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Cross-Calibration Measurements Using Back-to-Back Fission Chambers

By

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A thesis

Submitted in partial fulfillment

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To the Graduate Faculty:

The members of the committee appointed to examine the thesis of Kevin Tsai find it satisfactory and recommend that it be accepted.

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TABLE OF CONTENTS

LIS	ST OF FIGURES	vi
LIS	ST OF TABLES	viii
LIS	ST OF ABBREVIATIONS	ix
AE	BSTRACT	X
1.	INTRODUCTION	1
2.	BACKGROUND	3
	2.1. Description of Fission Chambers	3
	2.1.1. Description of MPFD	4
	2.1.2. Description of BTBFC	5
	2.2. Method of Cross-Calibration	9
	2.2.1. Direct Cross-Calibration	9
	2.2.2. Indirect Cross-Calibration (Gold Foil Activation)	10
3.	EXPERIMENTAL SETUP	12
	3.1. AGN-201 Setup	12
	3.2. BTBFC System Setup	13
	3.2.1. Detector Setup	13
	3.2.2. Data Acquisition System (DAS) Setup	15
	3.2.3. Gas Supply System	17
	3.3. Neutron Activation Analysis Setup	18
4.	METHODS AND RESULTS	19
	4.1. Direct Cross-Calibration Ratios	19

	4.2. Gold Foil Coupled Cross-Calibration Measurements	21
5.	SUMMARY AND CONCLUSIONS	24
6.	REFERENCES	26
AF	PPENDIX A	27
AF	PPENDIX B	37
AF	PPENDIX C	40

LIST OF FIGURES

Figure 2-1. Schematic overview of a MPFD where the wire connections can interchangeably be
the anode or cathode depending on power supply connection. [1]4
Figure 2-2. Schematic overview of the OD 1.4 BTBFC
Figure 2-3. Sample spectrum from OD 1.4 BTBFC with U-235 Standard fission foil
Figure 2-4. Schematic overview of OD 0.8125" BTBFC7
Figure 3-1. Schematic overview of the AGN-201 reactor
Figure 3-2. Sample preparation for loading into OD 0.8125" BTBFC14
Figure 3-3. Assembled BTBFC with samples loaded, clamped, and attached to 6-foot rod14
Figure 4-1. Comparison between count-rate ratios $(\pm 1\sigma)$ from BTBFC and mass-ratios $(\pm 1\sigma)$ from
KSU. The notation is such that the integer refers to the deposit designation as given in Table 4.2.
i.e., NFT.U.250.6 is shown as .6 in this figure
Figure 4-2. BTBFC measured mass comparison with KSU reported mass. Potential explanations
for the discrepancies in measured mass is discussed in the following section (Summary and
Conclusions)
Figure 4-3. Extended plot of Figure 4-1 to include Table 4-5
Figure A.T.1-1. The initial spectrum measured using OD 1.4" BTBFC and ID-U-5 standard29
Figure A.T.1-2. Modifying the Ortec 142PC pre-amplifier to include a 1 μ F capacitor (temporarily
held in place with a plier)
Figure A.T.1-3. The built attenuator and circuit diagram
Figure A.T.1-4. Comparison of spectra measured from attenuation tests
Figure A.T.2-1. Measured spectra of ID-U-5 and NFT.U.250.5 in OD 1.4" BTBFC

Figure A.T.3-1. Background spectra measurements
Figure A.T.3-2. Direct cross-calibration spectrum measurement
Figure A.T.3-3. Setup variation test
Figure A.T.3-4. Operational Voltage evaluation measured with NFT.U.250.1 inserted
Figure B-1. Original pulse-height spectrum measured by OD 0.8125" BTBFC
Figure B-2. Resulting spectrum after 5 iterations of the smoothing function
Figure B-3. Region selected from half-max of the first spectrum in the smoothed spectrum with a
3 rd degree polynomial regression
Figure B-4. Region selected from the zeros calculated from the polynomial given in Fig. B-3 that
is used to calculate total counts, standard error, and count-rate
Figure C-1. Design dimensions (in inches) of OD 0.8125" BTBFC40

LIST OF TABLES

Table 3-1. List samples irradiated, with materials, mass, and accompanying detector	13
Table 3-2. BTBFC DAS setup for all experiments	16
Table 3-3. DAS operational Settings	16
Table 3-4. HPGe DAS setup for gamma counting	18
Table 4-1. Table of samples used in direct cross-calibrations and calculated ratios	20
Table 4-2. List of samples used in indirect cross-calibrations	21
Table 4-3. Calculated mass in comparison with KSU reported mass	22
Table 4-4. Ratios calculated from values given in Table 4-3	23

