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STUDY OF A MOLECULAR SIEVE FIRE

C. R. Schmitt
J. R. DeMonbrun

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

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

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STUDY OF A MOLECULAR SIEVE FIRE

C. R. Schmitt
J. R. DeMonbrun

Oak Ridge Y-12 Plant

P.O. Box Y, Oak Ridge, Tennessee 37830

operated for the U.S. ATOMIC ENERGY COMMISSION
by UNION CARBIDE CORPORATION—NUCLEAR DIVISION
under Contract W-7405-eng-26

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ABSTRACT

A fire occurred in a molecular sieve column which was used to refine the fluid of a hydraulic press. The fire started spontaneously when hot air (400° F) was admitted to the column during a regeneration treatment. It was concluded that an accumulation of organic residues on the molecular sieves was probably responsible for the fire.

CONTENTS

SUMMARY	5
INTRODUCTION	6
STUDY OF A MOLECULAR SIEVE FIRE.	7
Study Procedure	7
Discussion	13
ACKNOWLEDGEMENTS	19
APPENDIX A	20
Mass Spectrometric Analysis of Molecular Sieve Materials	20
APPENDIX B	22
Standard Operating Procedure - Hydraulic Fluid Refining System for a 7500-Ton Press	22
APPENDIX C	26
Purchasing Specifications 11-AC-1, Code HC - Lubricants and Cutting Coolants	26

SUMMARY

A fire occurred in a sodium aluminosilicate molecular sieve column of a hydraulic press at the Oak Ridge Y-12 Plant^(a) which used a triarylphosphate hydraulic fluid. The molecular sieve bed had ignited upon admitting air at 400° F during a regeneration treatment. Laboratory analyses and studies have indicated that organic residues from the triarylphosphate accumulated on the sieves and that these were probably responsible for the fire.

As a safety precaution, it is recommended that the molecular sieves be replaced every six months, or annually, to prevent a dangerous accumulation of organic residues that could provide the ingredients for a damaging fire.

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Atomic Energy Commission.

INTRODUCTION

A 7,500-ton hydraulic press at the Oak Ridge Y-12 Plant uses a proprietary, fire-resistant triarylphosphate fluid which is pumped from the press reservoir through a refining system and then returned to the press reservoir. The refining system consists of a gear-type pump, a prefilter unit containing six cellulose filter cartridges, and a molecular sieve bed (8 - 12-mesh sodium aluminosilicate beads) for removing any water and acidity from the triarylphosphate hydraulic fluid.

Regeneration of the molecular sieve bed is accomplished by first washing the molecular sieves with low-pressure steam followed by passing instrument air at 400° F countercurrently through the sieve column. This regeneration procedure had been used effectively for the past nine years; however, on September 1, 1971, the molecular sieve bed ignited spontaneously upon admitting the hot instrument air during regeneration. Although no one was hurt, the fire produced such an intense heat that it burned through a section of steel piping located just below the molecular sieve column. A study was therefore made by development personnel in an attempt to determine the cause of this unexpected fire.

STUDY OF A MOLECULAR SIEVE FIRE

STUDY PROCEDURE

Miscellaneous analytical studies were performed on selected samples of the triarylphosphate hydraulic fluid, molecular sieves, and cellulose filter cartridges that were obtained from the building refining system immediately after the fire. A photomicrograph showing a typical fused cluster of molecular sieves removed from the bottom of the molecular sieve column where the fire was most intense is presented in Figure 1. As shown, some of these beads exhibited an unburned residue of carbon, indicative of the presence of some organic compound which served as a carbon precursor. Results of the carbon and spectrographic chemical analyses of the molecular sieves sampled from the top and bottom sections of the molecular sieve column are given in Table 1.

