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## Characterization of Ft. St. Vrain Reactor Dust

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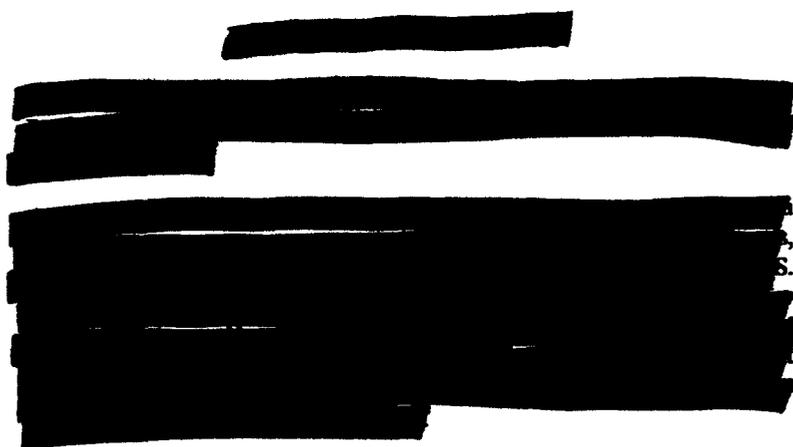
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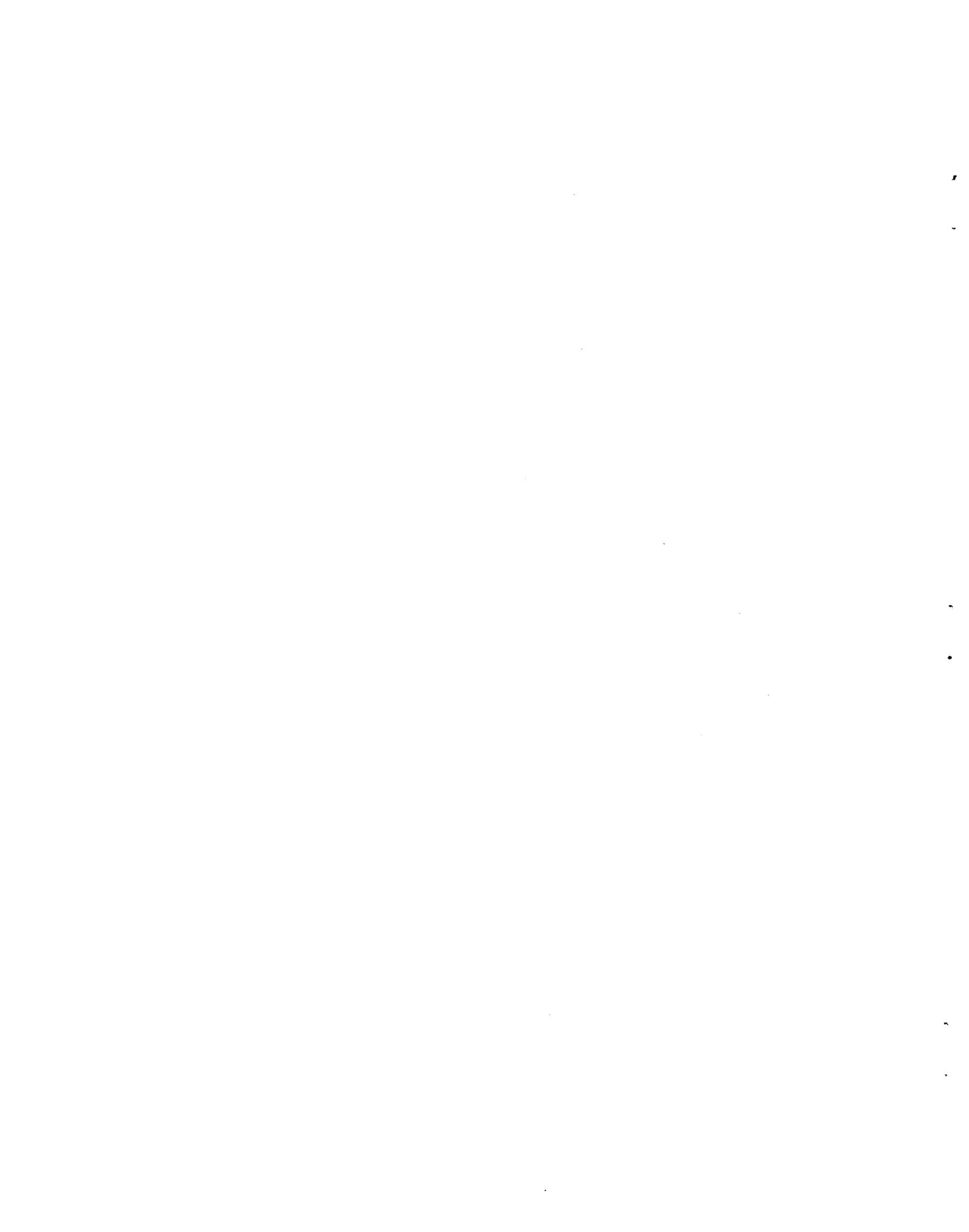
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## CONTENTS

	Page
SUMMARY .....	1
1. OBJECTIVE .....	1
2. INTRODUCTION .....	2
3. RESULTS .....	3
4. REFERENCES .....	3
APPENDIX A: RESULTS OF ANALYSIS OF FORT SAINT VRAIN REACTOR DUST BY X-RAY DIFFRACTION AND FLUORESCENCE .....	5
APPENDIX B: EXAMINATIONS OF DUST BY ELECTRON MICROSCOPE .....	15



# CHARACTERIZATION OF FT. ST. VRAIN REACTOR DUST

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## SUMMARY

Dust samples were obtained from a circulator which was removed from service during FY 1989. These samples were examined by scanning electron microscope and by x-ray diffraction and fluorescence. The latter technique showed that the bulk of the major dust sample consisted of  $\text{Fe}_3\text{O}_4$  with about 10 wt%  $\text{MoS}_2$  and 5% other materials. The  $\text{MoS}_2$  is thought to be present from its use as a lubricant on bolts used to assemble the circulator to the reactor. No graphite was observed. The presence of  $\text{Fe}_3\text{O}_4$  was either the result of significant moisture ingress during the shutdown before removal of the circulator or to exposure of the dust to air following removal of the circulator. The back-scattered electron (BSE) examination of the dust showed the  $\text{MoS}_2$  to be present as discrete flakes adhering to the other particles. The scanning-electron microscopy (SEM) examination of the dust showed that the bulk of the iron oxide particles were less than 1  $\mu\text{m}$  in diameter.

