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BY OVERNIGHT MAIL

June 9, 2000

U. S. Nuclear Regulatory Commission
ATTN: Document Control Desk
Washington, DC 20555-0001

Subject: Holtite-A Neutron Shielding Material

References: 1. Holtec Project No. 1020
2. Holtec letter to NRC dated May 26, 2000

Dear Sir:

In accordance with our commitment made in the Reference 2 letter, enclosed please find one copy of the non-proprietary version of Holtec Report No. HI-2002396, *Holtite-A: Development History and Thermal Performance Data*, Revision 0.

Please contact me at (856) 797-0900, extension 668 if you have any questions or require additional information.

Sincerely,

Brian Gutherman, P.E.
Licensing Manager

Document I.D.: HL1020-009

Cc: Mr . Kirk Lathrop, USNRC (w/o encl.)

Enclosure: Holtec Report No. HI-2002396, Revision 1, non-proprietary version

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HOLTITE-A: DEVELOPMENT HISTORY AND THERMAL PERFORMANCE DATA

Holtec Report No. HI-2002396

Holtec Project No. 5014

Report Category: I

Report Class: Safety Related

NON-PROPRIETARY VERSION

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QA AND ADMINISTRATIVE INFORMATION LOG

(To Be Filled In By the Principal Author of the Document and Placed After the Title Page)

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Holtec Project No **5014**	

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REVIEW AND CERTIFICATION LOG FOR MULTIPLE AUTHOR DOCUMENTS†

REPORT NUMBER: HI-2002396 PROJECT NUMBER: 5014 REPORT TYPE: Technical Report

No.	Document Portion††	REVISION No. 0		REVISION No. ____		REVISION No. ____	
		Author & Date	Reviewer & Date	Author & Date	Reviewer & Date	Author & Date	Reviewer & Date
1.	Chapters 1-2,4-6	S.E. TURNER 4/17/00	D. M-MAJUMDAR 4/17/00				
2.	Chapter 3	Indresh Rampall (IR) 4/17/00	D. M-MAJUMDAR 4/17/00				
3.							
4.							
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THE REVISION CONTROL OF THIS DOCUMENT IS BY A "SUMMARY OF REVISIONS LOG" PLACED BEFORE THE TEXT OF THE REPORT. Form: CL3.2.5

TABLE OF CONTENTS

List of Tables

List of Figures

List of Figures (continued)

CHAPTER 1: INTRODUCTION AND BACKGROUND

Holtite-A™ is Holtec International's neutron shield material used in the company's HI-STAR 100 dual-purpose overpacks. Appendix 1.B in the HI-STAR 100 TSAR [1] provides detailed information on Holtite-A which formed the basis for the regulatory acceptance of the Holtite-A material for its use in the HI-STAR 100 cask. The object of this report is to document the information underlying the statements made in References [1] and [2], and to provide additional information of the thermal characterization studies performed on Holtite-A by Holtec as part of the company's test program which has been ongoing since early 1998. For convenience of reference, the entire body of information provided in the SAR documents on Holtite-A is reproduced herein as Appendix 1.

It is noted from Appendix 1 that Holtite-A emulates the neutron shielding material sold under the trade name NS-4-FR by the company Bisco Products, a division of Brand, Inc. Bisco, located in the vicinity of Chicago's O'Hare airport, which was actively involved in the development and marketing of neutron absorber materials in the 1980s. The principal author of this report (Dr. Stanley Turner) served as Bisco's consultant, mainly for testing and qualification of coupons. The principal developer of NS-4-FR was Mr. Larry Dietrick, who, at this writing, is a senior consultant of Scientech, Inc. Bisco's test data on NS-4-FR is contained in a letter by Mr. Dietrick dated April 20, 1987 [6] (please see page 1.B-20 in Appendix 1). Bisco's test data on NS-4-FR and corroborating work by others [5,7]* led to widespread acceptance of NS-4-FR in the U.S. nuclear industry. NAC's transport cask, NAC-STC, for example, utilizes NS-4-FR as the neutron shield material. Bisco utilized a commercially available resin (phenolic type, CAS No. 25068-38-6) and a commercially available fire retardant, aluminum trihydrate (ATH) and a common neutron absorber (Boron Carbide) to make the shielding material it named NS-4-FR. The product could not be patented because numerous patents on similar resin-based polymers had been granted in the past by the U.S. Patent Office, making the proof of novelty of NS-4-FR impossible to establish. Bisco did not even see it fit to trademark the NS-4-FR name. Bisco's only defense against potential competitors copying its technology was the mixing process, which required that the mixing chamber be evacuated of air. Bisco guarded the secrecy of its mixing operation with

* References [5] and [6] are also part of the HI-STAR 100 SAR documentation (Appendix 1).

great care, permitting only a select few from outside the company to tour its factory. Holtec's personnel Dr. K.P. Singh and Dr. Stanley Turner were among the few granted the privilege of touring the factory floor and observe the operations, chiefly because Holtec was both a consultant to Bisco and a substantial user of its product used in the wet storage business (Boraflex).

Bisco, unfortunately, fell on hard times and ceased operations in the beginning of the 90's. Before completely shuttering its doors, Bisco, however, managed to locate a small Japanese company (Genden) to purchase its mixing procedures and associated know-how. The Japanese firm, in turn, gave rights to NAC International to market NS-4-FR in the United States. NAC's Japanese principal dissolved quickly as a functioning entity in late 1998, when a company employee identified and alleged deliberate acts of deception by the company's personnel in the manufacturing of NS-4-FR. The parent of the defunct company, Japan Atomic Power Company (JAPCO), came into possession of the technology purchased by Bisco, after Genden's collapse. In 1992, Holtec International faced the need to select a neutron shield material for the company's newly launched HI-STAR 100 program. The chief concern with neutron shield material, as is explained in the next chapter, is its physical integrity at elevated temperatures. Commercially available neutron shields such as NS-4-FR have limited thermal resistance at temperatures above 325°F. The rate of material loss increases geometrically with rising temperatures in the manner of the classical Arrhenius equation. Anticipating that the heat loads for transport casks will increase with time, leading to a concomitant increase in the bulk temperature of the neutron shield material, Holtec set out (in 1992) to develop a neutron shield material which would remain stable at 370°F. The company's research and development effort was led by Dr. Stanley Turner. Dr. Turner experimented with a variety of promising polymeric forms, none of which held up at 370°F. Dr. Turner had secured the consulting services of Bisco's ex-president, Mr. James Anderson, to help Holtec avoid repeating the errors of Bisco's R&D program in the 80's. Many years of experimentation at Holtec's Florida laboratories (where all neutron absorber testings have been centralized by the company since 1987) showed that improving the temperature resistance to 370°F was an elusive undertaking. By late 1997, the company had decided to follow the incremental approach whereby a neutron shield suitable for the near-term heat load is employed in the first generation of HI-STAR 100s. The company however does and will continue to test other promising materials that may prove increased thermal endurance. This quest to develop a high temperature neutron shield is scheduled to continue. However, for the first generation of HI-STARs (HI-STAR 100s), Holtec decided to follow the path of guaranteed success, namely, either use the NS-4-FR material manufactured by a qualified supplier, or manufacture NS-4-FR itself.

The first path of action, namely, to utilize a qualified supplier to provide NS-4-FR was a preferred course of action because it involved no direct investment in product development by the company. The second path of action, namely, to produce NS-4-FR ourselves, was less attractive because it meant an uncertain amount of R&D expense, but it posed no insurmountable technical problems. The company's personnel were most familiar with NS-4-FR since its inception at Bisco, and the developer of NS-4-FR, Mr. Larry Dietrick, was available as a consultant. In summary, Holtec had determined after careful consideration that making NS-4-FR by the company was quite feasible. Holtec decided that self-manufacturing should be a fallback option, not the first choice.

Accordingly, the company contacted NAC International, the U.S. supplier of NS-4-FR appointed by Genden of Japan, to possibly supply the NS-4-FR material for the company's HI-STAR 100 overpacks due to enter fabrication in 1999. The company's QA program required that all safety significant[*] products be subject to independent testing and suppliers of safety significant material be evaluated and qualified to be put on the company's supplier's list before a purchase order for safety significant goods or services can be placed.

To initiate the process to secure NAC as a supplier of NS-4-FR (a safety significant material), Holtec placed a Safety Related Purchase Order in January, 1998 with NAC to provide a limited quantity of NS-4-FR under NAC's Part 72 Subpart G and Part 71 Subpart H QA programs. NAC accepted the purchase order (provided in Appendix 2 herein) and delivered 4" dia. 24" long bars of NS-4-FR to Holtec for testing and evaluation. NAC also provided a Certified Material Test Report (as required by the Holtec purchase order) attesting to the properties of the supplied material (Appendix 3).

The long bars of NS-4-FR supplied by NAC were "sliced" into 0.5" and 1.3" thick disks for thermal testing in Holtec's Florida laboratory. The sectioned pieces revealed a disconcerting condition: the disks contained "voids". Dr. Turner, alarmed by the physical condition of the test specimens supplied by NAC under a safety-related contract, recommended in an internal company memo (please see Appendix 4) that NAC be disqualified as a supplier of NS-4-FR. Holtec's Mark Soler relayed the company's findings on the test specimens to NAC in a letter dated April 20, 1998. Holtec's letter, and NAC's responses on this matter, are reproduced in Appendix 4. NAC, in its response to Mark Soler's concerns, asserted that the voids are inconsequential to the behavior of the material and that NAC possesses the necessary know-how to make a void-free installation of NS-4-FR in a cask system such as Holtec's HI-STAR 100. Holtec's experts agreed with NAC that the presence of voids should not alter the resistance of the test specimens. NAC saw no need to provide improved (void-free) samples to Holtec. Before permitting NAC to install it's neutron shield into HI-STAR 100, however, Holtec would have needed to see definitive evidence that NAC would be able to make void-free installation in our casks. Many within Holtec were skeptical of NAC's ability to install void-free NS-4-FR columns, given the supplier's seeming inability to provide even void-free samples for testing.

To ensure success, Holtec International also launched the parallel path of reproducing NS-4-FR itself under the name Holtite-A, the annex "A" added to separate this material from future enhanced variations of Holtite which our long-term R&D program (mentioned earlier) might yield. Holtite-A, in other words, was to be a replicate of NS-4-FR. The company's TSAR appropriately identified Holtite-A as a material that emulates NS-4-FR (please see Appendix 1). The TSAR document also left open the possibility that Holtec could use NAC to install NS-4-FR in HI-STAR 100 casks, should NAC's supplied test specimens pass qualification tests and NAC's QA program pass Holtec's QA audit.

Fifteen NS-4-FR specimens, prepared from the NS-4-FR bars supplied by NAC, were subjected to thermal aging tests for ninety-seven days at 325°F, as described in the TSAR (Appendix 1). The average weight loss of 2.41% was judged to be consistent with Bisco's data (Appendix 1,

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* "Safety Significant" is defined in Holtec's QA Manual.

page 1.B-20). The HI-STAR TSAR concludes that, "on the strength of other published work [5, 6, 7] and the company's own thermal testing, Holtite-A meets all of the requirements for an acceptable neutron shield material in the manner of NS-4-FR which was licensed previously in Docket 71-9235 (NAC-STC)".

The testing of the NAC-supplied NS-4-FR samples, however, continued beyond ninety-seven days. As we describe in Chapter 5, the specimens began to exhibit anomalous behavior upon prolonged exposure. This data, further discussed in Chapter 5, essentially disqualified NAC as a prospective supplier of NS-4-FR and left Holtec with the fallback option (namely, to produce Holtite-A ourselves) as the sole remaining path of action.

Fortunately, our program to produce Holtite-A has turned out to be an outstanding success. Material specimens made in the laboratory, as well as those taken from the production pours, have exhibited excellent thermal stability under prolonged exposure at 325°F. Chapter 5 contains the necessary details. Before providing the test data and results in Chapters 4 and 5, we describe the correspondence between Holtite-A and Bisco's NS-4-FR in a rigorous manner in Chapter 2. In Chapter 3, the thermal environment for the neutron shield material in the HI-STAR 100 cask is defined. Results of thermal-aging tests on candidate NS-4-FR materials for the neutron shield material in the overpack for Holtec's HI-STAR 100 dual-purpose casks are contained in Chapters 3, 4, and 5.

Using the thermal environment information in Chapter 3, the expected thermal performance (stability) of Holtite-A is quantified in Chapter 6. Results of the testing, and the evaluation of the thermal environment expected confirm that Holtite-A is thermally stable and acceptable for use in the Holtec HI-STAR 100 cask.

CHAPTER 2: HOLTITE-A AND ITS RELATIONSHIP TO NS-4-FR

As discussed in Chapter 1, in the mid-to-late 80's, a shielding material was developed and tested by Bisco Products, Inc. of Park Ridge, Illinois. This material, named NS-4-FR by its developer, was composed of 62% by weight of $Al_2O_3 . 3H_2O$ (called commercially known as aluminum tri-hydrate or ATH) in a 2-part epoxy resin, to which B_4C (boron carbide) is added to enhance its neutron absorption property. The original formulation of NS-4-FR used a resin (Part A) with a CAS number of 25068-38-6 and an amine hardener mixture with CAS number of 112-57-2 and 68953-36-6. The mix ratio of Part A and Part B is variable but the vendor-recommended mixture is 55 parts of the resin to 45 parts hardener (or catalyst).

NAC International markets a product similar to NS-4-FR, whose behavior will be described and compared to the original NS4-FR developed by Bisco in Chapter 4. To distinguish between the two different versions of NS-4-FR discussed in this report, the original Bisco version will be designated as Bisco-NS4FR and the NAC version as NAC-NS4FR. The shielding material used in HI-STAR casks is same as the original Bisco version*, Bisco-NS4FR, containing 1% B_4C and is called Holtite-A. The mixture composition of the product and its elemental composition are summarized in Tables 2.1 and 2.2. NAC has not disclosed the mixture composition of NAC-NS4FR material to Holtec. The ingredients are NAC's proprietary information (See Appendix 5, NAC Technical brochure, Section II). A close similarity in the chemical composition of Holtite-A (Bisco-NS4FR) and NAC-NS4FR indicates that the NAC-NS4FR uses a similar mix of raw materials although the resin formulation may be different.

The epoxy resin materials used in Holtite-A play an important role in the manufacture and performance of the finished product. The relevant physical properties of the resin are provided in Table 2.3. The ATH is a mined product that is subsequently cleaned and supplied in various grades. The ATH grades used in the manufacturer of Holtite-A is SB-30 or KC-30. These materials have an aluminum content of 33.5% or more.

* This is independently corroborated in a letter from Larry Dietrick (developer of Bisco-NS4FR) to Holtec (Appendix 7).

TABLE 2.1: Bisco-NS4FR and Holtite-A Composition

Constituent	Weight %	CAS #
ATH	62	[redacted]
B_4C	1	
Resin	37	

TABLE 2.2: Elemental Chemical Composition
(Comparison of Holtite-A (Bisco-NS4FR) and NAC-NS4FR, Wt%)

Element	Bisco-NS4FR	NAC-NS4FR Product Datasheet (Appendix 5)
B-10	0.143	Not Available
B-11	0.639	Not Available
Aluminum	21.46	21.5
Hydrogen	5.94	6.0
Carbon	27.19	27.7
Nitrogen	1.95	2.0
Density, g/cc	1.693	1.68
H_2 Density, g/cc	0.106	0.101

TABLE 2.3: Properties of Resin Formulation Materials

Parameter	Method of Measurement	Part A (Resin)	Part B (Hardener)
Specific Gravity	ASTM-D297-93 ASTM-D792-91	[redacted]	[redacted]
Brookfield Viscosity	ASTM-D2393	[redacted]	[redacted]
EEW (Epoxy Equivalent Weight)	ASTM-D1652 (Method B)	[redacted]	[redacted]

CHAPTER 3: HOLTITE A THERMAL ENVIRONMENT IN HI-STAR 100

3.1 Introduction

The HI-STAR 100 overpack is a dual-purpose device for storage and transport of Multi Purpose Canisters (MPC) containing Spent Nuclear Fuel (SNF) [1, 2]. Cross sectional views of the HI-STAR System are depicted in Figures 3.1 and 3.2. The overpack is a multi layered metal structure with a bolted cavity sized for emplacement of an MPC. On the outside of the metal structure, forty annular spaces formed with half-inch thick metal plates are filled with Holtite-A neutron shield material. When used with an MPC, the overpack structure is designed to safely transfer SNF heat and dissipate to the environment from the exposed surfaces.

For the purpose of engineering a suitable thermal environment for Holtite-A, pertinent characteristics of neutron shield materials and nature of the application are considered. Neutron shielding materials (primarily a blend of Aluminum Trihydrate in a polymer matrix) are relatively poor heat conductors (compared to steel) and have a much lower temperature limit than metals for long-term operation. It is heuristically apparent that the outward flow of heat in the ovepack structure elevates the temperature of the interior regions relative to the outer layers. To ensure that the neutron shield material is protected from high temperatures reached in the interior, it is arrayed on the outside as shown in Figure 3.2. In the HI-STAR 100 overpack design, wherein neutron shield materials are interposed in the primary (radial) path for heat flow for proper function (i.e. capture of neutrons emitted by SNF), it becomes imperative that they not unduly interfere with the heat dissipation function. Accordingly the Holtite-A region is embedded with radial heat conducting carbon steel channels to substantially alleviate heat dissipation limitations from neutron shielding materials.

In dry storage the neutron shield material is non-uniformly heated by the stored SNF in the MPC. This is due to axial variations in decay heat emission and cooling from the ends of the cask by axial heat conduction. The local rate of heat emission by SNF reaches a peak value at about active fuel mid-height and attenuates towards the ends. The SNF decay power is not constant as the heat rate exponentially decays with time. Thus, as the cask heat load drops, the Holtite-A temperatures will also attenuate with elapsed time in dry storage. In this chapter, the HI-STAR 100 thermal solutions presented in the Holtec safety analysis reports [1,2] are examined to fully characterize the spatial and temporal temperature variations in the neutron shield region.

3.2 Temperature Field of Holtite-A

The HI-STAR 100 safety analysis reports [1,2] provide maximum neutron shield temperatures at design basis maximum heat load (5-year cooled fuel) at the start of dry storage. The thermal solution for the HI-STAR 100 System was based on an array of conservative assumptions listed below:

1. Maximum annual average temperature of 80°F.
2. Insolation with a bounding solar absorbtivity of unity.
3. Most adversely located HI-STAR cask in an ISFSI array (i.e. maximum radiation cooling blockage from neighboring casks).
4. No credit considered for conduction through neutron shield.
5. No credits for effect of wind (i.e. stagnant air assumption).
6. Classical correlation for natural convection employed in analysis understates heat dissipation.

To quantify the thermal field attenuation with elapsed time (τ_s) in dry storage with 5-year cooled SNF prior to dry storage, the Reg. Guide 3.54 [3] is employed. Using the information provided in this Reg. Guide, the cask heat load function $Q(\tau_s)$ is obtained. The function, starting with design basis maximum heat load for storage ($Q(0) = 19$ kW), is shown depicted in Figure 3.3. To bound the heat load reduction over a 20-year licensed storage period, a HI-STAR thermal solution is obtained at 40% of Q(0). An additional thermal model run at 70% of Q(0) gives a three-point solution for interpolation of HI-STAR temperatures as a function of heat load $Q(\tau_s)$. Using this interpolation data, the peak and radial average neutron shield temperature attenuation curves are constructed as depicted in Figure 3.4. In Table 3.1, bounds on Holtite-A spatial temperature variations at design maximum (Q(0)) and at 70% and 40% of Q(0) heat loads established from the thermal solutions are summarized. The computed results are conservative upperbound values as they include the array of conservatisms stated above. A discussion of the results obtained from the analysis are presented next.

The peak temperature, at the start of dry storage (i.e. the maximum in space and time) is computed as 270°F. The maximum radial average temperature is 250°F. Axial temperature profiles for the neutron shield inner surface and radial average* (parameterized by τ_s) are presented in Figures 3.4 and 3.5. From these plots it is apparent that the temperature at the ends of the neutron shield region are at about or below 200°F throughout the duration of dry storage. From the plot of temperature vs. time (Figure 3.4), Holtite-A peak temperature drops by about 60°F over a 2-year dry storage period to 210°F. After about 3 years in dry storage, the neutron shield temperature is below 200°F. At the end of the dry storage period of 20 years, the temperature falls off to about 120°F. In summary:

- The bulk average temperature of Holtite-A in HI-STAR 100 at design basis maximum heat load (19 kW) corresponding to 5-years cooled fuel prior to dry storage is well below 250°F. In other words, at the beginning of dry storage when the SNF is at the hottest, the bulk average temperature of Holtite-A is significantly less than 250°F.

- The bulk average temperature of Holtite-A as well as its peak temperature will drop with time. After 2 years of storage, the bulk temperature is well below 200°F.

- After about 7 years of storage, the bulk temperature drops to less than 150°F.

* Defined as the line integral of radial temperature distribution from inner surface of Holtite-A region to outer surface.

TABLE 3.1: HOLTITE A STORAGE TEMPERATURES SUMMARY

Location	@19 kW (°F)	@13.3 kW (°F)	@7.6 kW (°F)
Top Axial Location Inner Surface Outer Surface	 143 141	 131 130	 119 118
Top Axial Location Inner Surface Outer Surface	 270 231	 221 193	 169 154
Top Axial Location Inner Surface Outer Surface	 194 173	 166 151	 136 127