An attempt was made by chromatographic, solvent extraction, and infrared techniques to separate and identify any organics other than the triarylphosphate that might have been present in the used hydraulic fluid. Table 2 summarizes the alumina-chromatographic adsorption and solvent extraction test results obtained on five-milliliter samples of the

H-510

Figure 1. FUSED CLUSTER OF MOLECULAR SIEVE BEADS TAKEN AFTER THE FIRE. (5X)

Table 1
ANALYSES OF THE MOLECULAR
SIEVES AFTER FIRE

Carbon (%)	Top of Bed 4.52	Bottom of Bed 2.41
Spectrographic Results (%)		
Ag	< 0.005	< 0.005
Al	12	8
Au	< 0.02	< 0.02
B	< 0.01	< 0.01
Ba	< 0.002	< 0.002
Be	< 0.001	< 0.001
Bi	< 0.01	< 0.01
Ca	0.6	0.6
Cd	< 0.04	< 0.04
Co	< 0.005	< 0.005
Cr	< 0.01	< 0.01
Cs	< 0.6	< 0.6
Cu	0.015	0.005
Fe	0.8	0.8
Hf	< 0.02	< 0.02
K	0.2	0.3
Li	< 0.02	< 0.02
Mg	2.2	2.2
Mn	0.012	0.02
Mo	< 0.002	< 0.002
Na	5	6
Nb	< 0.02	< 0.02
Ni	< 0.01	< 0.01
Pb	< 0.01	< 0.01
Rb	< 0.15	< 0.15
Sb	< 0.04	< 0.04
Si	> 20	> 20
Sn	< 0.005	< 0.005
Ta	< 0.15	< 0.15
Th	< 0.04	< 0.04
Ti	0.05	0.06
U	< 0.3	< 0.3
V	< 0.005	< 0.005
W	< 0.08	< 0.08
Zn	< 0.08	< 0.08
Zr	0.002	0.002

hydraulic fluid sampled from the inlet cellulose cartridge filter of the refining system after the fire. As reported in Table 2, one extraction test gave evidence of a residue product having a strong phenolic or cresolic odor. An infrared spectrogram made on the ether-alcohol extract (Figure 2) could not be identified in the Sadtler series of standard infrared spectra, but was significantly different from that of the proprietary triarylphosphate fluid.

Thermogravimetric, differential thermal, and quadrupole mass spectrometric analyses were conducted on the molecular sieves removed from the refining system in an attempt to identify any organic species that may have been present. Results, as noted in Table 3, showed the presence of Masses 44, 32, 28, and 18 which are indicative of carbon dioxide (44), oxygen (32), nitrogen or carbon monoxide (28), and water (18). These components were definitely higher than normally found in mass spectrometer analyses of this type. A typical thermogravimetric analysis-differential thermal analysis (TGA-DTA) scan of the molecular sieves sampled from the refining unit after the fire is given in Figure 3. Both of the nonfused molecular sieve samples taken from the top and bottom of the column showed a weight loss and an endotherm at a temperature range of 62 - 179° C. In a TGA-DTA scan of the fused molecular sieve beads, no such endotherm (or exotherm) was observed.

These data indicate that the presence of some organic material, possibly phenol or cresol or a derivative thereof, had accumulated on the molecular sieve column over the past nine years of operational use and was ignited by the hot air (400° F) during the regeneration treatment.

Semiquantitative spectrographic and X-ray diffraction analyses of solid particulates sampled from the inlet cellulose filter cartridge of the hydraulic fluid refining unit are reported in Table 4. These data indicate the presence of iron, iron oxide, and silicate-type inorganics, as expected. A photograph showing the appearance of the inlet and outlet filter cartridges after the fire is presented in Figure 4.

Table 2

SUMMARY OF THE ALUMINA-CHROMATOGRAPHIC COLUMN, SOLVENT EXTRACTION, AND
INFRARED TESTS WITH THE HYDRAULIC FLUID REMOVED
FROM THE REFINING SYSTEM AFTER THE FIRE

