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TECHNICAL REPORT ARLCD-TR-77053

DEVELOPMENT OF AN EXPLOSIVE SLURRY MONITOR

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An environmentally acceptable method of disposing of waste explosive material involves wet-grinding the material and pumping the resulting slurry into an incinerator where it is burned. For safety and economy, the homogeneity of the slurry must be maintained at a specified concentration. Therefore, the mixture must be monitored as it flows in a 9-millimeter pipe. Such a monitoring requirement is not satisfied by any commercially available instruments.

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20. Abstract (Continued)

A monitoring system using a bifurcated fiber-optic probe has been developed and tested. Various typical concentrations of several materials gave useable response levels, and a number of abnormal conditions (simulated in the tests) were detected.

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INTRODUCTION

With the advent of the Clean Air Act and the subsequent restrictions on the emission of air pollutants, the disposal of waste energetic materials by open burning had to be discontinued. One alternative that avoids air pollution is a process involving wet grinding of the waste energetic material and pumping the resulting aqueous slurry into an incinerator.

Although the slurry is made up to a nominal concentration, there is no assurance that homogeneity will prevail throughout the pumping process. Variations in homogeneity will result not only in inefficient fuel consumption, but also in an explosive hazard. For these reasons, continuous monitoring was considered necessary.

Although the monitoring of slurries is well established in many industries, it has not been done at the concentration levels envisioned for this process. In addition, instruments that would be hazardous in combination with the energetic material could not be used. Finally, the design of the pumping system imposed the restriction that the instrument must be usable within a pipe having an inside diameter of 0.364 inches or about 9 millimeters. These considerations led to the selection of a bifurcated fiber-optics probe as the basic element of the monitor.

EQUIPMENT

Major components are described below; a diagram of the apparatus is shown in Figure 1, and pilot-plant apparatus, in Figure 2.

Probe

The probe, Model P-1, manufactured by Freeman Laboratories, Inc., uses bifurcated fiber optics. One part transmits light into the slurry while the other part transmits light reflected from the suspended solids to a photoconductive cell. The electronic circuitry of this instrument is simple, affording easy maintenance and trouble-free operation.

Recorder

The model FS01W6D recorder, manufactured by Texas Instruments, Inc. was used to show the response of the probe. This recorder was selected because of its 100-millivolt range, which matches the output

level of the probe, its built-in calibration features, and its "suppressed-zero range" in which the zero value of the measured variable is less than the lower range value so that zero does not appear on the scale. Using these last two features, the span of signals from the probe can be set readily, and the zero suppression can be made accurately.

Pump

The pump, Series 110, is manufactured by the Tat Engineering Corporation of North Bradford, Connecticut. It is a peristaltic type in which the slurry is forced through the line at a rate of 4.5 gallons (17 liters) per minute by periodic compression of the tubing. The tip of the probe is inserted in the discharge side of the line.

Mixer

A Benco Model 4R, manufactured by Bench Scale Equipment of Dayton, Ohio, is used. This consists of a glass container with a capacity of 26 liters, baffles, a stirrer with a speed controllable from 90 to 5000 rpm, and mixing blades of either turbine or propeller type.

Test Materials

Type	Color	Particle size (through mesh no.)
TNT, finely divided	Tan	20
Comp B, finely divided	Tan	20
Comp B, granular	Tan	8
RDX, finely divided	White	20
Nitrocellulose fines	White	None-Fibrous material
Propellant A-7	White	8
Propellant M-1, granular	Yellow-green	8
Propellant M-9, flake	Graphite coated	None - Nominal length 0.007 in. (0.18 mm)
Propellant M-10	Yellow	8
Ion-exchange resin	White	200

EXPERIMENTAL PROCEDURES

The following experimental procedures were used to test the effectiveness of the bifurcated fiber-optics probe.

Zero Adjustment

1. In the mixing vessel, make a slurry consisting of 15% energetic material and 85% water (by weight).

2. Maintain homogeneity of the slurry by adjusting the mixer speed and baffles.

3. Circulate the slurry by turning on the pump.

4. Using the zero and span controls on the fiber-optics control box, adjust the output on the recorder to the low level (toward zero). This establishes the baseline for the 15% concentration of energetic material.

Range Adjustment

After the base line is set for the initial (15%) concentration, add additional solid material to produce a slurry of 30% concentration. Adjust the zero suppression and recorder span controls to give the maximum value to be observed on the recorder. These settings will serve to fix the response of the optic probe so that all readings are within the range of the recorder.

Calibration

After obtaining the reading for a 15% slurry, transfer into the mixing vessel weighed amounts of energetic material to increase the concentrations to 20%, 25%, and 30% in order to establish a four-point calibration curve for this material.

Testing for Upset Conditions

After the calibration curve has been established and the 30% slurry is in the system, upset the homogeneity of the slurry by decreasing the mixer speed to induce settling. Raise and lower the suction end of the pump line to observe the output when probing different concentration

levels. If settling, or layering, does not readily occur due to the nature of the material under test (for example, finely divided Composition B), pour 50 to 100 milliliters of clear water near the suction end of the pipe line thereby allowing the line to become filled with diluted slurry. Then return the suction line into the highest concentration and observe the difference in response. This will give the range of reflectivity to be expected for the specific material.

Application

For a sample of RDX (lot No. HOL 21-22) containing 24.09% water, the probe was set for 50 millivolts and the recorder span was set at 5 millivolts full-scale. Water, in the amount of 9,112 grams, was poured into the container and 2,238 grams of the wet RDX was added to make the initial concentration of 15%. To attain the 20% concentration, an additional 1,012 grams of RDX was added; 1,189 grams, for the 25% level; and 1,462 grams, to make the final concentration of 30%. The response of the probe to changes in RDX concentration is shown in Figure 3.

From the calibration test it was established that a 20 millivolt span, full-scale, was required for the recorder, and a zero suppression of 26.42 millivolts was required to bring the output for this RDX into a zone that could be recorded.