3.3 Computer Files

Thermal models developed for licensing of HI-STAR System have been employed in this study. The FLUENT version 4.43 CFD code was used to compute the thermal solutions. The list of files are provided below:

```
 Volume in drive F is VOL1
 Directory of F:\PROJECTS\5014\IR\holtite
MPC24_1  CAS        206,514  03-27-00  2:50p mpc24_1.cas
MPC24_1  DAT        482,840  03-27-00  2:50p mpc24_1.dat
MPC24_2  CAS        206,514  03-27-00  2:39p mpc24_2.cas
MPC24_2  DAT        482,840  03-27-00  2:39p mpc24_2.dat
MPC24_3  CAS        206,514  03-27-00  2:47p mpc24_3.cas
MPC24_3  DAT        482,840  03-27-00  2:47p mpc24_3.dat

P19KW    LOG            580  11-03-97  4:04p P19KW.LOG
P13P3KW  LOG            579  03-27-00  2:32p p13p3kw.log
P7P6KW   LOG            576  03-27-00  2:34p p7p6kw.log


 Volume in drive F is VOL1
 Directory of F:\PROJECTS\5014\IR\holtite\plt
P19KW_I  PLT          1,217  03-27-00  2:57p p19kw_i.plt
P19KW_O  PLT          1,217  03-27-00  2:59p p19kw_o.plt
P13P3K~1 PLT          1,217  03-27-00  3:05p p13p3kw_i.plt
P13P3K~2 PLT          1,217  03-27-00  3:06p p13p3kw_o.plt
P7P6KW_I PLT          1,217  03-27-00  3:13p p7p6kw_i.plt
P7P6KW_O PLT          1,217  03-27-00  3:16p p7p6kw_o.plt
TCURVES  XLS         91,648  03-28-00  3:27p Tcurves.xls
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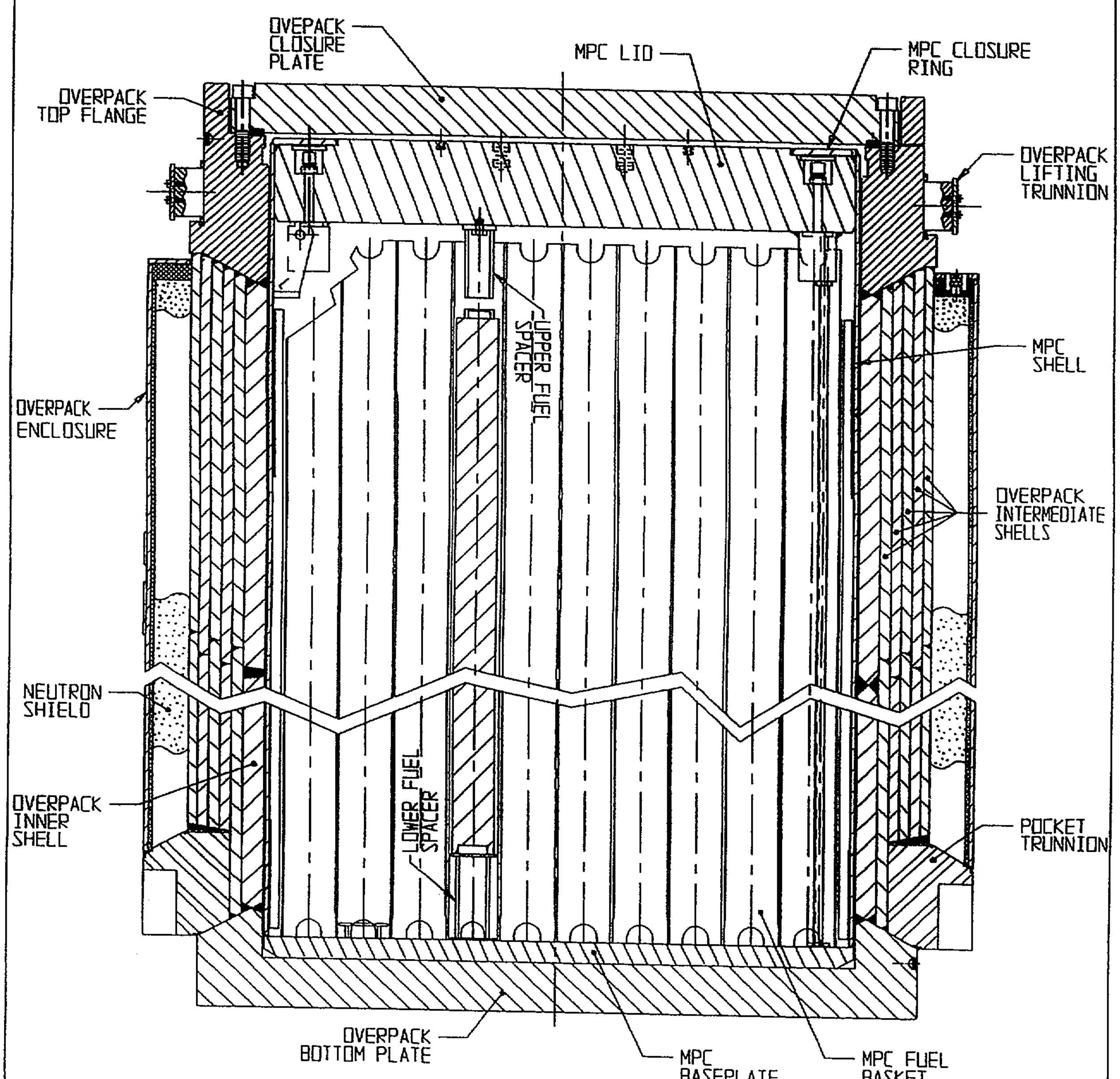

FIGURE 3.1: CROSS SECTION ELEVATION VIEW OF HI-STAR 100 SYSTEM

REVISION 7

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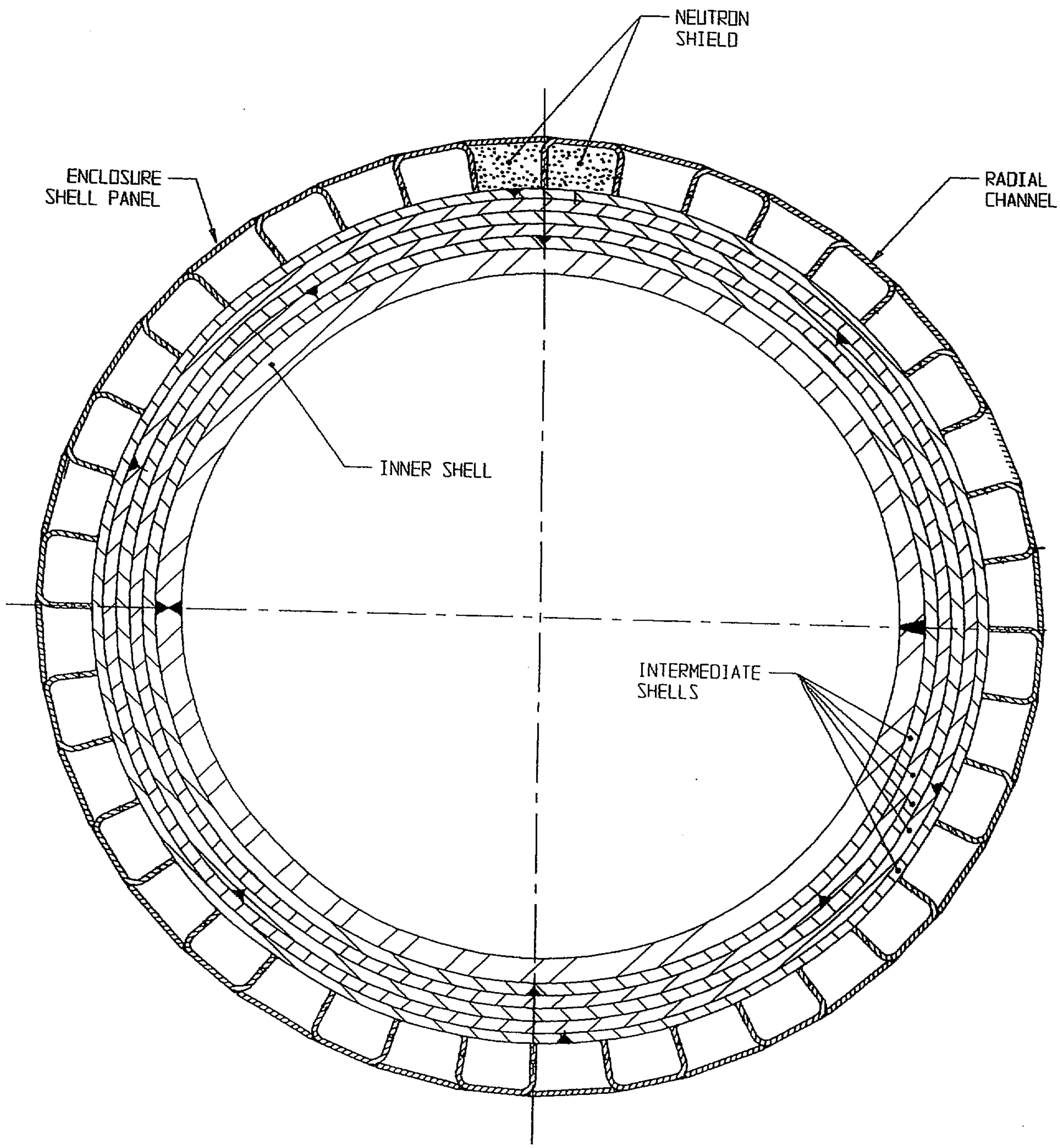


FIGURE 3.2: OVERPACK MID-PLANE CROSS SECTION

Figure 3.3: Cask Decay Heat (Reg. Guide 3.54 with initially 5-year cooled SNF) as a Function of Elapsed Time in Dry Storage

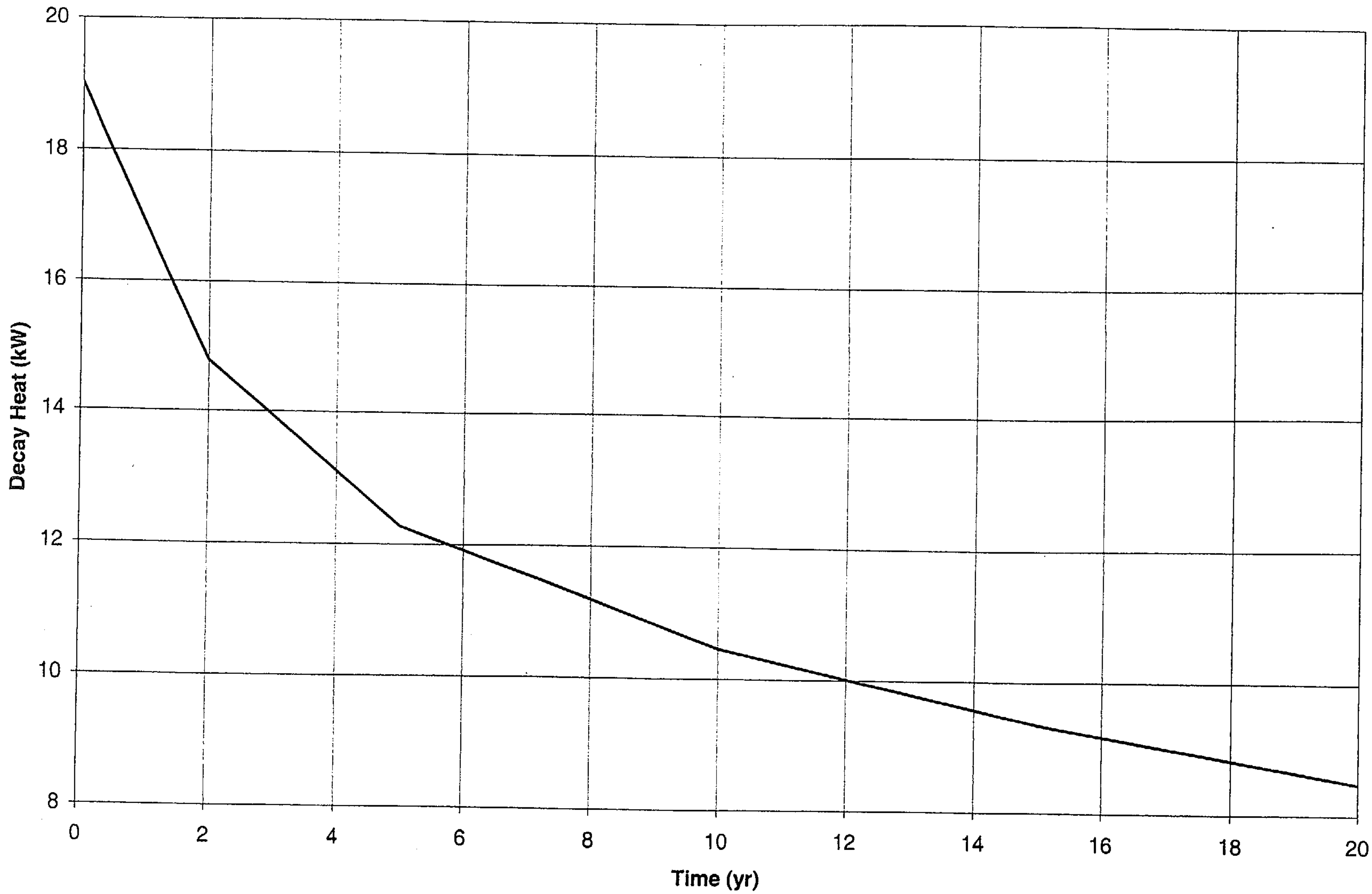
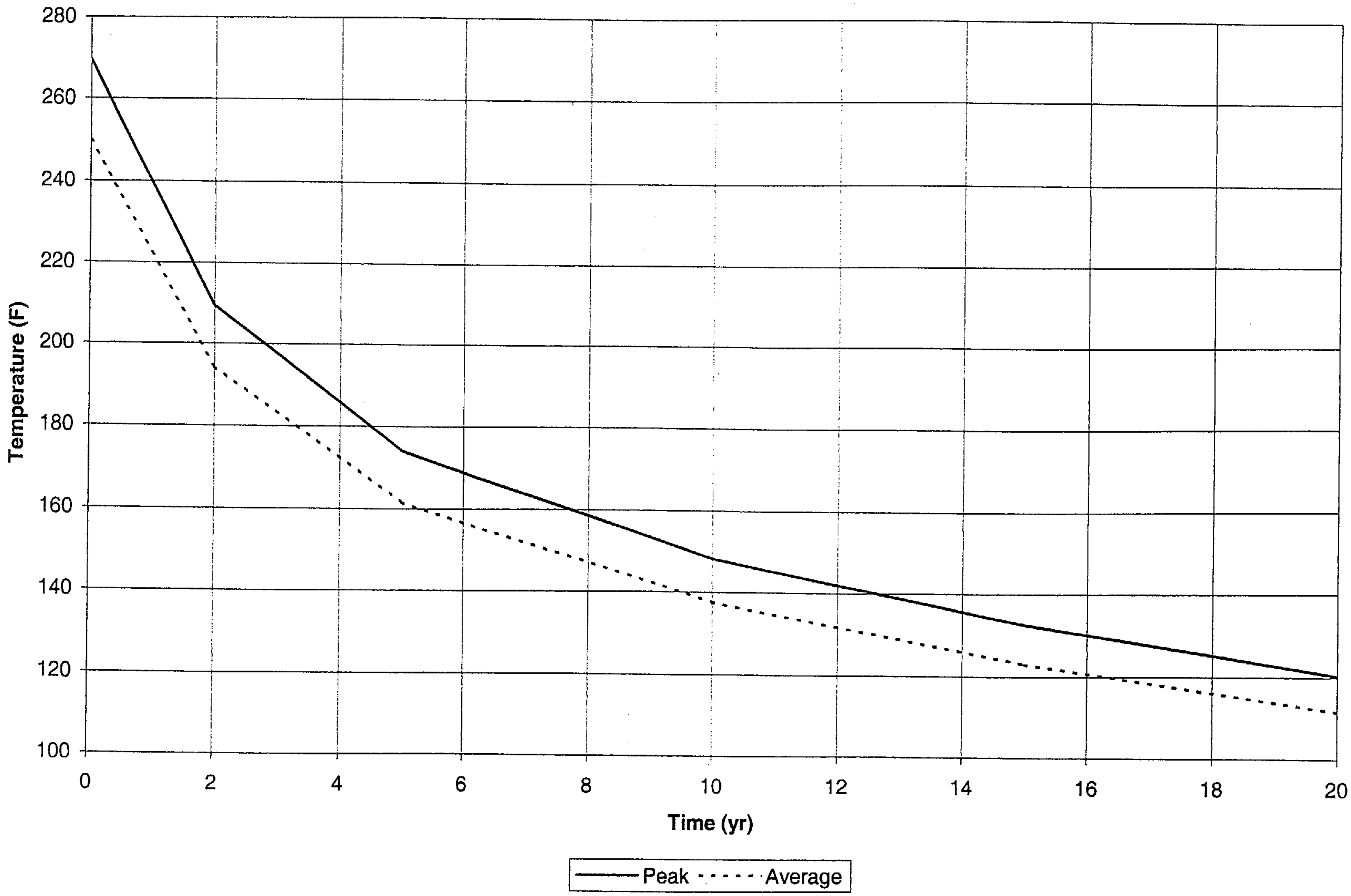


Figure 3.4: Peak and Highest Radial Average Temperatures as Functions of Elapsed Time in Dry Storage



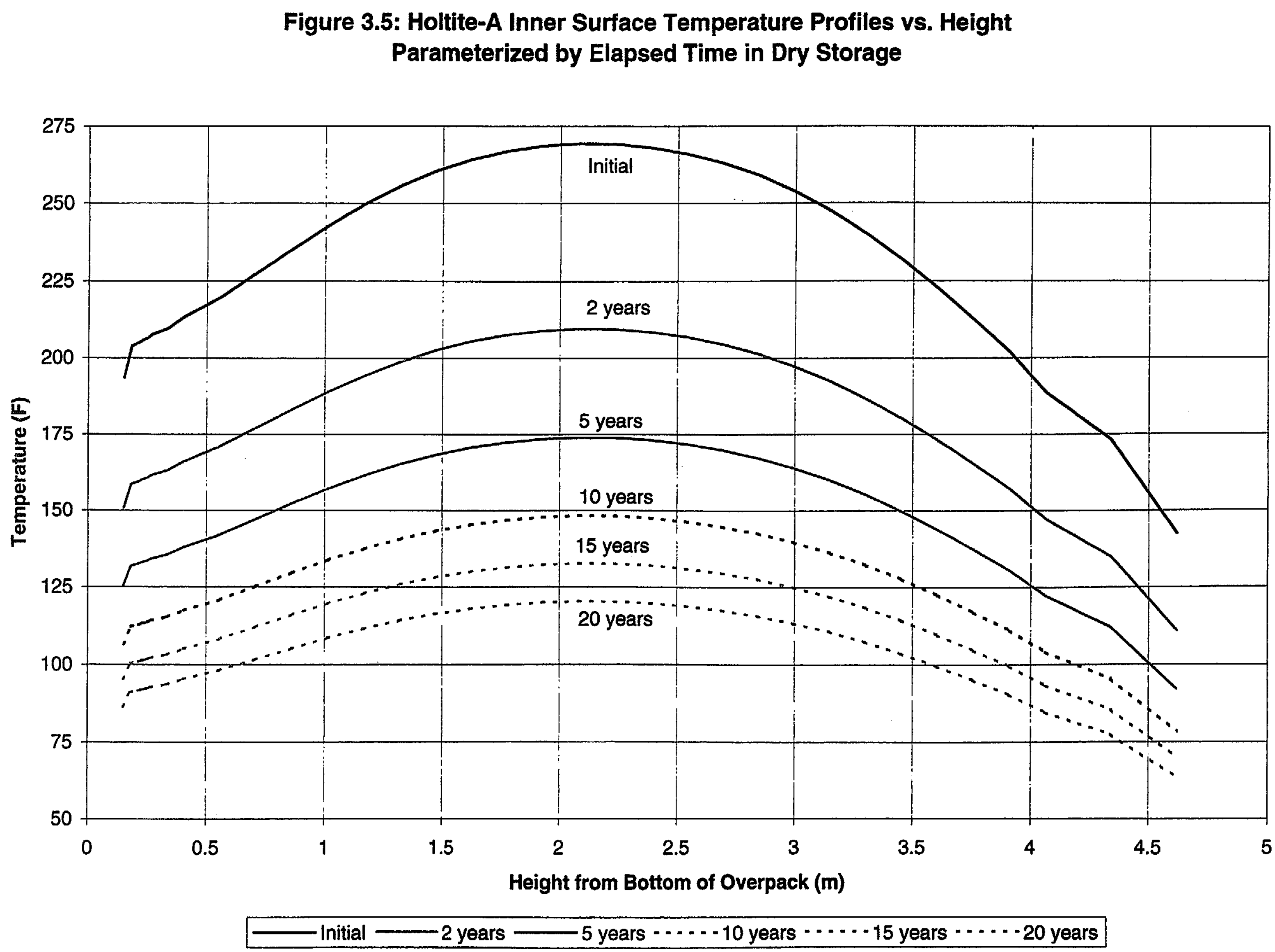
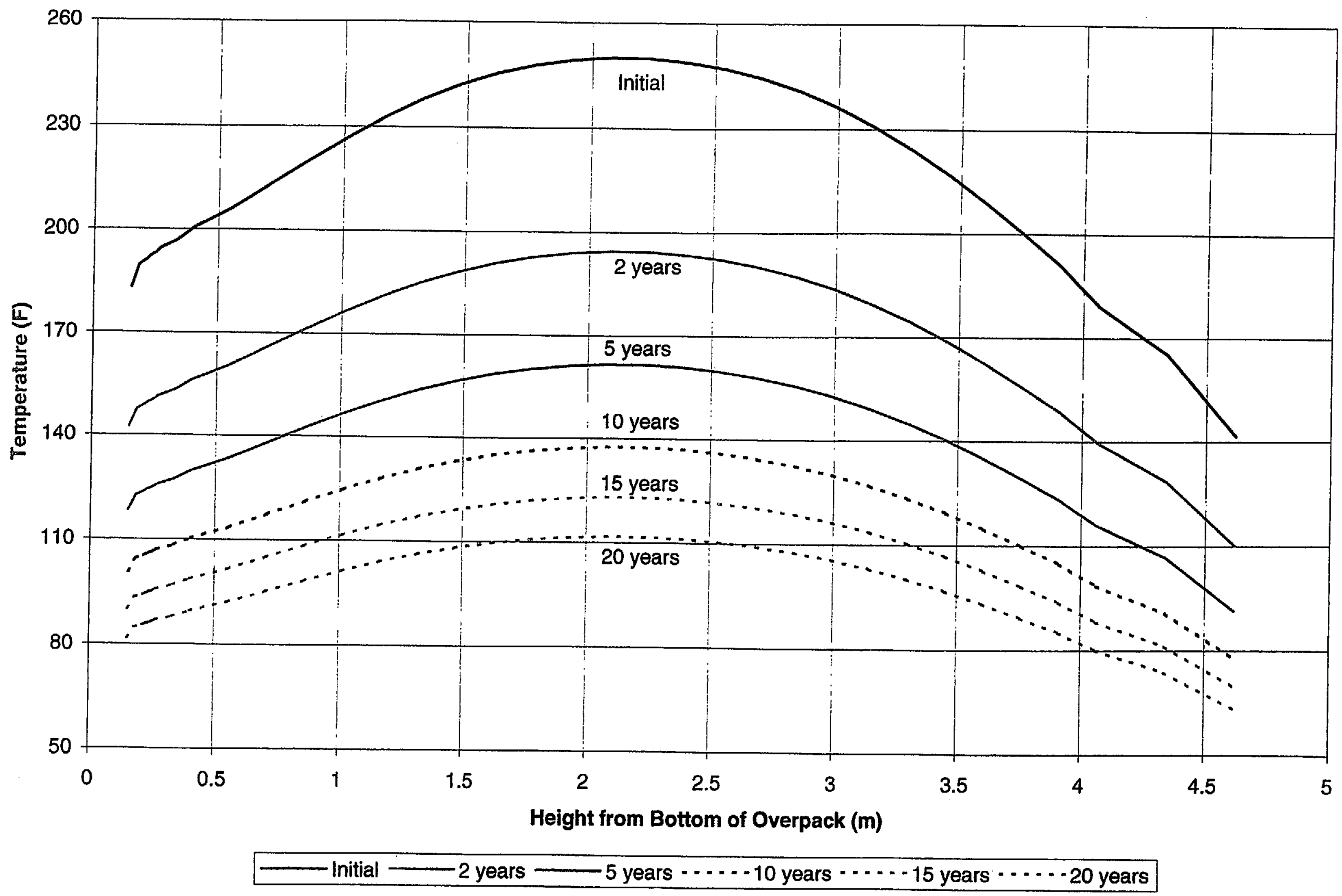


Figure 3.5: Holtite-A Inner Surface Temperature Profiles vs. Height
Parameterized by Elapsed Time in Dry Storage
Initial
2 years
5 years
10 years
15 years
20 years
275
250
225
200
175
150
125
100
75
50
Temperature (F)
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
Height from Bottom of Overpack (m)
Initial
2 years
5 years
10 years
15 years
20 years

Figure 3.6: Holtite-A Radial Average Temperature Profiles vs. Height Parameterized by Elapsed Time in Dry Storage



CHAPTER 4: PRIOR WORK ON HOLTITE-A THERMAL STABILITY TESTING

Having established a sound shielding material for use in HI-STAR casks (Bisco-NS4FR), material data sheet specifications were prepared and incorporated in the HI-STAR licensing documents under review by the NRC (Appendix 1). To independently confirm the thermal stability characteristics of NS4FR under Holtec's QA program, the advertised supplier of this product (NAC International) was contacted to supply samples with certification to 10CFR71 Subpart H and 10CFR72 Subpart G (Holtec PO, Appendix 2). Samples of the NAC-NS4FR were purchased and fifteen test coupons sliced with a saw cutter, surfaces ground smooth and prepared for testing. Holtec's own program for in-house manufacture of Holtite-A samples of Bisco-NS4FR material formulation from the mixture data (Tables 2.1 and 2.3) was underway. The NS4-FR testing from the NAC supplied material historically appeared first (early 98). The Holtite-A samples production and testing as described in Chapter 1 was to appear later.

It is noted from the results presented in Chapter 3 that the thermal environment in which Holtite-A is to work as a neutron shield material is generally in the range of 150°F to 250°F in the HI-STAR cask in a sealed configuration (i.e. protected from the outside elements). For the purpose of qualifying the material supplier in a timely manner, the conditions employed in the test were severe relative to the service requirements. The test conditions employed were 325°F temperature and specimens were exposed to air. After ninety-seven days of testing the samples exhibited stable characteristics and the test results were determined consistent with published data on Bisco-NS4FR. The test results were reported in the HI-STAR 100 TSAR (Rev. 10). The statements, while not made explicitly in the TSAR, bound Holtec to a regulatory commitment to perform confirmatory testing of poured samples of shielding material before the first manufactured hardware (cask) is placed in service.

Before a discussion of the results of Holtec thermal tests on the NS4-FR material is undertaken, we digress to touch upon the testing done by others and thus place the work done by Holtec in proper perspective. The original shield material, Bisco-NS4FR, has been tested by Bisco Products, Inc in the 80's and later by others [5,7]. Unpublished information garnered from discussions with original developers show that the material holds up to continuous exposure under extreme conditions (338°F, 14 months, ~4% weight loss). Published data from Larry Dietrick (of Bisco Products) confirms a weight loss of between 3.09% to 3.15% at 338°F continuous exposure for 146 days. The Japanese [5] have conducted long duration (~2500 hr) tests in the 150°C(302°F) to 200°C(392°F) range on the Bisco supplied NS-4-FR material. At the lower end of the temperature range, less than 2% weight loss is reported. At the higher end of the test temperature, weight loss approaching 7% is discernible from the reported plots. An extended duration aging test on this material (lasting for about an year) was conducted at temperatures approaching those in casks (150°C [302°F]). From this work it was concluded that the NS-4-FR material is satisfactory for shielding in Holtec's casks. Both the Bisco series of prior testing and independent investigations by the Japanese confirm the thermal stability of the Bisco-NS4FR. The information available in the public domain [5,6,7] confirms the suitability of Bisco-NS4FR (Holtite-A) for use in spent nuclear fuel storage casks.

As weight loss characteristics for the NAC-NS4FR material are not certified by NAC International (January 28 letter from NAC, Appendix 2), sample testing had begun early at Holtec's test laboratory in Florida in March of 1998. NAC's confirmation that the material was supplied suitable for thermal degradation testing (June 2, 1998 letter, Appendix 4) cleared a cloud of uncertainty surrounding an unrelated matter (the discovery of voids in the material). The fifteen samples made from NAC supplied NS4FR billots were continuously exposed to test temperature (325°F) in heated ovens. The samples were periodically withdrawn weighed, weight loss computed and then introduced back in the ovens.

The cumulative weight loss results from this long duration (97-day) test are shown plotted in Figures 4.1 through 4.15. The weight loss results from the tests on fifteen samples (~3%) confirmed the NS4-FR material behavior was consistent with published data in continuous exposure at high temperatures. Later (in 1999), two sample batches of Holtite-A manufactured by Holtec were tested. These were the four lab samples (Batch 1) and three shop floor samples (Batch 2) of the Holtite-A material poured in the first production HI-STAR cask meant for delivery to plant Hatch. These were tested under same conditions (325°F) for a period of 90 days (Batch 1) and 109 days (Batch 2). Results of this testing (cumulative weight loss) are presented in Figures 4.16 through 4.19 (Batch 1) and 4.20 through 4.22 (Batch 2). The Holtite-A results are consistent with the published data for Bisco-NS4-FR, (viz. a weight loss of about 3%). This accelerated testing of laboratory and shop produced Holtite-A confirmed that it's thermal stability characteristics are correctly represented in the HI-STAR 100 TSAR [1].

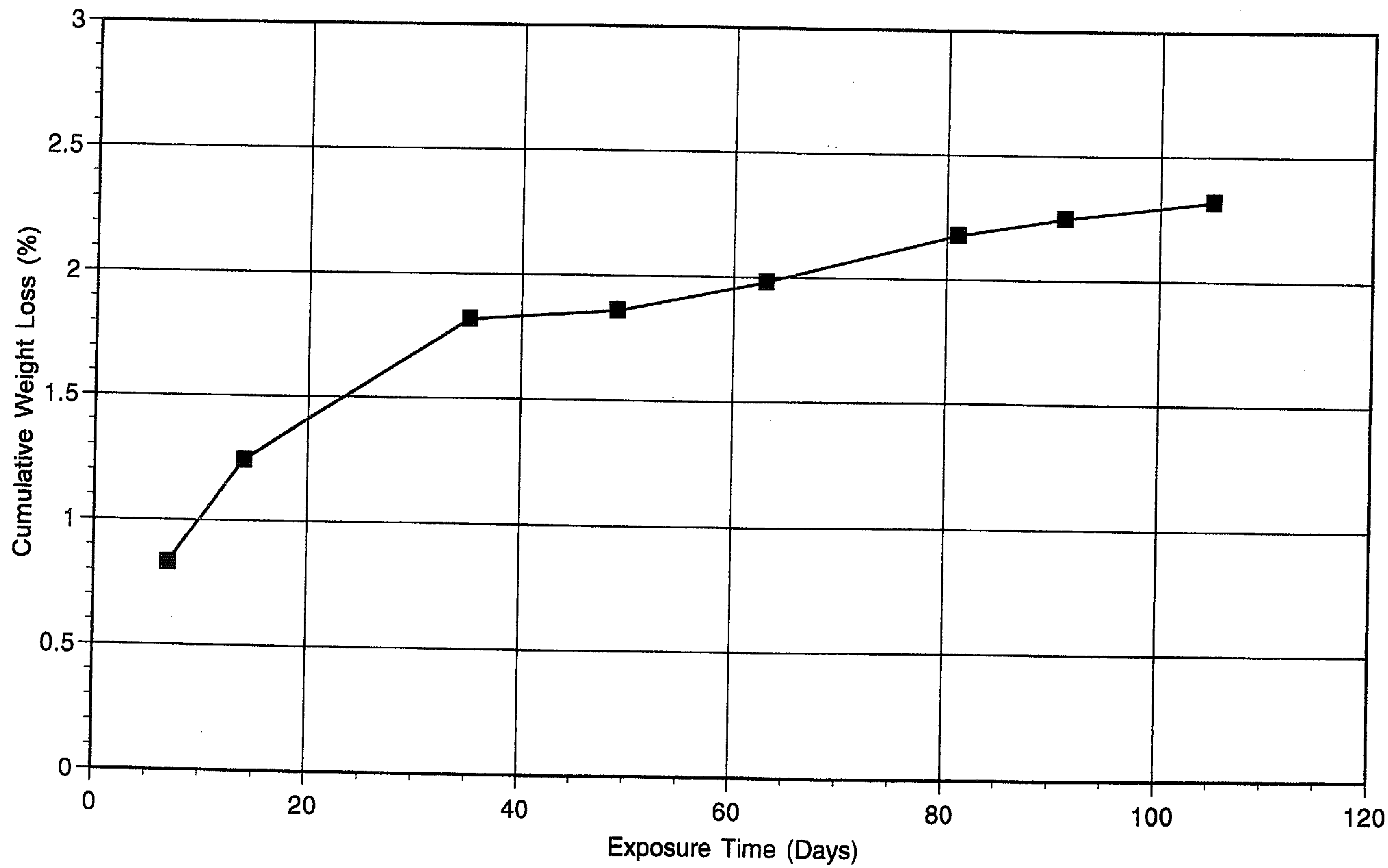


FIGURE 4.1: NAC-NS4FR Thermal Degradation Test Result (Sample #1-1)

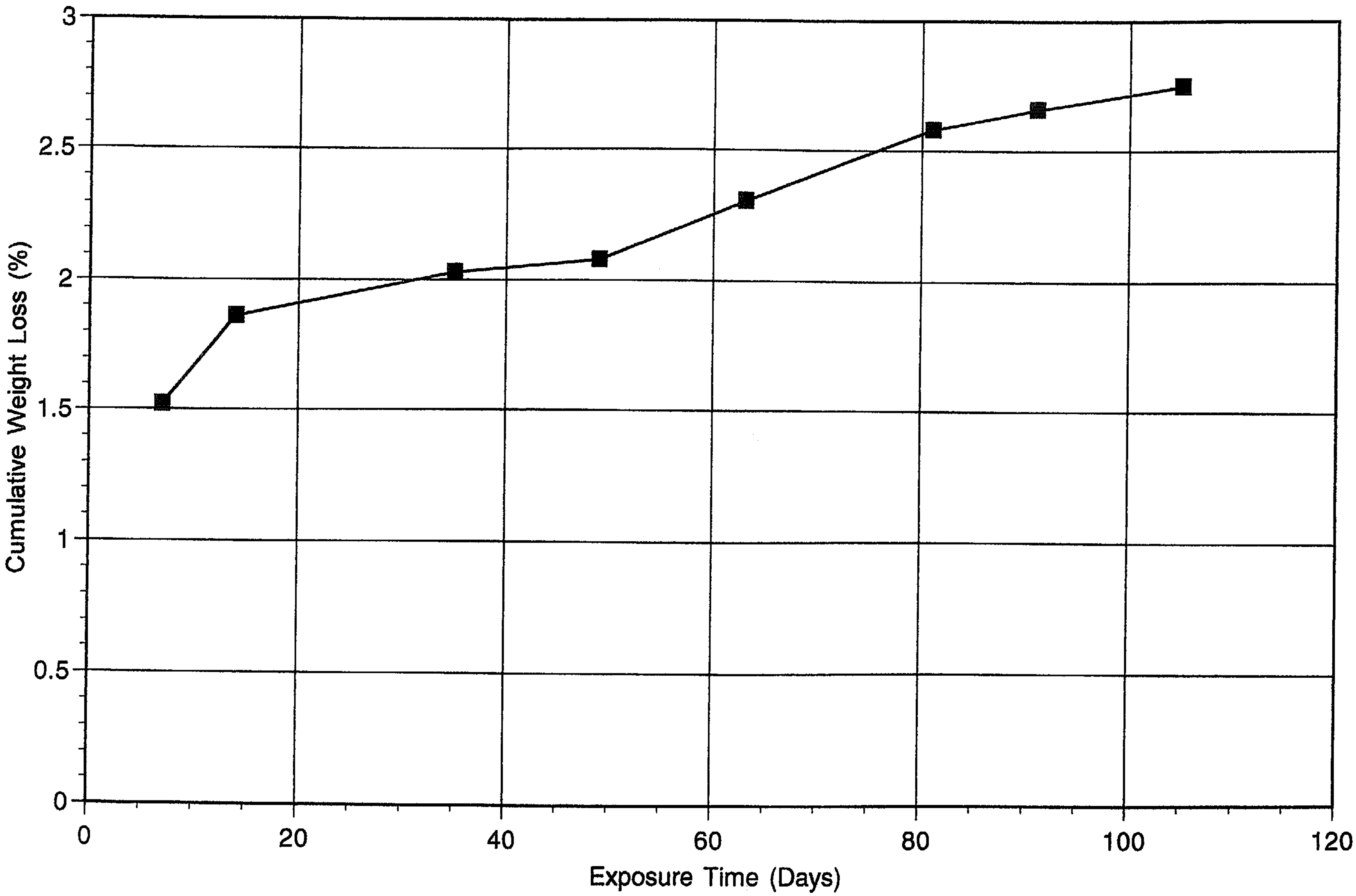


FIGURE 4.2: NAC-NS4FR Thermal Degradation Test Result (Sample #1-2)

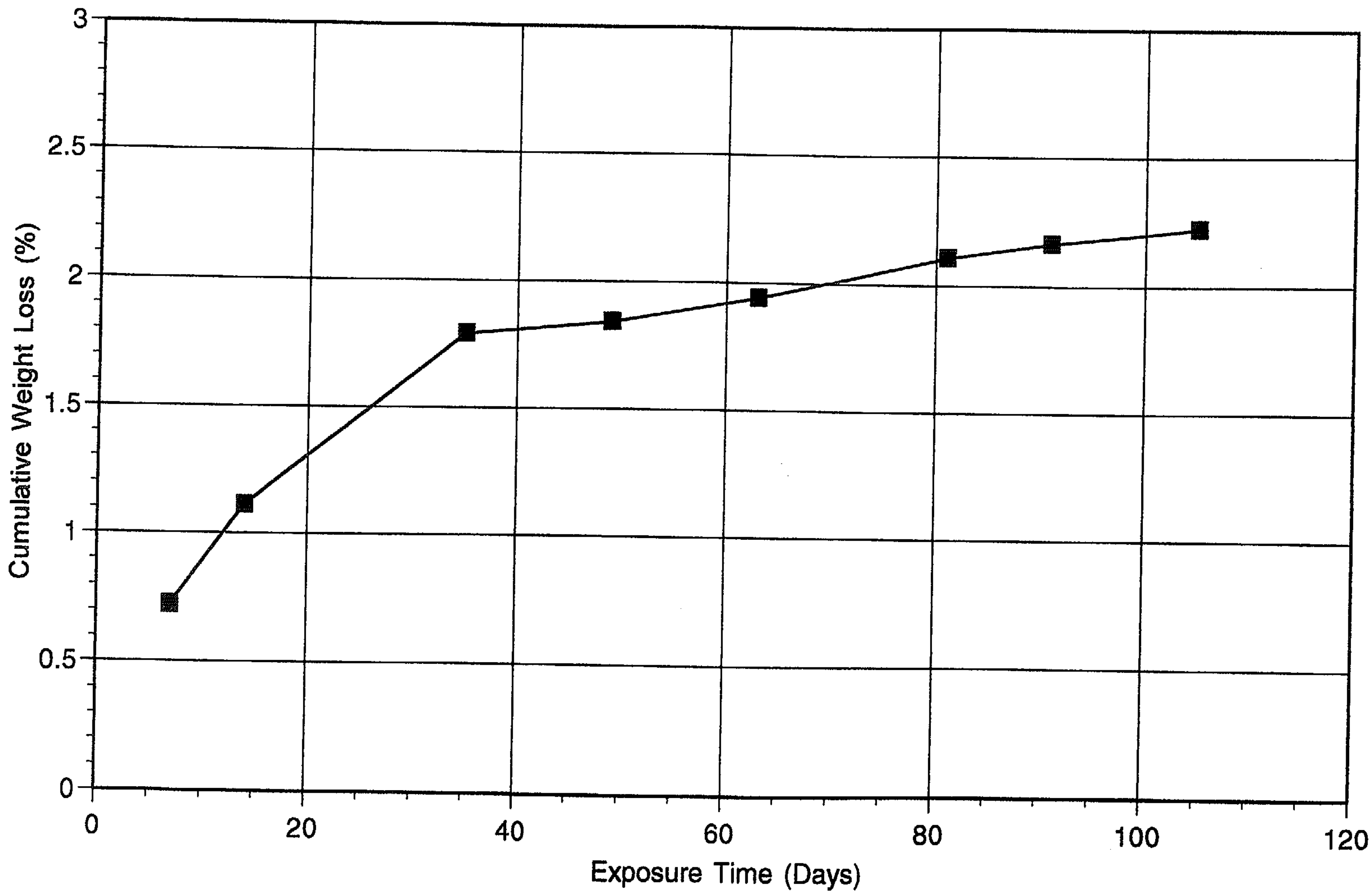


FIGURE 4.3: NAC-NS4FR Thermal Degradation Test Result (Sample #1-6)

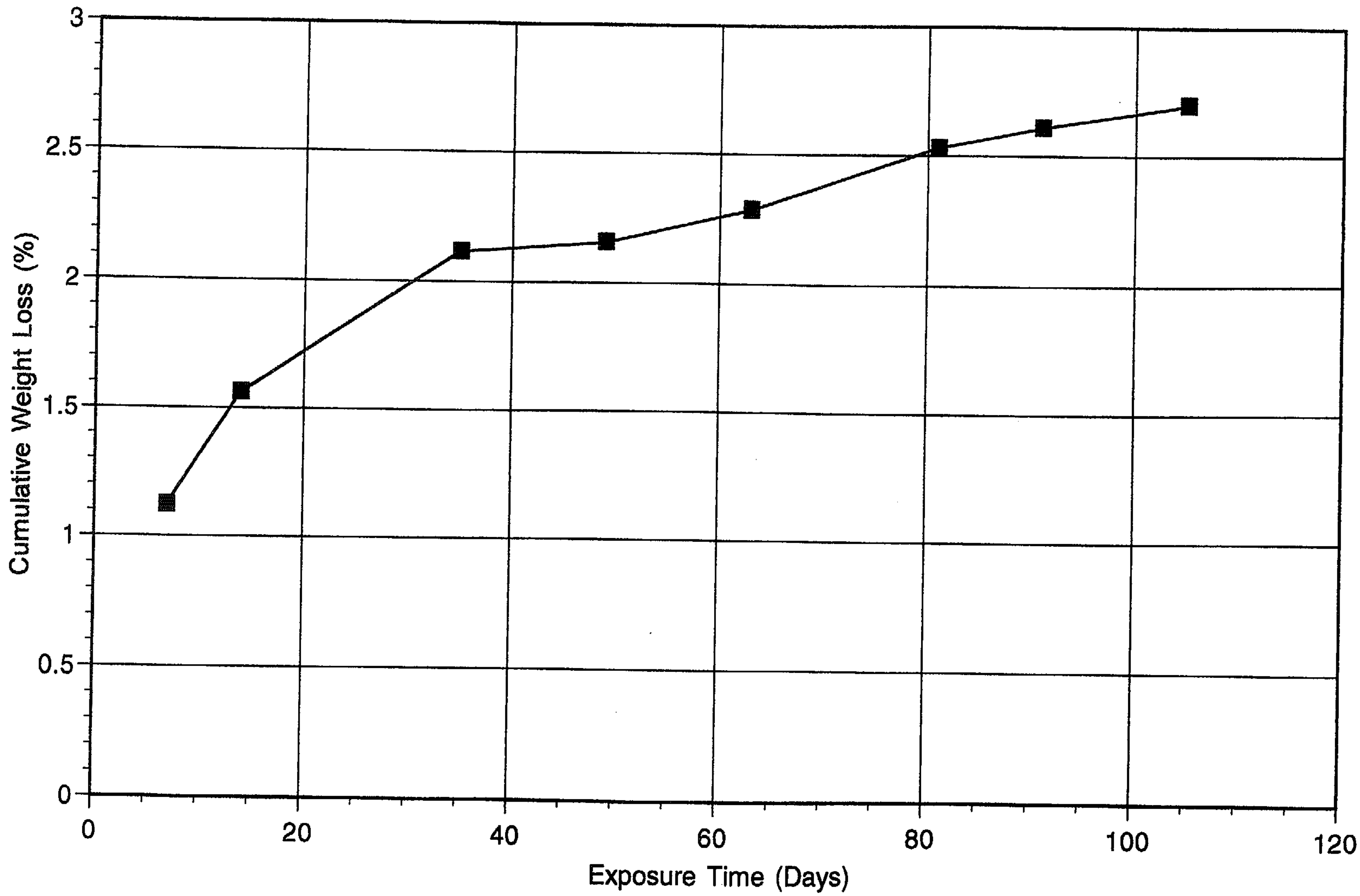


FIGURE 4.5: NAC-NS4FR Thermal Degradation Test Result (Sample #1-15)

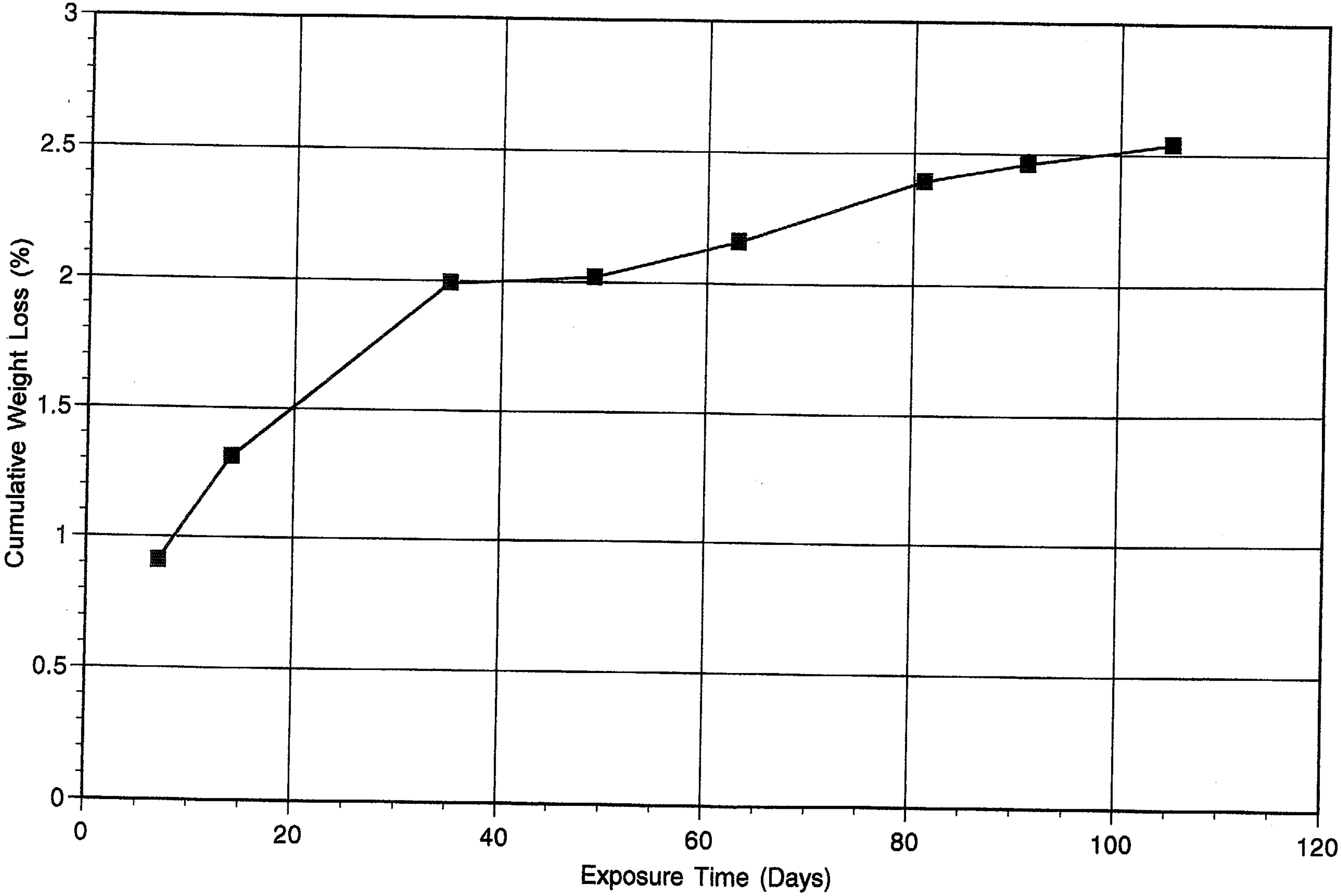


FIGURE 4.4: NAC-NS4FR Thermal Degradation Test Result (Sample #1-14)

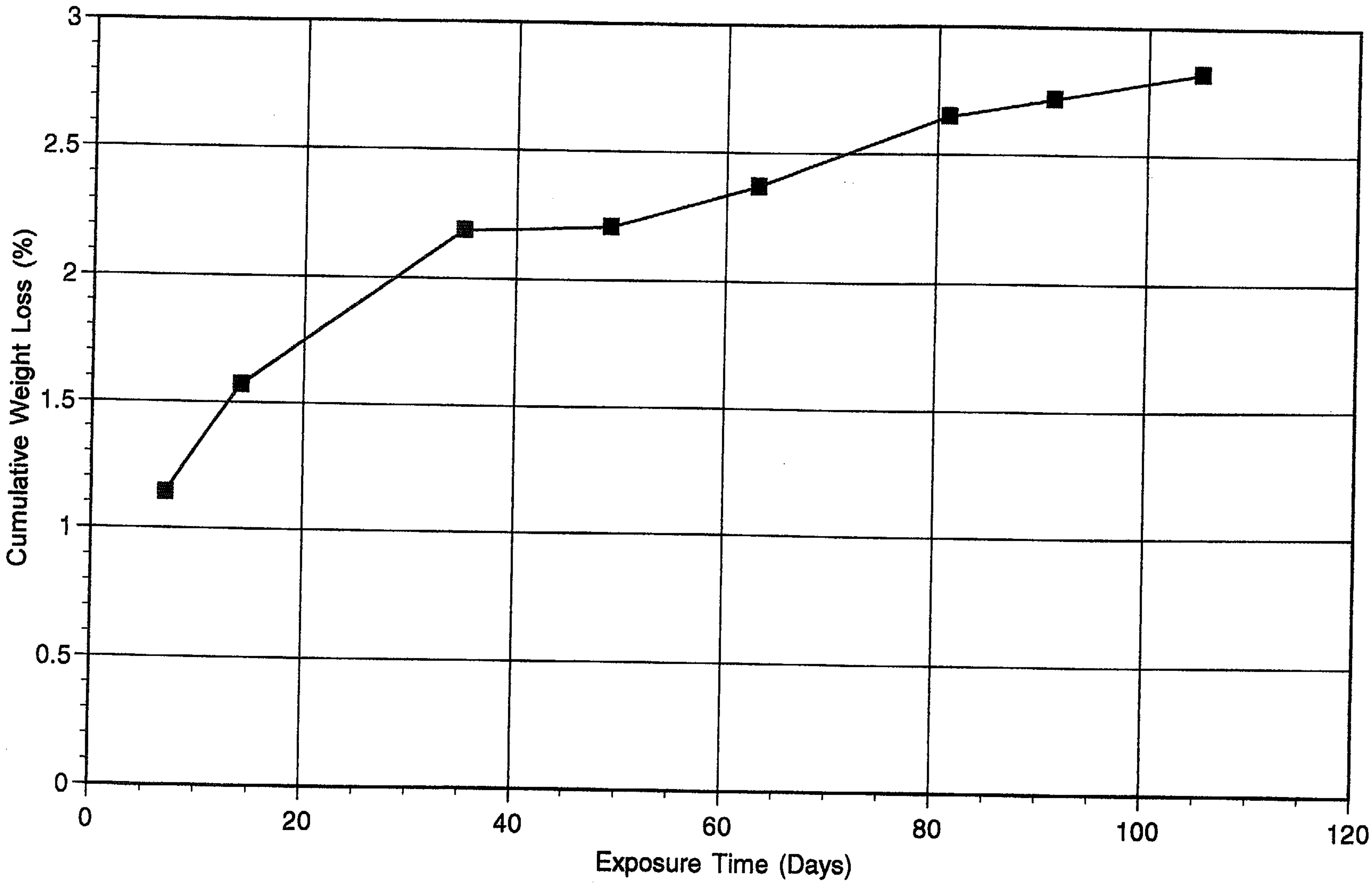


FIGURE 4.6: NAC-NS4FR Thermal Degradation Test Result (Sample #1-4)

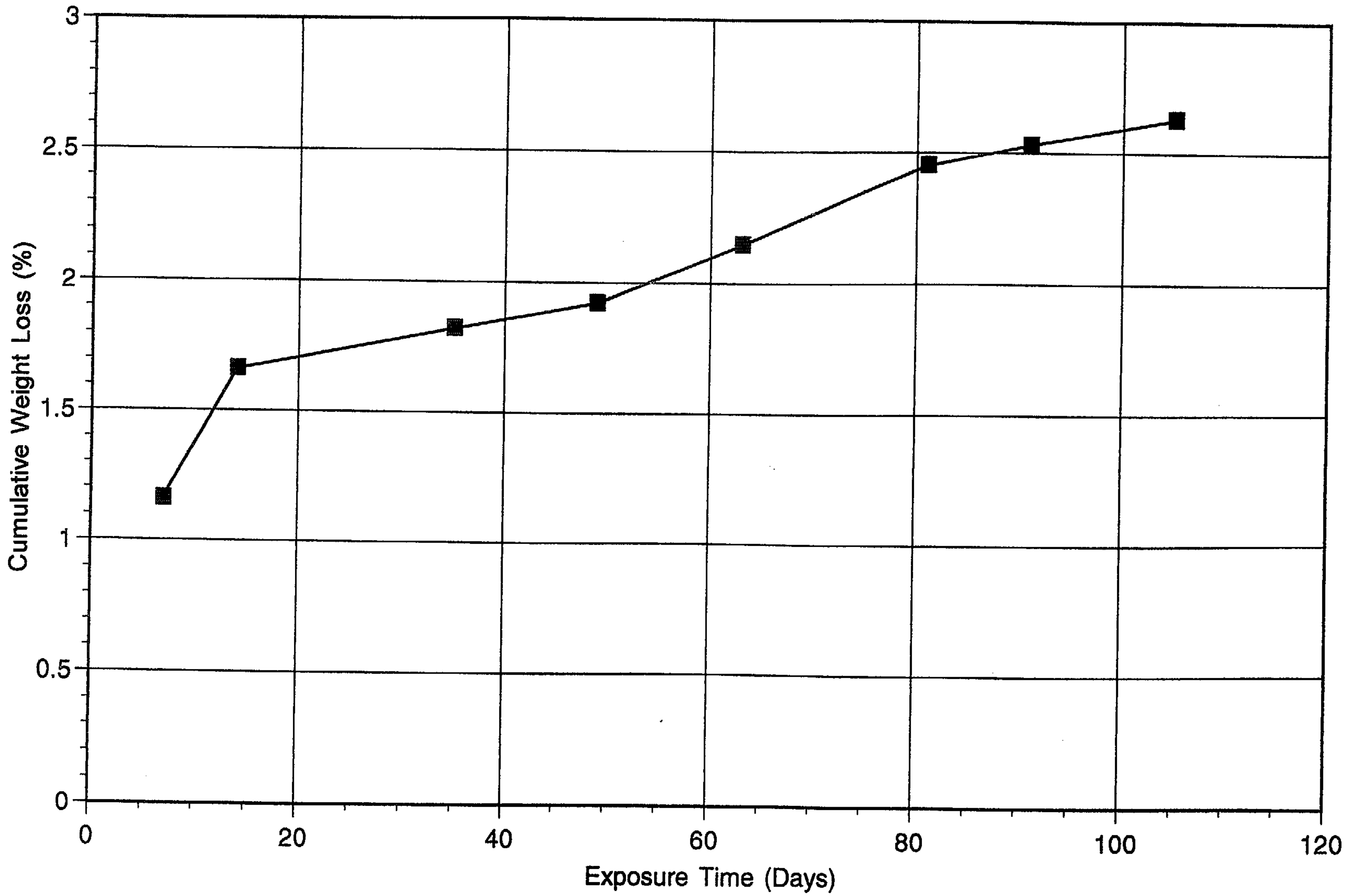


FIGURE 4.7: NAC-NS4FR Thermal Degradation Test Result (Sample #1-5)

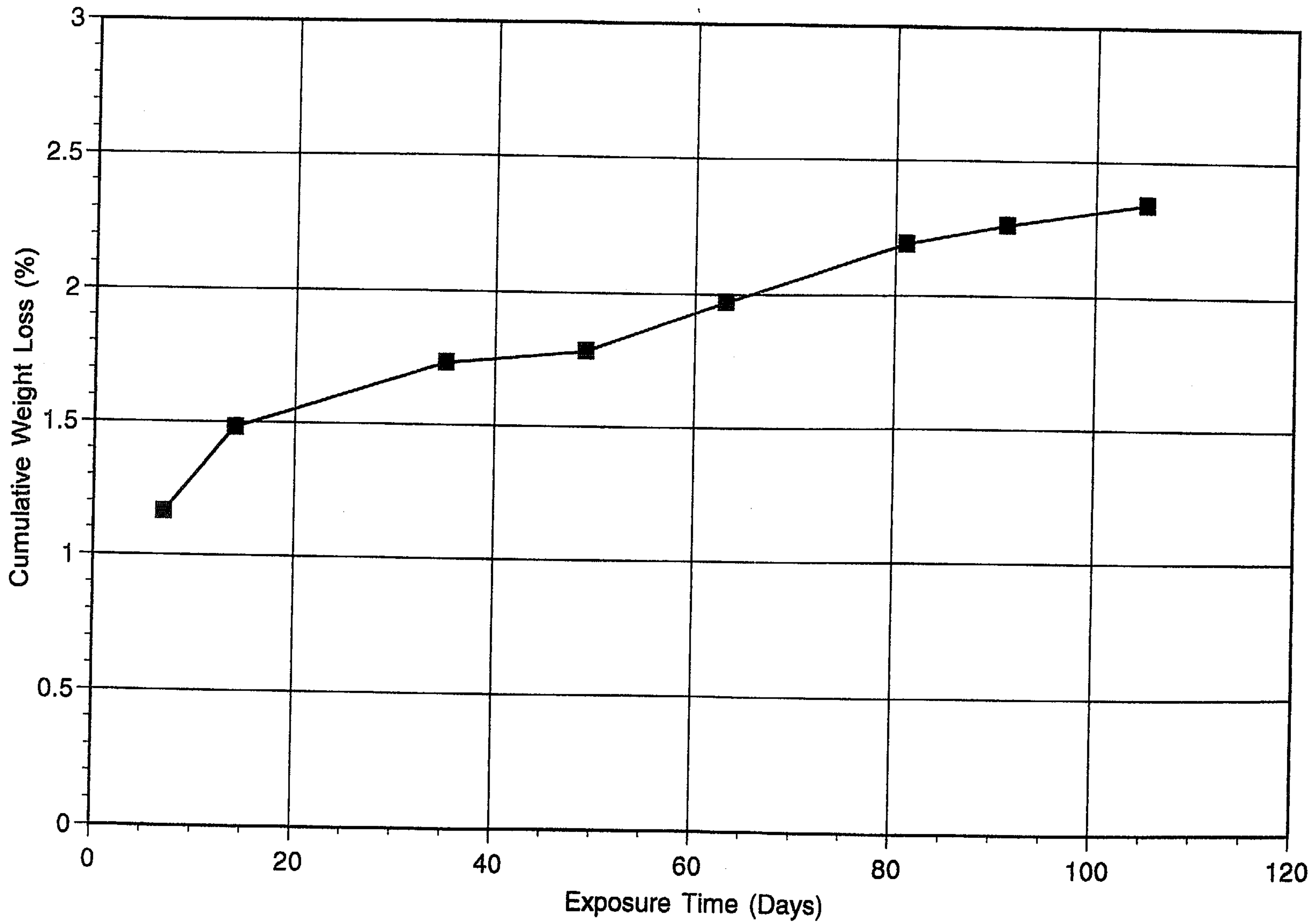


FIGURE 4.8: NAC-NS4FR Thermal Degradation Test Result (Sample #1-10)

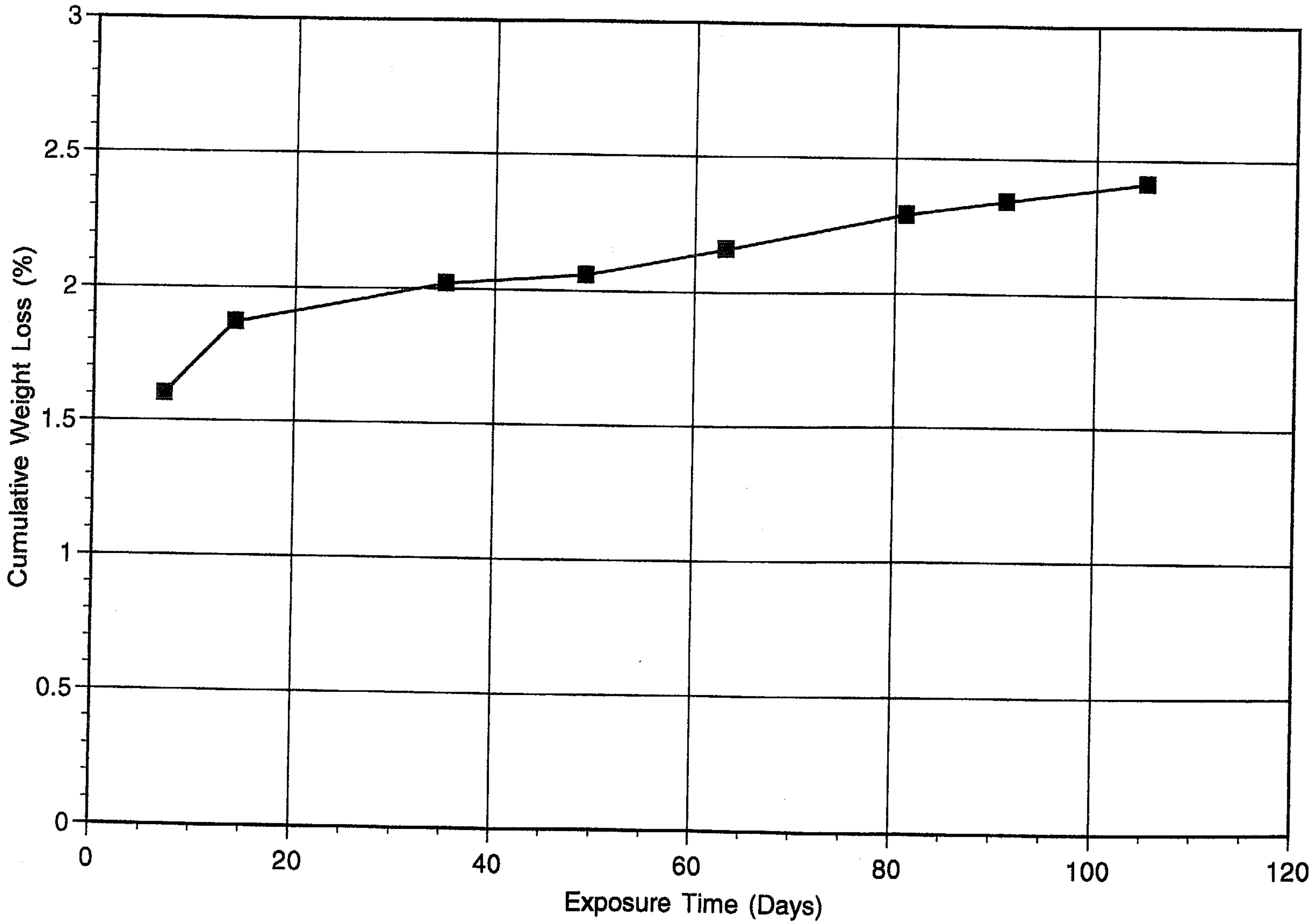


FIGURE 4.9: NAC-NS4FR Thermal Degradation Test Result (Sample #1-11)

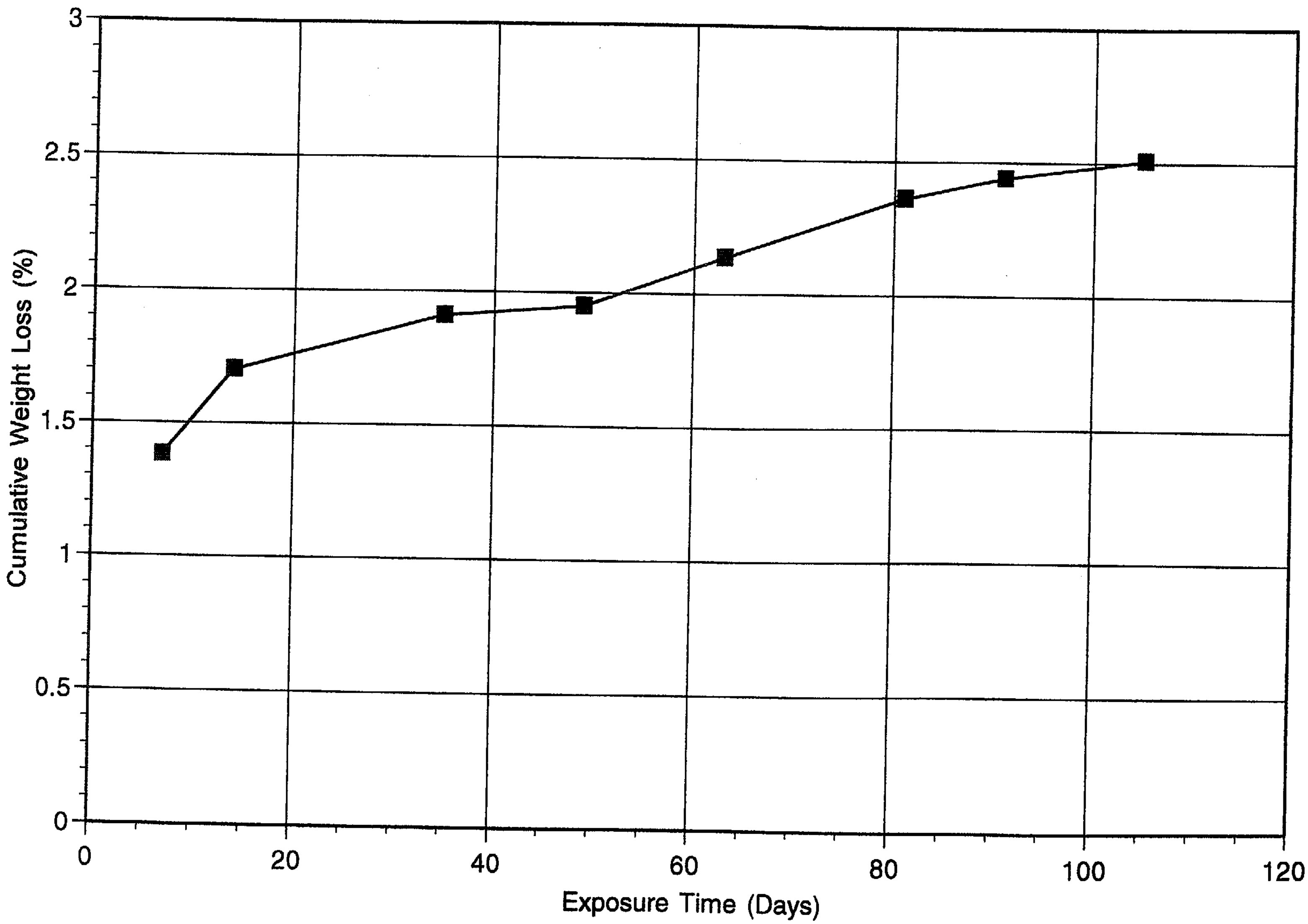


FIGURE 4.10: NAC-NS4FR Thermal Degradation Test Result (Sample #1-12)

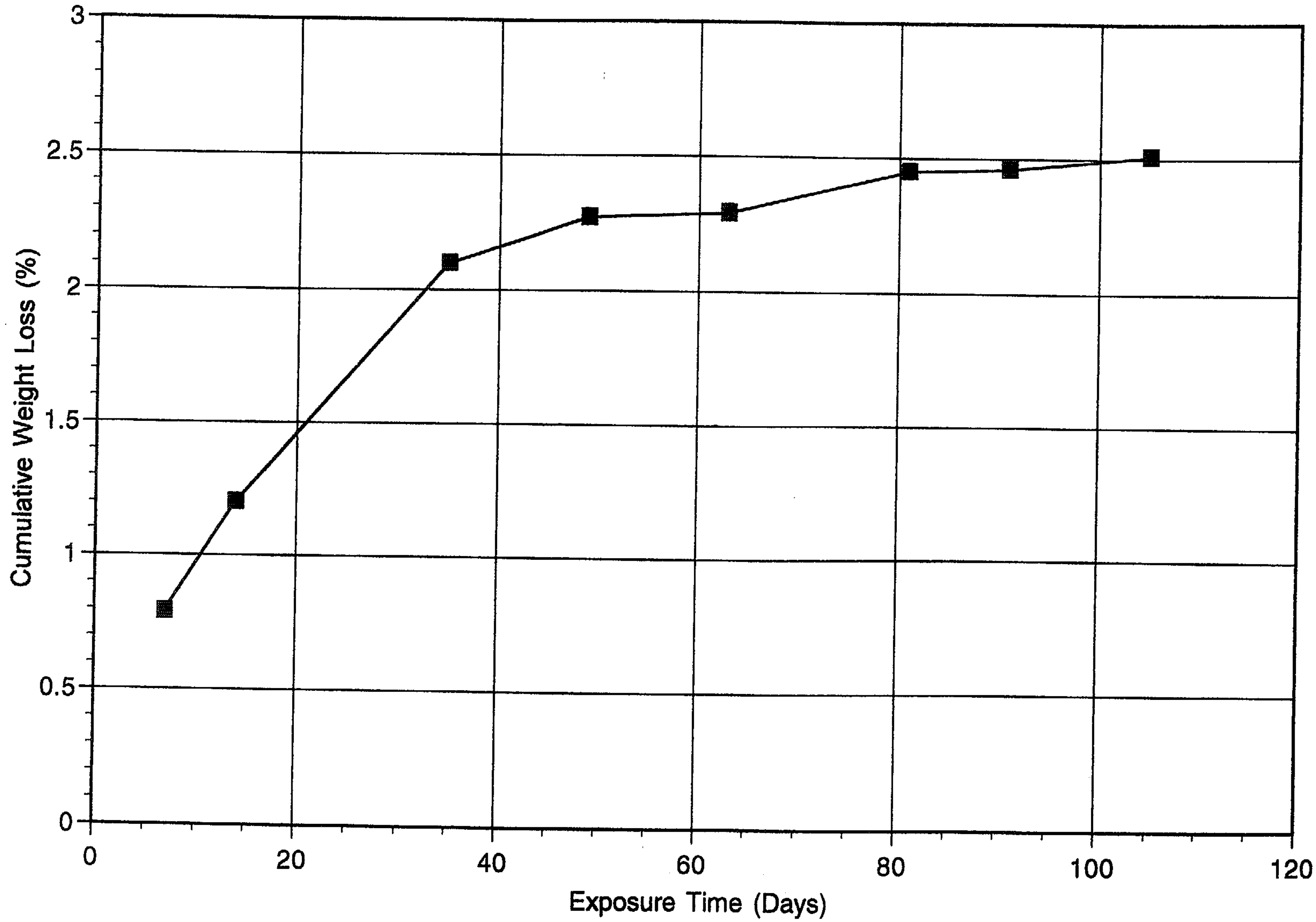


FIGURE 4.11: NAC-NS4FR Thermal Degradation Test Result (Sample #1-7)

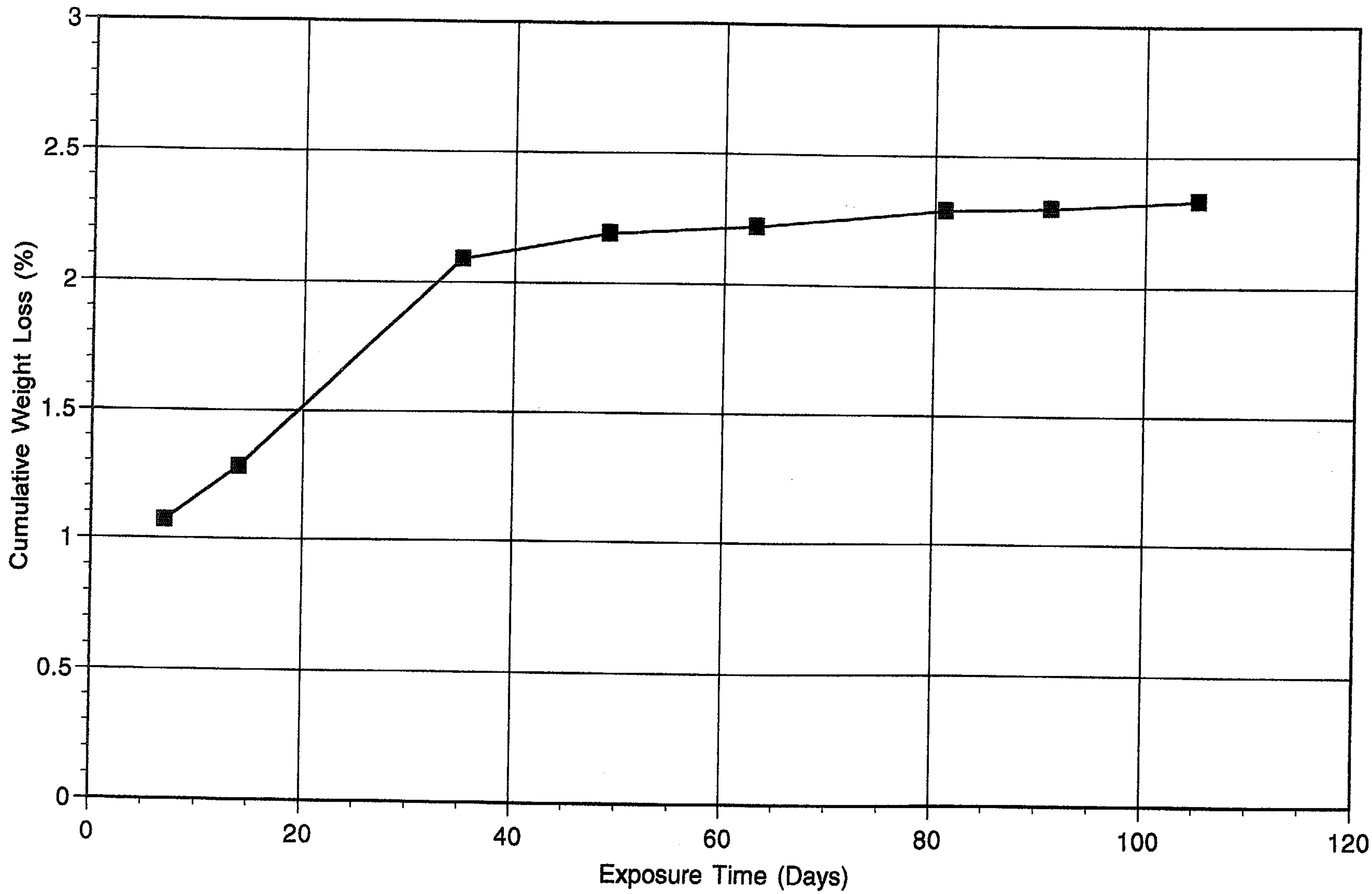


FIGURE 4.12: NAC-NS4FR Thermal Degradation Test Result (Sample #1-3)

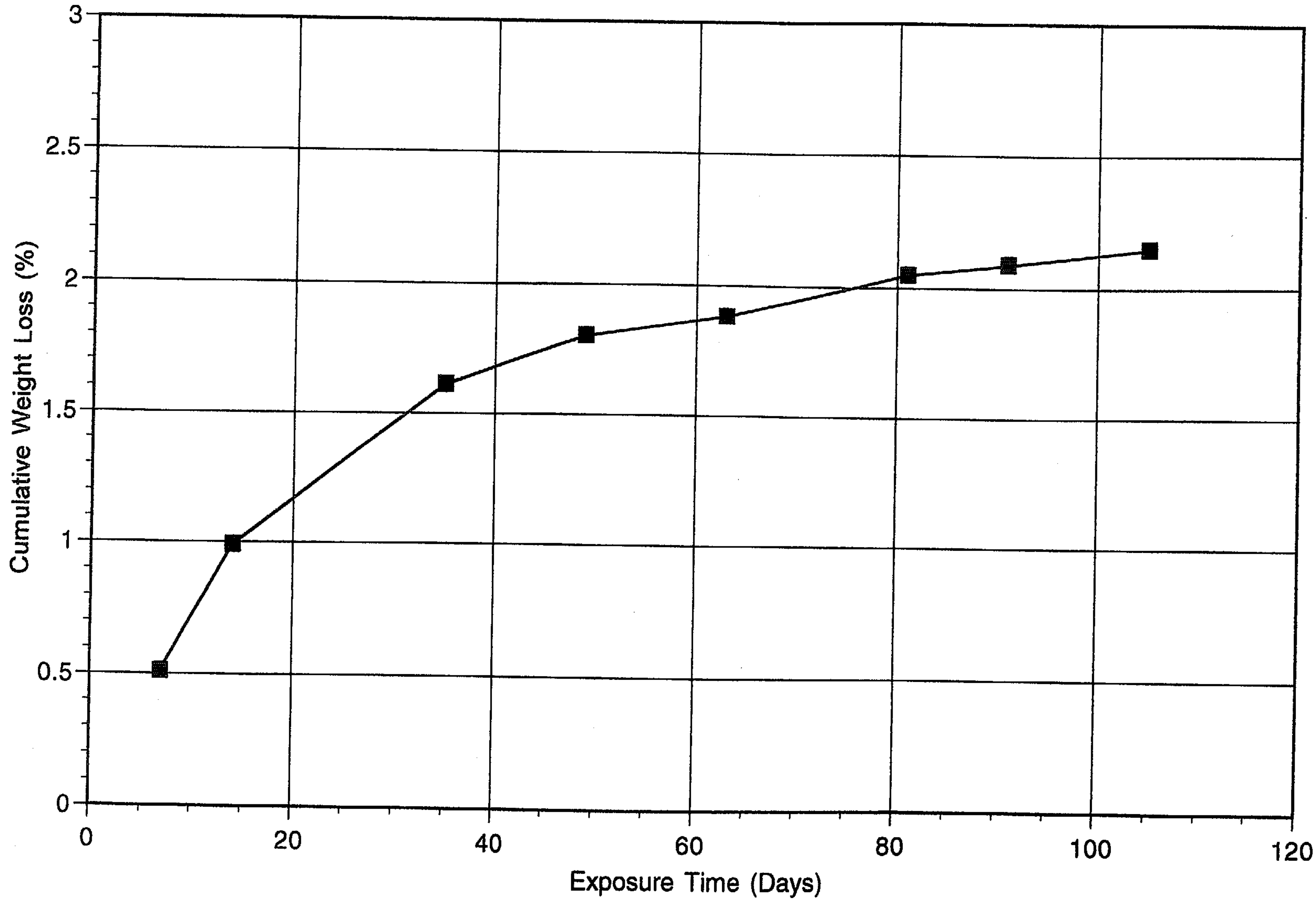


FIGURE 4.13: NAC-NS4FR Thermal Degradation Test Result (Sample #1-8)

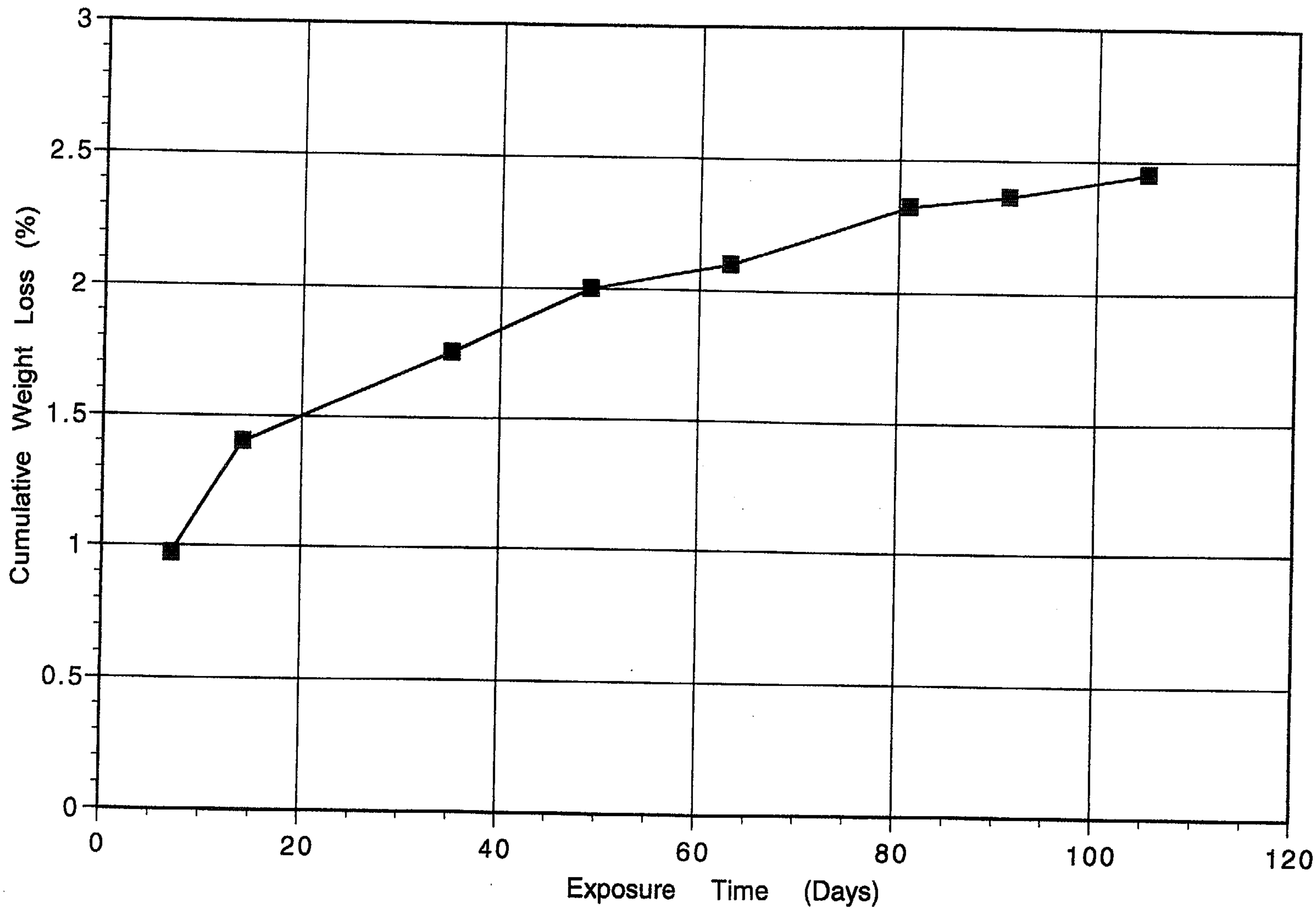


FIGURE 4.14: NAC-NS4FR Thermal Degradation Test Result (Sample #1-9)

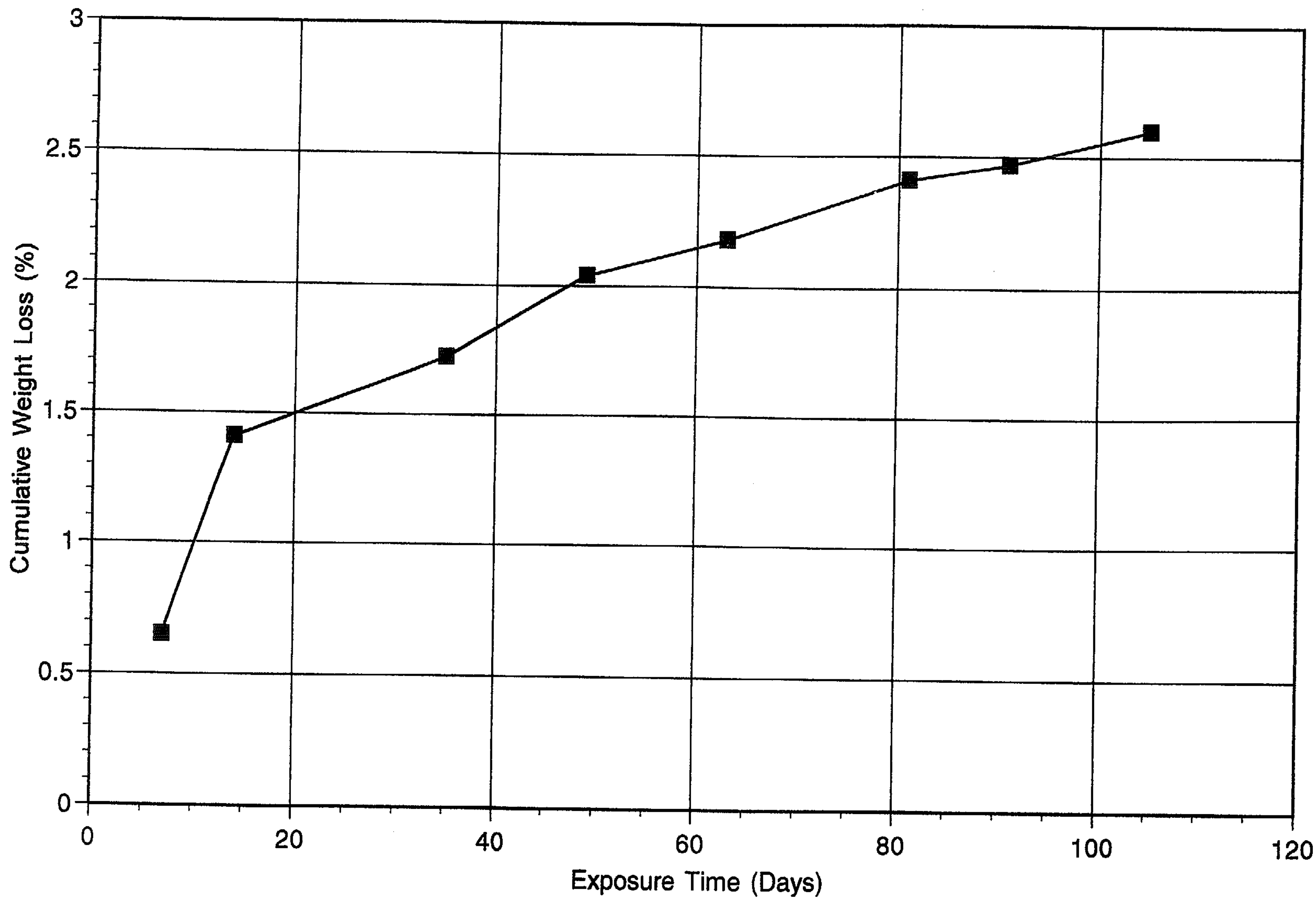


FIGURE 4.15: NAC-NS4FR Thermal Degradation Test Result (Sample #1-13)

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PROPRIETARY INFORMATION

FIGURES 4.16 THROUGH 4.22

CHAPTER 5: RECENT WORK ON THERMAL AGING TESTING OF HOLTITE-A AND NAC-NS4FR BY HOLTEC INTERNATIONAL

5.1 Introduction

In the previous chapter, the Holtite-A and NAC-NS-4-FR test data obtained by Holtec to identify the supply of the appropriate neutron shield in conformance with the HI-STAR 100 TSAR was described.

In this chapter, we summarize test results obtained from the continuous thermal aging studies on Holtite-A and NAC-NS4FR at the company's Florida's laboratories. The test results presented herein provide data collected on Holtite-A and NS-4FR collected up to March 2000. Thermal-aging tests have been made on Holtite-A and NAC-NS4FR. The samples were all tested at 325°F temperature in an air-exposed environment. Samples of Holtite-A prepared in the laboratory and from pours at the UST&D manufacturing facility were employed in the thermal aging tests.

5.2 NAC-NS4FR Testing

As described in Chapter 1, the NAC-NS4FR was purchased from NAC International under a safety-related purchase order. This material, as shown in Chapter 2, has a close similarity in the chemical composition to Holtite-A (Bisco-NS4-FR). The material was advertised by NAC International as NS-4-FR (the trade name used by the original developer, Bisco, Inc.). The material behavior, as confirmed by prior Holtec testing work, was consistent with the published work on NS4-FR by others [5,6].

The NAC-NS4FR was received from NAC International moulded in plastic tubes 4 in diameter and 24 in long. The NAC-NS4FR was removed from the plastic tubing and cut into samples of a suitable size for testing. The sectioned samples had revealed voids in the material as shown in photographs produced in Appendix 4. Although voids in an installed condition for cask use are considered undesirable, testing of the material proceeded upon NAC's assurances that the supplied material was suitable for thermal aging tests and that they are in the know of producing voids free installations in casks.

A total of 15 samples of NAC-NS4FR were tested, ten of which were maintained at 325°F for slightly over two years. For the remaining five samples, the temperature was reduced to 300°F after 462 days of testing and maintained at 300°F for an additional 289 days. Early test data on these samples was discussed in Chapter 4. After 751 days at 325°F, the average weight loss of the ten NAC-NS4FR samples was 7.7% including one sample (#1-5) showing a weight loss approaching 12%. Figures 5.1 through 5.10 illustrate the weight loss history of these samples. The test data is archived in Appendix 6. It is noted from these plots that several samples took on an abrupt increase in the rate of weight loss* (starting at about 150 days of cumulative exposure) and lasts more than 100 days or so before leveling out to a more gradual pattern. It is postulated that decomposition within the samples initially produced gases that were trapped inside the sample. As fissures formed in the samples to release the decomposition gases, an increase in rate of weight loss was observed. Evidence corroborating this postulate is discernible from photographs of the NAC-NS4FR samples attached as Photos A through H after 751 days of exposure. Cracking, fissures formation and blisters are observed by visual inspection of these photos. The photos also show some charring (carbon formation) in the samples. Photo F shows swelling that was observed in one sample (#1-11).

5.3 <u>Holtite-A Thermal Aging Tests</u>

The initial testing had focused on NAC-NS4FR based on supplier's assurances that the material was same as the original Bisco-NS4FR. Once it was realized that the NAC material may not muster qualification, testing began on generic versions of the family of original Bisco formulations. These tests encompassed samples with variations in composition (early 1998 testing) and later testing (in 1999) focused entirely on material with the same composition as the original Bisco-NS4FR formulation, named Holtite-A by Holtec International. Results of the

* It is noted that there are isolated instances of weight increase between successive data points. The mass increase is likely caused by a temporary weight gain from retention of oxidation products in samples.

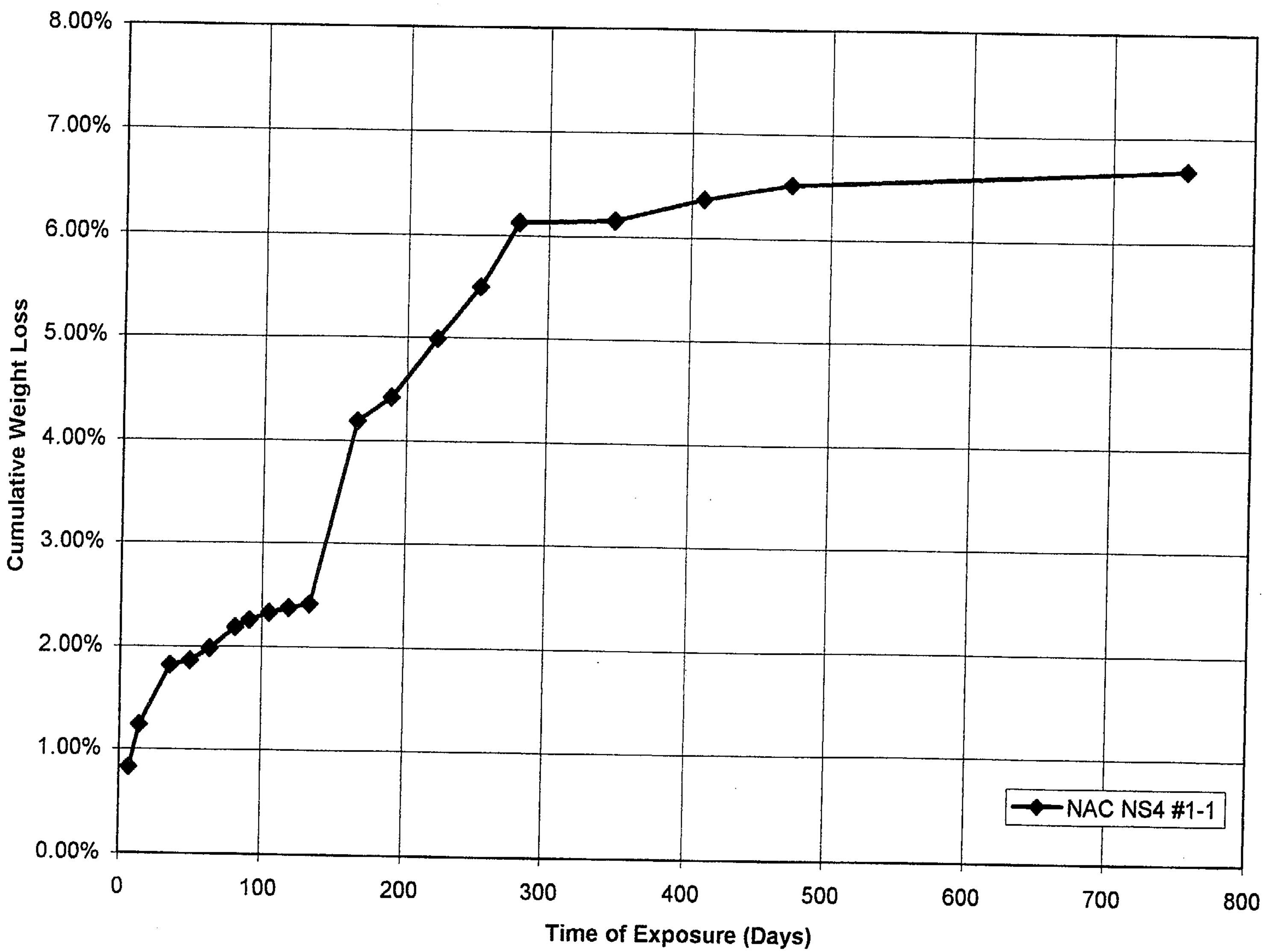

Figure 5.1 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

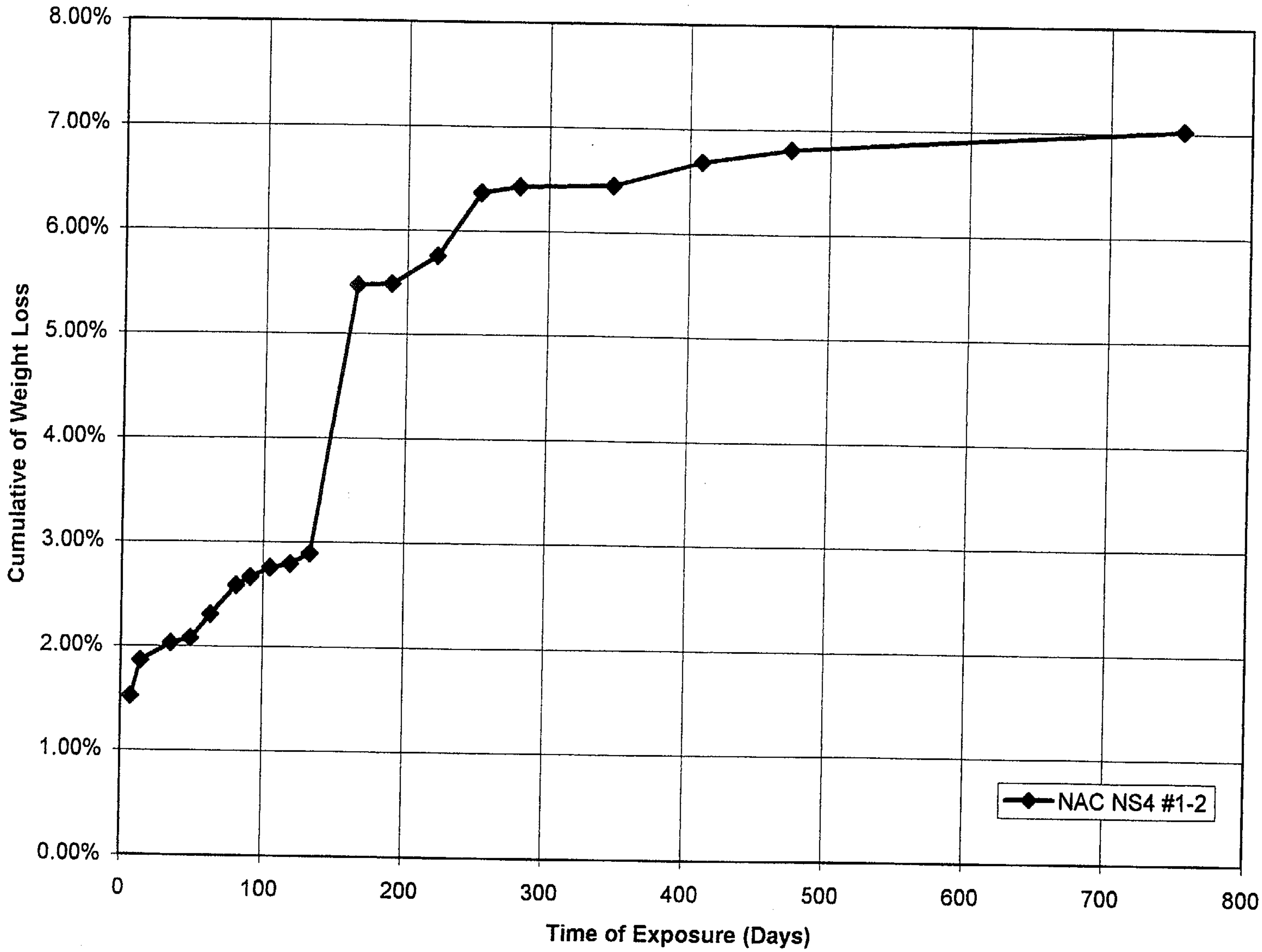

Figure 5.2 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

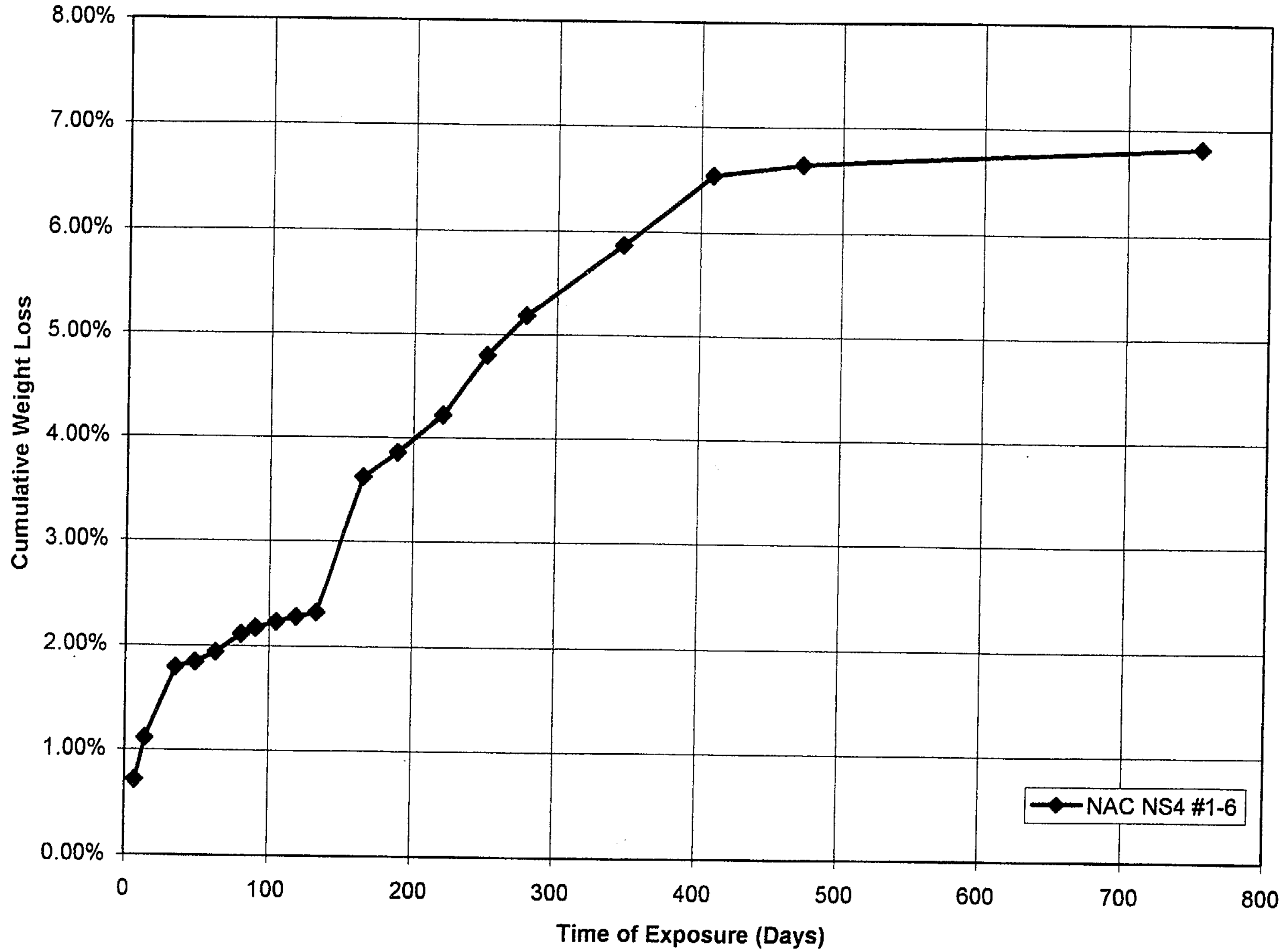

Figure 5.3 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

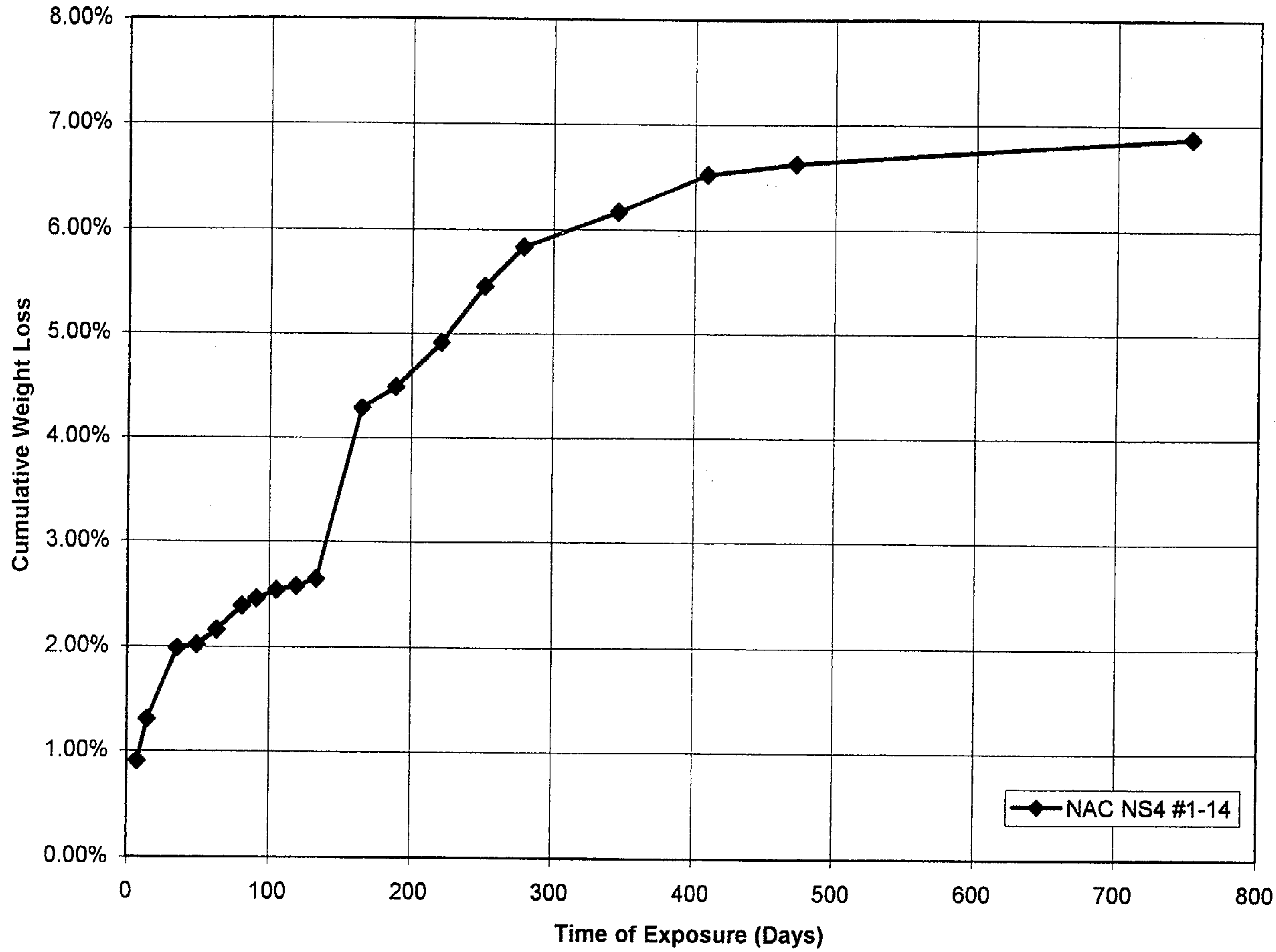

Figure 5.4 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

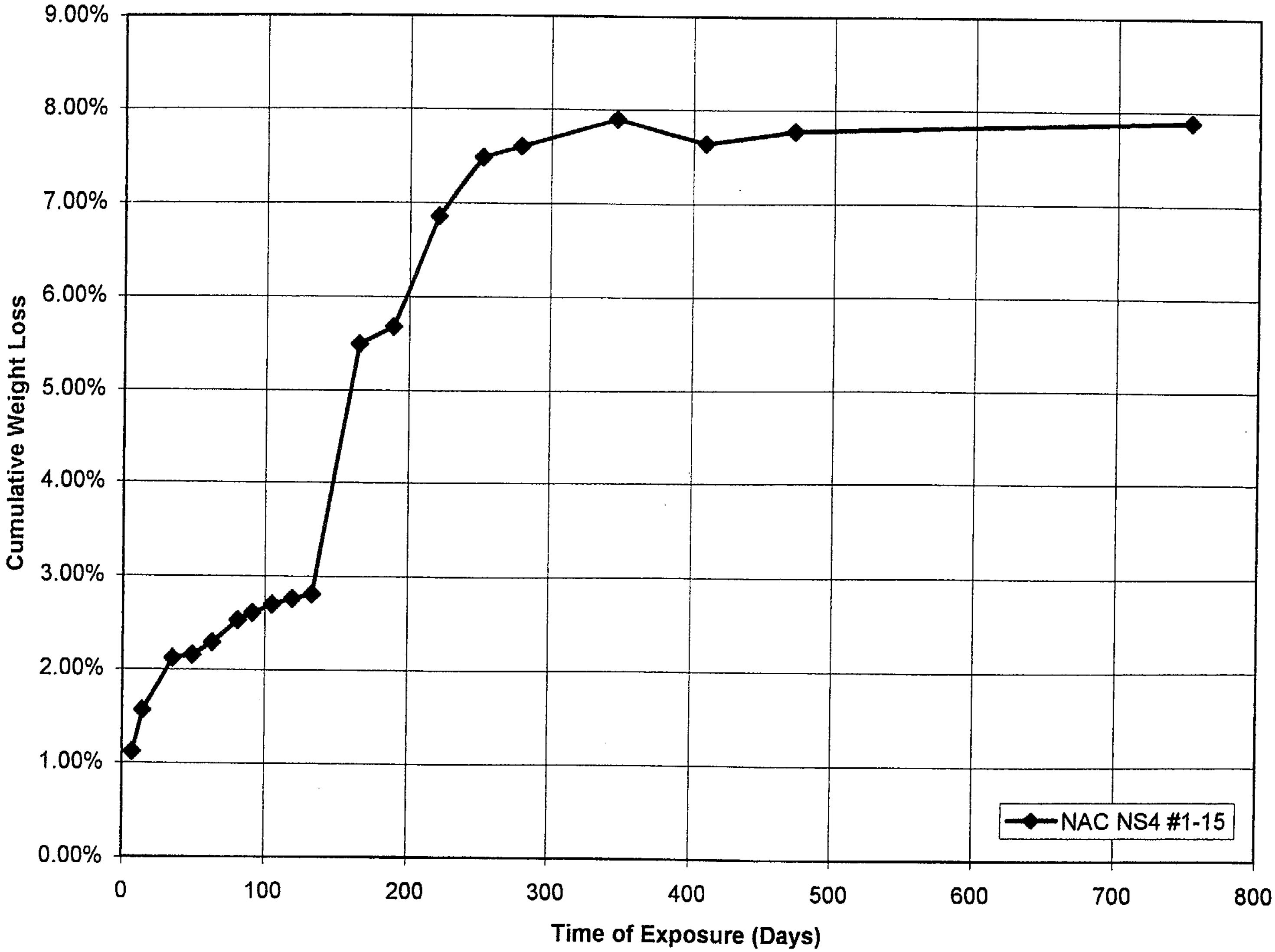

Figure 5.5 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

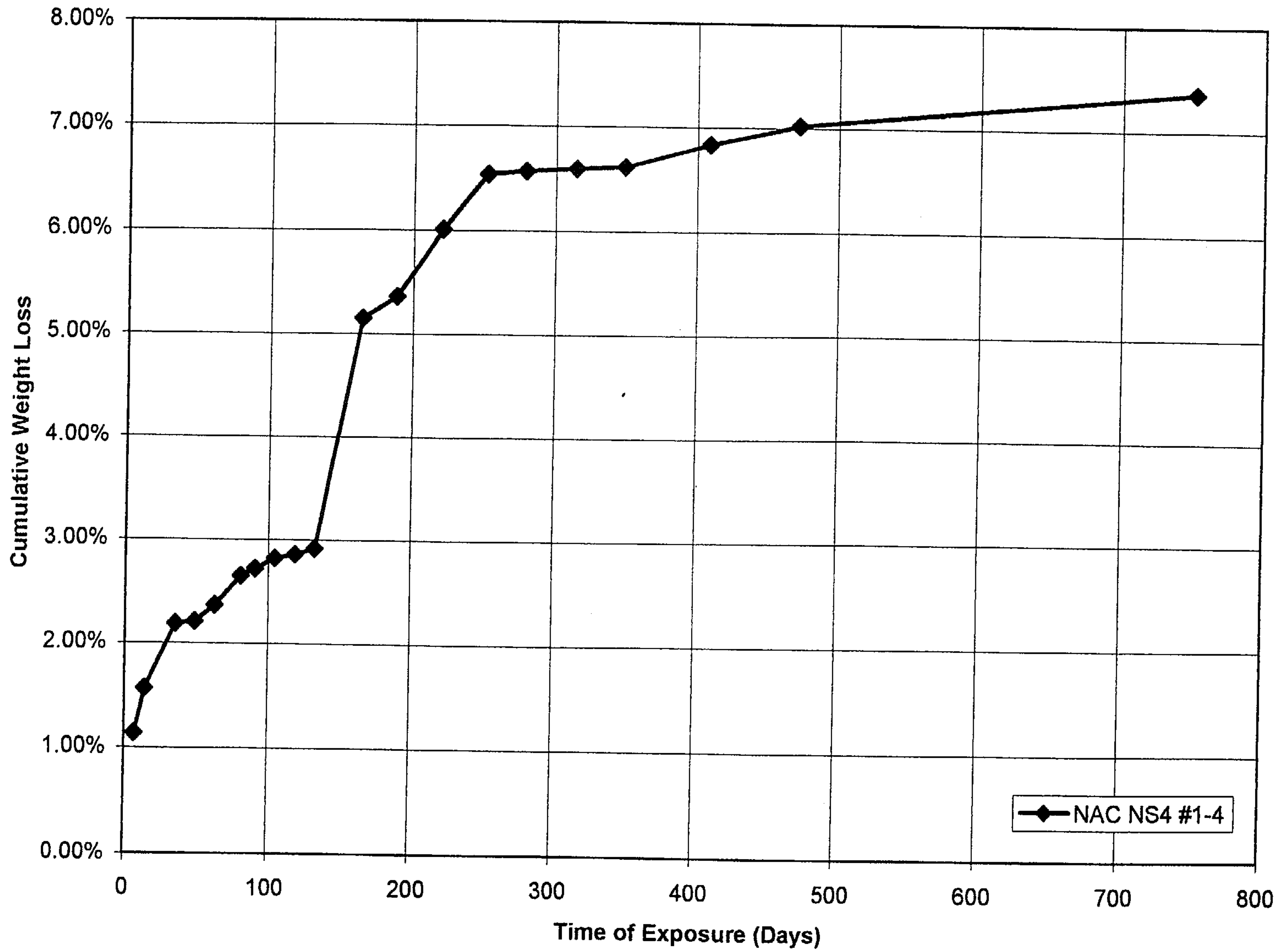

Figure 5.6 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

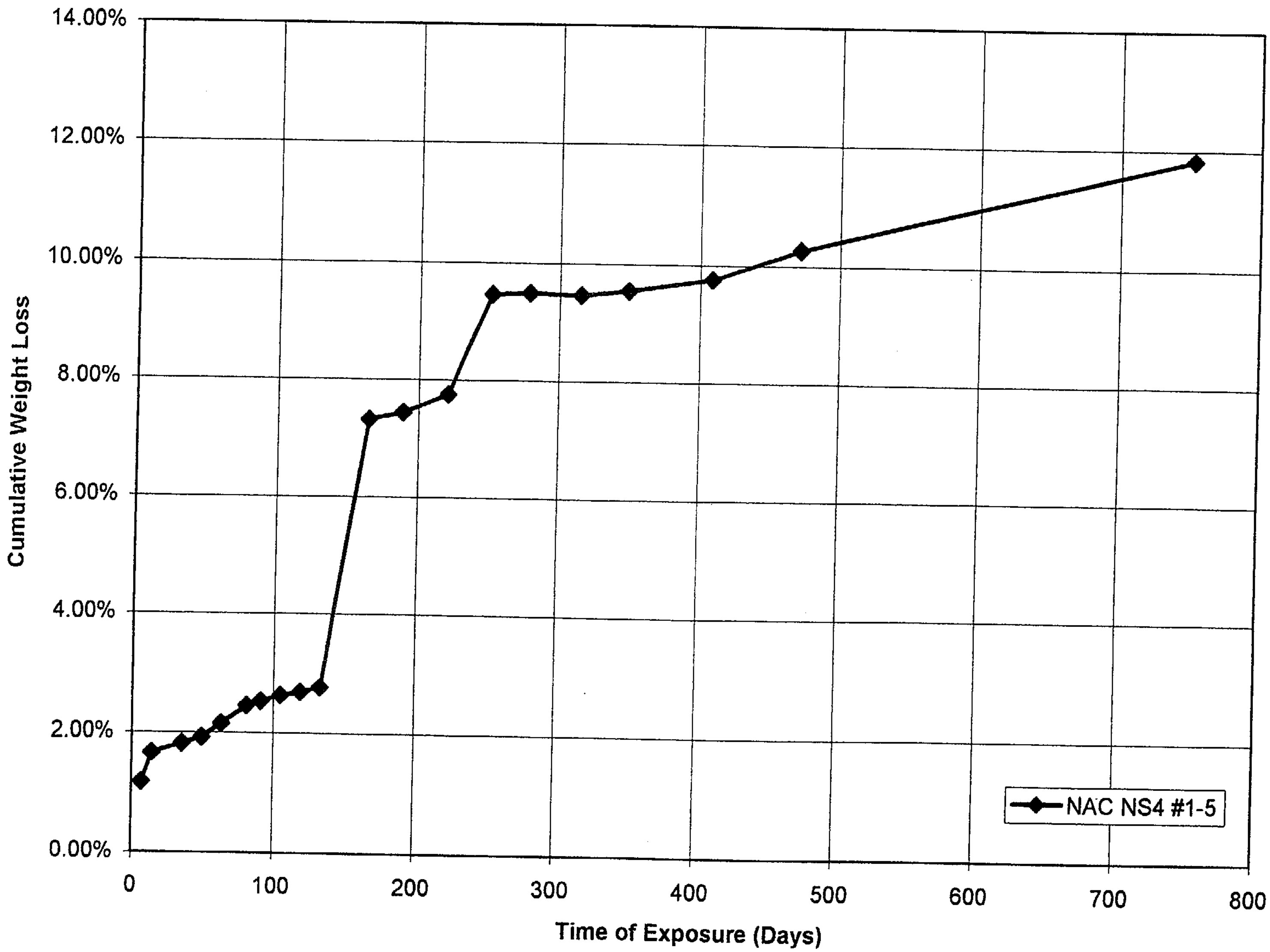

Figure 5.7 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