Fraction Number(1)	Solvent	Solvent Volume (mls)	Observations	Infrared Spectrogram
1	Petroleum Ether	10	No detectable solute phase.	Corresponds to triarylphosphate.
2	Petroleum Ether	50	No detectable solute phase.	Corresponds to triarylphosphate.
3	Benzene-Chloroform (50 vol % mixture)	75	No detectable solute phase. Clear liquid formed when solvent was removed by evaporation.	Corresponds to triarylphosphate.
4	Chloroform	75	Yellow-green fluorescence band observed. Viscous yellow liquid formed when solvent was evaporated.	Corresponds to triarylphosphate.
5	Ethyl Ether-Ethyl Alcohol (50 vol % mixture)	100	Evaporation of solvent gave a light-pink, glassy film which had a strong phenolic or cresolic odor.	Gave an unidentified infrared pattern (see Figure 2).
6	Demineralized Water	100	Evaporation of solvent left a clear film residue. No phenolic or cresolic odor detected.	Gave an unidentified infrared pattern corresponding to that of Fraction 5 except for one peak at the 950-micrometer wavelength.

(1) Five milliliters of hydraulic fluid used in each test.

Figure 2. INFRARED SPECTROGRAMS TAKEN FROM THE HYDRAULIC FLUID.

Table 3

THERMOGRAVIMETRIC, DIFFERENTIAL THERMAL,
QUADRUPOLE MASS SPECTROMETRY ANALYSES
OF MOLECULAR SIEVES FROM THE BUILDING
REFINING UNIT AFTER THE FIRE
(Flowing Dry Air at 5.7 l/hr; Heating Rate of 4° C/min)

	Weight Loss (%)	Mass Numbers
From Top of Column	12.8	44, 32, 28, 18
From Bottom of Column	15.0	44, 32, 28, 18
Fused Sieves (column bottom)	1.3	44, 32, 28, 18

atoms. This mass is often encountered in spectra of trimethylsilyl compounds and is assigned the formula:

When the black molecular sieves were heated to temperatures above 200° C, a peak corresponding to Mass 221 became significant.

High-resolution mass spectrographic analyses of the white molecular sieve beads that had been fused and clustered together at the bottom of the column after the fire showed only very-low-intensity spectra which were very similar to those of the black molecular sieve beads.

Hydraulic press operations were resumed during September 1971, using a new charge of molecular sieve beads in the triarylphosphate hydraulic fluid refining system. During December 1971, ten complete cycles of operation had been completed, which included ten consecutive adsorption cycles to remove any water and acid from the triarylphosphate fluid, ten treatments with low-pressure steam to wash out any residual acid and hydraulic fluid from the sieves, and ten regeneration treatments using instrument air preheated to 400° F. A complete description of the hydraulic fluid refining system and operational sequences on the sieves is given in Appendix B.

Samples of the molecular sieves were obtained during December 1971 after ten cycles of operation had been completed so that thermogravimetric, solvent extraction, and high-resolution mass spectrographic analyses could be made. High-resolution mass spectrographic analyses showed that a series of organic fragments corresponding to Masses 410 (the predominant peak), 424, 438, and 452 were present. Each number differed from the next by an atomic mass unit of 14 and corresponds to the splitting off of -CH₂ fragments, which have a molecular weight of 14.

Another series of organic fragments having Masses 305 (a very intense peak), 298, 291, 284, and 179 were also identified. Numbers in this series differed from the next number by a mass number of 105. These same series of mass numbers were also identified by making a Freon-alcohol extraction of the molecular sieves after ten cycles of operation had been completed. A summary of the high-resolution mass spectrographic analyses is given in Table

In an attempt to further identify the presence of any organic residues on the molecular sieves, high-resolution mass spectrographic analyses were conducted. A copy of the report is included (Appendix A). Some of the black molecular sieve beads that were sampled from the bottom of the column after the fire (which exhibited an unburned residue of carbon) showed major peaks corresponding to Masses 147 (predominant), 148, and 149. The isotopic abundance suggested the presence of two silicon

Figure 3. THERMOGRAVIMETRIC ANALYSIS-DIFFERENTIAL THERMAL ANALYSIS OF THE MOLECULAR SIEVES REMOVED FROM THE REFINING UNIT AFTER THE FIRE.

5. A typical high-resolution thermal mass spectrogram showing the organic species found in the molecular sieves after ten cycles of operation with triarylphosphate is given in Figure 5.

