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AUTHOR(S) K. L. Dykes, M. L. Meyer SRL PHONE NO. 5-6477

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Cover Letter S C UCNI U		

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File(WSRC-TR-90-165)
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May 21, 1991

Ms. W. F. Perrin, Technical Information Officer
U. S. Department of Energy
Savannah River Operations Office
Aiken, SC 29801

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367454

WSRC-TR-90-165

TRANSURANIC DRUM HYDROGEN EXPLOSION TESTS (U)

by

K.L. Dykes and M.L. Meyer

Publication Date: June 1991

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

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TRU DRUM HYDROGEN EXPLOSION TESTS (U)

by K.L. Dykes and M.L. Meyer

Westinghouse Savannah River Company
Savannah River Laboratory
Aiken, South Carolina 29808

SUMMARY

Radiolysis of transuranic (TRU) waste can produce flammable (>4%) mixtures of hydrogen gas in 55 gallon vented waste storage drums. Explosion testing was conducted at the E. I. duPont Explosion Hazards Laboratory to determine the minimum concentration at which a drum lid removal occurs. A secondary objective was to investigate the maximum pressure and rate of pressure rise as a function of hydrogen concentration.

Prior to beginning any drum explosion tests, small-scale pressure vessel tests and drum mixing tests were completed. The pressure vessel tests established a relationship between hydrogen concentration and the maximum pressure and pressure rise. These small-scale tests were used to establish the concentration range over which a drum lid removal might occur. Mixing tests were also conducted to determine the equilibration times for two different hydrogen-air mixtures in a TRU drum.

Nine successful drum explosion tests were conducted over a hydrogen concentration range of 13-36 % (v/v), test results suggest total integrity failure via drum lid removal will not occur below 15 % (v/v). Controlled small-scale pressure vessel tests were conducted over a range of 5-50 % (v/v) to determine the pressure and pressure rise as a function of hydrogen concentration. No similar relationship could be established for the drum explosion tests due to the variability in drum lid sealing and retaining ring closure. Mixing tests conducted at 5% and 25 % (v/v) indicate adding pure hydrogen to the middle of a drum causes some initial stratification along the drum length, but the air and hydrogen become well-mixed after 50 minutes.

BACKGROUND

TRU waste is currently disposed of at Savannah River Site (SRS) in polyvinylchloride (pvc) and polyethylene bags and sealed using a "horsetail". A "horsetail" is a standard waste bag closure method. It is created by tightly twisting the bag top and then taping the entire twisted bag length to secure the waste container. Waste cuts are contained in multiple bag layers for contamination control purposes and placed inside a high-density polyethylene drum liner. The liners are loaded into vented 55 gallon galvanized steel drums and the liner lid is sealed by coating the lid edge with Rayco® adhesive. The TRU drums are placed on concrete pads for interim storage[1].

Radiolysis causes bag degradation which may produce enough hydrogen to exceed the lower flammability limit of a gas-air mixture in the bags or liner. To prevent hydrogen buildup in the drum and liner, both lids are equipped with carbon composite filter vents to allow hydrogen to escape by natural diffusion. This filter vent, however, does not prevent the possibility of a flammable hydrogen-air mixture forming in the individual waste cut bags.

Tests to determine the hydrogen-air concentration required to cause a 55 gallon TRU waste drum deflagration were performed at the E. I. DuPont Explosion Hazards Laboratory, Remote Test Site, Chambers Works, N.J.

TEST PLAN

The major test objective was to determine the hydrogen-air mixture capable of causing a TRU drum integrity failure (drum lid removal) during an explosion. Additional objectives were to obtain the maximum pressure (Pmax) and rate of pressure rise (dP/dt) versus hydrogen concentration.

The tests took place in three segments: a small-scale (1.7 liter) pressure vessel test, TRU drum hydrogen-air mixing test, and a large-scale (55 gal.) TRU drum explosion test.

Pressure Vessel Test

The test objective was to determine the hydrogen concentration range where drum integrity failure would most likely occur. A 1.7 liter vessel was filled to slightly above ambient pressure with a known hydrogen concentration (5-50 % v/v) then ignited to determine the maximum pressure and rate of pressure rise. These parameters were monitored and transmitted to a computer for recording and storage purposes. Using this data as a baseline measurement for the drum explosion test, a hydrogen concentration range was selected by noting any steep increase in the maximum pressure and pressure rise plots. The baseline measurement could also be used to make a comparison and correlation between the pressure test vessel and TRU drums.

Drum Mixing Tests

A mixing test was conducted using two different hydrogen-air mixtures to determine the time necessary for equilibration. A modified TRU drum with sampling ports along the drum length was evacuated and filled with a gas mixture of 5 and 25% hydrogen (v/v). Gas samples were drawn from the drum every 20-30 minutes and analyzed using gas chromatography to determine the hydrogen equilibration time.

Drum Explosion Tests

The final segment of testing was to determine the concentration necessary to cause a TRU drum lid removal during a hydrogen explosion. The drum was filled with a hydrogen concentration between 12-36% (v/v) and allowed to equilibrate by natural diffusion. Prior to ignition by a hot-wire device, a gas sample was drawn into a 1 liter sampling Hoke® for verification of the hydrogen concentration by gas chromatography. The maximum temperature, pressure, and rate of pressure rise were recorded.