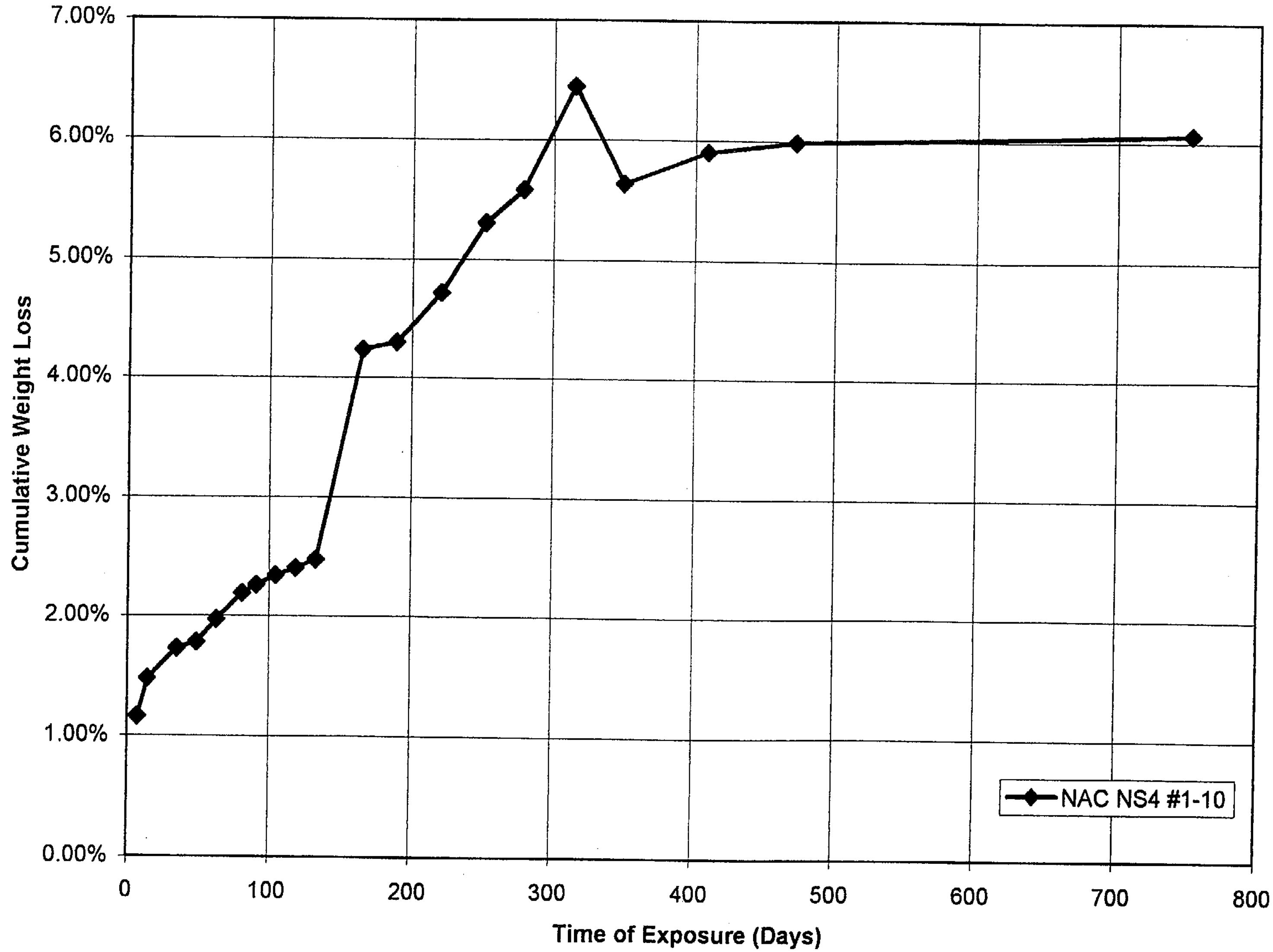

Figure 5.8 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

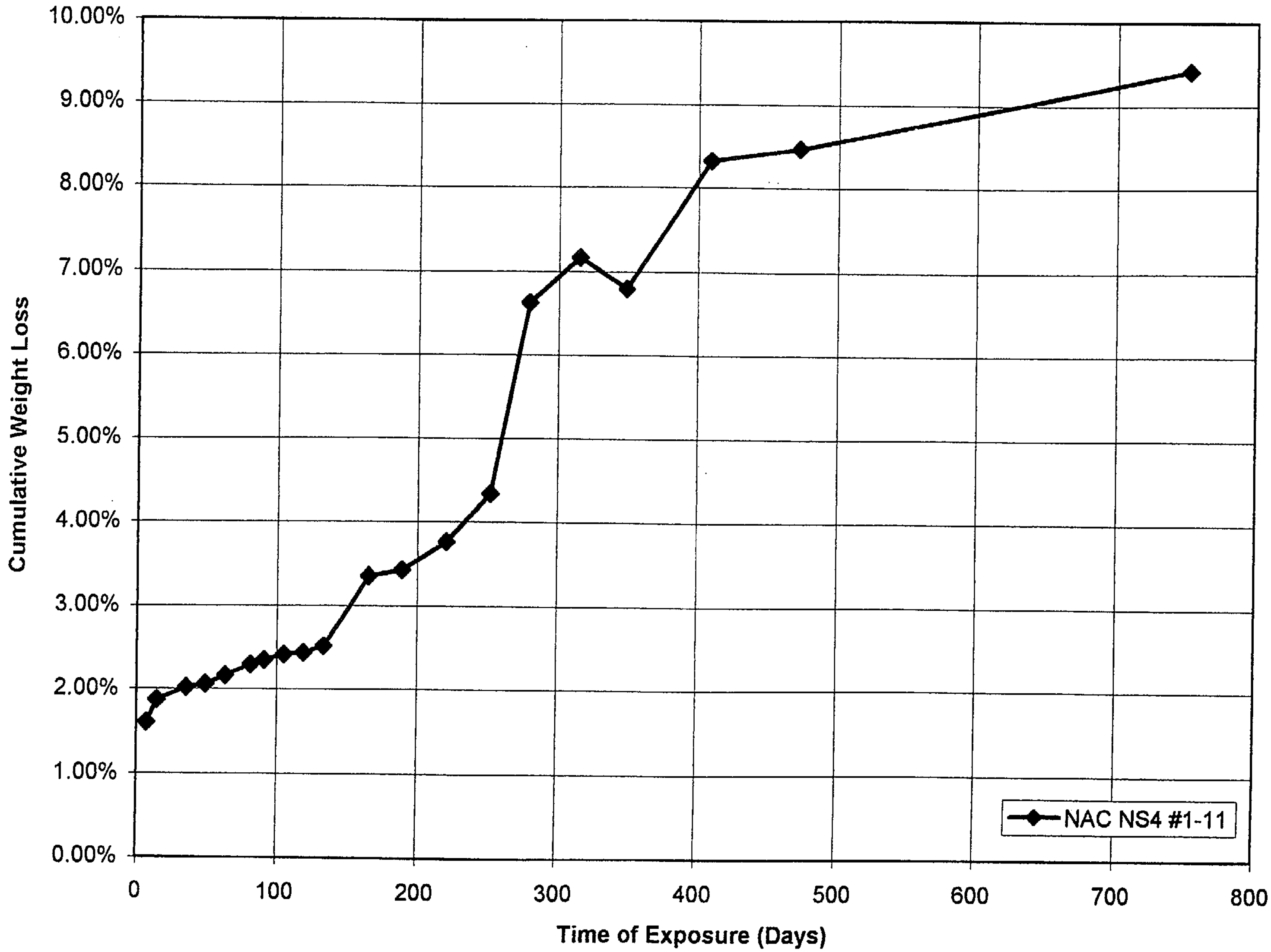

Figure 5.9 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

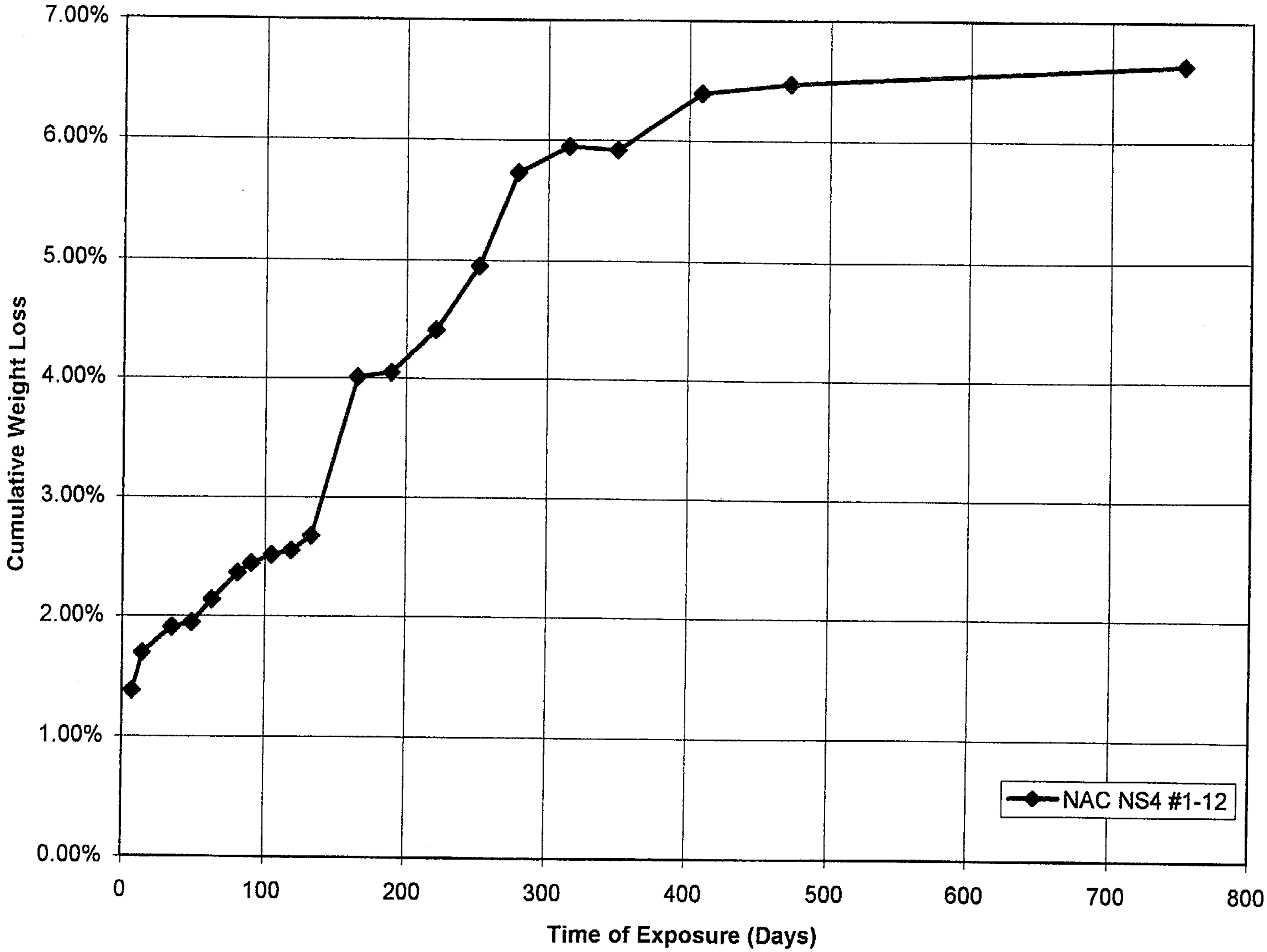

Figure 5.10 : Thermal Aging Time History of NAC-NS4FR at 325 Degrees F.

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PROPRIETARY INFORMATION

FIGURES 5.11THROUGH 5.17

testing of Holtite-A are shown in Figures 5.11 through 5.17*. A total of seven samples were tested at 325°F (four lab prepared and three shop floor samples). The average weight loss was [redacted] in the thermal aging test [redacted] exposure at 325°F). The sample average weight loss and mean exposure duration is computed as [redacted] and 238 days. Photographs of the Holtite-A at the end of the test are shown in Photos I through P.

An examination of the Holtite-A thermal aging data provided in this chapter leads to the following observations:

- The samples are free of large voids and gaps.
- The material behavior (weight loss) is consistent with Bisco-NS4FR (Japanese results) on their long-term (56 week) thermal aging test [5].
- Visual examination of test samples confirm material is stable (i.e. warping, swelling or cracking not observed).
- Lab to shop floor scale up of Holtite-A production confirmed.
- Assurance that weight loss results (<4%) muster regulatory compliance.

* The figures in this chapter include the data from early testing presented in Chapter 4.

CHAPTER 6: DISCUSSION AND CONCLUSIONS

In the shielding analysis of the HI-STAR 100, a 4% weight loss of Holtite-A was conservatively stipulated for the 20-year projected service life of the cask. The earlier Bisco tests on Bisco-NS4FR [7] reported a weight loss of 3.15% after 146 days at 338°F. Holtec tests on samples of Holtite-A, a material with the same formulation as Bisco-NS4FR, gave similar results. An average sample weight loss of 3.1% for a mean exposure of 238 days at 325°F was obtained from the test data. The test temperatures are much above the expected range of temperatures to which the Holtite-A would be exposed in the HI-STAR 100 cask during the 20-year storage period (discussed in Chapter 3). Thermal analysis, summarized in Chapter 3, indicates that the maximum temperature of the inner surface of the Holtite-A inner surface is 270°F and decreases to less than 200°F in three years (no thermal degradation expected at 200° and below). The Holtite-A temperature also decreases substantially on either side of the midplane. Thus, the bulk temperature of the Holtite-A will be substantially below the initial maximum temperature at the inner surface of the Holtite-A. After three years, even the maximum temperature decreases to less than 200°F.

The Japanese experiments, "Experimental Studies on Long-Thermal Degradation of Enclosed Neutron Shielding Resin" [5], were performed on a material identified as "NS4-FR supplied by Bisco Co." The results for the 2500 hr testing are reproduced from the study (Fig. 3 of [5]) in Figure 6.1. Observations from the Japanese study [5] pertinent to an evaluation of Bisco-NS4FR material performance in a cask are:

- In an enclosed space (such as the shield material cavity of the cask), the decomposition will be less than that observed in an open system.
- For different shaped samples of the same weight, the one with a larger surface area has a higher weight loss.
- Weight loss is a monotonically rising function of temperature

Based on these observations it is noted that the service conditions of Holtite-A in a HI-STAR cask are relatively benign. A large bulk of this material (~12,000 lbs) is poured and cured in-situ in the forty annular spaces and sealed. The exposed surface area per lb of the material is practically nil compared to the tested samples which weigh about 1 lb and thus subject to a much higher weight loss in an open (oxidizing) environment. The material is tested at a much higher than the maximum reported temperatures for Holtite-A in HI-STAR (the temperatures themselves computed in a conservative manner at design maximum heat loads with effects of wind completely ignored). The bulk temperature is much lower than the maximum computed temperature that monotonically reduces as a result of an exponential attenuation of fuel decay heat with time in dry storage. From these considerations, there is assurance that Holtite-A is in a safe thermal environment for use in the HI-STAR cask. To provide a quantitative upperbound to Holtite-A weight loss in the HI-STAR cask projected for a 20-year duration, the following numerical exercise is undertaken. The Holtite-A thermal decomposition time-history is fitted to a logarithmic form [5] presented below:

[redacted]

Postulating a constant T (at maximum 270°F) for a period of 20 years in HI-STAR cask for Holtite-A in the above equation, a projected weight loss of [redacted]. In other words, even if an overly conservative assumption for the thermal environment in the HI-STAR cask is made, the licensing basis of a commitment of 4% weight loss for Holtite-A is shown to be satisfied.

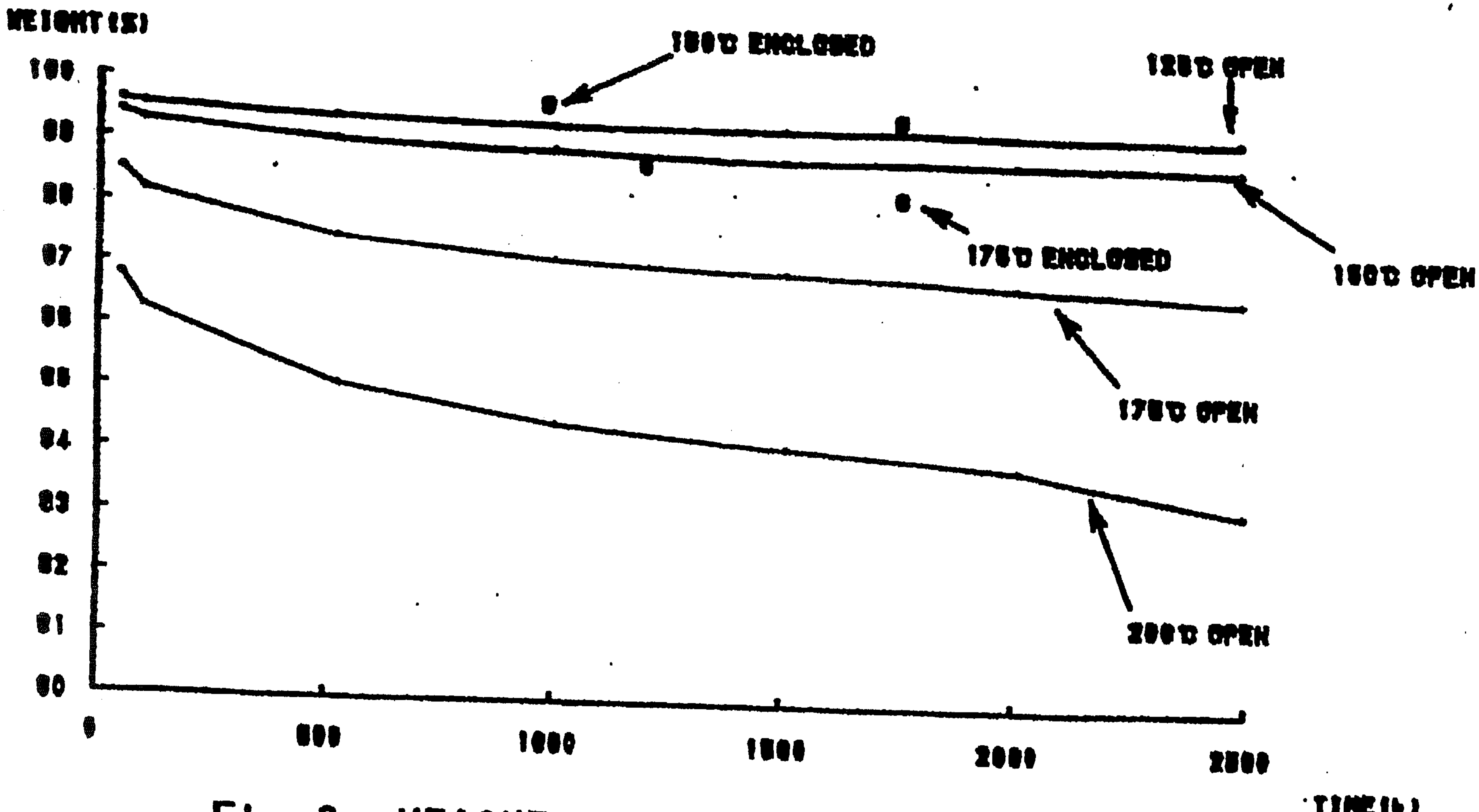


FIg.3 WEIGHT LOSS BY OPEN AND ENCLOSED TEST

Figure 6.1 Japanese Experimental Studies; Extracted from Reference 5

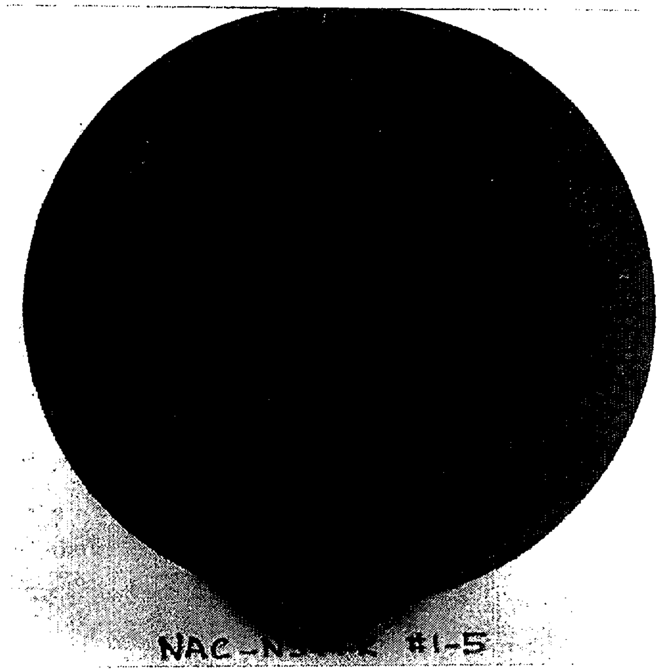


PHOTO A NAC–NS4 FR
752 DAYS @ 325 F

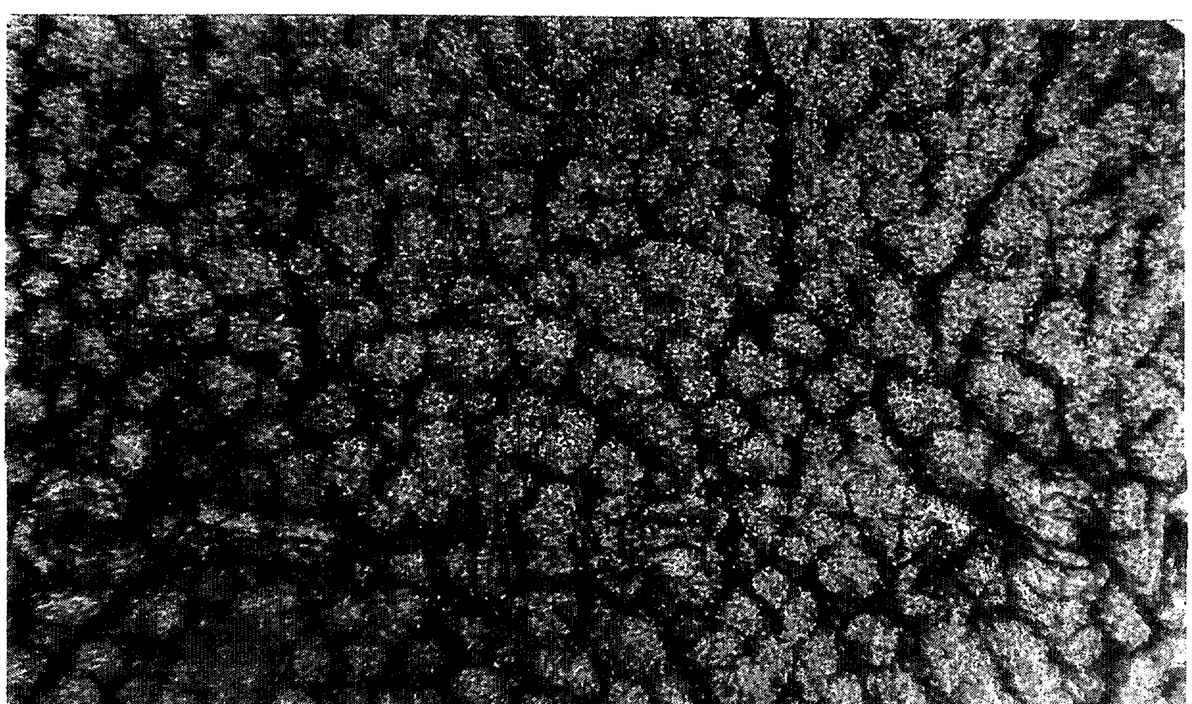


PHOTO B NAC–NS4 FR
752 DAYS @ 325 F

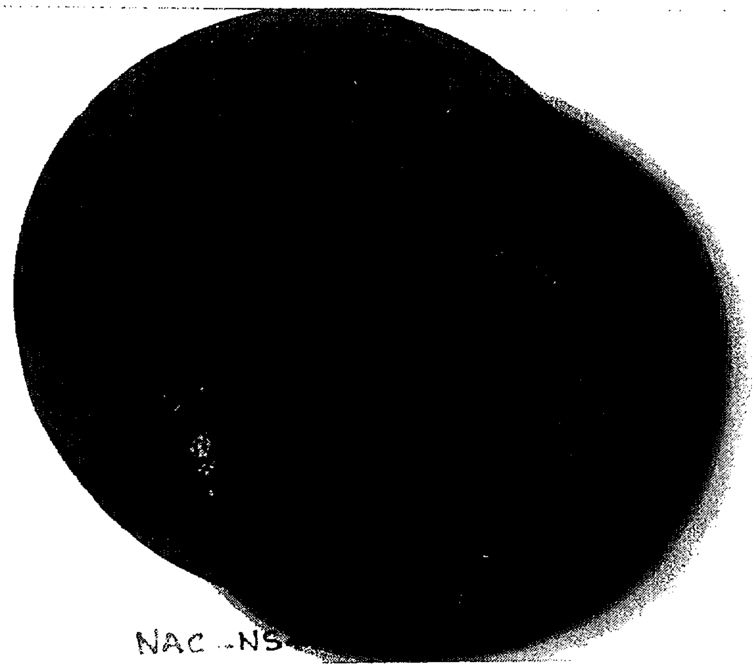


PHOTO C NAC–NS4 FR
752 DAYS @ 325 F

PHOTO D NAC–NS4 FR
752 DAYS @ 325 F

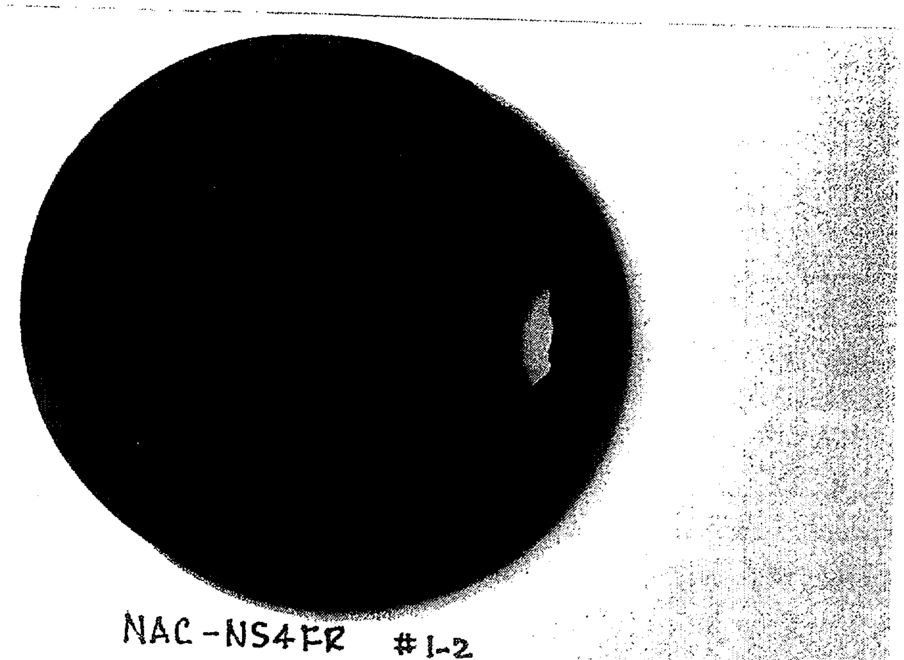


PHOTO E NAC–NS4 FR
752 DAYS @ 325 F

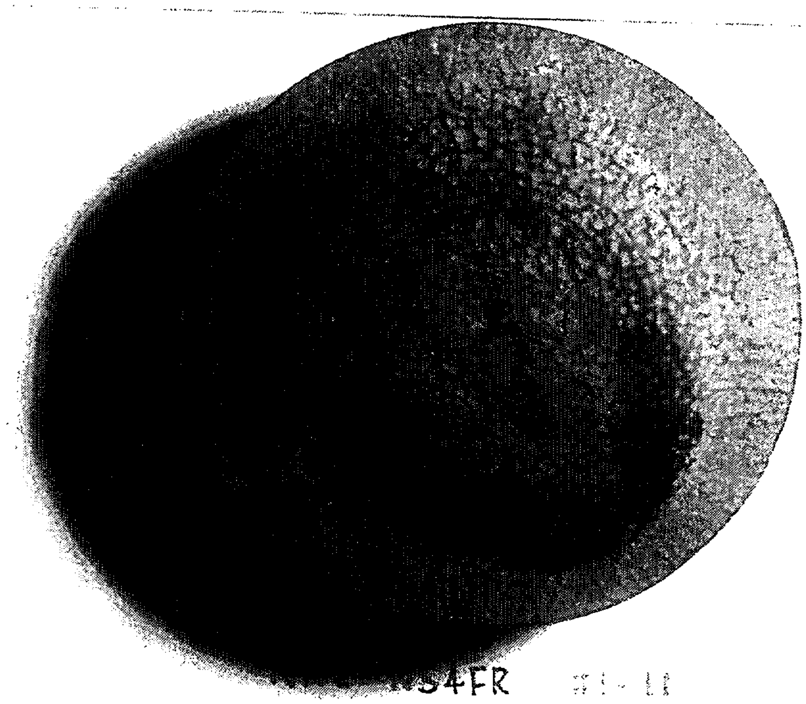


PHOTO F NAC–NS4 FR
752 DAYS @ 325 F

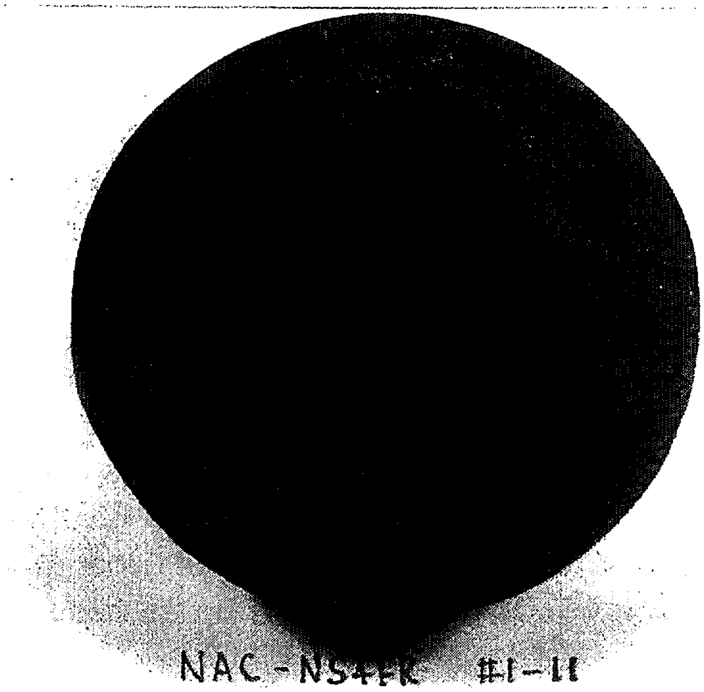
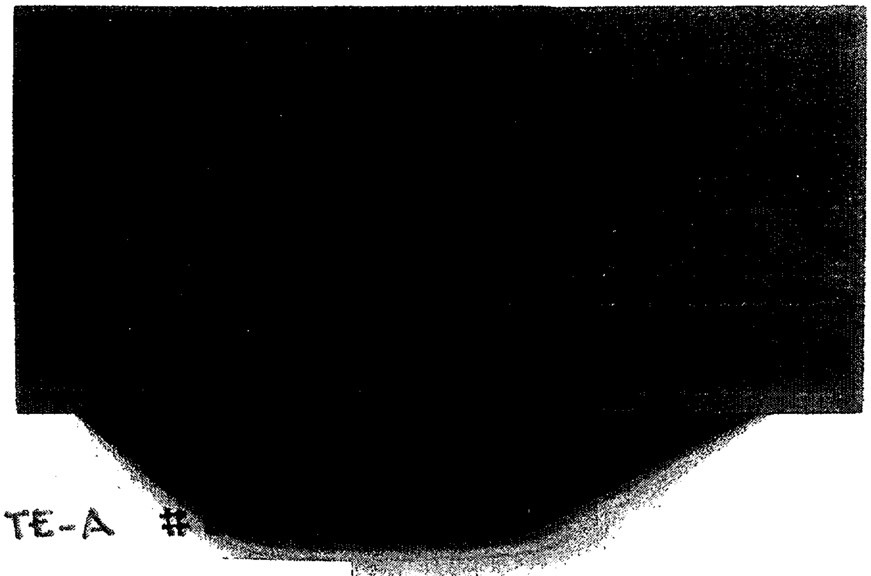


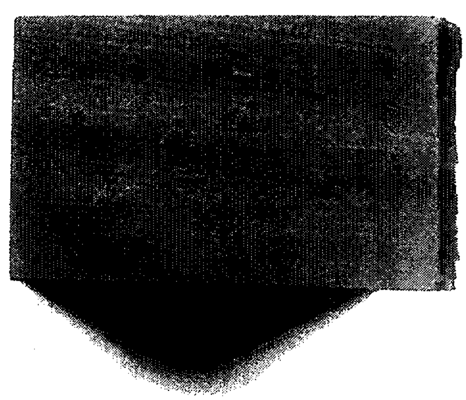
PHOTO G NAC-NS4 FR
752 DAYS @ 325 F

PHOTO H NAC-NS4 FR
752 DAYS @ 325 F



HOLTITE-A #

PHOTO I HOLTITE-A
249 DAYS @ 325 F

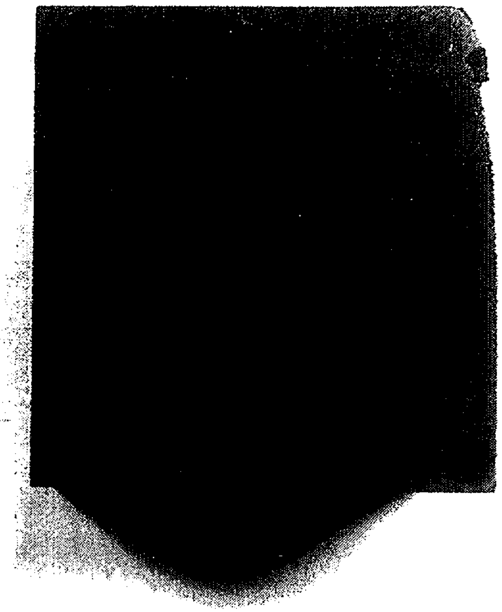


HOLTITE -A # A-3

PHOTO J HOLTITE-A
249 DAYS @ 325 F

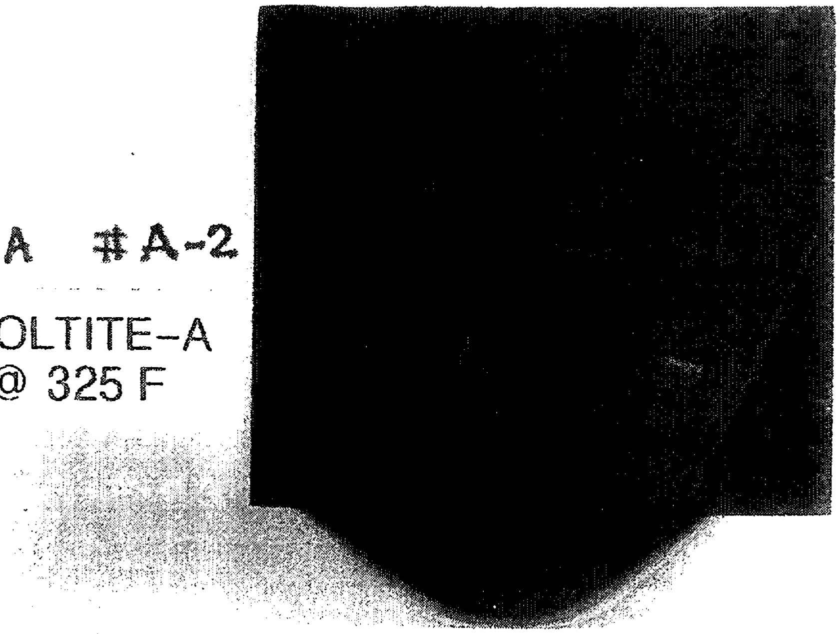
HOLTITE -A # A-3

PHOTO K HOLTITE–A
249 DAYS @ 325 F



HOLTITE -A # A-2

PHOTO L HOLTITE–A
249 DAYS @ 325 F



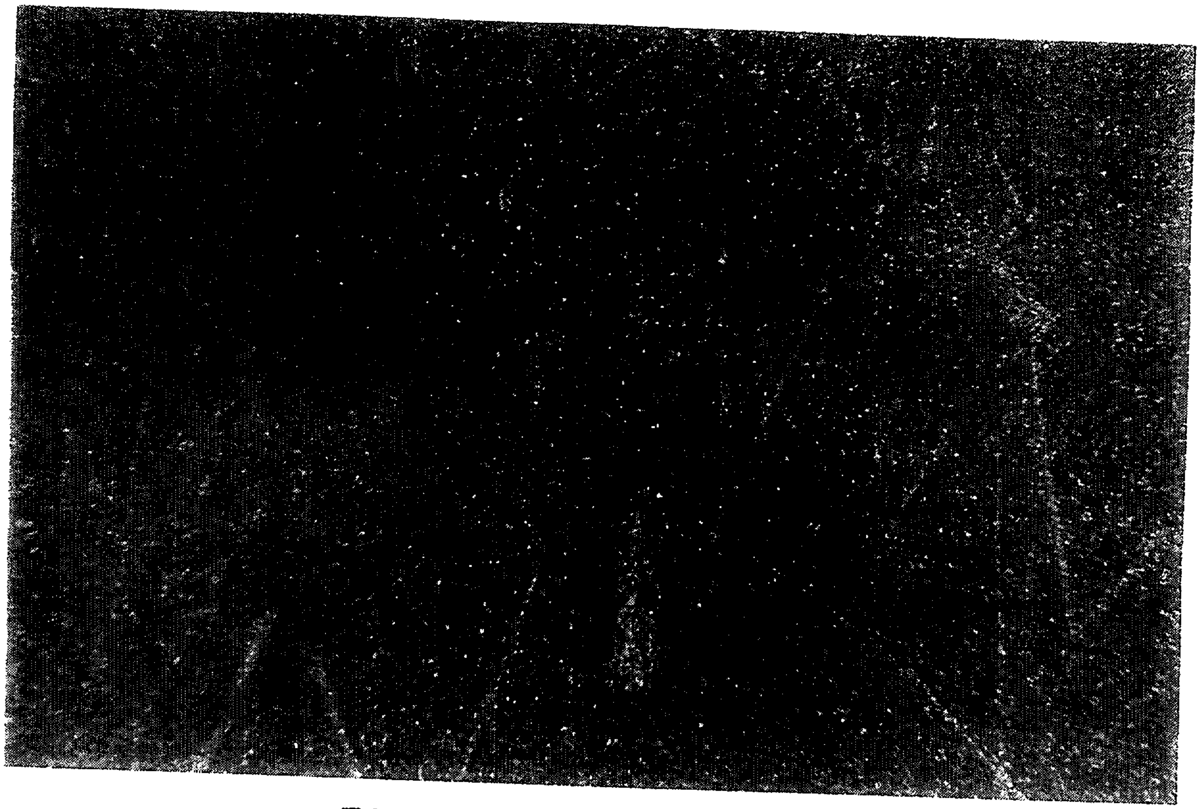
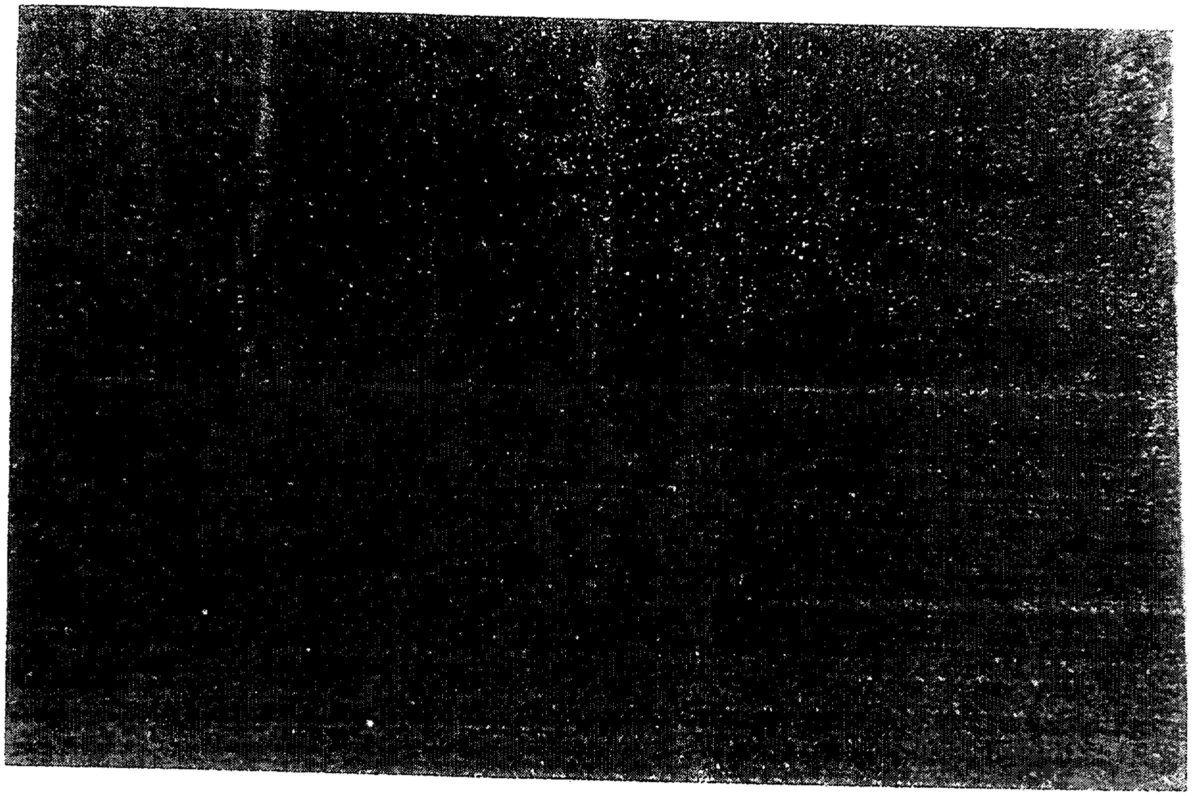


PHOTO M HOLTITE–A
249 DAYS @ 325 F

PHOTO N HOLTITE–A
249 DAYS @ 325 F



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249 DAYS @ 325 F

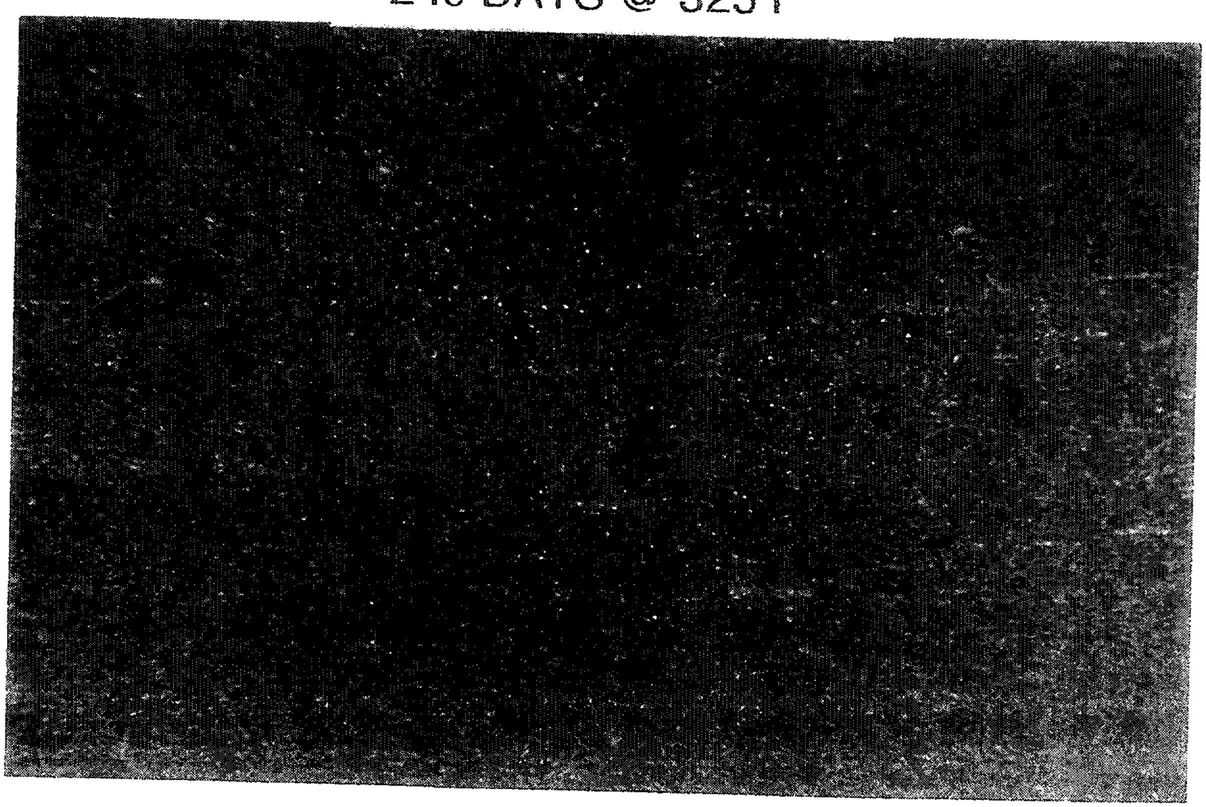

PHOTO P HOLTITE--A
249 DAYS @ 325 F

CHAPTER 7: REFERENCES

[1] "HI-STAR 100 Topical Safety Analysis Report", Holtec Report HI-941184, Rev. 10.

[2] "HI-STAR 100 Safety Analysis Report", Holtec Report HI-951251, Rev. 8.

[3] "Spent Fuel Heat Generation in an Independent Spent Fuel Storage Installation", Reg. Guide 3.54, Rev. 1, (January 1999).

[4] "HI-STAR 100 System Storage & Transport Condition Thermal Evaluation", Holtec Report HI-971826, Rev. 4.

[5] "Experimental Studies on Long-Term Thermal Degradation of Enclosed Neutron Shielding Resin", Asano, Ryoji and Nagao Niomura

[6] Letter Dated 4/30/87 from Mr. Larry Dietrick, Bisco Products, Inc., to Mr. Todd Lesser, of NAC International, on the subject of weight loss of Bisco-NS4FR.

[7] R. Boonstra, "Thermal Testing of Solid Neutron Shielding Materials", Department of Energy sponsored work at General Atomics.

APPENDIX 1

EXCERPTS FROM HI-STAR 100 TSAR, REVISION 10

APPENDIX 1.B: HOLTITE ™ MATERIAL DATA
(Total of 20 Pages Including This Page)

The information provided in this appendix describes the neutron absorber material, Holtite-A (also known commercially as NS-4-FR) for the purpose of confirming its suitability for use as a neutron shield material in spent fuel storage casks.

NS-4-FR contains aluminum hydroxide ($Al(OH)_3$) in an epoxy resin binder. Aluminum hydroxide is also known by the industrial trade name of aluminum tri-hydrate or ATH. ATH is often used commercially as a fire-retardant, hence the "FR" designation in NS-4-FR. NS-4-FR is a generic name for the material which was originally developed by Bisco Inc. and used for many years as a shield material with B_4C or Pb added. NS-4-FR contains approximately 62% ATH supported in a typical 2-part epoxy resin as a binder. Holtite-A, the Holtec International version of NS-4-FR, contains 1% by weight B_4C, a chemically inert material added to enhance the neutron absorption property. Pertinent properties of Holtite-A are listed in Table 1.B.1.

The essential properties of Holtite-A are:

1. the hydrogen density (needed to thermalize neutrons),

2. thermal stability of the hydrogen density, and

3. the uniformity in distribution of B_4C needed to absorb the thermalized neutrons.

ATH and the resin binder contain nearly the same hydrogen density so that the hydrogen density of the mixture is not sensitive to the proportion of ATH and resin in the NS-4-FR mixture. B_4C is added (1% in Holtite-A) as a finely divided powder and does not settle out during the resin curing process. Once the resin is cured (polymerized), the ATH and B_4C are physically retained in the hardened resin. Analysis for B_4C throughout a column of Holtite-A has confirmed (Holtec International qualification tests) that the B_4C is uniformly distributed with no evidence of settling or non-uniformity. Furthermore, an excess of B_4C is specified in Holtite-A as a precaution to assure that the B_4C concentration is always adequate throughout the mixture.

NS-4-FR material has been extensively tested for thermal stability, as indicated in the following documents (copies of these documents are attached).

- Letter dated 4/20/87 from Mr. Larry Dietrick, Bisco Products to Mr. Tod Lesser, NAC International, "Weight loss of NS-4-FR under extreme temperature conditions"

- "Experimental Studies On Long-Term Thermal Degradation of Enclosed Neutron Shielding Resin", Asano, Ryoji and Nagao Niomura

- "Thermal Testing of Solid Neutron Shielding Materials", Boonstra, Richard H.

The specific gravity specified in Table 1.B.1 does not include an allowance for weight loss. The specific gravity specified in Chapter 1 includes a 4% reduction to conservatively account for potential weight loss at the design temperature of 300°F. However, the BISCO letter dated 4/20/87 provides information stating that samples had been exposed to a continuous temperature of 338°F for 146 days and a maximum of 3.15% weight loss had been experienced. Thus, there is a substantial level of conservatism in the Holtec allowance for weight loss.

Tests on the stability of Holtite-A were also performed by Holtec International. Results of these independent tests at 325°F on 15 samples gave an average weight loss in 97 days of 2.41% and a maximum weight loss of 2.72%. The observations are consistent with published tests on NS-4-FR.

The paper entitled "Experimental Studies on Long-Term Thermal Degradation of Enclosed Neutron Shielding Resin " provides information which corroborates the information provided in the BISCO letter dated 4/20/87. The paper suggests that enclosures of the NS-4-FR material can further decrease the percent weight reduction at elevated temperatures. The NS-4-FR is encapsulated in the HI-STAR 100 overpack and therefore should experience a very small weight reduction during the design life of the HI-STAR 100 System. It should be noted that the shielding analysis conservatively assumes 4% loss in density.

The paper entitled "Thermal Testing of Solid Neutron Shielding Materials" provides information regarding NS-4-FR material stability during a fire accident. Results of the study suggests that NS-4-FR could withstand a fire accident with minimal damage. This data is provided for information only, as the post-accident shielding analysis very conservatively assumes complete degradation of the neutron shield and replaces the neutron shield with a void.

The data and test results presented here confirm that

1. Holtite-A with 1% B_4C has the same thermal stability and characteristics as the previously approved NS-4-FR material,

2. The hydrogen density meets or exceeds minimum NS-4-FR specifications (measured at 0.105 gH_2/cc compared to the NS-4-FR specification of 0.096 gH_2/cc), and

3. The B_4C is uniformly distributed, with no evidence of settling or non-uniformity.

Based on the information described above, Holtite-A meets all of the requirements for an acceptable neutron shield material in the manner of NS-4-FR, which was licensed previously in Docket No. 71-9235 (NAC-STC).

Table 1.B.1

PROPERTIES OF HOLTITE-A NEUTRON SHIELD

PHYSICAL PROPERTIES (Reference: NAC International Brochure)	
% ATH	62 maximum (confirmed by Holtec in independent analyses)
Specific Gravity	1.68 g/cc maximum
Thermal Conductivity	0.373 Btu/hr/ft-°F
Max. Continuous Operating Temperature	300°F
Specific Heat†	0.39 Btu/lb-°F
Hydrogen Density	0.096 g/cc minimum (confirmed by Holtec in independent analyses)
Radiation Resistance	Excellent
Ultimate Tensile Strength	4,250 psi
Tensile elongation	0.65%
Ultimate Compression Strength	10,500 psi
Compression Yield Strength	8,780 psi
Compression Modulus	561,000 psi
CHEMICAL PROPERTIES (Nominal)	
wt% Aluminum	21.5 (confirmed by Holtec)
wt% Hydrogen	6.0 (confirmed by Holtec)
wt% Carbon	27.7
wt% Oxygen	42.8
wt% Nitrogen	2.0
wt% B_4C	up to 6.5 (Holtite-A uses 1% B_4C)

† BISCO Products Data from Docket M-55, NAC-STC TSAR.

Experimental Studies on Long-term Thermal Degradation of Enclosed Neutron Shielding Resin

Ryoji ASANO[1], *Nagao NIOMURA*[2]

[1]Hitachi Zosen Corporation, Japan
[2]Ocean Cask Lease Co.,Ltd, Japan

INTRODUCTION

Resins which have high Hydrogen atom content are effective for Neutron shielding and are recently used for neutron shielding material of spent fuel shipping casks. As the resins themselves are easily burned at relatively low temperature, which could be the problem during the fire test condition, mixture of resin and fire retardant which main component is a hydroxide compound is usually used as shielding material. The fire retardant prevents resin from burning by decomposing of the hydroxide compound under fire test condition.

When these resins are used for neutron shielding material of cask, their temperature rises during the transportation by decay heat of spent fuel. Therefore, thermal degradation of resin (hereafter called as "heat weight loss") at the operating temperature should be paid attention.

