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COMBUSTIBILITY OF TETRAPHENYLBORATE SOLIDS (U)

by
D. D. Walker

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

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Richard M Wallace
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cc: M. D. Boersma, 773-42A
W. C. Perkins, 773-42A
D. T. Hobbs, 773-A
D. F. Bickford, 773-A
L. F. Landon, TNX
C. T. Randall, TNX
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N. R. Davis, 703-H
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SRL Records, 773-A

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To: G. T. Wright, 773-A

From: D. D. Walker, 773-A D. D. Walker

Combustibility of Tetraphenylborate Solids (U)

Summary

Liquid slurries expected under normal in-tank processing (ITP) operations are not ignitible because of their high water content. However, deposits of dry solids from the slurries are combustible and produce dense, black smoke when burned. The dry solids burn similarly to Styrofoam and more easily than sawdust. It is the opinion of fire hazard experts that a benzene vapor deflagration could ignite the dry solids. A tetraphenylborate solids fire will rapidly plug the waste tank HEPA ventilation filters due to the nature of the smoke produced. To prevent ignition and combustion of these solids, the waste tanks have been equipped with a nitrogen inerting system.

Introduction

In the in-tank process, radioactive salt solutions are decontaminated by precipitating radioactive cesium as its insoluble tetraphenylborate salt. Non-radioactive potassium is also precipitated. The potassium tetraphenylborate (KTPB) constitutes the majority of the precipitate. On a weight basis, a large fraction of the precipitate has an elemental composition similar to benzene, which is a flammable liquid. During normal operations, the organic precipitate is mixed with water and does not exceed 11 wt % solids. Because of the high water content, these slurries are not expected to be combustible. However, during the batch process the liquid level in the processing tank will vary greatly. Solids deposited on tank walls and cooling coils as the liquid level falls can potentially dry and pose a flammablilty hazard while the liquid level in the tank is low. The purpose of this investigation was to determine the potential for deposits to form, their flammability when dry, and the impact of a fire on the tank ventilation system.

Modern practice in the literature on fire safety tends to avoid the use of "flammable" to describe the combustion properties of solids. The following are general categories of fire hazards of solids: non-combustible, fire resistant, combustible, and spontaneously combustible. "Flammable" is used to describe liquids and implies a greater hazard than "combustible." Flammable liquids have flash points below 100°F, whereas combustible liquids have flash points above 100°F. In the appended reports, "flammable" is often substituted for "combustible."

Combustibility Studies

Samples of simulated KTPB slurries were prepared at the Savannah River Laboratory and were sent to J. E. Johnston at the Du Pont Engineering Test Center, Newark, DE, for combustibility experiments. The report of that work is given in Appendix A. The composition of the slurries is given in Table I.

J. E. Johnston and T. P. Garret (a senior consultant on fire and explosion physical properties in the duPont Engineering Services Division) concluded that:

(a) there is a significant fire potential from KTPB solids,

Dry KTPB is more combustible than wood dust and is similar to Styrofoam. A KTPB fire will propagate in both vertical and horizontal deposits. When burned, it produces sooty smoke similar to that from benzene fires.

(b) a benzene vapor deflagration is a credible ignition source for the solids,

KTPB is ignitible from a relatively brief exposure to flame, but not from small electrical sparks. An aqueous 10 wt % KTPB slurry could not be ignited by direct application of a flame.

(c) to prevent fires, tanks containing KTPB solids should be inerted unless all KTPB deposits can be kept saturated with water at all times.

A flame will propagate at oxygen levels as low as 12 volume %, but they may smolder below that level.

TABLE I
Composition of Tetraphenylborate Slurries

Slurry	A	A'	B	B'	C	C'*	D*
Wt% solids***	1%	2%	10%	10%	10%	10%	10%
	(unwashed)		(unwashed)		(washed)		(washed)
Radiation dose(rads)**	0	0	0	3.6×10^7	0	3.6×10^7	2.7×10^8
Component			Concentration (molar)				
Na^+	5.0		5.0		0.42		0.42
OH^-	1.6		1.4		0.076		0.076
NO_3^-	1.82		1.92		0.28		0.28
NO_2^-	0.59		0.59		0.026		0.026
AlO_2^-	0.27		0.31		0		0
SO_4^{2-}	0.14		0.14		0.006		0.006
CO_3^{2-}	0.16		0.16		0.007		0.007
Cl^-	0.022		0.022		0.0012		0.0012
F^-	0.015		0.015		0.0007		0.0007
PO_4^{3-}	0.008		0.008		0.0004		0.0004

---

*The component concentrations are for the slurry prior to the radiation treatment.

**A radiation dose of 1.45×10^8 rads is equivalent to 1 year of tank farm storage.

***The solids are 95.2 wt% KTPB, 4.0% sodium titanate, and 0.8% CsTPB.

Deposit Characterization

KTPB slurries which are unirradiated or have received only small radiation doses were found to coat metal surfaces and build up a layer of solids. A 10 wt% slurry which had received a large radiation dose, simulating two years of tank farm storage, drained rapidly from metal surfaces and did not build up a significant solids deposit. Three different slurries were used in this test, simulating three different times in the in-tank processing cycle. The first slurry was an unirradiated, unwashed 2 wt% solids slurry with a high salt content (Table I, Slurry A'). It simulates the process during the decontamination and production stages in Tank 48. The second slurry was an irradiated, washed, 10 wt% solids slurry (Table I, Slurry C'). It received a radiation dose of 3.6×10^7 rads, equivalent to three months of residence in the tank farm. It simulates the slurry after washing and during transfer to Tank 49, the precipitate feed tank for the Defense Waste Processing Facility (DWPF). The third slurry was a 10 wt % slurry which had received a radiation dose equivalent to two years of storage in the tank farm (Table I, Slurry D). This slurry simulates the material stored in the DWPF precipitate feed tank (Tank 49). To simulate the effects of changes in tank liquid level, small steel coupons with mill-scale surfaces similar to the tank walls were dipped into the slurries once a day for three weeks. The results are shown in Figure 1. The test clearly demonstrated the potential to accumulate solids on tank walls containing fresh or slightly irradiated slurries. However, slurries which have received large radiation doses drain easily from the coupons and do not build up significant amounts of solids. Details of the deposits are given in Table II.

Samples of slurry deposits were taken from a ventilated slurry tank (Tank W2) located at the Savannah River Laboratory's Engineering Test Facility (TNX). The tank holds simulated washed precipitate prior to feeding it to the Precipitate Hydrolysis Experimental Facility (PHEF). A water-line deposit of solids was present when the tank was emptied after use. The deposit varied considerably around the tank, but appeared to be between three and ten millimeters thick and between three and ten centimeters wide. Two samples were taken: the first, a few days after the tank level was lowered below the water-line deposit, the second five weeks later (during which time the tank was not used). The results from the analyses of these samples are listed in Table III. The second wall sample was a free flowing powder which was easily ignited despite the presence of water (27 wt%). The nitrate/organic ratio decreased on drying, whereas the ratio of nitrate to water in the deposit increased. These changes indicate that the wet deposit dried by a combination of drainage and evaporation.

The potential range of water content in KTPB deposits was studied in controlled humidity tests. Samples of four slurries (Table I, Slurries A', B', C, and D) were filtered and dried, leaving KTPB deposits with varying amounts of soluble salts. These dried slurries were placed in controlled humidity chambers, allowed to adsorb water, and were then analyzed for water content. The results are listed in Table IV. The major variables controlling the water content in a deposit are the salt content (whether the

TABLE II
Results of Slurry Deposit Tests on Metal Coupons

Slurry:	A'	B'	D
solids	2 wt%	10 wt%	10 wt%
radiation dose	0	3.6×10^7	2.9×10^8
Deposit thickness: after 10 days:	11 mm	5.4 mm	<.5 mm
after 22 days:	18 mm	16 mm	--
Deposit density (g/L)*	0.38	0.42	--
Water (wt%)	18	18	--
Insoluble solids (wt%)**	20	36	--
Soluble solids (wt%)***	62	46	--

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*Dried at 23±2°C at relative humidity of 60±10%.
**KTPB and sodium titanate.
**Sodium nitrate, nitrite, sulfate, and hydroxide.

TABLE III
Tank W-2 Sample Analyses

Component	Composition (wt %)		
Sample:	Bulk Slurry	Wall #1	Wall #2
Water	84.0	77.5	27.0
Insoluble solids	9.7	16.2	66.0
Soluble solids	6.3	6.3	7.0
Nitrate	1.35	1.50	2.4

TABLE IV
Water Content of KTPB Solids (23°C)

Source of Solids*	Weight % Water		
Relative Humidity:	31	52	93
A' (unwashed, unirradiated)	3.6±.3	26±1	74±1
B' (unwashed, irradiated)	0.2±.1	8.3±.1	72±1
C (washed, unirradiated)	0.2±.1	2.5±.1	32±2
C (washed, irradiated)	0.4±.1	2.2±.2	26±1

---

*See Table I. The slurries were drained of liquid and dried before use.

dried slurry was washed or unwashed), and the relative humidity of the environment. High water content is favored by high humidity and salt. The radiation history of the slurry appears to have less impact on the water content.

Typically, the ambient relative humidity levels are 40 to 100% in the tank farm. Inside of inerted tanks, this will be decreased due to dilution with the dry nitrogen. Dry nitrogen will comprise about 75% of the total gas flow through the tanks, so the humidity could be as low as 10 to 25% (ignoring evaporation of water in the tank). Thus, it is probable that the solids deposits in a tank will contain sufficiently low amounts of water to make them combustible under expected humidity conditions. Washed, irradiated precipitate (similar to the slurry expected in Tank 49) has the greatest tendency to dry out, but has the least tendency to deposit on the walls. Unirradiated, washed precipitate (similar to slurries expected in Tank 48 after the washing cycle) has a high tendency to dry out and also tends to form deposits.

It is expected that this material under most potential humidity conditions in the tank will be dry enough to ignite. The second sample removed from Tank W-2 is representative of these conditions and was found to be ignitible.

Smoke Characterization

A sample of simulated and dried KTPB slurry was prepared at the Savannah River Laboratory and was sent to L. E. Butler of the Du Pont Engineering Department for smoke characterization studies. The composition of the dried sample is given in Table V along with analyses of other samples described below. The report of Butler's work is reproduced in Appendix B. Additional information on smoke was obtained in by J. E. Johnston (Appendix A) and at SRL.

