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Evaluation of the Potential for Gas
Pressurization and Free Liquid
Accumulation in a WVDP Canister

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December 1993

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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SUMMARY

A full-scale canister provided by the West Valley Demonstration Project (WVDP), fired during the SF-11 vitrification qualification test, was tested to determine its potential for gas generation (non-radiolitic only) and liquid accumulation. The canister was sealed and held at a temperature of about 500°C for eight weeks. Gas samples obtained during the test were analyzed using mass spectroscopy to determine the composition of gases within the canister. At the end of the eight weeks the canister gases were evacuated through a desiccant to capture any water that had been released by the glass during the test. In addition, an analysis of the glass using fourier transform infrared (FTIR) spectroscopy was performed to determine the water content in the glass both before and after the temperature exposure.

Gas samples obtained during the test showed that some oxygen consumption through metal oxidation and oxidation (presumably) of the ferrous iron and other components in the glass occurred. The composition in mole % of the gas within the canister after eight weeks of exposure at 500°C was helium, 76.8; nitrogen, 22.2; carbon dioxide, 0.58; argon, 0.28; oxygen, 0.08; carbon monoxide, <0.1; and hydrogen, methane, and nitrogen oxides each <0.01, the minimum laboratory reporting points. The high helium concentration can be attributed to backfilling the canister with helium for the canister lid weld leak checks.

The glass itself was not found to be a significant source for free water at the conditions of the test. The amount of water recovered from the canister was calculated to be about 0.7 g. This is 0.00004 wt% of the total amount of glass (about 4340 lb) in the canister. A direct measure of water content in the glass, using FTIR spectroscopy, determined that glass samples taken from the canister before and following the test contained equivalent amounts of water, approximately 0.15 wt%. This value is quite consistent with the water content found in commercial glasses.

Based on the results of this experiment it is concluded that the waste form product and canister will satisfy Waste Acceptance Preliminary

Specifications 3.1 and 3.2, which prohibit free liquids and free gases other than cover and radiogenic gases.

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INTRODUCTION

Specifications 3.1 and 3.2 of the Waste Compliance Plan for the West Valley Demonstration Project (WVDP) High Level Waste Form address prevention of free liquids entering and gases accumulating in the canistered glass waste form (Cadoff 1989). The first specification states, "After closure the canistered waste form shall not contain free liquids that could be drained from the canister either initially or after having been subjected to the transition temperature of Specification 1.4(a)." The second specification, 3.2, states, "After closure the canistered waste form shall not contain free gases other than cover and radiogenic gases." Studies to confirm that these specifications will be met have been undertaken at Pacific Northwest Laboratory (PNL)^(a) in support of the WVDP Waste Qualification Report (WQR). The waste qualification gas pressurization test is one component of these studies.

The overall objectives of support work for these two specifications are 1) to demonstrate that the glass itself is not a source of free liquids and 2) to provide an estimate of the quantities and compositions of any non-radiogenic gases that might accumulate inside the canister after the canistered waste form has been exposed to temperatures up to the glass transition temperature. These objectives were to be met by reviews of the relevant literature and by testing a full-scale canister containing simulated waste glass at the transition temperature for several weeks. The testing is referred to as the waste qualification gas pressurization test.

A literature review supported the contention that the glass itself is not a direct source for free water. Another review also revealed that other gas-forming materials will not be present in the final glass waste form as they will be either absent from the melter feed stream or oxidized or vaporized in the melter and exhausted as gases before filling the canister. The findings of the reviews are presented elsewhere (Letter, W. A. Ross to

(a) Pacific Northwest Laboratory is operated by Battelle Memorial Institute for the U. S. Department of Energy under contract DE-AC06-76RLO 1830.

