addition, the unit was installed on the building wall and required very little floor space. Complete analysis of the oil is performed on a three-month basis; however, the dielectric strength is checked monthly. The Molecular Sieve adsorber constantly maintained the dielectric strength of the oil at 35 kv for 16 months before it required regeneration. The regeneration procedure of this unit is done at the Experimental Test Facility and requires the unit to be out of service for 24 hours. The savings in electric power for operating the sieve adsorber versus the flash vaporization system has been \$1250/year. There has been no maintenance on the sieve system and there is no fire hazard in the operation of the unit. The cost of installation was \$475.

Molecular Sieve adsorbers have also proved to be very economical at the AEC's Paducah Plant, operated by Union Carbide Corporation-Nuclear Division. A self-cooled, 14-kv grounding transformer contained oil with a dielectric strength of 30 kv and 37 ppm total water content. This unit was deenergized and the oil passed through a Molecular Sieve adsorber.⁽⁴⁾ In 20 hours the dielectric strength was increased to 33kv and the total water reduced to 17 ppm. A field test on an energized power transformer (100,000 kva, 161,000/14,000 volts) indicated that moisture could be removed from the transformer oil. Therefore, if a transformer requires only a conventional "dry out", the operation can be performed with a Molecular Sieve adsorber. This work would eliminate much expense in labor and a 30-day outage time to perform a conventional "dry out".

VACUUM PUMP OIL RECLAMATION SYSTEM

A vacuum pump Molecular Sieve reclamation system (Figures 15 and 16) refines the oil for a complex of mechanical vacuum pumps. All vacuum pump oil is directed to a dirty oil tank, then pumped at the rate of three gallons per minute through a ten-micron filter, the Molecular Sieve adsorber, a one-micron filter, and through three separate lines to three clean oil tanks. The clean oil tanks have overflow lines which are directed back to the dirty oil tank. The reclamation system operates continuously to refine the dirty oil. The filters are of the replaceable cartridge type. The adsorber is 10 inches in diameter and 42 inches in length and contains 71 pounds of Type 4A Molecular Sieves (8 x 12-mesh beads). The three-gallon-per-minute fluid flow through the adsorber is equivalent to a fluid velocity of 0.49 foot per minute. The sieve adsorber is regenerated with instrument air (-75° F dew point measured at atmospheric pressure) at 450° F and with 15 psig steam at 235° F.

Due to the large quantity of free and dissolved water, visible sludge deposits, and a high neutralization number, the filters have required changing every 30 days and the Molecular Sieve adsorber has been regenerated every 60 days. All filter cartridges are oven dried at 175° F prior to installation and the Molecular Sieve adsorber is regenerated at the Experimental Test Facility.

119494(U)

Figure 15. RECLAMATION SYSTEM FOR THE MINERAL-BASE OIL USED IN THE VACUUM PUMPS.

Table 7 presents a tabulation of the oil analyses performed on this system.

This Molecular Sieve reclamation system replaced a flash vaporization-clay filtration system which had high maintenance and operating costs. These costs were quite prohibitive in keeping the oil clean. Sludge deposits were accumulated in all vacuum pumps and pump maintenance costs were excessive. The oil in the entire system was being replaced once each month. Since installation of the Molecular Sieve unit in November 1965, none of the oil has been replaced. The savings in operating, maintenance, oil, and utilities costs per year are \$4000. This total does not include the possible savings in pump maintenance due to clean oil operation.

ISOSTATIC PRESS OIL RECLAMATION SYSTEM

The isostatic press reclaiming system (Figures 17 and 18) was designed to maintain both mineral oil systems used in the press operation (SAE 5 and SAE 20 mineral oil). When one system has been refined to reduce the moisture content to a very low range (10 - 20 ppm), the filters are removed from the filter casing and the casing of each filter is steam cleaned and air dried; the Molecular Sieve adsorber is also regenerated. The flexible hose couplings are then changed to the other oil system and the unit is placed into service.

This reclamation system has regeneration facilities for steam cleaning and air drying. Each of the ten and one-micron filters contains 12 replaceable cartridges. The adsorber is 10 inches in diameter and 36 inches long and contains 60 pounds of Type 4A Molecular Sieves. This unit is operated for 16 hours, five days/week and requires eight hours for one cycle. The flow rate of 2.5 gallons per minute is equivalent to a flow velocity of 0.5 foot per minute.

Figure 16. COMPONENTS OF THE RECLAMATION SYSTEM FOR THE MINERAL-BASE OIL USED IN THE VACUUM PUMPS.

