71-9315

BWXT Y-12
Protecting America's Future

April 27, 2005 Y/HDPO/DT05-17

Mr. Shawn Williams
U. S. Nuclear Regulatory Commission
Mail Stop: O13D13
Washington, DC 20555-0001

Submittal of ES-3100 container data as requested by phone on April 27, 2005

Dear Mr. Williams:

Per your phone request of April 27, 2005, BWXT Y-12 is herein submitting the following items:

- U. S. Patent 6,299,950 (Kaolite 1600™) [attached]
- Specifications for Thermo Electron Corporation Catalog No. 277-4 Special Dry Mix - as provided by the manufacturer [attached]. Other specifications can be found in Equipment Specification JS-YMN3-801580-A005, page 1-109 of SAR; Table 2.17, page 2-25 of SAR; Appendix 6.9.3.3, page 6-91 of SAR.
- Letter No. COR-NDA-04-93 from Canberra Oak Ridge, LLC January 5, 2005, Results of Prompt Gamma-ray Neutron Activation Analysis and Neutron Transmission Measurements on Prototype Confinement Vessel Inner Liners and Spacers. [attached]

The specifications of the Cat 277-4 material are included in various places. If the composite of this information as provided in the second bullet is insufficient for the reviewer's needs, please let me know and I will look into it further.

If you have any questions, please contact me at (865) 576-8254 or George Singleton at (865) 241-3854.

Very truly yours,

J. G. Arbital

Jeffrey G. Arbital
Containers Program Manager

JGA:acj

NMSS01

Attachments: As stated

C w/o att: D. R. Tousley, DOE NA-26
J. M. Shuler, DOE EM-24
D. P. Sooter, Y-12
E. D. Ragos, YSO

US006299950B1

(12) **United States Patent**
Byington et al.

(10) Patent No.: **US 6,299,950 B1**
(45) Date of Patent: ***Oct. 9, 2001**

(54) FIREPROOF IMPACT LIMITER AGGREGATE PACKAGING INSIDE SHIPPING CONTAINERS

(75) Inventors: Gerald A. Byington, Knoxville; Raymon Edgar Oakes, Jr., Kingston; Matthew Rookes Feldman, Knoxville, all of TN (US)

(73) Assignee: BWXT Y12 LLC, Oak Ridge, TN (US)

(*) Notice: This patent issued on a continued prosecution application filed under 37 CFR 1.53(d), and is subject to the twenty year patent term provisions of 35 U.S.C. 154(a)(2).

Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 08/940,295

(22) Filed: Sep. 30, 1997

(51) Int. Cl.[7] G21F 5/00; G21C 19/00

(52) U.S. Cl. 428/34.5; 252/478; 250/506.1; 376/272; 976/323; 976/333; 976/344

(58) Field of Search 352/478; 428/34.4, 428/34.5, 34.6; 264/71; 250/506.1; 376/272; 976/320, 323, 324, 333, 344, 350, 353

(56) References Cited

U.S. PATENT DOCUMENTS

3,982,134	9/1976	Housholder et al.	250/506
4,612,046	9/1986	Orcutt	75/96
5,385,873	1/1995	MacNeill	501/95
5,626,665	5/1997	Barger et al.	106/706
5,658,634	8/1997	Ragland et al.	428/75

FOREIGN PATENT DOCUMENTS

1460196A * 12/1976 (GB).

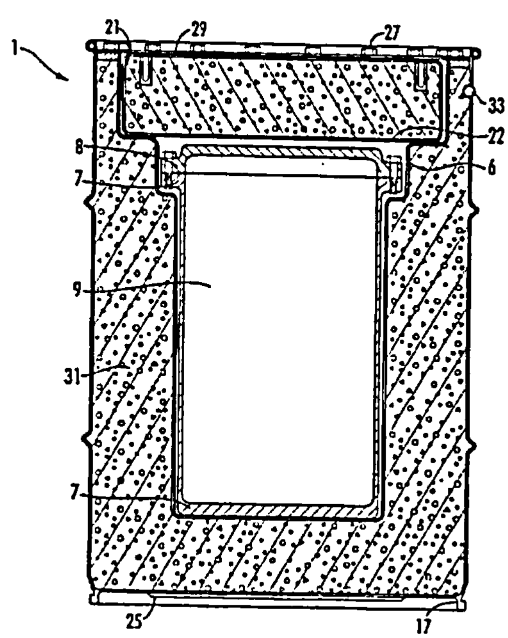
* cited by examiner

Primary Examiner—Rena L. Dye
(74) *Attorney, Agent, or Firm*—J. Herbert O'Toole; Hardaway/Mann IP Group

(57) ABSTRACT

The invention is a product and a process for making a fireproof, impact limiter, homogeneous aggregate material for casting inside a hazardous material shipping container, or a double-contained Type-B nuclear shipping container. The homogeneous aggregate material is prepared by mixing inorganic compounds with water, pouring the mixture into the void spaces between an inner storage containment vessel and an outer shipping container, vibrating the mixture inside the shipping container, with subsequent curing, baking, and cooling of the mixture to form a solidified material which encapsulates an inner storage containment vessel inside an outer shipping container. The solidified material forms a protective enclosure around an inner storage containment vessel which may store hazardous, toxic, or radioactive material. The solidified material forms a homogeneous fire-resistant material that does not readily transfer heat, and provides general shock and specific point-impact protection, providing protection to the interior storage containment vessel. The material is low cost, may contain neutron absorbing compounds, and is easily formed into a variety of shapes to fill the interior void spaces of shipping containers.

