Recovery of Perchloroethylene Scrubbing Medium Generated in the Refabrication of High-Temperature Gas-Cooled Reactor Fuel

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RECOVERY OF PERCHLOROETHYLENE SCRUBBING MEDIUM
GENERATED IN THE REFABRICATION OF HIGH-TEMPERATURE
GAS-COOLED REACTOR FUEL

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RECOVERY OF PERCHLOROETHYLENE SCRUBBING MEDIUM GENERATED IN THE
REFABRICATION OF HIGH-TEMPERATURE GAS-COOLED REACTOR FUEL

M. S. Judd,¹ J. E. Van Cleve, Jr., W. T. Rainey, Jr.²

ABSTRACT

During the refabrication of high-temperature gas-cooled reactor (HTGR) fuel, perchloroethylene (C_2Cl_4) is used as the nonmoderating scrubbing medium to remove condensable hydrocarbons, carbon soot, and uranium-bearing particulates from the off-gas streams. This paper discusses the process by which the contaminated perchloroethylene is recycled.

INTRODUCTION

In the HTGR fuel cycle, the recycle fuel fabrication is that portion where uranium that has been recovered from spent fuel elements at a reprocessing plant is fabricated into fuel and incorporated into recycled fuel elements. Two types of uranium will be fabricated at the recycle fabrication plant — that containing the residual ^{235}U recovered from the fissile particles in fresh fuel elements and that containing the ^{233}U bred from the ^{232}Th in the fertile particles.

The processing steps necessary to incorporate the recovered uranium into a fuel element suitable for use in an operating HTGR are illustrated in Fig. 1. The fissile ^{233}U and ^{235}U are received from the reprocessing plant as uranyl nitrate solutions, which are converted to an acid-deficient uranyl nitrate solution. The uranyl ions are loaded onto ion exchange resin microspheres. The uranium-loaded resin microspheres are dried, carbonized, converted to uranium oxide-carbide kernels, and then coated with multiple layers of pyrolytic carbon and silicon carbide in a fluidized-bed coating furnace. In fuel rod fabrication, pyrolytic-carbon-coated ThO_2 particles obtained from a fresh-fuel fabrication plant are blended with coated recycled uranium particles and bonded into fuel rods with pitch and graphite.

¹Now with U.S. Steel, Monroeville, Penn.

²Analytical Chemistry Division.

Fig. 1. Process for HTGR recycle fabrication.

The fuel rods, which are 5/8 in. (1.59 cm) in diam and 2-1/2 in. (6.35 cm) long, are loaded into hexagonal graphite fuel blocks, approximately 31 in. (78.7 cm) high and 14 in. (35.6 cm) across the flats. The fuel elements are heated to carbonize and anneal the fuel rods and then loaded with poison rods, end plugs, and dowels.

Unique off-gas cleanup problems are created by processes used to carbonize, convert, and coat the uranium-loaded fuel kernels and carbonize the fuel rods. The gaseous effluents from these processes are passed through a spray column scrubber, which employs perchloroethylene

(C_2Cl_4) as the nonmoderating scrubbing medium (Fig. 2). The perchloroethylene traps carbon soot, uranium-bearing particulates, and a large number of complex polynuclear aromatic hydrocarbons.

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Fig. 2. Perchloroethylene scrubber for removing soot and hydrocarbons from coater effluent.

Recycling the perchloroethylene scrubbing medium is desirable in order to (1) reduce the volume of waste material, (2) recover the entrained uranium, and (3) reduce hazards encountered during disposal. Table 1 lists the amounts of raw scrubbing medium that would be processed in the recovery system of a commercial plant. A commercial plant will produce fuel elements containing approximately 84 kg of uranium daily. This will correspond to a recovery system with a capacity of 6200 liters/day.