Table 7
VACUUM PUMP OIL ANALYSES

Date of Analysis	Dirty Oil Tank				Effluent Molecular Sieves		
	Free Water Observed?	Sludge Observed?	Water Content (ppm)	Neutralization Number (mgs KOH/gm)	Color	Water Content (ppm)	Neutralization Number (mg KOH/gm)
11/10/65	Yes	Yes	6,300	1.59	Clear	20	0.064
11/12/65	Yes	Yes	1,000	0.194	Clear	40	0.026
11/16/65 ⁽¹⁾	Some	Some	1,100	0.088	Clear	30	0.026
11/17/65	No	No	112	0.060	Clear	7	0.02
12/1/65	No	No	5,767	0.128	Clear	115	0.044
12/16/65	No	No	425	0.043	Cloudy	225	0.022
Molecular Sieves Regenerated							
12/21/65	No	No	780	-	Clear	82	-
1/3/66	No	No	98	-	-	8	-
1/11/66	No	No	53	0.022	Clear	12	0.021
1/19/66	No	No	687	0.041	Clear	21	0.021
2/1/66	Yes	Yes	2,374	0.034	Clear	18	0.026
2/7/66	No	No	152	0.06	Clear	20	0.02
2/16/66	Yes	Yes	1,012	0.11	Clear	500	0.08
2/23/66	No	No	744	0.083	Clear	56	0.063
3/11/66	No	No	750	0.27	Cloudy	267	0.084
Molecular Sieves Regenerated							
4/18/66	Yes	Yes	19,900	0.490	Clear	53	0.051
5/5/66	Yes	Yes	-	-	Clear	-	-

(1) A sample of the effluent oil was tested in a test vacuum pump. In 15 minutes the vacuum obtained was 10 microns. A similar test of new oil gave a vacuum of 10 microns in 10 minutes. Before refining the oil it required 30 minutes to obtain 20 microns of vacuum.

This system was installed in January 1966 and replaced three flash vaporization-clay filtration units. These units were a constant maintenance problem. The Experimental Test Facility studies had indicated that improvement could be made in the fluid using the Molecular Sieve unit. Table 8 is a tabulation of the data obtained from the analyses of the oil.

The neutralization number data indicate that there are compounds present in this particular oil that were not removed by the flash vaporization-clay filtration units nor have they been removed by the Molecular Sieve adsorber. However, these compounds do react with the sodium butylate used in the neutralization number test and appear as acidity or other compounds that will react with sodium butylate. A study was made to identify these compounds and to find a way to remove them. A laboratory test column was set up to determine if removal could be made by the following methods:

119495(U)

Figure 17. RECLAMATION SYSTEM FOR THE MINERAL-BASE OILS USED IN THE ISOSTATIC PRESS.

Table 8
ISOSTATIC PRESS OIL ANALYSES

Date of Analysis	Sample Taken	Water Content (ppm)	Neutralization Number (mg KOH/gm)
2/5/66(1)	1600-Gallon Tank	83	0.43
2/7/66	Sieve Effluent	12	0.45
2/9/66	Sieve Effluent	19	0.45
2/16/66	1600-Gallon Tank	21	0.53
2/16/66	Sieve Effluent	14	0.47
2/23/66	Sieve Effluent	18	0.42
3/11/66	Sieve Effluent	28	0.49
4/18/66	Sieve Effluent	65	0.62
5/4/66	Sieve Effluent	18	0.47

(1) This sample represents the effluent from the flash vaporization-clay filtration units.

1. The oil was passed through a column of regenerated pelletized activated alumina. (Neutralization numbers before and after indicated no change.)
2. A mixture containing about 0.5 percent water in oil was premixed and then passed through a column of the regenerated activated alumina. (The neutralization number after the oil was passed through the column indicated a 50 percent decrease.)

PI Pressure Indicator
TI Temperature Indicator

Figure 18. COMPONENTS OF THE RECLAMATION SYSTEM FOR THE MINERAL-BASE OILS USED IN THE ISOSTATIC PRESS.

3. Ten milliliters of water were mixed with 50 milliliters of oil, then centrifuged. (The neutralization number was very low, indicating about a 90 percent reduction.)
4. Oil was passed through a wetted bed of pelletized activated alumina. (The neutralization number was reduced by 50 percent.)
5. Oil was passed through a bed of diatomaceous earth. (The neutralization number was not affected.)

A second sample of the oil was thoroughly washed with water and then centrifuged. The water extracted from the mixture was then evaporated and the residue analyzed on an infrared analyzer. This analysis indicated that the residue was a paraffin or a wax that is soluble in water and not actually an acid. It has not been determined how this material entered the oil system or if it is a compound resulting from an oxidation of the oil.

"BONUS" NUCLEAR REACTOR HYDRAULIC FLUID RECLAMATION SYSTEM

The BONUS reactor reclamation system⁽⁵⁾ is shown in Figures 19 and 20. This side-stream system continuously processes 100 gallons of tri-aryl phosphate ester hydraulic fluid at a rate of three gallons per minute through a one-micron filter, a

116170(U)

Figure 19. RECLAMATION SYSTEM FOR THE TRI-ARYL PHOSPHATE ESTER FLUID USED IN THE "BONUS" NUCLEAR REACTOR PRESSURE CONTROL SYSTEM.

Figure 20. COMPONENTS OF THE RECLAMATION SYSTEM FOR THE TRI-ARYL PHOSPHATE ESTER FLUID USED IN THE "BONUS" NUCLEAR REACTOR PRESSURE CONTROL SYSTEM.

Molecular Sieve adsorber, a one-micron filter, and a rotameter. Each filter consists of six cotton replaceable cartridge-type tubes. The adsorber contains 60 pounds of Type 4A Molecular Sieve (8 x 12-mesh beads).