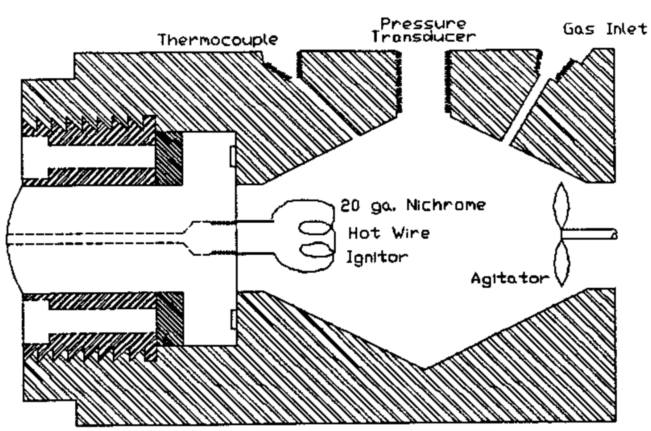
TEST EQUIPMENT

Three major pieces of equipment were used to perform testing: a 1.7 liter pressure vessel, a modified TRU drum to quantify mixing time, and a modified TRU drum used for the explosion testing. The test site provided the pressure testing vessel and SRS provided the modified TRU drums.

Pressure Vessel Test

The pressure vessel, a Nixon Reactor (Figure 1), was equipped with a thermocouple, pressure transducer, ignitor, agitator and a valved gas inlet. The pressure transducer and thermocouple relayed data to a computer and chart plotter for recording and processing.