Although no definite organic structures have been assigned to these mass numbers at the present time, the data show clearly that organic residues accumulate on the molecular sieves and that these were probably responsible for the fire.

Table 4

ANALYSES OF SOLID PARTICULATES FROM THE
INLET CELLULOSE FILTER CARTRIDGE

Semiquantitative Spectrographic Analyses			
Element	Concentration (%)	Element	Concentration (%)
Ag	< 0.001	Mo	0.002
Al	0.1	Na	0.04
Au	< 0.01	Nh	< 0.01
B	< 0.003	Ni	0.01
Ba	0.001	P	0.6
Be	< 0.001	Pb	0.01
Bi	< 0.003	Rb	< 0.1
Ca	0.07	Sb	< 0.01
Cd	< 0.01	Si	0.2
Co	< 0.001	Sn	0.01
Cr	0.01	Ta	< 0.1
Cs	< 0.2	Th	< 0.01
Cu	0.1	Ti	0.01
Fe	1.5	U	< 0.1
Hf	< 0.1	V	< 0.01
K	0.02	W	< 0.02
Li	< 0.01	Zn	0.04
Mg	0.1	Zr	< 0.001
Mn	0.002		

X-Ray Diffraction Analyses

Major Constituents	Amorphous
Intermediate Constituents	Alpha $\text{Fe} \cdot \text{Fe}_3\text{O}_4$, Gamma $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$
Minor Constituents	SiO_2

144255
Figure 4. INLET AND OUTLET CELLULOSE FILTER CARTRIDGES THAT WERE REMOVED FROM THE REFINING SYSTEM AFTER THE FIRE.

A computer search of the best empirical formulas that could be assigned to the two principal mass numbers (410 and 305) showed $\text{C}_{24}\text{H}_{27}\text{PO}_4$ and $\text{C}_{16}\text{H}_{18}\text{PO}_4$, respectively. Computer calculations showed the 105-mass-number loss to be C_8H_9 .^(b) A summary of the computer calculations made to identify the best empirical formulas of $\text{C}_{24}\text{H}_{27}\text{PO}_4$ and $\text{C}_{16}\text{H}_{18}\text{PO}_4$ that were assigned to the principal mass numbers (410 and 305) is presented in Table 6.

DISCUSSION

The hydraulic fluid used in the hydraulic press is essentially a pure triarylphosphate ester that is manufactured by a major US chemical company and which has widespread industrial usage. It is used for many industrial applications requiring fire-resistant properties since it does not support or propagate flame when removed from an ignition source by "wicking"

(b) Rainey, W. T., Jr; "Mass Spectrometric Analysis of Molecular Sieve Materials", *Letter*; Union Carbide Corporation—Nuclear Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; January 17, 1972.

Figure 5. THERMAL MASS SPECTROGRAM OF THE MOLECULAR SIEVE EXTRACT.

action. According to Union Carbide Corporation—Nuclear Division Purchasing Specification 11 AC-1, shown in Appendix C, it has a viscosity of 575 SSU at 100° F, a flash point of 450° F (minimum), a fire point of 660° F (minimum), and an auto-ignition point of 1,165° F (minimum).

According to technical information recently supplied by the manufacturer, this hydraulic fluid contains no flammable additives and is essentially a pure tricresylphosphate ester. It is a mixture of the ortho, para, and meta tricresylphosphates, but predominately contains the para isomer to provide the desired viscosity.

As noted in Table 1 and Figure 1, the molecular sieves removed from the refining unit after the fire showed a considerable residue of carbon, ranging from 2.4 to 4.5 percent. It is probable that the carbon content of the molecular sieves was much higher than this prior to ignition and burning. A sample of fused molecular sieves removed from the bottom of the bed analyzed 0.67 percent carbon inasmuch as the fire zone was most intense at this location and most of the carbon had been burned away. The molecular sieve fire was so intense that the heat generated had partially melted a steel flange and some steel piping that were located immediately below the molecular sieve bed, indicating that a temperature of at least 1,375° C (the melting point of steel) had been reached. A photograph showing some typical steel piping sections removed from the bottom of the sieve bed that had become fused by the fire is presented in Figure 6.