LIST OF ABBREVIATIONS

ANL	Argonne National Laboratory
ATRC	Advanced Test Reactor Critical Facility
BTBFC	Back-to-Back Fission Chamber
INL	Idaho National Laboratory
ISU	Idaho State University
KSU	Kansas State University
MPFD	Micro-Pocket Fission Detector
NAA	Neutron Activation Analysis
NEET	Nuclear Energy Enabling Technologies Program
OD 0.8125" BTBFC	Outer diameter 0.8125" Back-to-Back Fission Chamber
OD 1.4" BTBFC	Outer diameter 1.4" Back-to-Back Fission Chamber
TREAT	Transient Test Reactor
TREAT-IRP	Transient Test Reactor-Integrated Research Project
ZPPR	Zero Power Physics Reactor

Cross-Calibration Measurements Using Back-to-Back Fission Chambers Thesis Abstract—Idaho State University (2018)

An integrated research project (IRP) for advanced instrumentation for Transient Reactor (TREAT) at Idaho National Laboratory (INL) was initiated. A task within the IRP is the continuing development of the Micro-Pocket Fission Detector (MPFD) as a real-time, in-core neutron flux sensor. Fissionable deposits of the MPFD need to be well characterized to provide accurate neutron flux measurements. Cross-calibration measurements of MPFD fissionable deposits were made using Back-to-Back Fission Chambers (BTBFC) as a method of characterization of fissionable deposits. BTBFC ratio and mass measurements are compared to the mass measurements reported by Kansas State University (KSU). BTBFC ratio measurements provided partial verification of the mass reported by KSU. BTBFC mass determinations indicated a discrepancy in comparison to KSU reported mass. Overall results indicate the need for further study on the use of the BTBFC in cross-calibration measurements as well as the need for destructive analysis of the MPFD fissionable deposits.

Key Words: BTB, TREAT, MPFD, Fission Chamber, Cross-calibration

1. INTRODUCTION

An integrated research project (IRP) for advanced instrumentation for transient reactor testing was initiated in support of the efforts of restarting and resumption of activities at the Transient Reactor Test (TREAT) Facility at Idaho National Laboratory (INL). The continuing development of the Micro-Pocket Fission Detector (MPFD) at Kansas State University (KSU) became a task within the TREAT-IRP. The MPFD is developed to provide real-time in-core neutron flux measurements of TREAT during transients [1]. Currently, the neutron reactive materials within the sensor regions of the MPFD are both produced and characterized at KSU [2].

The purpose of this project is to support the development and deployment of MPFD at TREAT by providing independent verification and further characterization of the neutron reactive material, fissionable deposits, used in the sensor region of the MPFD—a project funded through the TREAT-IRP and Nuclear Energy Enabling Technologies (NEET) [1]. The mass of the fissionable deposits in MPFD samples will be measured through the method of crosscalibration Back-to-Back Fission Chambers (BTBFC) in comparison with KSU's method of alpha-particle spectroscopy.

BTBFC, developed by Argonne National Laboratory (ANL), are coupled 2π fission chambers with accessible sensor regions where two fission foils can be placed back-to-back to measure relative fission rates [3][4]. Two BTBFCs are used in this project—the outer diameter (OD) 1.4-inch BTBFC (known as "legacy detectors" from ANL) and a newly designed and fabricated OD 0.8125-inch BTBFC. The standard used in mass cross-calibration against MPFD samples is a U-235 fission foil that has been well calibrated by ANL [3]. The results of the cross-

1

calibration mass measurement will be used as independent verification of alpha-particle measurements and be provided to KSU and INL to further the development of the MPFD.

This paper provides background information regarding the MPFD and BTBFC. The paper will also cover the design and operating characteristics of both OD 1.4" and OD 0.8125" BTBFC. Measurements made from cross-calibration of the U-235 standard and three MPFD samples will be presented along with their analysis and derived conclusions.

2. BACKGROUND

2.1. Description of Fission Chambers

Neutron detectors are generally based on detection of charged particle released from neutroninduced reactions [5]. One category of neutron detectors are fission chambers. Typical fission chambers are ion chambers with neutron reactive (fissionable) materials thinly deposited within the sensor region. Neutron-induced fission releases a back-to-back pair of highly energetic and highly charged fission fragments. Provided that the deposits are sufficiently thin, one fission fragment will be released towards/into the fill-gas region of the chamber with high efficiency while the other will be absorbed in the backing material surface that the neutron reactive materials are deposited on. The fission fragments that escape into the chamber's fill-gas region ionizes fill-gas, typically argon. Electrons from ionizing events are collected at the anode resulting in an output signal in the form of electric pulses or current representing fission events. Signals generated from fission fragment ionization are generally discernable from others generated from other particles such as alpha, beta, or gamma particles due to the large differences in energy deposition. With proper characterization of the detector, it is possible to calculate the neutron flux as a function of the signals generated from fission fragments.