## 1. OBJECTIVE

The possibility of recovering Ft. St. Vrain dust from the circulator was viewed as a unique opportunity. It is recognized that the frequent incursions of moisture into the reactor may have resulted in different chemical forms for the metallic impurities since moisture ingress into this reactor has been a recurrent problem. It was desired to determine whether the major portions of the dust were graphite (or other carbon forms) or were metallic materials derived from scaling of metal surfaces. It is also expected that the general size of the particles which were deposited in the circulator would be similar, independent of the chemistry of the gas system.

## 2. INTRODUCTION

Fission products deposit on both the surfaces of the materials contacting the MHTGR gas phase and on dust particles suspended in the gas or residing on the surfaces. The chemistry of the interactions and the relative surface areas of the bulk materials and the dust determine the significance of the effect of dust on the behavior of the volatile fission products.

In general, the dust particles to be found in the MHTGR fall into three classes; 1) metals or metallic oxides, 2) graphite or other carbonaceous, and 3) extraneous materials derived from impurities. The latter category includes environmental dust such as clay particles, construction debris, etc. Of particular interest is the fact that the oxidation potential for normally operating HTGR reactors (when the impurity levels are low) is slightly reducing to iron oxide. This is indicated by gas analyses from the Peach Bottom and Ft. St. Vrain reactors taken when the reactors had minimum gas impurities (1) (Table 1).

**Table 1.** Primary coolant impurities (best conditions)

Reactor	Impurity levels (vppm)					
	H <sub>2</sub>	CH <sub>4</sub>	CO	CO <sub>2</sub>	H <sub>2</sub> O	H <sub>2</sub> /H <sub>2</sub> O
Ft. St. Vrain	3	0.3	5.5	1.2	0.2	15
Peach Bottom	10	1.0	0.5	<0.5	-0.5	20

The H<sub>2</sub>/H<sub>2</sub>O ratio of 15-20 observed in both of the reactors when significant in-leakage was absent gives an oxygen potential which is very close to that required to oxidize iron to FeO. Under normal conditions the system will probably reduce the FeO to iron metal and, with only a fairly minor in-leakage of steam will oxidize the iron to FeO. Thus only minor perturbations will cycle the iron alloy surfaces from metal to oxide; this will certainly generate dust particles through the expansion and contraction of the surface layer and is the likely source of the iron oxide particles observed in the Ft. St. Vrain dust. Graphite dust could either result from chemical erosion of the surface of the graphite blocks or from mechanical abrasion from vibration. Carbon particles can also result from the carbon cycle through decomposition of carbon species.

The relative quantities of the various types of dust and the absolute quantity of total dust will vary from reactor to reactor depending on many design and operational variables. However, based on the above discussion it appears that either metallic or metallic oxide particles would be expected.

About 0.7 g of dust was obtained from the hub of the circulator removed from the Ft. St. Vrain reactor. Minor quantities were obtained from gas deflectors in the same general area, but these quantities were not sufficient for detailed examination. The dust was exposed to air both before the recovery and afterward. As mentioned earlier, this could have resulted in the oxidation of iron or FeO particles to Fe<sub>3</sub>O<sub>4</sub>.