This system is a portable reclamation unit and was shipped from the Y-12 Plant to the BONUS Site^(a) to alleviate problems which had occurred for several months in the reactor pressure control system. Laboratory analyses revealed a high moisture content (~2000 ppm compared to a recommended maximum of 100 ppm) in the hydraulic fluid which was used in the servo-valve system which activates the major reactor steam valves. The excessive moisture hydrolyzed the ester to yield an organic acid form. The acid, in turn, corroded the steel components of the system to produce abrasive corrosion products in the form of fine particles that eroded the servo-valve metering surfaces. Excessive leakage at the high-pressure drop ($\Delta P = 3000$ psi) through the scored valves caused the hydraulic fluid to overheat which led to its partial decomposition and a buildup of sludge in the valves.

Since the reclamation system was installed in April 1965, no operating difficulties with the hydraulic system have been encountered. Table 9 is a tabulation of the laboratory analyses of the BONUS hydraulic fluid.

Table 9
LABORATORY ANALYSES OF "BONUS" REACTOR HYDRAULIC FLUID

Date of Analysis	Water Content (ppm)	Neutralization Number (mg KOH/gm)	Interfacial Tension (dynes/cm)
4/1/65 ⁽¹⁾	2010	0.043	22.7
4/2/65 ⁽²⁾	1180	0.043	23.0
4/6/65	80	0.032	23.3
5/4/65	10	0.021	23.6
7/15/65	10	0.022	-
9/22/65	19	0.018	-
10/27/65	71	0.037	-
11/16/65 ⁽³⁾	-	-	-

(1) Dirty fluid from sump.

(2) New fluid which was used to replace the dirty fluid.

(3) New Molecular Sieve desiccant installed in the vessel since low pressure steam for regeneration was not available.

(a) BONUS Reactor is located in Puerto Rico.

PORTABLE OIL RECLAMATION SYSTEM

Figure 21 shows a portable unit. Figure 22 is a flow diagram of the unit used to refine the oil in 18 tape-controlled machines. This system has a spare Molecular Sieve adsorber that is regenerated and in standby. When the adsorber on the portable unit is depleted it is disconnected at two flanges and the standby unit installed. The depleted unit is then regenerated at the Experimental Test Facility and placed in standby.

118207(U)

Figure 21. PORTABLE OIL RECLAMATION SYSTEM.

Another portable sieve unit has been used to refine all types of oil. It has been used to remove water, acidity, and dirt from 6500 gallons of transformer oil and 2000 gallons of tri-aryl phosphate ester fluid, which has resulted in savings of \$10,500 to the plant. It is now refining 5000 gallons of transformer oil to be returned to the plant oil system.

Figure 22. COMPONENTS OF THE PORTABLE OIL RECLAMATION SYSTEM.

PROPERTIES OF THE MOLECULAR SIEVE DESICCANT

CHEMISTRY

Molecular Sieves are synthetic crystalline zeolites of metal aluminosilicates. In contrast to other adsorption materials, the pores of any of the particular types of Molecular Sieves are precisely uniform in size and of molecular dimensions. Depending upon the size of these pores, a molecular structure may be readily adsorbed, it may be slowly adsorbed, or can be completely excluded from adsorption. Sieves retain adsorbates by strong physical forces rather than by chemisorption. The external surface area of Molecular Sieves, about one percent of the total surface area, is available for the adsorption of molecules of all sizes. However, the internal area is available only to those molecules small enough to enter the pores. The adsorption and desorption of molecules from Molecular Sieves are reversible, and when the adsorbate is driven off, the crystal does not deform or collapse.

Not only will sieves adsorb molecules by size and configuration, they will adsorb molecules based on polarity or degree of unsaturation. In a mixture of molecules that are small enough to enter the pores, the less volatile, the more polar, or the more unsaturated the molecule, the more firmly the molecule is held in the crystalline structure.

Molecular Sieves are available in many types (3A, 4A, 5A, 10X, and 13X). These materials may be obtained either in the powder, bead, or pellet form, depending upon the requirements. The material evaluated and now used for oil purification is Type 4A, 8 x 12-mesh beads, with an effective pore opening of 4.2 angstroms. The basic unit cell formula is: $\text{Na}_{12}[(\text{AlO}_2)_{12}(\text{SiO}_2)_{12}] \cdot 27\text{H}_2\text{O}$. This bead form of the material contains about 20 percent inert clay binder.

REGENERATION OF MOLECULAR SIEVES

Regeneration of Molecular Sieves is required when influent and effluent sample analyses of the undesirable adsorbants show essentially no decrease across the sieve unit. The sieve unit may be regenerated in place if such a facility is large enough to warrant a steam supply and heated air source. In the case of small or portable systems, only one source of regeneration is necessary. The sieve units are removed from the piping or pulled by truck to the regeneration facility.

When it is determined the sieve unit requires regeneration, it is isolated and drained free of the fluid. This material may be returned to the system to reduce loss. A low-pressure steam supply (10 psig or less at 230 - 280° F) is valved into the bed to "strip" or "wash" the fluid and any other sludge formation from the sieve bed. Steam flow may be in either direction, upstream or downstream, depending upon the adsorption

cycle flow and quantity of sludge accumulated on the sieve material. The steam stripping process continues until samples of the condensate show no visible traces of fluid.