2.1.1 Description of MPFD

MPFDs utilizes the same concept of typical fission chambers with two parallel electrode wires and a neutron reactive material hermetically sealed in an argon-filled sensor region. A schematic overview of the MPFD is shown in Figure 2-1 [1]. The design of the MPFD consist of stacked ion chambers with varying neutron reactive materials for measuring neutron flux and changes in spectral indices. The neutron reactive material of MPFD samples are natural uranium that is 2 mm in diameter and unknown thickness [2]. The samples' substrate on which natural uranium is deposited on are temperature resistant ceramic made of alumina (Al₂O₃) disks 0.185 inch in diameter and 0.059-inch-thick with platinum-titanium layers 2mm in diameter and 500 Å-50 Å thick evaporated on top [2]. The metallic layer is used as an electrode for electrodeposition of natural uranium. The mass of uranium electrodeposited were chosen to withstand high neutron fluence from TREAT transients, therefore the mass is ~1 µg Nat U to avoid melting the fissile material.



Figure 2-1. Schematic overview of a MPFD where the wire connections can interchangeably be the anode or cathode depending on power supply connection [1].

The MPFD design differs from traditional fission chambers due to its signal generation. Traditional fission chambers are designed based on full energy deposition of the fission fragments in the fill-gas, but the MPFD does not require full energy deposition of the fission fragments in the fill-gas. The amount of ionization from fission fragments, despite not depositing their full energy, still remains significantly larger than the amount of ionization from background radiation such as electrons and gamma particles allowing easy discrimination between fission events and background [6]. This departure from traditional fission chambers allows the MPFD to attain smaller detector geometries and lower fill-gas pressure.

2.1.2 Description of BTBFC

BTBFCs also utilizes the same concepts as traditional fission chambers with parallel plate electrodes and neutron reactive material deposited on top of the cathode plate. A schematic overview of the OD 1.4" BTBFC is shown in Figure 2-2. The BTBFC contains two symmetric 2π ion chambers oriented back-to-back. The ion chambers are bisected by two fission foils. Fission foils are commonly metal backings with neutron reactive coatings deposited on top. BTBFC fission foils are made of stainless steel (SS-316) disks 34.3 mm in diameter and 0.15 mm thick (1.35" dia. 0.006" thick) [3].



Figure 2-2. Schematic overview of the OD 1.4 BTBFC.

The design of the BTBFC allows easy access to the sensor region and swapping of fission foils, therefore the sensor region is not hermetically sealed. Constant flow of P-10 (90% argon and 10% methane) fill-gas flows through the detector keeps the sensor region purged of gas impurities.

The BTBFC generates its signals using full energy deposition of fission fragments. Pulseheight measurement of the signal will produce a spectrum proportional to particle energy shown in Figure 2-3. Pulses above (to the right of) the "valley", known as the geometrical cut-off, are from fission events—the two "smeared" peaks represent the energy peaks of the large fission fragment (first peak) and the small fission fragment (second peak). Pulses below (to the left of) the cut-off are from background particles [3]. However, signal generation from full energy deposition makes miniaturization difficult. The distance between the neutron reactive material and the anode plate is limited to a minimum of 0.20 inches to account for fission fragment range in atmospheric P-10 gas.



Figure 2-3. Sample spectrum from OD 1.4 BTBFC with U-235 Standard fission foil.

An additional function of the BTBFC besides real-time flux measurements is crosscalibration mass measurements. The mass of one fission foil can be determined relative to the other by measuring the reaction rate ratio of two fissile foils simultaneously in the BTBFC (see following section).

The two BTBFCs used in this TREAT-IRP are the OD 1.4" BTBFC and the OD 0.8125" BTBFC. The OD 1.4" BTBFC and U-235 standard fission foil used in this TREAT-IRP are the same "legacy" BTBFC and fission foil developed and used by ANL for the Zero Power Physics Reactor (ZPPR) cross-calibration measurements [3][4]. Additionally, this detector was also used and tested in the Advanced Test Reactor Critical Facility (ATRC) at INL as a real-time neutron flux detector [7]. Experimental setup from the ATRC test was used as the reference point for reusing the OD 1.4" BTBFC within this TREAT-IRP.

The OD 0.8125" BTBFC is designed and fabricated within the TREAT-IRP for crosscalibrating MPFD samples. The schematic overview of the OD 0.8125" BTBFC is given in Figure 2-4. The design specifications are given in Appendix C.



Figure 2-4. Schematic overview of OD 0.8125" BTBFC.

This BTBFC is designed with a 0.259-inch gap to compensate for the thickness of the alumina substrate in MPFD samples. The size is designed for accessing the AGN-201 reactor core center to compensate for the low reaction rate of MPFD samples having significantly less deposited fissile materials.

2.2. Method of Cross-Calibration

Calibration, the comparison of an unknown, or the performance thereof, with a calibrated standard of known accuracy and precision, is a critical procedure in sensor development and deployment. A primary function, by design, of the BTBFC is to provide cross-calibration measurements of fissile materials—the measurement of mass ratios by fission counting. The BTBFC method of fission counting through neutron induced fission has the potential to provide significantly faster counts-rates used in calibration calculations within a significantly shorter time in comparison to alpha-particle spectroscopy. The use of the BTBFC can therefore provide measurement verification of another method, alpha-spectroscopy, within significantly less counting time and better counting statistics.