In this test series, when the mixer speed was slowed to 400 rpm, sedimentation layers occurred and the recorded reading dropped from 9.3 inches (23.6 cm) to 4.5 inches (11.4 cm). When a 'slug' of solids was drawn into the line, a high off-scale reading was obtained. When clear water was drawn into the line, a low off-scale reading was obtained.

DISCUSSION OF RESULTS

Table 1 shows the response in millivolts to concentration changes for nine different materials. In general, there was an increase in probe output for concentration increase. However, the amount of change was not always of sufficient magnitude to give adequate sensitivity. The response of the probe to changes in concentration of energetic slurries is directly related to the particle size of the suspended matter; that is, sensitivity increases with increased particle size. Figure 4 shows the results of an idealized experiment using ion-exchange resins (Rohm and Haas Amberlite spheres).

The results in Table 1 include data obtained using white light only and white light with a red filter. This was done to see if the filter would increase the differential response, that is, the amount of change resulting from incremental changes in concentration. The results show that the differential increase did not occur. However, the use of the filter did greatly reduce the response, particularly with the Composition B. With white light, the response was at the high millivolt output level. With the red B-650 filter the response moved to the very lowest millivolt level. The filter did not change the span (Δ millivolts) but did affect the operational level.

The data presented in Table 2 compares the responses of the white light as opposed to the filtered light. The results indicate that there was not enough change to warrant using a filter.

In Table 3 the results show that for finely divided Composition B a change in concentration from 15% to 20% produces less than a millivolt change in probe response. However, with the granular Composition B, the probe response was much higher (Fig 5). With all concentration changes, the coarse material gave not only a higher response but also adequate sensitivity for all the concentrations of interest. See Figures 6 and 7.

The finely divided material gave a response that was only adequate at the lower concentration levels. Another instance, as reported in Table 1, is that of nitrocellulose . In the very low concentrations, going from 3% to 5%, a change of 5.5 millivolts was obtained while from 7% to 9% the change was about half of that. Due to the nature of the material and the physical size of the vessel, it was impractical to try to obtain results at higher concentrations.

The sensitivity of the probe to changes in the color of energetic slurries is directly related to the brightness (whiteness) of the color; that is, sensitivity will increase as brightness increases. A suitable example of this is shown in Table 1; RDX, a white crystalline compound, gave a change of about 20 millivolts over all the operational ranges. With the propellant M-9, a graphite-coated, flaked material, a change of about 5 millivolts was recorded over the entire concentration range.

In Table 4 are shown the results of placing the probe in two different locations, at 12 o'clock and at 6 o'clock, to determine if the response would be uniform regardless of the angle that the probe was presented to the flowing slurry. Here again, Composition B, in both the fines and granules, was used as a test medium. The results (Fig 8), indicate that with both granular and fines the response from the 6 o'clock position may be more uniform.

In Table 5, a comparison was made of the effect of the amount of light allowed to pass from the source through the transmitting bundle of fibers to be reflected by the particles in the slurry. This was accomplished by blocking out half the transmitting bundle. The instrument uses a fluorescent light source without controls to vary light intensity. The results in Table 5 show that when light is attenuated the corresponding results are also attenuated, but, interestingly, the responses to concentration changes remain fairly constant. These results are plotted in Figure 9. The information in Table 6 represents an effort to determine if the particle size changes during pumping. The particle size measurements were made by methods 204.1 and 204.2 in MIL-STD-650, depending on the material. The results indicate that particles do decrease in size as they are pumped.

The data presented in Table 7 and Figure 10 show the minimum and maximum responses obtained for all energetic materials to date, providing an overall view of all the results and showing which materials have the best response.

Nitrocellulose yields more than a 16-millivolt change for concentrations of 9% to 10%. The best response in the operational ranges of most interest is probably from RDX with a 22.4-millivolt change. A number of materials produced a change of approximately 5 millivolts for the operational ranges: M-9, M-1, Composition B fines, and TNT fines with filter. Granular Composition B, with a change of 21 millivolts over the operational ranges, is a very suitable material to monitor. As a quick guide, Table 7 and Figure 10 show windows of reflectivity for the materials tested.

To ascertain the effect of particle size and concentration on turbidimetric and/or nephelometric measurements, experimentation was conducted with a Brinkman Colorimeter, model PC1000W, in conjunction with a Brinkmen Chemputer 3 using glass beads of known size. See Figure 11.

A solution of 50% water and 50% glycerine was used in order to improve the suspendibility of the glass beads. Known weights of glass beads were added to the glycerine and water mixture to increase the concentration in 2.5% steps over the range from zero to 20%.

In a typical test, 475 milliliters of the 50/50 glycerine and water solution was put into an Erlenmeyer Flask, the optic probe was inserted into the solution, and the resulting reading was set to indicate 100% transmittance. Then glass beads were added to the flask to increase the concentration in 2.5% increments. A magnetic stirrer was employed to keep the particles in suspension. Test data and results are shown in Table 8 and Figure 8. The results confirmed those obtained in testing energetics which indicated that small particle size causes the probe to be insensitive at higher concentrations. Increasing particle size will enhance the operation of the fiber optic probe and allow greater concentrations to be monitored. Further studies will be required to minimize the effect of particle size.

CONCLUSIONS

The sensitivity of the optic probe to changes in concentration of energetic slurries is directly related to the particle size of the suspended matter, that is, sensitivity increases with increased particle size and vice versa (prolonged pumping action reduces particle size as shown by a decrease in probe output). Also the sensitivity of the optic probe to changes in the color of energetic slurries is directly related to the brightness of the color, that is, sensitivity increases as brightness (whiteness) increases.

The best orientation for the probe is at the 6 o'clock position. In this position the effect of bubbles and foam is minimized.

The probe was sufficiently sensitive to respond to the changes in concentration of energetic material in an aqueous slurry and to give a reliable, useful response to upset conditions.

