# Testing and Validation of Threaded Lid Vented Nuclear Materials Storage Containers

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A type 304 stainless steel nuclear materials storage container (NMC) has been developed and tested to verify leak tightness under severe impact. The nuclear materials containers were designed to meet requirements established in DOE's Recommendation 94-1, "Criteria For Interim Safe Storage of Plutonium-Bearing Solid Material". Containers used for the interim storage (less than 20 years) of nuclear materials must allow a means for retrieval and observation of stored contents, while also meeting stringent structural and leak-proof requirements. The new, all stainless steel container has a threaded lid closure that is equipped with a sintered stainless steel high efficiency particulate air (HEPA) filter vent. The container, which can be hand tightened, provides a positive seal for storing nuclear material, yet allows the controlled release of explosive gases including hydrogen.

The nuclear materials container, which was designed for easy retrieval and monitoring, features a lid which can be tightened and released by hand via a handle. To allow the controlled release of gas and simultaneous retention of radioactive particulate, a type 316 stainless steel HEPA filter is welded to the interior surface of the lid. A semi permeable membrane is adhered to the top surface of the container to prevent water entry up to 2 PSI. A handle allows retrieval and lid installation. Figure -1 shows a photograph the nuclear materiel storage container.

Results of air leak tests after a 1.21 meter drop test, hydrogen diffusivity measurements, particle retention and gas permeability, and water intrusion tests on the nuclear material containers (NMC) will be presented.

## DROP TESTS

One test criterion for the container is that, with its packaged contents, it must remain leak tight after a 1.21 meter drop onto an un-yielding surface. The containers were dropped from the specified height in five orientations; flat on the bottom, 45 degrees off of normal on the bottom corner, directly on the handle, direct side impact, and at roughly 45 degrees off of normal on the screw-top. Observations were made of any visible changes to container integrity or appearance. Also, when possible, the exact angle of impact was measured and recorded. Leak tests were conducted by observing the inverted container, immersed in water, for bubbles after the container had been internally pressurized to a minimum of 5" water column.

The package in the test configuration was as nearly duplicated to an actual package as possible. A pewter container, which serves as a radiation shield for 60 Kev gamma radiation, was filled with about 3.6 kilograms of dry sand to simulate the stabilized nuclear material. The pewter can was double bagged; the first bag was a 12 mil polyvinylchloride (PVC) bag with a NucFil®-030 (patented) snap-fit HEPA filter affixed to it, and the second layer was a 3 mil polyethylene bag, also with a NucFil-030 snap-fit HEPA filter affixed to it. Each bag was thermally heat sealed and then trapped air evacuated. The double bagged pewter can full of sand was then inserted into the threaded nuclear material storage container and the screw top threaded on and hand tightened. Figure-2 shows a photograph of the double bagged lead free pewter can being installed into the nuclear material storage container.

The required drop test height is from greater than 1.21 meters onto an unyielding surface. Each container was dropped from about 1.21 meters by hand which allowed the best control for providing impact with the cement floor in the desired orientation.

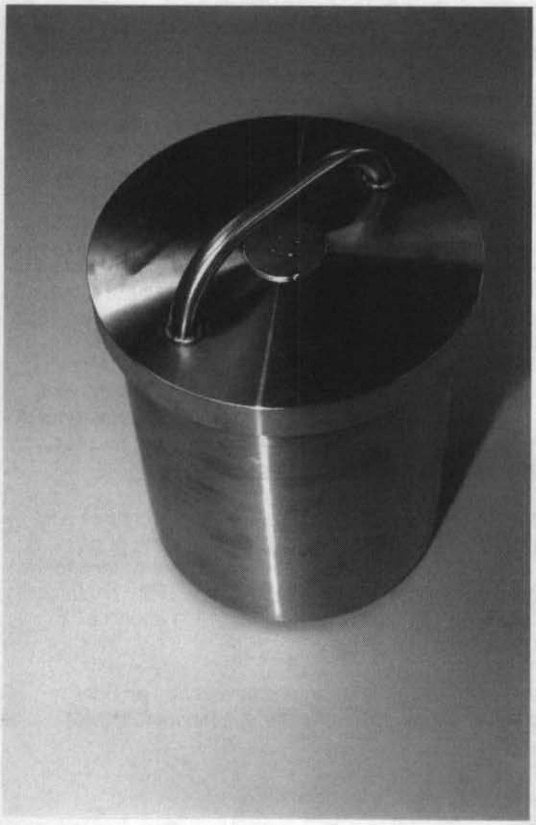


**Figure –2 Doubled bagged lead free pewter can**

**LEAK RATE TESTS**

The leak rate test was done by internally pressurizing the container to about 5" water column pressure differential. Then the container was immersed upside down in a basin of water . The water temperature was maintained at nearly room temperature (22 degrees centigrade). Figure - 3 shows a photograph of two of the pressurized containers before immersion into the water basin. Figure -4 shows a photograph the two containers immersed in the basin of water with the pressure gage reading slightly above 5 inches water column. Upon the initial immersion, air bubbles escape out from the threads for about 30 seconds. The container was rotated by hand while tapping on the lid to encourage the trapped air in the threads to escape, prior to monitoring for container leakage. Leaks are detected quite easily if they exist because air bubbling continues. A passing result is one where there are no bubbles detected for more than three minutes. A continuous stream of a air bubbles would be grounds for failing the container.