2.2.1 Direct Cross-Calibration

Direct cross-calibration measurements are performed by loading a single BTBFC with two fission foils/samples and measuring the two pulse-height spectra simultaneously to determine fission counts/count-rates (an example of a pair of measured spectra is shown in Figure A.T.3-2 in Appendix A). Since both foils are loaded back-to-back, the flux experienced by both samples are identical. The counts/count-rates from each of the fission foils/samples are proportional to mass, detector efficiency, cross-section, and flux given by:

$$C \propto \left(\frac{m\epsilon}{A}\right) \left(\sum_{i} \sigma_{f}^{i} W^{i} \varphi_{f} + \sum_{i} \sigma_{s}^{i} W^{i} \varphi_{s}\right)$$

where m is the mass, C, is the total counts or count-rate from fission, ε is the counting efficiency (based on fission fragment escape probability and detector efficiency), A is the atomic weight of

the material, σ is the cross-section, and W is the weight percent from all of the fissionable isotope for neutron induced fissions by fast neutron flux ϕ_f and thermal neutron flux ϕ_s [3]. The mass ratios calculated from the proportionality is then given by:

$$\frac{m_1}{m_2} = \left(\frac{C_1 \varepsilon_2 A_1}{C_2 \varepsilon_1 A_2}\right) \left(\frac{\sum_i \sigma_{f_2}^i W_2^i \phi_{f_2} + \sum_i \sigma_{s_2}^i W_2^i \phi_{s_2}}{\sum_i \sigma_{f_1}^i W_1^i \phi_{f_1} + \sum_i \sigma_{s_1}^i W_1^i \phi_{s_1}}\right)$$

Direct cross-calibration within this project will only apply to MPFD samples (see Appendix A). In the context of direct cross-calibrating nearly identical MPFD samples (with respect to flux, counting efficiency, cross sections, and isotopic composition), the equation above simplifies to:

$$\frac{\mathbf{m}_1}{\mathbf{m}_2} = \frac{\mathbf{C}_1 \mathbf{\varepsilon}_2}{\mathbf{C}_2 \mathbf{\varepsilon}_1} \approx \frac{\mathbf{C}_1}{\mathbf{C}_2} \ .$$

2.2.2 Indirect Cross-Calibration (Gold Foil Activation)

While direct cross-calibration is performed between MPFD samples, cross-calibration between the U-235 standard and MPFD samples requires an indirect method (see Appendix A). A medium is needed to reference relative fission rates of the U-235 standard and MPFD samples measured from two separate BTBFC—neutron flux. This is also necessary due to the lack a standard that will fit into the smaller fission chamber required for core center irradiations.

The measured reaction rates from the fissile material must be scaled based on measured neutron flux. The mass ratios must be calculated though the original equation, without simplification, mentioned in the prior subsection.

Neutron activation analysis (NAA) of gold foils was chosen as the method of neutron flux determination. Gold foils loaded in the BTBFC with the fission foils experiences a neutron flux and undergoes (n, γ) reaction emitting 411keV gammas with a half-life of 2.695 days. By gamma-particle counting of activated gold in a radiation counter, the neutron flux experienced by the gold can be calculated through the following set of equations [5]:

$$A_{\infty} = \phi \Sigma V$$

$$A_{\infty} = \frac{\lambda(C - B)}{\epsilon(1 - e^{-\lambda t_0})e^{\lambda t_0}(e^{-\lambda t_1} - e^{-\lambda t_2})}$$

where:

 A_{∞} = saturated activity

- φ = neutron flux
- $\Sigma = macroscopic cross section$
- V = volume
- $\lambda = decay constant$
- C B = Net counts measured in radiation counter
- $\epsilon = \text{counting efficiency}$
- $t_0 = total irradiation time$
- t_1 = radiation counter start time
- t_2 = radiation counter stop time

3. EXPERIMENTAL SETUP

3.1. AGN-201 Setup

Cross-calibration measurements are performed in the AGN-201 test reactor shown in Figure 3-1. Measurements made with the OD 1.4" BTBFC are performed in the lower right "Access port" center. Measurements made with OD 0.8125" BTBFC are performed at the core-center accessed through the central irradiation facility (CIF). The reactor power is maintained at ~3.2 W steady-state for all experiments.



Figure 3-1. Schematic overview of the AGN-201 reactor.

3.2. BTBFC System Setup

3.2.1. Detector Setup

Samples used in cross-calibration measurements are given in Table 3-1. The samples to be loaded in the OD 0.8125" BTBFC are first adhered to individual SS-316 disks (OD 0.72", thickness 0.006") shown in Figure 3-1. The loaded BTBFC is attached to a 6-foot rod via the clamp screw shown in Figure 3-3. Signal cables and gas tubes are connected, and the entire assembly is wrapped in a plastic bag secured at the sensor region with a zip-tie.