After the steaming process, dry, oil-free instrument air (< 50 ppm water) is passed through the bed to remove the excess moisture and partially dry the bed. Again, the time required for this phase of regeneration depends upon the size of the unit. After partial drying, the dry instrument air is preheated by means of an electric heater; then passed through the bed in a counterflow direction to the adsorption cycle. The air temperature is usually controlled around 350° F entering the bed but can be elevated to 600° F without damage to the sieve material. As a safety precaution, it is good practice to keep the air temperature at least 50° F below the flash point of the fluid being purified. Then, should the steam cycle not be complete or inadvertently not performed, the chance of a fire hazard is reduced. The air flow required for each system will vary according to the size of the sieve bed and heat losses. By trial and error, air flow is adjusted to maintain the heater energized and to obtain the highest effluent air temperature possible for a particular bed. The bed is regenerated until the highest effluent temperature is obtained and is maintained at this level for 30 minutes. If wet air and low temperatures are used, the capacity of the adsorbing cycle is significantly reduced.

Cooling of the Molecular Sieve bed may be accomplished either by convection or by turning off the electric heater and allowing the air to flow through the sieve bed. However, in convection cooling, air pressure should be applied to that part of the bed in the normal direction of fluid flow. Upon cooling, the sieve will create a vacuum and the dry-air source will eliminate moisture inleakage during the cooling.

If the bed requires cooling at the termination of the heating cycle, the cool dry air should be purged through the bed in the opposite direction from the heating cycle. When the effluent air temperature is approximately 100° F, the unit is cooled sufficiently. Convection cooling is preferred since air-flow cooling tends to load a small amount of moisture in the bed.

SERVICE LIFE

Experimental studies have not been performed during this investigation to determine the number of cycles of regeneration with steam and hot air that will cause degradation of the Molecular Sieve material. The Experimental Test Facility Molecular Sieve desiccant has been used in the investigation of all types of oils and phosphate ester fluids. It has been regenerated approximately 50 times in 4 1/2 years with no significant change in adsorption capacity. The vendor's literature⁽⁶⁾ states that units used in drying other hydrocarbon fluids can be designed on a basis of 3000 cycles.

RECLAMATION SYSTEM DESIGN CONSIDERATIONS

GENERAL

In the design of a Molecular Sieve oil reclamation system, a detailed study should be made to determine the following:

1. The type of equipment in which the oil is to be used (eg, electrical transformers, mechanical vacuum pumps, hydraulic press, servo controls).
2. The type of oil that is to be used and the chemical and physical characteristics of the oil.
3. The amount of oil in the system including storage tanks, head tanks, and equipment.
4. The present condition of the oil (eg, free and dissolved water content, acidity, interfacial tension, sludge, particulate matter content).
5. The method in which the oil is stored (eg, open head tanks, tanks vented to atmosphere, closed tanks with dry inert gas blanket over the liquid, vented tanks with a desiccant installed in the vent line).

The method of oil storage is of primary importance. If a vent line is extended outside of the operating building, provisions should be made to prevent snow or rain from entering the vent. If an oil return line empties into a storage tank, it should extend well below the lowest operating level of the tank to prevent splashing. Any unnecessary agitation causes more of the oil to come in contact with the moist atmospheric air.

TYPES OF RECLAMATION SYSTEMS

After analyzing the general conditions, the type of reclamation system best suited for the particular service can be determined. One of the following three types of systems will usually suffice:

Side Stream System

A side stream system is usually best suited for applications such as hydraulic press installations, servo control systems, and mechanical vacuum pump installations. In these systems a small amount of water continually enters the oil systems and should be removed as it accumulates.

Portable Reclamation Systems

Portable systems have performed well in removing water and acid from transformer oil. The water loading is usually low since water adversely affects the dielectric strength of the oil. A system of this type can be mounted on a trailer for towing to various transformer sites. During the reclamation period it is not necessary to take the transformer out of service. Periodic samples should be analyzed to determine when the water and acid contents have been sufficiently reduced and the dielectric strength has been restored.

Small portable units have also been used to reclaim oil on tape-controlled lathes and similar equipment. This unit is mounted on a small dolly and is used to service a number of machines.

Central Station System

A permanent and centrally located system may prove more economical for the reclamation of oil or the regeneration of Molecular Sieves from smaller systems which are scattered throughout the plant. Dirty oil can be transported to the central station in tank trucks where it can be circulated directly from the truck, through the Molecular Sieve reclamation system, and back into the truck, or dirty and clean storage tanks can be installed at the central station.

Small vessels containing Molecular Sieves can be detached from the particular system in which they are installed and transported to the central station. Usually they can be regenerated and placed back into service in an eight-hour shift.

In summary, it is rarely justified to install a large and expensive reclamation system for the services just described. The best practice is to keep the oil clean and to remove moisture and acid as they accumulate. Even on systems that have been in service for several months and have accumulated large quantities of water, it is not necessary to install large reclamation units. Regeneration of the Molecular Sieves after they have become saturated is inexpensive and can be performed by a competent chemical operator.