A tetraphenylborate solids fire produces dense, black smoke which forms large stringy pieces of soot which cling to available surfaces. Electron microscopy of soot collected on filters showed the ultimate particle size was of the order of 0.1 micron. Approximately 3 to 6% of the burned solids are converted to smoke (Table V). Analysis of the smoke indicates that it becomes depleted in the non-carbon components (B, K, and Cs) relative to the fuel solids, but that Cs is less depleted than potassium. From its black color, the smoke appears to be mostly carbon from the incomplete combustion of the phenyl rings in the tetraphenylborate solids. Under the temperature conditions of a flame, cesium and potassium could be volatilized as the metals, cations, or metaborates, which would explain the presence of these elements in the smoke. The lower boiling point of Cs relative to K may explain the enrichment of cesium in the smoke. From the composition of the smoke, it is possible to calculate the cesium radioactivity that would be released from a fire in the Tank Farm (see Appendix C).

A single HEPA filter used in the tank farm has a surface area of 250 ft^2 and is designed to stand a constant pressure drop of 10 inches of water without failing. Based on the results in Appendix B, it would require 0.95 pounds of smoke solids to reach a pressure drop of 10 inches of water in the standard HEPA filter. From this information it is possible to calculate the amount of dry solids which can be burned in a fire before the HEPA filters will break through (see Appendix C).

Explosivity

No evidence has been obtained that indicates that dried deposits of tetraphenylborate salts are explosive. Confined heating to 325°C, mechanical impact, and hot wire or flame contact did not produce hazardous responses (Appendix A). However, deposits derived from salt solutions contain both organic compounds and nitrate salts. When intimately mixed in proper proportions, this combination can form explosive mixtures. In spite of the evidence to the contrary, the potentially explosive nature of nitrate/organic mixtures should not be forgotten. Insofar as possible, these deposits should not be allowed to accumulate, dry out, or be heated.

Differential thermal analysis (DTA) was used to determine if the dried solids exhibit any low temperature exothermic reactions.

TABLE V
Composition of Tetraphenylborate Solids and Amount of Smoke Produced During Combustion

Sample:	1* 10 wt % washed	2 10 wt % unwashed	3** 10 wt % washed
Element		Composition (wt%)	
B	2.8	1.7	2.8
K	10.8	4.4	10.1
Cs	0.25	0.095	0.040
Smoke (% of initial solids)	3.0±1.5	6.1±.4	5.2±.2
Residue after burning (% of initial solids)	43±3	54±2	39±1
Smoke composition (wt%)			
B	0.61		
K	0.63		
Cs	0.20		

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*This sample was used by L. E. Butler in his smoke studies (Appendix B).

**This was sample "E" used by J. E. Johnston (Appendix A).

No evidence of an unusual thermal decomposition mode was found. The DTA curves are shown in Figure 2. A description of the samples is given in Table I. All samples, including reagent grade sodium tetraphenylborate, exhibited an exothermic reaction which was initiated around 300°C. The reaction peaked near 375°C. High levels of salt (Samples A' and B') decreased the exothermic response (presumably by dilution of the reactive organic compound) and increased the temperature of initiation. These results agree closely with the AIT (autoignition temperature) and AOT (auto-oxidation temperature) tests described in Appendix A.

Quality Assurance

Experimenal work at the Savannah River Site reported here was obtained in accordance with the SRL Quality Assurance Program. Experimental data and calculations are recorded in the following laboratory notebooks: DPSTN-4443, DPSTN-4611, and DPSTN-4749.

DDW:bcs

Figure 1.
Results of Dipping Tests in Different Slurries

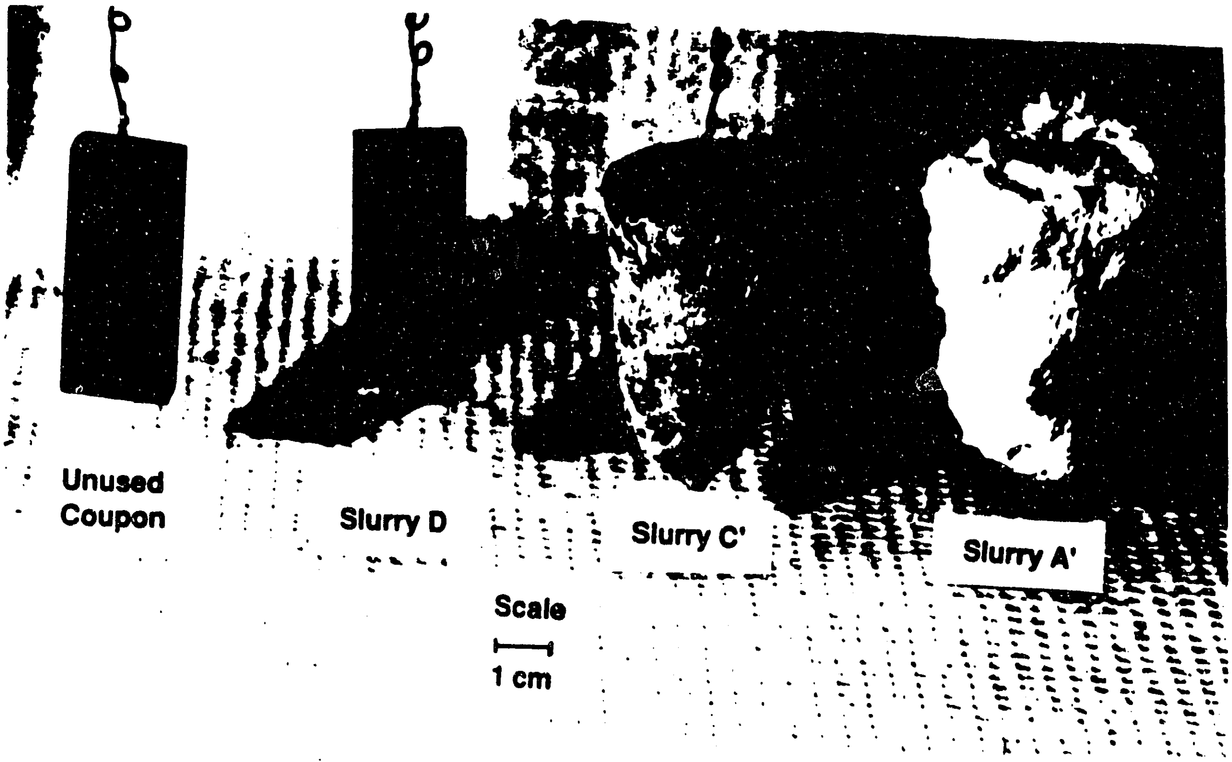
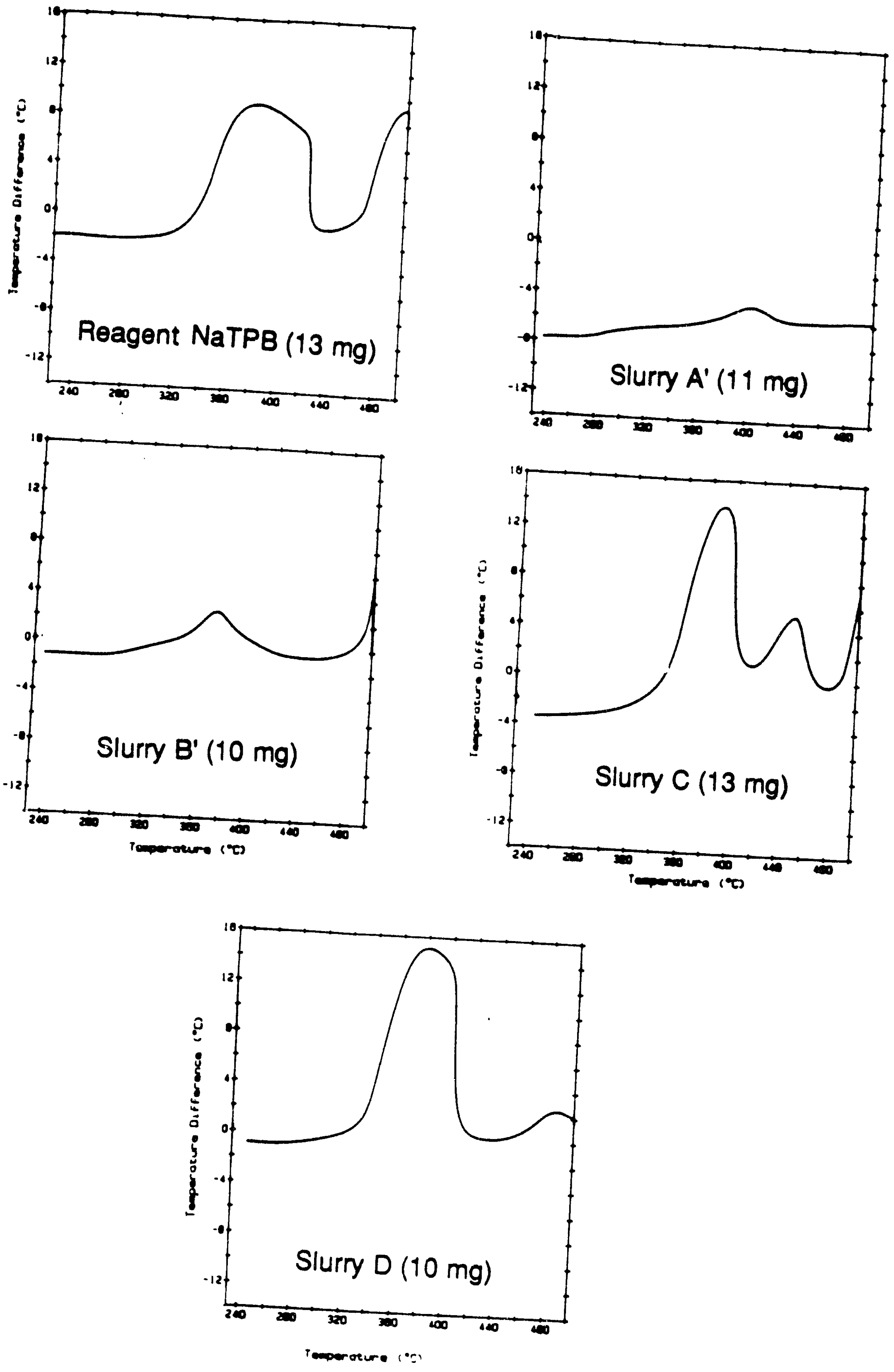


Figure 2.
DTA Curves for Dried KTPB Slurries



Appendix A

Report: Potassium Tetraphenyl Borate Flammability

The following report on the flammability and ignition characteristics of potassium tetraphenylborate slurries was prepared by James E. Johnston, Engineering Test Center, Engineering Service Division, E. I. du Pont de Nemours and Company, Newark, Delaware.