6 Claims, 4 Drawing Sheets



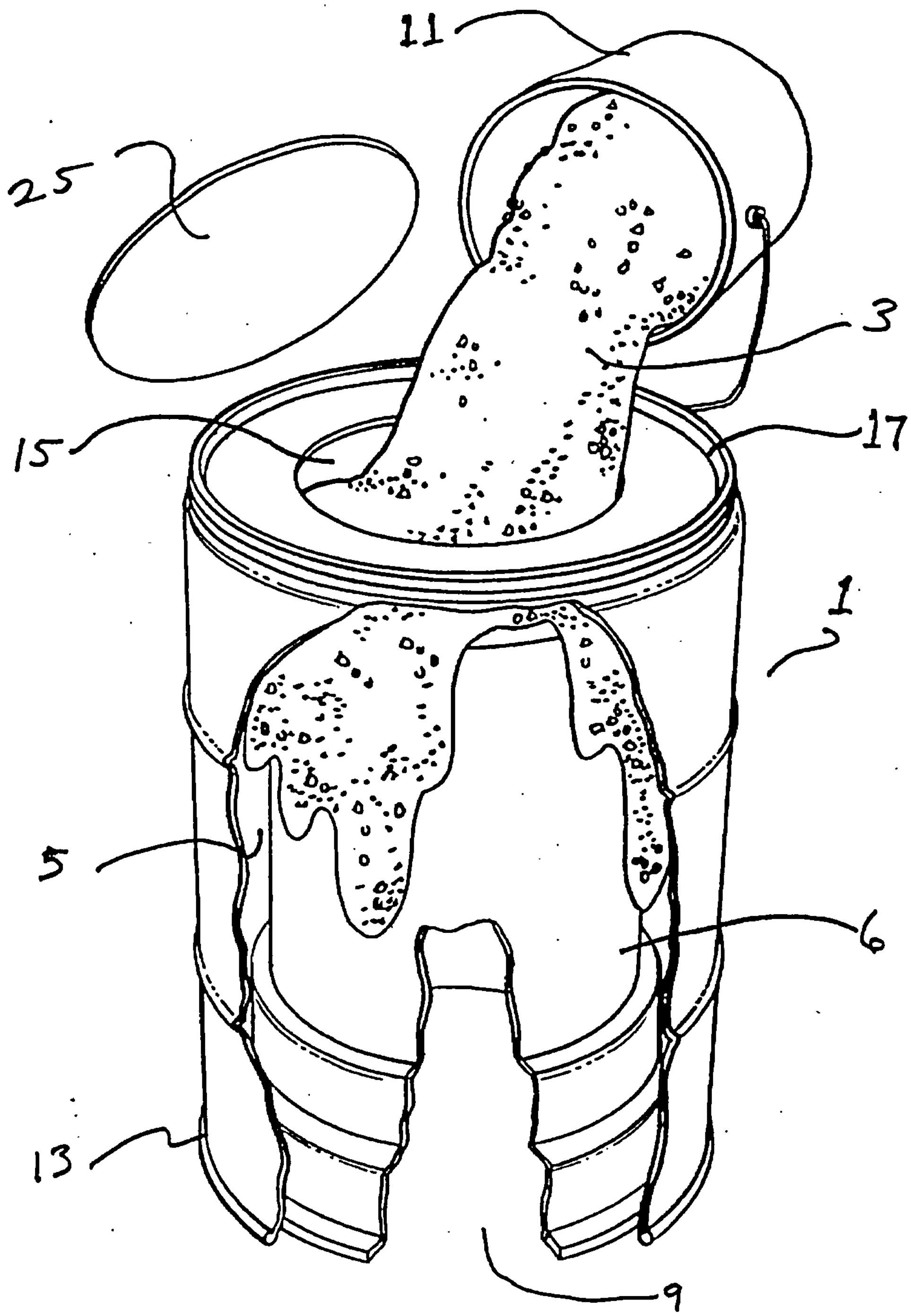


Fig. 1

FIG. 1A

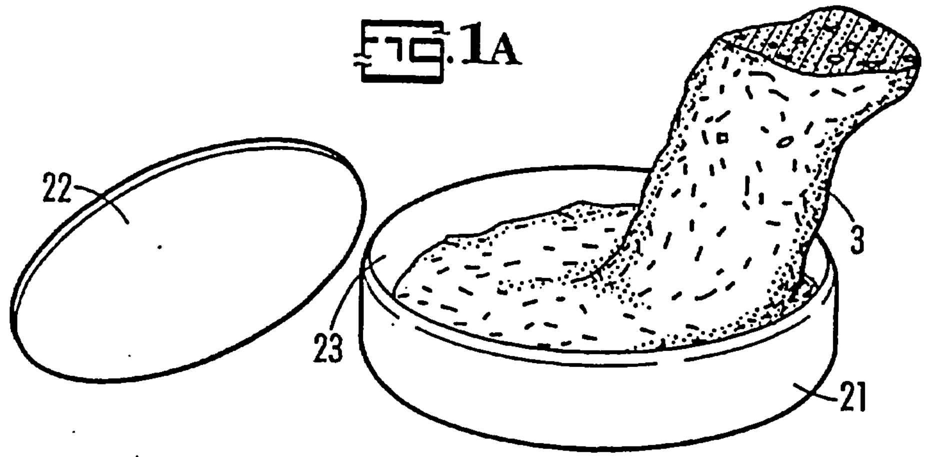
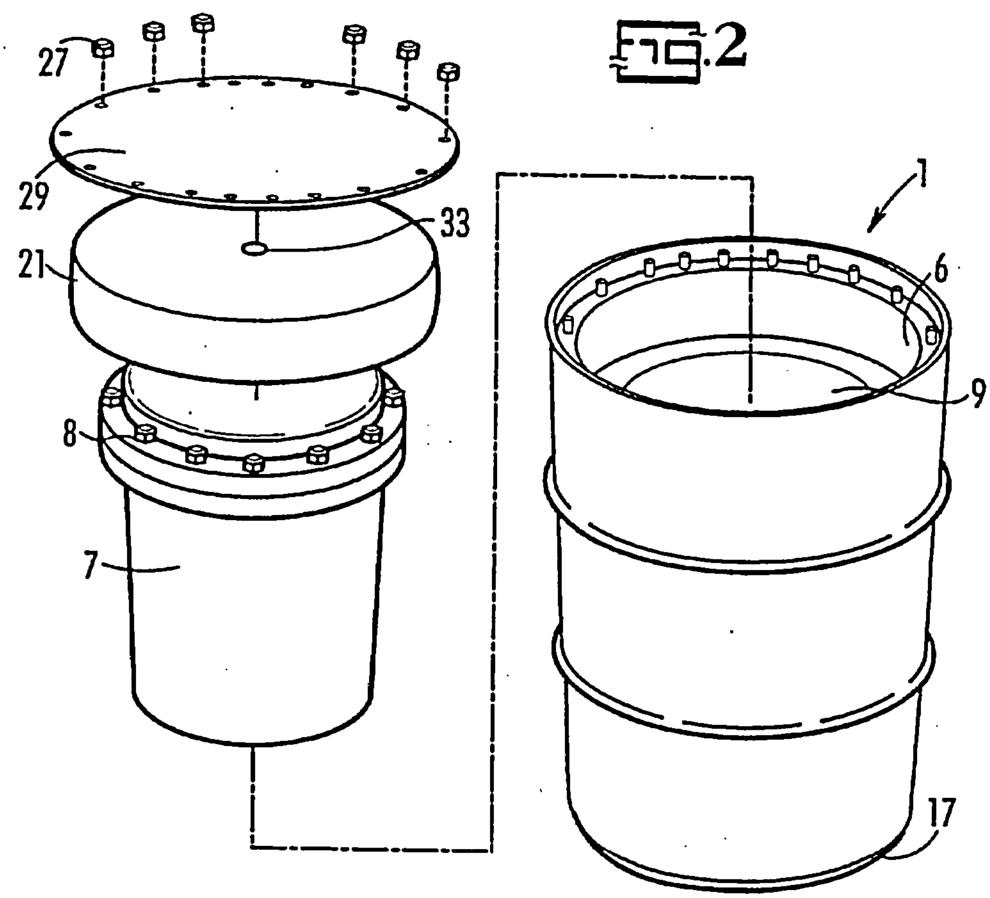