Table 5

MAJOR MASS NUMBERS IDENTIFIED BY HIGH-RESOLUTION MASS SPECTROMETRY OF MOLECULAR SIEVES AFTER TEN CYCLES OF OPERATION WITH TRIARYLPHOSPHATE HYDRAULIC FLUID

Mass Unit Difference	Mass Number	Mass Unit Difference
	452	
14	438	
14	424	
	410(1)	105
	396	
	319	105
14	305(2)	
14	298	105
14	291	
	284	105
	209	
	208	
	193	
14	179	105

(1) Most predominant mass found.

(2) Very intense peak.

Although the analytical data presently available do not present conclusive evidence to explain completely the origin and causative factors pertaining to the molecular sieve fire, some definite conclusions and a considerable amount of isolated information have been accumulated to construct the following explanation for its occurrence: The presence of carbon in the molecular sieves, which consist chemically of sodium aluminosilicate (a

noncombustible material), shows that some organic residue must have been present for combustion to have occurred. It was suspected that an accumulation of a considerable amount of some organic residue, possibly a derivative of cresol (or phenol) on the molecular sieves, had probably occurred over the past nine years of use because of the hydrolysis of tricresylphosphate according to the reaction:

The ease of this hydrolysis reaction was readily demonstrated in a supplementary laboratory test by heating a one-to-one volume ratio of triarylphosphate fluid with water that had been adjusted to a pH of 4.0. After six hours on a steam bath, the pH had decreased to 3.7, indicating the formation of some phosphoric acid. Since it is also well known that molecular sieves represent a highly active surface and can retain water and possibly also some phosphoric acid as irreversibly adsorbed components, it is likely that some hydrolysis of the hydraulic triarylphosphate fluid occurred upon repeated regeneration treatments with air at 400° F over the years. Samples of new molecular sieves that had been removed from the building hydraulic oil system after ten adsorption cycles and steam cleanings were obtained prior to regenerating with air at 400° F. These sieves showed a residual hydraulic triarylphosphate fluid content of from 3.1 to 5.5 weight percent for samples taken at the top and bottom of the bed, respectively. Thus, steam cleaning of the sieves prior to regeneration with air at 400° F is not completely effective in removing all traces of hydraulic fluid from the sieve bed.

Table 6

COMPUTER CALCULATIONS FOR BEST EMPIRICAL
FORMULA ASSIGNED TO MASS NUMBERS 410
AND 305 FOUND ON MOLECULAR SIEVES
AFTER TEN CYCLES OF OPERATION
WITH TRIARYLPHOSPHATE

The Following Compounds Have Molecular
Weights Lying in the Range
305.09070 - 305.10070

Min No of Atoms is C 10 H 10 O 0 P 0
Max No of Atoms is C 30 H 40 O 10 P 2
Measured Mass 305.09570

Exact Mass	Deviation	Rings	Number of Atoms of Each Element			
			C	H	O	P
305.09667	- 0.00097	17.5	23	13	1	0
305.09445 ⁽¹⁾	0.00124	8.0	16	18	4	1

The Following Compounds Have Molecular
Weights Lying in the Range
410.16150 - 410.17150

Min No of Atoms is C 10 H 10 O 0 P 0
Max No of Atoms is C 30 H 40 O 10 P 2
Measured Mass 410.16650

Exact Mass	Deviation	Rings	Number of Atoms of Each Element			
			C	H	O	P
410.16487 ⁽¹⁾	0.00162	11.5	24	27	4	1
410.16265	0.00384	2.0	17	32	7	2
410.17093	0.00443	2.5	17	31	9	1

(1) Most logical assignment of molecular structure.

The following conclusions can be drawn as a result of this study:

1. The presence of carbon on the sieves indicates the presence of some organic as a carbon precursor which served as the combustion material.

144256

Figure 6. PIPE SECTIONS ATTACHED TO THE VALVE AND STEM THERMOMETER THAT WERE FUSED AS A RESULT OF THE MOLECULAR SIEVE FIRE.