**DROP TEST RESULTS**

Figure-1 shows a photograph of the canister prior to being dropped from the test height.. Figure 5 shows a photograph of a test container just prior to drop test from a height of about 1.21 meters. The results of leak rate testing after drop tests in five orientations are given in TABLE -1.

**TABLE - 1 RESULTS OF 1.21 (4 Feet) METER DROP TEST**

| DROP TEST ORIENTATION | CAN # | OBSERVATIONS NOTED | LEAK RATE TEST PASS / FAIL |
|---|---|---|---|
| DIRECT ON BOTTOM | RFP-2 | SLIGHT INDENTATION AT POINT OF IMPACT 10 mm long, 1 mm | PASS |
| BOTTOM EDGE | RFP-2 | IMPACTED AT 22 DEGREES SOME INDENTATION 41 mm long, 5.61 mm deep | PASS |
| DIRECT ON HANDLE | RFP-3 | FLATTENED TOP EDGE OF HANDLE SLIGHTLY 1mm, LID REMAINS FLAT | PASS |
| DIRECT ON EDGE OF LID | RFP-3 | ONLY SLIGHT BLEMISH AT POINT OF IMPACT. | PASS |
| DIRECT ON SIDE | RFP-4 | SLIGHT INDENTATION ON BOTTOM END, SLIGHT BLEMISH ON EDGE OF LID | PASS |