J. M. Pope, September 29, 1989, "Literature Review for WQR Reports").

The specific objective of the waste qualification gas pressurization test was to produce quantitative and composition data on any free liquids and gases generated in a representative WVNS waste canister filled with simulated, nonradioactive glass by its extended exposure at the glass transition temperature. The canister, filled at West Valley during vitrification process qualification run SF-11, was shipped to PNL where it was maintained at about the glass transition temperature (500°C) for eight weeks in PNL's in-can melter (ICM). Provisions were made to sample gases periodically and to collect free liquids that may have accumulated in the canister plenum during the eight-week test.

Conclusions, a description of the test equipment, and experimental results for the eight-week exposure test are covered in the following sections.

CONCLUSIONS

The glass itself was not found to be a significant source for free water at the conditions of the test. The amount of water sorbed when the canister was depressurized after 8 weeks at temperature and the released gases were passed through a desiccant bed was about 0.4 g. Taking into account the 0.4-g recovered, the volume of gas remaining in the canister, and the relative humidity of the air in the canister plenum when the lid was welded in place, the amount of water released from the glass was calculated to be about 0.7 g. This comes to 0.4 ppm, or 0.00004 wt% of the total amount of glass (about 4,340 lb) in the canister. This amount of water is insignificant assuming all the glass contributed to the water's release.

Oxygen was depleted from the canister plenum during the first week of testing, falling from 4.81 mole % in the first sample taken upon reaching test temperature to 0.05 mole % in a sample taken at the end of the first week. Concurrently, carbon dioxide content rose from 0.10 mole % for the first sample to 0.47 mole % for the second sample taken after one week of testing.

Although analytical data indicated a further rise in carbon dioxide content to 0.58 mole % by test end, the rise is judged as insignificant because of the precision of the analytical method. Some oxygen consumption through metal oxidation and oxidation of the ferrous iron and other components in the glass was expected. For example, the inner surface of the canister lid had not been exposed to high temperatures prior to the test.

The composition (in mole %) of the gas within the canister following eight weeks of exposure at 500°C was helium, 76.8; nitrogen, 22.2; carbon dioxide, 0.58; argon, 0.28; oxygen, 0.08; carbon monoxide, <0.1; and hydrogen, methane, and nitrogen oxides each <0.01, the minimum laboratory reporting points. The high helium concentration was from backfilling the canister with helium for the canister lid weld leak checks.

Canister pressure fell from 14.0 psig to 11.4 psig during the test. Most of the decrease was due to the weekly collection of samples, which amounted to a pressure loss of 0.3 to 0.5 psi per sample. A small decrease unmeasurable in this test may have been due to various oxygen depletion reactions, such as with the glass and the canister.

The test results did not demonstrate any pressurization of the canister by the generation and release of gases from the glass during the eight weeks of exposure at the transition temperature. In fact, the results showed a consumption of free gas as oxygen in the plenum reacted. There was no indication that any free gas, other than the cover and radiogenic gases, would accumulate in the canister plenum and thus cause canister pressurization.

The top 24 inches of the canister protruded above the ICM out of the heating zone. Although this exposed section was insulated, its exposure resulted in a temperature gradient along the exterior surface of the canister wall such that the temperatures 17 in. and 24 in. below the top of the canister lid were, respectively, 408°C and 485°C. The glass surface before heating was 17-3/4 in. below the top of the canister lid, or about the same level as one of the temperature profile measurements. Any potential effects from the lower temperature glass on gas or liquid vapor release would be small

considering that the predominant path for migration should be out to the side of the canister. The gas would then pass up between the canister and glass to the head space.

DESCRIPTION OF TEST SYSTEM

The principal elements of the test system were the glass canister, the PNL ICM with temperature controls for heating and maintaining the canister at the test temperature, a pressure gauge for measuring the canister pressure during the test, a gas-sampling system, and a water condensing and sampling system fitted to a PNL-built lid for the canister.