2. Literature data and laboratory studies have shown that tricresylphosphate, an ester, can hydrolyze to form cresol and phosphoric acid.

3. Analyses of the replacement molecular sieves used in the refining system after only ten cycles of operation have shown that steam cleaning is ineffective in removing all tricresylphosphate fluid from the sieves prior to regeneration with hot air.
4. High-resolution mass spectrographic analyses of the replacement molecular sieves used in the refining system after only ten cycles of operation identified the presence of several series of organic residues having principal mass numbers ranging from 179 to 472, showing that steam cleaning and hot-air regeneration treatment of the molecular sieves does not prevent such organic residues from accumulating in the bed of sieve beads.
5. High-resolution mass spectrographic analyses of the replacement molecular sieves used in the refining system after only ten cycles of operation identified Masses 410 and 305 as the most intense organic residues present. These masses have been assigned empirical formulas corresponding to $C_{24}H_{27}PO_4$ and $C_{16}H_{18}PO_4$, respectively, by computer calculations.
6. As a safety precaution, the molecular sieves in the refining system should be replaced more frequently (eg, semiannually or annually) to prevent a cumulative buildup of organic residues on the sieves that could sustain combustion.

ACKNOWLEDGEMENTS

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APPENDIX A

INTERNAL CORRESPONDENCE

UNION CARBIDE NUCLEAR COMPANY

POST OFFICE BOX Y, OAK RIDGE, TENNESSEE

To (Name) C. R. Schmitt
Company
Location

Date January 17, 1972

Originating Dept.

Answering letter date

Copy to

Subject Mass Spectrometric Analysis of
Molecular Sieve Materials

Samples of the molecular sieves were introduced into the mass spectrometer using the direct inlet probe, and spectra were recorded as the sample temperature was increased from room temperature to ca 400°C.

Black beads from burned sieve. This sample gave indication of a rather volatile material containing an organic silicon compound. The major peaks were very intense at m/e 147, 148, and 149. The isotopic abundance suggested the presence of two silicon atoms. This mass is often encountered in spectra of trimethylsilyl compounds and is assigned the formula $(CH_3)_3Si-O = Si(CH_3)_2$. This is, of course, a fragment ion derived by rearrangement of a higher mass ion. The exact mass measurement of the m/e 147 peak by high resolution mass spectrometer gave a mass in good agreement with the suggested formula. No higher mass peaks were of significant intensity, so the molecular ion is probably not stable.

At temperatures above 200°C an m/e 221 peak became significant. Isotopic abundances at m/e 221 and 222 suggest the presence of three silicon atoms. There is no evidence that the m/e 221 ion is related to the m/e 147 ion; neither intensity vs temperature nor metastable ions suggest such relationship.

White beads from burned sieve. These beads gave only very low intensity spectra which were very similar to those from the black beads.

New sieve beads. Only the expected air gas peaks were obtained from these beads.

Recycled beads. These beads gave a very intense spectrum of an organic compound with an apparent molecular weight of 410. This peak was the most intense peak in the spectrum, so one would expect it to be a very stable molecule without many functional groups. Other groups of peaks above and below 410 in multiples of 14 mass units suggest the presence of homologous compounds or that the m/e 410 peak is a hydrocarbon fragment of unusual stability.

C. R. Schmitt

-2-

January 17, 1972

Other intense peaks were noted at m/e 305, 209, 208 and 193 with metastables to indicate

$$410 \rightarrow 305 + 105$$

$$208 \rightarrow 193 + 15$$

Exact mass measurement of the m/e 410 and 305 peaks gave results which must be in error, although the precision appeared to be very good. The best empirical formulas seemed to be $C_{22}H_{18}O_8$ (410) and $C_{16}H_{17}O_8$ (305) suggesting the 105 mass loss to be C_6HO_2 , a rather improbable fragment. Inclusion of phosphorous into the calculations does not make the results more plausible.*

Extract of recycled beads. This liquid gave the same mass spectrum as the recycled beads. It gave a clean gas chromatogram, showing it to be predominantly a single compound. Attempts to convert it to a trimethylsilyl derivative failed. Apparently it contains no active hydrogens, and introduction into the mass spectrometer through the chromatograph inlet showed no change.