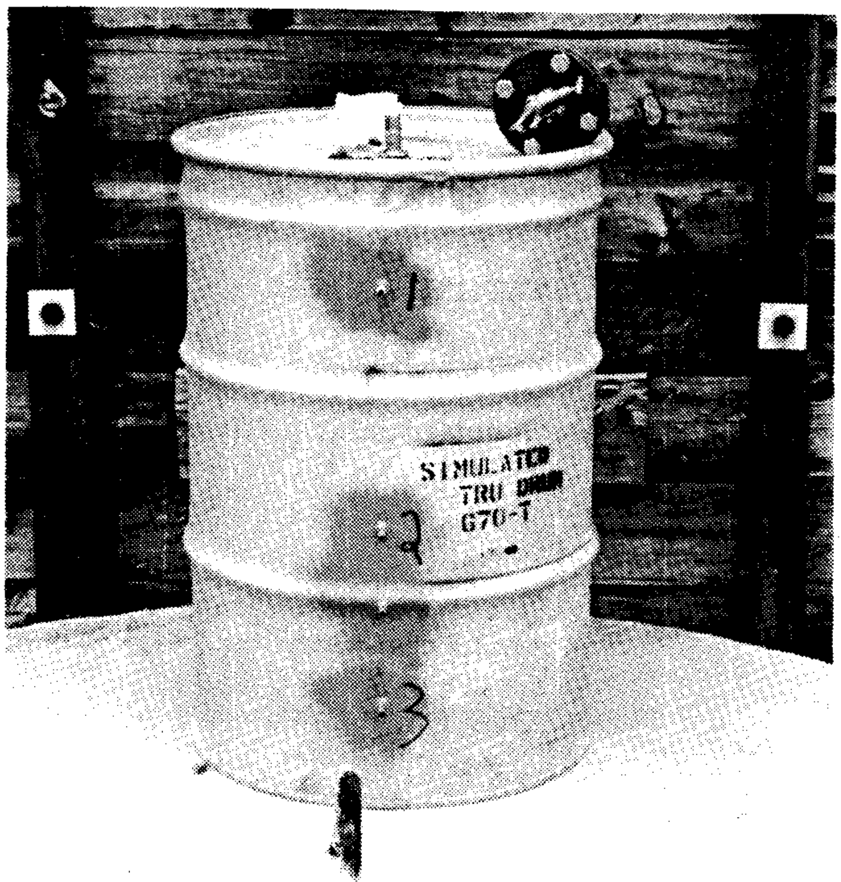
Figure 1: Sketch of the Nixon Reactor



Drum Mixing Tests

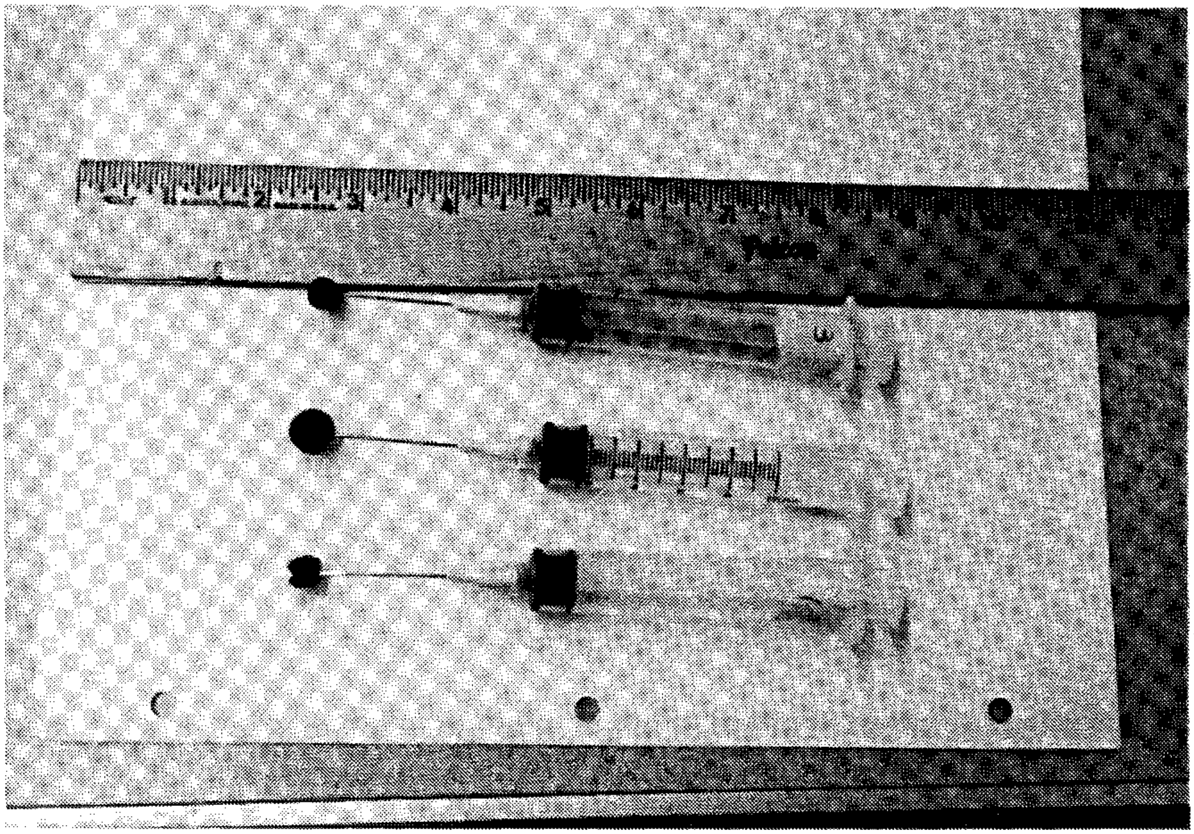
The mixing drum was a standard galvanized TRU drum containing a polyethylene liner with carbon-media filter vents in the drum and liner lids. A modified filter element allowed the drum vent to be plugged during testing and three ports along the drum height were used for gas sampling (Figure 2). Sampling ports were 1/8" NPT carbon steel pipe extending 2" from the side of the drum that were welded to the drum and sealed to the liner using RTV®.

Figure 2: Drum Sampling Ports



For sampling ease, septa were attached to the drum ports and 10 cc gas samples were drawn from the drum using plastic syringes (Figure 3). To ensure there was no measurable gas diffusion each syringe was subjected to a qualification test. The qualification consisted of filling the syringes with a 10% (v/v) hydrogen standard and allowing the syringes to sit for 30 minutes before injection into the gas chromatograph. No measurable hydrogen loss out of the syringes was detected.

Figure 3: Gas Sampling Syringes



Drum Explosion Tests

To perform drum testing a 1/2" - 14 NPT stainless steel pipe extending 2" from the top of the NucFil® filter vent was added to the standard filter vent1 (Figure 4). The pipe extension allowed the filter vent to be opened or closed during the drum evacuation and filling processes. Both the mixing and explosion test drums (Figure 5) were fitted with a 2" NPT carbon steel pipe located midway along the height of the drum for insertion of ignition and data acquisition devices. The pipe was inserted through and sealed to the galvanized drum and the polyethylene liner by welding to the drum and using a lock nut and RTV® sealant at the pipe-liner interface. Ignition and data acquisition devices were sealed to the drum using a modified flange (Figure 6). The flange had access ports for a pressure transducer, thermocouple, Nichrome ignitor, and gas inlets.