FIG. 2



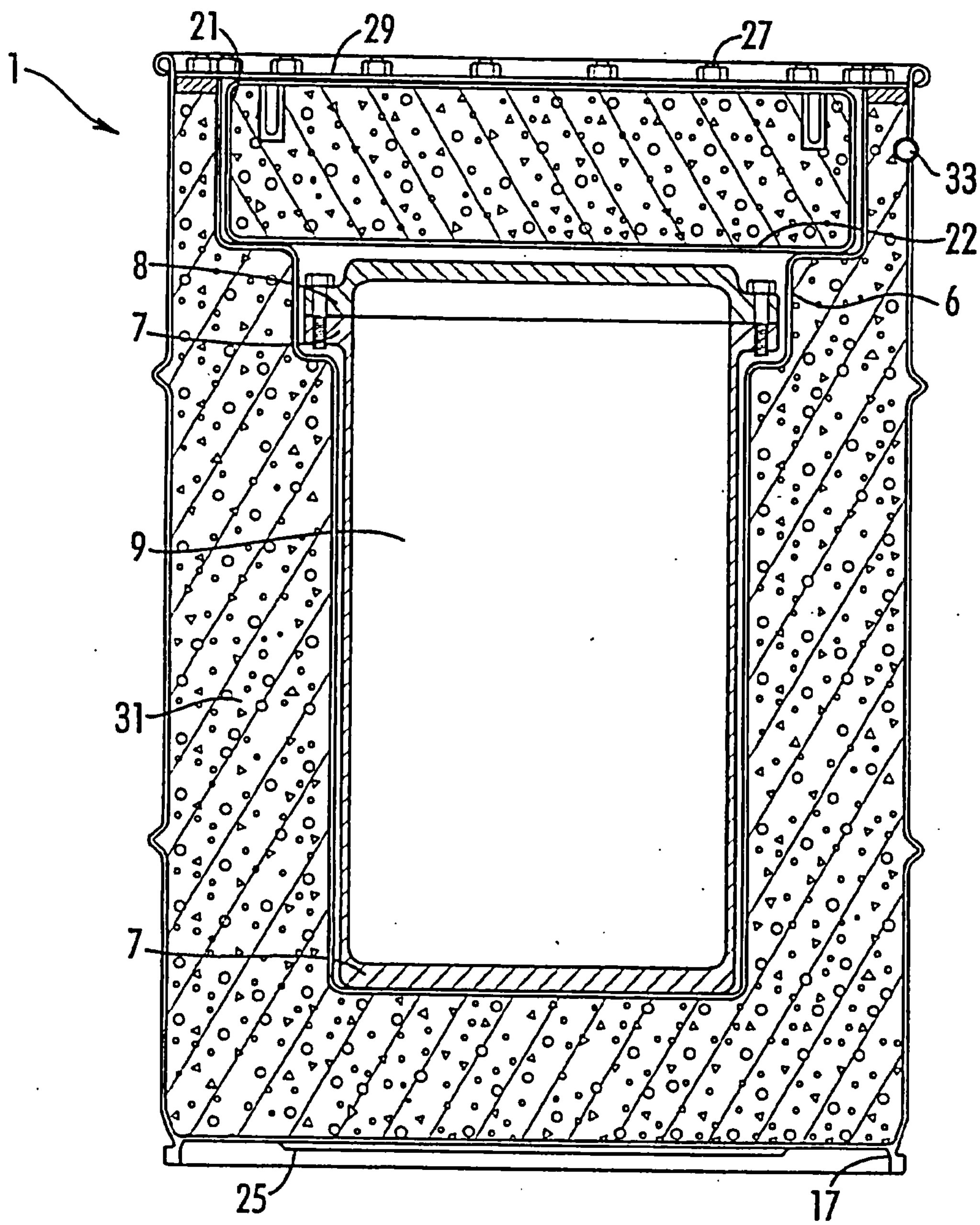


FIG. 3

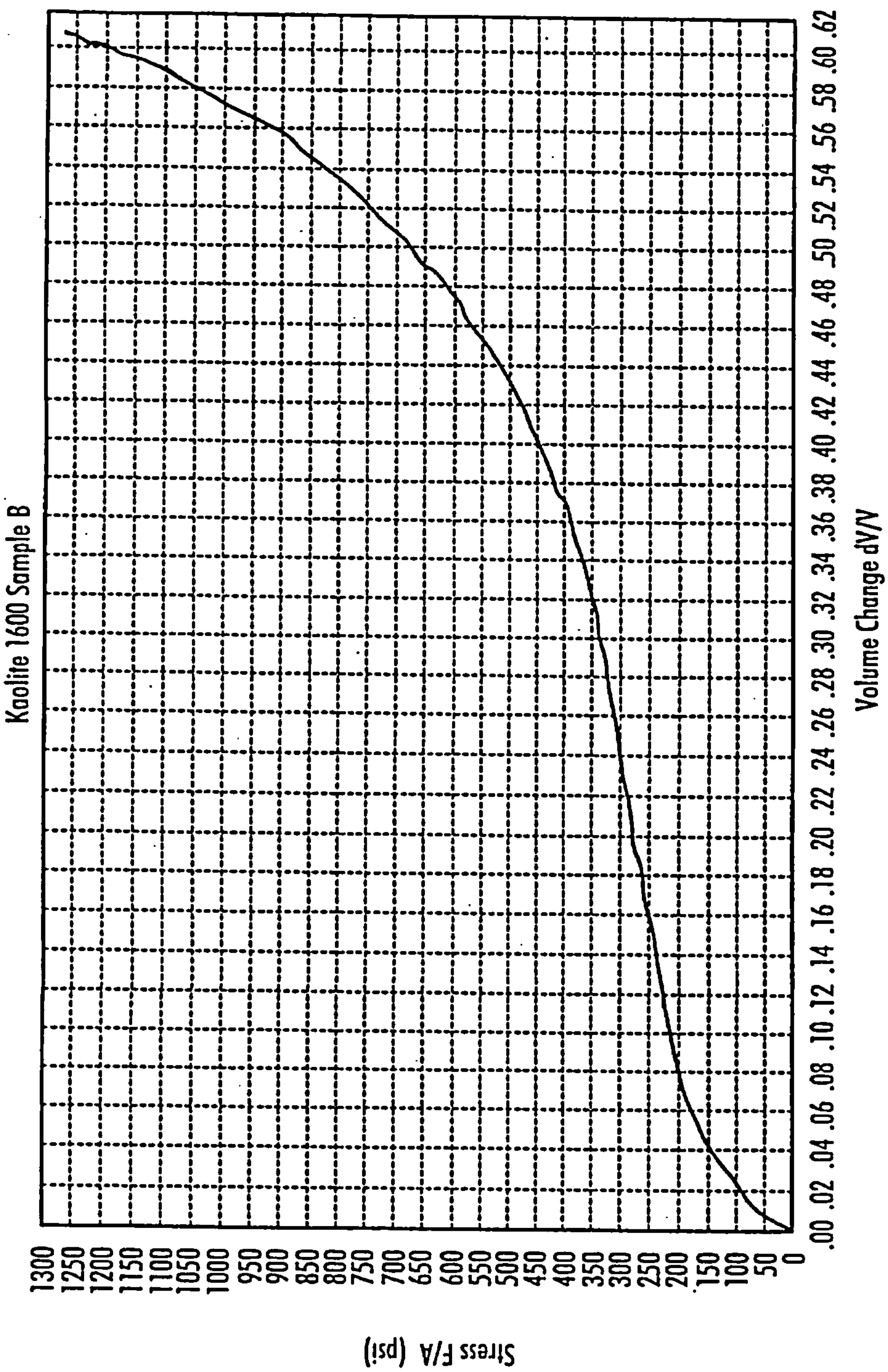

Kaolite 1600 Sample B
Stress F/A (psi)
1300 1250 1200 1150 1100 1050 1000 950 900 850 800 750 700 650 600 550 500 450 400 350 300 250 200 150 100 50 0
Volume Change dV/V
.00 .02 .04 .06 .08 .10 .12 .14 .16 .18 .20 .22 .24 .26 .28 .30 .32 .34 .36 .38 .40 .42 .44 .46 .48 .50 .52 .54 .56 .58 .60 .62

FIG. 4

FIREPROOF IMPACT LIMITER AGGREGATE PACKAGING INSIDE SHIPPING CONTAINERS

United States Government has rights in this invention pursuant to Contract No. DE-AC05-84OR21400 between the U.S. Department of Energy and Lockheed Martin Energy Systems, Inc.

BACKGROUND OF THE INVENTION

The invention relates generally to a packaging for shipping containers. Specifically, the present invention relates to a product for preventing damage to an interior containment vessel from impacts to exterior shipping containers. The invention is more specifically related to a product which is fireproof, castable, crushable, and non-toxic, which provides protection for nuclear material and/or other hazardous materials inside an interior containment vessel, when the exterior container is utilized for shipping of hazardous or radioactive materials.

Shipping containers with single, double, and triple containment have been utilized extensively in the nuclear materials and hazardous waste shipping industry. There continues a need for a product that provides shock and fire-resistant material for placement inside the exterior container, to protect and cradle the interior containment vessel. Ragland, et al., U.S. Pat. No. 5,658,634, describes a thin laminated metal foil and nonwoven fiber material that provides insulation for automotive systems. Barger, et al., U.S. Pat. No. 5,626,665, describes a cementitious system which utilizes low amounts of water plus calcined clay and other materials to produce high strength cement. MacNeill, U.S. Pat. No. 5,385,873, describes a high temperature resistant material containing ceramic fibers and inorganic vermiculite to withstand the high temperatures within catalytic converters. Orcutt, U.S. Pat. No. 4,612,046, describes an insulating composition formed from a mixture of Kaolite™ and amorphous quenched slag, to thermally insulate the exposed surface of molten metal in a cast vessel or furnace vessel. Householder, et al., U.S. Pat. No. 3,982,134, describes a container for nuclear materials transport that has on the interior, a pressure vessel, gamma radiation protection, insulating material, and neutron absorbing shielding in the form of beads or globules of water encapsulated in plastic. The prior art provides for materials that are heat insulating, shock protective, or radiation shielding when placed in separate layers into the interior of a nuclear materials shipping container. These and other materials, when added to containers, have shortcomings based on the inability to provide a light-weight, low-cost, heat insulating, shock protective, and radiation shielding material that can be castable inside the interior void spaces of a nuclear materials shipping container. Thus there exists room for improvement within the art.

SUMMARY OF THE INVENTION

It is an object of this invention to provide a process for making a product for encapsulating containers for shipping.

It is a further object of this invention to provide a process for making a product for encapsulating containers of nuclear materials and/or hazardous materials for shipping.

It is a further object of the present invention to provide a process of encapsulating a container for shipping.

It is an additional object of this invention to provide a product for encapsulating containers for shipping that is not flammable.

It is a further additional object of this invention to provide a rigid product that provides shock and impact protection to encapsulated containers by compaction of the rigid product.

It is yet a further and more particular object of this invention to provide a product that insulates an encapsulated container.

It is yet an additional and more particular object of this invention to provide a product that serves as a radiation absorber to reduce the amount of radiation measured at the exterior surface of the shipping container.

These and other objects of the present invention are accomplished by a process for making an aggregate product, and the aggregate product composed of a rigid homogeneous aggregate material utilizing portland cement and inorganic vermiculite. The material made by the process is non-flammable, provides both thermal insulation and impact protection to any containment vessel that the material surrounds.

The above and other objects of the present invention are also accomplished by a process of protecting a containment vessel inside a container for shipping nuclear materials, comprising the steps of: mixing a portland cement material with an inorganic vermiculite and water; pouring the mixture into the interior voids of an outer container for shipping; vibrating the mixture inside the outer container for shipping; curing the vibrated mixture at ambient temperature inside the exterior container for shipping; baking the cured mixture inside the exterior container for shipping at elevated temperatures; cooling the baked mixture to ambient temperature; welding a cover plate over the fill hole; and assembling the shipping package.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention's features and advantage will become apparent from a reading of the following detailed description, given with reference to the various figure of drawing, in which:

FIG. 1 is a view of the aggregate product of the present invention in a mixing container;

FIG. 1*a* is a view of the aggregate product poured into a top plug unit;

FIG. 2 is a cross-sectional view of the aggregate product poured into the exterior container of the present invention;

FIG. 3 is a cross-sectional view of the solidified product of the present invention inside a double-containment nuclear shipping container; and

FIG. 4 is a graph of the compressive stress and fractional volume change when compressive stress is applied to the solidified product of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

In accordance with this invention, it has been found that a protective material is needed that is fire-resistant and is crushable to serve as an shock absorbing and impact limiting material for insertion into the void spaces 5 formed between the inside walls of a shipping container 1, and the outer walls of a containment vessel 7 for hazardous and/or nuclear materials. In accordance with FIGS. 1–4, the preferred embodiment for the present invention is a fireproof, impact limiting, homogeneous aggregate material (HAM)3. The homogeneous aggregate material 3 is in a granular form when dry, and is in a porable form after water is mixed in (see FIG. 2), until the mixture is poured into a shipping container, vibrated and allowed to stand, cure and solidify for numerous days, and baked at high temperatures for numerous days, forming a solid mass inside the shipping container (see FIG. 3) after a cover plate is welded over the fill hole.

The homogeneous aggregate material is a combination of portland cement and inorganic vermiculite. The material of the present invention does not contain hydrocarbon compounds as does the prior art. Most existing, commonly-used, internal packaging materials utilized in hazardous and/or radioactive shipping containers, contain some type of carbon, or hydrocarbon-based, internal packaging and insulating material which is flammable after the appropriate combustion temperature is reached during test scenarios. The internal impact limiting and insulating material 3 (HAM) is placed in the void spaces 5 of an outer container 1 which occur between:

(a) the interior surface 6 of the walls of the outer shipping container 1;

(b) the walls of an interior encapsulating jacket 6 or stainless steel liner of material that covers the exterior surface of the internal nuclear material containment vessel 7 placed inside the outer shipping container 1; and

(c) the top lid 29 and top plug unit 21 that seals the upper portion of the shipping container 1.

The homogeneous aggregate material 3 is also placed in the void space 5 in the top plug unit 21 of the shipping container 1. The top plug unit 21 forms an upper barrier for impact absorption and insulation from the top of the interior containment vessel 7, then a drum lid 8 is bolted onto the top of the outer shipping container 1 (see FIGS. 2 and 3). The lid 8 of the interior containment vessel 7 has separate bolts, and O-ring seals made of ethylene-propylene material, for sealing of the lid 8 of the interior vessel 7 onto the interior containment vessel 7.

The invention provides for utilization of a homogeneous aggregate material 3 as an internal containment vessel 7 packing or encapsulating material, which solves numerous problems incurred by the use of hydrocarbon-based packaging materials because the invention is fireproof, shock absorbent, and castable into any shape, providing additional safety to reduce the possibility of a worst-case breach of nuclear material transport containers.

The homogeneous aggregate material 3 is composed of two main inorganic components. One of the main inorganic components is portland cement, which is typically composed of: lime, alumina, silica, iron oxide, tetracalcium aluminoferrate, tricalcium aluminate, tricalcium silicate, and dicalcium silicate in varying amounts, along with small amounts of magnesia, sodium, potassium, and sulfur. The other main component of the homogeneous aggregate material is an inorganic vermiculite, which is mixed with the portland cement.