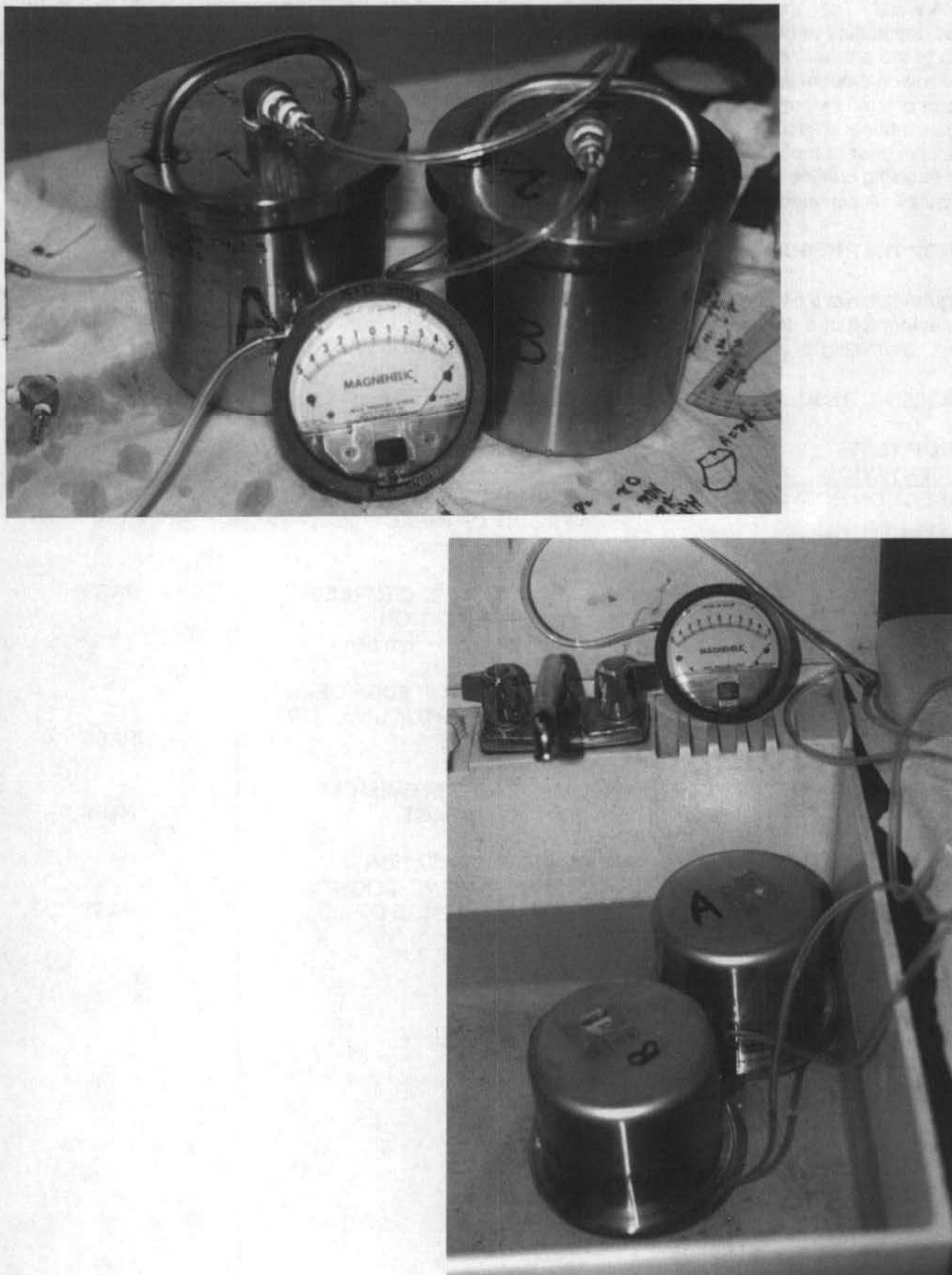



Figure –4 Pressurized Leak Test

After successfully passing the required drop test from 1.21 meters, the containers were dropped in the same orientations from 2.5 meters (8 feet). The drop tests were conducted at NFT Inc. Laboratory, Golden, CO USA and data recorded in laboratory notebook -01 pages 56 through 58. The tests were also videotaped and witness by an independent party. The results are shown in Table -2 below:

**TABLE-2 RESULTS OF 2.5 (8 feet) METER DROP TEST**

| ORIENTATION | CAN # | OBSERVATIONS NOTED | LEAK RATE TEST PASS / FAIL |
|---|---|---|---|
| DIRECT BOTTOM EDGE | RFP-4 | IMPACT AT 34 DEGREES OFF NORMAL, SEVERE WRINKLES ON LOWER EDGE, 88 mm long, 12.4 mm DEEP | PASS |
| DIRECT ON HANDLE | RFP-4 | INDENTED HANDLE 2.44mm | PASS |
| DIRECT ON EDGE OF LID | RFP-4 | MARRED SURFACE FINISH NO DENTS | PASS |

**HYDROGEN DIFFUSIVTY**

A hydrogen diffusivity test was conducted to verify that gases generated would diffuse out of the container through the filter and membrane. The hydrogen diffusivity test quantifies the rate at which radiolytically and chemically generated hydrogen and other gases will diffuse with zero pressure gradient across the membrane. The Waste Isolation Pilot Plant Safety Analysis Report section 1.3.5 requires a value of 1.9 E-06 mole/second/mole fraction. A gas chromatograph equipped with a thermal conductivity detector was used to measure the concentration of hydrogen gas (initially at 4.0%, certified) with a balance of nitrogen over a period of about 35 minutes.

Figure-6 shows a photograph of the test chamber where the hydrogen diffusion rate through the filter and membrane was measured. A precise mixture of hydrogen gas ($H_2$) and nitrogen ($N_2$) is circulated by a peristaltic pump through the gas chromatograph and returned to the test vessel. At the start of a test and at five minute intervals, a sample of the gas is injected into the detector for analysis. Seven samples are take over a 35 minute period. The amount of hydrogen in the test vessel decays over time due to diffusion through the filter. The hydrogen diffusivity losses through tubing and connections have been measured and are about three orders of magnitude lower than diffusion rates through the filter. Therefore, no adjustments to filter diffusion coefficients are made.

The chromatogram peak area is related to the amount of hydrogen in the container. The decay rate, or hydrogen diffusivity , D, through the assembly is given by:

$$D = PV/t\ RT\ Ln(H_o/H_t)$$

Where P is atmospheric pressure, V is the volume of the vessel with connective tubing, t is elapsed time in seconds, R is the Ideal Gas Constant, and T is the temperature in Kelvin. The diffusion rate, D, is in moles/ second / mole fraction. The initial concentration of hydrogen given by the chromatogram peak area is $H_o$ and the amount of hydrogen after time t is $H_t$.

A special test fixture was assembled that nearly duplicated the transport path of the gas through the filter and membrane in the containers. Chromatograms and spreadsheet calculations are shown in Appendix - 1 attached. Only two samples were measured for hydrogen diffusivity because the test is very long. The hydrogen transport rate through the filter and membrane averaged 15.75 E-06 mole/ second/ mole