ESTABLISHED 1802
E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
P. O. Box 6090
NEWARK, DE 19714-6090

ENGINEERING DEPARTMENT

CC: B. A. Hamm-PD-SRP
R. A. Jacobs-SRL-SRP (704T)
R. W. Cox-Engg-L33W14
T. P. Garrett-ESD-L1355
R. T. Harris, Jr.-ESD-ETC
W. P. Flynn-ESD-ETC
IC-ETC

January 5, 1988

D. D. WALKER
PETROCHEMICALS
SAVANNAH RIVER PLANT (773A)

PETROCHEMICALS (ATOMIC ENERGY DIVISION) - SAVANNAH RIVER LABORATORY
ENVIRONMENTAL TESTING - EXPLOSION HAZARDS
POTASSIUM TETRAPHENYL BORATE FLAMMABILITY

The preparation of cesium waste for processing in the DWPF involves in-tank precipitation using potassium tetraphenyl borate (KTPB). There is concern that accumulation of potentially flammable KTPB solids on the interior of the tank could constitute a hazard. We have been asked to provide a summary statement on the implications of test work from the Explosion Hazards Lab of the Engineering Test Center, which was asked to perform a variety of tests on simulated KTPB deposits that would demonstrate:

- Thermal stability
- Ignition sensitivity
- Burning characteristics including minimum oxygen
- Ease of extinguishing with water

SUMMARY OF TEST RESULTS

Confined heating to 325°C, mechanical impact, and hot wire or flame contact with KTPB-water mixtures (or with foam over such mixtures containing benzene) do not produce hazardous responses. However, by other measures, the test results summarized below indicate significant fire potential for KTPB solids.

BENZENE FIRE: KTPB deposits fires produce a very sooty smoke. The carbon content of washed KTPB deposits is greater than 75%. The smoke is like that of a benzene fire. If the fire is primarily a benzene fire fed by thermal decomposition of KTPB, we would expect that the minimum oxygen concentration required to support combustion of KTPB deposits would be at least as high as it is for free benzene (10.5%). Test results below show 12-15.5%.

EXTINGUISHING THE FIRE: Horizontal surface fires of KTPB are readily extinguished by an overhead water mist, even when a physical barrier is present to prevent direct impingement from the nozzle. However, fires from a vertical surface produce a strong updraft that requires direct impingement of a water mist to extinguish.

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IGNITION: Although a discreet electrical spark of 11 joules will not ignite dried bulk solids, less than two seconds exposure to either the flame of a paper match or a continuous AC spark will ignite KTPB solids.

PROPAGATION: In horizontal burning tests which show no propagation for sawdust, a fire will propagate with KTPB deposits.

HUMIDITY: Humidity tests reported by D. D. Walker (SRP) indicate that humidity of 50-90% is needed to keep the moisture content of KTPB solids above 25% water, which Walker reports is still readily ignitible.

CONCLUSIONS

KTPB is a "combustible" solid that propagates a fire in atmospheres as low as 12% oxygen and smolders readily after its flames are extinguished until a significant water content is established. If its flames were not so readily extinguished with water, it would probably be considered a "flammable" solid.

The following considerations have guided our thinking:

1. The design of the tank and its associated airflow do not assure high humidity for all KTPB deposits.
2. Dry KTPB is more flammable than wood dust and is ignitible from a relatively brief (2 second) exposure to a flame.
3. The most credible, worst-case ignition sources are a vapor deflagration (which for benzene is a relatively slow phenomenon) or a lightning strike (which, though rare, would certainly have the necessary energy to ignite any combustible solid).
4. The ignition source of industrial fires and explosions is identified in only about 50% of the incidents.
5. In evaluating engineering controls for storage or processing of flammable materials, there is a major difference between conventional flammable materials and this case in which a radioactive release from the unsealed tank is totally unacceptable.

This leads to the further conclusion that, unless all KTPB deposits can be kept saturated with water at all times, the oxygen level in all portions of the tank must be reduced with an inert gas flow. T. P. Garrett concurs with this conclusion, based on a thorough review of both the above considerations and the test data presented below.

TEST RESULTS

SAMPLE IDENTIFICATION: All samples were provided by SRP containing a mixture of potassium tetraphenyl borate (KTPB) and related salts that simulate the expected composition of Tank 48, either in the form of aqueous slurries or dry solids, as follows: ("%" means percent solids)

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A: 1% unwashed
B: 10% unwashed
C: 10% washed
D: (dry) irradiated
E: Mixture of "C" and "D" provided in 2 lb lot for final burning tests.

All samples were dried overnight at 110°C before testing unless otherwise indicated.

THERMAL STABILITY BY CONFINED HEATING: Thermal stability was studied using 5 grams of Sample D under one atmosphere of air in a 100 cc Nixon Reactor which is a 316 stainless steel heavy wall vessel specially designed for explosion hazards determinations. It was equipped with a strain gauge pressure transducer mounted centrally in the side wall, 1/8" diameter stainless steel sheathed thermocouple in the sample and touching the bottom inside wall, and external electrical heating. The pressure transducer and thermocouple were connected to appropriate signal recording instrumentation. The reactor was heated at 10-12°C/minute.

No deviations from the applied heating rate were observed in the temperature record. Similarly, no deviations were observed in the pressure record.

Similar results were obtained for an oven-dried 5-gram portion of Sample B and a wet 10-gram portion of the same sample.

IMPACT SENSITIVITY: Drop weight tests were performed on our Du Pont 5 (or 2)kg impact apparatus. Approximately .02-.05 grams of sample were placed in a copper cup which was placed on a steel anvil, with a striker pin set on top of the sample. A 5-kg weight was dropped onto the striker pin from a maximum height of 55", thereby transmitting the impact to the sample. Observations may include flame, sparks, burnt streak on the piston, cup rupture, noise (sharp report), or burnt odor. Five repetitions were performed for each sample, with the following results:

Sample A:	No go
Sample B:	No go
Sample C:	No go
Sample D:	No go

AUTOIGNITION TEMPERATURE (AIT) AND AUTOOXIDATION TEMPERATURE (AOT): The AIT and AOT were determined in our spinning disk apparatus, which consists of a 1" x 12" quartz tube equipped with external electrical heating and a spinning disk axially mounted in its center. Various amounts of sample are dropped onto the disk and spun out against the heated side wall. A thermocouple mounted through the wall just above the disk is used to detect the AIT and AOT temperatures. The AIT is the lowest temperature that causes the sample to ignite; the AOT is the lowest temperature that causes an exothermic reaction. In conditions where heat cannot be dissipated and the sample temperature is at or higher than the AOT, exothermic decomposition heat may slowly increase until the AIT is reached.

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The results of spinning disk measurements are as follows:

	AIT, °C	AOT, °C
Sample A	> 600	None below AIT
Sample B	305	" " "
Sample C	320	290

FOAM IGNITION: A bottle of wet "A" sample was shaken well and transferred to an open beaker to sit for three days. A 0.5" layer of floating salt covered with a 0.1" layer of foam formed during that time. Direct contact with a fused (1350°C) nichrome wire had no effect on the foam. Direct contact by a propane torch flame caused the foam to collapse but no other effect.

Similar results were obtained for a freshly shaken portion of Sample C (no wait period was used).

IGNITION OF VAPOR OVER SLURRY: A mixture of 25 grams of dried Sample C with 225 grams of water (10% solids) with 0.25 cc of benzene in a 500 cc vessel (like the one discussed under Thermal Stability) was stirred slowly (30-35 RPM) for 1.5 hours to distribute the benzene, then rapidly for 1-minute to create foam. Hot wire ignition produced a normal deflagration, but inspection after the test suggests that the high speed mixing did not produce any foam. A similar test with overnight high speed agitation produced a thick 1-phase cream that would not ignite.

FLASH POINT OF BENZENE-SLURRY: A Fisher-Tag Closed Cup flash point apparatus shows no flash point for a sample prepared as above ("Ignition of Vapor over Slurry") in the temperature range 5-60°C.

VAPOR IGNITION OVER DRY SOLIDS: Initiation of a benzene deflagration over a 2-gram portion of Sample D (dry) produced no visible charring of the sample. An atmosphere of 2.8% benzene in air at 14.7 PSIA was ignited with a hot wire in a closed 1750 cc vessel, producing an explosion pressure of greater than 50 PSIG.

SPARK IGNITION OF SOLIDS: Point-to-Plate Apparatus: These tests were made on our "point to plate" test apparatus. A small amount of sample was placed into a dimple recess in a brass plate and a 10 kV continuous AC spark or 11 joule capacitance discharged spark (DC, transient), was passed through the sample from a point electrode 3/8" above the sample. (A human is capable of generating a spark of 0.015 joules.) Test results are as follows:

Sample A: No Go (AC or DC)
Sample B: No Go (AC or DC)
Sample C: Ignited in less than 2 seconds & propagated (AC)
No Go (DC)
Sample D: Ignited in less than 2 seconds & propagated (AC)
No Go (DC)
Sample E with 25 wt.% added water: No Go (DC)
No Go (AC < 2 sec)
Ignited & propagated (AC 4-5 sec)

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MINIMUM OXYGEN CONCENTRATION (MOC) FOR IGNITION OF DISPERSED DUST: A portion of sample E was ground with a mortar and pestle and dispersed by a blast of air containing various levels of oxygen in a 1.3 liter Hartman Dust Apparatus. Ignition was by a continuous 10 kV ac spark. The test result was:

MOC = 15.5% oxygen

MINIMUM OXYGEN CONCENTRATION (MOC) FOR HOT WIRE IGNITION: Direct contact ignition of 2-gram postions of sample C solids (dried overnight in a 110°C oven) in a 100 cc closed vessel (see Thermal Stability discussion above) in an atmosphere of air with reduced oxygen produced low pressure and temperature responses (2-9 psi, 2-30°C) that were approximately proportional to the oxygen concentration. Since there was insufficient oxygen in the vessel to react with all the relatively large sample, and since visual inspection of the residue showed only charring at the point of contact with the hot wire ignitor, the results indicate only that forced heating in a fuel rich environment will consume all available oxygen regardless of the oxygen concentration.

MINIMUM OXYGEN CONCENTRATION (MOC) FOR PROPAGATION OF IGNITED PILE OF SOLIDS: A small pile of Sample E was ignited with a 20-gauge nicrome hot wire on the bottom of a 1-liter glass resin kettle. The kettle contained a vapor phase agitator and an air supply distributor consisting of a loop of 0.25" diameter copper tube with holes positioned around the pile of solids on the bottom of the kettle. The air supply containing various levels of reduced oxygen was provided from a 2-gallon stirred autoclave. Test results are as follows:

15% oxygen:	Small even flame.
12% oxygen:	Flame went out when hot wire was deactivated.
10.5% oxygen:	No flame. Sample charred.
9 % oxygen:	No flame. Sample charred.