Glass Canister

The West Valley Nuclear Services (WVNS) canister (Figure 1) has a 24-in. outside diameter, is 117-3/4-in. tall (WVNS Dwg. No. 900E-1092) without a lid, and was constructed of 304L stainless steel (ASTM-240), the cylinder wall being No. 10 gauge metal. The identification number, which was welded on the surface near the top of the canister, is WV065A. The canister was filled at West Valley with glass to 17 in. from the top face of the canister flange, giving a glass height of about 100-3/4 in., which in turn gives a glass volume of about 25.8 ft³. By using a glass density of 2.695 g/cm³ or 168 lb/ft³, the glass weight was calculated to be approximately 4,340 lb. The density used is that given by Eisenstatt (1986). The plenum space above the glass in the canister was calculated to be about 3.89 ft³; this includes the volume above the glass in the main canister cylinder, the volume of the ASME flanged and dished head and its straight flange section, and the volume of the cylindrical throat of the canister. A 3/4-in.-thick stainless steel lid capable of withstanding the expected maximum temperature and pressure of the gas pressurization test was designed, built, and welded to the top flange of the canister. The lid was fitted with piping, tubing, and valves for a pressure gauge and gas-sampling system connections. With the lid in place, the canister was tested for helium leaks and found to be leak free. A calibrated, Type K thermocouple was tacked onto the outside of the canister shell about the center of the canister length for temperature measurement using a digital

indicator. This served as the principal measurement point for test temperature control.

In-Can Melter

The ICM furnace (Figure 2) was used as the heater for the glass canister. It is full-scale with respect to the diameter of the heated chamber; it can accept 12-to 24-in.-diameter canisters. It is insulated with fiber-ceramic materials that allow rapid temperature changes to be made because of the materials' low heat capacity and their resistance to thermal shock. The electrical resistance furnace is power rated at 210 kVA and will operate at temperatures up to 1200°C. The furnace, having six independently controlled zones, is heated with Kanthal alloy (Fe, Cr, Al) resistance elements, which are supported in the dense refractory. Two thermocouples are located in each zone between heaters in the ICM melt cavity; one is for controller operation, the other for chart recording. The zone temperatures are controlled by Honeywell Dialatrol temperature-indicating controllers, and they are recorded by a Honeywell Electronic 112 multipoint chart recorder. (McElroy 1978 and Blair 1980).

Twenty-four inches of the canister body, including the canister's flanged and dished head and throat, protruded above the ICM such that the glass level was out of the top heater zone. Two inches of fiberglass insulation was placed around this portion of the canister to diminish heat loss.

Pressure Measurement and Gas and Water Sampling Systems

A Bourdon-tube type pressure gauge was used for indicating the pressures throughout the test. Sampling systems were used for collecting gas and water samples. The pressure gauge was a Marsh Instrument Co. Mastergauge, Type 100-SS, 316 stainless steel, 0-60 psi, with 1/2-psi subdivisions, which allowed visual interpolation of pressure readings to about 0.1 psi. Schematics of the apparatus are shown in Figure 3.

The gas-sampling system consisted of the valve connected to the canister lid, a short length of stainless steel tubing, and a gas sample bottle. The

FIGURE 1. WVP Canister

FIGURE 2. In-Can Melter Furnace

water condensing and sampling system, substituted for the gas-sampling system as originally planned, consisted of a heat-traced, 1/4-in. stainless steel line leading from the canister, a glass condenser, and a water-collection flask with a sidearm. Because the literature indicated that little or no water should be present in the glass, a gas-drying unit filled with moisture-indicating Drierite was added to the sampling system downstream of the condenser and flask. Air was passed through the condenser with an air pump for cooling the off gases from the canister. A valved 20-SCFH rotameter was installed in the line from the canister to measure and control the outflow of gas at between 0.5 and 1.5 SCFH to ensure that a pressure drop of no more than 5 psi/h would occur in the canister. As a control, the low velocity through the tube and condenser also helped ensure that only gases at near room temperature would reach the drying unit. The heating tape used for line tracing was also partially removed from the canister's gas outlet line near the condenser entry to help promote gas cooling. Use of the heating tape was initially intended to make certain that no moisture would condensate in the system before reaching the condenser so that any moisture present could be condensed and collected.