RECOMMENDATIONS

Further work should be performed to improve the response to changes in concentration of materials of small particle size, to find the minimum particle size to which the probe shows an acceptable response over the concentration range of interest, and to determine the effect of the intensity and wavelength of the light source on response.

Table 1

Effect of concentration changes on probe response*

	Concentration (percent solids)	Recorded response (millivolts) Run A	Run B	Run C
Finely divided TNT	15	47.30	46.75	41.88
Lot: KNK 3096	20	49.65	49.50	44.31
Test equip: Lab	25	50.93	50.95	45.76
	30	51.36	51.20	46.16
	30	50.76	50.20	45.06
	> 30	51.86	51.45	46.56
Lot: Rad-6 0804	15	88.91	88.56	86.94
Test equip: Lab	20	91.16	90.26	89.59
Light: Red, B-650	25	91.96	91.21	89.59
	30	91.96	91.36	91.29
	0	88.56	89.56	87.74
	> 30	93.91	91.26	91.24
Lot: KNK 2-3096	15	76.48	74.36	78.04
Test equip: Pilot	20	78.98	80.96	80.94
	25	79.98	83.36	81.84
	30	80.38	83.96	82.74
	30	81.48	83.09	82.74
	30	78.38	82.56	82.54
	> 30	82.98	90.66	89.14
Comp B fines	10	_	92.25	96.10
Lot: Hol 050-5414	15	92.60	92.60	96.90
Test equip: Lab	20	92.85	92.95	97.45
	25	93.10	93.20	97.30
	30	93.45	93.20	97.70
	> 30	93.60	93.20	97.20

*Unless indicated otherwise, white light was used.

Table 1 (cont)

	Concentration (percent solids)	Recorded response (millivolts) Run A	Run B	Run C
Comp B fines	10.8	-	1.73	2.83
Lot: Hol 050-5414	15	11.25	2.01	2.99
Test equip: Lab	20	11.29	2.13	3.08
Light: Red, B-650	25	11.27	2.07	3.05
	30	-	2.03	3.02
Lot: Hol 35-9085	15	69.82	69.38	67.25
Test equip: Pilot	20	72.50	68.68	66.90
	25	71.50	67.75	66.20
	30	70.04	67.10	65.25
Lot: Hol 35-9085	5	60.70	59.70	67.90
Test equip: Pilot	10	66.00	64.70	73.30
	15	66.90	65.50	74.20
	20	67.00	65.40	74.10
	> 20	67.70	65.90	74.40
Comp B granular	15	38.20	33.00	37.20
Lot: Hol 20-2004	20	44.40	38.00	43.60
Test equip: Pilot	25	50.40	42.50	49.20
	30	54.00	46.00	53.60
	> 30	56.00	47.00	56.00
RDX	15	27.22	21.80	21.90
Lot: Hol 21-22	20	33.62	29.60	29.30
Test equip: Lab	25	37.72	35.60	35.00
	30	42.57	44.20	40.00
	> 30	42.77	46.10	40.00
Nitrocellulose	3	8.44	4.72	7.85
Test equip: Lab	5	13.94	9.52	13.05
	7	17.94	12.92	17.25
	9	21.14	15.52	19.45

Table 1 (cont)

	Concentration (percent solids)	Recorded response (millivolts) Run A	Run B	Run C
Propellant A7	15	38.90	44.30	42.41
Test equip: Lab	20	38.95	45.10	42.61
	25	40.95	46.10	44.41
	30	44.45	49.50	47.71
	<15->30	53.00	52.40	50.70
Propellant M1	15	23.70	21.80	22.90
Lot: Rad 68869	20	26.00	24.40	24.60
Test equip: Lab	25	25.80	26.00	25.80
	30	25.70	26.80	26.40
	<15-> 30	24.00	27.70	25.90
Propellant M9	15	7.00	7.65	6.60
Test equip: Lab	20	9.40	8.95	8.20
	25	10.7	10.75	9.90
	30	12.0	11.95	11.20
	<15-> 30	12.6	12.55	9.40
Propellant M10	15	6.28	5.40	5.89
Test equip: Lab	20	7.38	6.20	6.94
	25	8.18	7.00	7.59
	30	8.38	7.55	7.94
	<15->30	8.38	5.00	10.34

Table 2

Effect of light source on probe response

Material	Concentration (percent solids)	Change in response (millivolts)					
		White Light			Red Light		
		Run A	Run B	Run C	Run A	Run B	Run C
TNT fines	15-20	2.35	2.85	2.43	2.25	1.70	2.65
	20-25	1.28	1.45	1.45	0.80	0.95	1.30
	25-30	0.43	0.25	0.40	0.00	0.15	0.40
	<15->30	4.56	4.70	4.68	2.70	2.70	4.30
Comp B fines	10.8-15	-	0.35	0.80	-	0.28	0.16
	15-20	0.25	0.35	0.55	0.04	0.12	0.09
	20-25	0.25	0.25	0.55	-0.02	-0.06	-0.03
	25-30	0.35	0.00	0.30	0.00	-0.04	-0.03
	<15->30	1.00	0.95	1.10	-	-	-

Table 3

Effect of particle size on probe response

Material	Concentration (percent solids)	Change in response (millivolts)					
		Fine (20 mesh)			Coarse (8 mesh)		
		Run A	Run B	Run C	Run A	Run B	Run C
Comp B	15-20	0.25	0.35	0.55	6.20	5.00	6.40
	20-25	0.25	0.25	0.55	6.00	4.50	5.60
	25-30	0.35	0.00	0.30	3.60	3.50	4.40
	<15->30	1.00	0.95	1.10	17.80	14.00	18.80

Table 4

Effect of probe orientation on probe response

Material	Concentration (percent solids)	Change in response (millivolts)			
		Probe at 12 o'clock		Probe at 6 o'clock	
		Run A	Run B	Run A	Run B
Comp B granular	15-20	10.22	5.00	6.40	6.20
	20-25	11.12	4.50	5.60	6.00
	25-30	5.86	3.50	4.40	3.60
	>30	16.20	14.00	19.80	17.80
Comp B fines	5-10	4.40		5.00	
	10-15	0.45		0.80	
	15-20	-0.25		-0.10	
	>20	4.90		6.20	