One type of inorganic vermiculite and portland cement mixture utilized in tests is commercially available from Thermal Ceramics, Inc., of Augusta, GA, under the trade name of Kaolite™ 1600. The inorganic mixture tested was composed of:

approximately 10% aluminum oxide (alumina),

approximately 37% of silicon dioxide (silica),

approximately 6.7% of ferric oxide,

approximately 1.2% of titanium oxide,

approximately 30% of calcium oxide,

approximately 13.1% of magnesium oxide, and

approximately 2% of sodium monoxide.

The portland cement and inorganic vermiculite aggregates form a homogeneous, rigid mass after water is added and the mixture is allowed to stand in a shipping container and cure for approximately two days, followed by a high temperature baking for approximately two days.

One preferred embodiment of the process of mixing and forming the homogeneous aggregate material 3 in an outer shipping container 1, for protection of the interior containment vessel 7, includes the following steps in reference to FIGS. 1-3.

(A) Provide a stainless steel shipping container 1 weighing approximately 95 pounds and having approximately 5.0 cubic feet of void space 5 between the outer shipping container 1, and the walls of an interior encapsulating jacket 6 or stainless steel liner, into which an inner containment vessel 7 is placed. The void space 5 is to be filled with the wet mixture 35 of inorganic vermiculite, portland cement and water, for a wet cast weight of approximately 400 pounds.

(B) Mix approximately 122 pounds of inorganic vermiculite and portland cement with approximately 183 pounds of water slowly in a mixer container 11 until thoroughly mixed (see FIG. 1).

(C) Place the drum shipping container 1 upside down 13 onto a shaking or vibrating table (not shown). Shake or vibrate the shipping container 1 at approximately 1.5 to 2 times the wet cast weight (750 pound-force) at 2,000 vibrations per minute, while pouring the wet mixture 35 into the drum shipping container void space 5 through a bottom pour hole 15 (see FIG. 2).

(D) Continue the vibrations for a time period of at least five minutes after the drum of the shipping container 1 is full.

(E) Shake or vibrate the shipping container 1 at approximately 750 pound-force at 2,000 vibrations per minute, while pouring the wet mixture 35 into the bottom pour hole 15 in the bottom surface or bottom head 17 of the outer shipping container 1, and vibrate for at least 5 minutes after filling.

(F) While the mixture 35 is solidifying inside the shipping container 1 (see next step), a similar wet mixture 35 of inorganic vermiculite, portland cement, and water is poured into the top plug unit 21, through an opening, into the void space 23 (see FIG. 1A), while vibrating the top plug unit 21 for at least 5 minutes after filling.

(G) Allow the mixture to solidify within the shipping container 1 and within the top plug unit 21, over approximately 24 to 48 hour period, at room temperature. The temperature of at least approximately 60, and up to approximately 90 degrees fahrenheit is preferable.

(H) Bake the solidified mixture 31 (FIG. 3), inside the shipping container 1, and in the top plug unit 21, in a gas-fired or forced convection fresh air circulating electric furnace (not shown), over at least approximately 48 hours, beginning at 200 degrees fahrenheit for approximately 4 hours, and increasing the temperatures by approximately 75 degrees every hour, until approximately 500 degrees is reached, with baking at approximately 500 degrees for approximately 36 to 40 hours, for a total bake period of approximately 48 hours.

(I) Cool the solidified and baked mixture 31 within the shipping container 1, and within the top plug unit 21, to approximately room temperature. The finished weight for the solidified and baked mixture 31 in the shipping container 1 is approximately 245 pounds, and the finished density is approximately 30 pounds per cubic foot.

(J) Weld a bottom cover plate 25 over the bottom pour hole 15 in the outer shipping container 1 (see FIGS. 1 and 3).

(K) Weld the fill hole cover plate 22 over the top pour opening in the top plug unit 21 (see FIG. 1a).

(L) Assemble the finished shipping container 1 (FIG. 3) in the following order; load the containment vessel 7 with the radioactive and/or hazardous materials, seal the containment vessel top 8, and fasten with bolts, lower the assembled containment vessel 7 into the interior encapsulating jacket 6 inside the outer containers center void 9 (now filled with solidified homogeneous material 31, place the top plug unit 21 over the containment vessel 7, install the shipping container lid 29 with its fasteners 27.

After baking, cooling, and assembly of the shipping container, the homogeneous aggregate material has approximately 4–5 pounds per cubic foot of residual water bound in the solidified material, potentially serving as a neutron absorbing and heat dissipating component of the homogeneous aggregate material 31.

A second embodiment for shipping containers utilized for transport of neutron emitting nuclear materials, is the addition of natural boron, enriched boron, compounds containing boron (i.e. boron carbide), or compounds containing gadolinium, cadmium, europium, hafnium, samarium, indium alloys, or other neutron absorbing compounds mixed into the homogeneous aggregate material. The addition of boron compounds, or other neutron absorbing compounds, to the mixture before solidification, provides a nonvolatile neutron absorbing additive to the homogeneous aggregate material 3. The above described steps of mixing, pouring, curing, baking, and cooling are utilized, with the boron compounds, or other neutron absorbing compounds, mixed into the mixture of portland cement, vermiculite, and water at the mixing step, before the wet mixture is poured into the voids of the shipping container. The percentage of boron, boron containing compounds, or other neutron absorbing compounds added to the wet mixture is variable and is dependent on the radioactivity of the materials stored in the inner containment vessel. As explained earlier, the baked and cooled homogeneous aggregate material 3 has approximately 4–5 pounds per cubic foot of water remaining in the solidified material, potentially serving as a neutron absorbing component of the homogeneous aggregate material 3.

The benefits of the homogeneous aggregate material 3 are numerous when compared to the prior art. Current packaging and shock-inhibiting materials utilize hydrocarbon- or carbon-based materials for the interior voids 5 of shipping containers 1 containing interior nuclear material containment vessels 7. The carbon-based materials will eventually burn and release toxic fumes, or may add to the internal heating of a containment vessel 7 of nuclear materials. The silicon, aluminum, ferric, magnesium and calcium composition of inorganic vermiculite and portland cement will not burn when cured and hardened inside a shipping container 1. The cured mass of homogeneous aggregate material provides a castable, non-flammable, packaging material that serves as a thermal insulator for any enclosed containment vessel 7 of hazardous chemical and/or nuclear materials. The cured mass has a very low capacity to store heat, therefore providing outstanding insulating properties from exposures to high or low temperatures. The cured mass does not expand appreciably when heated to high temperatures. The melting temperature of the cured mass is approximately 2335 degrees fahrenheit, which is higher than the stainless steel outer container 1 that is typically utilized for transport of nuclear materials.

Testing results for the solidified, cured, and baked homogeneous aggregate material 3 inside a shipping container 1 have verified the insulating capabilities of the claimed invention. Testing has subjected stainless steel shipping containers 1, with an internal containment vessel 7 surrounded by the solidified, cured, and baked homogeneous aggregate material 31 encapsulated by a ductile jacket 6, to temperatures of 1525 degrees Fahrenheit at the outer surface of the walls of the shipping container 1 for over 34 minutes. The ductile jacket 6 is composed of the stainless steel interior wall and the exterior wall of the drum shipping container (see FIG. 2) 1. The maximum temperature measured on the interior wall 6 of the encapsulated material was approximately 215 degrees Fahrenheit, with a maximum measured temperature at the exterior of the containment vessel 1 of 150 degrees Fahrenheit. The heat is also dissipated by some of the approximate 4–5 pounds per cubic foot of water left inside the HAM after curing and baking, that evaporates during the fire test, venting steam away through vent holes from the interior wall that protects the containment vessel. Vent holes in the containment vessel 7 and shipping container 1 are drilled after the HAM 3 is cured, and fusible plastic hole plugs 33 are placed in the vent holes. In summary, internal nuclear and/or other hazardous containment vessels 1 are protected from destructive temperatures at the containment vessel lids 8, which have O-ring seals which deteriorate over 350 degrees Fahrenheit, by the homogeneous aggregate material, placed inside the shipping container 1 and serving as an encapsulating jacket 6 around the containment vessel 7.

A second major benefit is the impact limiting properties of the homogenous aggregate material 3 when solidified in a ductile jacket 6 within the shipping container 1, provides a brittle structure that is frangible when subjected to impacts (see FIG. 4). As the shipping container 1 is subjected to impacts, the brittle structure of the HAM fractures, crushes, and powders, dissipating the force around the interior containment vessel 7. Because the brittle homogenous aggregate material 3 directs fractures from the impact in many different directions, the HAM 3 does not delaminate along one specific plane. The stress-strain curve (see FIG. 4) shows the energy absorbing capabilities of the brittle structure of the homogenous aggregate material 3 formed into an encapsulating jacket 6. Therefore, the encapsulating jacket 6, and the brittle structure of the homogenous aggregate material 3, provides insignificant pathways for flames, radiation of heat, or hot gasses to reach the internal containment vessel 7.

A third and less obvious benefit of the homogeneous aggregate material 3 is that the inorganic vermiculite material is non-toxic in a dry condition, and is castable into a multitude of shapes when water is added, with the final form being non-toxic also. During curing of the aggregate material inside the shipping container 1, and during any thermal testing for package certification purposes of the shipping container 1 with the aggregate material inside, the only offgas formed is water vapor, which is non-toxic. As discussed above, the cured inorganic mass does not burn, and no toxic offgasses such as hydrocarbons or tars are formed when the cured mass approaches its melting temperature.

A fourth benefit of the homogeneous aggregate material 3 is the low cost for the materials, and the low cost to prepare a rigid mass of the material. Costs of $12.00 to $14.00 per cubic foot have been calculated for the raw materials, which is significantly less than current rigid polyurethane foam and high-density fiberboard insulation utilized for Type-B nuclear and/or hazardous material shipping containers 1. As emphasized above, insulating material with hydrocarbon- or carbon-based materials, such as wood, polyurethane foam, and high-density fiberboard insulation, are prone to ignite above each material's combustion temperature, which requires complete removal and cleaning of a container containing hydrocarbon-based insulation when subjected to high, ignition temperatures. The invention of homogeneous aggregate material 3 does not ignite, and will not require interior cleaning of a container utilizing the material when subjected to high temperatures up to the material's melting point, which is above the melting point of the exterior stainless steel shipping container 1.