DESIGN PROCEDURE

The design of an oil reclamation system for a 7500-ton hydraulic press will serve to outline the general approach used in sizing a "side stream" reclamation system. The hydraulic press has a head tank with a submerged inlet pipe and an open vent into the building. Raising and lowering the ram causes the oil level in the head tank to fluctuate.

PARAMETERS

Initial Conditions

Volume of Oil in System - 7700 gallons

Water Content (free and dissolved) - 600 ppm by weight

Type of Oil - tri-aryl phosphate ester

Specific Gravity - 1.1514

Total Water = $7700 \text{ gal} \times 8.34 \text{ lbs/gal} \times 1.1514 \times 6 \times 10^{-4} = 44.4 \text{ lbs}$

Condition after Reclamation

Water Content (free and dissolved) - < 20 ppm

Total Water = $7700 \text{ gal} \times 8.34 \text{ lbs/gal} \times 1.1514 \times 2 \times 10^{-5} = 1.6 \text{ lbs}$

Pounds of Molecular Sieve Desiccant Required

Based on pilot plant studies and vendor's literature,⁽⁶⁾ a weight percent of approximately 16 was used for design. (Weight Percent = Pounds of Water Adsorbed per 100 Pounds of Desiccant).

Pounds of Desiccant = $44.4 \text{ lbs of water} \times 100 \text{ lbs of desiccant} / 16 \text{ lbs of water} = 278 \text{ lbs.}$

Desiccant Vessel Size

A superficial velocity (velocity of the liquid through the empty vessel) of 0.5 - 1.0 foot per minute is recommended for drying fluids not miscible with water.⁽⁷⁾

Height-to-Diameter Ratio

The height of the vessel should be two to three times the diameter and in no case less than 30 inches.

With these parameters, a standard Schedule 20 18-inch OD pipe (17.25-inch ID) was investigated using a pump with a capacity of 10 gallons per minute.

$$V = \frac{Q}{A},$$

where:

V represents the velocity in feet per minute,

Q the flow rate in cubic feet per minute, and

A the cross sectional area.

$$Q = \frac{10 \text{ gal/min}}{7.48 \text{ gal/ft}^3} = 1.33 \text{ ft}^3/\text{min}, \text{ and}$$

$$A = 1/4 \pi D^2 = (1/4)(3.142)(17.25)^2 = 2332.8 \text{ in}^2 = 1.62 \text{ ft}^2.$$

Therefore:

$$V = \frac{Q}{A} = \frac{1.33 \text{ ft}^3/\text{min}}{1.62 \text{ ft}^2} = 0.825 \text{ ft/min}.$$

This value is within the velocity limits of 0.5 to 1.0 foot per minute.

Molecular Sieve Type 4A (8 x 12 mesh) has a bulk density of 45 pounds per cubic foot. If 278 pounds of desiccant are needed, the height of the vessel must be:

$$\frac{278 \text{ lbs}}{1.62 \text{ ft}^2 \times 45 \text{ lbs/ft}^3} = 3.8 \text{ ft} = 45.6 \text{ in}.$$

Therefore:

$$\text{Height-to-Diameter Ratio} = 46 \text{ inches}/17.25 \text{ inches} = 2.7.$$

An 18-inch-diameter by 42-inch-long vessel was fabricated from Schedule 20 steel pipe with standard weld caps for ends.

$$\text{Cycles per Day} = \frac{(10 \text{ gals/min})(60)(24)}{7700 \text{ gals}} = 1.87.$$

This unit has been in continuous service since February 1964 and will reduce the water content to < 20 ppm in 7 days. At the end of eight months, laboratory analysis indicated that the desiccant had reached equilibrium and was no longer removing water; therefore, the system was removed from service and regenerated.

REGENERATION

In order to reuse the desiccant, the adsorber unit is regenerated to remove the water and acids adsorbed on the desiccant and to remove sludge deposits, dirt, and lint.

Frequency

The frequency of regeneration is based on several factors:

1. The maximum water content allowed for the particular service.
2. The total water content at the time the reclamation system is placed in service.
3. The amount of desiccant installed in the system.
4. The rate of water accumulation in the system.
5. The amount of residual water remaining in the desiccant after the previous regeneration.

The regeneration of a desiccant used in oil reclamation requires that the desiccant be first washed with steam to remove all of the oil from the beads. This process prevents the oil from forming a hard crust and "blinding" the desiccant when the desiccant is heated to remove the moisture. The steam wash also removes any sludge that may have accumulated.

After washing with steam, the desiccant is heated by using dry, oil-free air. The air first passes through an electric heater then into the desiccant vessel.

Temperature of Regeneration Air

The temperature of the oil-free air used in regeneration is usually maintained at approximately 50° F below the flash point of the oil being reclaimed to prevent the possibility of a fire in the event all of the oil is not completely washed from the desiccant vessel. The temperature and dew point of the air determine the amount of residual moisture left in the desiccant.⁽⁸⁾ For example, if the desiccant is heated to 300° F with air having a dew point of -75° F (measured at atmospheric pressure), the residual moisture is two weight percent or approximately 12 percent of the useful capacity of the desiccant. If air having a dew point of +35° F is used at 300° F, the residual weight percent is 5.8 or approximately 36 percent of the useful capacity of the desiccant. It is therefore desirable to use air that has the lowest dew point available. Other gases may be used for regeneration such as nitrogen provided the hot gas does not form a flammable mixture with any residual oil that may remain in the desiccant vessel. Vacuum regeneration may be possible.