Table 5

Effect of light intensity on probe response

Material	Concentration (percent solids)	Light transmitted (percent) 100 Meter reading	100 Change (millivolts)	50 Meter reading	50 Change (millivolts)
Comp B fines	5	67.90	-	32.70	-
	10	73.30	5.40	36.30	3.60
	15	74.20	0.90	37.20	0.90
	20	74.10	0.10	37.50	0.30
	>20 (overall)	74.40	6.50	36.70	4.00

Table 6

Effect of pumping action on particle size

	As received		Pumped			
			Run A		Run B	
Sieve	(% on)	(% thru)	(% on)	(% thru)	(% on)	(% thru)
			Composition B, Granular			
60	98.3	1.7	93.6	6.4	94.5	5.5
80	0.2	1.5	1.6	4.8	1.5	4.0
325	1.4	0.1	4.7	0.1	3.9	0.1
on pan	0.1	-	0.1	-	0.1	-
			Composition B, Fines			
60	1.2	98.8	1.0	99.0	1.0	99.0
80	2.0	96.8	1.6	97.4	0.6	98.4
325	1.4	95.4	1.0	96.4	2.8	95.6
on pan	45.4	-	96.4	-	95.6	-
			Composition A-7			
60	37.5	62.5	4.0	59.0		
80	45.0	17.5	33.5	25.5		
325	17.3	0.2	0.5	-		
on pan	0.2	-	0.5	-		
			TNT Fines			
60	61.5	38.5	72.8	27.2		
80	31.9	5.6	23.7	3.5		
325	5.5	0.1	3.4	0.1		
on pan	0.1	-	0.1	-		
			M-1 Propellant			
60	97.4	2.6	94.5	4.5		
80	1.5	1.1	2.0	2.5		
325	0.9	0.2	2.3	0.2		
on pan	0.2	-	0.2	-		

Table 7

Reflectivity achieved on tested energetics

Material	Response (mv)* Minimum	Maximum
Comp B, fine	92.25	97.70
Comp B, fine (red filter)	1.73	11.29
Comp B, granular	33.00	54.00
Trinitrotoluene, fine	41.88	51.36
Trinitrotoluene, fine (red filter)	86.94	91.96
RDX	21.80	44.20
Nitrocellulose, fine	38.90	49.50
Propellant A-7	21.80	26.80
Propellant M-1	6.60	12.00
Propellant M-10	5.40	8.38

*Minimum response is lowest reading obtained with a concentration; maximum, the greatest reading obtained for the largest known concentration.

Table 8

Effect of particle size on transmittance

Concentration (percent solids)	Particle size (microns) 13-44 Meter	13-44 Trans (%)	45-62 Meter	45-62 Trans (%)	88-125 Meter	88-125 Trans (%)
0	.240	100	.241	100	.240	100
2.5	.073	30	.080	33	.117	48
5.0	.071	29.5	.074	30.5	.091	39
7.5	.072	29.5	.074	30.5	.083	35
10.0	.072	29.5	.074	30.5	.080	33
					.079	32
					.079	33

Concentration (percent solids)	Particle size (microns) 126-177 Meter	126-177 Trans (%)	178-250 Meter	178-250 Trans (%)	251-300 Meter	251-300 Trans (%)
	.240	100	.240	100	.240	100
2.5	.145	48	.127	54	.155	65
5.0	.100	37	.105	44	.133	55
7.5	.089	35	.092	38	.112	46
10.0	.082	33	.086	35	.100	42
12.5	.080	32	.082	33.5	.092	38
15.0	.078	33	.080	33.0	.087	36
17.5			.078	32.0	.083	35
20.0					.080	33
22.5					.079	32