EXPERIMENTAL TESTING AND RESULTS

Initial and Final Observations of Open Canister

Before the lid was welded onto the canister and after its removal following the testing, the glass and metal surfaces were visually inspected for evidence of moisture. No moisture was observed nor found by wiping the interior surfaces with filter paper. About eight to ten pounds of glass shards were observed lying in the canister plenum before and after the test; it is presumed that the shards were broken free from the top of the glass log.

Helium Leak Check

After the lid was welded into place, the canister was placed into the ICM. The canister plenum was pressurized with dry (<5-ppm moisture), high purity helium to 40 psig and checked to a leak rate of $<1 \times 10^{-7}$ cm³/s by a certified technician using a model CEC24-120B leak detector (Consolidated Electrodynamics Corporation). Pressure stability was also observed by

GAS SAMPLING ARRANGEMENT

LIQUID SAMPLING ARRANGEMENT

FIGURE 3. Gas and Liquid Sampling Arrangements

monitoring the pressure gauge while the system was left pressurized for one week. The helium pressurization and subsequent venting of the canister to atmospheric pressure before the test began provided the gas mixture determined by the composition analyses of the periodic gas samples taken during the test. This composition was considered to be prototypic since helium, air, or a combination of the two are among the alternatives for cover gases for waste glass canisters (Cadoff 1989).

Startup and Test Conditions

The canister was heated from 24°C to 511°C (measured by the digital indicator and the thermocouple tacked to the canister at the center of the canister's length) in about 6 hours; the pressure concurrently rose from 0 to 10.0 psig. The first 500 mL gas sample was taken at this condition. It was observed with this gas sampling that the pressure fell momentarily to about 9.75 psig. A pressure drop was typical with each gas sampling during the 8-week test. After the sampling the pressure again rose since equilibrium control conditions had not yet been established, reaching a maximum of 14.5 psig at 523°C by the third day of the test. Temperature controllers on the ICM were adjusted to lower the temperature and, consequently, the pressure. The maximum temperature reached during the test was 529°C (near the end of week 8 and the end of the test), but generally the temperature was about 521°C. The pressure at this highest temperature was 11.4 psig; it was at this level because several gas samples had been taken previously. Temperatures and pressures were recorded each work day throughout the test, and gas samples were taken about weekly.

Canister Wall Temperature Profile

A wall temperature profile for the upper portion of the canister was made at the end of the first week of the test. Measurements were made vertically from the top of the canister lid, or 17-3/4 in. above the starting temperature glass level. A Type K thermocouple was inserted down the outside of the canister so that it touched the wall. The profile is given below:

<u>Distance Below Lid (in.)</u>	<u>Temperature (°C)</u>
On Lid	300
5	274
12	376
17	408
24	485
36	504

Canister Pressure History During Test

A pressure history for the canister during the test is shown in Figure 4. The pressure fell off immediately about 0.3 to 0.5 psi with each gas sampling, but otherwise, with a brief exception described below, remained relatively steady between samplings. The pressure at the end of the first week before the second sampling was 14.0 psig, and at the end of the eighth week before the final sampling, 11.4 psig. The pressure drops thus can be attributed primarily to the gas sampling, although some small pressure drop is attributed to depletion of oxygen in the plenum space by various oxidation reactions.

If the gases in the plenum space were at 500°C, the canister pressure estimated by ideal gas relationships would have been 23.4 psig. Since the pressure was only about 14.0 psig during the first week of the test, the average plenum temperature had to be lower because the plenum volume was constant. Again using ideal gas relationships, the average plenum temperature was calculated to be 307°C.

During the fifth week of the test, a control switch was inadvertently shut off, causing a temporary loss of power to the ICM. The canister pressure fell as shown by the profile, but recovery occurred within a day following the return of power.