A fifth benefit of the homogeneous aggregate material 3 encapsulated inside a stainless steel shipping container 1 is the low cost to maintain the shipping container 1. The organic compounds described earlier for use as impact limiting and thermal insulating materials tend to age and breakdown when exposed to severe temperature, humidity changes, and rough handling, will require periodic replacement. Replacement of the impact limiting and thermal insulating material generates a recurring maintenance cost for the life of the shipping package 1. The homogeneous aggregate material 3 of the present invention, when placed inside the exterior shipping container 1, has been subjected to over 42,000 miles of simulated endurance vibration testing with a fully loaded containment vessel 7. Radiographs have shown that the internal structure of the homogeneous aggregate material 3 will fracture, crush, and be reduced to powder from the endurance vibration and impact testing. Additional thermal (direct flame or indirect heating) testing on this container 1 has shown no significant loss of effectiveness in its impact limiting and thermal insulating properties, when incorporated with the internal homogeneous aggregate material 3. Therefore, there is no projected cost to replace the internal contents of the exterior shipping container 1, even if the structure of the internal homogeneous aggregate material fractures. There is no appreciable loss of reduction of properties of the internal homogeneous aggregate material 3 within the shipping container 1 over the life of the shipping container 1.

Although the present invention has been described in considerable detail with reference to a preferred version thereof, other versions are possible. For example, the materials of the apparatus may utilize a different inorganic vermiculite composition, or a similar composition of a non-carbon based, homogeneous aggregate of materials which include a solidifying agent such as portland cement. The percentage of alumina, silica, and other non-carbon oxide compounds may be varied from the percentages described above.

The configuration of the inorganic homogeneous aggregate material can be of any shape when solidified, providing impact protection and crush limitations for any stress on the exterior shipping container 1 in any direction. The shape of the material conforms to the shape of the container in which the material is cured (see FIG. 3). The solidified inorganic homogeneous aggregate material 3 fills the void spaces 5 between the interior contaminent vessel 7, and the exterior walls of the outer shipping container 1. The containment vessel 7 is placed inside the outer container 1 with the top plug 21 over the containment vessel's lid 8. By this method of encapsulating a container 1 with a rigid, nonflammable, inorganic matrix of crushable material, the internal containment vessel's high hazard materials is protected from temperature extremes and from impacts to the exterior shipping container 1.

The process mixing and curing steps can be varied by allowing for additional mixing and vibrating time for the mixture inside the shipping container 1, and by providing a longer curing and heating time without detriment to the final rigid form of the inorganic homogeneous aggregate material 3.

Many variations will undoubtedly become apparent to one skilled in the art upon a reading of the above specification with reference to the drawings. Therefore, the spirit and scope of the appended claims should not be limited to the description of the preferred versions contained herein.

What is claimed is:

1. A shipping container comprising:

1) an outer container having a wall, a bottom, a dismountable cover and a hollow interior;

2) a liner having sides and a bottom being smaller in every dimension than said outer container;

3) an inner container having a wall, a bottom, a dismountable cover and a hollow interior, said inner container being smaller in every dimension than said liner; and

4) a thermally insulating and energy-absorbing material disposed between said inner container and said outer container in all directions, said thermally insulating and energy-absorbing material consisting essentially of a mixture of

a) vermiculite;

b) portland cement;

c) water; and

d) air, constituting a void volume;

said mixture having been dried after being packed between said liner and said outer container to contain not more than 5 pounds per cubic feet of water and to exhibit a compressive stress no greater than 500 psi over a range of 10 to 40% compressive strain.

2. A container according to claim 1 wherein that portion of said thermally insulating and energy absorbing material disposed between the cover of said inner container and the cover of said outer container is removable.

3. A shipping container according to claim 1 wherein said thermally insulating and energy absorbing material has a density of approximately 30 pounds per cubic foot.

4. A shipping container according to claim 1 wherein said thermally insulating and energy absorbing material further comprises a component having a high neutron absorption cross section.

5. A shipping container according to claim 4 wherein said component having a high neutron absorption cross section is selected from the group consisting of boron, boron compounds, gadolinium, gadolinium compounds, cadmium, cadmium compounds, europium, europium compounds, hafnium, hafnium compounds, samarium, samarium compounds, and indium alloys.

6. A shipping container according to claim 1 further comprising at least one pressure relief valve in said outer container.

* * * * *

Thermo
ELECTRON CORPORATION

Catalog 277-4 Special Dry Mix

Note: Read all instructions before starting

Catalog Type 277-4 special mix containing 4% boron content minimum

	Percent	For 300 lb batch use
Component A		
Component B		
Component C		
Component D		
Component E		

Mixing Room Preparations

A. Clean mixer by vacuuming out any previous use.
B. Place all needed materials in mixing room , insure lot numbers maintained on those items . (B4c)
C. Using calibrated weight verifies platform scales are accurate (done at beginning and mid shift as well as end of shift . Note in small lots beginning and end is adequate. Note use scale log sheet and second person to verify weight.
D. Get labels for every batch or barrel . Note use day of manufacture for batch # and sequence barrels 1 through ?

Mixing

A. Verify boron carbide material is of correct mesh size –40+200 mesh and that the content is no less than 78% minimum boron. See supervisor or check work sheet.
B. Weigh materials out in plastic buckets. " use scale check form "
C. Pour components into mixer
D. Close and lock lid . Mix for 3-5 minutes
E. While mixing is in process weigh another batch.
F. Place barrel under the mixer on platform
G. Shut off mixer , rotate hopper to lower position this allows material to fill drum .
H. Turn on mixer , re-engage the paddles to empty mixer . Some shaking of barrel may be necessary to insure material fits container.
I. Disconnect the lid from barrel and rotate mixer to upright position .
J. Install barrel lid and place ring on unit . Note visual required as to material level.
K. While next barrel is mixing move barrel to platform scale and verify weight . Notify supervisor of any discrepancies. Weight 300 lbs.
L. Move barrel off scale and stage for palletizing .

CANBERRA

Canberra Oak Ridge, LLC
1133-C Oak Ridge Turnpike, Suite 260
Oak Ridge, TN 37830-6442

Ltr. No.: COR-NDA-04-93

Date: January 5, 2005

To: D. B. Miller

c: G. A. Byington, J. F. DeClue, L. C. Ostrowski, D. A. Tollefson, File

From: R. W. Brandenburg

Ref: BWXT-4300038751

Subject: **Results of Prompt Gamma-ray Neutron Activation Analysis and Neutron Transmission Measurements on Prototype Confinement Vessel Inner Liners and Spacers**

INTRODUCTION

Canberra was contracted by BWXT to perform prompt gamma-ray neutron activation analysis (PGNAA) on confinement vessel inner liners (CVIL) for the ES-3100 prototype replacement for the 6M shipping container and spacers to be used in the containers. Neutron transmission measurements on the CVILs are also part of the project. The CVILs are cylinders with an inside diameter of 6.5 in. and outer diameter of 8.5 in. The shell of the CVIL is made of 304L stainless steel and the annulus is filled with high alumina-content cement. For test purposes Canberra was supplied with three CVILs and ten spacers. The cement in two of the test CVILs contains boron in the form of 4 wt% boron carbide (B_4C). The third CVIL contains no boron. The portion of the CVIL filled with the cement is approximately 31 in. tall. The spacers are in 4-in. diameter tin coated steel cans that are 1.25 in. thick. Four of the ten spacers contain 4 wt% boron carbide while the other six contain no boron. Some of the cement mixture from each pour is used to make spacers as well as fill the annulus of the cylinder.

The measurements for the project had three parts:

1. Twelve PGNAA measurements using a BEGe gamma-ray detector were made at 30° increments around each CVIL at the center height of the CVIL.

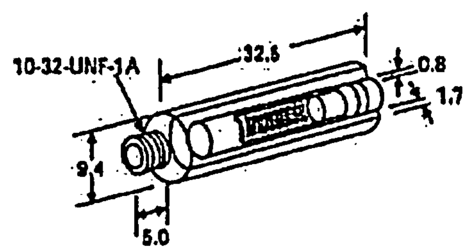
2. Two PGNAA measurements using a BEGe gamma-ray detector were made on each spacer with a 180° rotation of the spacer between the measurements.

3. Sixty neutron transmission measurements were made on each CVIL using a BF_3 neutron detector. The measurements were made at 30° increments around each CVIL at five heights starting at five inches from the bottom of the CVIL and at five inch increments up the CVIL.

NEUTRON SOURCE CONFIGURATION

A 1.8-µg ^{252}Cf spontaneous fission neutron source was purchased from AEA Technology to be the neutron source for the tests. The source was rated at $4.4 * 10^6$ neutrons/sec. The source is sealed in a 32.5 mm long, 9.4 mm diameter stainless steel cylinder (Fig. 1).