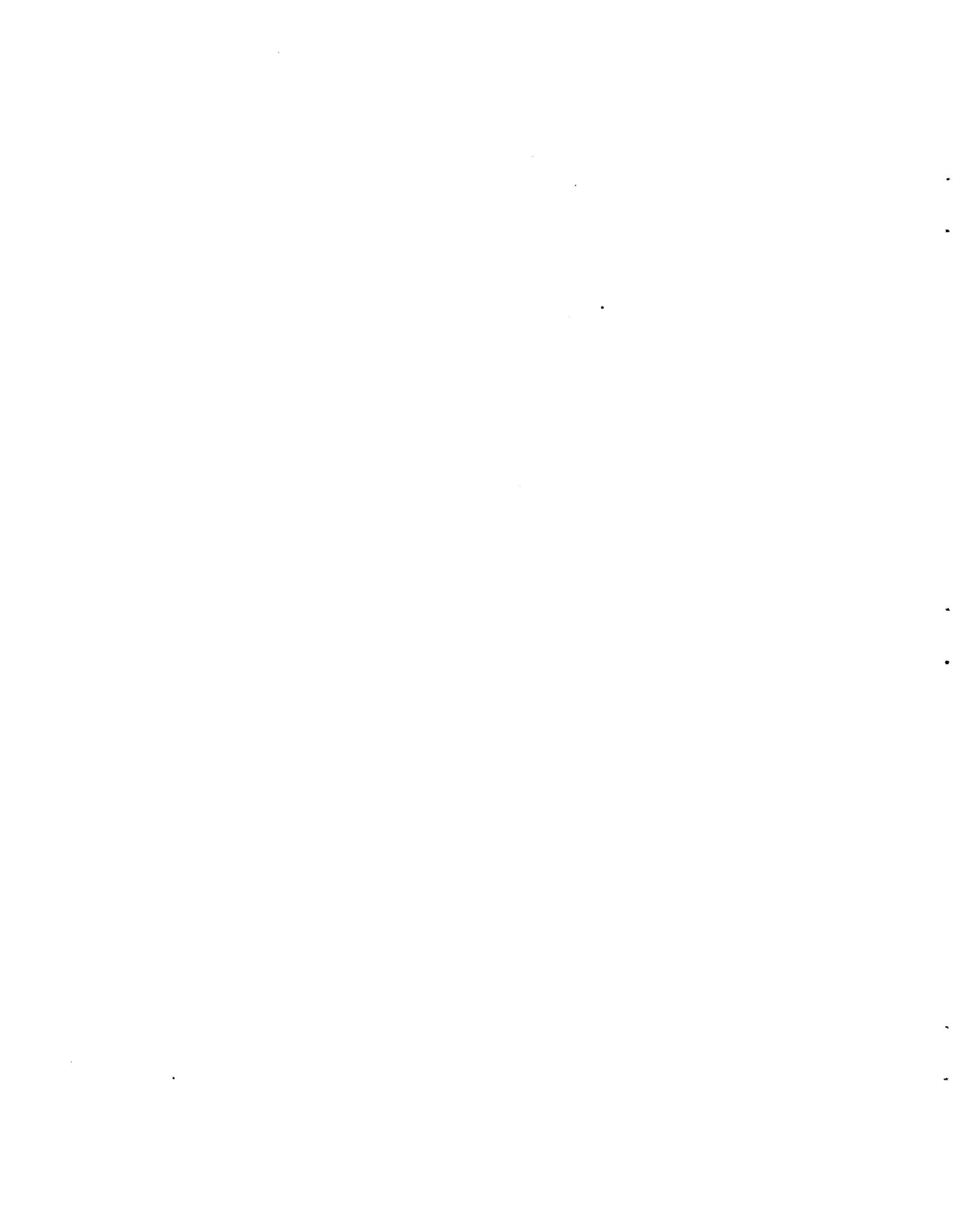
### 3. RESULTS

The dust sample was examined by x-ray diffraction to determine the composition of the dust. This examination is reported in Appendix A.

The dust was examined by SEM and BSE to determine the morphology of the particles and obtain an estimate of the particles sizes. This examination is reported in Appendix B.

### 4. REFERENCES

1. R. D. Burnette and N. L. Baldwin, *Primary Coolant Chemistry of the Peach Bottom and Ft. St. Vrain High-Temperature Gas-Cooled Reactors*, General Atomic Company (November, 1980).



**APPENDIX A**  
**RESULTS OF ANALYSIS OF FORT SAINT VRAIN REACTOR DUST**  
**BY X-RAY DIFFRACTION AND FLUORESCENCE\***

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Abstract

Both the X-ray diffraction and fluorescence analysis of the Ft. St. Vrain dust gives the overall average composition of the sample. The major compounds determined by diffraction are  $\text{Fe}_3\text{O}_4$  (85 wt %) and  $\text{MoS}_2$  (9.5 wt %). X-ray fluorescent analysis revealed that Ca, Ti, Cr, and Ni comprised the remaining 5% of the elements above atomic no. 15. An air environment greatly reduced the sensitivity to elements below Ca; however, Ca-Al-Si or other low atomic no. minerals of a crystalline nature comprising more than 5 wt % of the sample would have been detected by diffraction. No graphitic carbon was detected which indicates 0.5 wt % as the upper limit in the sample. Amorphous material could comprise up to 5 wt % of the sample. If other analytical methods find more than 5% of low atomic no. elements other than sulfur and oxygen our compositions would need some adjustment.

Summary

The results of our analysis are shown in Table A.1.

The sample of Ft. St. Vrain dust was encapsulated in a lucite plastic sample holder with a thin ~6  $\mu\text{m}$  thick mylar film window to permit X-ray analysis with a minimum of background scattering and yet contain the radioactive sample (~10 mrad on contact). Powder diffraction analysis revealed the dominant compounds to be  $\text{Fe}_3\text{O}_4$  and  $\text{MoS}_2$  both in crystalline form, Fig. A.1. We determine the relative weight fractions of the two compounds in the sample by comparing intensities with two standards mixed in the proportions of 0.5  $\text{Fe}_3\text{O}_4$  + 0.5  $\text{MoS}_2$  and .90  $\text{Fe}_3\text{O}_4$  + 0.10  $\text{MoS}_2$ . After analysis, we concluded that the Ft. St. Vrain sample contained  $90 \pm 2$  wt %  $\text{Fe}_3\text{O}_4$  and 10 wt %  $\text{MoS}_2$ . Comparison of the X-ray diffraction pattern

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\*Research sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

**Table A.1. X-ray diffraction and x-ray fluorescent analysis of Ft. St. Vrain reactor dust**

X-ray diffraction		X-ray fluorescence	
Compound	Weight %	Element	Weight %
Fe <sub>3</sub> O <sub>4</sub>	85 ± 2	Fe	62 ± 2
		O	24 ± 1 calculated*
MoS <sub>2</sub>	9.5 ± 2	Mo	5.7 ± 0.5
		S	3.8 ± 0.3
Graphite	<0.5		
Others	<6	Ca	1.9 ± 0.9
Amorphous	<5	Ti	1.1 ± 0.7 (could partly be Fe escape peak)
		Cr	1.0 ± 0.3
		Ni	1.4 ± 0.3

Radioactive isotopes

Fe<sup>55</sup> → Mnka X ray

Cd<sup>109</sup> → Agka X ray

La → BaKa X ray

No measurements made beyond 40 keV

\*X-ray fluorescence measurement was made in an air environment which drastically attenuates the X-ray signal for elements below atomic no. ~18.