MINIMUM IGNITION ENERGY (MIE): A portion of sample C-D was ground with a mortar and pestle and dispersed by a blast of air in a 1.3 liter Hartman Dust Apparatus. The largest available capacitance discharge of 11 joules failed to ignite the sample. Therefore, the minimum ignition energy is greater than 11 joules.

BURNING: OPEN PILE: A 2-gram portion of Sample C was ignited on a piece of wood. It burned with a 3" high flame and produced an impressive amount of soot that floated 6' upward to the ceiling, leaving large stringy pieces on the wall.

A 5-gram portion of Sample E was directly contacted with a match. A small, slow burning flame started at the point of contact in 1-2 seconds. Within 30 seconds, the entire pile was burning, producing dense, black smoke but not a lot of heat.

BURNING: SOOT CHARACTERIZATION: Electron microscopy with energy dispersive X-ray analysis of soot collected on a common Millipore filter over a small burning pile of Sample C showed agglomerates with ultimate particle size of the order of 0.1 microns with no indications of the metallic elements contained in the salt.

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JEJ6:15

BURNING: HORIZONTAL (TROUGH): Dried sample was placed 3/8" deep in a 44" long V-shaped horizontal aluminum trough constructed from 3/16" thick 90° angle stock. The trough was located in a laboratory hood perpendicular to the airflow (cross-flow). Samples were ignited as indicated below and visually observed for propagation of flame with the following results:

	IGNITOR	IGNITION	PROPAGATION
Sample A:	Propane torch	None	
Sample B:	Propane torch	Yes	None. Self extinghished in 30 sec. Small flare-ups (1 sec or less).
Sample C:	Paper book match	Yes	0.5 ft/min Smoldered after flame extinguished. Required 28 cc of water from a spray bottle to fully extinguish both the flame and smoldering.
Sample D:	Propane torch	Yes	0.33 ft/min
Sawdust:	Propane torch	Smoldered	None
Flour:	Propane torch	Smoldered	None

BURNING: VERTICAL AND HORIZONTAL THIN CAKE: Metal lids taken from 2-gallon friction top metal cans were coated with wet solids and dried at 105°C. Final cake thickness was approximately 1/16".

	IGNITOR	IGNITION	PROPAGATION
Sample A:	Propane torch	None	
Sample B:	Propane torch	Yes (in 5 sec)	Horizontal: burned for 5 sec before self-extinguishing. Vertical: burned for 10 sec before self-extinguishing. Very sooty. Small flare-ups (1 sec or less).
Sample C:	Propane torch	Yes	Horizontal: burned for 5-10 sec before self-extinguishing. Vertical: burned for 20 seconds and propagated to 3X original area. Very Sooty.

BURNING: CAST HORIZONTAL THICK CAKE: A 12" x 1" x 1/4" form was set on a piece of 1/2" thick marinite insulation, filled with slurry, and dried at 110°C. Final cake thickness was 1/8-1/4".

JEJ:ese
JEJ6:15

	IGNITOR	IGNITION	PROPAGATION
Sample A:	(Too thin to cast)		
Sample B:	Propane torch	Yes	1.07 ft/min Material consumed in 1.5 min. Yellow flame & black sooty smoke.
Sample C:	Propane torch Very Sooty	Yes	.95 ft/min Material consumed in 3.6 min. Yellow flame & black sooty smoke.

BURNING: VERTICAL WITH DIPPED THICK CAKE: A 6" x 1/2" x 1/2" piece of marinite insulation was repeatedly dipped in slurry, and dried at 110°C. Final cake thickness was 1/8-1/4".

	IGNITOR	IGNITION	PROPAGATION
Sample A:	(Too thin to cast)		
Sample B:	Propane torch	Yes	Yes. (Total burn = 1.4 min.)
Sample C:	Propane torch	Yes	Yes. (Total burn = 5.8 min.)
Sample D:	Propane torch	Yes	Yes. (Total burn = 4.3 min.)

BURNING: TORCH IGNITION OF SLURRY SURFACE: Application of flame from a propane torch to the surface of the original wet slurries showed no ignition.

BURNING: EXTINGUISHING WITH WATER: A 20 gram pile of Sample C in a 4 x 5 x 1.5" aluminum foil dish was ignited with a propane torch. The flames were extinguished with 0.4 grams of water mist. Reignition of the benzene vapors still coming off the smoldering solids was accomplished 3 minutes after the flame was extinguished using an electric spark.

BURNING: EXTINGUISHING WITH FOG: The fire from a burning 50-gram pile of as-received Sample E could not be extinguished with a very fine water mist (ie., one that would barely wet your hand when placed in front of the fog nozzle), either by indirect of direct impingement. This and other tests with a documented set of nozzles are shown in the next table. A color videotape record of these tests was also made. These tests should not be used to compute water requirements for firefighting or to select nozzles for plant installation.

JEJ:ese
JEJ6:15

EXTINGUISHING FIRES OF SAMPLE E (As received)
Using Horizontal Water Fog Pattern 14" Above Sample Fire
Water Usage = 300 cc

RUN NO.	SAMPLE Wt., g	NOZZLE CODE/ CC H_2O	WATER PRESS. PSIG	EFFLUX TIME SEC	TEST MODE	EFFECT ON FIRE
1	-	(Very fine) ("Dry")			Unobstructed	None
2	30	40/300	100	17	Unobstructed	Flame & smoldering extinguished
3	30	24/300	100	37	Unobstructed	Flame extinguished but smoldering continued
4	30	40/300	80	37	4" wall next to burning pile	Flame extinguished smoldering fumes easily reignited
5	30	40/<1500	80	Partial H_2O use	4" wall next to burning pile	Flame & smoldering extinguished Residue could not be reignited with match
6	30	24/1500	80	"	"	"
7	Wetted solids dried in metal tray. Set vertical Nozzle at 1/2 ht of tray	40/1500	80	-	Pan facing away from fog Pan turned into fog	None (nozzle not above flames; fire draft strong) Flame and smoldering extinguished

Note: Fog nozzle hole diameter increases with nozzle number.
Nozzles obtained from Bete Fog Nozzles, Inc., 324 Wells Street, Greenfield, MA 01301
Nozzle Code 8 = 1/4FPJ8
Nozzle Code 24 = 1/4PJ24
Nozzle Code 40 = 1/4PJ40

Tests have been performed by L. C. Nixon, K. L. Branner, J. M. Chase, and E. B. Moore at the Explosion Hazards Laboratory at Carneys Point, New Jersey, and W. P. Flynn at the Engineering Test Center.

ENGINEERING SERVICE DIVISION
Engineering Test Center

James E. Johnston

J. E. Johnston

JEJ:ese
JEJ6:15

Appendix B
Report: Smoke from a KTPB Fire

The following report on the characteristics of smoke from a potassium tetraphenylborate solids fire was prepared by L. E. Butler, Particle Technology Group, Engineering Service Division, E. I. du Pont de Nemours and Company, Newark, Delaware.

DU PONT
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E. I. DU PONT DE NEMOURS & COMPANY
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P. O. Box 6090
NEWARK, DE 19714-6090

ENGINEERING DEPARTMENT

CC:
B. W. Wolstenholme-ESD-ETC
J. M. Wesley-ESD-L2316
R. W. Sylvester-ESD-L1363
J. B. Dunson-ESD-L1368
D. McIntosh-ESSD-L13W76
IC 13 - PT

November 28, 1988

D. D. WALKER
PETROCHEMICALS DEPARTMENT
SAVANNAH RIVER LABORATORY

PETROCHEMICALS - SAVANNAH RIVER LABORATORY - CHARACTERIZATION OF SMOKE FROM A KTPB FIRE

The attached report will summarize the work to characterize the smoke which would result from the a fire in the potassium tetraphenyl borate, KTPB, waste storage tank. The concern being what effect such smoke would have on the performance of the HEPA filters presently installed on the tank vents.

ENGINEERING SERVICE DIVISION

Larry E. Butler

L. E. Butler
Particle Technology Group

LEB:cu
leb1.18
Atch

SUMMARY

The characterization of the physical properties of the KTPB smoke yielded the following properties:

Mass median particle size(D50): 0.60 microns
Geometric standard deviations((D84/D16)^1/2)): 3.6
Concentration: 0.004 grains/dscf (5.71e-7 lb/dscf)
Conversion ratio: 3.58% (3.58 lb particulate/100 lb KTPB)

It is important to note that the reported conversion ratio was achieved under the following conditions:

1) with dry KTPB
2) with constant stirring of dry KTPB
3) direct flame ignition

KTPB is self extinguishing and flame propagation will be severely retarded in the presence of moisture or the absence of surface renewal.

The effect on the HEPA filters will, therefore, depend on the burning conditions inside the storage tank. If the above burning conditions are somehow established, the filter would be overwhelmed by the resulting smoke. It is believed the HEPA filters would be able to withstand the smoke from a limited fire in the tank since these conditions cannot be maintained in the tank should a fire occur. However, if a scenario in which these conditions can be maintained is believed possible, thought should be give to such options as more filters, prefilters, or fire suppression.

TEST SUMMARY

Two test setups were used to determine the above properties. Figure 1 illustrates the cascade impactor train used to determine the particle size distribution and concentration. Figure 2 shows the sample train for measuring the pressure drop vs particulate loading across a filter of similar efficiency as a HEPA filter. These figures are straight forward and require no explanation.

Tables 1 and 2 and their associated graphs, Figures 3 and 4, represent a summary of the particle size characterization. The mass mean particle, D50, is taken from the Figure 3 and 4. The D50 is a standard reference point in particle work and represents the particle diameter for which equal weights of particle are larger and smaller. The average D50 for the two test runs was 0.60 microns. The HEPA filters should be >99.9% efficient at collecting particle of this size.

The dP vs particulate loading tests were conducted using a Type AE glass fiber paper. This type of filter paper has the same

99.99% collection efficiency as a HEPA filter. The face velocity of the 0.1167 sq ft test filter was 9.0 acfm/sq ft. This is about double the design face velocity of 4.0 cfm/sq ft in order to accelerate testing. The resulting data is plotted on Figure 6 in the form of differential pressure, dP, vs particulate loading in pounds per square foot, lb/sq ft, of surface area. The results back calculated to 4.0 cfm/sq ft are also plotted on Figure 6. The complete test data summary is presented in Table 3.