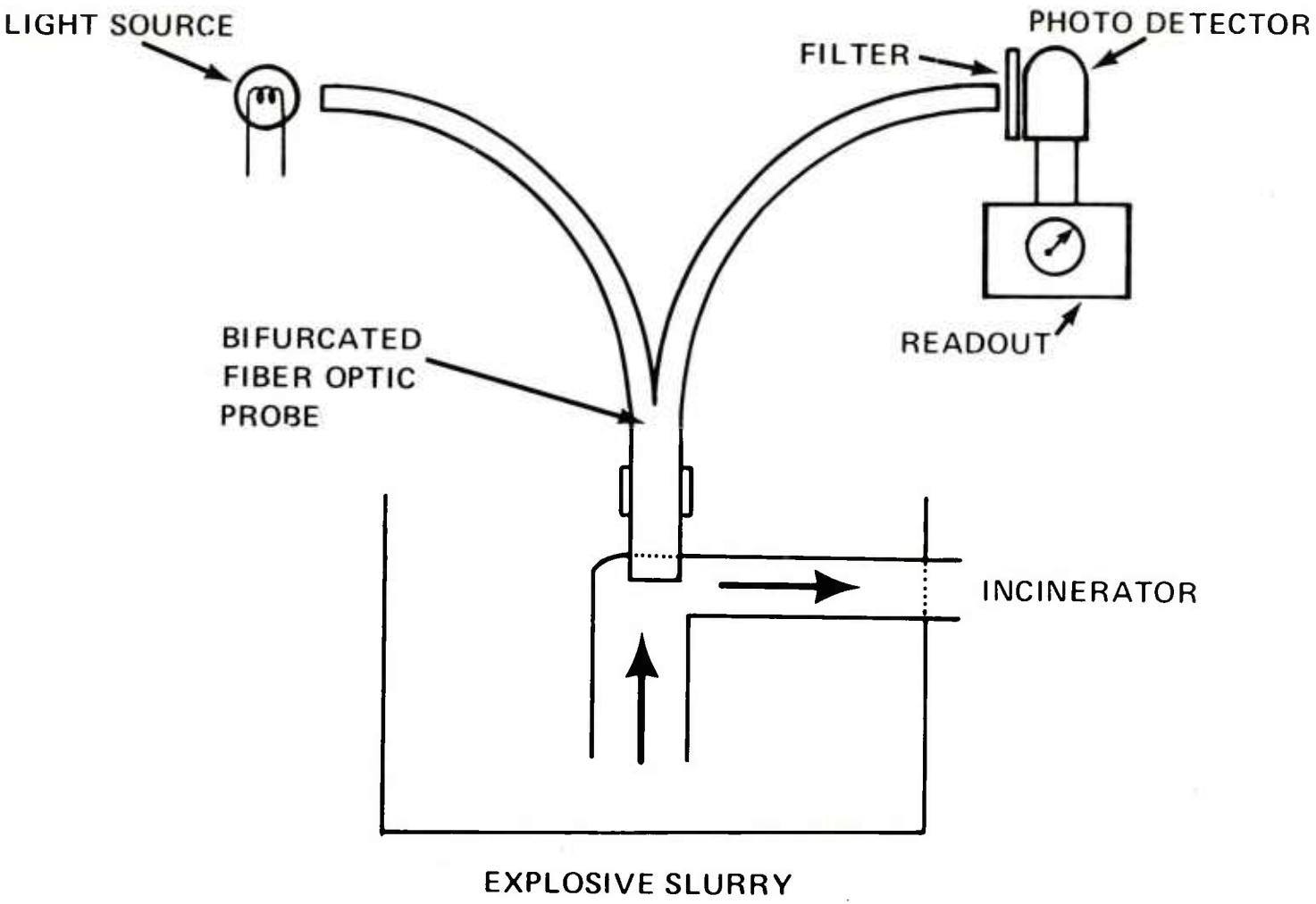

Fig 1 Explosive slurry monitor

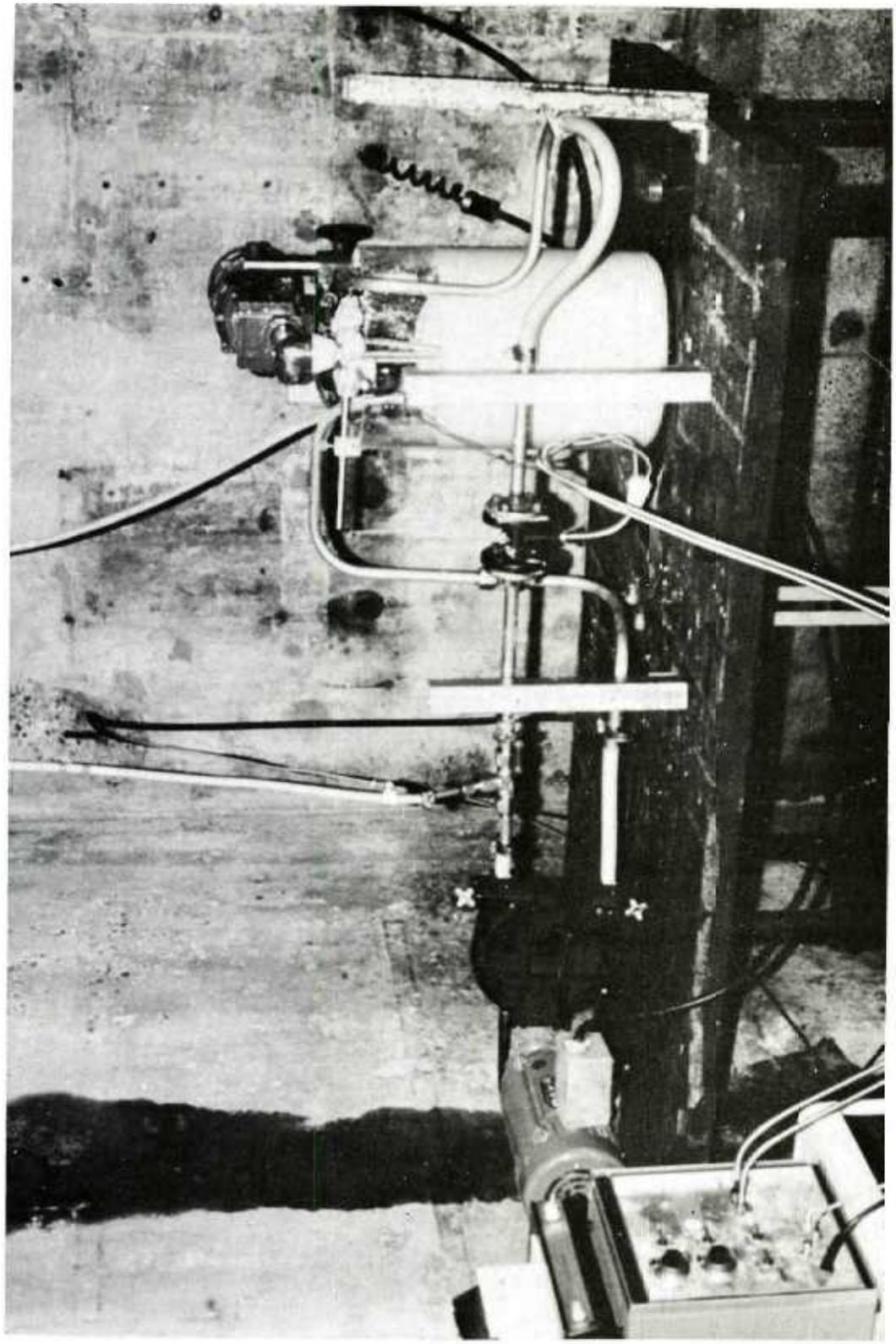

Fig 2 Pilot-plant apparatus