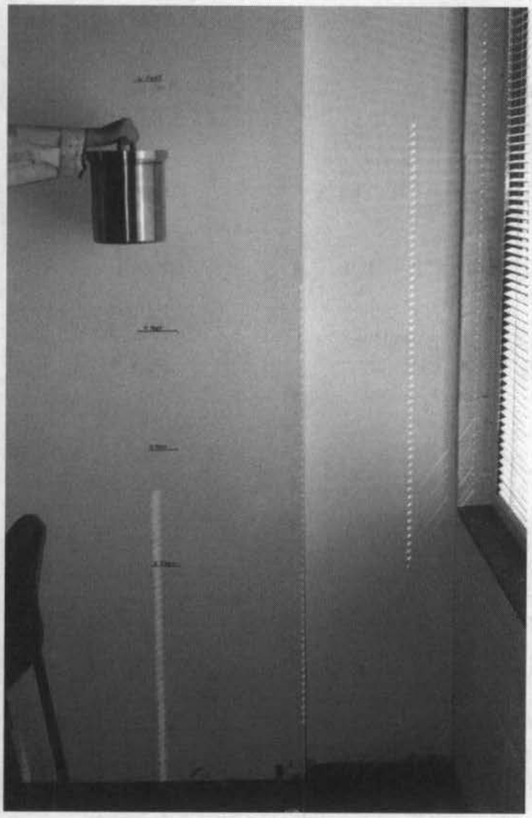


Figure –5 Drop Test From 1.21 Meters

Figure –6 Test chamber for hydrogen diffusion measurements fraction which is 8 times greater than the minimum established by the WIPP TRUPACT II SAR. Oxygen peaks begin to appear after about 10 minutes which is the result of air diffusing back into the container.

## AIR PERMEABILITY AND FILTRATION EFFICIENCY

Each container lid is equipped with a type 316 stainless steel HEPA grade filter media (a carbon-bonded-carbon filter may also be used). The lid, with the integral filter, is shown being tested for flow rate and particle removal efficiency in Figure - 7 and 8. Air permeability and filtration efficiency are measured using an Air Techniques Inc., aerosol photometer with a 0.3 to 0.5 micron dioctyl phthalate (DOP) aerosol. The optical photometer verifies that the filter vent qualifies as a HEPA grade filter, removal of greater than 99.97% of aerosol. Given an air flow rate of 200 ml/min across the filter, the resistance across the filter must be 1.0 inches water column or less. In production, all filters will be tested for air permeability and particle retention. Filter efficiency tests were conducted on 10 container lids with integral stainless steel filters. Listed in Table -2 are the results showing the airflow rates of 210 ml/min, percent penetration of DOP, and resistance to air flow (inches water column).

**TABLE -2**

| SERIAL I.D. | PERCENT PENETRATION | RESISTANCE TO FLOW (inches W.C.) | FLOW RATE ( ml/min ) |
|---|---|---|---|
| RFP-NMC-201 | 0.004 | 0.55 | 210 |
| RFP-NMC-202 | 0.004 | 0.55 | 210 |
| RFP-NMC-203 | 0.008 | 0.55 | 210 |
| RFP-NMC-204 | 0.004 | 0.55 | 210 |
| RFP-NMC-205 | 0.008 | 0.60 | 210 |
| RFP-NMC-206 | 0.010 | 0.65 | 210 |
| RFP-NMC-207 | 0.010 | 0.65 | 210 |
| RFP-NMC-208 | 0.004 | 0.55 | 210 |
| RFP-NMC-209 | 0.004 | 0.55 | 210 |
| RFP-NMC-210 | 0.004 | 0.55 | 210 |

Of the first 1000 cans in production, there were no failures due to percent of penetration or high resistance to flow.

## WATER ENTRY TEST

To verify that no water intrusion will occur, for example from fire suppressant systems, the containers were subject to a 1 psig water column for a period of 12 hours. After a 12 hour period there was no visible water detected on the interior surface of the container. A second water intrusion test subjected the containers to a constant spray of water, at a rate of 30 gallons per minute for 1 hour, with no visible water entry. Figure -9 shows a photograph of the water spray test. A third water spray test utilized an industrial dishwasher where the canister was subjected to a high pressure spray (greater than 40 PSI) at all angle at approximately 90 degrees Celsius for 1.5 minutes. There was no visible water entry after the high pressure spray test. Figure -10 shows a photograph of the high pressure water spay generated from the industrial dishwasher.

## CONCLUSION

Nuclear materials storage containers fabricated entirely from type 304 stainless steel have been developed and tested to verify leak tightness under severe impact. The nuclear materials containers were designed to meet requirements established in DOE's Recommendation 94-1, "Criteria For Interim Safe Storage of Plutonium-Bearing Solid Material". Containers used for the interim storage (less than 20 years) of nuclear bearing materials must allow a means for retrieval and observation of stored contents, while also meeting stringent structural and leak-proof requirements. The new, all stainless steel, container has a threaded lid closure that is equipped with a sintered stainless steel HEPA filter vent. The hand tightenable container provides a positive seal for storing nuclear material, yet allows the controlled release of explosive gases including hydrogen.

The results of air-leak after a 1.21 meter drop test indicates that the container seal was not compromised from the drop impact. Hydrogen diffusivity measurements indicate hydrogen gas will be transported though the integral filter and semipermeable membrane at a rate of about 15 E-06 moles/second/mole fraction. Filter efficiency tests demonstrate that a reliable seal is formed with the sintered stainless steel filter media. Particle retention of 0.3 to 0.5 micron DOP aerosol was measured at greater than 99.97% at an air flow of greater than 210 milliliters per minute. Through three different water entry test, one pressurized at 1 PSIG and two water spray tests, it is demonstrated that water will not enter the container.