Figure 4: Modified Filter Vent

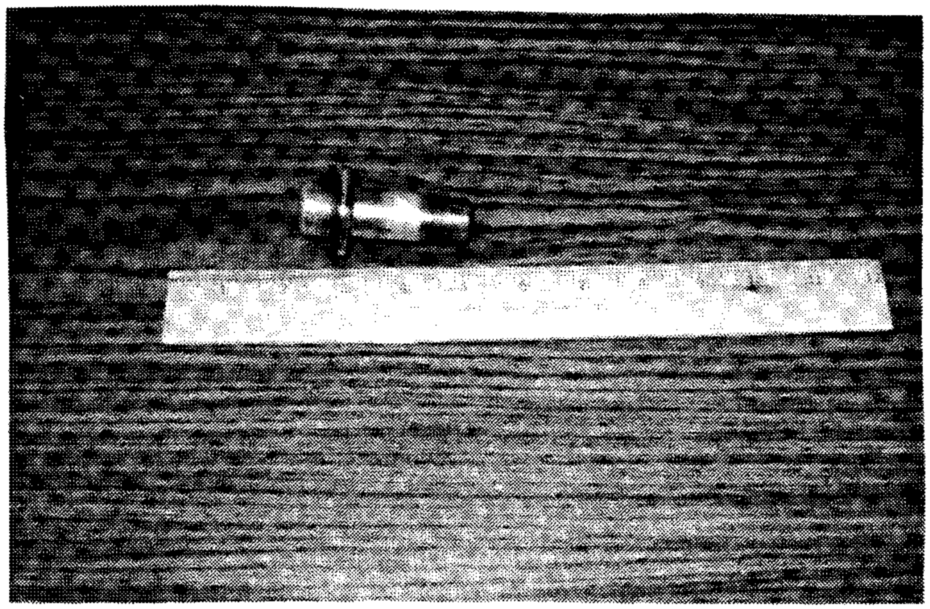


Figure 5: Modified Explosion and Mixing Test Drum

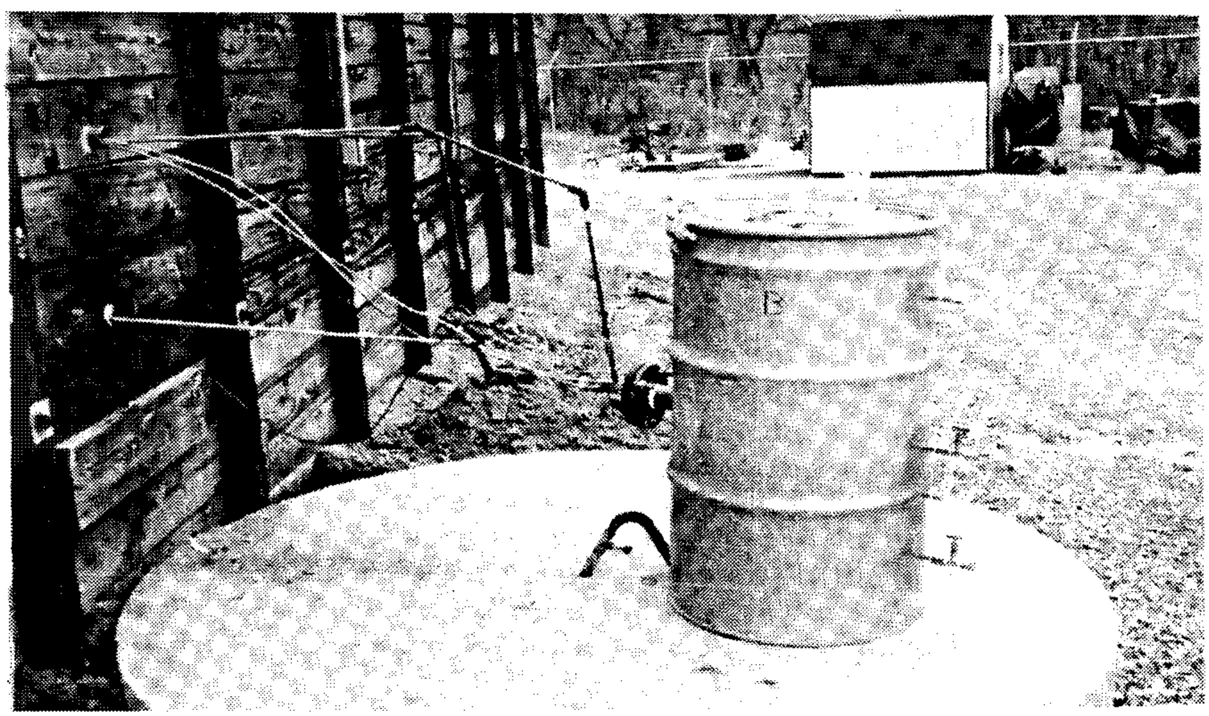
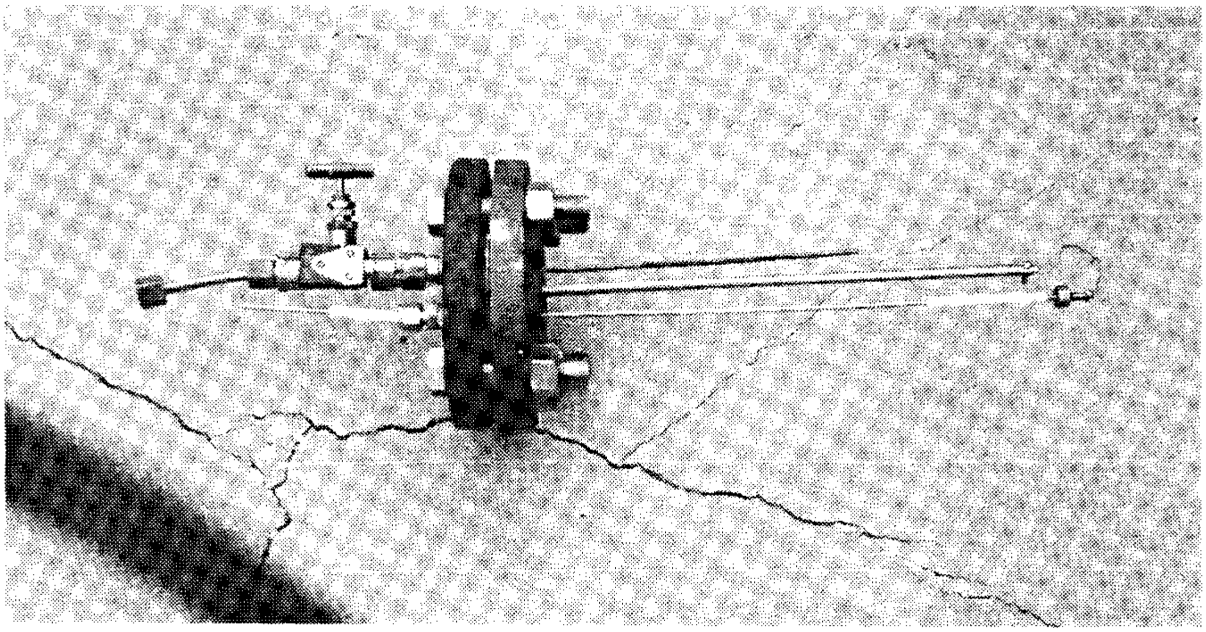