Furthermore, when the resin is used for neutron shielding material, there are two cases. One is to put it on the outside surface of the cask and the other is to enclose it between two layers. In former case, the heat weight loss occurs in air of which study report can be obtained. On the other hand, the latter is the reaction in the enclosed environment which study report can be seldom obtained. Therefore, the study of the heat weight loss in the enclosed environment was carried out for long term period assuming the operating time of the real cask .

TEST MATERIAL

Test material is NS-4-FR supplied by BISCO CO. LTD, U.S.A. Raw materials are epoxi resin, hardener and fire retardant. They are mixed together and hardened according to the manufacturing manual supplied by BISCO. NS-4-FR is the neutron shielding material which contains about 60% of aluminium hydroxide as fire retardant.

TEST

Tests were carried out in order of basic material test, open test, enclosed test and long term cyclic test which simulates the operation term of cask. The test results are explained as follows.

Basic material test
TG tests which can be performed comparatively easily were carried out in order to study basic thermal characteristics of the test material. The test conditions are as follows.

Condition	Case 1	Case 2
Atmospheric gas	Air&N_2	Air&N_2
Gas Flow Rate(cc/min)	150	200
Temp. Rising Rate(°C/min)	3	10
Max.Temperature(°C)	220	530

Heat weight loss could not be detected in the Case 1. The results of Case 2 are as follows.

(1)The weight loss of the test specimen in nitrogen gas was much smaller than that in air between 300°C and 380°C which were shown in Fig 1. It indicates that the test materials are decomposed and loose its weight by oxygen in air and by heat within the temperature range.

(2)Comparing the results between test material and NS-4- FR without fire retardant, the weight loss of latter is less than that of former until 360°C as shown in Fig. 2. It indicates that the weight loss of former is mainly due to the decomposition of aluminium hydroxide as fire retardant. This result means that the decomposition of aluminium hydroxide is important for the weight loss during low temperature. And it is necessary to select a suitable grade of aluminium hydroxide, as the decomposition temperature depends on the purity and grain size of aluminium hydroxide.

TG test results can not be used directly for long term degradation data because the test specimen was pulverized to very small size, and reaction and diffusion is very rapid, but they can be good reference information.

Open test

Open tests were performed varying the shape of test specimen and temperature to study heat weight loss in air. The results are as follows.

(1) Effect of shape of test specimen
To study the effect of the shape, cubic and cylindrical test specimens with nearly equal weight were tested. It was observed that the heat weight loss of latter which had larger surface area was always larger than that of the former. It indicates that effects of surface oxidation and surface diffusion are important factors for the heat weight loss.

(2) Effect of temperature
The heat weight loss at 125°C, 150°C, 175°C and 200°C are shown in Fig.3 as a function of time. The increase of the heat weight loss is observed in 200°C test after 1000 hr. It is supposed that generation of continues crack inside of the test specimen makes it easy to diffuse the decomposed resin component and water.

Enclosed test

Supposing that the neutron shielding material is filled in the enclosures tests were conducted to study the effects of enclosed condition to the heat weight loss. The tests were performed on test specimen in the sealed stainless steel container with Ar atmosphere.

(1) Sealed stainless steel container
Seal container is shown in Fig. 4, of which lid is welded to seal the cavity of the container perfectly. Enough height of container cavity is provided to avoid the effect of welding heat to the test specimen. Ar gas seal hole is seal-welded and cooled immediately by water after replacing air with Ar gas.

(2) Test Condition
Continuous test and cyclic test of 110 hr heating and 58 hr cooling which simulated the actual operating condition of cask were performed at 125°C, 150°C and 175°C. The test duration was from 8 to 16 weeks.

(3) Test Results
Test results are shown also in Fig.3. Main results are as follows.

(a)Test Results at 125°C
The heat weight loss at 1512 hr continuous test and that at 1760 hr cyclic test were negligible. The heat weight loss at this temperature is regarded as insignificant.

(b)Test Results at 150°C
The heat weight losses at both 1224 hr continuous tests, 990 hr cyclic test and 1760 hr cyclic test were almost 1/3 of that of open test.

(c)Test Results at 175°C
The heat weight losses at both 1600 hr continues test, 1210 hr cyclic test and 1760 hr cyclic test were almost half of that of open test.

(d)Due to the few test specimens and short test period, data scattering was observed in the test results . However, heat weight loss of enclosed test is clearly less than that of open test except those at 125°C when no heat weight loss was observed.

Long term cyclic test at 150°C
In order to avoid scatter in test results and to estimate heat weight loss during long term use of cask, long term cyclic tests at 150°C were conducted, where temperature supposed was the maximum working temperature of neutron shielding material during transportation. The results are shown in Fig. 5. Total test specimens were 18 and maximum test period was 56 weeks. One cycle is composed of 110 hr heating and 58 hr cooling which is same as the enclosed test above. The heat weight loss for 56 weeks was about 1.1%.

DISCUSSION

The relation between heat weight loss W(%) and test period D(day) is given from Fig. 5, as follow.

$$W=100.63-0.218 \times \log D$$

Using this equation the heat weight loss for 20 years W_{20}(%) can be estimated as follow.

$$W_{20}=100.63-0.218 \times \log(20 \times 365)$$
$$=1.87(\%)$$

From the calculation above it is enough to have the cask design margins of 2.0% heat weight loss even if the scattering in the test results are taken into account.

Water drops were observed on inside surface of sealed container when the lid was cut off to open and to take out the test specimen from the container after test. It is considered that these drops prevented the temperature rise of test specimen by evaporating during the test and reduced the heat weight loss of the specimen.

From the results, it is concluded that NS-4-FR is effective as neutron shielding material of cask, especially when it is used in enclosed condition.

BISCO (150°C HEATED)
TG <Sample> <Comment> <Temp.program(C) (C/min) (min)>
10°C/min 1* 20.0-500.0 10.00 0.00
9.552mg --------------- <Gas>
<Date> (9.552mg) --------------- N_2 AND AIR 150.0ml/min
90/04/10 16:51 <Reference> --------------- --------------- 0.0ml/m!
--------------- ---------------

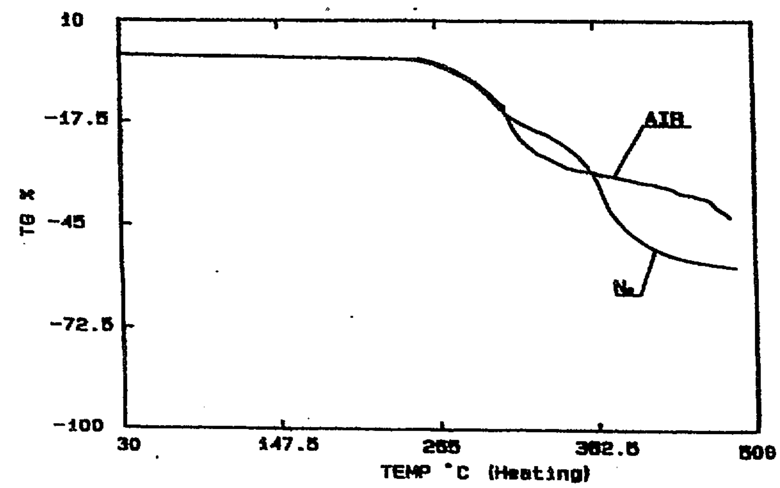

Fig 1 TG OF NS-4-FR IN AIR AND NITROGEN

BISCO
TG <Sample> <Comment> <Temp.program(C) (C/min) (min)>
RESIN AND NS-4-FR --------------- 1* 25.0-500.0 10.00 0.00
7.985mg --------------- <Gas>
<Date> (7.985mg) --------------- N_2 200.0ml/min
90/04/26 14:00 <Reference> --------------- --------------- 0.0ml/min
A1203 ---------------

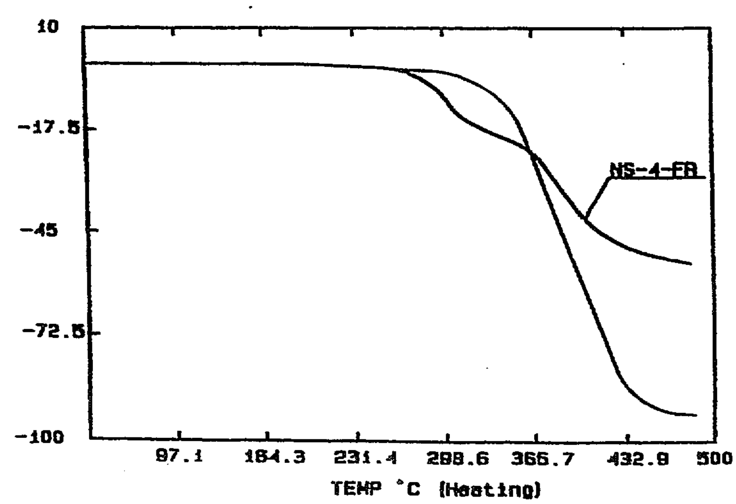

Fig 2 TG OF NS-4-FR W/O FIRE RETARDANT

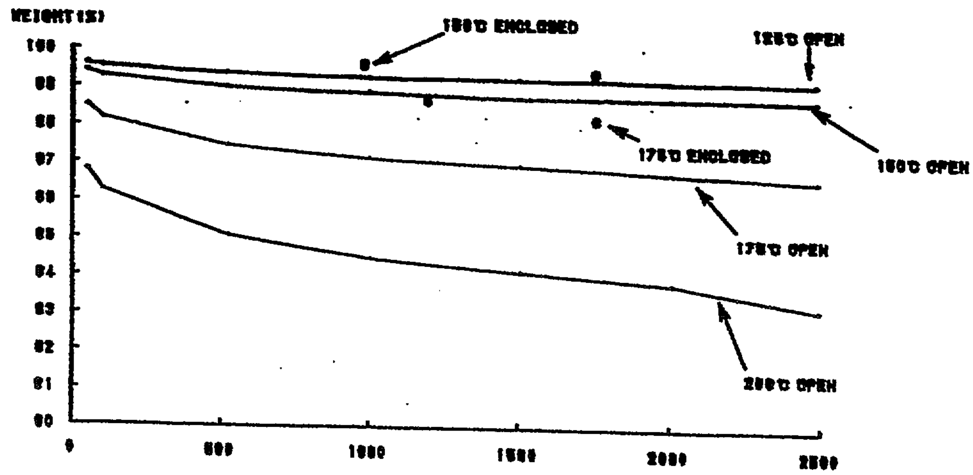

Fig.3 WEIGHT LOSS BY OPEN AND ENCLOSED TEST

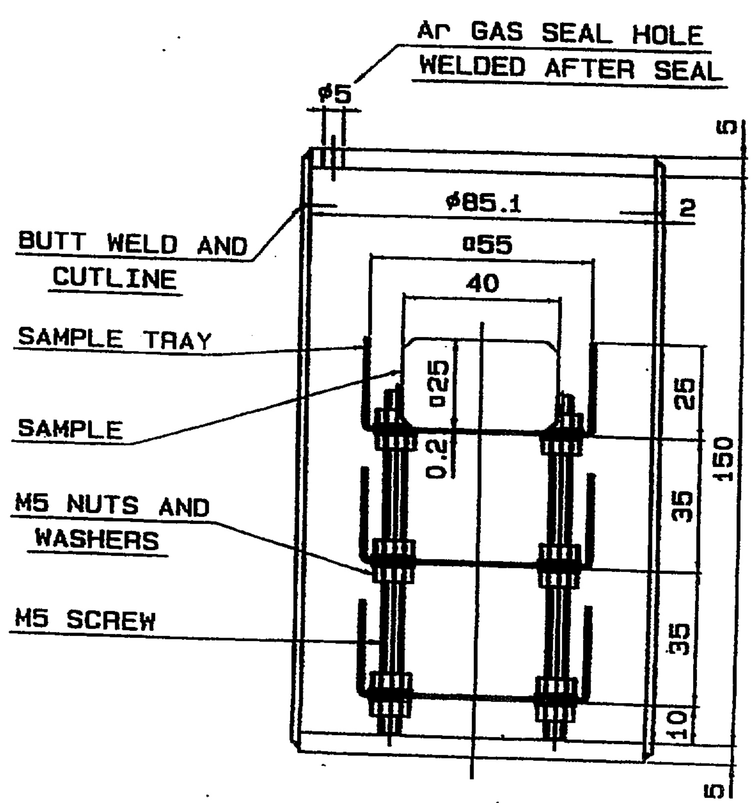

Fig 4 SEALED STAINLESS STEEL CONTAINER

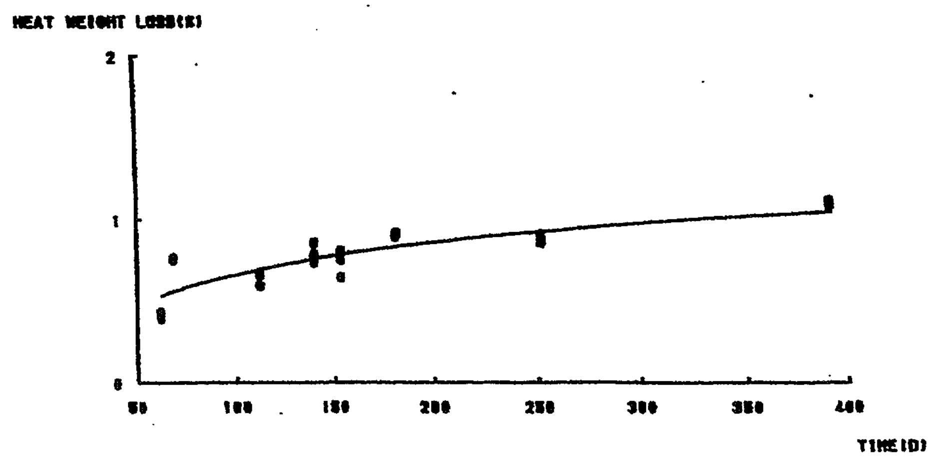

Fig.5 WEIGHT LOSS OF LONG TERM CYCLIC TEST

Thermal Testing of Solid Neutron Shielding Materials*

Richard H. Boonstra

General Atomics, San Diego, California

INTRODUCTION

General Atomics (GA) is currently developing two legal-weight truck casks for the Department of Energy (DOE), Office of Civilian Radioactive Waste Management (OCRWM). These casks, the GA-4 and GA-9, will carry four PWR and nine BWR spent fuel assemblies, respectively. Each cask has a solid neutron shielding material separating the steel body and the outer steel skin. In the thermal accident specified by NRC regulations in 10CFR Part 71 the cask is subjected to an 800°C environment for 30 minutes. The neutron shield need not perform any shielding function during or after the thermal accident, but its behavior must not compromise the ability of the cask to contain the radioactive contents.

In May-June 1989 the first series of full-scale thermal tests was performed on three shielding materials: Bisco Products NS-4-FR, and Reactor Experiments RX-201 and RX-207. The tests are described in *Thermal Testing of Solid Neutron Shielding Materials*, GA-A19897, R. H. Boonstra, General Atomics (1990), and demonstrated the acceptability of these materials in a thermal accident. Subsequent design changes to the cask rendered these materials unattractive in terms of weight or adequate service temperature margin. For the second test series a material specification was developed for a polypropylene based neutron shield with a softening point of at least 280°F. Table 1 lists the neutron shield materials tested. The Envirotech and Bisco materials are not polypropylene, but were tested as potential backup materials in the event that a satisfactory polypropylene could not be found.

TESTING SETUP AND PROCEDURE

Setup

Figure 1 shows a representative test article and thermocouple positions. Each test article consists of blocks of neutron shield contained in a box of dimensions 36 in. x 36 in. x 4.5 in. The box is 11-gage type 304 stainless steel with continuous welds along all seams and a 6-in. x 12-in. hole in the center of a 36-in. x 36-in. face. The hole simulates damage from the hypothetical drop and puncture events and extends 1.5 in. into the material. Six inches of mineral fiber insulation surround the box except for the face with the hole.

Six KNBS (Chromel-Alumel) 20-gage thermocouples (TCs) measure temperatures on the test article. The environment temperatures are determined using five TCs of the same type as on the test article, but shielded to prevent radiant heat loss to the test article surface. These TCs are positioned 6 in. away from the test article surface.

* Work supported by the U.S. Department of Energy, Office of Civilian Radioactive Waste Management, under DOE Field Office, Idaho, Contract DE-AC07-88ID12698.

As shown in Fig. 2, a vertical exposure furnace heats the uninsulated face of the test article. The back wall of the furnace contains a bank of seven natural gas burners. A single globe valv regulates the overall gas pressure to control the test exposure to the temperature range of 800° to 900°C.

TABLE 1 NEUTRON SHIELD MATERIALS IN THERMAL TESTS	
Supplier	**Material**
Kobe Steel, Ltd.	PP-R01 polypropylene, 1% boron
Envirotech Molded Products, Inc.	High-density polyethylene (HDPE), 0.8% boron
Bisco Products, Inc.	Modified NS-4, 4.5% boron
Reactor Experiments, Inc.	High melt index polypropylene (HMPP), 1% boron

Procedure

The completed test article is conditioned at room temperature for at least 24 hr, then moved position directly in front of the furnace. Recording of the TC data begins 5 minutes prior to ignition of the burners. After ignition, the average furnace temperature (i.e., average of the fi furnace TCs) is maintained between 800° and 900°C for 30 minutes. The burners are then off and the test article is pulled away to cool in ambient air. When all temperatures have pea the test article is again conditioned at room temperature for at least 24 hr and then disassem for inspection.

Material Acceptance Criterion

The neutron shield must not provide a source of thermal input to the cask sufficient to degra containment integrity. More specifically, the material is acceptable if (1) temperatures on the back surface do not at any time exceed the maximum temperature of the thermal accident environment and (2) it shows no evidence of prolonged combustion (i.e., combustion lasting period of several hours) following the thermal accident.

OBSERVATIONS AND RESULTS

Two of the materials, the modified NS-4 and the HMPP, passed the test and are acceptable use as the neutron shielding. The results for the PP-R01 and HDPE materials are inconclusiv because the tests were stopped prematurely due to intense smoke combined with inadequat ventilation. Table 2 provides time-related observations for the four tests. Post-test inspecti results and temperature plots are discussed below.

Kobe PP-R01

Disassembly of the test article after cooling indicated that despite the observed combustion relatively small amount of material had been lost. The polypropylene was heavily charred a broken into loose tiles on the front surface. Some individual blocks had fused together but contrary to expectations, there was little evidence of extensive melting. The total weight l was 36.3 lb out of an initial 200.1 lb, or 18%.

Figure 3 gives the average environment temperature and the thermal response on the back surface of the test article, where the peak temperature is about 340°C. The responses ex abrupt increases within the first several minutes, a characteristic of all the tests. All temperatures on the back surface meet the acceptance criterion that they not exceed the accident environment temperature, but acceptability of the material cannot be established a the test was halted.

Envirotech High-Density Polyethylene (HDPE)

The test article was opened to reveal that most of the material remained in the box. Charring was concentrated mostly on material near the hole and in the lower region beneath the hole. Here molten material had collected and resolidified as indicated by a layer of dark material. In the upper portion, above the hole, charring was confined mainly to the surface and virgin, white material in the form of individual blocks was still identifiable. There was very little loose material. The total weight loss was 27.0 lb from an initial weight of 199.6 lb, or about 14%.

The average environment temperature and back surface TC responses are shown in Fig. 4. The responses display the sudden rises seen in the preceding test, although the peak response is less than 150°C. In this respect the HDPE performed better than the PP-R01 and its temperatures also meet the acceptance criterion. However results are indecisive due to termination of the test.

Bisco Modified NS-4

This material is similar to the NS-4-FR tested in 1989 but has a slightly lower hydrogen content and a lower weight. In this test the heating phase successfully proceeded the full 30 minutes, and the material was deemed acceptable.