X.224 - stainless steel capsule



Safety performance testing

ANSI/ISO classification	IAEA special form	Model no.
C64545	GB/204/S	CVN.CY6

Fig. 1. Neutron Source Capsule

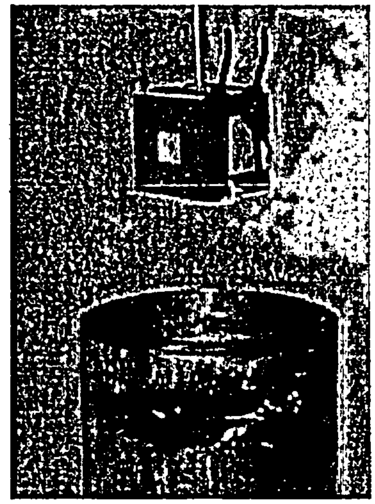


Fig. 2. Neutron Source Holder

A collimator was fabricated from high density polyethylene to hold the source. The collimator is a 3-in. cube of polyethylene covered on five sides with 2 mm of cadmium. The sixth side is covered with cadmium except for a 1-in. square in the center. A 9.5-mm hole in the center of the 1-in. square is the source location (Fig. 2). The collimator, which is suspended from a rod attached to the ceiling, is small enough to fit down the center of the CVILs. The source in the collimator is approximately 54 in. from the concrete floor. The detectors were placed on a Genie lift and centered on the source. The CVIL was placed on a second Genie lift and centered within ± 1/16 in. under the collimator. The lift was used to raise the CVIL to the designated height within ± 1/16 in. for the measurements. The spacers were clamped in a small vise and placed on the second lift and centered 1/2 ± 1/16 in. in front of the source. For the gamma-ray measurements, the detector to CVIL distance was 30 ± 1/8 in. and the detector to spacer distance was 20 ± 1/8 in. The neutron detector to CVIL distance was 4 ± 1/8 in.

GAMMA-RAY METHOD

The gamma-ray detector was a BEGe detector from Canberra in a 2-in thick lead shield.

attached to the face of the detector and counted for 60 seconds. One gamma-ray peak from each of the sources was analyzed for peak area and width (FWHM) and compared with previous measurements.

Prompt neutron activation of boron (specifically ^{10}B) gives rise to a 478 keV gamma ray produced by the recoil of the lithium nucleus from the neutrons captured in ^{10}B. The gamma ray is Doppler broadened by 10-15 keV. Gamma-ray spectrum collection and analysis was performed using the Genie2K gamma-ray analysis software package from Canberra. The peak fitting routine in Genie2K does not expect a peak as broad as 10-15 keV, therefore a region-of-interest (ROI) was set around the broad boron neutron activation peak. The data has three components. The gross count is the total count of the gamma rays detected with an energy that falls within the ROI. The Compton background is the portion of the gross count that is caused by scattered gamma rays. The net count is the portion of the total count that is from the boron. The net count is the difference between the gross and Compton counts.

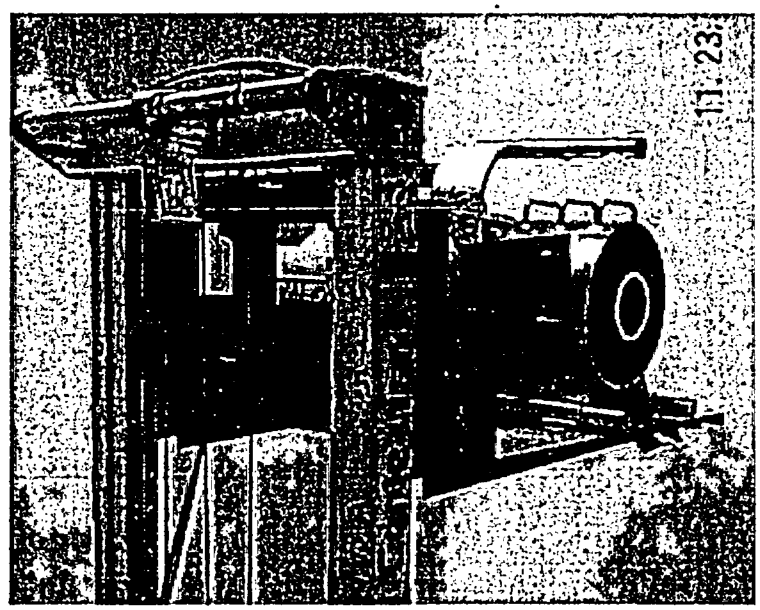


Fig. 3. BEGe Gamma-Ray Detector and Collimator

GAMMA-RAY RESULTS

The two CVILs containing boron were labeled 277-4-C1 and 277-4-C2. The CVIL containing no boron was labeled 277-0. CVIL 277-4-C1 was filled with three pours. CVILs 177-4-C2 and 277-0 were each filled with one continuous pour. Each CVIL in-turn was centered under the source collimator on the turntable on the Genie lift. The CVIL was raised to position the center of the CVIL at the level of the source for the twelve measurements (Fig. 4). Each measurement consisted of collecting a spectrum for 10 minutes. The spectrum was saved and counts in the ROI were recorded. Table 1 contains the results of the PGNAA measurements on the three CVILs. Chart 1 is a plot of the results. The average net counts for the two boron containing CVILs, agree very well. The net count for the non-boron containing CVIL is small and probably due to some minor activation of other materials in or near the CVIL. In all cases, CVILs and spacers, the standard deviation of the net counts and gross counts was less than or close to the standard deviation that would be expected based on counting statistics. The Compton background for all three CVILs agrees well, implying that there are no major effects from other materials.

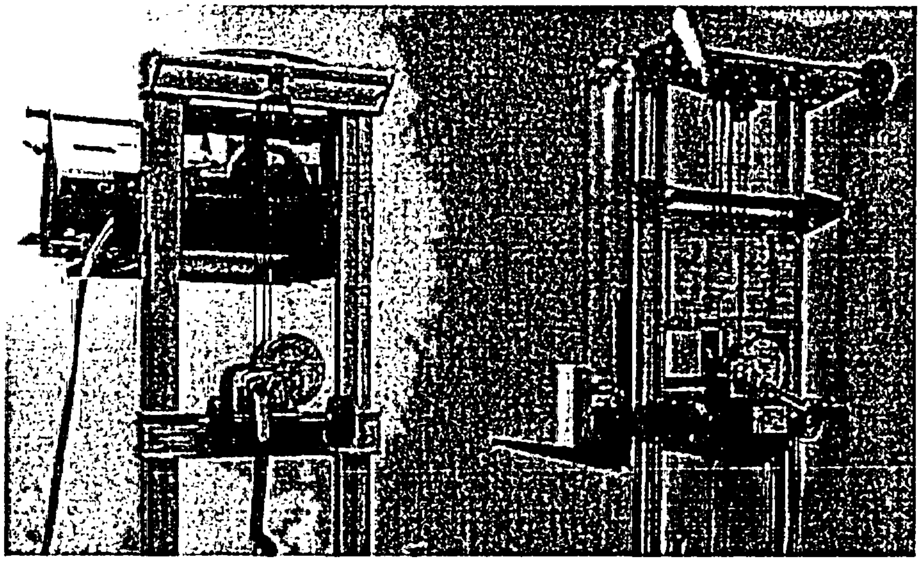


Fig. 4 BEGe Detector and CVIL Positioned for PGNAA Measurement

CVIL	277-4-C1			277-4-C2			277-0		
Angle	Gross Counts	Compton	Net Counts	Gross Counts	Compton	Net Counts	Gross Counts	Compton	Net Counts
0	39,002	20,658	18,344	39,403	20,816	18,587	21,552	20,066	1,486
30	39,038	21,152	17,886	39,681	21,211	18,470	21,394	20,392	1,002
60	39,378	21,429	17,949	39,110	20,925	18,185	21,145	20,184	961
90	39,306	20,036	19,270	39,890	21,732	18,158	21,347	21,123	224
120	39,287	20,421	18,866	39,407	21,123	18,284	21,463	19,661	1,802
150	39,126	20,895	18,231	39,573	20,905	18,668	21,367	20,500	867
180	39,218	21,241	17,977	39,696	21,093	18,603	21,327	19,977	1,350
210	39,356	21,468	17,888	39,693	20,856	18,837	21,547	19,682	1,865
240	39,258	20,570	18,688	39,565	20,965	18,600	21,317	20,382	935
270	39,351	20,658	18,693	39,762	21,300	18,462	21,424	19,918	1,506
300	39,281	20,431	18,850	39,470	20,955	18,515	21,576	20,155	1,421
330	39,406	21,034	18,372	39,574	20,708	18,866	21,460	20,984	476
Average	39,251	20,833	18,418	39,569	21,049	18,520	21,410	20,252	1,158
Std. Dev.	132	444	456	204	274	227	122	457	503
Rel SD	0.3%	2.1%	2.5%	0.5%	1.3%	1.2%	0.6%	2.3%	43.4%

Table 1. Results of PGNAA on CVILs

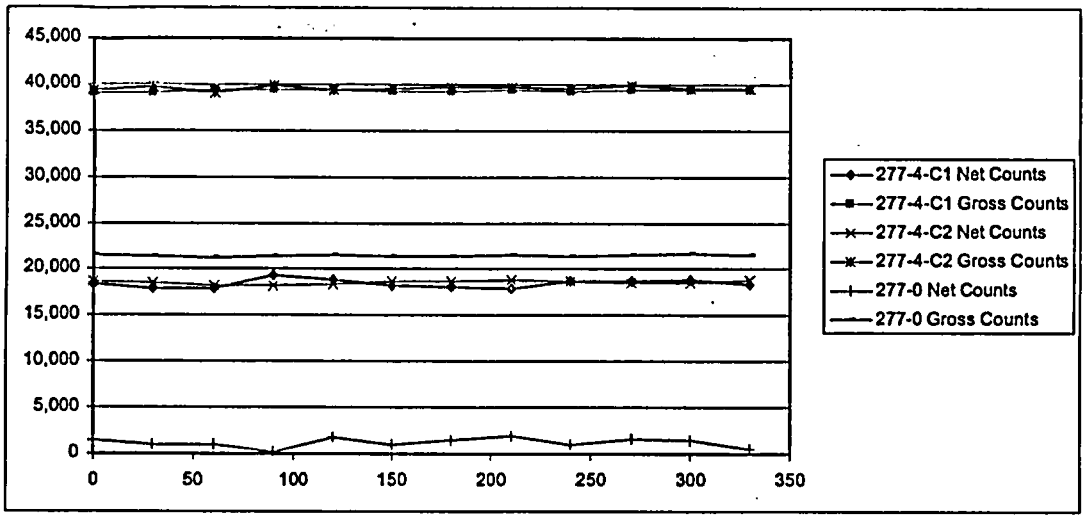


Chart 1: Results of PGNAA on CVILs

The spacers were measured by centering them vertically in front of the source (Fig. 5).