Table 3-1. List samples irradiated, with materials, mass, and accompanying detector.			
Sample ID	Material/Mass	BTBFC used	
ID-U-5 ^a	$119.82 \pm 0.22 \ \mu g \ (98.4409 \ wt\% \ U-235)$	OD 1.4"	
NFT.U.250.1 ^b	$0.817 \pm 0.0164 \ \mu g \ (NatU)$	OD 0.8125"	
NFT.U.250.5 ^b	$0.768 \pm 0.0159 \ \mu g \ (NatU)$	OD 0.8125"	
NFT.U.250.6 ^b	$0.732 \pm 0.0155 \ \mu g \ (NatU)$	OD 0.8125"	
Gold Foil #1°	0.0058 g Au	OD 0.8125"	
Gold Foil #2 ^c	0.0056 g Au	OD 0.8125"	
Gold Foil #3 ^d	0.0138 g Au	OD 1.4"	
Gold Foil #4 ^c	0.0062 g Au	OD 0.8125"	
^a Referenced as NEW5-01 [3].			
^b MPFD samples from KSU with mass determined through alpha-particle spectroscopy.			

^oMPFD samples from KSU with mass determined through alpha-particle spectroscopy. ^cGold foil is placed on top of a blank MPFD alumina disk.

^dGold foil is placed on top of a bare SS-316 disk (OD 1.35", thickness 0.006").



Figure 3-2. Sample preparation for loading into OD 0.8125" BTBFC.



Figure 3-3. Assembled BTBFC with samples loaded, clamped, and attached to 6-foot rod.

3.2.2 Data Acquisition System (DAS) Setup

The DAS setup is shown in Table 3-2. The core components of the DAS setup at the ATRC was replicated with the exception of an attenuator (see Appendix A). The BTBFC is powered by the high voltage power supply through the preamplifiers creating sufficient electric fields between the electrodes of the chamber for signal generation. Pulses are passed from each half of BTBFC through two individual sets of pre-amplifier, attenuator, amplifier, and MCA. The settings for HV power supply output and amplifier gain is given in Table 3-3. The initial detector evaluations for determining operational settings is given in Appendix A.

The Ortec Maestro 32 MCA Emulator software was used to plot pulse-height spectra from the BTBFC system during experiments in real-time. Background signals contributes a significant amount of dead-time in the MCA and requires discrimination. Since the background signal level varies between experiments, the lower level discriminator (LLD) setting on the MCA is determined at the start of each experiment.

The pulse-height spectrum following the completion of an experiment is exported as a text file and replotted on Microsoft Excel for analysis and calculations.

Table 3-2. BTBFC DAS setup for all experiments.				
Component (amount)	Model	Input	Output	
BTBFC	OD 1.4"	HV Power Supply	Pre-amplifier	
	OD 0.8125"	(Pre-amplifier)		
Pre-amplifier (2)	Ortec 142PC	BTBFC,	Attenuator	
		HV Power Supply		
High Voltage Power	Ortec 556	NIM BIN	Pre-amplifier	
Supply				
NIM BIN	Tennelec TB-3	Building Power	HV Power Supply,	
	TC-911		Amplifier	
Attenuator (2)	See Appendix A	Pre-amplifier	Amplifier	
Amplifier (2)	Ortec 572A	Attenuator	MCA	
Multi-Channel	Ortec Easy-MCA	Amplifier	CPU	
Analyzer (2)				

Table 3-3. DAS operational Settings.				
BTBFC	Voltage (V)	Shaping (µs)	Amplifier Gain	Polarity
OD 1.4"	200	0.5	12.5	Positive
OD 0.8125"	300	0.5	12.5	Positive

3.2.3 Gas Supply System

Continuous flow of P-10 fill-gas is supplied from the gas cylinder. A regulator/flowmeter is attached to the cylinder outlet to facilitate proper flow. A plastic gas tube connects the flowmeter to the SS-316 gas tube of the BTBFC. Another plastic gas tube connects the outflow SS-316 gas tube to the MSA P100 filter.

3.3. Neutron Activation Analysis Setup

Neutron activation analysis is performed in a High-Purity Germanium (HPGe) detector. The HPGe detector is placed in a Ortec High Performance Low Background Lead Shield (HPLBS). The setup including DAS is given in Table 3-4. Irradiated gold foils are removed from the alumina and SS-316 backing and placed on top of the HPGe within the HPLBS via plastic holders.

Ortec GammaVision-32 is used to measure the pulse-height spectrum. Net count-rates from gold foil decay (411 keV) is measured from the software.

Table 3-4. HPGe DAS setup for gamma counting.			
Component Model		Input	Output
HPGe	GEM-C5970P4	HV Power Supply	Amplifier
Coolant	Ortec X-Cooler III	Building Power	HPGe
High Voltage Power	Ortec 459	NIM BIN	HPGe
Supply			
NIM BIN	NH-84A	Building Power	HV Power Supply,
			Amplifier
Amplifier	Ortec 575A	HPGe	MCA
Multi-Channel Analyzer	Ortec Easy-MCA	Amplifier	CPU

4. METHOD AND RESULTS

4.1. Direct Cross-Calibration Ratios

Three direct cross-calibration measurements were conducted in the OD 0.8125" BTBFC. Selected samples with corresponding placements within the BTBFC are given in Table 4-1. Measured integrated counts from each spectrum is used to determine an average count-rate and standard error (see Appendix B). The count-rates from each individual measurement are used to calculate count-rate ratios also given in Table 4-1. Additionally, mass ratios from KSU's mass measurements are calculated and presented in Table 4-1. Figure 4-1 plots the count-rate ratios presented in Table 4-1.