A post-test inspection indicated immediately that the majority of the material had been retained. This was confirmed by a weight loss of only 6%, 14.5 lb out of 244.6 lb. There was a fairly uniform, black char layer about 1/8 to 3/16 in. thick on the front surface. With the exception of this char layer, which was fragile and separated easily from the remainder of the material, all blocks appeared nearly intact. An average of 2-1/16 in. of undamaged material remained in the front layer of blocks. Some charring was also observed along the sides of blocks that joined at the locations of TCs 1 and 2 and TCs 4 and 5. The back layer of blocks revealed localized discoloration near the top comers and a black, oily material that had condensed on the block surfaces. Apart from this, very little damage was noticed on these blocks.

Figure 5 gives the average environment and back surface temperatures The maximum back surface response is just less than 100°C. The initial peaking of TCs 4 and 6 before 50 minutes is followed by a gradual increase of these temperatures with subsequent maximums between 400 and 500 minutes. The later gradual increase is obviously due to conduction of heat through the material to the back surface; the initial peaks are therefore due to some other phenomenon.

Reactor Experiments High-Melt Index Polypropylene (HMPP)

This material also went through the full 30 minutes of heating and post-test cooldown. It was subsequently judged acceptable for use in the cask.

After complete cooling in ambient air, the total material weight loss was determined to be 109 lb from the initial 194 lb, some 56%. Disassembly of the test article confirmed a significant absence of material. No obvious char layer was noted. The space above the bottom edge of the hole was empty except for a column of partially melted blocks along the left side of the box. In the lower section, beneath the bottom edge of the hole, the material had softened and fused into a solid mass, although gaps between individual blocks could still be seen. The thickness of the fused material at the center of its top edge was approximately 5 in., indicating that the front steel had bowed outward 0.5 in. and allowed additional material to flow down from the top portion. Melted and resolidified material could be seen as distinct layers on the exterior surfaces of the fused, bottom portion.

Temperatures of the average environment and on the back side of the test article are shown in Fig. 6. The maximum back face temperature, 211°C, occurs in TC 6 at about 6.5 minutes. The sudden increase in back face temperatures at the beginning of the test is again noted, particularly in TCs 4 and 6, which were located in the bottom portion of the test article.

CONCLUSIONS

The Bisco modified NS-4 and Reactor Experiments HMPP are both acceptable materials from a thermal accident standpoint for use in the shipping cask. Tests of the Kobe PP-R01 and Envirotech HDPE were stopped for safety reasons, due to inability to deal with the heavy smoke, before completion of the 30-minute heating phase. However these materials may prove satisfactory if they could undergo the complete heating.

Table 3 compares key results for all four materials. The Bisco modified NS-4 is best in terms of survivability and back side temperatures. Despite the more intense combustion of the HMPP during the 30-minute heating phase, the test article did not sustain this combustion when moved away from the furnace. Had the molten material not become entrapped on the test article insulation (which is not a part of the actual cask), the combustion would have expired much earlier. The back side maximum temperature of 211°C is well within the criterion of 800°C. This material therefore is also acceptable. It is preferable to the Bisco modified NS-4 since it is about 20% lighter and at the same time has a hydrogen density 20% higher.

The tests of the PP-R01 and HDPE materials were terminated for safety due to the extremely heavy smoke in the indoor test facility. The combustion of the materials without any external heat input led to the initial belief that they were unacceptable. However the HMPP test showed the same phenomenon, and combustion in the test article eventually ceased when it was moved from the furnace. This fact led to the conclusion that placing the test article against the well-insulated furnace formed an effective heat-retaining environment that kept the temperature high enough to support combustion. Neutron shield material became sufficient fuel in place of the gas burners. Moving the test article to cool in ambient air disrupted this process and the combustion ended. It is thus plausible that the PP-R01 and HDPE would behave like the HMPP and prove acceptable if these tests could proceed the full duration. Note that backside temperatures (see Figs. 3(b) and 4(b)) remained well below the acceptance criterion of 800°C.

TABLE 3
COMPARISON OF NEUTRON SHIELD THERMAL PERFORMANCE

Material	Time to End of Flames (min.)	Weight Loss (%)	Peak back side temps (°C)			
			TC 1	TC 3	TC 4	TC 6
PP-R01	Test terminated	18	341	169	328	336
HDPE	Test terminated	14	83	140	146	78
NS-4	5	6	96	41	47	42
HMPP	11.5 (1), 50 (2)	56	127	203	152	211

(1) Material inside test article
(2) Material outside test article entrapped on insulation

ACKNOWLEDGMENT

The author would like to express his appreciation to Omega Point Laboratories (San Antonio, TX) for performing the tests.

REFERENCES

Boonstra, R.H., *Thermal Testing of Solid Neutron Shielding Materials*, GA-A19897, General Atomics (1990).

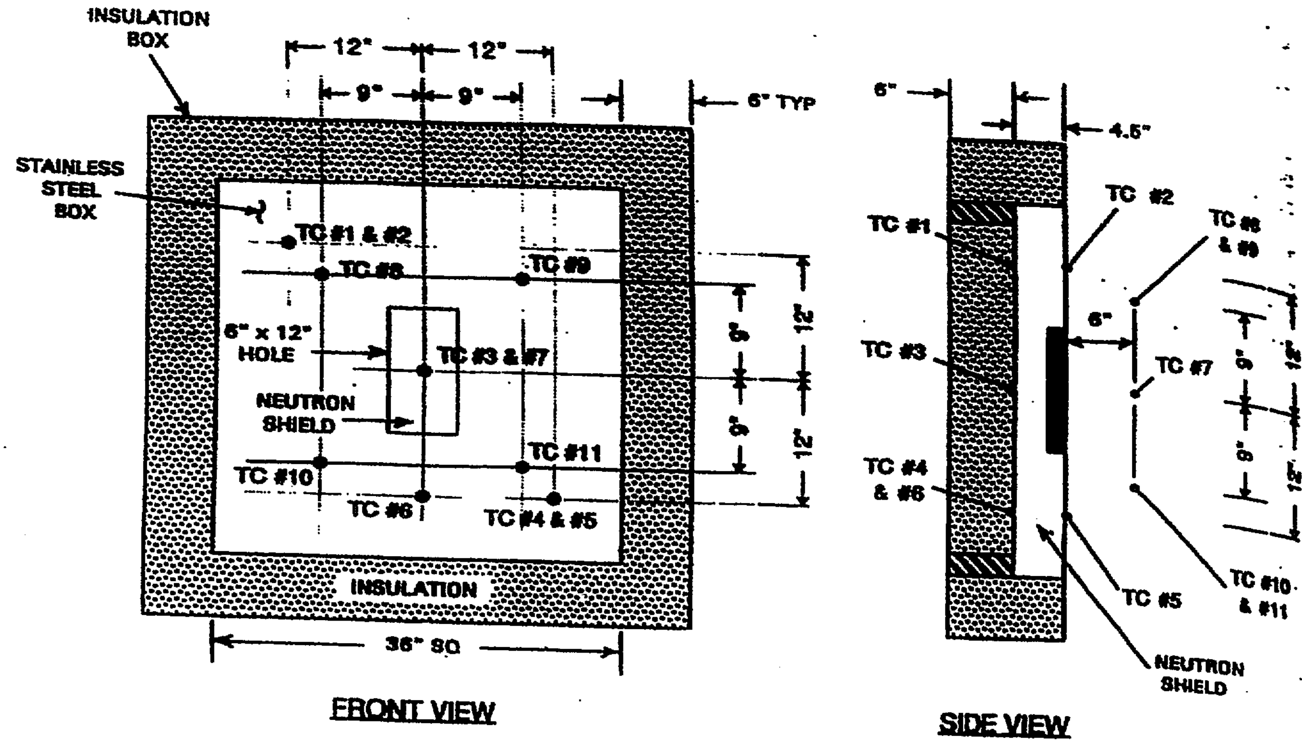

Fig. 1 Neutron shield test article with thermocouple locations

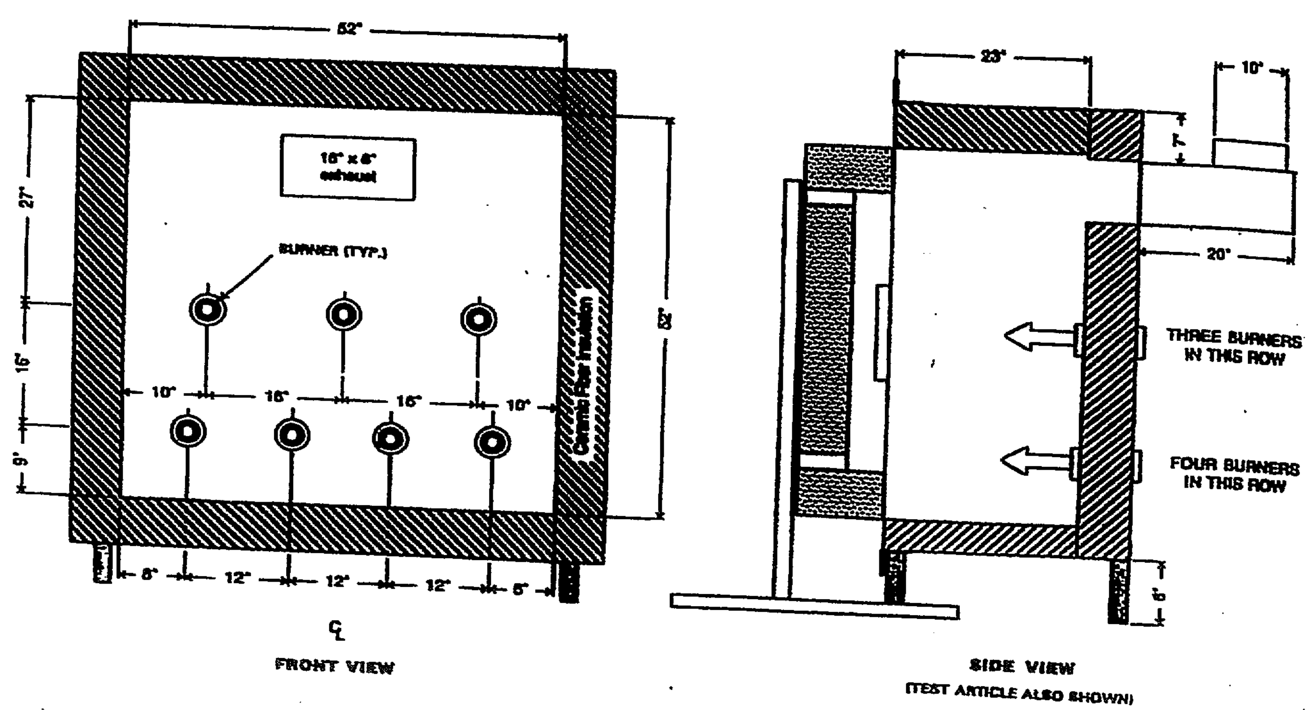

Fig. 2 Neutron shield test furnace

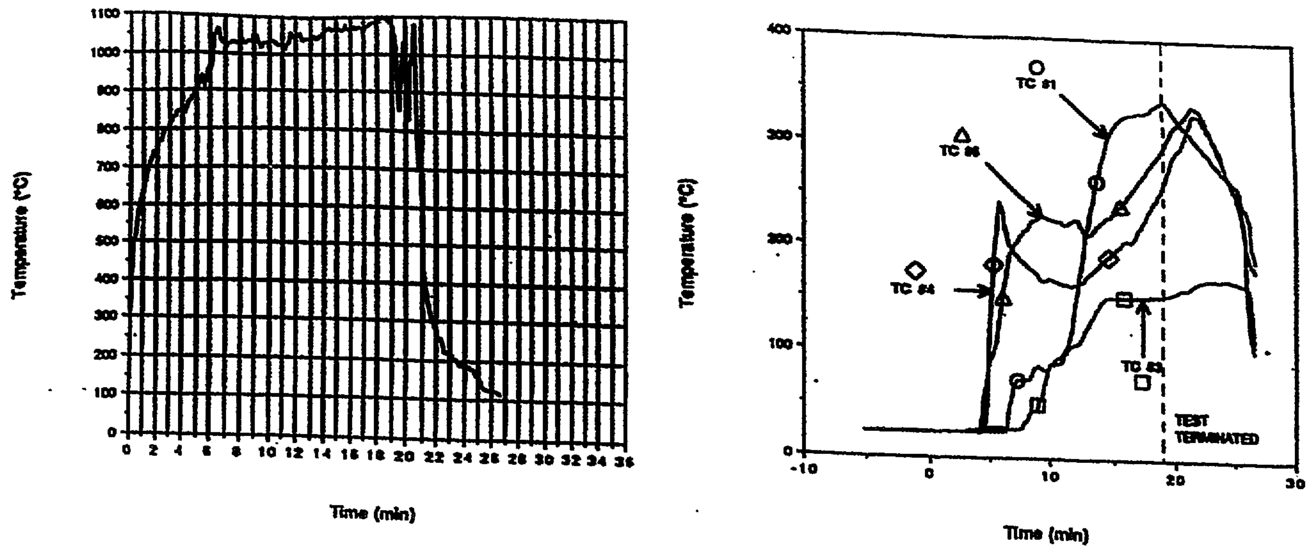

Fig. 3   **Kobe PP-R01 test temperatures: (a) average environment, (b) back side response.**

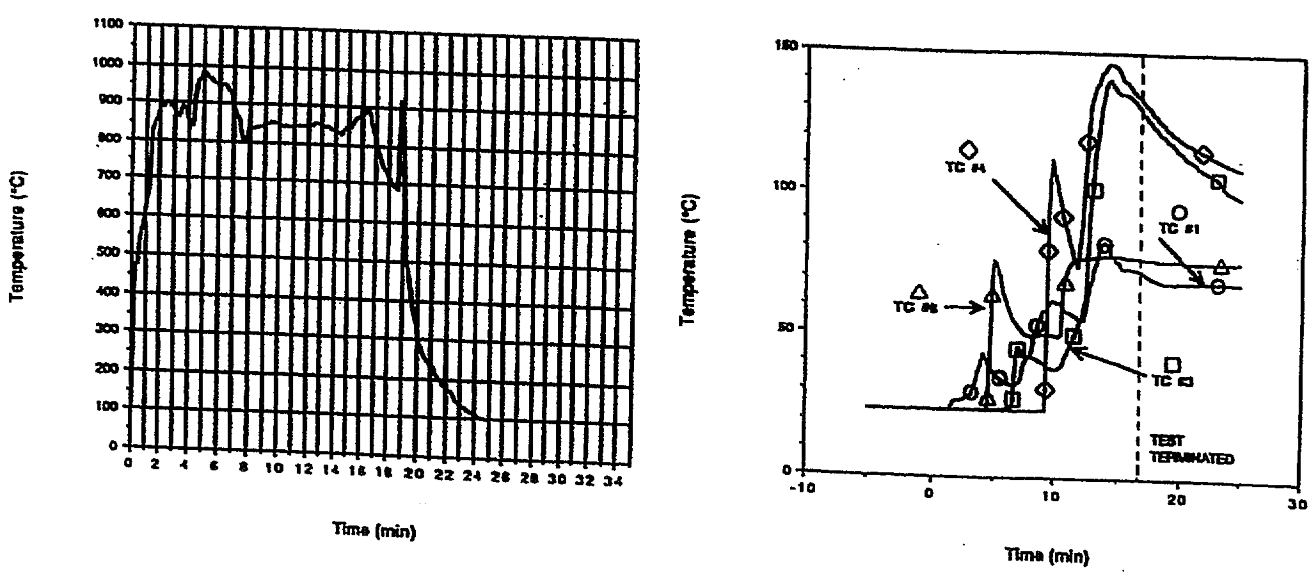

Fig. 4   **Envirotech HDPE test temperatures: (a) average environment, (b) back side response.**

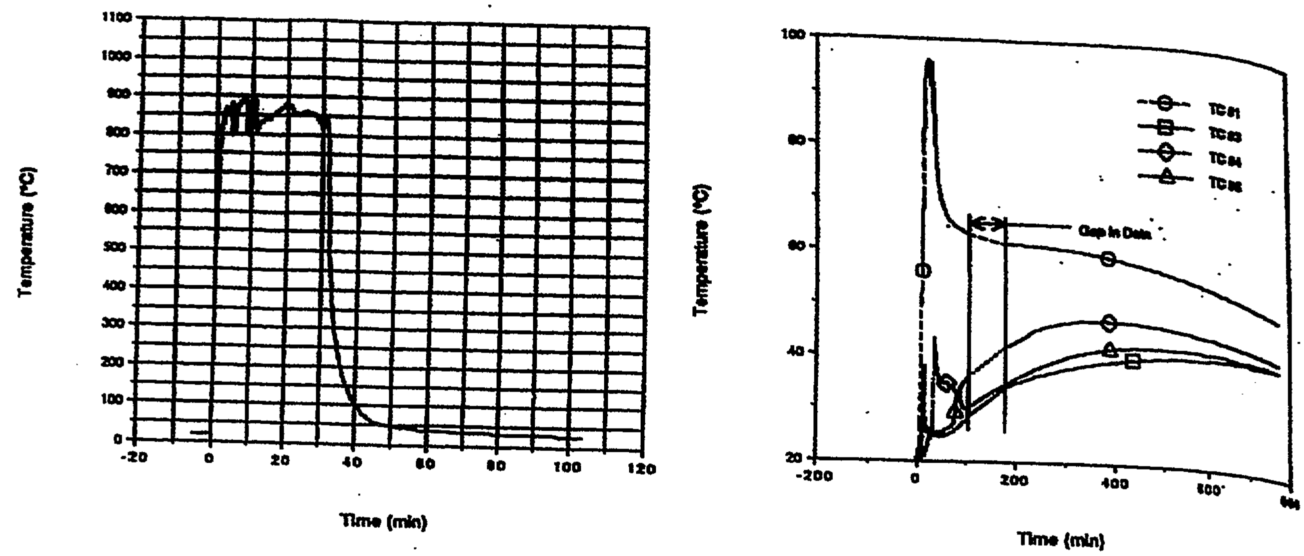

Fig. 5 **Bisco Modified NS-4 test temperatures: (a) average environment, (b) back side response.**

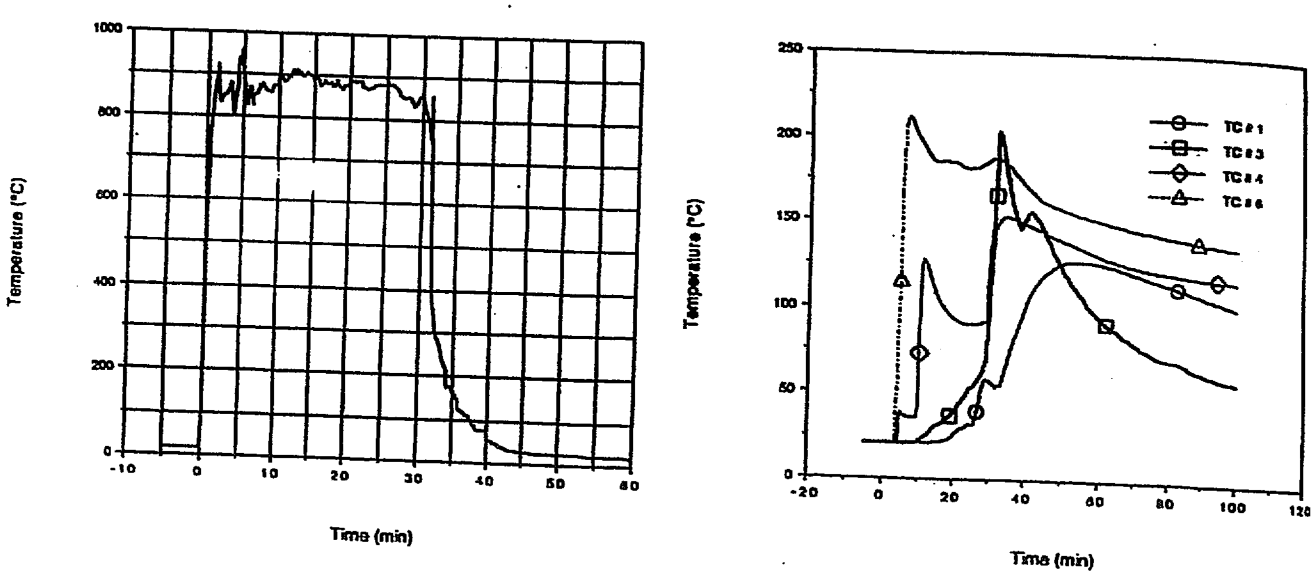

Fig. 6 **Reactor Experiments HMPP test temperatures: (a) average environment, (b) back s[illegible] response.**

TABLE 2
THERMAL TEST OBSERVATIONS

PP-R01 polypropylene, 1% boron	High-density polyethylene (HDPE), 0.8% boron	Modified NS-4, 4.5% boron	High melt index Polypropylene (HMPP), 1% boron
3 min — Temp. = 800°C, slight smoking and flame from exhaust stack. 5.5 min — Temp. > 900°C, smoke and flames fill furnace interior. Furnace burners turned down, then shut off. 6.5 min — Temp. > 1000°C, extremely heavy smoke. 18.5 min — Temp. = 1100°C, decision made to terminate test. 19 min — Two failed attempts to extinguish flames with CO_2. Test article moved from furnace and flames extinguished with water.	2 min — Temp. = 800°C 2.5 min — material ignited 4.5 min — Temp. > 900°C, material flowing out of box front with vigorous flaming and smoking from exhaust port. Furnace burners turned down, then shut off. 5 min — Temp. ≈ 1000°C, exhaust port damping reduced temp. to within test range. 17 min — Test terminated due to copious smoke production, flames visible along bottom of test article. Test article moved from furnace. Flames observed inside furnace indicating material had been discharged into furnace. Flames extinguished with water.	Additional ventilating capability supplied. Environment achieved desired test range and maintained there for 30 min without difficulty. Some smoke was observed and some flames could be seen from the exhaust port, but intensity of combustion was less than in previous tests. 30 min — Test article moved from furnace. Flames approximately 4 ft high issue from hole in test article. After about 5 min, the flames diminish and self extinguish. White smoke persists another 3.5 min.	3 min — smoke begins issuing from exhaust port. 5 - 6 min — 3-ft column of flame appears, burners adjusted to control temp to specified range. 17 min — Furnace burners shut off. Internal combustion maintains temp. in test range. Molten material observed flowing into water-filled catch pan. 30 min — Test article moved from furnace. Flames issuing from hole and from material that had flowed out of the hole and become trapped in the insulation. Flames from hole gradually diminish and self extinguish after 12 min. Flames from material in insulation self extinguish after another 38 min.

bisco

bisco products, inc.
[illegible]
park ridge, illinois [illegible]
[illegible]
telex [illegible]

April 20, 1987

Mr. Todd Lesser
Nuclear Assurance Corporation
5720 Peachtree Plaza
Norcross, GA 30092

SUBJECT: WEIGHT LOSS OF NS-4-FR UNDER EXTREME TEMPERATURE CONDITIONS.

Dear Todd,

As a follow-up to our telephone conversation last week, this letter will confirm the results of the temperature testing that has been performed on NS-4 FR to date.

TEST 1: Weight loss of NS-4-FR at -70°F:

A sample of NS-4-FR was weighed at room temperature, and was then exposed to a low temperature of -170° F for a period of time sufficient to bring the entire sample to a temperature below -70° F. The sample was then weighed and allowed to gradually return to room temperature. A final weight measurement was then taken after the sample had reached room temperature. The beginning and ending weights were identical. The weight at -170° F was slightly higher than the initial weight, probably due to condensation.

CONCLUSION: Since there is no weight loss when exposed to temperatures below -70 F, it can be concluded that there is also no hydrogen loss in the NS-4-FR at that temperature.

TEST 2: Weight loss of NS-4-FR at 338o F (170o C):

This is an update of the thermal aging test begun on November 20, 1985. As of April 17, 1987, the two NS-4-FR samples have been exposed to a continuous temperature of 338oF for 146 straight days. The samples have been periodically pulled out and weighed.

CONCLUSION: The cumulative weight loss as of April 17, 1987 is 3.05% and 3.16% for the two bricks. This test will be continued until such time that the additional weight loss is zero or negligible.

Please let me know if you require any additional information regarding BISCO NS-4-FR material.

Very truly yours,

Larry J. Dietrick
Project Engineer
LJD/hf

APPENDIX 2

HOLTEC PURCHASE ORDER NO. 7071MI DATED 1/26/98
TO NAC INTERNATIONAL
AND NAC'S CLARIFICATION LETTER DATED JANUARY 28, 1998

M³

HOLTEC INTERNATIONAL
HOLTEC CENTER
555 LINCOLN DRIVE WEST
MARLTON, NJ 08053
PHONE 609-797-0900
FASX 609-797-0909

PURCHASE ORDER

Purchase Order Number: 7071MI

Purchase Order Date: 01/26/98

Page: 1

To: NAC INTERNATIONAL
655 ENGINEERING DRIVE
NORCROSS, GA.
30092

Ship To.: HOLTEC INTERNATIONAL
230 NORMANDY CIRCLE
PALM HARBOR, FL 34683

Ship Via..: UPS
Receive By: 02/13/98
Terms.....: NET 30 DAYS
F.O.B.....: PLANT

Confirm To:
Buyer.....: MARK SOLER
Phone.....: 609-797-0900
Vendor....: NAC

Item ID	Description	Unit	Quantity	Unit Price	Total Price
	NA		1.00	0.0000000	0.00

THIS PURCHASE ORDER CAN ONLY BE AMENDED THROUGH A DULY EXECUTED CHANGE ORDER. PROVISIONS OF THE LATEST CHANGE ORDER SHALL GOVERN.

THE ATTACHED REFERENCES, DRAWINGS, ATTACHMENTS, ETC. AS APPLICABLE, CALLED OUT IN THIS PURCHASE/CHANGE ORDER TOGETHER CONSTITUTE THIS PURCHASE CONTRACT. IN CASE OF DISCREPANCY BETWEEN THE VARIOUS DOCUMENTS THE VENDOR [illegible] RESOLUTION FROM HOLTEC'S PROJECT MANAGER BEFORE PROCEEDING.

THE COPY OF THIS PURCHASE DOCUMENT MUST BE ACKNOWLEDGED AND RETURNED WITHIN (10) DAYS OF ISSUANCE TO THE VENDOR TO VALIDATE THIS CONTRACT. FAILURE TO RETURN THE COPY MAY LEAD TO REJECTION OF VENDOR'S INVOICE BY HOLTEC'S ACCOUNTING DEPARTMENT.

HOLTEC'S PROJECT MANAGER IS THE SOLE COMMERCIAL, CONTRACTUAL AND TECHNICAL CONTACT FOR THE VENDOR.

VENDOR'S INVOICE ON PURCHASE/CHANGE ORDERS MUST BE SUBMITTED IN DUPLICATE TO

HOLTEC INTERNATIONAL
HOLTEC CENTER
555 LINCOLN DRIVE WEST
MARLTON, NJ 08053
PHONE 609-797-0900
FASX 609-797-0909

PURCHASE ORDER

Purchase Order Number: 7071MI

Purchase Order Date: 01/26/98

Page: 2

To: NAC INTERNATIONAL
655 ENGINEERING DRIVE
NORCROSS, GA.
30092

Ship To.: HOLTEC INTERNATIONAL
230 NORMANDY CIRCLE
PALM HARBOR, FL 34683

Ship Via..: UPS
Receive By: 02/13/98
Terms.....: NET 30 DAYS
F.O.B.....: PLANT

Confirm To:
Buyer.....: MARK SOLER
Phone.....: 609-797-0900
Vendor....: NAC

Item ID	Description	Unit	Quantity	Unit Price	Total Price

HOLTEC. VENDOR'S INVOICE MUST IDENTIFY ITEM I.D., ITEM, DESCRIPTION, QUANTITY, AND UNIT PRICE PRECISELY IN THE MANNER OF THE PURCHASE ORDER TO RECEIVE PROMPT PAYMENT. DISCREPANT DESCRIPTIONS WILL DELAY INVOICE PROCESSING. WHERE APPLICABLE, INVOICES FOR CHANGE ORDERS MUST INCLUDE THE SAME INFORMATION.

ALL PURCHASE/CHANGE ORDERS REQUIRE THE AUTHORIZED SIGNATURE BY A HOLTEC CORPORATE OFFICER, TO BE CONSIDERED BINDING CONTRACTUAL INSTRUMENT.

SAFETY RELATED PURCHASE/CHANGE ORDERS REQUIRE QA CONCURRENCE AND SIGNATURE.

V. Gupta 1-26-98
QUALITY ASSURANCE MANAGER

Mark Soler 1/26/98
PROJECT MANAGER

SELLER'S ACCEPTANCE

[illegible]: 0.00
[illegible]: 0.00

Authorized Signature:

HOLTEC INTERNATIONAL
HOLTEC CENTER
555 LINCOLN DRIVE WEST
MARLTON, NJ 08053
PHONE 609-797-0900
FASX 609-797-0909

PURCHASE ORDER

Purchase Order Number: 7071MI

Purchase Order Date: 01/26/98

To: NAC INTERNATIONAL
655 ENGINEERING DRIVE
NORCROSS, GA.
30092

Ship To.: HOLTEC INTERNATIONAL
230 NORMANDY CIRCLE
PALM HARBOR, FL 34683

Ship Via..: UPS
Receive By: 02/13/98
Terms.....: NET 30 DAYS
F.O.B.....: PLANT

Confirm To:
Buyer.....: MARK SOLER
Phone.....: 609-797-0900
Vendor....: NAC

Item ID	Description	Unit	Quantity	Unit Price	Total Price
1	NS-4-FR SAMPLES		1.00	1500.0000000	1500.00

THE FOLLOWING ATTACHMENTS ARE APPLICABLE ON THIS ORDER:

ATTACHMENT A: ITEM DESCRIPTION AND MISCELLANEOUS REQUIREMENTS
ATTACHMENT B: QA REQUIREMENTS

ORDER CATEGORY: SAFETY RELATED

PROJECT: 70718

Subtotal: 1500.00
Total...: 1500.00

Authorized Signature:

HOLTEC
INTERNATIONAL

Holtec Center, 555 Lincoln Drive West, Marlton, NJ 08053
Telephone (609) 797-0900
Fax (609) 797-0909

ATTACHMENT A TO PURCHASE ORDER 7071MI

ITEM DESCRIPTION AND MISCELANEOUS REQUIREMENTS

1.0 Item Description

Supply a set of samples of NS-4-FR with B_4C loading between 1 and 1.2 weight percent and a weight concentration of hydrogen of 6.0% (+/-0.1%). Sample sizes shall be as follows:

a) Use a minimum two foot long section of thin walled plastic pipe (ie. PVC piping) with a minimum inside diameter of four inches. Place an end cap on one side. Pour NS-4-FR into pipe. Make two samples, each a minimum of two feet long.

b) Make three samples with minimum dimensions of 16" (length), 8" (width) and 2" (depth). An aluminum lasagna tray or other similar commercial item may be used.