Figure 6: Modified Flange



TEST PROCEDURE

Pressure Vessel Test

Hydrogen was added to the pressure vessel in specific concentrations using partial pressure addition based on Dalton's law which states:

$$\frac{p_1}{p_t} = \frac{n_1}{n_t} = \text{mole fraction} = y_1$$

p_1 - partial pressure of component 1
p_t - total pressure
n_1 - moles of component 1
n_t - total moles

Before beginning a test, the pressure vessel was opened to the atmosphere for equilibration. When barometric pressure was reached in the vessel, the exhaust valve was closed and hydrogen was added through an inlet valve to the selected concentration. Once the desired hydrogen concentration was obtained, the hydrogen inlet valve was closed and the agitator started. The agitator was turned off after 10 minutes of mixing and current was increased in the hot wire until ignition developed. Data was recorded via a computer and pen recorder.

Drum Mixing Tests

Hydrogen concentrations for all drum tests were obtained by first reducing the drum pressure, and then adding the appropriate volume of hydrogen to return the drum pressure to atmospheric pressure. This was accomplished by plugging the filter vent with a rubber stopper and reducing the pressure in the drum via a vacuum line at the flange-drum interface (Figure 5). After allowing the pressure in the evacuated vessel to equilibrate (5-10 minutes), the apprtopriate volume of hydrogen was added through a valved inlet to the selected concentration. Drum pressure was always adjusted to atmospheric pressure to simulate existing conditions in the burial ground. At ≈ 20 minute intervals gas samples were simultaneously drawn from 3 different sampling ports (Figure 2). Gas concentrations were measured using gas chromatography.

Drum Explosion Tests

All explosion test drums were sealed and closed according to established procedures from the waste generators[2]. The filter vent was plugged and the drum pressure reduced via a vacuum line at the flange-drum interface. Drum pressure was then returned to atmospheric pressure by hydrogen addition to the selected concentration and allowed to equilibrate. Prior to igniting the mixture, a sample was drawn into a 1 liter evacuated Hoke® cylinder (sample vessel) for gas analysis. After the mixture was ignited and data recorded, samples were drawn from the Hoke® cylinder and analyzed using gas chromatography to determine the exact concentration.

RESULTS

Pressure Testing

As expected, maximum pressure and pressure rise per unit time were highly dependent on hydrogen concentration. Figures 7 and 8 are graphical representations of the empirical relationships established from the data in Table 1. A third-order polynomial gives a good fit for both figures with correlation coefficients of 0.99. Both the maximum pressure and pressure rise values occur at slightly above the stoichiometric concentration of hydrogen in air[3,4]. This can be explained using basic combustion theory. In combustion processes, excess oxidant is always introduced to overcome the nonideal conditions in the system, such as reaction kinetics and thermodynamics, while fuel is considered the limiting reagent. In a closed system, such as the 1.7 liter pressure vessel, there is a limited amount of fuel and oxidant present in the system. As the stoichiometric hydrogen concentration is exceeded, oxygen becomes the limiting reagent and hydrogen would be in excess. Under nonideal conditions, with a limited supply of oxygen, excess hydrogen would be required for complete combustion.