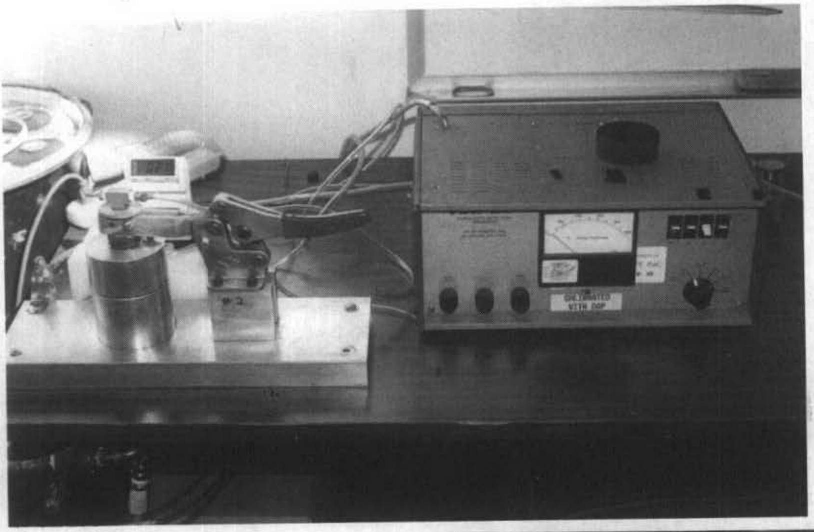


Figure –7 Optical photometer used for filter Efficiency test

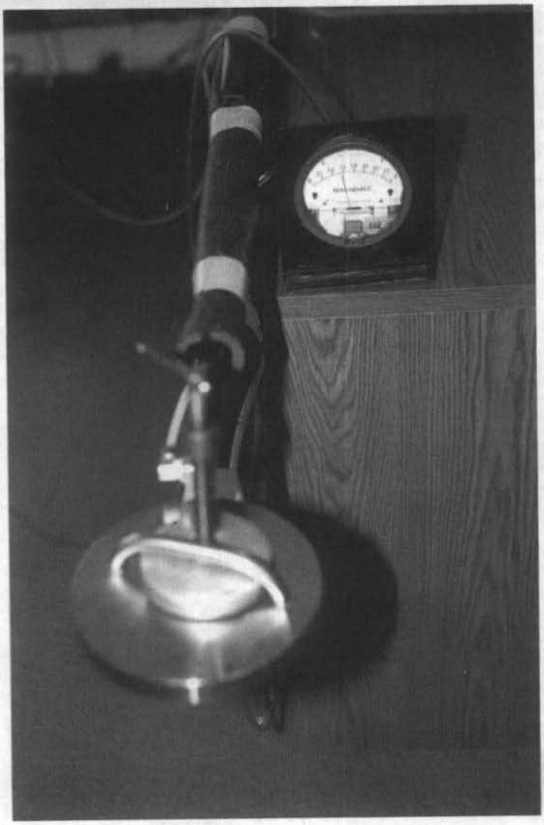


Figure – 8 canister lid with integral filter, resistance flow is 0.65" W.C.