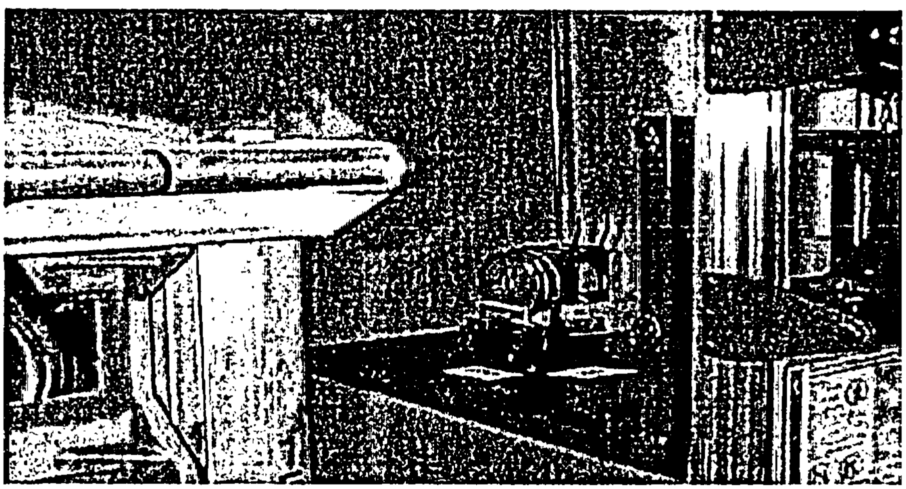


Fig. 5. Spacer Positioned for PGNAA Measurement

A 15-minute spectrum was collected then the spacer was rotated 180° and a second 15-minute spectrum collected. Each spectrum was saved and the counts in the ROI were recorded. The spacers with no boron were labeled 277-0-S1 through –S6 and the boron containing spacers were labeled 277-4-S1 through –S4. Side A is with the cover of the can facing the detector and side B is with the cover facing the source. Table 2 contains the results from the measurements of the ten spacers. Chart 2 is a plot of the results.

Spacer Number	Gross Count	Compton	Net Count	Spacer Number	Gross Count	Compton	Net Count
277-4-S1-A	72,749	61,156	11,593	277-0-S1-A	65,385	61,738	3,647
277-4-S1-B	71,471	59,605	11,866	277-0-S1-B	63,964	59,280	4,684
277-4-S2-A	74,181	62,844	11,337	277-0-S2-A	63,861	59,744	4,117
277-4-S2-B	73,478	60,761	12,717	277-0-S2-B	62,261	56,850	5,411
277-4-S3-A	75,877	62,548	13,329	277-0-S3-A	64,510	59,526	4,984
277-4-S3-B	73,651	60,415	13,236	277-0-S3-B	63,653	59,684	3,969
277-4-S4-A	75,194	61,778	13,416	277-0-S4-A	64,952	60,544	4,408
277-4-S4-B	72,634	59,833	12,801	277-0-S4-B	62,865	59,961	2,904
Average	73,654	61,118	12,537	277-0-S5-A	65,635	60,188	5,447
Std Dev	1,427	1,197	826	277-0-S5-B	63,881	59,161	4,720
Rel SD	1.9%	2.0%	6.6%	277-0-S6-A	64,041	59,921	4,120
				277-0-S6-B	64,154	60,366	3,788
				Average	64,211	59,919	4,293
				Std Dev	839	455	786
				Rel SD	1.3%	0.8%	18.3%

Table 2. Results of PGNAA on Spacers

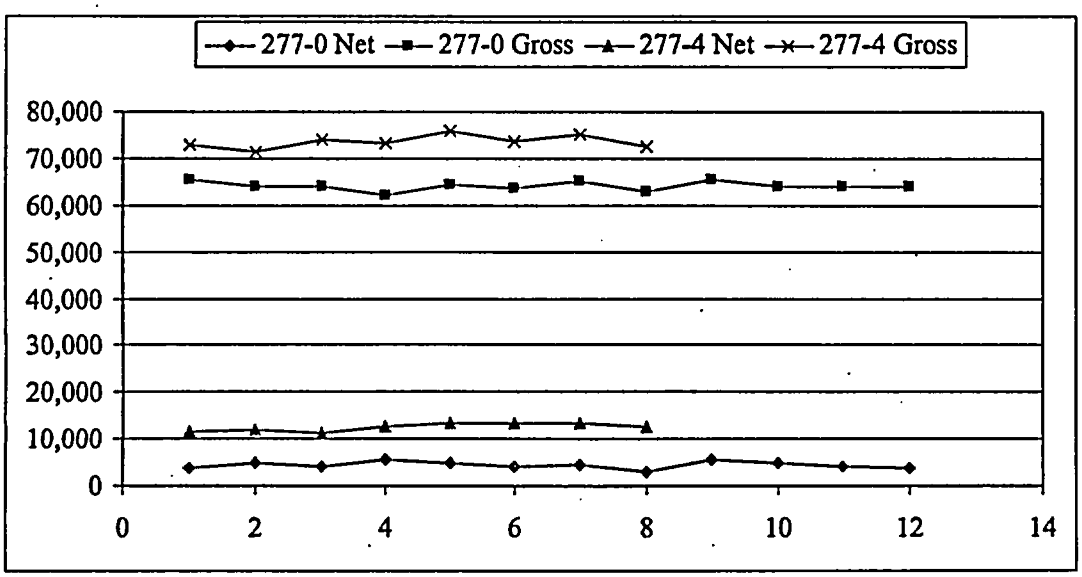


Chart 2. Results of PGNAA on Spacers

The results for the spacers show less difference between the spacers with and without boron than the CVILs. The variation among the measurements is larger for the spacers. Direct comparison between the CVIL results and the spacer results is difficult because the source to sample distance, the detector to sample distance, the area of the sample exposed to the source, and the count times are all different.

NEUTRON METHOD

The neutron detector was a 1-in.diameter BF_3 tube surrounded by a cylindrical block of polyethylene. The polyethylene is inside an aluminum case lined with a sheet of cadmium. The cadmium liner is intended to stop thermal neutrons from the surroundings and allow only fast neutrons from the source through to be moderated by the polyethylene and detected by the BF_3 tube. There was no additional shielding around the detector (Fig. 6). The detector assembly and 2241-7 handheld electronics package were from Ludlum Measurements, Inc. Quality assurance (QA) measurements were made by counting the neutron source with no CVIL in the beam of neutrons. The measurement results were entered into the Measurement Control software used by Canberra at ETTP.

The CVIL was positioned as in the gamma-ray measurements. Measurements were made at 60 points on each CVIL. As with the gamma-ray measurements, 12 measurements were made at 30° increments around the CVIL but these measurements were made at five different heights on the CVIL. The measurements were made at heights of 5, 10, 15, 20, and 25 inches from the bottom of the CVIL. The neutrons were counted for five minutes at each position then the CVIL was rotated to the next measurement position. The zero degree starting positioned was remeasured after the full rotation. The CVIL was the moved to the next height and the process repeated.

NEUTRON RESULTS

For all three CVILs, the standard deviation of the counts at any level on the CVILs was about 1.5 times the standard deviation expected from counting statistics. This indicates that there is little true variation in the neutron transmission around the circumference of the CVILs. The variation of the counts with height was larger. The standard deviation of the counts at different heights was 3 to 4 times the standard deviation expected from counting statistics for CVILs 277-4-C1 and 277-0, the three pour CVILs, but only 1.7 for 277-4-C2, the one pour CVIL. For both of the CVILs containing boron, the measurements 5 inches from the bottom yielded the highest transmission and the measurement at 25 inches from the bottom yielded the lowest transmission. This effect is presumed to be due to neutron scattering from the floor. The floor was more exposed to the neutron source when the CVIL was at maximum height (the 5-in position) than when the CVIL was down further and provided some shielding. The average transmission when compared to the measurement of the source with no CVIL is 81.4% for 277-4-C1, 83.6% for 277-4-C2, and 85.3% for 277-0. One interesting point is on 277-4-C1 at 150° and 25 inches. The results for that point were 4.4% higher that the average at the 25-in. level (Chart 3). The measurement was repeated with similar results. No clear cause of this anomaly could be determined without further investigation. Tables 3, 4, and 5 and Charts 3, 4, and 5 show the results of the neutron measurements on the three CVILs.