W. T. Rainey, Jr.
W. T. Rainey, Jr.

WTR/dcs

Note: Subsequent studies at an improved resolution of the mass spectrometer have identified $C_{24}H_{27}PO_4$ (m/e 410), $C_{16}H_{18}PO_4$ (m/e 305), and C_8H_9 (m/e 105) as the best empirical formulas.

APPENDIX B

STANDARD OPERATING PROCEDURE - HYDRAULIC FLUID REFINING SYSTEM FOR A 7500-TON PRESS

Introduction

The hydraulic fluid refining system for a 7500-ton press (Figure B-1) is designed upon the principle of a continuous side-stream filter, removing the hydraulic fluid from the press reservoir, pumping the fluid through the refining system, and then returning it to the press reservoir. The system consists of a gear-type pump, a prefilter, two rotameters, a molecular sieve bed, and a sieve after filter. The gear-type pump has a built-in relief system. The relief set point is determined by the size of the spring installed in the pump head. The prefilter and the sieve after filter contain six cartridges with an effective particle size removal of 10 micrometers. The two rotameters are located at the inlet to the sieve bed and behind the press operating console. The molecular sieve bed consists of a sodium aluminosilicate bead material of 8 - 12 mesh. In addition to acting as a good filter, the sieve will remove water and acidity from the hydraulic fluid. However, it has more affinity for water removal; therefore, if it becomes "loaded" with water, it requires regeneration to further reduce the acidity. Laboratory analysis of the hydraulic fluid will determine the frequency of regeneration.

Regeneration is accomplished by first washing or sparging the sieve with low-pressure steam. This process will remove the hydraulic fluid and also wash out any acid. Then low-pressure instrument air, preheated by a 12-kw electric heater to 400° F, is passed countercurrently through the sieve. When the air temperature leaving the sieve remains constant (range of 250 - 300° F), the sieve has been regenerated. Then cold instrument air is passed by normal flow through the sieve to the sump for partial cooling. The sieve is then allowed to cool at ambient conditions for at least 2 - 4 hours. The system is then filled with hydraulic fluid and normal flow rates established.

Safety Precautions and Safety Equipment

There have been no reported cases of toxic effects from the use of the hydraulic fluid. However, accidental swallowing is the chief potential source of entry into the body. No eating should be permitted around the material, and personal cleanliness should be stressed. Exposure to the skin presents a minimal hazard; but, if exposure should occur, particular attention should be paid to a thorough cleaning of the skin and removal of soiled clothing if extensive and prolonged skin contact occurs. Under usual operating conditions, inhalation of vapors is almost impossible due to the extremely low volatility of the fluid. No maximum allowable concentration (MAC) for this particular hydraulic fluid in air has been established. Care should be exercised during the steaming process of the sieve to avoid inhalation of any vapors as a result of this part of the procedure. Safety equipment for the operation of this system should include safety glasses or goggles, heavy work gloves for operation of the hot valves, and a respirator (if deemed necessary during the steam cycle of regeneration).

Figure B-1. HYDRAULIC FLUID REFINING SYSTEM.

Procedure

PART I - NORMAL OIL FLOW (Assume All Valves Closed)

1. Open Valves 21, 19, 20, 1, and 3.
2. Partially open Valve 17; start pump; partially open Valve 2 to fill sieve bed, venting air at Valve 17.
3. When hydraulic fluid is observed at Valve 17, close valve and stop pump.
4. Fully open Valve 2, then Valves 5, 7, and 8.
5. Start pump and adjust Valve 1 to give a 10-gpm flow rate through the rotameter.
6. Check the line at the sump to be sure Valve 6 is not leaking.