Table 1: Pressure Vessel Test Data

Hydrogen Concentration (%)	Initial Pressure (psig)	Maximum Pressure (psig)	dP/dt (psi/s)
5	0.77	1.8	0.1
10	1.63	45.3	368.8
15	2.59	78.9	4012
20	3.68	121.5	13039
25	4.9	186.4	30591.7
30	6.3	240	44132.3
35	7.92	253.5	51153.4
40	9.8	260.5	51780
45	12.03	268	49774
50	14.7	252	39994.9
30	6.3	236	46444
30	6.3	240.1	42188
15	2.59	76.8	3755
25	4.9	189.1	34051.1
5	0.77	20.5	229.2
10	1.63	45.3	329.8
20	3.68	119.8	13645
50	14.7	185	22784
47	13.04	263.6	46444
35	7.92	250.8	51102
45	12.03	258.3	47344
40	9.8	251.5	48346
5	0.77	1.5	0.1

Figure 7: Maximum Pressure versus Hydrogen Concentration

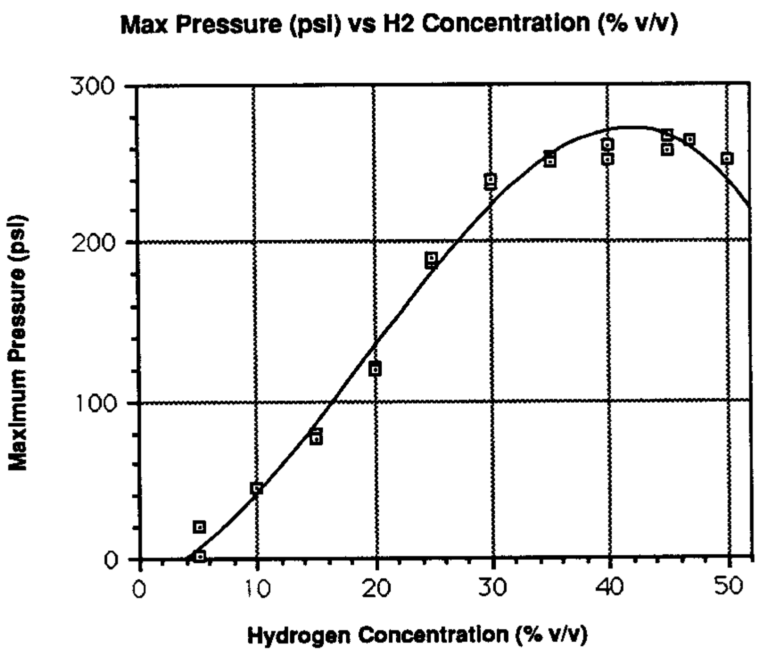
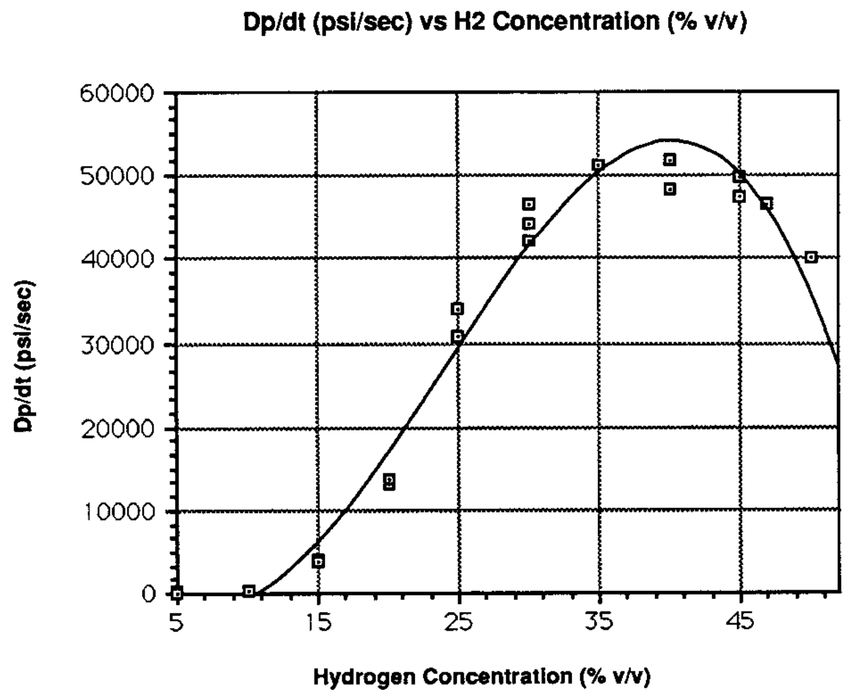