Characterization of Raw Perchloroethylene

Table 2 shows the data obtained from four raw perchloroethylene samples, which should be considered representative. Variations in the composition of the samples are characteristic of the process system, the amount of fuel processed, and the residence time of the perchloroethylene in the scrubber. Specific gravity measurements were made by weighing a 25-ml pycnometer filled with a well-mixed sample. The weight percent of perchloroethylene was determined by calibrated gas chromatography, and it is estimated to be within 3% of the true value. The error was primarily due to the difficulty in measuring the major peak on the chromatogram. Solids content was determined by weighing the solids that were removed from the samples by filtration. The solids were ignited, the residue was dissolved in acid, and the uranium content was determined by fluorimetric analysis. The hydrogen ion concentration was measured by shaking the sample with a known volume of standard alkali and back-titrating the excess alkali. Chloride is a measure of the total ionic chlorine in the sample. For all samples, this value was less than the minimum concentration (5 ppm) detectable by potentiometric titration.

The variations in specific gravity of the raw perchloroethylene samples are primarily due to variations of the amount of dissolved hydrocarbons and, to a lesser extent, the amount of suspended soot. Reagent perchloroethylene has a specific gravity of 1.63, as compared with specific gravities ranging from 1.47 to 1.63 for the raw perchloroethylene samples. Sample P-4 had a specific gravity of 1.47 because the effluent from resin carbonization consists mainly of condensable hydrocarbons, with a minor amount of carbon soot. The effluent from coating operations consists mainly of carbon soot, with a minor amount of condensable hydrocarbons. The values for weight percent perchloroethylene and weight percent solids are determined by the type and number of runs.

The uranium content of the raw perchloroethylene is determined by operating parameters. Particle entrainment and abraded uranium particulates account for the uranium in the scrubbers. The hydrogen ion and chloride concentrations are both very low, which is significant because

Table 1. Material entering C_2Cl_4 recovery system
per kilogram of uranium product

Source	C_2Cl_4 (liters)	U (kg)	C_xH_y (kg)	C soot (kg)
Resin carbonization	3.16	0.0133	0.44	
Resin conversion and coating	46.8	0.0061	0.60	6.3
Fuel rod carbonization	23.4		3.3	
Totals	73.4	0.0194	4.34	6.3

Table 2. Characterization of raw C_2Cl_4

Sample code	Process history	Specific gravity at 25°C	C_2Cl_4 (wt %)	Solids (wt %)	Uranium content (ppm)	$[H^+]$ (N)	Chloride (ppm)
P-4	Resin carbonization	1.47	87.4	0.54	80	$2(10^{-4})$	<5
P-7	Conversion and coating	1.63	96.7	0.50	429	$2(10^{-4})$	<5
P-8	Conversion and coating	1.62	93.1	1.52	10	$2(10^{-4})$	<5
P-10	Conversion and coating	1.57	88.7	4.00	260	$<1(10^{-4})$	<5

hydrochloric acid is undesirable from a materials compatibility standpoint.

Several perchloroethylene samples were analyzed using combined gas chromatography and mass spectrometry (GC-MS). This type of analysis was used to identify and compare the condensable hydrocarbons contained in the raw perchloroethylene obtained from the different refabrication processes. (A detailed description of the GC-MS method of analysis is given in the Appendix.) The chromatogram for P-3, obtained from fuel rod carbonization, is shown in Fig. 3, and the probable identification of the compounds found in P-3, which corresponds to the numbered peaks, are given in Table 3. Although slight shifts were noted in absolute retention times due to small variations in chromatographic conditions, Table 3 lists the compounds in order of relative retention times.

Although the chromatograph shown in Fig. 3 is representative of the effluent from rod carbonization, this chromatograph is also typical of the effluents from resin carbonization and microsphere coating. The important difference in the composition of the effluents noted between rod carbonization and resin carbonization and microsphere coating was the presence of several thiophene derivatives (peaks 30, 37, and 47). This was not unexpected, since the fuel-rod pitch binder contained 1 to 2% sulfur.