Reference data for comparing the laboratory test data to the performance of a HEPA filter was taken from "Nuclear Air Cleaning Handbook", Oak Ridge Laboratory, Contract No. W-7 405-ENG-26. Figure 5 shows a plot of permissible long term dP for a HEPA filter and the duration of a pressure pulse. A HEPA filter is designed to withstand a constant pressure drop of 10" water. Using this dP reference and reading the corresponding particulate loading from Figure 6, an average loading of 0.0038 lb/sq ft is obtained. To equate the test filter loading to a HEPA filter, we use the fact that a proper sized HEPA is rated at 250 sq ft/1000 acfm. Your flow is 1000 acfm therefore you probably have 250 sq ft of surface area in the tank filters. Scaling up this loading to this size HEPA, we obtain a particulate loading of 0.95 lb of particulates. This would be equivalent to the burning of 26.5 lb of dry KTPB under ideal condition. The question which you must consider is whether or not 26.5 lb of dry KTPB is present in the tank. If the answer is yes, you will have to evaluate the likelihood of a potential ignition source being present to start a fire in the tank and keep it going.

You will also note on Figure 6 that the two test runs' curves differ. This difference in the upper end of the curves is believed due to the collapse of the filter cake lattice structure in Run 2 and the subsequent "packing" of the additional layers of particulates. Figure 5 shows that a normal HEPA filter will have collapsed before the filter cake structure does (Reference Figure 5).

Figure 7 is a confirmation that performance of the glass fiber filters dP and the filter cake dP vs flow rate is a linear relationship. This is an expected relationship for laminar flow through a filter.

FIGURE 1
KTPB SMOKE CHARACTERIZATION SET-UP

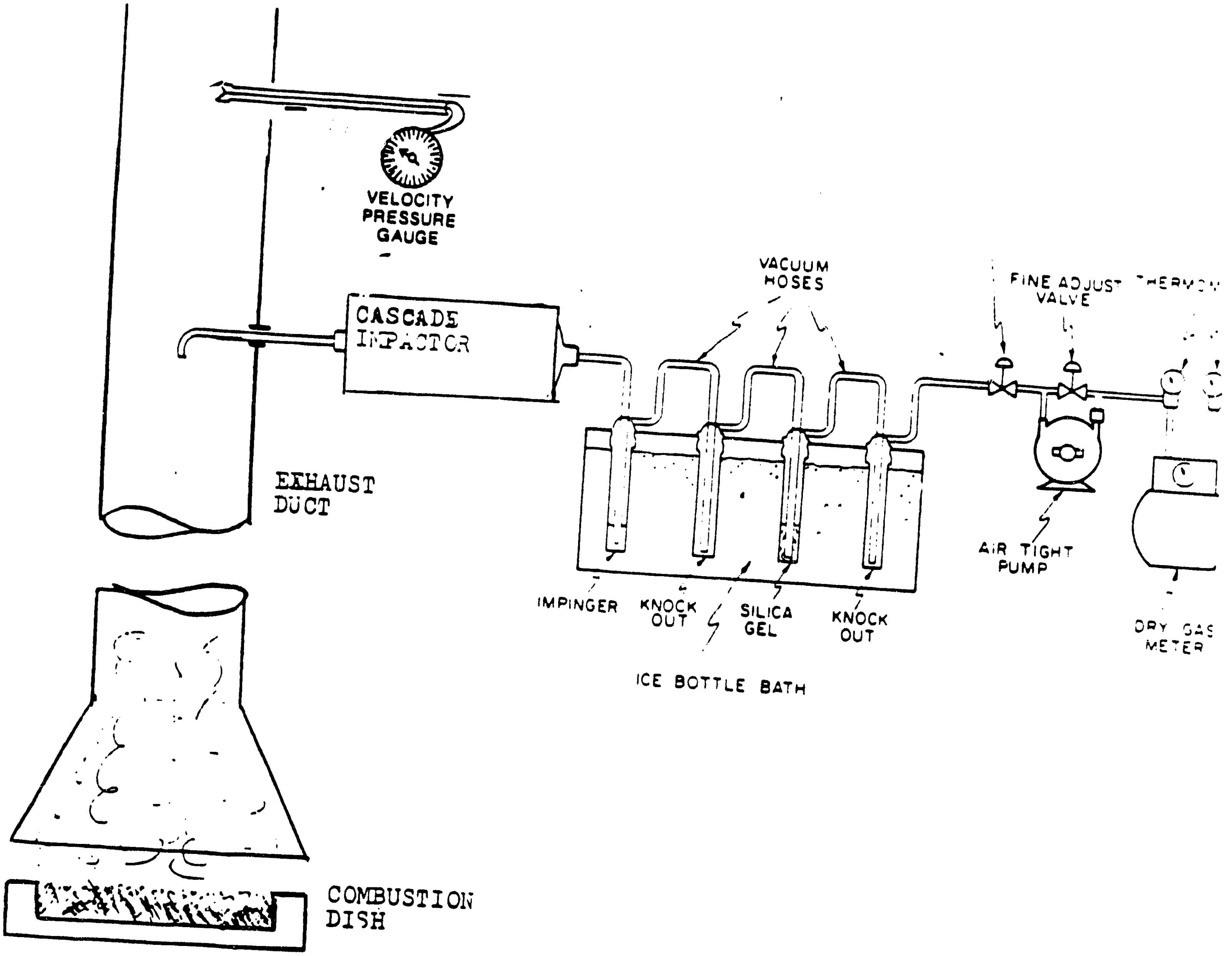


FIGURE 2

dP vs. PARTICULATE LOADING TEST

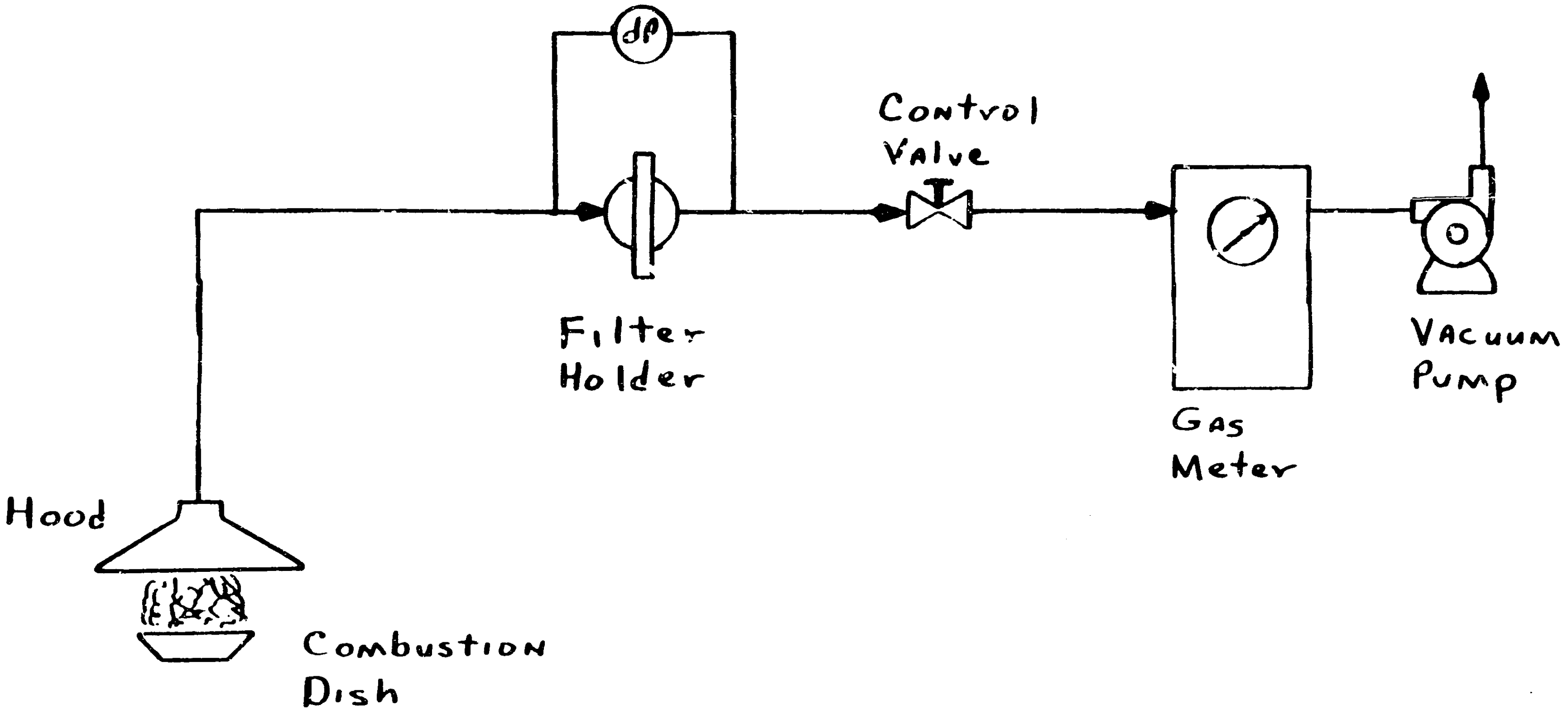


TABLE 1

PARTICLE SIZE ANALYSIS BY CASCADE IMPACTOR

For: KTPB	**RUN:** #1
DATE: 4/7/88	**Operator:** R.WILLIS

COLLECTION DATA

Cyclone Catch 0.000mg. Imp. Catch 2.080mg. Filter Catch 0.0[illegible]mg.
Particle Weight Conc.= .0037Gr/Dry SCF
Velocities: Duct= 610cm/sec : Nozzle= 736cm/sec 120.620% of Isokinetic
Wt. Conc. Corrected to Isokinetic = .0037Gr/Dry SCF

PARTICLE SIZE DISTRIBUTION:

STAGE No.	HOLE SIZE cm.dia.	HOLES No.	VELOCITY cm/sec	CUTSIZE micron	CATCH mg.	CUMULATIVE Wt. %
1	1.5000	1	206.02	17.486	0.100	5.2[illegible]
2	0.0757	90	831.78	1.966	0.210	14.9[illegible]41
3	0.0508	110	1634.93	1.093	0.230	25.5[illegible]
4	0.0299	110	2656.90	0.725	0.230	36.137[illegible]
5	0.0343	110	3612.06	0.568	0.190	44.670
6	0.0300	105	4996.70	0.436	0.250	56.05[illegible]
7	0.0259	105	6806.90	0.329	0.250	67.84[illegible]
8	0.0259	78	9456.49	0.244	0.240	78.9[illegible]
9	0.0237	56	14110.40	0.196	0.180	87.1[illegible]
10	0.0259	40	23740.55	0.120	0.060	89.8[illegible]
11	0.0259	36	56113.19	0.030	0.140	96.[illegible]