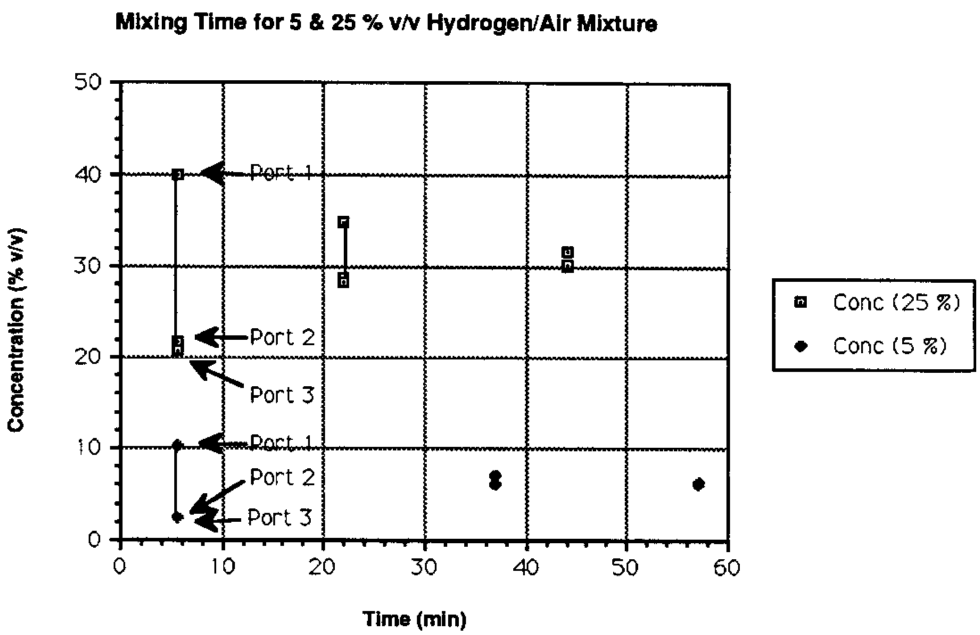
Figure 8: Maximum Pressure Rise versus Hydrogen Concentration



Mixing Drum Tests

Initially partial pressure addition was tried to obtain various hydrogen concentrations, but to obtain a stoichiometric hydrogen-air mixture (≈30% H2) using partial pressure addition in a 55 gallon drum a 6.0 psig increase in the drum pressure is necessary. As hydrogen was added to the drum, severe leaking occurred at the drum lid interface at a pressure of 1.5 psig. To minimize leaking, the lid was removed and the drum lid and lip were sanded smooth. The drum lid was reattached and sealed using a moldable gasketing compound. Pressurization of the drum to 1.5 psig after this sealing resulted in drum lid deformation. To prevent any future lid deformation and eliminate leaks at the drum lid interface, drums were evacuated prior to hydrogen addition to achieve concentrations of 5 and 25 % v/v. Figure 9 indicates the time necessary for equilibration at the three equispaced sampling ports (port 1,2,3). The ports are pictured in Figure 6 with port 1 being the drum top and port 3 the drum bottom. As seen in the plot with a 25 % (v/v) hydrogen-air mixture, there was a significant gas layering effect for the first 10-30 minutes. Based on these results, 50 minutes was chosen as an adequate equilibration time for the drum explosion tests.

Figure 9: Mixing Times for 5 and 25% Concentration Hydrogen-Air Mixtures



TRU Drum Explosion Tests

Hydrogen concentrations in the explosion tests were obtained using the same evacuation method used for the mixing test. The test results are shown in Table 2. During tests 1-8 successful reaction was only achieved twice.

Table 2: Drum Explosion Data

Test #	Filled Hydrogen Concentration (%)	Actual Hydrogen Concentration (%)	Standard Deviation (±)	Maximum Pressure (psig)	dP/dt Maximum (psi/s)	Observations
1†	15	N/A	N/A	N/A	N/A	No go
2	15	14.61	N/A	N/A	N/A	No go
3	25	N/A	N/A	124	9905	Lid Blown
4	20	12.843	0.604	93.4	23	Flame propagation
5	20	13.594	1.283	N/A	N/A	No go
6	25	28.219	0.134	N/A	N/A	No go
7	25	28.708	0.632	N/A	N/A	No go
8	20	18.806	1.572	N/A	N/A	No go
9††	20	35.329	0.458	105.2	15412	Lid Blown
10	15	16.95	N/A	137.5	14473	Lid Blown
11	15	14.893	0.098	69	566	Bulged
12	15	14.053	0.021	138	14149	Bulged
13	15	16.49	0.706	121.4	37970	Bulged
14	12	13.346	0.253	70.4	17795	Bulged
15†††	18	17.549	0.429	8.8	1	No go
16*	18	22.72	3.04	320	99624	Lid Blown
17	18	17.966	1.094	211.1	71303	Lid Blown
18	12	13.945	0.277	69.1	12688	Bulged

† - used 4 1/2 inch nichrome wire for ignition (Tests 1-8)
†† - used 3 inch wire for ignition (Tests 9-18)
††† - thermocouple shorted out power source
* - calculated concentration based on chromatography peak mass

Since the lower flammability limit of hydrogen in air is 4% (v/v), experimental procedures and the equipment set-up were reviewed after test #8. Although 2 1/2" of 20 gauge Nichrome wire had been used in the pressure vessel tests, a 4 1/2" length of wire was used for the drum explosion tests. This additional length effectively increased the resistance and reduced the wire heat load. These conditions would result in a cooler resistor and prevented the gas mixture from reaching autoignition temperature under the test conditions. Reduction of the wire length to 3" resulted in successful reactions for all of the tests except #15. Equipment inspection showed a high probability the ignition source in test #15 was shorted out by a thermocouple resting against the area where power was supplied to the drum. Repositioning and insulation of the thermocouple prevented any recurrence of this problem.