The desiccant vessel is usually insulated to obtain uniform heat throughout the bed. When the effluent gas temperature is within 20 to 50° F of the influent temperature, most of the moisture has been driven from the desiccant. The low-dew-point air flow is maintained until the desiccant has cooled to room temperature. The direction of flow for the hot air regeneration is the same as when the unit was in service (downward). To prevent atmospheric air from entering the vessel during the cooling cycle, a pressure of five to 10 psig of low-dew-point air is maintained in the vessel until it is placed back in service. This step is particularly important if the desiccant is regenerated at a central station and transported to the site.

The desiccant, as received from the desiccant manufacturer, must be regenerated before it can be placed in service. However, since the desiccant has not been exposed to oil at this time, it is not necessary to steam wash the unit prior to purging with hot air.

OTHER DESIGN CONSIDERATIONS

Oil Temperature

The capacity of the Molecular Sieve desiccant is influenced by the temperature of the oil. At higher temperatures, the weight percent is reduced to as much as one half if the oil temperature entering the desiccant is increased from 100 to 220° F. Therefore, it is desirable to limit the oil temperature to approximately 120° F. Since the oil flow rate is low, a small heat exchanger is adequate. Oil-to-water heat exchangers must be leak tight to prevent the cooling water from contaminating the oil. Such heat exchangers should be inspected regularly.

Economy

If an oil reclamation unit is to be installed on an existing oil system that contains a large amount of water, the water may be removed in steps. The reclamation unit can be sized for the anticipated day-to-day moisture loading and according to the estimated regeneration period. First, allow the desiccant to become saturated at a high weight percent (~20%). Regenerate the desiccant and remove the remainder of the water and repeat the procedure. Once the moisture has been reduced, the reclamation may be used for normal moisture loading. This method of operation eliminates the need for large equipment that has no real use after the initial water has been removed.

If the oil system is small and the moisture loading requires only a small amount of desiccant, it is generally more economical to construct a central regeneration station. Instead of regenerating the desiccant at the site, it may be removed and transported to the central station for regeneration. Usually a small oil system does not accumulate water to the extent that the desiccant cannot be removed from service for regeneration.

AUXILIARY COMPONENTS

Screens

Screens are usually installed in the inlet and outlet of the vessel containing the desiccant. For smaller vessels, a removable upper screen may be adequate for desiccant replacement. For larger vessels which would be difficult to invert to remove the desiccant, both inlet and outlet screens should be removable (see Appendix B). The largest screen opening should be approximately one half the size of the smallest desiccant pellet to prevent loss of desiccant from the unit.

Filters

Filters should be installed on either side of the desiccant vessel along with a rotameter (see Figure 12, Page 31). The upstream filters remove sludge and particulate matter and prevent the desiccant bed from becoming clogged with solids. The desiccant also serves as an excellent filter. The downstream side of the desiccant vessel should contain a filter to prevent any desiccant carryover into the oil circulating system. The rotameter provides an inexpensive, direct-reading flowmeter. The flow may be controlled by direct throttling of a valve or by a simple bypass line from the discharge of the pump back to the suction of the pump. It may be desirable to arrange the upstream filters for two different sizes of particle removal. The first filter should be a roughing filter; the second filter a final filter. This arrangement provides for longer filter life and lengthens the time between filter changes. Units now in service have roughing filters of 10 to 1 microns and final filters of one micron. The filters on the downstream side of the desiccant vessel are usually one micron. Any filter medium may be used provided it is compatible with the oil being clarified. All units described in this report are of the cotton basket-weave type. When the filters are loaded they are removed and replaced with new ones.

Since it is assumed that all filter elements which are received from filter manufacturers have been exposed to wet atmospheric air, the filters should be heated to approximately 175° F for four to six hours and installed in the filter vessel before they are allowed to cool.

Pumps

A small gear pump (30 to 50 psig discharge pressure) is particularly well suited for this application. A built-in pressure relief valve installed as part of the pump or a separate relief valve installed directly downstream from the pump must be provided. Gear pumps are essentially positive displacement pumps and will build pressure rapidly should the flow be stopped due to plugged filters or incorrect valving.

Materials of Construction

Unless other design parameters dictate, only the usual carbon steel materials for the temperature and pressure encountered are required. Screwed fittings are difficult

to make leak tight in oil service, therefore, welded joints are recommended except at such locations as rotameters and pressure gauges. Valves adjacent to the desiccant vessel should be selected for a temperature of not less than 500° F to allow for regeneration. Figure 12, Page 31 shows the valving and instrumentation arrangements.