P00839 12/15/89 DIF S=0.05 T=51.1 FORT ST. VRAIN REACTOR DUST

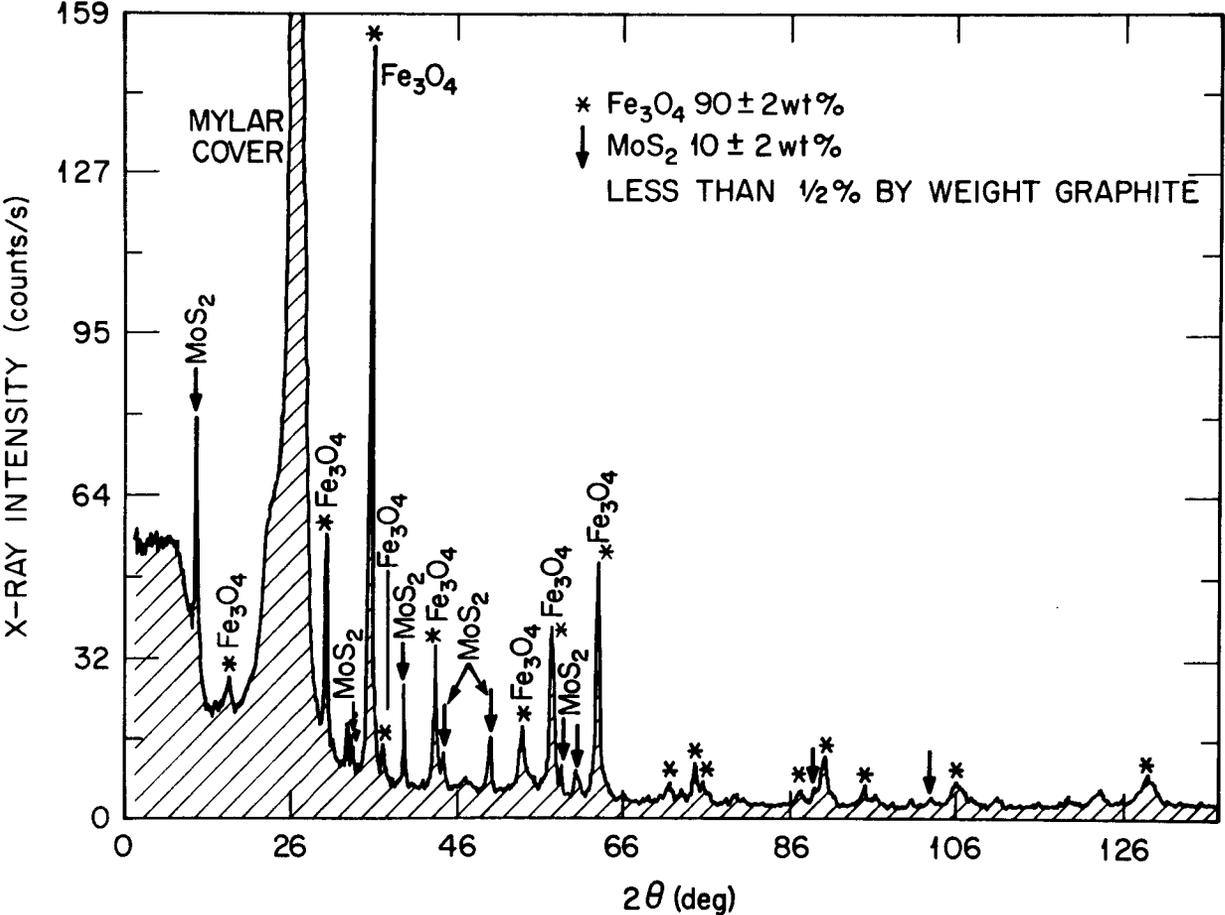


Figure A.1. X-Ray diffraction pattern of dust from Ft. St. Vrain reactor circulator.

of the Ft. St. Vrain sample (Fig. A.1) with the standard 90 wt %  $\text{Fe}_3\text{O}_4$  + 10 wt %  $\text{MoS}_2$  (Fig. A.2) shows how well the standard reproduces the pattern. Identical sample holders were used. Careful analysis for crystalline graphite showed no graphite peaks. If any graphite is present, it must be less than 0.5 wt %. Similarly, no other compounds were found. Amorphous material may be present producing only background scattering. A careful study of the background scattering produced no evidence of any amorphous phase. We conclude that if an amorphous phase is present, the quantity would be less than 5 wt %. Fluorescent analysis would detect any amorphous phase whose elements have atomic numbers above Ca and S. Table A.2 gives the powder diffraction patterns from which a positive identification was made for  $\text{Fe}_3\text{O}_4$  and  $\text{MoS}_2$ . There is no possibility that these compounds are other than stated.

X-ray fluorescence analysis was performed on the same reactor dust and the 90 wt %  $\text{Fe}_3\text{O}_4$  + 10 wt %  $\text{MoS}_2$  standard for comparison. The sample containers were identical to eliminate elemental contaminants not part of the reactor sample. Table A.3 gives a printout of the signals detected. Those elements not belonging to the reactor sample were identified by comparison with the standard. In addition, we identified the radioactive nuclides. Radioactivity in the sample was measured by counting with the Si(Li) detector and with no external source of X-ray impinging on the sample. The results are shown in Fig. A.3. Figures A.4-A.6 show the fluorescent spectrum excited by operating a Rh target at 40 keV with the reactor dust as the sample.

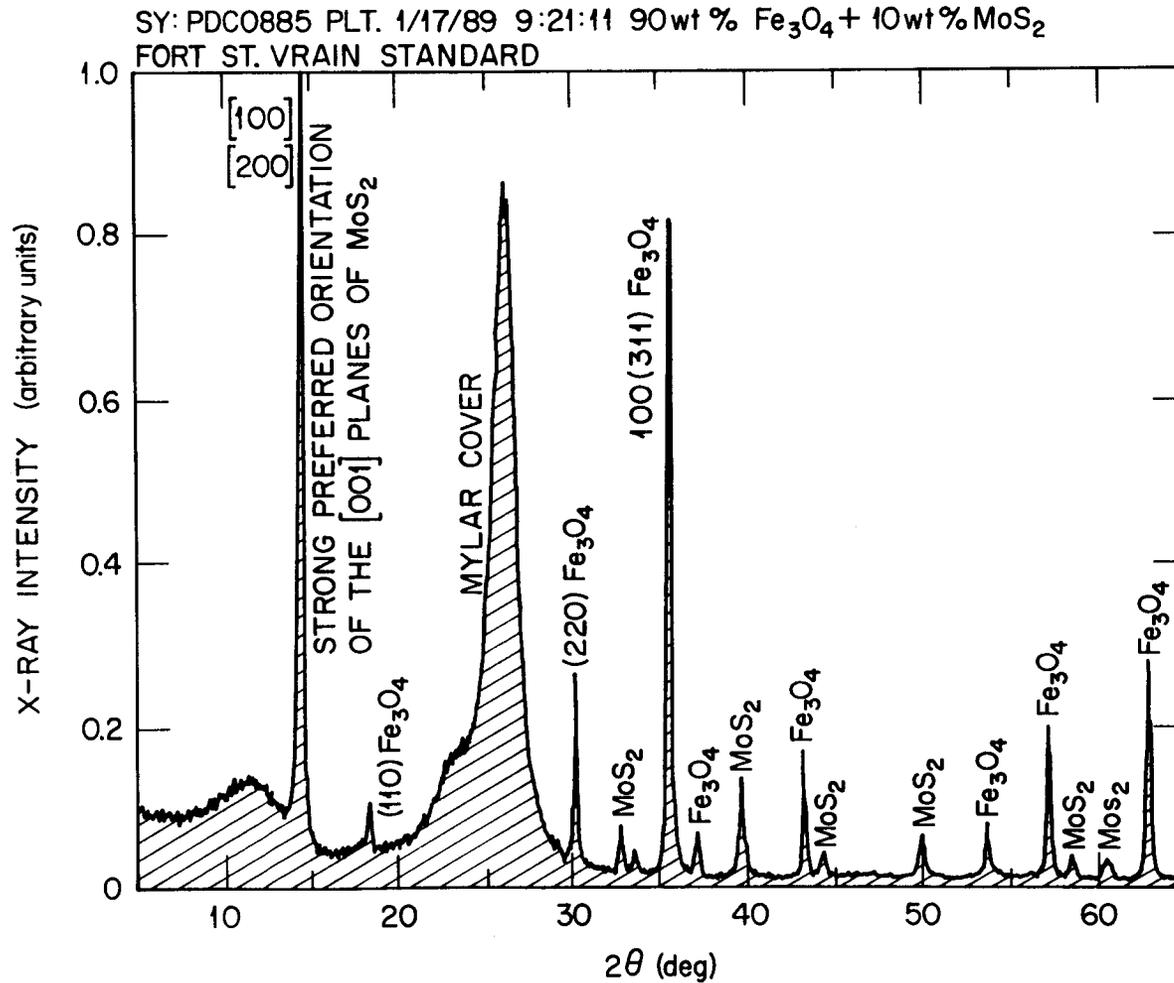
Since  $\text{Fe}_3\text{O}_4$  is in between the fully oxidized state of Fe,  $\text{Fe}_2\text{O}_3$ , and the least oxidized from, FeO, the likely conditions for formation of  $\text{Fe}_3\text{O}_4$  is a slightly reduced oxygen pressure in the presence of water vapor.  $\text{MoS}_2$  is a well known lubricant.