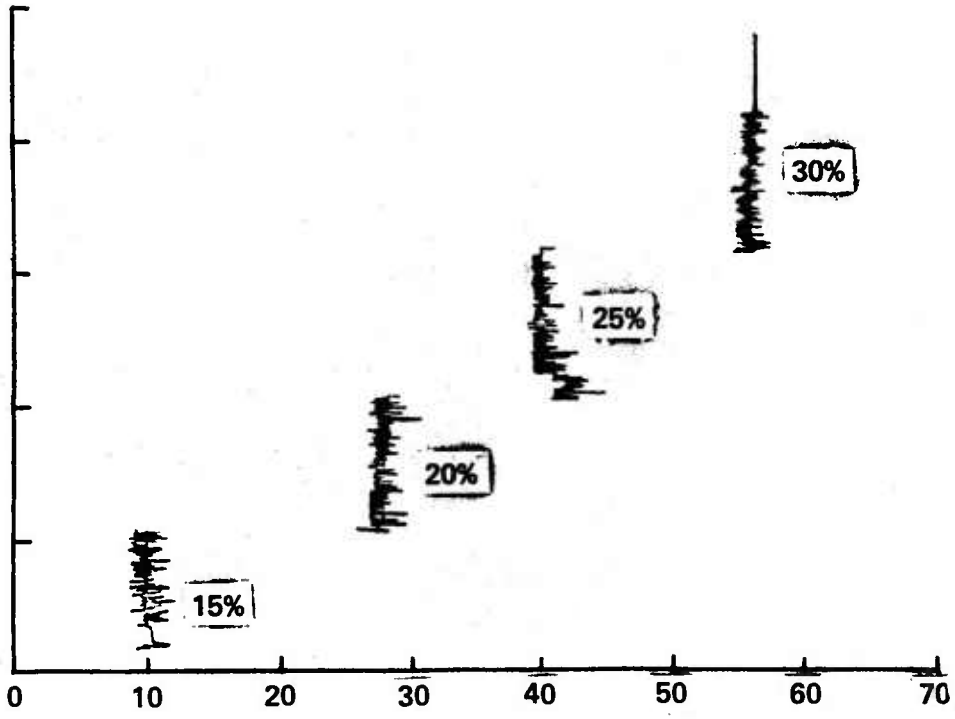

Fig 3 Optic probe monitoring of RDX

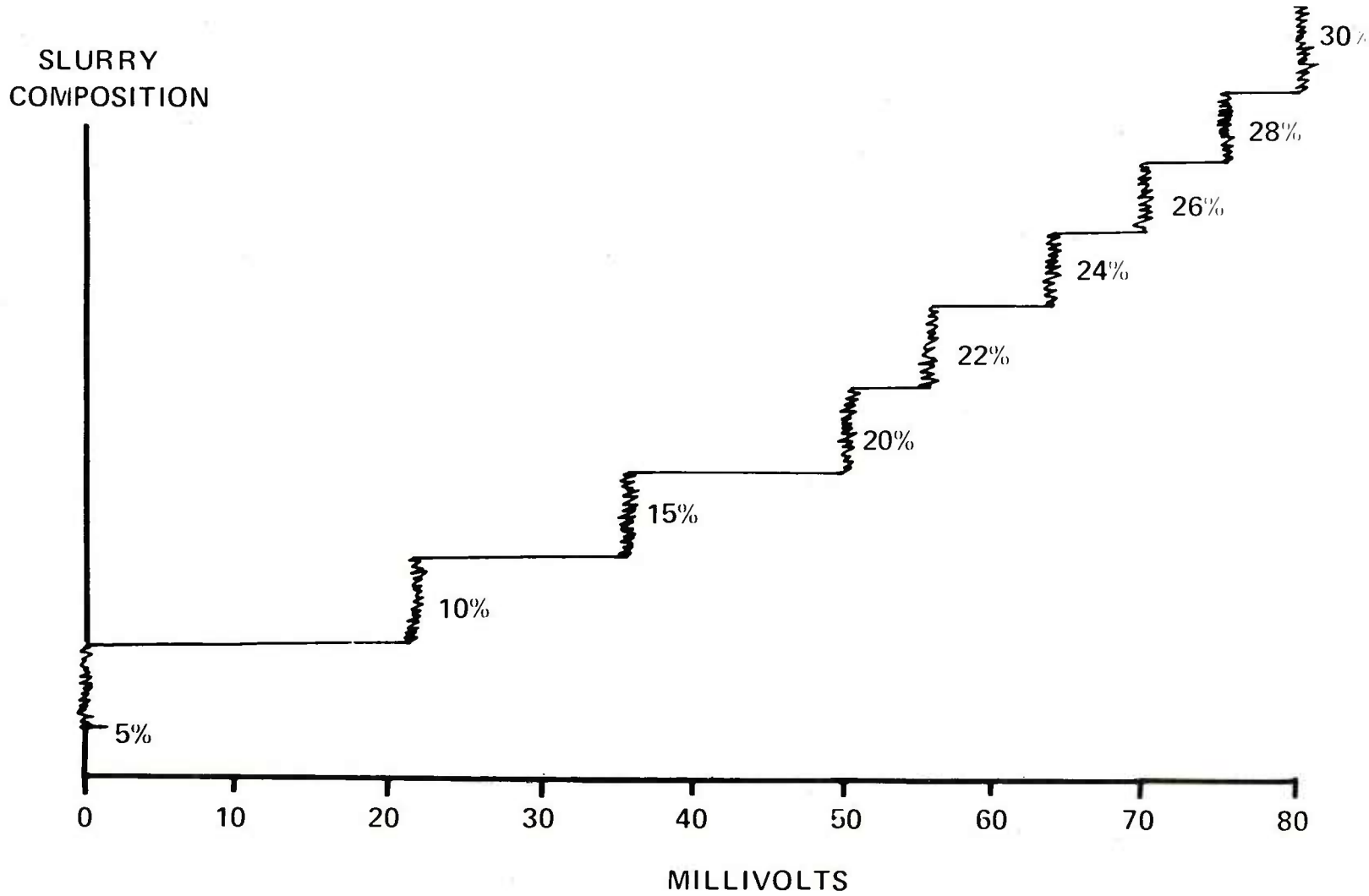

Fig 4 Optimal response