2.0 Miscellaneous Requirements

a) The manufacturing processes and materials used to make the NS-4-FR samples shall be the same as that which has been used and shall be used in the future when manufacturing NS-4-FR for dry cask storage use (reference NAC International technical data sheet for NS-4-FR).

b) The samples shall be tested by Holtec for weight loss in order to verify existing test data. Pending the results of the tests, Holtec may begin procurement of NS-4-FR material for use in its dry storage casks. An audit of NAC would be performed by Holtec prior to the procurement of any NS-4-FR material that would be used on dry storage casks.

c) NAC shall provide a letter to Holtec which defines the weight loss characteristics of NS-4-FR. If NAC cannot certify that the samples supplied to Holtec will meet specified weight loss criteria identified in previous test data, the NAC letter should clearly state this.

Attachment B to Purchase Order 7071MI

Quality Assurance Requirements

(Requirements with an 'X' or a check are applicable on this order.)

(X) 10CFR21 applies

( ) 10CFR50 Appendix B applies

(X) 10CFR71 Subpart H

(X) 10CFR72 Subpart G

( ) ASME Code Section III, Subsection _________, _________ edition with _________ addenda

( ) Supplier shall perform work under its ______ ASME Certificate.

( ) Supplier shall perform work under its ASME _____ Stamp Program.

( ) All work to be performed in accordance with revision RevO (2nded) of the supplier's QA manual.

(X) A certificate of compliance is required and must include the following:

- (X) Certification to Holtec purchase order
- (X) Certification to 10CFR21
- ( ) Certification to 10CFR50 Appendix B
- (X) Certification to 10CFR71 Subpart H
- (X) Certification to 10CFR72 Subpart G
- ( ) ASME Section III, Subsection ______
- (X) Certification to QA manual revision
- (X) Other: Material, Procedures/Revisions Used to Mix kits and Manufacture/Test NS-4-FR

(X) All items/materials to be traceable to heat numbers/lot numbers.

( ) CMTR's required for all material.

Attachment B to Purchase Order 7071MI (cont.)

(X) CMTR's shall include:

- ( ) Certfication to applicable ASME or ASTM code
- (X) Actual chemical and mechanical test results
- ( ) ASTM A262 Pr. E
- ( ) No weld statement
- ( ) Heat treatment statement
- ( ) Material supplied in unsensitized condition statement.
- ( ) U.S. Origin
- (X) Other: Density, B_4C content, hydrogen content test results and/or certification

( ) No weld repairs allowed.

( ) Applicable requirements of this purchase order shall be imposed by the supplier on its vendors.

(X) All nonconformances that are dispositioned by the supplier as " use-as-is" or repair must be submitted to Holtec for review and approval.

(X) Holtec shall have the right of access to facilities of the supplier for the purpose of inspection of materials/items, surveillance of in process activities and review of documents.

(X) Items shall be marked as follows:

Lot #/Batch # to allow traceability back to COC's and CMTRs.

Attachment B to Purchase Order 7071MI (cont.)

( ) The following hold/witness/notification points apply and require 5 days written notification to Holtec:

H,W or N	Description

Notes: Hold points must be witnessed by Holtec unless waived in writing by Holtec. Work may not proceed without Holtec witnessing the activity or waiving the requirement to witness the activity.

Witness points must be witnessed by Holtec unless waived in writing by Holtec. However, work may proceed on the scheduled day whether or not Holtec is at the facility to witness the activity unless Holtec identifies otherwise through written correspondence.

Notification points do not need to be witnessed by Holtec. The activity may proceed on the specified date without Holtec's presence.

(X) Additional QA requirements- see attachment A

doc: qapurch

NAC INTERNATIONAL

Atlanta Corporate Headquarters
655 Engineering Drive
Norcross, Georgia 30092
770-447-1144
Fax 770-447-1797
www.nacintl.com

January 28, 1998

min 16"

Mr. Mark Soler
Project Manager
Holtec International
Holtec Center
555 Lincoln Drive West
Marlton, NJ 08053

Subject: Purchase Order 7071MI
NS-4-FR Samples

Dear Mr. Soler:

NAC International is in receipt of your purchase order dated January 26, 1998.

Our review of the terms and conditions has resulted in the following clarifications to the purchase order:

Section 1.0, Boron Carbide Content
Should be stated as > 1.0% ok ms

Section 1.0, Hydrogen Content
Should be stated as >5.58% ok ms

Section 2.0 (c) Weight Loss
Weight loss characteristics are not certified properties of the material. ok ms

Attachment B, Page 2
Cannot provide mechanical or chemical test results. Can provide actual density, boron carbide, and hydrogen content. ok ms

Attachment B, Page 2
Cannot allow access as mixing / testing will not be performed at NAC facility. ok ms

If you have any questions, please do not hesitate to contact the undersigned at (770) 447-1144.

Sincerely,

Thomas A. Danner
Test Manager

ED980104

APPENDIX 3

"CERTIFIED MATERIAL TEST REPORT"
ON NS-4FR SOLD BY NAC TO HOLTEC INTERNATIONAL
WITH COVERING LETTER DATED 3/5/98 BY T. A. DANNER

NAC INTERNATIONAL

Atlanta Corporate Headquarters
655 Engineering Drive
Norcross, Georgia 30092
770-447-1144
Fax 770-447-1797
www.nacintl.com

March 5, 1998

Mr. Mark Soler
Project Manager
Holtec International
Holtec Center
555 Lincoln Drive West
Marlton, NJ 08053

Subject: Purchase Order 7071MI
NS-4-FR Samples and Certified Material Test Report

Dear Mr. Soler:

NAC International is pleased to submit the following Certified Material Test Report per the requirements of the subject purchase order.

Report 354-R-01, Revision 0
Holtec International
NS-4-FR Sample Development
Certified Material Test Report

Samples have been sent to the Florida address stated in your purchase order. Our invoice for $1,500 will be submitted under separate cover.

NAC International is pleased to have provided these services. If you have any questions or comments, please do not hesitate to contact Jeff Dargis or the undersigned at (770) 447-1144.

Sincerely,

Thomas A. Danner
Test Manager

ED980243

Holtec International

NS-4-FR Sample Development

Certified Material Test Report

Report Number 354-R-01
Revision 0

Provided By:

NAC International
655Engineering Drive
Norcross, Georgia 30092

NAC INTERNATIONAL	REVISION CONTROL SHEET	DOCUMENT NUMBER: 354-R-01

DOCUMENT TITLE:

Holtec International
NS-4-FR Sample Development
Certified Material Test Report

APPROVALS:

NAME	TITLE	REVISION	DATE
Thomas A. Danner	TEST MANAGER	0	2-17-98
Thomas A. Danner	DIRECTOR, DESIGN AND ANALYSIS	0	2-17-98
R. Howard Smith	VICE PRESIDENT, QUALITY	0	2.18.98

AFFECTED PAGES	DOCUMENT REVISION	PREPARED BY & DATE	ACCURACY & CRITERIA CHECKED BY & DATE	REMARKS
1 - 9 A1-A6 B1-B2	0	Jeffrey R. Dargis 2-17-98	Thomas A. Danner 2-17-98	None

Table Of Contents

1.0 Purpose

The purpose of this report is to provide all necessary documentation associated with the delivery of five (5) samples of NS-4-FR Shielding Material to Holtec International (Holtec) as follows:

- Two (2) NS-4-FR samples contained in minimum two foot (2') long sections of thin walled plastic pipe (i.e., PVC piping), with a minimum inside diameter of four inches (4").
- Three (3) NS-4-FR samples with minimum dimensions of 16" (length), 8" (width), and 2" (depth).
- A spare NS-4-FR sample with minimum dimensions of 16" (length), 8" (width), and 2" (depth) is provided at no charge to the customer.

Samples are provided in accordance with Reference [2.1.1] as modified by Reference [2.1.2]. The material has been procured by Holtec International for the purpose of testing.

Holtec PO Number:	7071MI
Supplier Information:	NAC International 655 Engineering Drive Norcross, Georgia 30092
Date of Manufacture:	February, 1998

2.0 References

[2.1] Purchase Orders

[2.1.1] Holtec International Purchase Order Number 7071MI, dated 1/26/98.

[2.1.2] NAC Letter ED980104, dated 1/28/98.
Subject: Exceptions to Holtec Purchase Order

[2.2] Letters/Faxes

[2.2.1] Laboratory Report, Boron and Hydrogen Test Results

[2.2.1.1] Report dated Feb. 16, 1998 for NAC Purchase Order 98-0122-01.

2.0 References (Continued)

[2.3] NAC Procedures

[2.3.1] NAC Document No. NS-P-05, Revision 1.
Specific Gravity Determination For Solid Materials.

[2.3.2] NAC Document No. NS-P-01, Revision 1.
Specific Gravity Via Gardner Sample Cup - 100 cc Volume.

[2.3.3] NAC Document No. NS-P-08, Revision 0.
Density & Elemental Analysis of NS-4-FR.

[2.4] NAC Test Specification

[2.4.1] NAC Document No. 354-S-01, Revision 0.
Holtec International Sample Development.
Test Specification.

3.0 Documentation Requirements

The documentation requirements are compiled in Table 3-1.

Table 3-1
Documentation Requirements

Document	Holtec Purchase Order 7071MI Page / Section Reference	Report Location
• Certification to Holtec Purchase Order	Attachment B	Appendix B
• Certification to 10CFR21	Attachment B	Appendix B
• Certification to 10CFR71, Subpart H	Attachment B	Appendix B
• Certification to 10CFR72, Subpart G	Attachment B	Appendix B
• Certification to NAC QA Manual Revision	Attachment B	Appendix B
• Procedures and Specifications used to manufacture samples	Attachment B	Appendix B
• Lot numbers of raw materials used in samples	Attachment B	Table 4-2
• Density Test results	Attachment B	Table 4-1
• Boron & Hydrogen Content Test Results	Attachment B	Table 4-1 and Appendix A
• Test Methods	Attachment B	Appendix A

4.0 Density & Elemental Analysis Of NS-4-FR Formulation

4.1 Specific Gravity / Density

The theoretical specific gravity of the NS-4-FR formulation is 1.67. The corresponding theoretical density of the NS-4-FR formulation is 1.67 g/cm^3.

Acceptable levels for specific gravity (sp $gr_{(req)}$) and density ($\rho_{(req)}$) are based on the tolerances identified in Reference [2.4.1].

sp $gr_{(req)}$ 1.67 ± 0.05
$\rho_{(req)}$ 1.67 ± 0.05 g/cm^3

4.2 Boron Content

Acceptable levels of boron carbide in this particular NS-4-FR formulation, $B_4C_{(min)}$, are based on the requirements of Reference [2.1.1] as modified by Reference [2.1.2].

$B_4C_{(min)} > 1\%$.

The level of boron in any sample is based on the laboratory measured boron content of the sample.

$B_{(actual)} = B\%_{(sample)}$

The theoretical amount of boron in boron carbide is 77%. Therefore the amount of B_4C in any given sample is:

$B_4C_{(actual)} = B_{(actual)} \div 0.77$

$B_4C_{(actual)}$ is then compared versus $B_4C_{(min)}$ to determine acceptability.

4.3 Hydrogen Content

Acceptable levels of hydrogen in this particular NS-4-FR formulation, $H_{(min)}$, are based on the requirements of Reference [2.1.1] as modified by Reference [2.1.2].

$H_{(min)} > 5.58\%$.

4.0 Density & Elemental Analysis Of NS-4-FR Formulation

4.3 Hydrogen Content (Continued)

The level of hydrogen in any sample is based on the laboratory measured hydrogen content of the sample.

$$H_{(actual)} = H\%_{(sample)}$$

$H_{(actual)}$ is then compared versus $H_{(min)}$ to determine acceptability.

4.4 Analysis of Laboratory Results

An analysis of the results for specific gravity (density), boron carbide, and hydrogen is found in Table 4-1.

Table 4-1
Holtec International
NS-4-FR Test Results **

Kit Sample ID	Lab ID**	Dry Density Requirement (g/cm^3)*	Actual Dry Density (g/cm^3)	Density Acceptable?	Minimum Boron Carbide Requirement (%)	Actual Boron Carbide (%)	Boron Carbide Content Acceptable?	Minimum Hydrogen Requirement (%)	Actual Hydrogen Content (%)	Hydrogen Content Acceptable?
HOL020498-01	W-8692	1.62 - 1.72	1.663	yes	1%	1.24%	yes	5.58%	6.24%	yes

* Specific Gravity Value = Density Value in g/cm^3.
** Lab ID and Test methods described in Appendix A.

Table 4-2
Lot Numbers of Raw Materials Used In Sample Development

Corresponding Kit Test Sample ID	Lot Identification			
	Raw Material 1	Raw Material 2	Raw Material 3	Raw Material 4
HOL020498-01	01218001	01218002	FR3588	M71

5.0 NS-4-FR Material Dedication

This section establishes the requirements for the safety related dedication of NS-4-FR shielding materials procured under Reference [2.1.1] as modified by Reference [2.1.2].

The only material falling under the auspices of the NAC Quality Assurance Program is the NS-4-FR shielding material. NS-4-FR consists of a formulation of commercial grade raw materials. The NS-4-FR material was manufactured and the kits prepared in accordance with Reference [2.4.1]. The NS-4-FR material dedication process consists of the following steps:

- Manufacture of the kits in accordance with approved procedures.
- Documentation (i.e., individual batch sheets) of commercial grade raw material formulations for each of the kits manufactured (Proprietary).
- Mixing of samples from each combination of raw material lots.
- Testing of samples for density, boron content, and hydrogen content.
- Meeting test acceptance criteria as defined in the purchase order and/or test specification (dedication point).
- Documenting dedication process and test report results in a Certified Material Test Report.

6.0 Open Items

None.

Appendix A
Galbraith Laboratories
Boron & Hydrogen Test Report

There are a total of six (6) pages in this appendix, including this cover sheet.

GALBRAITH® LABORATORIES, INC.

Accuracy with speed - since 1950

LABORATORY REPORT

Jeffrey Dargis
NAC International
655 Engineering Drive
Norcross GA 30092

Report Date:	02/16/98
Sample Received:	02/09/98
Purchase Order #:	98-0122
FAX #:	770-447-1797

I. Report Cover Sheet

1. Report Date 2/12/98
2. Client name: NAC International, Inc.
   655 Engineering Drive
   Norcross GA 30092
3. NAC PO No.: 98-0122-01
4. Hydrogen Testing by ASTM D-5373, GLI #ME-11
   Boron Testing by ASTM C-791 Modified, GLI #E5-3 G56C
5. Sample Ids: ID No. HOL020498-01
6. Sample Size: Hydrogen: 40.5 mg, 40.4 mg
   Boron: 60.42 mg, 57.22 mg
7. Statement 1: As identifed by NAC International, boron and hydrogen testing of the samples identified on this report cover sheet are provided in support of:
   - Holtec International Sample Testing

GALBRAITH® LABORATORIES, INC.

Accuracy with speed - since 1950

LABORATORY REPORT

Jeffrey Dargis

8. Statement 2: Results: Duplicate testing was performed to indicate degree of test repeatability.
9. Statement 3: Certificate of Conformance: Tests were performed in accordance with procedures listed on this cover sheet for sample numbers HOL020498-01. Test results have not been falsified or misrepresented in any manner.