The following calculations were made to standardize the X-ray fluorescent measurements to weight percent. We assumed that the X-ray diffraction measurement of 90 wt %  $\text{Fe}_3\text{O}_4$  + 10 wt %  $\text{MoS}_2$  gave us the elemental composition as follows:

$$\frac{3 \times 55.85 \text{ Fe}}{231.55 \text{ Fe}_3\text{O}_4} \times .9 = 65 \text{ wt \% Fe}$$

$\text{Fe}_3\text{O}_4$

$$\frac{4 \times 16 \text{ O}_{xy}}{231.55} \times .9 = 25 \text{ wt \% O}$$



**Figure A.2.** X-Ray diffraction pattern taken on standard composed of 90 wt.%  $\text{Fe}_3\text{O}_4$  and 10 wt.%  $\text{MoS}_2$  for comparison with Ft. St. Vrain circulator dust. Strong preferred orientation occurs in the  $\text{MoS}_2$  pattern.

Table A.2. Standard X-Ray diffraction powder patterns of MoS<sub>2</sub>, Fe<sub>3</sub>O<sub>4</sub> and graphite

6-0097

6-0097 MAJOR CORRECTION

d	6.15	2.28	1.83	6.15	MoS <sub>2</sub> -2H
I/I <sub>1</sub>	100	45	25	100	MOLYBDENUM DISULFIDE (MOLYBDENITE-2H)
Rad. CuKα	λ 1.5405	Filter Ni			
Dia.	Cut off	Coll.			
I/I <sub>1</sub> DIFFRACTOMETER	d corr. sha.?				
Ref. SHANNON ET AL., NBS CIRCULAR 539 VOL. 7 (1952)					
Sys. HEXAGONAL	S.G. P6 <sub>3</sub> /mmc (194)				
a <sub>0</sub> 3.160	b <sub>0</sub> 12.295	A C 3.891			
c β	γ	Z 2 Dx 5.00			
Ref. Ibid.					
εα	nωβ	εγ	Sign		
2V	D 5.06 mp	mp	Color LEAD-GREY		
Ref. Ibid.					
Ref. Ibid.					
SAMPLE FROM THE ALPHA CORP., GREENWICH CONN.					
SPECT. ANAL. <0.1% AL, CA, CU, FE, SI					
<0.01% MG, PB, TI <0.001% MN.					
X-RAY PATTERN AT 26°C.					
SEE ALSO DATA FOR MOLYBDENITE-3R					

19-629

d	2.53	1.49	2.97	4.85	(Fe <sub>3</sub> O <sub>4</sub> )56F
I/I <sub>1</sub>	100	40	30	8	Iron Oxide (Magnetite)
Rad. CuKα	λ 1.5405	Filter Ni			
Dia.	Cut off	Coll.			
I/I <sub>1</sub> DIFFRACTOMETER	d corr. = 4.9				
Ref. National Bureau of Standards, Monograph 25, Sec. 5, 31 (1967)					
Sys. Cubic	S.G. Fd3m (227)				
a <sub>0</sub> 8.3967*	b <sub>0</sub>	c <sub>0</sub>	A C		
α β	γ	Z 8 Dx 5.197			
Ref. Ibid.					
εα	nωβ	εγ	Sign		
2V	D mp	mp	Color Black		
Ref. Ibid.					
*Refined in 1975.					
Sample obtained from the Columbian Carbon Co., New York					
17, New York. Spinel structure.					
Spectrographic analysis showed the following major					
impurities: 0.01 to 0.1% Co, 0.001 to 0.01% Ag, Al, Mg,					
*N, Mo, Ni, Si, Ti and Zn. Pattern taken at 25°C.					
Internal standard: W. Merck Index, 8th Ed., p. 455.					

GRAPHITE

23 64	C
20	I/I <sub>max</sub> x 100 Carbon/Graphite 2H CuKα X rays
26.508	100
42.402	10
44.599	50
50.675	5
54.651	80
59.854	10
77.401	30
83.394	50
85.201	5
86.906	20
93.913	5
101.600	40
132.677	10
136.619	40
148.170	5

**Table A.3.** Measured fluorescent intensities of elements identified in Fort St. Vrain circulator dust and their conversion to weigh fraction of the collected sample