Results from the GC-MS analyses, as well as other studies made by Lee et al. using a time-of-flight mass spectrometer³ to monitor the gaseous effluents from the same processes, show that similar products should be expected from pyrolysis of organic compounds in nonoxidizing atmosphere. The work of Bard et al.⁴ showed that the same products were

³D. A. Lee, D. A. Costanzo, D. P. Stinton, J. A. Carpenter, Jr., W. T. Rainey, Jr., D. C. Canada, and J. A. Carter, "In-Line Monitoring of Effluents from HTGR Fuel Particle Preparation Processes Using a Time-Of-Flight Mass Spectrometer," *Nuclear Technology*, to be published.

⁴R. J. Bard, H. R. Boxman, J. P. Bertino, and J. A. O'Rourke, "Pyrolytic Carbons Deposited in Fluidized Beds at 1200° to 1400°C from Various Hydrocarbons," *Carbon* 6, 603-16 (1968).

Fig. 3. Chromatogram from perchloroethylene P-3, recovered from scrubber after fuel rod carbonization run. Injection volume: 0.8 μ l undiluted.

Table 3. Probable identification of the chromatogram of sample P-3

Peak number	Molecular weight	Probable identification
1	78	Benzene
2	92	Toluene
3	94	Perchloroethylene
4	106	Xylene
5	104	Styrene
6	120	C ₃ benzene ^a
7	118	Methylstyrene
8	134	C ₄ benzene
9	132	C ₂ styrene
10	148	C ₅ benzene
11	146	C ₃ styrene
12	128	Naphthalene
13	130	Divinylbenzene
14	160	C ₄ styrene
15	144	Methyldivinylbenzene
16	142	Methylnaphthalene
17	158	C ₂ divinylbenzene
18	154	Biphenyl
19	156	C ₂ naphthalene
20	168	Methylbiphenyl
21	152	Biphenylene
22	168	Methylbiphenyl
23	170	C ₃ naphthalene
24	182	C ₂ biphenyl
25	166	Fluorene
26	184	C ₄ naphthalene
27	196	1,2-Diphenylpropane
28	196	C ₃ biphenyl
29	180	1,1-Diphenylethylene
30	184	Dibenzothiophene
31	198	C ₅ naphthalene
32	178	Phenanthrene
33	194	Methylvinylbiphenyl
34	210	C ₄ biphenyl
35	192	Methylphenanthrene
36	190	Methylenephenanthrene
37	198	Dimethylnaphthothiophene
38	206	C ₂ phenanthrene
39	202	Pyrene
40	202	Fluoranthene
41	216	Methylpyrene
42	230	Terphenyl
43	234	C ₄ phenanthrene
44	310	C ₂₂ H ₄₆
45	226	Benzofluoranthene
46	228	Chrysene
47	248	Methylbenzonaphthothiophene
48	242	Methylchrysene
49	256	C ₂ chrysene
50	252	Benzopyrene
51	266	Methylbenzopyrene
52	264	Methyleneperylene
53	276	Benzoperylene

^aThe subscripted number after C in this table refers to the number of alkyl carbons attached to the aromatic nucleus. No indication of chain length(s) or position of attachment is to be inferred unless stated.

obtained from pyrolysis of seven different hydrocarbon gases in fluidized beds under conditions similar to those reported here. Generally, the products are progressively converted to condensed-ring materials of decreasing hydrogen-to-carbon ratio, until carbon is finally deposited.

The presence of polynuclear aromatic hydrocarbons (PAH) in a process stream should be a warning of the potential hazards to all personnel who might be exposed to those materials during processing or to effluent materials during disposal operations. Certain of the compounds are known to be dangerous as carcinogens, cocarcinogens, or tumor promoters, and relative ratings of the dangers have been published.⁵⁻⁷ However, there are really no good quantitative data on the dangers of exposure to many of these compounds. The testing is far from complete, and until more information is available, adequate protection must be furnished to limit exposure of personnel in plant operations as well as those in the environs of the operation. The continued expansion in biological testing of PAHs and related compounds should result in the near future in development of regulations relating to occupational exposure.