10. Statement 4: Hydrogen and boron testing was performed in accordance with regulations and reporting requirements defined in 10CFR71, Subpart H and 10CFR72, Subpart G.

II. Report

SAMPLE ID	LAB ID	ANALYSIS	RESULTS	
ID No. HOL020498-01	W-8692	Hydrogen	6.24	%
			6.46	%
		Boron	0.96	%
			0.96	%

III. Appendices:

1. Equipment List: Leco 1000 GLI #483
   Perkin Elmer P-2000 GLI #694
2. See attached method summaries

Lee Bates
Manager of Technical Services
LB:yd

Robert L. Logan
Director of Quality Assurance

U.S. Mail: P.O. Box 51610 · Knoxville, TN 37950-1610
Other Carriers: 2323 Sycamore Drive · Knoxville, TN 37921-1750
Tel: (423) 546-1335 · Fax: (423) 546-7209

GALBRAITH® LABORATORIES, INC.
Accuracy with speed - since 1950

Galbraith Laboratories, Inc.

Method Summary

Procedure #: ME-11 Rev 1 **Analyte:** CHN **Range:** See *

Title: Carbon, Hydrogen, and Nitrogen by Leco CHN 1000 Determinator

Effective Date: 04/23/96 **Superseded:**

Procedure: Weigh 10 to 100 mg of sample into a tin capsule to the nearest 0.001 mg. Seal.
Alternate A - for difficult-to-combust samples - add tin powder or V_2O_5.

Instrument: Leco CHN 1000 Determinator

Calibration: Acetanilide (2-40 mg)

Control: s-1483 Cyclohexanone-2,4-dinitrophenylhydrazone ; % C:H:N 51.79: 5.07: 20.14; s-2483 Acetanilide % C:H:N 71.09: 6.72: 10.36; s-3483 Acetanilide % C:H:N 71.09: 6.71: 10.36; s-6483 EDTA % C:H:N 41.10: 5.52: 9.59; s-0606 Carbon Powder % C 100.00 One every ten samples

Precision and Accuracy:

	RSD	RE
s-3483 (H)	1.97 %	0.03 %
s-3483 (N)	1.26 %	0.59 %
s-3483 (C)	0.32 %	0.05 %
s-0606 (C)	0.20 %	0.03 %

Determination: Combustion at 1050 °C in constant O_2 stream ; Infrared absorption for CO_2, H_2O ; Thermal conductivity of sampled gas stream for N_2 (reduced nitrogen oxides). Quantitation limit is 0.5 %. Method detection limit is 0.3 %. Alternate A) Combust at 1100°C

Calculations: Microprocessor calculates response factors and results from sample weights manually entered.

References: Leco Manual 200-370

ASTM D5291-92, Standard Test Methods for Instrument Determination of Carbon and Hydrogen.

Other procedures: ME-2, ME-7

*Comments: Range C: 0.5-100% H: 0.5-15% N: 0.5-50%

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354-R-01
Rev. 0
PAGE A4 OF 6

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Galbraith Laboratories, Inc.

Method Summary

Procedure#: G-56C Rev 0 **Analyte:** B, nonvolatile Si **Range:** ppm-%

Title: Sodium Carbonate Fusion for Boron and Non-Volatile Si Analyses by Inductively Coupled Plasma-Optical Emission Spectroscopy and Flame Atomic Absorption

Effective Date: 03/19/92 **Superseded:**

Procedure:

Decomposition: The sample is fused with sodium carbonate over a bunsen burner. The resulting melt is dissolved in ASTM type I water with the addition of tracemetal hydrochloric acid. When the melt has been dissolved the solution is transferred to a plastic volumetric flask. An internal standard is added and the solution is brought to volume. The sample is now ready for analysis.

References: R. Bock, Decomposition Methods in Analytical Chemistry, T. & A. Constable Ltd., Edinburgh 1979.

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GALBRAITH® LABORATORIES, INC.
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Galbraith Laboratories, Inc.

Method Summary

Procedure #: E 5-3 Rev 1 **Analyte:** B **Range:** ppm-%

Title: Boron by Inductively Coupled Plasma Emission Spectroscopy

Effective Date: 07/28/92 **Superseded:**

Procedure:

Decomposition: Choose an appropriate digestion method

Instrument: Perkin-Elmer P2000 or Optima 3000

Calibration: Standards to bracket sample concentration

Sample Intro.: Plasma (15 l/min; 1400 watts)

Determination: Primary wavelength at 249.77 nm; Direct readout; detection limit @ 0.010 mg/L

Interferences: Spectral only

Calculations: Microprocessor; (ppm x v /10 x D) / mg sample = %
(v = sample volume in mL; D = dilution factor)

Precision/Accuracy: RSD = 5.57 %; RE = 2.66 %

References:

Wallace et. al. Analytical Methods for Inductively Coupled Plasma Spectrometry, Perkin-Elmer Corp., Norwalk 1981.

"Standard Practice for Describing and Specifying Inductively Coupled Plasma Optical Emission Spectrometers", ASTM E1479-92, Annual Book of ASTM Standards, American Society for Testing and Materials

USEPA Test Method 200.7 Inductively Coupled Plasma -AES Method for Trace Element Analysis of Water and Wastes, Dec. 1982.

Other Procedures: E 5 series

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354-R-01
Rev. 0

Appendix B
Certificate Of Conformance

There are a total of two (2) pages in this appendix, including this cover sheet.

NAC INTERNATIONAL

CERTIFICATE OF COMPLIANCE

Customer:	Holtec International Holtec Center 555 Lincoln Drive West Marlton, NJ 08053	Holtec PO No.:	7071MI

Regulatory and Reporting Requirements: 10CFR71, Subpart H; 10CFR72, Subpart G; 10CFR21

Quality Assurance Standard / Revision: NAC International Quality Assurance Manual Edition 2, Revision 0

Test Specification: NAC International Document 354-S-01, Revision 0. Holtec International, NS-4-FR Sample Development

Test Procedures:

NAC International Document NS-P-01, Revision 1. Specific Gravity Via Gardner Sample Cup, 100 cc Volume

NAC International Document NS-P-05, Revision 1. Specific Gravity Determination For Solid Materials

NAC International Document NS-P-08, Revision 0. Density and Elemental Analysis of NS-4-FR

Line	Qty	U/M	Description
1	2	each	NS-4-FR samples contained in minimum two foot (2') long sections of thin walled plastic pipe (i.e., PVC piping), with a minimum inside diameter of four inches (4").
2	3	each	NS-4-FR samples with minimum dimensions of 16" (length), 8" (width), and 2" (depth).
3	1	each	Spare NS-4-FR sample with minimum dimensions of 16" (length), 8" (width), and 2" (depth).

Exceptions:
None.

NAC International certifies that the samples listed above were furnished to Holtec International (Holtec) in conformance with the requirements of Holtec Purchase Order 7071MI as modified by NAC International Letter ED980104, dated 1/28/98. Exceptions (i.e., if any) to the specification requirements are listed above and have been reported and approved. Non-proprietary records which provide objective evidence supporting this certification have been transmitted to the Purchaser and/or are in Seller's possession and are available for review from the purchaser upon request.

Roy W. Barr for
R. Howard Smith
Vice President, Quality
NAC International

Thomas A. Danner 2-17-98
Thomas A. Danner
Test Manager
NAC International

APPENDIX 4

HOLTEC LETTER TO NAC DESCRIBING OBSERVED VOIDS IN THE NS-4-FR MATERIAL PROVIDED BY NAC, AND RELATED CORRESPONDENCE BETWEEN HOLTEC AND NAC (ca. 1998)

HOLTEC INTERNATIONAL
Holtec Center, 555 Lincoln Drive West, Marlton, NJ 08053
Telephone (609) 797-0900
Fax (609) 797-0909

April 20, 1998

Mr. Tom Danner
NAC International
655 Engineering Drive
Norcross, GA. 30092

Reference: Holtec Purchase Order 7071MI

Subject: Voids in NS-4-FR Samples

Upon receiving the samples of NS-4-FR material from the above referenced purchase order, Holtec began sectioning the samples in order to prepare actual test specimens. During sectioning, large voids were discovered in the material. Copies of photographs showing these voids are provided as an attachment to this letter.

The Holtec purchase order required that the samples be supplied using the same manufacturing processes and materials which have been used and shall be used in the future when manufacturing NS-4-FR for dry storage casks. The purchase order also imposed NAC's QA manual, 10CFR21, 10CFR71, Subpart H and 10CFR72, Subpart G requirements. We were advised that the samples were being made at the same time as the material being installed for the Japanese NFT casks so we have to believe that the requirements of our purchase order were fully met by NAC.

Therefore, the voids found in these samples raise significant concerns regarding the manufacturing capabilities of NS-4-FR and the acceptability of the material for use in dry storage casks. Clearly, unknown voids in the material will have a detrimental affect on the shielding capabilities of the cask. Since there is no clear way to verify that the material is void free other than full neutron scanning and as it appears that the manufacturing process used by NAC does not preclude the creation of voids in the NS-4-FR, the preparation and installation methods of the NS-4-FR material as well as the material itself needs to be questioned.

Holtec kindly requests NAC to advise us as to how this condition could have occurred in the samples provided to Holtec for testing. This situation has potential 10CFR21 implications since the NAC-STC and other designs have been licensed and/or manufactured utilizing this material and manufacturing methods. Your response to this very important issue is most critical. If you have any questions, please contact me at 609-797-0900 (x619).

Mark Soler
Director of Quality Assurance

attachment: 2 pages

document: 5014-I042098

HOLTEC
INTERNATIONAL

MEMORANDUM

To: Vik Gupta,
QA Department,
Holtec International

From: Stanley E. Turner, PhD, PE
Senior Vice President
Holtec International

Subj: Validation Testing of NS4 Neutron Absorber Material

Date: April 6, 1998

Attachments: Two Photographs of NS4 QA/QC Samples

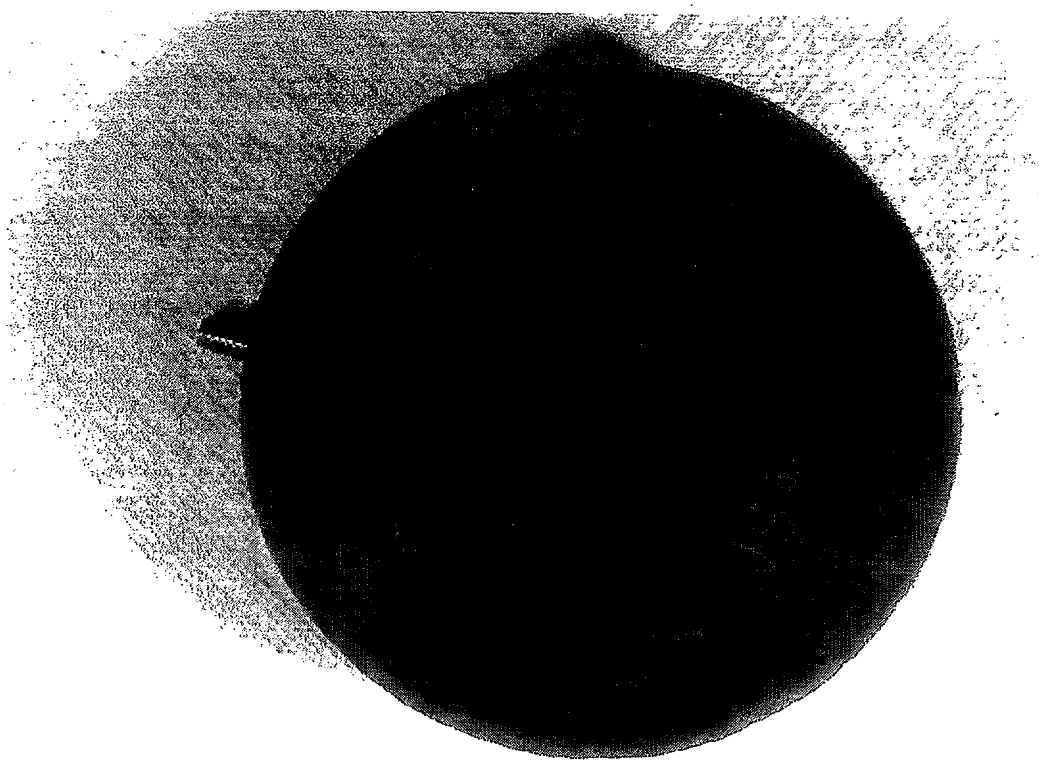
cc: Dr. K. P. Singh, President and CEO
Bernard Gilligan, Project Manager

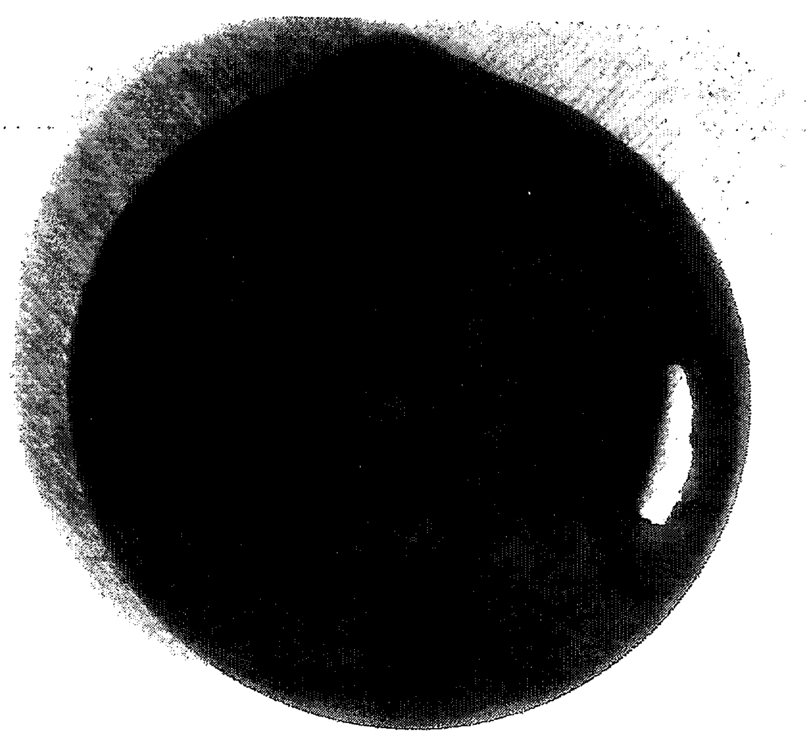
Holtec International recently ordered samples of NS4 neutron absorber material for validation testing to confirm acceptability of the material. This material was manufactured by the NAC Corporation, presumably using their standard manufacturing procedures. The two most relevant samples were cast in 4 inch ID cylinders each about 20 inches long, simulating the possible use of the material in our HI-STAR cask.

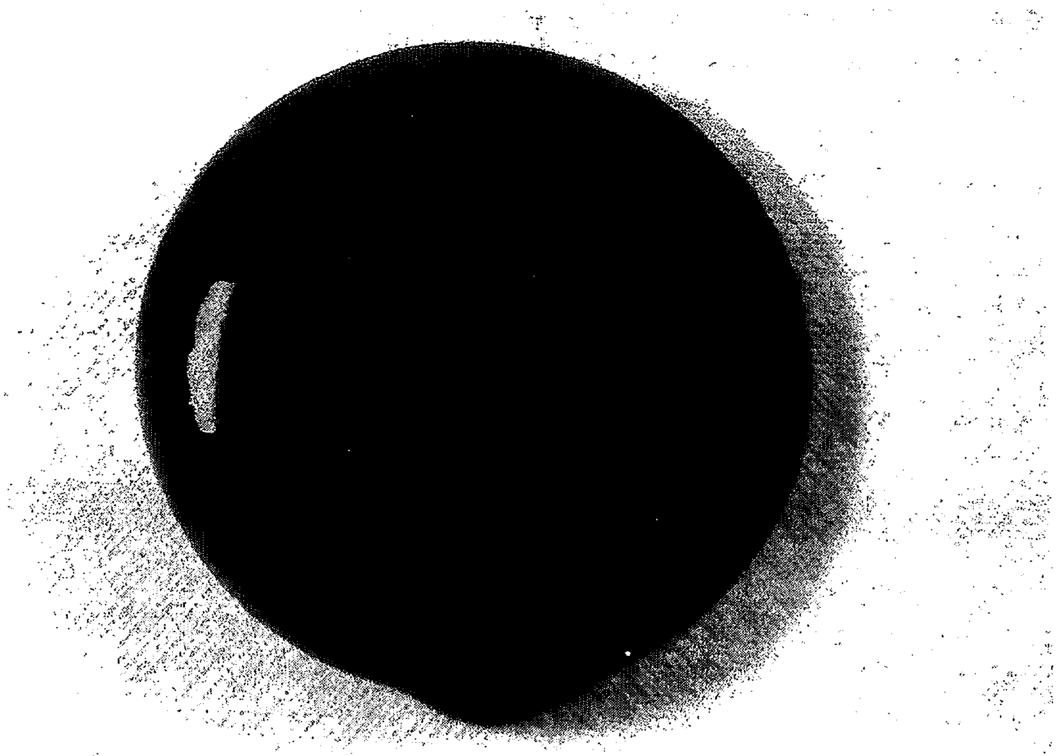
In preparation for thermal-stability testing, one of the cylindrical samples was cut into disks of a size suitable for weighing on our analytical balance. These sections revealed substantial size voids in the NS4 material as manufactured by NAC. In view of this unexpected observation, the second cylindrical sample was sectioned and also revealed similar voids. Attached hereto are photographs documenting the voids observed.

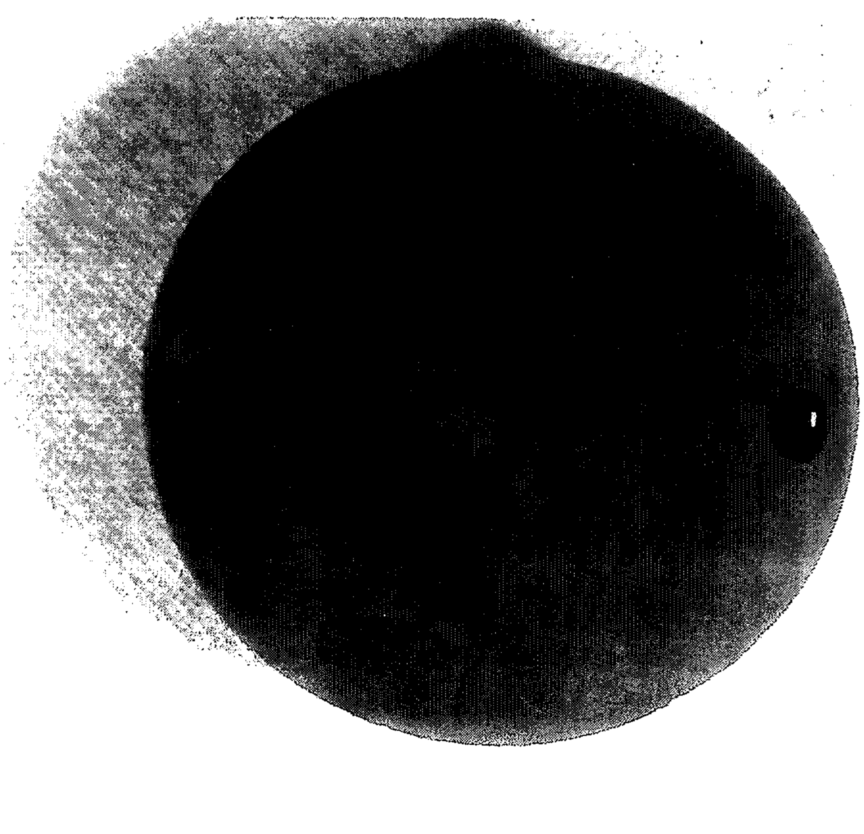
Therefore, it is my conclusion (and recommendation) that NS4 material, as manufactured by NAC is not an acceptable material for use as the neutron absorber material in the HI-STAR cask systems.

FLORIDA OFFICE: 230 NORMANDY CIRCLE EAST • PALM HARBOR, FL 34683 • (813) 787-4625 • FAX (813) 787-4719









NAC INTERNATIONAL

Atlanta Corporate Headquarters
655 Engineering Drive
Norcross, Georgia 30092
770-447-1144
Fax 770-447-1797
www.nacintl.com

70718

April 23, 1998

Mr. Mark Soler
Director of Quality Assurance
Holtec International
Holtec Center
555 Lincoln Drive West
Marlton, NJ 08053

APR 27 1998

Subject: Purchase Order 7071MI

Reference: Holtec Letter dated April 20, 1998, document number 5014-1042098

Dear Mr. Soler:

The sample NS4FR material provided as the deliverable to the subject Purchase Order was formulated, mixed, and tested in compliance with controlled procedures and NAC's Quality Assurance program as required by the subject P.O. Observation of voids in these samples is not surprising. Material formulation and mixing were controlled. Mixed material was poured into a mold without development of a specific installation procedure to eliminate voids in the cured sample. The objective of these test samples was to provide material for thermal degradation testing and not to qualify an installation procedure which results in void free material.

The photographs provided in the referenced correspondence present the sample specimens as a reddish brown color. NS4FR with boron carbide has a gray to black color. Is the difference in color resulting from a filter lens used on the camera or perhaps lubricant used when cutting the samples? We are interested in understanding why these photos represent the material color so different that our base samples. Please provide a description of your process, materials and equipment used in cutting the samples and photography parameters and lens used when taking the photograph.

Note existing project specific cask installation procedures have been validated by destructive examination of full scale mockup specimens. Section cuts through the length and at numerous cross sections verified that the procedural methods did not induce any unacceptable void in the cavity filled geometry. These tests have been performed successfully at a number of fabrication facilities in both Japan and United States.

If you have any questions, please do not hesitate to contact the undersigned at (770) 447-1144.

Sincerely,

Thomas A. Danner
Director, Design, Analysis and Product Development

ED980405

HOLTEC INTERNATIONAL

Holtec Center, 555 Lincoln Drive West, Marlton, NJ 08053

Telephone (609) 797-0900
Fax (609) 797-0909

May 12, 1998

Mr. Tom Danner
NAC International
655 Engineering Dr.
Norcross, GA. 30092

Reference: 1) Holtec Letter to NAC dated April 20, 1998
2) NAC Letter to Holtec dated April 23, 1998

Dear Mr. Danner:

We have read your responses in reference (2) however the number and size of voids in the supplied samples still provide significant concern to us since we would have expected the samples to be supplied with the highest level of quality. While the pipe used for the mold has a somewhat smaller inner dimension than our cavity for the overpack, they are similar in nature and thus our concern over the ability of the NS-4-FR material to be installed in a void-free manner still exists. We would like to ask the following questions regarding the control of voids in NS-4-FR:

1) Whether pouring into a small cylinder or a large square tube, how would an installation process differ such that separate installation qualification procedures must supposedly be developed? It would seem that the pouring mechanism would be the same though the amount poured at any one time might differ.

2) If voids can appear in a tube over a two foot section as they did in our samples, how can one be assured that a cavity as long as 160" can be properly poured without having voids? Did NAC's qualification procedures evaluate a sample that was at least as long as cavities in manufactured casks?

3) Have any of the raw materials used in making NS-4-FR changed or been supplied by a different manufacturer since installation qualification activities were performed? If so, can NAC guarantee that these changes would not affect the ability to properly install the NS-4-FR without voids?