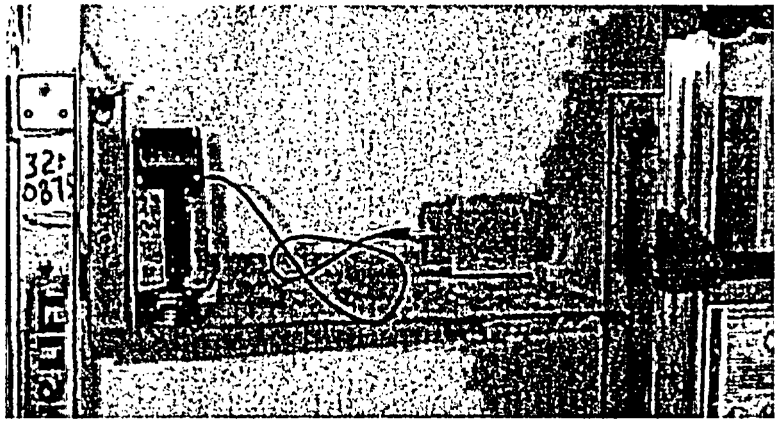


Fig. 6. Neutron Detector and CVIL Positioned for Transmission Measurements

Angle (°)	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Mean	Std Dev
0	5	20,954	10	20,249	15	20,142	20	20,041	25	19,544	20,186	507
30		21,072		20,319		20,463		20,329		19,533	20,343	548
60		21,387		20,600		20,739		20,260		19,655	20,528	637
90		21,310		20,371		20,398		19,889		19,620	20,318	645
120		21,168		20,834		20,500		20,500		19,425	20,485	654
150		21,288		20,665		20,741		20,434		20,398	20,705	357
180		21,185		20,675		20,840		20,282		19,624	20,521	598
210		21,237		20,581		20,162		20,025		19,361	20,273	695
240		20,980		20,593		20,273		20,046		19,300	20,238	631
270		20,933		20,342		20,189		19,966		19,287	20,143	598
300		21,126		20,754		20,404		20,064		19,586	20,387	597
330		20,909		20,345		20,295		20,086		19,707	20,268	437
360		20,831		20,220		20,490		20,111		19,725	20,275	415
Mean		21,106		20,504		20,434		20,156		19,597	20,359	
Std Dev		175		204		229		187		282	538	

Table 3. Results of Neutron Transmission Measurements on CVIL 277-4-C1

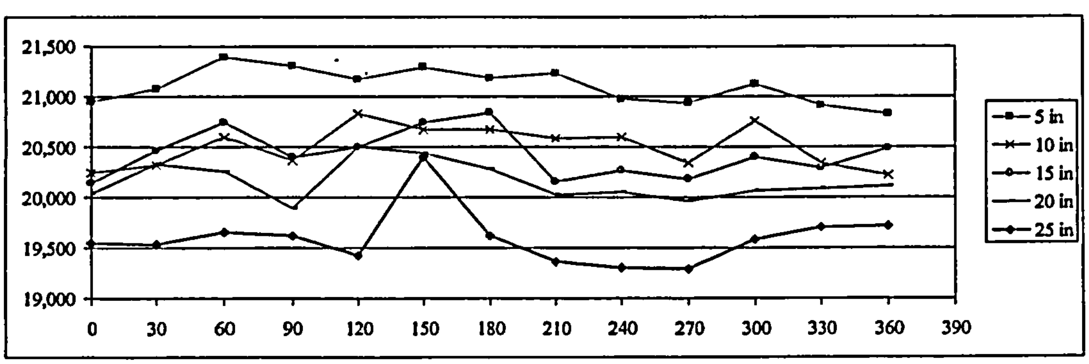


Chart 3. Results of Neutron Transmission Measurements on CVIL 277-4-C1

Angle (°)	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Mean	Std Dev
0	5	21,451	10	20,985	15	20,857	20	20,916	25	20,188	20,879	452
30		21,444		21,094		20,998		21,028		20,259	20,965	433
60		21,382		20,974		20,714		20,674		19,978	20,744	513
90		21,563		20,960		20,819		20,655		20,230	20,845	486
120		21,280		21,172		20,926		20,839		20,334	20,910	368
150		21,416		20,998		21,149		20,697		20,371	20,926	405
180		21,420		20,947		20,802		20,782		19,874	20,765	768
210		21,737		21,140		20,833		20,598		19,915	20,845	737
240		22,032		21,211		20,904		20,911		20,250	21,062	460
270		21,925		21,252		21,269		21,105		20,415	21,193	389
300		21,484		21,243		20,886		20,952		20,417	20,996	377
330		21,391		21,010		20,600		21,085		19,990	20,815	581
360		21,394		20,957		20,609		20,858		20,341	20,832	395
Mean		21,532		21,073		20,874		20,854		20,197	20,906	
Std Dev		226		117		190		166		193	468	

Table 4. Results of Neutron Transmission Measurements on CVIL 277-4-C2

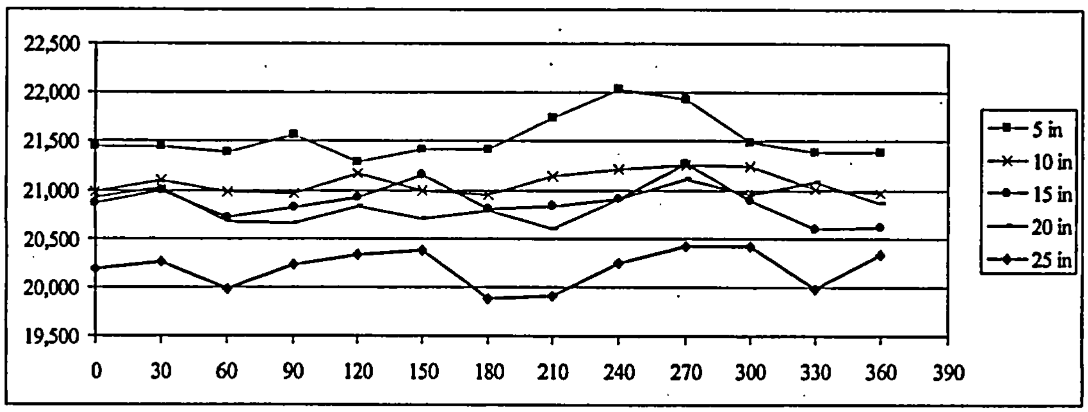


Chart 4. Results of Neutron Transmission Measurements on CVIL 277-4-C2

Angle (°)	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Height (in)	Counts	Mean	Std Dev
0	5	21,752	10	21,329	15	21,377	20	20,946	25	20,624	21,206	433
30		21,376		21,848		21,084		21,243		20,576	21,225	462
60		21,129		21,518		21,172		21,085		20,378	21,056	416
90		21,437		21,482		21,472		21,138		20,588	21,223	383
120		21,743		21,685		21,765		21,265		20,679	21,427	465
150		21,550		21,867		21,535		21,298		20,637	21,377	461
180		21,359		21,939		21,385		21,009		20,524	21,243	522
210		21,456		21,898		21,434		21,346		20,616	21,350	463
240		21,043		21,888		21,754		21,319		20,640	21,329	512
270		21,415		21,954		21,446		21,132		20,682	21,326	466
300		21,495		22,037		21,674		21,411		20,898	21,503	415
330		21,443		22,399		21,847		21,828		20,894	21,682	557
360		21,259		21,542		21,864		21,511		21,022	21,440	317
Mean		21,420		21,799		21,524		21,272		20,674	21,338	
Std Dev		204		283		246		231		172	438	

Table 5. Results of Neutron Transmission Measurements on CVIL 277-0

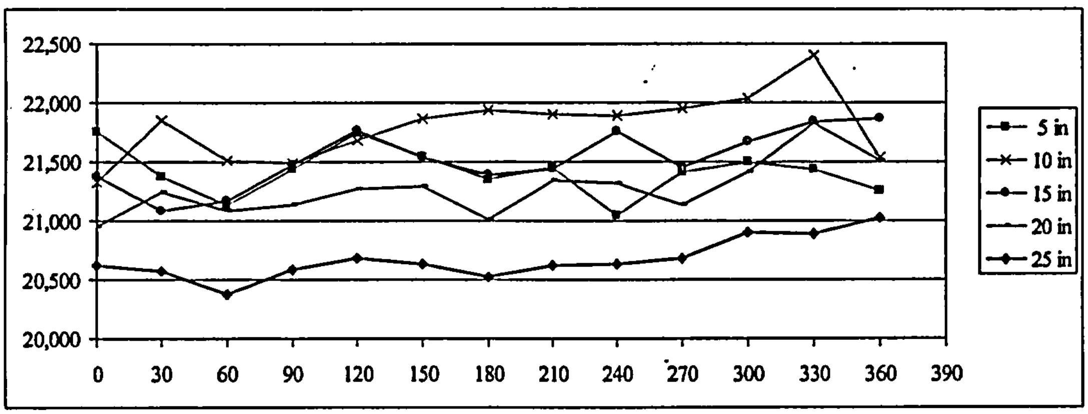


Chart 5. Results of Neutron Transmission Measurements on CVIL 277-0

SUMMARY AND CONCLUSIONS

For the measurement of gamma rays resulting from the prompt neutron activation of ^{10}B, the average net counts for the two boron containing CVILs, agreed very well. The net count for the non-boron containing CVIL is small and probably due to some minor activation of other materials in or near the CVIL. In all cases, CVILs and spacers, the standard deviation of the net counts and gross counts were less than or close to the standard deviation that would be expected based on counting statistics. The Compton background for all three CVILs agreed well, implying that there are no major effects from other materials.

The results for the spacers showed less difference between the spacers with and without boron than the CVILs. The variation among the measurements was larger for the spacers. Direct comparison between the CVIL results and the spacer results is difficult because the source to sample distance, the detector to sample distance, the area of the sample exposed to the source, and the count times were all different.

In the neutron transmission measurements for all three CVILs, the standard deviation of the counts at any level on the CVILs was about 1.5 times the standard deviation expected from counting statistics. This indicates that there was little true variation in the neutron transmission around the circumference of the CVILs. The variation of the counts with height was larger. The standard deviation of the counts at different heights was 3 to 4 times the standard deviation expected from counting statistics for CVILs 277-4-C1 and 277-0, the three pour CVILs, but only 1.7 for 277-4-C2, the one pour CVIL. The result for the measurement on 277-4-C1 at 150° and 25 inches is 4.4% higher that the average at the 25-in. level (Chart 3), but this anomalous result does not lead to any different conclusions than those derived from the other CVIL measurements.

For both of the CVILs containing boron, the measurements 5 inches from the bottom yielded the highest transmission and the measurement at 25 inches from the bottom yielded the lowest transmission. The average transmission when compared to the measurement of the source with no CVIL was in the range from 81.4 to 85.3%.