Since the qualitative identification of many of the components in these process streams is not unambiguous, rating the dangers associated with the streams is difficult. Mass spectral data indicate the presence of benzopyrene (or isomeric materials) but cannot determine whether the substance is benzo(a)pyrene or benzo(e)pyrene. Testing indicates that benzo(a)pyrene is a potent carcinogen, while benzo(e)pyrene is only a mild carcinogen. Therefore, the allowed exposures might be different; however, until unequivocal proof of identity is available, allowed exposure must be dictated by the most potent of the two. Similarly,

⁵National Research Council, National Academy of Sciences, *Particulate Polycyclic Organic Matter, Biological Effects of Atmospheric Pollutants*, Washington, D.C., 1972.

⁶D. Hoffman, I. Schmeltz, S. S. Hecht, and E. L. Wynder, "On the Identification of Carcinogens, Tumor Promoters, and Cocarcinogens in Tobacco Smoke," *Proceedings of the Third World Conference on Smoking and Health*, June 2, 1975, New York.

⁷I. Schmeltz, D. Hoffman, and E. L. Wynder, "Toxic and Tumorigenic Agents in Tobacco Smoke: Analytical Methods and Modes of Origin," *Proceedings of the Eighth Annual Conference on Trace Substances in Environmental Health*, June 11, 1974, Columbus, Mo.

and methylchrysenes have been rated as mild to strong carcinogens. Pyrene, fluoranthene, and benzoperylene have been determined to be cocarcinogenic. Several methyl-, dimethyl-, ethyl-, and trimethylnaphthalenes have shown promoter activity.

EXPERIMENTAL RESULTS AND DISCUSSION

The equipment used during the laboratory-scale perchloroethylene recovery work is shown in Fig. 4. During laboratory distillation experiments, the distilling flask was heated with an electric heating mantle. The perchloroethylene vapors passed through an insulated reflux condenser into the primary water-cooled condenser. The heat input to the system was controlled by the vapor temperature as it entered the primary condenser. A magnetic stirrer was used to prevent violent boiling and keep the carbon soot in suspension.

During the laboratory development work, several of the various raw perchloroethylene batches were found to contain anywhere from 2 to 14% water. The water is formed during carbonizing of the resin. Since a mixture of two immiscible liquids will boil at the temperature where the sum of their vapor pressures equals the system pressure, a phase separator had to be added to the system. If the still is vented to the atmosphere, a mixture of perchloroethylene and water will boil and steam distill around 87°C, which is lower than the boiling point of either water (100°C) or perchloroethylene (120°C). Although steam distillation was not adopted as the distillation method, it offers several advantages over straight distillation, primarily a lower distillation temperature and much better control of the pot temperature.

Measurements of the weight percent of perchloroethylene in the raw scrubbing medium, the still bottoms, and the distillates were made to determine the efficiency of the recovery system. Although the material balances over the system were only accurate to within plus or minus 5%, the performance of the recovery system was completely satisfactory. The percent purity and specific gravity of the distillates are given in Table 4, and the material balances for each sample are given in Table 5. The amount of perchloroethylene in the distillates ranged from 96 to over

Fig. 4. Distillation apparatus for perchloroethylene reclamation.

99 wt %. The high purity indicates that fractional distillation is not necessary and that conventional evaporators are adequate for recovery.