X-Ray energy	X-Ray counts	X-Ray lines	X-Ray production efficiency	Conversion of count to weight fraction	Weight fraction
2.30	410	S KA1, S KA2, S KB1	7	0.04 wt/410c = $0.98 \times 10^{-4}$ wt fraction/count	0.04*
2.96	489	AR KA1, AR KA2		From air	
3.69	606	CA KA1, CA KA2	23	$200/23 \times 606 \times .037 \times 10^{-4}$	0.019
4.51	1120	TI KA1, TI KA2	75	$200/75 \times 1125 \times .037 \times 10^{-4}$	0.011
5.41	1905	CR KA1, CR KA2	130	$200/130 \times 1854 \times .037 \times 10^{-4}$	0.01
5.90	1894	MN KA1, MN KA2		Fe <sup>55</sup> radioactive isotope	
6.40	175478	FE KA1, FE KA2, MN KB1, MN KB3	200	$65 \text{ wt}/175 \times 10^3 = 0.037 \times 10^{-4}$ wt fraction/count $65 \text{ wt}/175 \times 10^3 = 0.037 \times 10^{-4}$ wt fraction/count	0.65* 0.65*
7.05	24535	FE KB1, FE KB3			
7.48	3992	NI KA1, NI KA2	212	$200/212 \times 3992c \times .037 \times 10^{-4}$	0.014
17.46	14577	MO KA1, MO KA2	223	$0.06 \text{ wt}/14577c = 0.0412$ wt fraction/count*	0.06*
19.61	3315	MO KB1, MO KB3		$0.06 \text{ wt}/14577c = 0.0412$ wt fraction/count*	0.06*
20.18	1139	RH KA1, RH KA2		Target	
22.15	755	AG KA1, AG KA2		Cd <sup>109</sup> radioactive isotope	
22.74	310	RH KBa, RH KB3		Target of X-Ray tube	
24.95	222	AG KB1, AG KB3		From Cd <sup>109</sup> radioactive isotope	

REGION (keV)	INTEGRAL	CTS/SEC
0.48 - 40.00	8.9329E + 05	4466.47

FLUORESCENT SPECTRUM BACKGROUND  
REMOVED 200 SECONDS DEC 18, 1989

\*Known from X-Ray diffraction pattern.

QUANTEX-RAY GRAPHICS  
RADIOACTIVE NUCLIDES

FORT ST. VRAIN REACTOR DUST  
PR = 200s 200s 0 INT  
V = 1024 H = 40 keV 1:1Q AQ = 40 keV 1Q

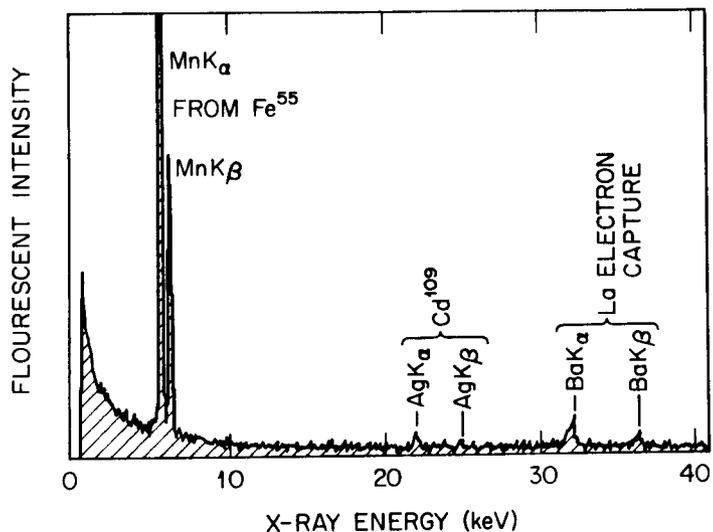


Figure A.3. Si(Li) detector measurement of extrinsic radioactivity of sample with no external X-rays on sample.

QUANTEX-RAY GRAPHICS  
FLOURESCENCE SPECTRUM, ELEMENTAL IDENTIFICATION

FORT ST. VRAIN REACTOR DUST  
PR = 2000s 200s 0 INT  
V = 1024 H = 40 keV 1:1Q AQ = 40 keV 1Q

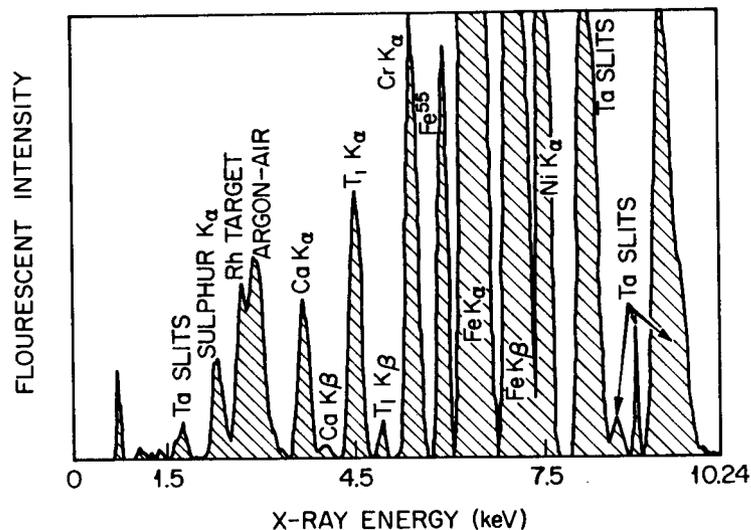


Figure A.4. X-Ray fluorescent spectrum of Ft. St. Vrain circulator dust excited with Rh target X-rays.

QUANTEX-RAY GRAPHICS

FLOURESCENCE SPECTRUM, ELEMENTAL IDENTIFICATION

FORT ST. VRAIN REACTOR DUST

PR = 2000s      200s      0 INT  
 V = 1024    H = 40keV    1:1Q    AQ = 40keV    1Q

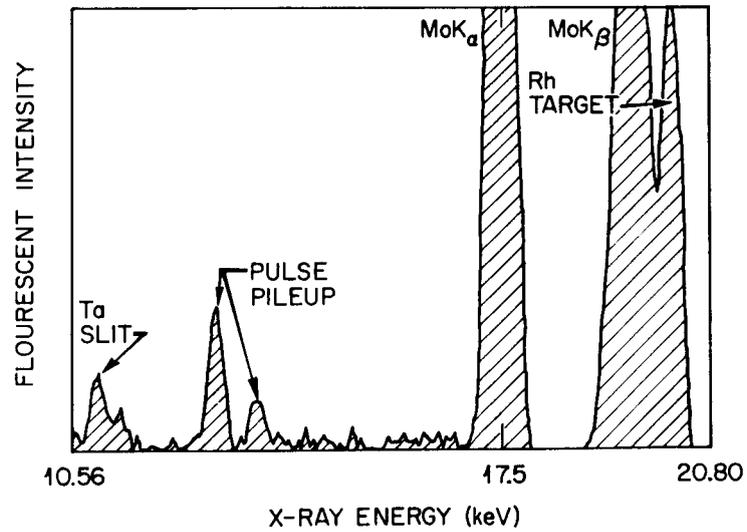


Figure A.5. X-Ray fluorescent spectrum of Ft. St. Vrain circulator dust above 10 keV.