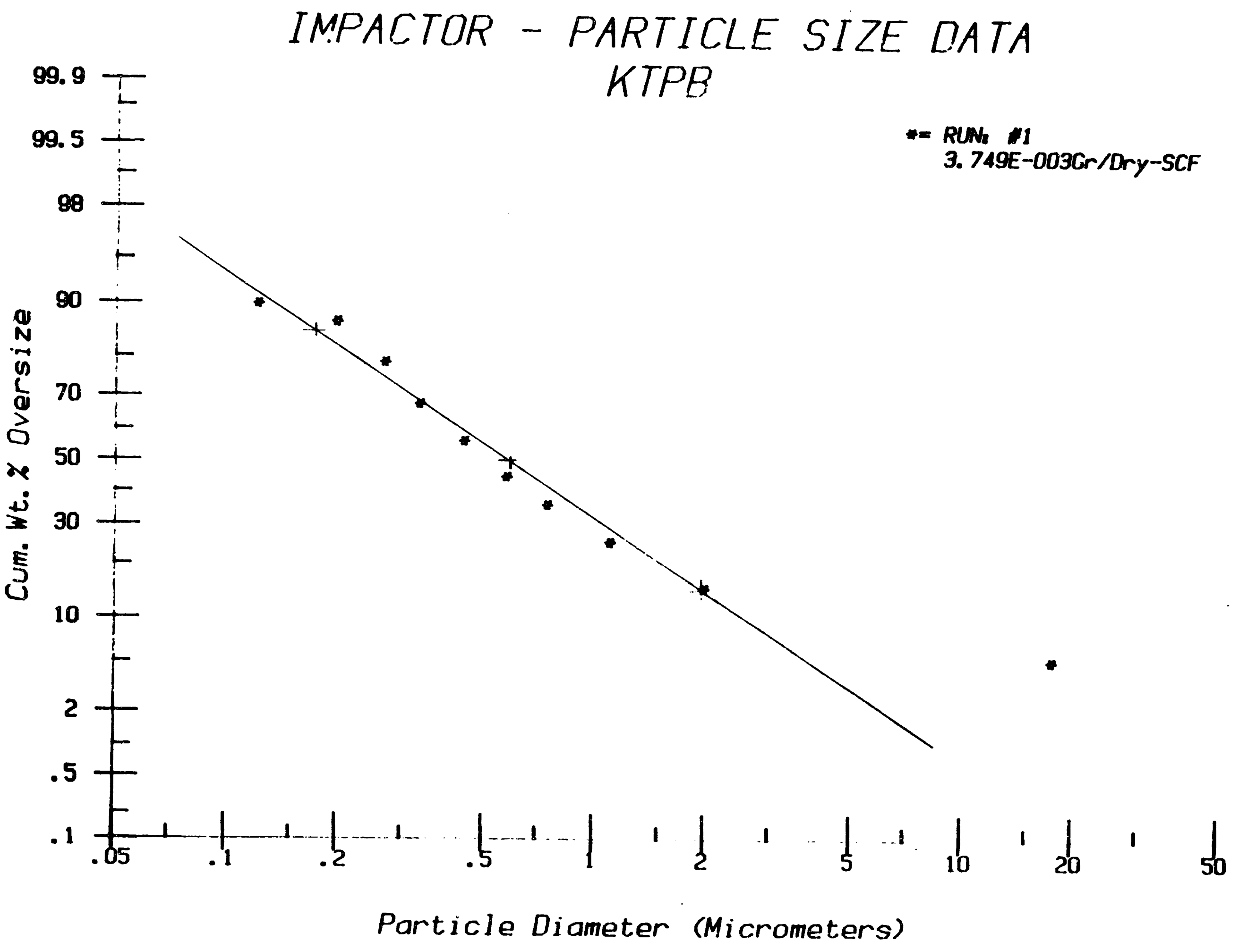
A calculated correction to Isokinetic has been made for Cumulative Wt. %

SAMPLING DATA
Nozzle Dia.= 0.794 cm.
Sample Time= 12.670 Min.
Sample Flow= 0.434 gm/sec.
Impactor dP= 500.0 mm-Hg.
Particle Dens. Assumed= 1.0 gm/cm^3

DUCT GAS DATA
Temp. 21.1 Deg.C
Press.= 760.0 mm-Hg Abs.
Visc.= 0.0001791 Cp
Density= 0.0011931 gm/cm^3
Humidity= 0.0128900 Mol/Mol
Wet Mol Wt.= 28.81
Ht.Cap.Ratio, k= 1.40

IMPACTOR DATA
Temp. 21.1 Deg.C
Press. 760.0 mm-Hg Abs.
Visc.= 0.0001791 Cp
Density= 0.0011931 gm/cm^3

FIGURE 3


IMPACTOR - PARTICLE SIZE DATA
KTPB
*= RUN: #1
3.749E-003Gr/Dry-SCF
Cum. Wt. % Oversize
99.9
99.5
98
90
70
50
30
10
2
.5
.1
.05
.1
.2
.5
1
2
5
10
20
50
Particle Diameter (Micrometers)

PARTICLE SIZE ANALYSIS BY CASCADE IMPACTOR

For: KTPB
DATE: 4/7/88

RUN: #2
Operator: R.WILLIS

COLLECTION DATA

Cyclone Catch 0.000mg. Imp. Catch 2.000mg. Filter Catch 0.020mg.
Particle Weight Conc.= .0036Gr/Dry SCF
Velocities: Duct= 610cm/sec : Nozzle= 697cm/sec 114.233% of Isokinetic
Wt. Conc. Corrected to Isokinetic = .0036Gr/Dry SCF

PARTICLE SIZE DISTRIBUTION:

STAGE No.	HOLE SIZE cm.dia.	HOLES No.	VELOCITY cm/sec	CUTSIZE micron	CATCH mg.	CUMULATIVE Wt. %
1	1.5000	1	195.11	17.991	0.130	7.0514
2	0.0787	90	787.75	2.023	0.260	19.8886
3	0.0508	110	1548.37	1.125	0.190	29.2386
4	0.0399	110	2516.24	0.758	0.210	39.5[illegible]36
5	0.0343	110	3420.85	0.566	0.140	46.4[illegible]4
6	0.0300	105	4726.49	0.450	0.180	55.2[illegible]5
7	0.0259	105	6446.53	0.341	0.230	66.5919
8	0.0259	78	8955.85	0.274	0.18[illegible]	75.435
9	0.0259	56	13363.37	0.204	0.120	81.3316
10	0.0259	40	22483.69	0.126	0.150	88.700
11	0.0259	36	53142.18	0.032	0.210	99.0175

A calculated correction to Isokinetic has been made for Cumulative Wt. %

SAMPLING DATA
Nozzle Dia.= 0.794 cm.
Sample Time= 12.850 Min.
Sample Flow= 0.411 gm/sec.
Impactor dP= 500.0 mm-Hg.
Particle Dens. Assumed= 1.0 gm/cm^3

DUCT GAS DATA
Temp. 21.1 Deg.C
Press.= 760.0 mm-Hg Abs.
Visc.= 0.0001791 Cp
Density= 0.0011931 gm/cm^3
Humidity= 0.0129900 Mol/Mol
Wet Mol Wt.= 28.81
Ht.Cap.Ratio, k= 1.40

IMPACTOR DATA
Temp. 21.1 Deg.C
Press. 760.0 mm-Hg Abs.
Visc.= 0.0001791 Cp
Density= 0.0011931 gm/cm^3

FIGURE 4

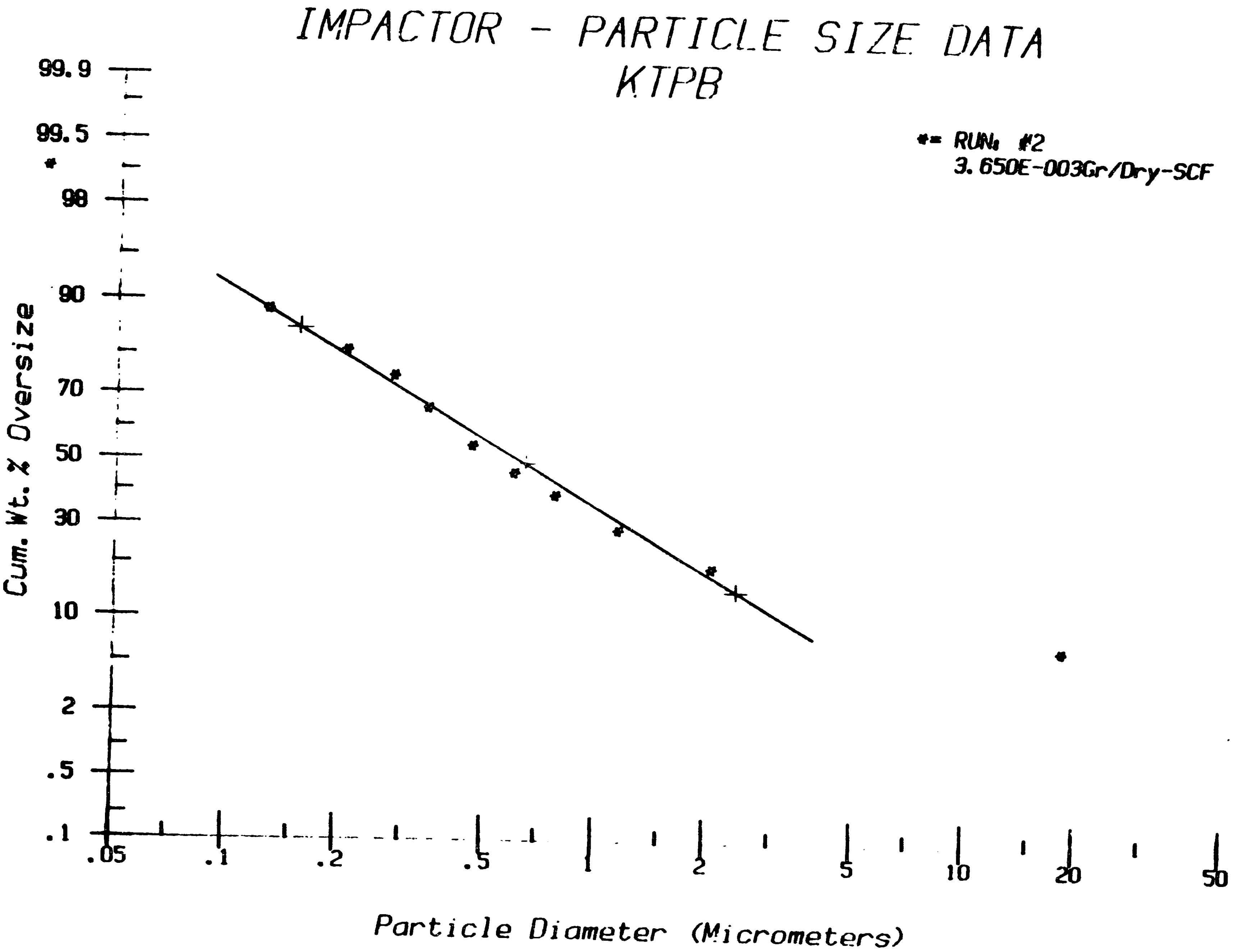

IMPACTOR - PARTICLE SIZE DATA
KTPB
*= RUN: #2
3.650E-003Gr/Dry-SCF
Cum. Wt. % Oversize
99.9
99.5
98
90
70
50
30
10
2
.5
.1
.05
.1
.2
.5
1
2
5
10
20
50
Particle Diameter (Micrometers)