Table 4. Characterization of distillates

Sample	C ₂ Cl ₄ (wt %)	Specific gravity at 25°C
P-4	96	1.60
P-7	99	1.62
P-8	98	1.61
P-10	99	1.62

Table 5. Material balance

Sample	History	C ₂ Cl ₄ (%)		
		In sample	In bottoms	Recovered
P-4	Resin carbonization	87	10	82
P-7	Conversion and coating	96	86	88
P-8	Conversion and coating	93	87	74
P-10	Conversion and coating	88	77	92

The amount of recovered perchloroethylene ranged from 74 to 92%. The primary reason these recovery values did not approach 100% was that material handling considerations required the distillations to be interrupted prior to completion. The most serious material handling problems are associated with removing the still bottoms. The problem with handling the still bottoms is that the raw perchloroethylene obtained from the microsphere coating operations contains considerable quantities of carbon soot. If the distillation is made to maximize perchloroethylene recovery, the resultant still bottoms consist of solids; since solid bottoms are much more difficult to transfer than liquid bottoms, this mode of distillation is unacceptable. Distillation of the raw perchloroethylene obtained from resin carbonization and fuel rod carbonization operations results in liquid still bottoms. Therefore, one solution to the solid still-bottoms problem is to blend the raw perchloroethylene from all operations in a manner resulting in liquid still bottoms. This approach has been tried and was successful.

Although not as desirable as the above solution, another solution to the solid still-bottoms problem is to stop the distillation before the suspended solids begin to settle out. This approach reduces the amount of perchloroethylene recovered, but it does result in liquid still bottoms. The latter approach was used to obtain the data shown in Table 5. The still bottoms resulting from the distillation of P-4 contained the smallest amount of perchloroethylene because this sample, unlike the others, came from the resin carbonization process and contained hydrocarbons, rather than soot. The amounts of perchloroethylene recovered are not particularly impressive, but there were not any difficulties in removing the still bottoms as a liquid.

Following completion of the laboratory experiments, the recovery flowsheet was verified in the engineering-scale equipment shown in Fig. 5. The 30-gal-capacity still consists of a jacketed vessel, two shell-and-tube heat exchangers for the condenser, a phase separator, a liquid agitator, a pressure gage and pressure relief valve, level and density probes, temperature instrumentation, and the necessary pumps and piping.

Fig. 5. Engineering-scale perchloroethylene recovery system.

The still was first checked out using water, and the pot temperature dropped only 2°C 16 hr after the steam supply was turned off. A cooling water loop was added to the steam jacket to rapidly cool the system when the distillation was terminated.

The engineering-scale still has been used to process a total of 129 gal of raw perchloroethylene. A temperature profile from a typical distillation is shown in Fig. 6. Material first began distilling around 87°C, which is indicative of a raw perchloroethylene solution containing

Fig. 6. Temperature profile for typical perchloroethylene distillation.

water. As soon as the water was removed, the still pot and vapor temperatures equilibrated at 122 and 120°C respectively; the condensate temperature remained around 13°C during the entire run; the cooling water flow to the condenser was approximately 20 liters/min, with an inlet temperature of 8°C and an exit temperature of 14°C; and the steam pressure varied between 5 and 40 psig during the distillation. This batch was distilled to recover 80% of the perchloroethylene to ensure liquid-bottoms discharge. After the desired amount of perchloroethylene was collected, the steam was turned off and the cooling water was turned on. The pot temperature dropped rapidly, while the vapor temperature dropped rapidly at first and then decreased gradually.

SUMMARY AND CONCLUSIONS

Perchloroethylene is proposed as a scrubbing medium for various process off-gas streams in the refabrication of HTGR fuels. In a large recycle plant, as much as 500,000 gal/year of perchloroethylene is estimated as necessary to operate the scrubbers. The recovery and recycle of this material is therefore important to the overall economics of refabricated HTGR fuels. Additionally, the volume of the liquid waste requiring disposition is materially reduced. The experiments described

in this report show that the perchloroethylene can be recycled by isothermal distillation.

The characterization of the used perchloroethylene showed that the material contained not only carbon soot but also a variety of polynuclear aromatic hydrocarbons. Compounds of this type have been shown to have biologically active properties. These properties require care in handling in order to limit personnel exposures.

Following the successful operation of a laboratory-scale still, which demonstrated the feasibility of distillation as a recovery process, an engineering-scale unit was assembled and operated with few problems. The engineering-scale unit will continue to be used to recycle the perchloroethylene produced during the developmental work on conversion and coating. A commercial unit is presently being evaluated for use in a large plant.