Table 4-1. Table of samples used in direct cross-calibrations and calculated ratios.				
Test #	Sample Position	(Side B/Side A)	KSU Mass	
		Count-Rate Ratio ^a $\pm 1\sigma$	Ratio ^b $\pm 1\sigma$	
4.1-1	Side A: NFT.U.250.6	1.200 ± 0.031	1.116 ± 0.033	
	Side B: NFT.U.250.1			
4.1-2	Side A: NFT.U.250.1	0.894 ± 0.021	0.940 ± 0.027	
	Side B: NFT.U.250.5			
4.1-3	Side A: NFT.U.250.5	0.899 ± 0.020	0.953 ± 0.028	
	Side B: NFT.U.250.6			
^a Ratios are calculated with $\pm 1\sigma$ from the count-rates measured by the BTBFC.				
^b Samples provided by KSU from alpha-particle spectroscopy measurements.				



Figure 4-1. Comparison between count-rate ratios $(\pm 1\sigma)$ from BTBFC and mass-ratios $(\pm 1\sigma)$ from KSU. The notation is such that the integer refers to the deposit designation as given in Table 4-1. i.e., NFT.U.250.6 is shown as .6 in this figure.

4.2. Gold Foil Coupled Cross-Calibration Measurements

Four gold foil coupled cross-calibration measurements were taken. The samples used and their positions within the BTBFC are given in Table 4-2. Gold foil activation analysis is performed with a High-Purity Germanium (HPGe) detector to calculate the thermal neutron flux experienced by each sample. Average count-rates from each fissile material is calculated by the same method described in Appendix B. Calculated MPFD sample fissile mass in comparison with KSU reported mass is given in Table 4-4 and Figure 4-2.

The measured mass given in Table 4-3 is used to calculate mass ratios similar to Table 4-1 and Figure 4-1. The calculated ratios are given in Table 4-4. An extended plot of Figure 4-1 containing the ratios from Table 4-4 is given in Figure 4-3.

Table 4	Table 4-2. List of samples used in indirect cross-calibrations.			
Test #	BTBFC	Sample Position		
4.2-1	OD 1.4"	Side A: ID-U-5		
		Side B: Gold Foil #3 (0.0138 g)		
4.2-2	OD 0.8125"	Side A: Gold Foil #1 (0.0058 g)		
		Side B: NFT.U.250.5		
4.2-3	OD 0.8125"	Side A: Gold Foil #2 (0.0056 g)		
		Side B: NFT.U.250.6		
4.2-4	OD 0.8125"	Side A: Gold Foil #4 (0.0062 g)		
		Side B: NFT.U.250.1		

Table 4-3. Calculated mass in comparison with KSU reported mass.				
Material ID	Measured Mass $\pm 1\sigma$ (µg)	KSU Reported Mass $\pm 1\sigma$ (µg)		
NFT.U.250.1	0.367 ± 0.0061	0.817 ± 0.0164		
NFT.U.250.5	0.309 ± 0.0056	0.768 ± 0.0159		
NFT.U.250.6	0.305 ± 0.0052	0.732 ± 0.0155		



Figure 4-2. BTBFC measured mass comparison with KSU reported mass. Potential explanations for the discrepancies in measured mass is discussed in the following section (Summary and Conclusions).

Table 4-4. Ratios calculated from values given in Table 4-3.				
Ratio	Table 4-5 Values Used	Calculated Ratio $\pm 1\sigma$		
Gold (.1/.6)	BTB (.1)/BTB (.6)	1.205392 ± 0.028461		
Gold (.5/.1)	BTB (.5)/BTB (.1)	0.842642 ± 0.020543		
Gold (.6/.5)	BTB (.6)/BTB (.5)	0.984529 ± 0.024296		



Figure 4-3. Extended plot of Figure 4-1 to include Table 4-4.

5. SUMMARY AND CONCLUSIONS

The performance and operational settings of the "legacy detector" OD 1.4" BTBFC based on past experiments were verified. A newly designed and fabricated OD 0.8125" BTBFC was also evaluated for use with the OD 1.4" BTBFC for cross-calibration measurements. Cross-calibration measurements of MPFD samples from KSU has been performed through the use of two BTBFC.

Key measurements made with BTBFC includes count-rate ratios and mass determination of the MPFD samples. Count-rate ratios were measured in two methods: direct cross-calibration and indirect cross-calibration. The count-rate ratios from indirect cross-calibration were also further used for mass determination of MPFD samples. The standard used in mass determination was the U-235 fission foil specifically made and calibrated for use with the BTBFC.