Gas Samples

Gas samples were taken from the canister plenum at the beginning of the test and weekly thereafter in evacuated 500 mL, Type 304 stainless steel,

Figure 4. Pressure History for Glass Canister Test

double sealed, single valve, gas sample bottles. The gas samples were analyzed by mass spectrometry, which has a precision (repeatability, reproducibility) of 1% to 2% relative on a species. The gas components were helium, nitrogen, oxygen, argon, carbon dioxide, and small amounts (<0.1 mole %) of other gases (Table 1). Helium, introduced during the pressurized leak test, constituted 74.7 to 77.5 mole % of the gas; nitrogen, 20.2 to 21.6 mole %. Oxygen, 4.81 mole % in the first sample, was depleted from the canister plenum during the test, and carbon dioxide was generated until the oxygen was essentially gone by the second sampling (0.05 mole %). The amount of oxygen in the gas before canister heatup is estimated to be about 5.4 mole % using the nitrogen-oxygen ratio in air and nitrogen in the first sample as bases. The first gas sample contained 0.10 mole % of carbon dioxide; the second, 0.47 mole %. The carbon dioxide content rose but slightly thereafter, averaging 0.55 mole % for the six following samples, which included the 0.58 mole % value of the eighth sample at the end of the test. The changes in gas composition are shown in Figure 5. After the second sampling (except for the small change in carbon dioxide) the gas composition remained nearly constant, the principal components being helium and nitrogen.

The composition in mole % of the gas within the canister after eight weeks of exposure at 550°C was helium, 76.8; nitrogen, 22.2; carbon dioxide, 0.58; argon, 0.28; oxygen, 0.08; carbon monoxide, <0.1; and hydrogen, methane, and nitrogen oxides each <0.01. The latter four values are the minimum laboratory reporting points for these gases.

Oxygen depletion can be attributed to any or a combination of three reactions: with the reducing glass; with the canister metal, especially the previously unexposed stainless steel of the canister lid; or with a foreign carbon source in the glass. Some oxygen consumption through metal oxidation and oxidation of the ferrous iron and other components in the glass was expected. The actual reactive source or sources are unknown, although the inner face of the canister lid, which had not been previously exposed to the high temperatures, was expected to oxidize, and upon the lid's removal to examine the glass surface it was observed to have a golden cast, indicating its oxidation. If oxygen depletion were entirely due to a completion reaction

Table 1. Mass Spectrometric Gas Analyses, Mole %.

Component	Sample Number and Time							
	WQR-01 WK 0	WQR-02 WK 2	WQR-03 WK 3	WQR-04 WK 4	WQR-05 WK 5	WQR-06 WK 6	WQR-07 WK 7	WQR-08 WK 8
He	74.7	77.3	77.4	77.1	77.5	77.5	77.5	76.8
N ₂	20.2	21.9	21.7	22.0	21.6	21.7	21.6	22.2
O ₂	4.81	0.05	0.04	0.05	0.05	0.06	0.06	0.08
Ar	0.23	0.27	0.28	0.27	0.27	0.28	0.27	0.28
CO ₂	0.10	0.47	0.52	0.55	0.56	0.55	0.55	0.58
CO	a	a	a	a	a	a	a	a
H ₂	b	b	b	b	b	b	b	b
CH ₄	b	b	b	b	b	b	b	b
NO _x	b	b	b	b	b	b	b	b

a. Less than 0.10 mole % for all samples.

b. Less than 0.01 mole % for all samples.

Figure 5. Gas composition as a function of testing time

with carbon, one mole of carbon dioxide would be formed for every mole of oxygen reacted, and the final mole % of carbon dioxide would be the same as that initially for oxygen, but such was not the case. The highest gas composition for carbon dioxide was that in the final sample, 0.58 mole % compared with the 4.81 mole % for oxygen in the first sample, or the estimated 5.4 mole % before heatup. This indicates that other oxidation reactions occurred, unless oxygen were bound to carbon in either the glass or the canister metal (304L stainless steel contains <0.03 wt% carbon). Although the analytical data for the second to the eighth gas sample shows a slight increase in carbon dioxide content (see Table 1), the increase is probably unreal and is due to analytical imprecision. The average of the six samples was 0.55 mole %, with the standard deviation being 0.02 mole %. A small amount of carbon dioxide might have been desorbed from the glass rather than formed by the oxidation reaction.