TABLE 3

SAVANNAH RIVER LABORATORY KTPB SMOKE DATA SUMMARY
PREPARED BY: LARRY E. BUTLER DATE: 11/28/88

GENERAL INFORMATIOM
FILTER AREA: 0.1167 sqft
HEPA DESIGN FACE VELOCITY: 4.0 acfm/sqft
SURFACE AREA RATING:250 sq.ft./1000 acfm

RUN #1

TEST VELOCITY: 1.04 acfm
FACE VELOCITY: 8.91 acfm/sqft

dP	dP	PARTICULATE LOADING			mg/g of BURNED
" H2O	" H2O	mg	lb	lb/sqft	MATERIAL
4.9	0	0.0	0.00000	0.0000	0.00
13.7	8.83	124.5	0.00027	0.0023	23.90
20.4	15.53	196.4	0.00043	0.0037	45.00
29.3	24.43	246.7	0.00054	0.0047	35.90
35.6	30.73	309.5	0.00068	0.0058	44.90
40.1	35.23	386.9	0.00085	0.0073	35.20
43.6	38.73	423.3	0.00093	0.0080	45.50

RUN #2

TEST VELOCITY: 1.05 acfm
FACE VELOCITY: 9.00 acfm/sqft

dP	dP	PARTICULATE LOADING			mg/g of BURNED
" H2O	" H2O	mg	lb	lb/sqft	MATERIAL
4.8	0	0.0	0.00000	0.0000	0.00
8.9	4.1	55.0	0.00012	0.0010	32.60
11.7	6.9	92.0	0.00020	0.0017	24.40
10.1	5.3	115.0	0.00025	0.0022	13.70
13.0	8.2	176.0	0.00039	0.0033	31.90
32.9	28.1	227.0	0.00050	0.0043	34.00
66.7	61.9	311.0	0.00069	0.0059	46.60
86.6	81.8	393.0	0.00087	0.0074	51.60

AVERAGE(ALL DATA): 35.78 mg/g
OVERALL RATIO: 3.58 %

NO. 31.288. 10 DIVISIONS PER INCH (70 DIVISIONS) BY FOUR 2½-INCH CYCLES RATIO RULING. codex IN STOCK DIRECT FROM CODEX BOOK CO., NORWOOD, MASS 02062 U.S.A.

FIGURE 5

PREMISSIBLE LONG TERM dP FOR HEPA FILTERS

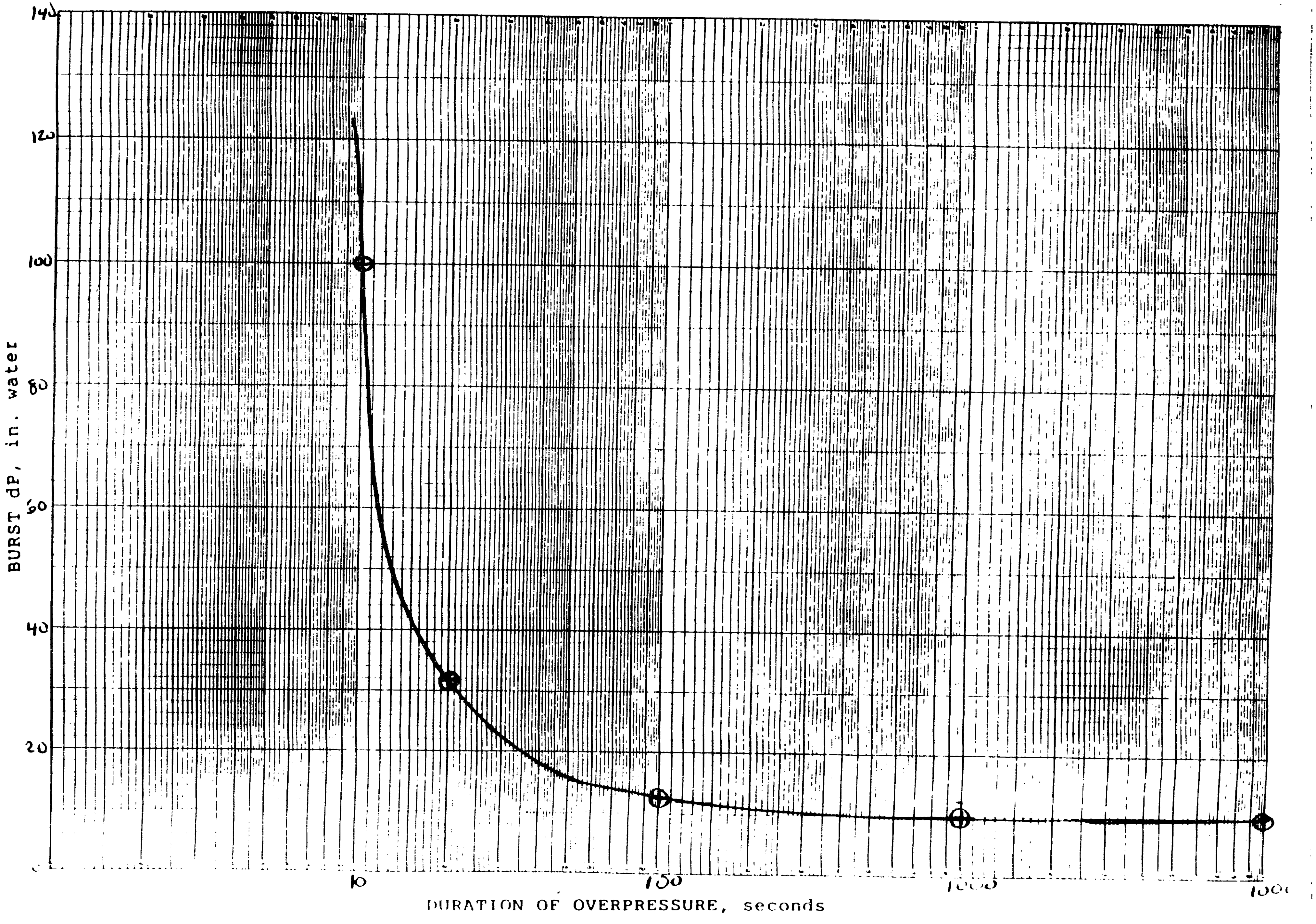


TABLE 3, continued

REFERENCE DATA

dP vs. VARYING FACE VELOCITY USING A SINGLE FILTER

SAMPLE RATE acfm	FACE VELOCITY acfm/sqft	dP in. H2O
0.25	2.14	0.64
0.48	4.11	1.20
0.73	6.26	1.70
0.94	8.05	2.50

dP vs. VARYING FACE VELOCITY USING MULTIPLE FILTERS

SAMPLE RATE acfm	FACE VELOCITY acfm/sqft	dP in. H2O
0.25	2.14	1.91
0.48	4.11	3.72
0.73	6.26	5.11
0.94	8.05	7.51

dP vs. VARYING FACE VELOCITY USING PARTICULATE LOADED FILTER

SAMPLE RATE acfm	FACE VELOCITY acfm/sqft	dP in. H2O
0.25	2.14	11.5
0.48	4.11	25.2
0.73	6.26	32.9
0.94	8.05	45.2