Measured count-rate ratios from both methods of cross-calibration lie within close proximity with the calculated ratios obtained KSU's mass measurements via alpha-particle spectroscopy. The results therefore verify the relative response of the fissionable deposit of MPFD samples in a neutron field.

Mass determinations of MPFD by indirect BTBFC cross-calibration with the U-235 standard fell significantly below KSU's reported amount. Determined mass of MPFD samples are all below the reported amount by a factor of 2-2.5. The large discrepancy in BTBFC measured mass against KSU reported mass can most likely be caused by the following claims:

The detector efficiency (fission fragment detected/ fission fragment emitted) of the OD
1.4" BTBFC and/or the OD 0.8125" BTBFC may not be near 100% nor equal. Therefore,
the assumption made in indirection cross-calibration (where ε was assumed to be 100%)

for both detectors) may not apply resulting in a factor difference in calculated mass.

- 2. Fission fragment escape probably of the fission fragment emitted towards the fill-gas from the MPFD samples may be less than that of the U-235 standard (ID-U-5). The mass measurements from indirect cross-calibration may then result in a factor difference lower than the alpha-spectroscopy measurements; since alpha-particles have higher escape probability than fission fragments, mass measurements involving fission counting methods will yield smaller values than alpha-particle counting measurements.
- 3. An error in calculating the mass either from BTBFC cross-calibration with U-235, or from alpha-particle spectroscopy, or both (where the error is cancelled when calculating ratios).

Efficiency measurements using a well characterized fission or alpha-particle source can be performed on both BTBFC to determine the validity of the first claim. Determination of absolute fissionable deposit on each MPFD sample through isotopic dilution analysis (destructive analysis) will be required to determine the validity of the second and third claim.

6. REFERENCES

[1]. T. UNRUH, et al., "Enhanced Micro-Pocket Fission Detector for High Temperature Reactors – FY 17 Final Project Report," INL/EXT-17-43397 (2017).

[2]. M. REICHENBERGER, et al., "Electrodeposition of uranium and thorium onto small platinum electrodes," *Nuclear Instruments and Methods in Physics Research A*, **812**, pp. 12-16 (2016).

[3]. W. P. POENITZ, et al., " 235 U(n,f), 238 U(n, γ), 238 U(n,f) and 239 Pu(n,f) Reaction Rate Measurement Calibrations at ZPPR," ANL-87-5 (1987).

[4]. S. B. BRUMBACH, D. W. MADDISON, "Reaction Rate Calibration Techniques at ZPPR for ²³⁹Pu Fission, ²³⁵U Fission, ²³⁸U Fission, and ²³⁸U Capture," ANL-82-38 (1982).

[5] G. F. KNOLL, Radiation Detection and Measurement. 4th ed. Wiley (2010).

[6]. M. REICHENBERGER, et al., "MCNP6 Simulated Performance of Micro-Pocket Fission Detectors (MPFDs) in the Transient REActor Test (TREAT) Facility," *Annals of Nuclear Energy*, **104**, pp. 191-196 (2017).

[7]. B. CHASE, et al., "Initial Back-to-Back Fission Chamber Testing in ATRC," INL/EXT-14-31830 (2014).

APPENDIX A

Several key initial tests were conducted with the setup shown in Table 3-1 and is given in Table A-1. The fissile samples used in initial tests as well as final measurements is given in Table 3-2. The measured spectra and conclusions made from the tests are given below.

Table A-1. Key initial tests performed in chronological order.					
Test #	BTBFC	Material Inserted	Purpose		
A.T.1	OD 1.4"	ID-U-5 only	Operational Settings		
A.T.2	OD 1.4"	ID-U-5	Spectrum measurement and		
		NFT.U.250.5	test feasibility of cross-		
			calibration		
A.T.3	OD 0.8125"	Blank	Background Test on		
			BTBFC #1		
	OD 0.8125"	NFT.U.250.5	Direct cross-calibration		
		NFT.U.250.6	feasibility		
	OD 0.8125"	NFT.U.250.1	Setup variation test on		
		NFT.U.250.6	BTBFC		
	OD 0.8125"	NFT.U.250.1	HV power supply variation		
		NFT.U.250.6	test on BTBFC #1		

A.T.1. OD 1.4" BTBFC Settings.

The initial test performed on the OD 1.4" BTBFC was to validate the DAS settings given in Table 3-1 to validate past test results. Measurements performed without the addition of an attenuator is given in Figure A.T.1-1. To validate operational settings, the HV power supply was set to 200V, and the amplifier was set to the minimum gain of 10. The spectrum measured in Fig. A.T.1-1 shows a clip in the spectrum at Channel 976. This indicates that the signal's pulse-height is too large (greater than 10V) for the MCA to process. Since the amplifier was already set to the minimum gain, another method of signal attenuation is needed. Two proposed methods of attenuation are: modifying the pre-amplifier by adding a 1 μ F capacitor (Fig. A.T.1-2) or including an attenuator between the preamplifier and amplifier (Fig. A.T.1-3). Both methods were tested. Comparison of resulting measured spectra is given in Figure A.T.1-4. Both methods of attenuation performed well, however, the decision was made to using the attenuator over the modified pre-amplifier due to stability concerns. With the addition of the attenuator, the remainder of the DAS setting for the OD 1.4" BTBFC given in Table 3-2 was verified.