PART II - REGENERATION OF THE MOLECULAR SIEVE

1. Removal of excess hydraulic fluid.
 - a. Stop hydraulic fluid pump; close Valves 3 and 5.
 - b. Open Valve 6 to drain oil from the sieve to the sump.
 - c. Open Valves 12 and 13.
 - d. Partially open Valve 14; open Valves 15 and 4. This action will purge the residual hydraulic fluid from the sieve.
 - e. Partially open Valve 11 to purge the hydraulic fluid from the piping below the sieve to the sump.
 - f. When the sump line indicates that the majority of the hydraulic fluid has been purged, close Valves 11 and 14.
2. Steam Washing or Sparging
 - a. Open Valve 10 to drain excess condensate from the steam supply header.
 - b. Partially open Valve 9 to direct the steam through Valves 15 and 4, through the sieve, and through Valve 6 to the sump. Watch TI-2 and do not exceed a steam temperature of 225° F. (Sieve will become very warm until the adsorption of steam condensate stops.)
 - c. When hot condensate is observed at the sump, open Valve 9 to completely steam the sieve.
 - d. Regeneration is complete when the condensate at the sump becomes clear.
 - e. Open Valve 11 for five minutes to steam clean the piping below the sieve.
 - f. Close Valves 9, 10, and 11.

NOTE: It is very important from a safety standpoint that all triarylphosphate hydraulic fluid be removed from the sieve and piping where hot instrument air will be used in the final regeneration. Triarylphosphate has a flash point of 455° F and a fire point of 665° F. There is little chance that any ignition would occur; however, do not exceed any air temperatures above 400° F at TI-1.

3. Instrument Air Regeneration

- a. With the 12-kw heater off, partially open Valve 14 to remove excess moisture or condensate from the molecular sieve.

- b. Open Valves 11 and 16 and close Valves 15 and 6. Air flow will now be directed counter currently through the sieve.
- c. Turn on the 12-kw heater safety switch; adjust the thermostat to 420° F.
- d. Adjust the air flow at Valve 14 to maintain the air temperature of 400° F at TI-1. Proper adjustment will keep the heater on at all times.
- e. Observe TI-2 on top of the sieve bed; and, when the temperature reaches the range from 250 to 300° F and remains constant for at least 15 minutes, the sieve is regenerated.
- f. Adjust the heater thermostat to 0° F and trip the heater safety switch.
- g. When the air at TI-1 is in the range of 250 to 275° F, open Valves 15 and 6 and close Valves 11 and 16. This action will direct the air down through the sieve for cooling (10 minutes).
- h. Close Valve 12 and let all the pressure bleed from the air heater.
- i. Close Valves 13, 14, 15, and 6. Open Valve 16. The sieve should now be allowed to cool at ambient conditions for four hours.
- j. Close Valves 16 and 4.
- k. Proceed to fill the sieve with hydraulic fluid, vent the air, and then establish the flow rates as described in Part I of this procedure.

APPENDIX C

PURCHASING SPECIFICATION 11-AC-1, Code HC

LUBRICANTS AND CUTTING COOLANTS

CLASSIFICATION

This data sheet covers an essentially pure triarylphosphate ester fluid for applications requiring fire-resistant properties.

support or propagate flame when removed from an ignition source by "wicking" action.

The bidder shall furnish representative typical test data in the space provided opposite the Company's specification value in the following table, or where asked to "please state".

DETAILED REQUIREMENTS

This fluid shall be suitable for applications requiring minimum flammability and shall not

	Requirement and Test	Code HC	
1.	Ester Content, % Min	99	
2.	Color	Clear Transparent	
3.	Viscosity, SSU at 100° F	575	
4.	Specific Gravity: 20/20° C	1.17	
5.	Flash Point, ° F, Min	450	
6.	Fire Point, ° F, Min	660	
7.	Foam Inhibited	Yes	
8.	Hot Manifold Test, ° F, Min	1,300	
9.	Auto-Ignition Point, ° F, Min	1,165	
10.	Pour Point, ° F, Max	20	
11.	Water and Sediment (ASTM D 96) % Max	Nil	
12.	Acidity, Mg KOH/g, Max	0.1	
13.	Federal or Military Specification Number	Please State	

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