K+E 10 X 10 TO THE CENTIMETER 18 X 24 CM
KEUFFEL & ESSER CO.
46 1610

FIGURE 6

dP vs. PARTICULATE LOADING

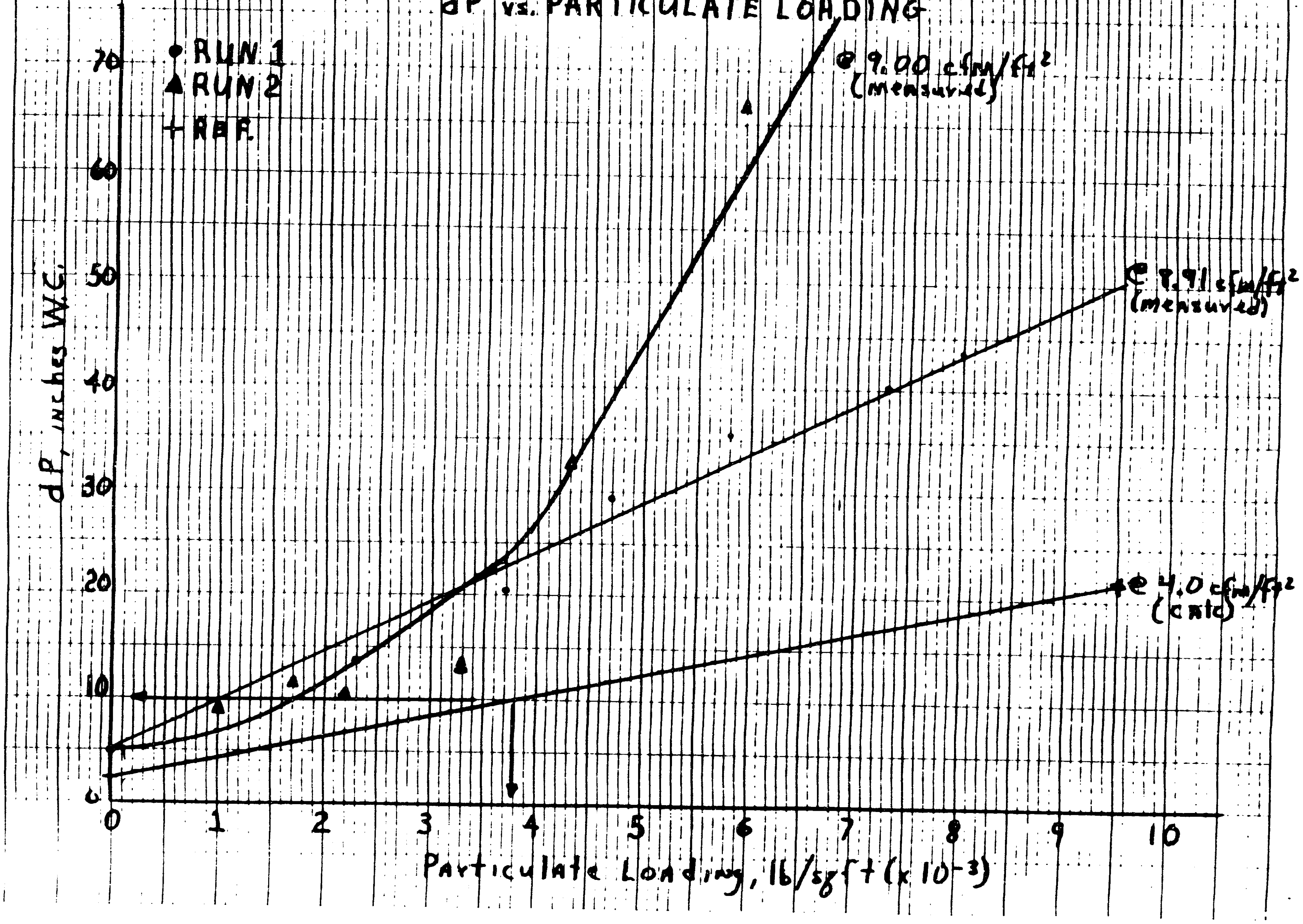
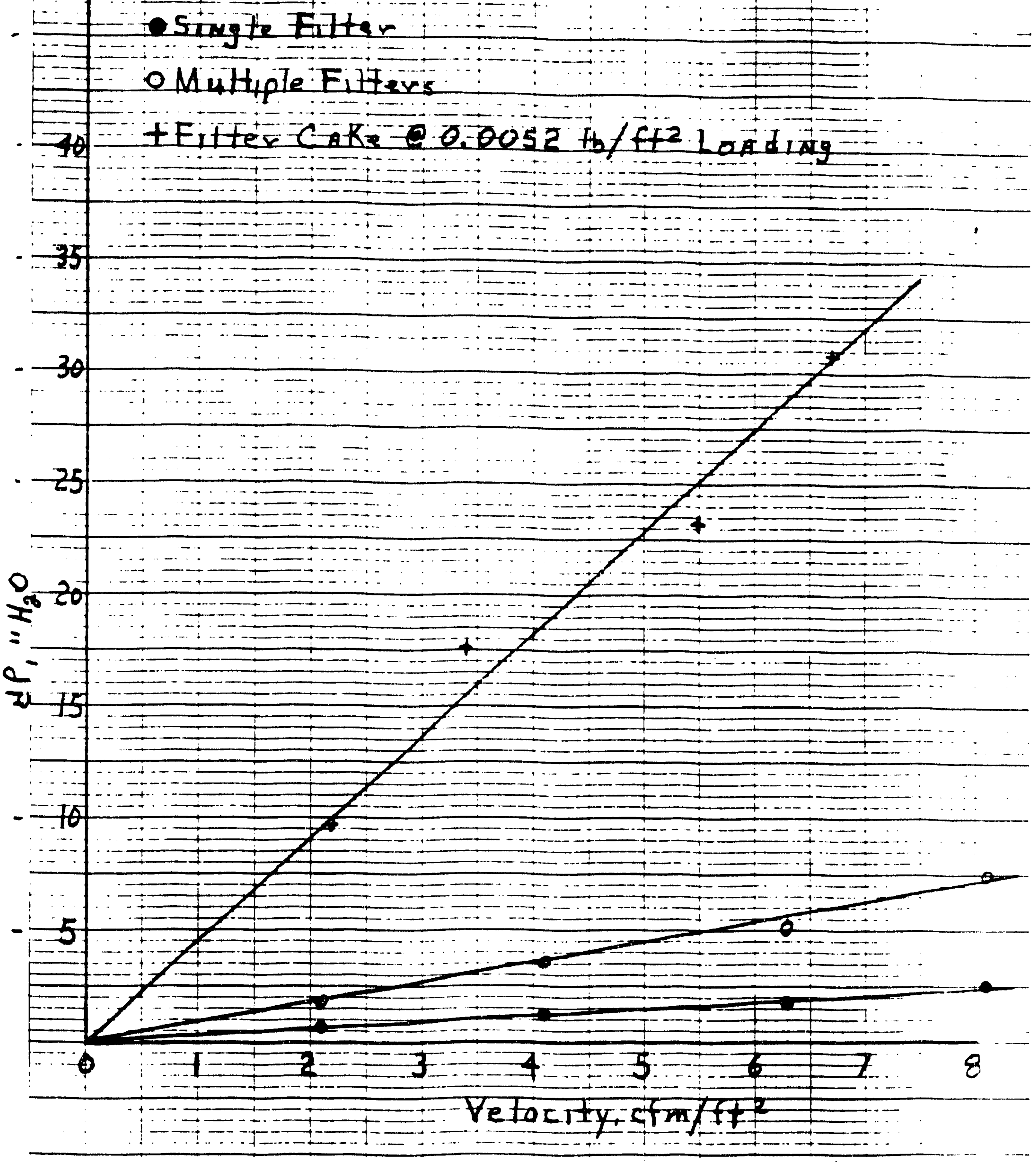


FIGURE 7



46 1610

K&E 5 X 5 TO THE CENTIMETER 18 X 24 CM
KEUFFEL & ESSER CO. MADE IN U.S.A.

Appendix C
Calculation of Radioactivity in Smoke and the Maximum Allowable KTPB Solids in the Filter Cell

Radioactivity in Smoke

The amount of cesium-137 activity released to the air in a tetraphenylborate solids fire can be calculated based on the following:

a) assume 6 wt % of the tetraphenylborate solids are converted to smoke (this is the highest percentage of smoke listed in Table II).
b) assume smoke solids contain 0.25 wt% cesium (see Table V)
c) Cs-137 comprises 38% of total cesium in 15-year aged waste (mole basis, the average atomic weight for cesium in waste is 134.6 amu, and there are 0.1153 grams Cs-137 per curie of Cs-137.*

The number of curies of Cs-137 released to the air by burning 1 lb of solids is given by:

$$453.6 \text{ g solids} \times \frac{.06 \text{ g smoke}}{\text{g solids}} \times \frac{.0025 \text{ g Cs}}{\text{g smoke}} \times \frac{137 \text{ g Cs-137}}{\text{mole Cs-137}}$$

$$\times \frac{1 \text{ mole Cs}}{134.6 \text{ g Cs}} \times \frac{.38 \text{ mole Cs-137}}{\text{mole Cs}} \times \frac{1 \text{ Ci Cs-137}}{.01153 \text{ g Cs-137}} = 2.3 \text{ Ci}$$

This calculation is based on the average waste composition, specifically a mole ratio of K:Cs::150:1. It is expected that this mole ratio will change as the waste in different tanks is processed and as changes occur in canyon operations .

Solids Fire Size Required to Plug Filter Cell Ventilation System

It is possible to estimate the amount of fuel which when burned will plug the Filter Cell ventilation filter system, causing it to rupture. The Filter Cell ventilation contains eight HEPA filter elements (each with 250 ft^2 surface area) and operates at 9500 cfm (4.75 cfm/ft^2 of filter surface area). In Appendix B, the loading calculations (yielding 0.95 lbs per filter) are based on a flow of 4 cfm/ft^2. This loading is corrected to 0.75 lbs/ft^2 for the higher flow in the Filter Cell design.

---

*See D.D. Walker and M. A. Schmitz, "Technical Data Summary: In-Tank Precipitation Processing of Soluble High-Level Waste," DPSTD-84-103, May 1984, Table 2.3.1.

The following conservative assumptions are also made:

1. At the start of the fire, the filters are already loaded to half of their maximum solids loading (0.38 lbs/ft^2).
2. The smoke solids do not distribute evenly over all the filter elements, but are deposited on only half of them (4 filters).
3. All of the smoke particles reach the filters (no credit for settling in the tank).
4. The maximum amount of smoke is generated (6 lbs smoke per 100 lbs KTPB).

The calculation is:

$$4 \text{ filters} \times \left(\frac{.76 \text{ lbs smoke}}{\text{filter}} - \frac{.375 \text{ lbs solids}}{\text{filter}}\right) \times \frac{100 \text{ lbs KTPB}}{6 \text{ lbs smoke}} = 25 \text{ lbs KTPB}$$

The 25 lbs of KTPB solids is approximately 1 cubic foot (based on a density of .4 g/mL, see Table II).

If it is assumed the solids are deposited on a wall similar to the observations in the TNX W2 tank (p. 4) with a thickness of 3 to 10 mm, then the surface area covered would be 30 to 100 ft^2. On a waste tank which is 85 ft in diameter, this deposit would form a band 1.3 to 4.5 inches wide around the circumference of the tank. A continuous deposit of this size is entirely credible based on the observed width of the band in the TNX W2 tank (10 cm wide).

It is also possible to calculate the amount of radioactivity that would get through the HEPA filters before they burst. Two pieces of information are required: (1) the particle size distribution of the smoke, and (2) the collection efficiency of HEPA filters for each particle size. Table C-I lists this information and the calculated amounts of cesium which will get through the filter. If 25 lbs of solids are assuming to burn, there will be:

25 lbs x 2.3 Ci/lb = 57.5 Ci of Cs-137 in the smoke.

From Table C-I, this will release approximately 25 mCi of Cs-137.

TABLE C-I
HEPA Filter Efficiency by Particle Size

Particle Size[1] Size (microns)	Amount (% of total)	HEPA Efficiency[2] (% penetration)	Cesium Released[3] (mCi)
>0.57	44.9	0.00	0.0
0.57-0.44	11.5	0.001	0.07
0.44-0.33	11.4	0.03	1.98
0.33-0.20	19.3	0.07	7.77
0.20-0.00	12.9	0.20	14.84
Total:	100.0		24.66

---

[1] Taken from Run #1, Appendix B.

[2] Taken from C. A. Burchsted, A. B. Fuller, and J. E. Kahn, "Nuclear Air Cleaning Handbook," 2nd edition, ERDA 76-21, 1976.

[3] Calculated from: (HEPA Effic.)x(Amount)x 57.5 Ci

END

DATE
FILMED
6/23/93