curve for fiber optic monitor

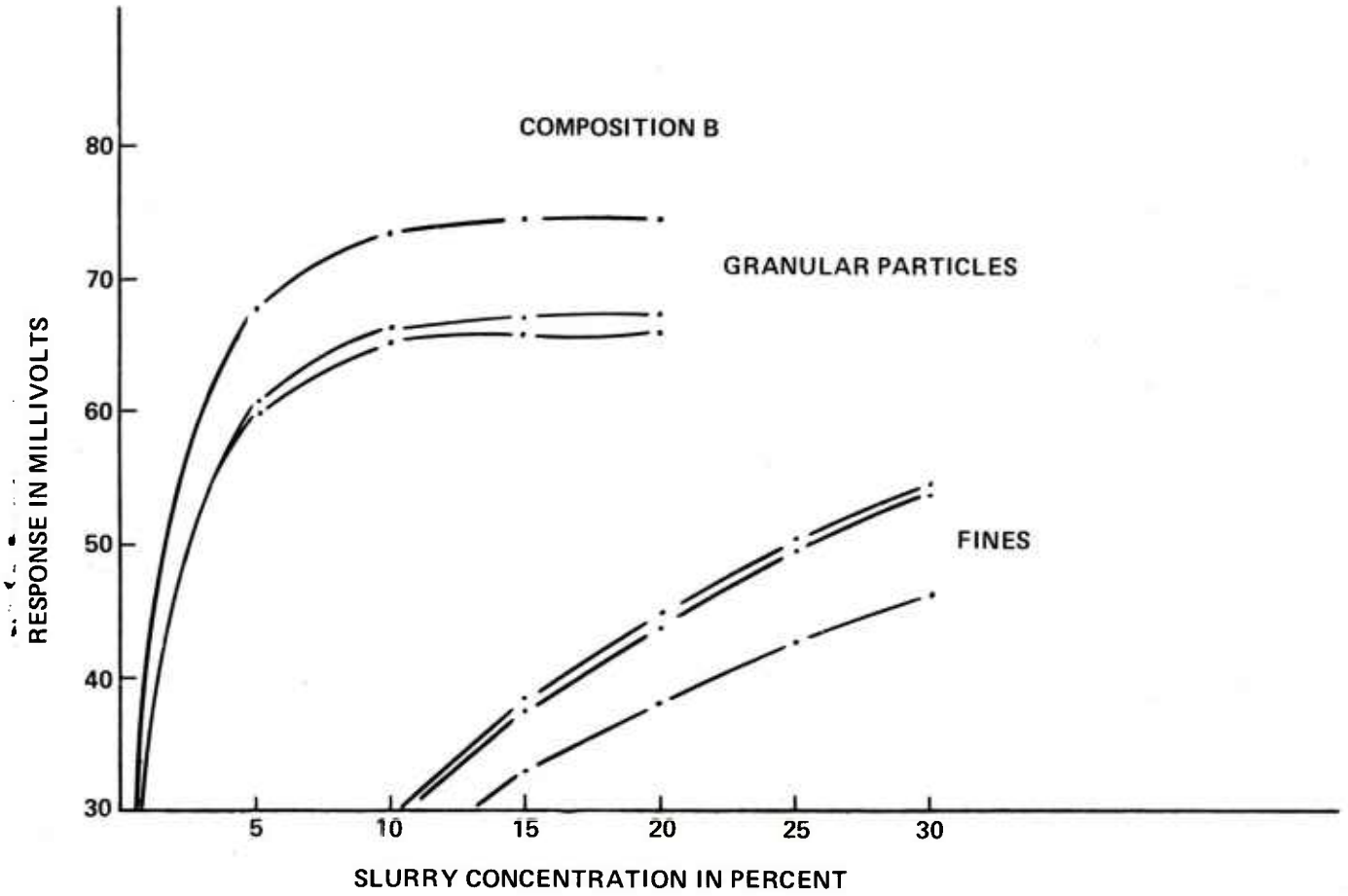

Fig 5 Effect of particle size on Comp B

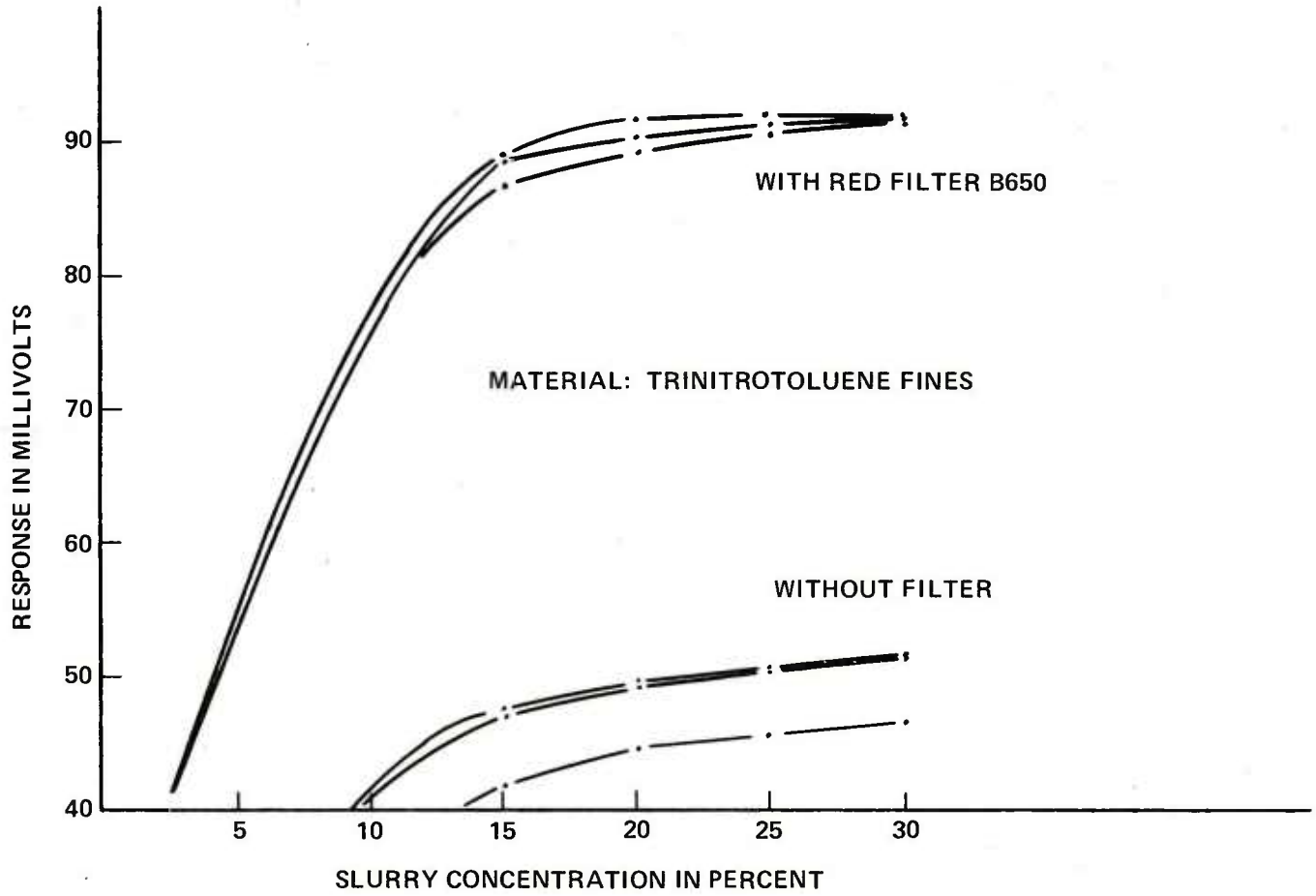