Figure A.T.1-1. The initial spectrum measured using OD 1.4" BTBFC and ID-U-5 standard.



Figure A.T.1-2. Modifying the Ortec 142PC pre-amplifier to include a 1 μ F capacitor (temporarily held in place with a plier).



Figure A.T.1-3. The built attenuator and circuit diagram.



Figure A.T.1-4. Comparison of spectra measured from attenuation tests.

A.T.2. OD 1.4" BTBFC Cross-Calibration

An MPFD sample, NFT.U.250.5, was inserted back-to-back with the standard, ID-U-5, in the OD 1.4" BTBFC. The test is to determine the feasibility of directly cross-calibrating between the MPFD samples with the standard in the OD 1.4" BTBFC. The resulting spectra is given in Figure A.T.2-1. The count-rate measured from the ID-U-5 is ~840 cps. The count-rate measured from the NFT.U.250.5 is ~0.03 cps. The count-rates are expected given the difference in reported mass. However, to obtain 5,000 counts from the NFT.U.250.5 sample in the only accessible part of the AGN-201 reactor the OD 1.4" BTBFC could reach, the irradiation time needed would be ~46 hours (the decision to perform only 5,000 counts instead of the standard 10,000 counts is to reduce count time.)

It was concluded that in order to obtain a good spectrum from the MPFD sample, significantly higher neutron flux is required.

The solution was to create a new BTBFC (OD 0.8125") that can access the core center of the AGN-201 test reactor. The OD 0.8125" BTBFC would be used to cross-calibrate MPFD samples.



Figure A.T.2-1. Measured spectra of ID-U-5 and NFT.U.250.5 in OD 1.4" BTBFC.

A.T.3. OD 0.8125" BTBFC Initial Evaluations

The OD 0.8125" BTBFC was designed and fabricated based on the conclusions made in experiment A.T.2. above. The design of the new BTBFC was to accommodate the use of MPFD samples as well as to operate with the same DAS setup as the OD 1.4" BTBFC. The initial evaluations of the OD 0.8125" BTBFC includes background measurements (Fig. A.T.3-1), direct cross-calibration measurements (Fig. A.T.3-2), setup variations (Fig. A.T-3-3), and operational voltage evaluations (Fig. A.T.3-4).

The background measurements showed only a 1-3% contribution to the total counts when MPFD samples were loaded. Direct cross-calibration feasibility was verified with the measurements results with MPFD samples, NFT.U.250.5 and NFT.U.250.6, inserted. Variation tests on setup indicates the need to maintain detector rotational position, therefore a 6' rod attachment, described in Section 3.2.1., was made to fix rotation and position of the detector between different experiments. Operational voltage evaluation was made by measuring the count-rate of NFT.U.250.1 as a function of voltage during reactor steady state power. The HV power supply was set to 300V based on the evaluation.

34



Figure A.T.3-1. Background spectra measurements.



Figure A.T.3-2. Direct cross-calibration spectrum measurement.



Figure A.T.3-3. Setup variation test.



Figure A.T.3-4. Operational Voltage evaluation measured with NFT.U.250.1 inserted.

APPENDIX B

All measurements made with the BTBFC are in the form of a pulse-height spectra. In order to obtain counts to the right of the geometric cut-off region representing fission events, a consistent method is desired. This section describes the method of determining the geometrical cut-off.

Original spectra typically measured is shown in Figure B-1. The spectrum is then passed to a smoothing function five times resulting in a spectrum shown in Figure B-2. The smoothing function is given by

$$C_{i+1}(x) = \frac{C_i(x-1) + C_i(x) + C_i(x+1)}{3}$$

where $C_i(x)$ are the counts (C) given in channel (x) on the i-th smoothing iteration.

After smoothing the spectra, the bin-interval between half-max values of the first spectrum peak is selected. A third-degree polynomial regression is performed (Fig. B-3). The corresponding root of the polynomial that intersects at the geometrical cut-off region is selected. All counts above the cut-off region is then integrated to provide the total count (Fig. B-4). Since the counts are based on fission events (poisson distribution), the square-root of the counts is calculated as the standard error.



Figure B-1. Original pulse-height spectrum measured by OD 0.8125" BTBFC.



Figure B-2. Resulting spectrum after 5 iterations of the smoothing function.



Figure B-3. Region selected from half-max of the first spectrum in the smoothed spectrum with a 3rd degree polynomial regression.



Figure B-4. Region selected from the zeros calculated from the polynomial given in Fig. B-3 that is used to calculate total counts, standard error, and count-rate.

APPENDIX C

The design of the OD 0.8125" BTBFC is based on the dimensions given in Figure C-1.



Figure C-1. Design dimensions (in inches) of OD 0.8125" BTBFC.