The test results did not show any gas pressurization of the canister by the generation and release of gases from the glass during the eight weeks of exposure at the transition temperature. In fact, there was a consumption of free gas as oxygen in the plenum reacted. There was no indication that any gas, other than the cover and radiogenic gases (generation of which could not be simulated), would accumulate in the canister plenum.

Water Samples

Following the test, the water condensing and sampling system previously described was put into place. The individual glassware items of the system were weighed before installation. The plenum gas was withdrawn, cooled in the bare stainless steel tubing and the condenser, and passed through the Drierite bed until the canister pressure reached 0 psig at 500°C. No droplets of moisture were observed in either the condenser or the collection flask. The external temperature of the collection apparatus did not increase when the plenum gases were released. When flow of gas ceased with pressure equilibrium, the condenser, collection flask, and Drierite container were removed and all openings sealed immediately, and then each item of equipment was reweighed. Only the Drierite container had a weight change; the initial weight was 1409.3 g, the final, 1409.7 g, a 0.4-g increase by water sorption.

(The balance was accurate to 0.1 g.) A small amount of the indicating Drierite at the bottom of the container at its inlet turned pink, also revealing the small amount of water sorption.

The water could be from two principal sources, the glass and the moisture originally present in the air in the canister plenum. The amount from the glass can be estimated from the difference between that in the air due to the air's relative humidity before canister closure, and the amount sorbed by the Drierite. The air remaining in the canister after it reached 0 psig has to be considered along with the air withdrawn with depressurization for determining a water balance. The relative humidity of the air in the building in which the unsealed canister was kept and in the canister, assumed in equilibrium, was about 35% at an air temperature of 24°C (75°F). This gives an absolute humidity of approximately 0.0065-lb H₂O/lb of dry air. With air being about 25 vol % of the 3.89 ft³ of gas in the canister plenum (dry helium being about 75 vol %) and dry air having a specific volume of 13.5 ft³/lb, the water content of the air and thus the plenum gas was calculated to be about 0.00047 lb, or 0.21 g, at the start of the test. With the pressure being 10.9 psig, i.e., 25.6 psia, in the canister at test completion before gas withdrawal and 14.7 psia afterwards, the volume of air withdrawn at the test control temperature (assumed constant during gas withdrawal) was 2.88 ft³. This left 3.89 ft³ in the canister plenum. Based on this data, the amount of total water that was in the plenum gas before canister depressurization is estimated to have been 0.9 g. By deleting the 0.21 g due to the air humidity, the water from the glass is estimated to be about 0.7 g. These rough calculations ignore the relatively small amount of water that was withdrawn with the weekly gas samples.

Considering that the amount of glass in the canister weighed about 4,340 lb or 1.97×10^6 g, the 0.7 g of water is insignificant, being equivalent to 0.4 ppm, or 0.00004 wt%. For comparison, commercial and synthetic silicate glasses contain about 0.01 to 0.03 wt% water as hydroxyl ions, and borate glasses about 0.2 to 0.5 wt% (Boulos and Kreidl 1972). The 0.4-ppm value is based on the assumption that all of the glass in the canister contributed to the water migration and release. Thus, it is concluded that the glass itself

was not a significant source of free liquids at the conditions of the test.

Glass samples taken from the canister prior to and following the eight-week test were also analyzed using fourier transform infrared (FTIR) spectroscopy to determine the water content. "Water content" here must be taken to include free and bound water as well as hydroxyl groups. The glass was determined to have a maximum water content of 0.15 wt%. The procedure and results are presented in the appendix. These results support the canister results in that the amount of water is very small and consistent for this class of glasses. It is also unlikely that the bound water would migrate from the glass matrix to become free water. However, this statement can only be taken as qualitative based on the accuracy of the FTIR procedure.