4) Do the samples supplied to Holtec comply with the chemistry data specified in the technical data sheet for NS-4-FR supplied by NAC?

HOLTEC INTERNATIONAL

Holtec Center, 555 Lincoln Drive West, Marlton, NJ 08053

Telephone (609) 797-0900
Fax (609) 797-0909

May 12, 1998

As a follow-up to question 3, we have some additional concerns regarding test data on NS-4-FR. Has any raw materials used in making NS-4-FR changed or been supplied by a different manufacturer since qualification testing for the NS-4-FR for weight loss, ability to withstand heat/fire and long term affects of radiation were performed? If so, has the material been requalified and can a copy of those reports be submitted to Holtec?

Your response to these questions will be greatly appreciated. Finally, in response to your question in reference (2), please be advised that the photographs sent to you for your examination were taken after the samples had been heated in the oven.

If you have any questions regarding this letter, please feel free to contact me.

Mark Soler
Director of Quality Assurance

NAC INTERNATIONAL

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770-447-1144
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www.nacintl.com

June 2, 1998

Mr. Mark Soler
Director of Quality Assurance
Holtec International
Holtec Center
555 Lincoln Drive West
Marlton, NJ 08053

Subject: Purchase Order 7071MI

Reference:
1) Holtec Letter dated May 12, 1998
2) NAC Letter to Holtec dated April 23, 1998
3) Holtec Letter to NAC dated April 20, 1998

Dear Mr. Soler:

This letter provides NAC's response to your correspondence dated May 12, 1998, Reference 1.

Review of our response to your original inquiry about voids in samples of material to be used for thermal testing may be of help to your staff in understanding the presence of voids in the thermal aging test samples. As stated in Reference 2, material was supplied for thermal age testing in full compliance with our Quality Assurance program and the project purchase order. Material handling from the mixer to the intended fill cavity is one of the most sensitive operations performed when installing NS4FR shielding. As stated in earlier correspondence, installation of NS4FR has been successfully accomplished at a number of facilities in both Japan and the United States. However, because of this operational sensitivity, it is most important that project specific proprietary installation procedures using delivery systems of specified and qualified design be developed when the product is required to be free of voids. Mixed material for the Holtec thermal aging test was simply poured into a mold without development of a specific installation procedure or a qualified delivery system to eliminate voids in the cured sample, since this was not the objective of pouring the samples. The objective of these test samples was to provide material for thermal degradation testing and not to qualify an installation procedure which results in void free material.

The following responses are provided to the Holtec questions submitted in your May 12, 1998, Reference 1, correspondence.

1) Installation procedures are proprietary. Geometry differences between cylindrical and square tubes have a minor influence. One of the most significant variables is the difference between free pour and controlled delivery. Free pour is rarely the preferred installation method for cask applications. As previously stated, the Holtec thermal aging test samples were prepared without exercising a controlled installation procedure. As stated in our previous correspondence, Reference 2, existing project specific cask installation procedures have been validated by

destructive examination of full scale mockup specimens. Section cuts through the length and at numerous cross sections verified that the procedural methods did not induce any unacceptable voids in the cavity filled geometry. These tests have been performed successfully at a number of fabrication facilities in both Japan and the United States.

2) As stated in Reference 2, NS4FR has been qualified for installation and installed in spent fuel transport and storage casks at a number of fabrication facilities in both Japan and the United States. Filled cavities have been similar to the tube geometry created by the radial heat transfer fins and cask shells typical of the Holtec cask. Lengths of these cavities are representative of your quoted 160 inch length , and NAC has evaluated samples of a full cask length.

3) Raw material constituents and supply have not changed since initial formulation and commercial supply. Raw material constituents continue to meet the same chemical specifications as those used for original material testing.

4) The samples supplied comply with the raw material chemical specifications for NS4FR. Hydrogen and boron concentration test reports have been provided to Holtec for the sample material. Additional chemistry testing was not contracted for and, therefore, was not performed.

If you have any questions, please do not hesitate to contact the undersigned at (770) 447-1144.

Sincerely,

Thomas A. Danner
Director, Design, Analysis and Product Development

APPENDIX 5

NAC INTERNATIONAL TECHNICAL BROCHURE FOR NS-4-FR

NAC INTERNATIONAL
GESC SHIELD MATERIALS

TECHNICAL DATA

NS-4-FR
Fire Resistant Neutron and/or Gamma Shielding Material

GESC NS-4-FR Fire Resistant Neutron Shielding Material is a high hydrogen structural shielding product designed for use in moderately high temperature applications. It has the unique characteristics of high strength, mechanical durability and fire resistivity. NS-4-FR may be loaded with lead and/or boron, offering excellent gamma or neutron shielding properties. NS-4-FR has been found to offer superior neutron shielding/attenuation properties over equivalently loaded polyethylene.

NS-4-FR Properties

Color	Brown
Specific Gravity	1.68
Hydrogen	6.07E+22 atoms/cc
Maximum Continuous Operating Temperature	300°F -X-
Radiation Resistance	Excellent
Ultimate Tensile Strength	4,250 psi
Tensile Elongation	0.65%
Ultimate Flexural Strength	7,600 psi
Ultimate Compression Strength	10,500 psi
Compression Yield Strength	8,780 psi
Compression Modulus	561,000 psi
Izod Impact Strength	2.9 ft. - lb./in.
Thermal Conductivity	0.373 BTU/hr.-ft-°F

Theoretical Elemental Composition

Carbon	27.7 wt.%	Nitrogen	2.0 wt.%	Hydrogen	6.0 wt.%
Oxygen	42.8 wt.%	Aluminum	21.5 wt.%		

Maximum B_4C And Lead Loadings

B_4C: 6.5 wt.%

Lead: 15 wt.%

Applications

Vessels, Closures, Structural Components, Doors, Bricks, Criticality Control.

Availability

Cast Special Shapes, Plates, Rounds, Squares, Structural Shapes (vessels, tanks, etc.)

Data is based on laboratory tests and should not be used for writing specifications. Each user should run independent tests to confirm material suitability for each specific application.

655 Engineering Dri

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NAC INTERNATIONAL
GESC SHIELD MATERIALS

MATERIAL SAFETY DATA SHEET

NS-4-FR Neutron Absorber

Section I—General Information

DOT Hazard Class:	Non-Regulated
Trade Name:	NS-4-FR Kit
Chemical Family:	Resin System: Proprietary
Formula:	3 Component System
Date Issued:	05/01/94

Section II—Compositional Information

	Ingredient	*TWA/TLU*
Part 1:	Proprietary	Non Established
Part 2:	Proprietary	Non Established
Part 3:	Proprietary	Non Established
Hazardous ingredients as defined in 29 CFR 1910 · 1200		None Present

Section III—Physical Data

Property	*Part 1*	*Part 2*	*Part 3*
Boiling Point (°F)	>350°F	>350°F	N/A
Vapor Pressure (68°F)	Nil	Nil	N/A
Vapor Density (Air=1)	1	N/A	N/A
Freezing Point (°F)	N/A	N/A	N/A
Specific Gravity (g/cc)	1.164	0.947	2.42
Melting Point (°F)	N/A	N/A	3700°F
Solubility in water	Nil	Nil	Nil
Odor	Sweet	Slight Ammoniacal	Negligible Odor
Percent Volatile	N/A	N/A	N/A
Physical State	Yellowish Clear Syrup	Thin Brown Liquid	Crystalline Powder

Section IV—Fire And Explosion Hazard Information

Property	*Part 1*	*Part 2*	*Part 3*
Flash Point (method-PMCC)	485°F	320°F	N/A
Flammable Limits	N/A	N/A	N/A
Extinguishing Equipment	CO_2/Dry Chemical, Foam	CO_2/Dry Chemical, Foam	CO_2/Dry Chemical, Foam
Special Fire Fighting Procedures	Wear full protective equipment including self-contained breathing apparatus. Cool exposed tanks with water.		
Unusual Fire and Explosion Hazards	None	None	None

Section V—Reactivity Data

Stability: Three parts are stable.

Incompatibility with Materials:

Part 1:	Oxidizing agents and mineral acids
Part 2:	Oxidizing agents and strong lewis or mineral acids
Part 3:	Soluble in strong acids and alkalies

Data is based on laboratory tests and should not be used for writing specifications. Each user should run independent tests to confirm material suitability for each specific application.

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Hazardous Decomposition Products:

Part 1:	CO, CO_2, and NO_x
Part 2:	CO, Aldehydes and acids from incomplete combustion
Part 3:	CO, CO_2

Polymerization will not occur in any part.

Section VI—Health Hazard Data

Routes of Entry:	Part 1	Part 2	Part 3
	Inhalation	Moderately Toxic Heated Part 1&2 will	Nuisance Dust
	Skin	Moderately Toxic cause minor Irritation	Not an irritant
	Ingestion	Moderately Toxic and be moderately toxic	Non expected

Health Hazard:

Part 1: May cause burns to skin and eyes. Liquid causes damage to mucous membranes.
Part 2: Resins may cause burns to skin and eyes. Avoid skin contact.
Part 3: Treat as a nuisance dust.

Signs of Exposure:

Part 1: May cause Asthma, skin sensitization or other allergic response.
Part 2: Itching skin or rash.
Part 3: None Known

NFPA Ratings:	Part 1	Part 2	Part 3
Health	3	3	0
Flammability	1	1	0
Reactivity	1	1	1

Emergency or First Aid Procedures:

Part 1: Eye contact, flush with plenty of water and get medical attention. Skin contact, wash with soap and water. If ingested, do not induce vomiting, get medical attention.
Part 2: Wash thoroughly with soap and water.
Part 3: Wash thoroughly with soap and water. Induce vomiting if large amounts are ingested. Flush eyes immediately with water for 5 minutes.

Section VII - Precautions For Safe Handling And Use

Spilled Material:

Part 1: Soak in industrial absorbent.
Part 2: Scrape up as much as possible, wipe remainder with rags and solvents. Proper precautions against fire and personal overexposure should be taken.
Part 3: Use dry clean up procedures; avoid dusting.

Waste Disposal:

Part 1: Should be sealed in metal containers at approved waste sites operated in compliance with Federal, State and Local Regulations.
Part 2: Same as Part 1.
Part 3: May be disposed at a sanitary landfill. Consult local regulations.

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MATERIAL SAFETY DATA SHEET

NS-4-FR Neutron Absorber

Storage and Handling:

Part 1:	Repair leaky drums. Keep lids tightly closed and store at temperatures between 50-95°F. Avoid temperatures above 110°F and below 45°F.
Part 2:	Same as Part 1.
Part 3:	Avoid prolonged storage and exposure to moisture.

Section VIII—Control Measures

Respiratory Protection:	NIOSH approved respirator along with using in well ventilated areas.
Local Exhaust:	Recommended.

Protecting Clothing:

Part 1:	Rubber gloves and gauntlets along with safety glasses with side shields must be worn. Coveralls and work shoes should also be worn at all times. Wash regularly at both the beginning and end of the work period.
Part 2:	Same as Part 1.
Part 3:	Dust respirator.

These data are offered in good faith as typical values and not as a product specification, no warranty, either expressed or implied, is hereby made. The recommended industrial hygiene and safe handling procedures are believed to be generally applicable. However, each user should review these recommendations in the specific context of the intended use and determine whether they are appropriate.

Prepared by: Thomas A. Danner
Revised: April 5, 1994

Data is based on laboratory tests and should not be used for writing specifications. Each user should run independent tests to confirm material suitability for each specific application.

655 Engineering Drive   Norcross, Georgia 30092   (404) 447-1144   Fax (404) 447-1797

APPENDIX 6

<u>PROPRIETARY APPENDIX (ALL PAGES)</u>

ARCHIVE OF THERMAL AGING TEST DATA FOR NAC-NS4FR AND HOLTITE-A

HOLTEC INTERNATIONAL

PROPRIETARY INFORMATION

APPENDIX 7

HOLTITE MATERIAL QUALIFICATION LETTER FROM LARRY DIETRICK

SCIENTECH.

September 30, 1999

Mr. Bernard Gilligan
Project Manager
Holtec International
555 Lincoln Drive west
Marlton, NJ 08053

SUBJECT: Holtite Material Qualification

Dear Mr. Gilligan:

The following information is provided regarding the Holtite neutron shielding material:

The material formulation is identical to the Bisco NS-4FR neutron shielding material that was developed by Bisco Products. The raw materials used in the formulation, as well as the proportions of each raw material used in the Holtite mix, are the same as Bisco NS-4FR. The Holtite material possesses the same properties as NS-4FR for all critical parameters (Specific gravity, hydrogen density, born density and thermal stability.)

During the seven years that I worked with NS-4FR, we placed the material in a variety of geometric shapes, (panels, containers, casks and custom designed fixtures). In each case during the curing cycle, some shrinkage of the material occurred. This normally took the form of a small pocket or pullback at the top edges of the material. In order to compensate for this, we normally would top-off the resin material with additional material after the initial curing and shrinkage had taken place. Tests were conducted to ensure that the top-off material adequately bonded to the cured material. The test showed that an excellent bond developed between the cured material and the top-off material.

In the normal course of placing this shielding material, it is possible that some air pockets can develop in the material, due primarily to the geometric shape of the cavity being filled. In every case during my experiences, the air pockets quickly rise to the top and escape before the material has cured. Please note that the Holtite material has a 24 hour cure period and does not take it's initial set until at least three hours after the material is placed. In my experience, there have not been any known cases of air pockets being trapped in the material in it's cured state.

Please contact me at 630-493-2210 if you have any further questions.

Sincerely,

Larry J. Dietrick
Senior Consultant