Safety

Always install the reclamation unit in a clean, well-lighted, and ventilated area. A detailed operating procedure with a flow diagram should be placed near the unit. The operator should be instructed in the operation of the unit, its purpose, and any safeguards deemed necessary. If the oil being processed is flammable, the plant policies governing such installations should be followed. Oil spills or leaks should be eliminated as soon as they are discovered. The system should be placed on the operator's check list and periodically observed unless automatic protective devices are installed such as high temperature and pressure cut outs and no-flow alarms. If the equipment on which the oil reclamation unit is installed is operated from a central control point, the flow and pressure of the oil reclamation unit may be extended to this area. No unusual safety hazards have been experienced with this type of equipment.

CONVENTIONAL OIL TESTS

INTRODUCTION

Throughout industry, large quantities of reclaimable oil are discarded; however, with a little knowledge of the composition and capabilities of the oil, needless waste can be eliminated. Also, many oil systems are never tested which causes high maintenance costs to expensive equipment. If laboratory tests can minimize these costs or can indicate if refining is advantageous, they can be easily justified and should be included in any plant oil reclamation program.

There are many conventional tests that may be performed on an oil system: flash point, pour point, viscosity, specific gravity, dielectric strength, residue content, color, ash content, interfacial tension, neutralization number, turbine oil stability, oxidation stability,⁽⁹⁾ molecular structure, water content, corrosion product contamination, and oil life.⁽¹⁰⁾ However, should all of these tests be used in monitoring an oil system, laboratory costs could exceed the cost of new lubricants. The most common tests used for the various systems in the Y-12 Plant have been: water content, neutralization number, dielectric strength, interfacial tension, molecular structure, and corrosion product contamination. A description of these tests, listed in the order of importance, is given in the sections that follow.

TYPES OF TESTS

Water Content

Testing for water content is performed on all fluids. Most of the oxidation products formed in hydrocarbon oils are soluble in water and thus the water becomes acidic. Water will also act as a catalyst with iron and copper resulting in accelerated oxidation. If water is removed, there still may be oxidation products; however, most of the detrimental effects of oxidation products occur in the presence of water. Water is very detrimental in the fire-resistant fluids as it will hydrolyze the ester resulting in the production of phosphoric acid. The water-content test is expressed as total water in parts per million.

Neutralization Number

This value is determined on all types of lubricating fluids. An increase in acidity gives warning of possible trouble in any operating fluid. The acidity can cause corrosion in tanks and valves, resulting in erosion of other components. This test determines the number of milligrams of potassium hydroxide necessary to neutralize one gram of oil sample and is expressed as mgs KOH/gm of sample.

Dielectric Strength

Dielectric strength of transformer oils is a very good indication of the presence of free water. The various systems using this fluid demand that the dielectric strength be maintained above 30 kv.

Interfacial Tension

Testing for interfacial tension is used on all fluids to determine the orientation of the polar molecules at the water-oil interface. This value gives an indication of the extent of sludging. The test units are expressed in dynes per centimeter.

Molecular Structure

Analyses of new oil versus used oils by infrared spectrophotometry can detect any major changes in a system. This test has been used on vacuum pump oil studies to determine if the sludge composition found in these systems was due to the breakdown of the lubricant or some other cause.

Corrosion Product Contamination

Tests by atomic absorption give a very good determination of the amount of such metals as iron and copper that may have been solubilized in the oil due to high water and acid contents.

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APPENDIX A

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SPECIFICATIONS OF FLUIDS WHICH HAVE BEEN RECLAIMED

7500-TON HYDRAULIC PRESS PHOSPHATE ESTER FLUIDS

Type	Phosphate Ester	
Nature	Tri-aryl	
Ester Content (% min)	99	
Color (max, ASTM D155)	1.5	4
Viscosity (ASTM D445-53T; SUS at 100° F)	220-230	550-650
Density (gms/cc)	1.14	1.17
Pour Point (ASTM D97, ° F max)	0	20
Flash Point (ASTM D92, ° F min)	455	455
Fire Point (ASTM D92, ° F min)	665	665
Autogeneous Ignition Point (° F min, ASTM D286)	1150	1150
Hot Manifold Test (° F min, ASTM D286)	1300	1300
Neutralization Number (max, ASTM D974)	0.1	0.1
Sulphur (% max, wt)	0.001	0.001
Corrosion	0	0
Surface Tension (dynes/cm at 68° F, ASTM D971)	40	41
Interfacial Tension (dynes/cm, min, ASTM D971)	24	20
Entrained or Emulsified Water (% max, Karl Fischer)	0.1	0.1
Free Water and Sediment (% max, ASTM D96)	0	0
Foaming Tendency (ml max, 5-min blowing period at 75° F, ASTM D892)	5	10
Emulsion Characteristics (SAE Method 3201.5, Federal Stand- ard Number 791)		
Separation Time (minutes, max)	2	6
Foam Depth (ml max at separation)	0	4
Solubility at 23° C (percent by wt)		
Fluid in Water	0.002	0.001
Water in Fluid	0.61	0.84
Hydrolysis (16 hrs in water at 210° F, mg KOH max)	0.001	0.06