QUALITY ASSURANCE STATEMENT

This work was performed under QA Impact Level II requirements per the PNL-MA-70 QA manual and WVSP quality assurance plan, WTC-007 (rev. 2). Testing was executed under the test plan, "Waste Qualification Gas Pressurization Test for WVDP," WTC-007-17 (rev. 0). Laboratory data and observations are documented in PNL laboratory record book, LRB 50964. A reproduction copy of the LRB entries and other supporting documentation are in the WVSP Technical File; T6, of the FY 1990 Task 03 file index.

ACKNOWLEDGEMENTS

The authors appreciate the work of R. S. Holeman for setting up and operating the equipment and gathering the data for this study.

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APPENDIX

ANALYSIS OF WATER IN WASTE GLASS BY FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

APPENDIX

ANALYSIS OF WATER IN WASTE GLASS BY FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

The following describes the methods and results of analysis of two glass samples, one sample taken before the canister test and one sample taken after the test was completed, to determine the amount of water present in the glass.

1. Procedure

- Crush glass to - 80 mesh,
- Dilute with spectroscopic grade KBr and mix thoroughly,
- Press a pellet approximately 0.2 cm thick at = 5000 psi,
- Obtain transmission FTIR spectra,
- Calculate fraction of water present based on infrared absorbance.

2. Limitations

- Water absorption from the air on high surface area, freshly crushed glass will cause erroneously high values to be obtained.
- Preferred to do all sample preparations in a water-free glove box; not done for present measurements.

3. Data and analytical results of glass sample #1, taken from the canister before the test started.

Absorbance at 3445 cm	=	0.071 absorbance units (see Figures A.1 & A.2)
Weight glass sample	=	0.0138 g
Wt KBr	=	0.7272 g
Total (glass + KBr)	=	0.7410 g
Wt glass + KBr Used to make pellet	=	0.2165 g
Pellet thickness	=	0.195 cm
Pellet diameter	=	0.708 cm

$$\text{Grams glass in pellet} = \frac{0.0138 \text{ g glass}}{0.7410 \text{ g (glass + KBr)}} \times 0.2165 \text{ g glass + KBr in pellet} = 4.03 \times 10^{-3} \text{ g}$$

$$\text{Pellet volume} = \frac{(0.7084 \text{ cm})^2}{4} * \pi * 0.1947 \text{ cm} = 7.67 \times 10^{-2} \text{ cm}^3 \\ = 7.67 \times 10^{-5} \text{ liters}$$

$$\text{Molar extinction coefficient} = \frac{81 \text{ L} \cdot \text{abs units}}{\text{mol} \cdot \text{cm}}$$

(from Dodd, J. Appl. Phys., 37, 3911 1965)

Therefore:

$$\frac{\text{g water}}{\text{g glass}} = \frac{0.071 \text{ abs units}}{0.1947 \text{ cm}} * \frac{\text{mol} \cdot \text{cm}}{81 \text{ L} \cdot \text{abs units}} * \frac{7.67 * 10^{-5} \text{ L}}{4.032 * 10^{-3} \text{ g glass}} * \frac{18 \text{ g H}_2\text{O}}{1 \text{ mol H}_2\text{O}} \\ = 1.5 \times 10^{-3} \text{ g water/g glass} \\ \approx 0.15 \text{ wt\% water}$$

This value should probably be taken as the upper limit, because adsorption of water from the atmosphere onto freshly ground glass surfaces does occur. Glass #2 (sample taken after the canister test) gave qualitatively identical results.

HAZE.001
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b
s
o
r
b
a
n
c
e

A
.3

Wavenumber (cm-1)

N1c/IR

FIGURE A.1. IR Spectroscopy Scan

HAZE.001
GLASS 1 HAZELTON
Tue, Jun 5, 1990, 1:38:33 PM

GLASS 1 HAZELTON

FIGURE A.2. Magnification of Figure A.1 Scan from Wavenumber 3700 Through 3200

**DATE
FILMED**

8/19/94

END