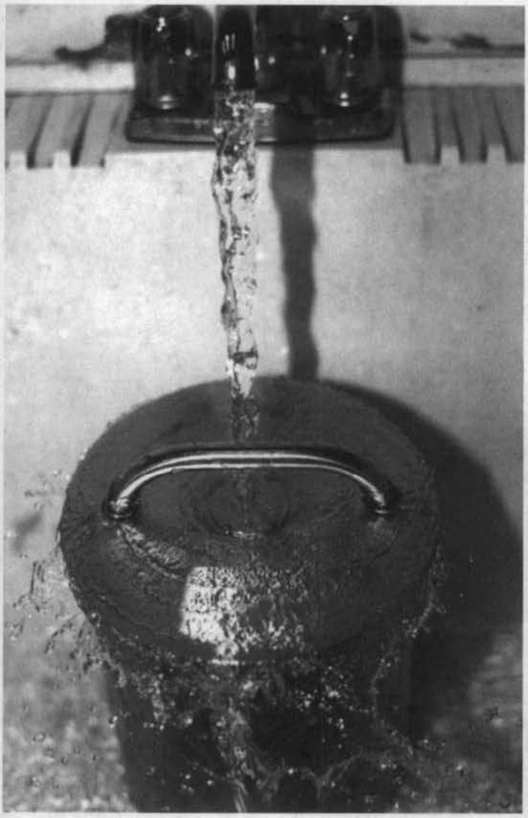


Figure –9 NMC under water spray test

Figure –10 NMC in high pressure industrial water spray test

# APPENDIX -1

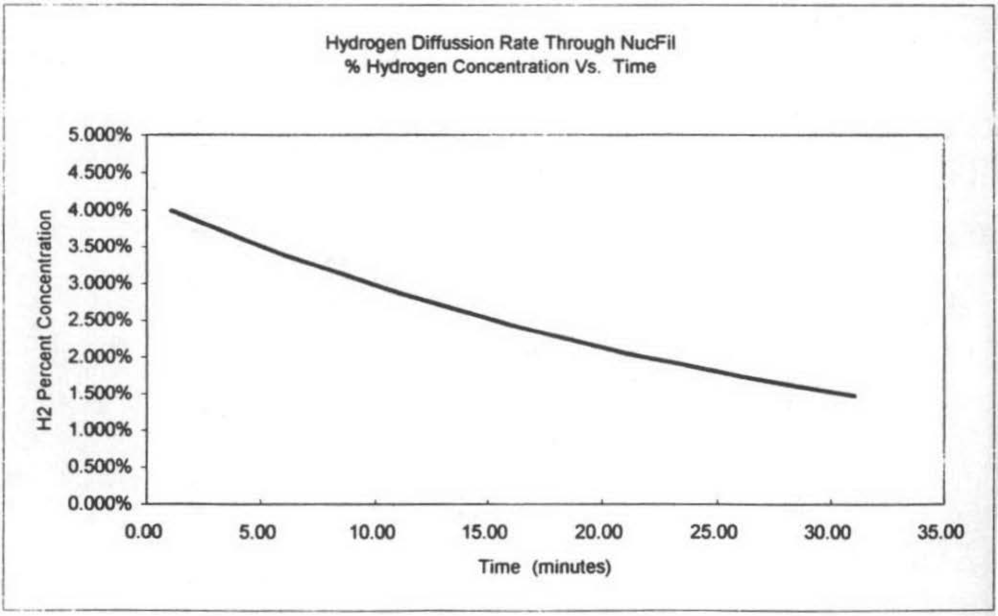
## Hydrogen Diffusion Chromatograms and Spreadsheet Calculations

| | | | |
|---|---|---|---|
| DATE: | 08-Apr-96 | FORM: | DIFFCALC.WQ1 |
| USER: | Terry W. | | |
| FILE: | C:\qpro\diff\sss200 | | |
| SAMPLE I.D | Sss 200 4/9/! | MODEL: | NUCFIL-013 |
| ATM. P (atı | 1 | | |
| TEMP. (K) | 297.6 | | |
| GAS CONSTAI | 0.08206 | | |
| VESS. VOl | 0.813 | | |

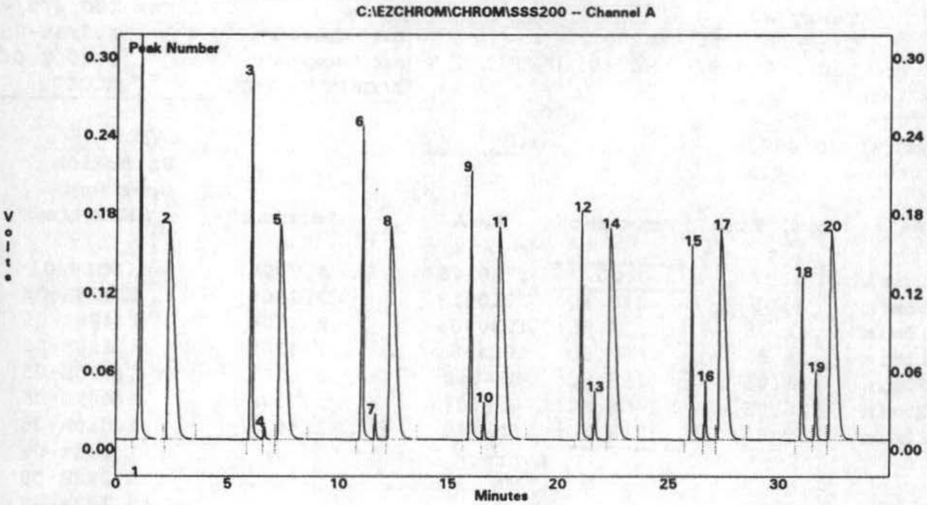
| RESULTS | |
|---|---|
| I.D.: | Sss 200 4/9/96 |
| Diff Constant: | 1.495E-05 |
| Min Accepted: | 1.900E-06 |
| ACCEPT/REJECT | ACCEPT |

QA________________________ DATE________

| H2 Pk | Ret. Time | Time sec) | AREA | Percent | Diffusion Constant Mol/MolFrac/S |
|---|---|---|---|---|---|
| - | - | - | - | - | - |
| H2 Initial | 1.07 | 0.00 | 1780125 | 3.990% | #DIV/0! |
| H2 @t=5min | 6.05 | 298.80 | 1510618 | 3.386% | 1.483E-05 |
| H2@t=10min | 11.05 | 598.80 | 1280486 | 2.870% | 1.486E-05 |
| H2@t=15min | 16.05 | 898.80 | 1084351 | 2.430% | 1.490E-05 |
| H2@t=20min | 21.05 | 1198.80 | 914869 | 2.051% | 1.503E-05 |
| H2@t=25min | 26.05 | 1498.80 | 774929 | 1.737% | 1.501E-05 |
| H2@t=30min | 31.05 | 1798.80 | 652840 | 1.463% | 1.510E-05 |
| | | | Average.... | . | 1.495E-05 |
| | | | STDV...... | ................ | 9.922E-08 |
| | | | Range..... | ................ | 2.743E-07 |



File : C:\EZCHROM\CHROM\SSS200 ml/MIN
Method : C:\EZCHROM\chrom\diffsn.MET
Sample ID : STRD-2 NUCFIL-013 SINTERED 20
Acquired : Apr 08, 1996 14:28:42
Printed : Apr 08, 1996 15:04:03
User : Terry W.