Based on the information obtained from this work, a flowsheet is being developed to recover the uranium present in the still bottoms.

APPENDIX

COMBINED GAS CHROMATOGRAPH — MASS SPECTROMETRIC ANALYSES

The samples were centrifuged to separate the perchloroethylene from suspended soot and water, and the resultant clear solution was diluted with pure perchloroethylene prior to injection into the gas chromatograph. Samples were injected into a Varian model 1200 gas chromatograph equipped with a splitter to allow use of the flame detector and mass spectrometer simultaneously. The splitter was constructed so that approximately 95% of the sample was diverted to the mass spectrometer. The chromatograph was interfaced to the mass spectrometer using a porous stainless steel effusion-type separator. All connecting tubing was heated stainless steel capillary, and all components were thoroughly silanized before use. The chromatographic column used for all analyses was a 180-cm- by 2-mm-ID glass column packed with 4% Dexsil 300 on 60-80 mesh Chromosorb W-HP operated isothermally at 28° for 6 min, then programmed at 4°/min (and in two cases at 10°/min after 45 min) to a maximum of 310° with a carrier flow rate of 20 ml/min. These conditions did not allow complete resolution of peaks, and so many of the peaks overlap because of interfering components.

The mass spectrometer was designed and constructed at Oak Ridge National Laboratory (ORNL) and was a 30-cm, 90° magnetic sector instrument operated at 4 kV accelerating voltage and 70 eV ionizing potential. Mass spectra were obtained as oscillograph recordings. The data were manually transferred to punched cards and were processed using an IBM 1130 computer and Calcomp plotter. Identification of compounds was usually made by comparison of spectra with a library of spectra of known compounds. However, in many cases, the data had to be interpreted to arrive at plausible structures, since all spectra were not available in the spectral libraries.

Mass spectral analysis does not lead to unequivocal structural identification in all cases. This is especially true for the types of compounds reported in this study, predominantly aromatic hydrocarbons and their alkylated homologs. Spectra of aromatic hydrocarbons are

known for their simplicity, being comprised usually of parent or molecular ions (M^+) and very few fragment ions of significant intensities. Usually, the spectra are also characterized by doubly charged molecular ions (M^{2+}), which appear at mass positions equal to $M^+/2$, and sometimes triply charged ions at $M^+/3$. Fortunately, the molecular ion is usually of reasonable intensity; thus the molecular weight can be determined. However, since the fragmentation pattern is usually quite simple, determining the desired structural information is often not possible. For instance, no significant differences are detectable between the mass spectra of phenanthrene and anthracene. Alkyl aromatic hydrocarbons yield spectra that may be useful in interpretation of structural information. Usually, the spectra will show other ions due to fragmentation of the alkyl group(s), and often this information is sufficient to determine the structure of the alkyl groups, but not necessarily their positions in the molecule. Thus, isopropylnaphthalene and *n*-propylnaphthalene yield spectra that are different and are recognizable.

Therefore, unambiguous identification by mass spectral analysis is difficult in these types of compounds. However, when used in conjunction with gas chromatography, definite structural information can often be obtained. In some cases in this study, identifications have been based on mass spectra and chromatographic retention times, but isomeric configurations are often possible and may not be eliminated with this information.

As indicated in Fig. 3, sensitivity changes were made during the run. These are indicated by notations (such as 10^{-12}) indicating the fraction of full instrument sensitivity. The actual peak heights or areas do not necessarily indicate relative concentrations of components because of inability to reset the flame-detector - mass-spectrometer split ratio to the same ratio each time and because of changes in flow rate during the run. In spite of the carrier-gas flow controller, small drifts in flow were experienced during runs, and any small drift caused a corresponding change in flow to the detector, resulting in an apparent variation in sensitivity. In addition, comparison of peak heights between different runs should also take into consideration the dilution factors used in making up the sample injected.

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