INSULATING OIL

Nature of Base Oil	Straight Petroleum
Oxidation Inhibited	No
Water and Sediment (ASTM D96)	Nil
Viscosity (ASTM D88, SUS at 100° F)	58 - 65
Specific Gravity at 60/60° F	0.86 - 0.91
Color (ASTM D1500, max)	1
Flash Point (ASTM D92, ° F min)	290
Pour Point (ASTM D97, ° F min)	-40
Dielectric Strength Upon Delivery at Destination (ASTM D877, kv min)	30
Interfacial Tension (ASTM D878)	35 - 55
Neutralization Value (ASTM D664, tan max)	0.03
Free Sulfur (ASTM D989)	Nil
Power Factor at 25° C (ASTM D924, max)	0.10%
Aniline Point (ASTM D611, ° C)	70 - 78
Sludge Accumulation at 120° C (ASTM D1314, % max)	
3 Days	0.08
7 Days	0.18
14 Days	0.30

MECHANICAL VACUUM PUMP OIL

Nature of Base Oil	Straight Mineral
Type of Base Oil	Paraffinic
Oxidation Inhibited	No
Corrosion Inhibited	No
Compounding	None
Water and Sediment (ASTM D96, % max)	Nil
Viscosity (ASTM D88, SUS at 100° F)	290 - 325
Viscosity (ASTM D88, SUS at 210° F)	51 - 53
Viscosity Index (ASTM D567, min)	90
Gravity (ASTM D287, min)	26
Color (ASTM D1500, max)	L 2.0
Flash Point (ASTM D92, ° F min)	430
Fire Point (ASTM D92, ° F min)	460
Pour Point (ASTM D97, ° F max)	20
Vapor Pressure at 150° F (mm Hg, max)	0.002
Carbon Residue (either method)	
(ASTM D189, % max)	0.03
(ASTM D524, % max)	0.11
Ash (ASTM D482, % max)	Nil
Sulfur (ASTM D129, % max)	0.03
Neutralization Number (ASTM D664, total acid max)	0.01
Foam (ASTM D892, 5 min blow at 75° F, mls/max)	10
Precipitation (ASTM D91, max)	0.00,
Corrosion (ASTM D130, 3 hrs at 210° F)	1
Moisture as Emulsion (Karl Fischer, % max)	Nil

ISOSTATIC PRESS MINERAL OIL
(Medium Viscosity)

Nature of Base Oil	Straight Mineral
Type of Base Oil	Paraffinic
Oxidation Inhibited	Yes
Corrosion Inhibited	Yes
Compounding	None
Viscosity (ASTM D88, SUS at 100° F)	290 - 325
Viscosity (ASTM D88, SUS at 210° F)	52 - 55
Viscosity Index (ASTM D567, min)	100
Water and Sediment (ASTM D96, % max)	Nil
Gravity (ASTM D287, min)	30
Color (ASTM D1500, max)	2.5
Flash Point (ASTM D92, ° F min)	435
Fire Point (ASTM D92, ° F min)	485
Pour Point (ASTM D97, ° F max)	5
Carbon Residue (either method)	
(ASTM D189, % max)	0.03
(ASTM D524, % max)	0.11
Ash (ASTM D482, % max)	Nil
Moisture as Emulsion (Karl Fischer, % max)	0.03
Sulfur (ASTM D129, % max)	0.19
Corrosion Appearance after 3 Hrs at 210° F (both) Copper (ASTM D130, max)	0
Steel (ASTM D66) Distilled Water	Bright
Steel (ASTM D66) Synthetic Sea Water	Bright
Foaming (ASTM D892, 5 min blow at 75° F, mls/max)	10
Precipitation Number (ASTM D91, max)	0.00
Neutralization Number (ASTM D664, tan max)	0.07
Oxidation Stability (ASTM D943 hrs to neutralization number of 2.0)	2500

ISOSTATIC PRESS MINERAL OIL
(Low Viscosity)

Nature of Base Oil	Straight Mineral
Type of Base Oil	Paraffinic
Viscosity (ASTM D88, SUS at 100° F)	85 - 95
Water and Sediment (ASTM D96, % max)	Nil
Gravity (ASTM D287)	0.860 - 0.870
Color (ASTM D156, min)	30
Bloom	Nil
Flash Point (ASTM D92, ° F min)	330
Pour Point (ASTM D97, ° F max)	0
Carbonizable Substances (ASTM D565)	Pass
Precipitation Number (ASTM D91, max)	0.00
Moisture as Emulsion (Karl Fischer, % max)	0.02

"BONUS" REACTOR PRESSURE CONTROL SYSTEM
PHOSPHATE ESTER HYDRAULIC FLUID

Appearance	Light Green
Viscosity (SUS at 100° F)	230
Viscosity (SUS at 210° F)	43
Odor	None
Flash Point (° F)	485
Fire Point (° F)	690
Autoignition Point (° F)	Over 1200
Boiling Point (° F)	Over 750
Pour Point (° F)	0
Density (lbs/gal at 60° F)	9.5
Specific Gravity at 20/20° C	1.15
Hot Manifold Test (° F)	Over 1200
Neutralization Number	0.0

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APPENDIX B

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ADSORBER VESSEL SCREEN DETAILS

Figure B-1. ADSORBER VESSEL SCREEN DETAILS.