Channel A Results

| Peak | Name | Time | Area |
|---|---|---|---|
| 1 | Hydrogen Initial | 1.08 | 1780125 |
| -- | Oxygen Initial | 1.65 | 0 |
| 2 | Nitrogen Initial | 2.34 | 3241179 |
| 3 | H2 @ t = 5 minutes | 6.07 | 1510618 |
| 4 | O2 @ t = 5 Minutes | 6.67 | 54667 |
| 5 | N2 @ t = 5 Minutes | 7.34 | 3208727 |
| 6 | H2 @ t = 10 Minutes | 11.06 | 1280486 |
| 7 | O2 @ t = 10 Minutes | 11.67 | 101196 |
| 8 | N2 @ t = 10 Minutes | 12.34 | 3177777 |
| 9 | H2 @ t = 15 Minutes | 16.06 | 1084351 |
| 10 | O2 @ t = 15 Minutes | 16.67 | 147762 |
| 11 | N2 @ t = 15 Minutes | 17.35 | 3151418 |
| 12 | H2 @ t = 20 Minutes | 21.06 | 914869 |
| 13 | O2 @ t = 20 Minutes | 21.67 | 188264 |
| 14 | N2 @ t = 20 Minutes | 22.35 | 3113861 |
| 15 | H2 @ t = 25 Minutes | 26.06 | 774929 |
| 16 | O2 @ t = 25 Minutes | 26.67 | 226979 |
| 17 | N2 @ t = 25 Minutes | 27.35 | 3091258 |
| 18 | H2 @ t = 30 Minutes | 31.06 | 652840 |
| 19 | O2 @ t = 30 Minutes | 31.68 | 264343 |
| 20 | N2 @ t = 30 Minutes | 32.36 | 3057786 |

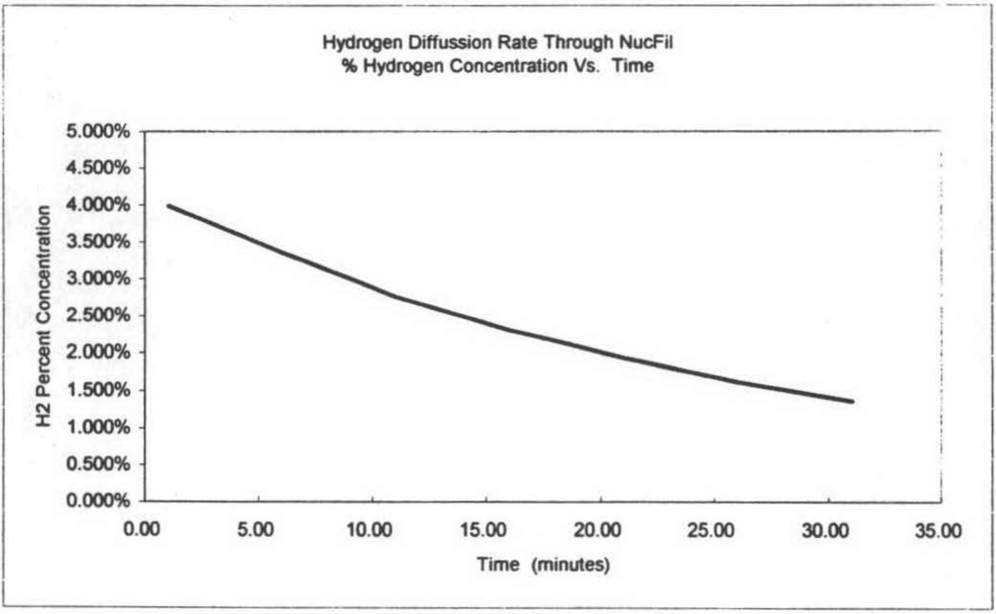
Totals :