QUANTEX-RAY GRAPHICS

FLOURESCENCE SPECTRUM, ELEMENTAL IDENTIFICATION

FORT ST. VRAIN REACTOR DUST

PR = 2000s      200s      0 INT  
 V = 1024    H = 40keV    1:1Q    AQ = 40keV    1Q

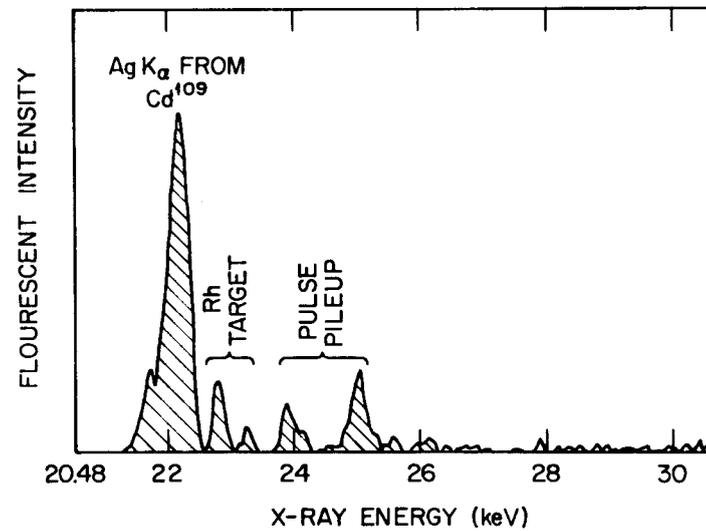


Figure A.6. X-Ray fluorescent spectrum of Ft. St. Vrain circulator dust above 20 keV.

$$\frac{95.64 \text{ Mo}}{160} \times .1 = 6 \text{ wt \% Mo}$$

MoS<sub>2</sub>

$$\frac{2 \times 32 \text{ S}}{160} \times .1 = 4 \text{ wt \% S}$$

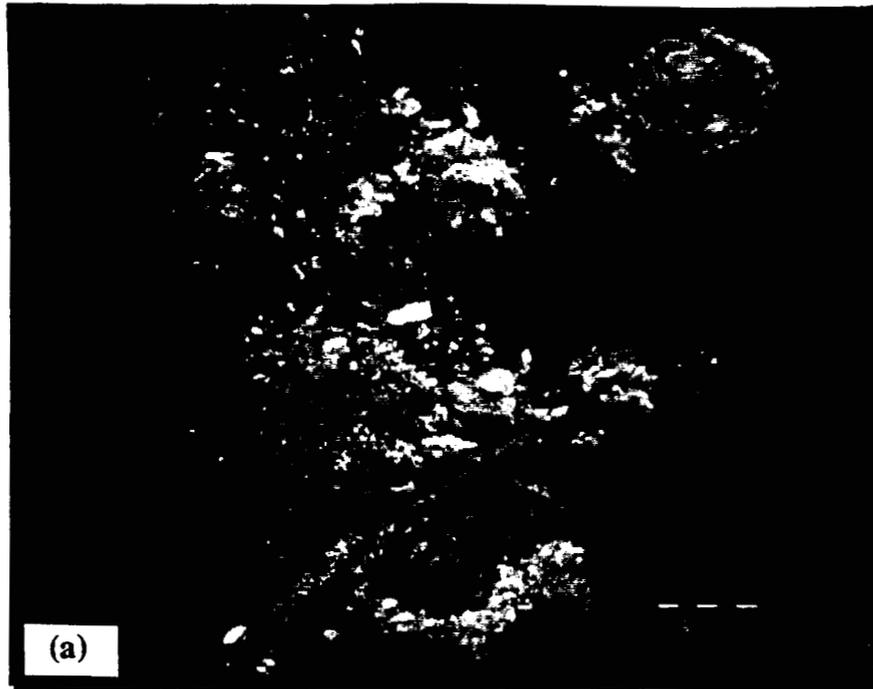
With these known weight percents, we calculated the weight percents of the other elements observed by fluorescence from their known X-ray fluorescent cross sections and absorption coefficients combined in a term we label X-ray Production Efficiency in Table A.3. These calculations for conversion to weight fractions and the measured fluorescent counts are shown on Table A.3. As the other elements comprised about 5 wt % of the sample, we iterated to obtain a sum for the components of 100%. Our standard composed of 90 wt % Fe<sub>3</sub>O<sub>4</sub> + 10 wt % MoS<sub>2</sub> was measured by X-ray fluorescent analysis under identical conditions and sample container as the Ft. St. Vrain dust to both help identify and standardize those lesser elements.

The results of these analysis are all presented in Table A.1.