Supplying hydrogen to the drums using partial pressure addition generally proved to be within ±2% of the desired concentration. The actual concentrations reported in Table 2 are the averages of the gas chromatography readings taken prior to ignition. Standard deviations have been reported whenever more than one sample could be drawn and successfully analyzed. Due to an integrator area calculation error, the values presented for the hydrogen concentration and standard deviation in test number 16 were hand-calculated. This was accomplished by comparing the peak area mass of an unknown chromatograph concentration to that of a known gas standard chromatograph. Uncertainty analysis calculations account for the large variation in the standard deviation as compared to the other test runs.

The maximum pressure and pressure rise/time as a function of hydrogen concentration for all tests is shown in Figures 10 and 11. Figure 10 follows a well-defined pattern with the exception of a point at ≈ 14 % hydrogen. Since the point cannot be dismissed as an outlier, additional testing would be required to verify any functional relationship. Figure 11 follows a widely scattered pattern and is independent of the gas concentration. This is not surprising due to the large number of variables associated with sealing the drum lid (drum manufacturing tolerances and deviations, lid gasket seal, retaining ring and bolt closure).

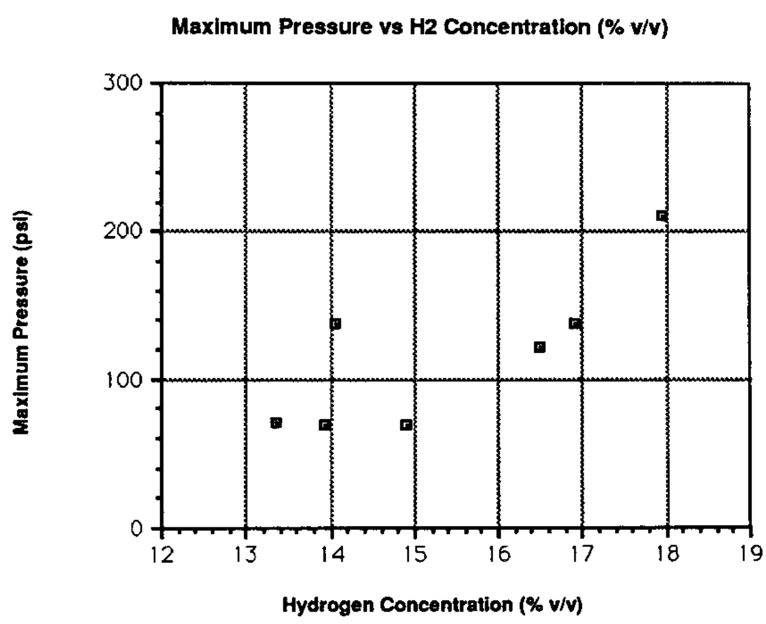


Figure 10: Maximum Pressure versus Hydrogen Concentration

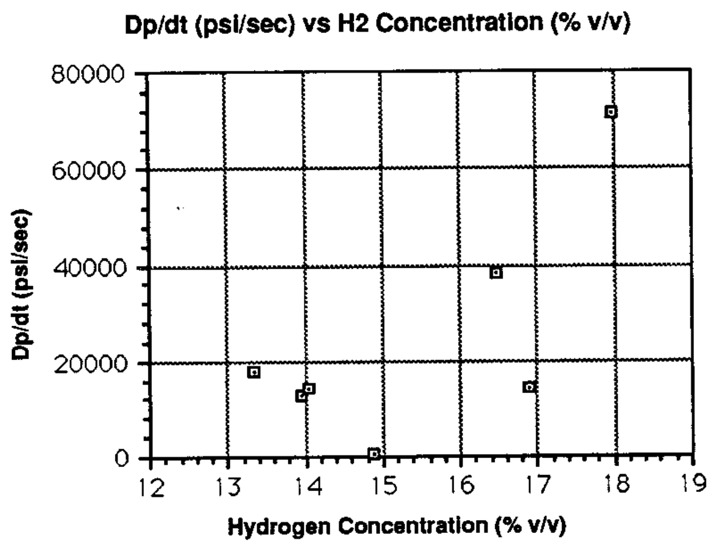


Figure 11: Maximum Pressure Rise versus Hydrogen Concentration

Observations made during each test show drum lid removal occurred at concentrations of ≈ 35%, 22%, 18%, and 17%. In the 5 successful tests at less than 17% only bulging of the drum top and bottoms occurred. Based on the limited number of data points, and lack of additional test time, a statistical probability for drum failure as a function of hydrogen concentration could not be determined.

Conclusions

Data suggests an explosive mixture up to 15 % (v/v) of hydrogen can be contained in a 55 gallon TRU drum without total integrity failure via lid removal. An empirical relationship was defined for the pressure and pressure rise as a function of hydrogen concentration for an experimental pressure vessel.

Since the limited number of tests and drum closure variables made it impossible to develop a similar description for the drum explosion tests, no direct correlation of the data could be made.

Quality Assurance

All data and detailed test procedures for this work are documented in DPSTN-4785 and DPSTN-4697 maintained by Keith Dykes and Michael Meyer respectively. The polynomial curves were developed using Cricketgraph 1.3 by Cricketsoftware. Detailed operating, test, and calibration procedures are documented in the above references.

Acknowledement

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