31023446

| | | | |
|---|---|---|---|
| DATE: | 08-Apr-96 | FORM: DIFFCALC.WQ1 | |
| USER: | Terry W. | | |
| FILE: | C:\qpro\diff'#54sss | | |
| SAMPLE I.D | SINTST.ST.01: | MODEL: NUCFIL-013 | |
| ATM. P (atı | 1 | | |
| TEMP. (K) | 297.6 | | |
| GAS CONSTAI | 0.08206 | QA ______ | DATE ______ |
| VESS. VOl | 0.813 | | |

| RESULTS | |
|---|---|
| I.D.: | SINTST.ST.013SS |
| Diff Constant: | 1.656E-05 |
| Min Accepted: | 1.900E-06 |
| ACCEPT/REJECT | ACCEPT |

| H2 Pk | Ret. Time | Time sec) | AREA | Percent | Diffusion Constant Mol/MolFrac/S |
|---|---|---|---|---|---|
| - | - | - | - | - | - |
| H2 Initia. | 1.07 | 0.00 | 1807004 | 3.990% | #DIV/0! |
| H2 @t=5min | 6.05 | 298.80 | 1519056 | 3.354% | 1.588E-05 |
| H2@t=10min | 11.05 | 598.80 | 1250552 | 2.761% | 1.700E-05 |
| H2@t=15min | 16.05 | 898.80 | 1045646 | 2.309% | 1.680E-05 |
| H2@t=20min | 21.05 | 1198.80 | 875293 | 1.933% | 1.667E-05 |
| H2@t=25min | 26.05 | 1498.80 | 734648 | 1.622% | 1.653E-05 |
| H2@t=30min | 31.05 | 1798.80 | 615775 | 1.360% | 1.646E-05 |
| | | | Average.... | . | 1.656E-05 |
| | | | STDV...... | ................ | 3.514E-07 |
| | | | Range..... | ................ | 1.124E-06 |



File : C:\EZCHROM\CHROM\#54ss2
Method : C:\EZCHROM\chrom\diffsn.MET
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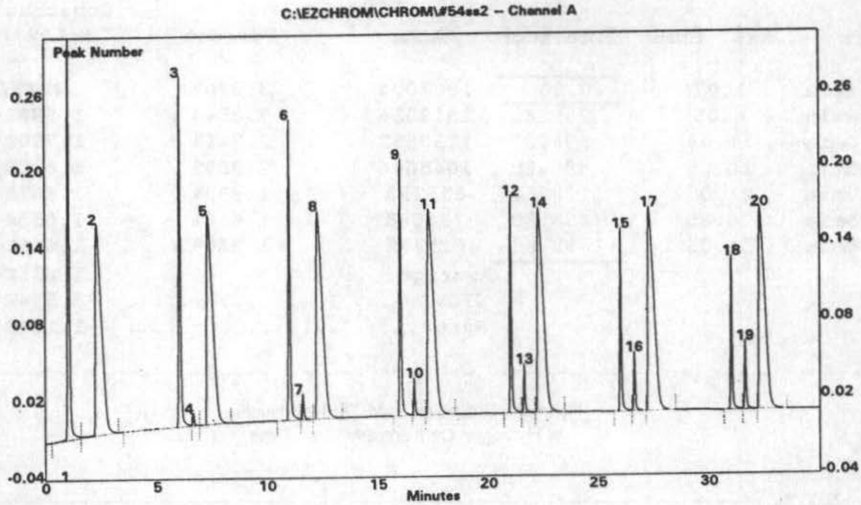
Acquired : Apr 08, 1996 06:28:21
Printed : Apr 08, 1996 07:03:48
User : Terry W.




Channel A Results

| Peak | Name | Time | Area |
|---|---|---|---|
| 1 | Hydrogen Initial | 1.07 | 1807004 |
| -- | Oxygen Initial | 1.65 | 0 |
| 2 | Nitrogen Initial | 2.32 | 3266442 |
| 3 | H2 @ t = 5 minutes | 6.05 | 1519056 |
| 4 | O2 @ t = 5 Minutes | 6.65 | 56765 |
| 5 | N2 @ t = 5 Minutes | 7.31 | 3177959 |
| 6 | H2 @ t = 10 Minutes | 11.05 | 1250552 |
| 7 | O2 @ t = 10 Minutes | 11.65 | 104877 |
| 8 | N2 @ t = 10 Minutes | 12.31 | 3130961 |
| 9 | H2 @ t = 15 Minutes | 16.05 | 1045646 |
| 10 | O2 @ t = 15 Minutes | 16.65 | 148489 |
| 11 | N2 @ t = 15 Minutes | 17.31 | 3091453 |
| 12 | H2 @ t = 20 Minutes | 21.05 | 875293 |
| 13 | O2 @ t = 20 Minutes | 21.65 | 190839 |
| 14 | N2 @ t = 20 Minutes | 22.32 | 3054571 |
| 15 | H2 @ t = 25 Minutes | 26.05 | 734648 |
| 16 | O2 @ t = 25 Minutes | 26.65 | 229171 |
| 17 | N2 @ t = 25 Minutes | 27.32 | 3023443 |
| 18 | H2 @ t = 30 Minutes | 31.04 | 615775 |
| 19 | O2 @ t = 30 Minutes | 31.65 | 265684 |
| 20 | N2 @ t = 30 Minutes | 32.33 | 2991993 |

Totals : 30580634