## APPENDIX B

### EXAMINATIONS OF DUST BY ELECTRON MICROSCOPE

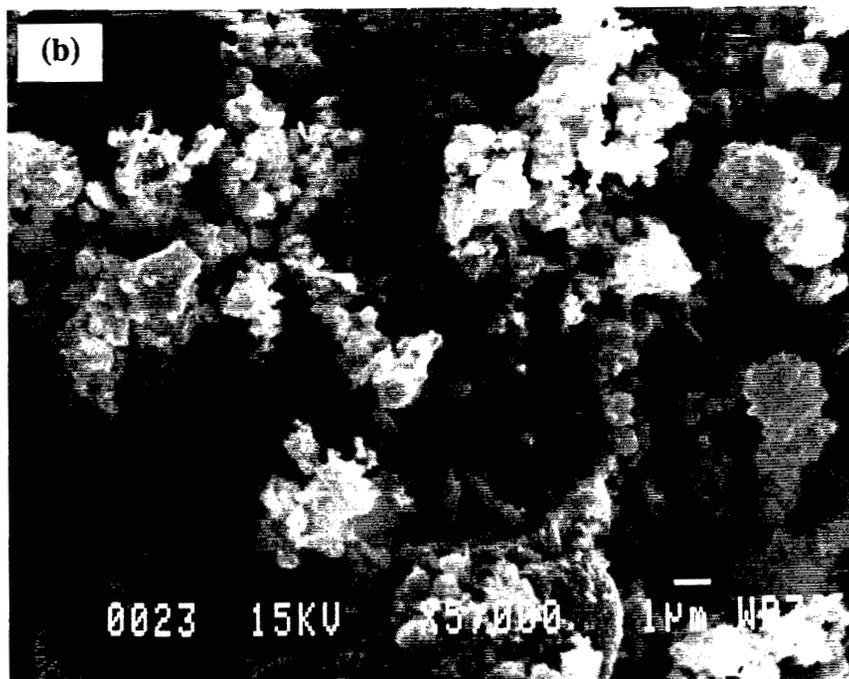
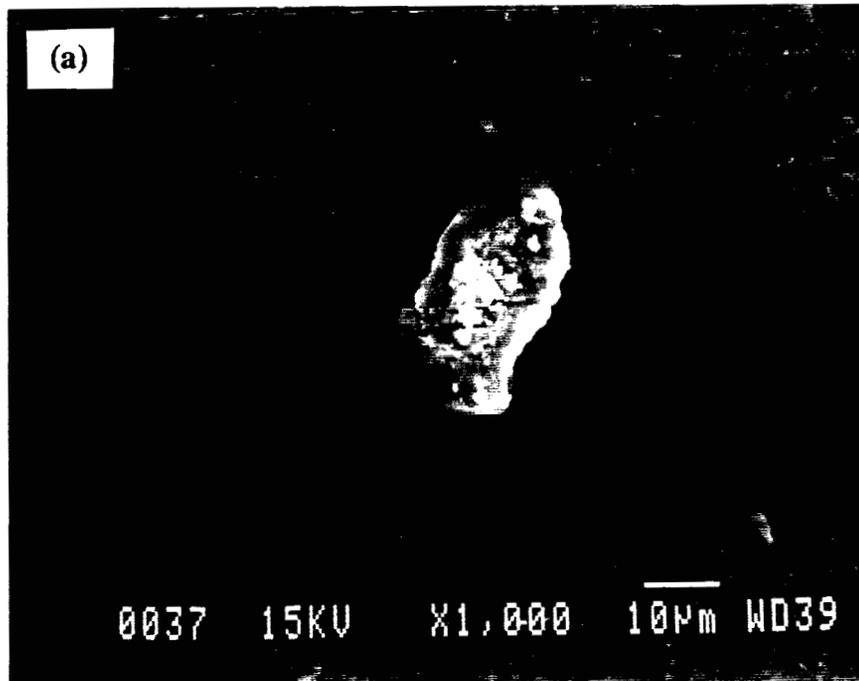
The dust was examined both by SEM and by BSE microscopy. Samples of photographs by BSE are shown in Fig. B.1. Photograph A1 (250X magnification) shows an isolated MoS<sub>2</sub> particle as a white flake. Also shown in this photograph, is a fiber identified as iron (Fe<sub>3</sub>O<sub>4</sub>). Photograph B1 (75X magnification) shows another particle of MoS<sub>2</sub>. Photograph C1 (250X magnification) shows a MoS<sub>2</sub> particle attached to an agglomeration of Fe<sub>3</sub>O<sub>4</sub> particles. These identified MoS<sub>2</sub> particles confirm the X-ray diffraction analysis showing MoS<sub>2</sub> as a significant component of the dust. The particles observed appear as discrete particles and are apparently not present as a component of the other materials. Fig. B.2 shows larger magnifications of dust by SEM. The individual particle shown in photograph A2 (1000X magnification) is high in calcium and silicon as would be expected for an environmental contaminant; this may have been introduced into the reactor or may have contaminated the recovered dust during handling. Photograph B2 (5000X magnification) shows particles identified as iron (Fe<sub>3</sub>O<sub>4</sub>). The great majority are <1 μm and are agglomerated. A very few particles appear to be non-agglomerated and >1 μm. Photograph C2 (10,000X magnification) again indicates that the majority of the particles are <1 μm with a significant number ≤0.2 μm.



**Figure B.1.** Photographs of Ft. St. Vrain dust by backscattered electron microscopy.



Figure B.1. (continued)



**Figure B.2.** Photographs of Ft. St. Vrain dust by scanning electron microscopy.

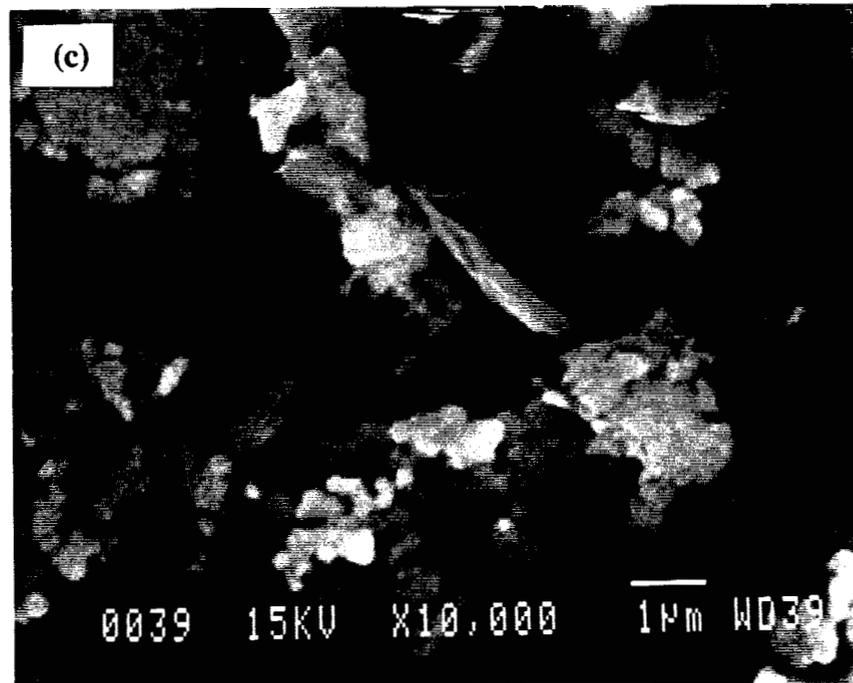


Figure B.2. (continued)



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