Fig 6 Effect of filtered light on TNT fines

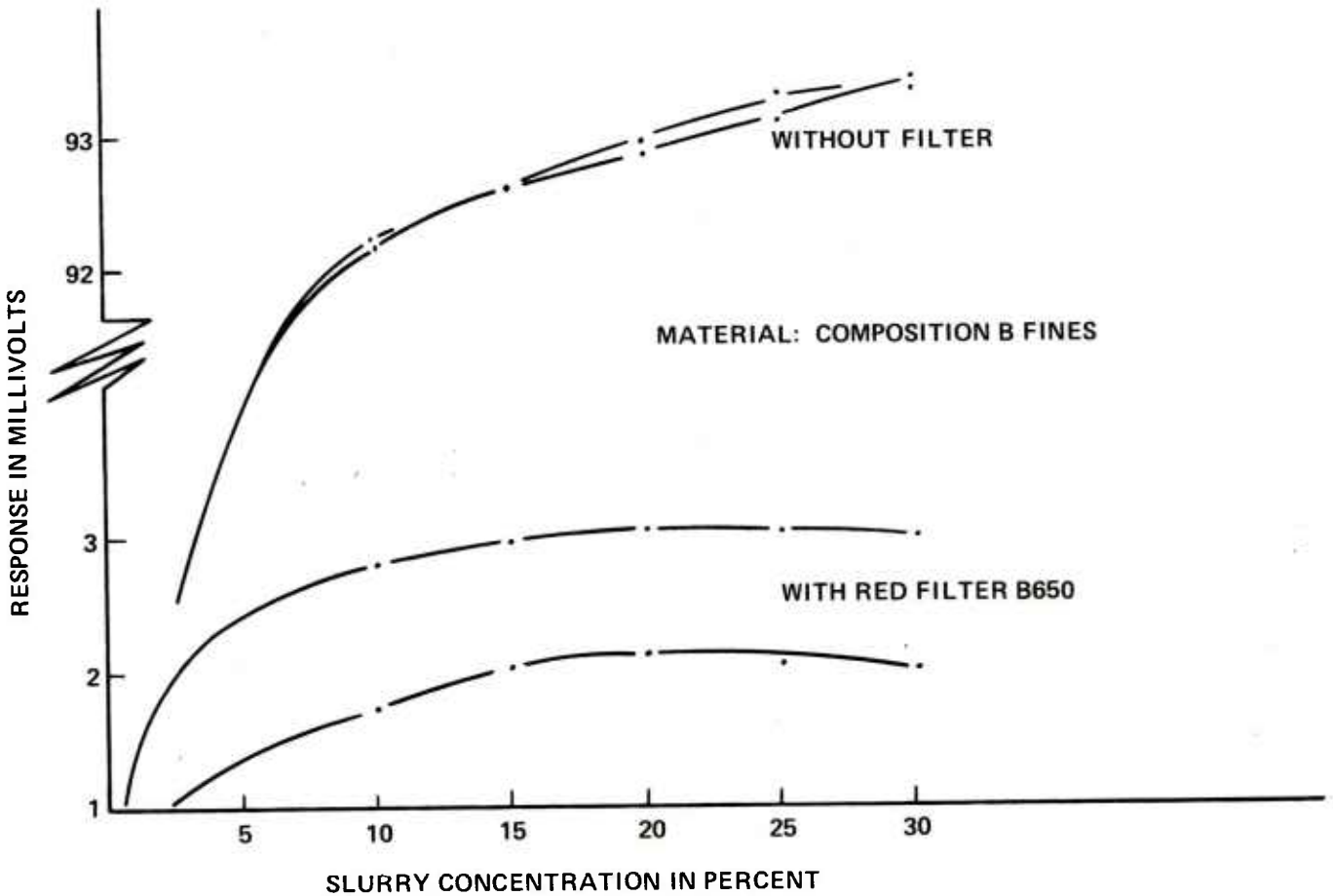

Fig 7 Effect of filtered light on Comp B fines

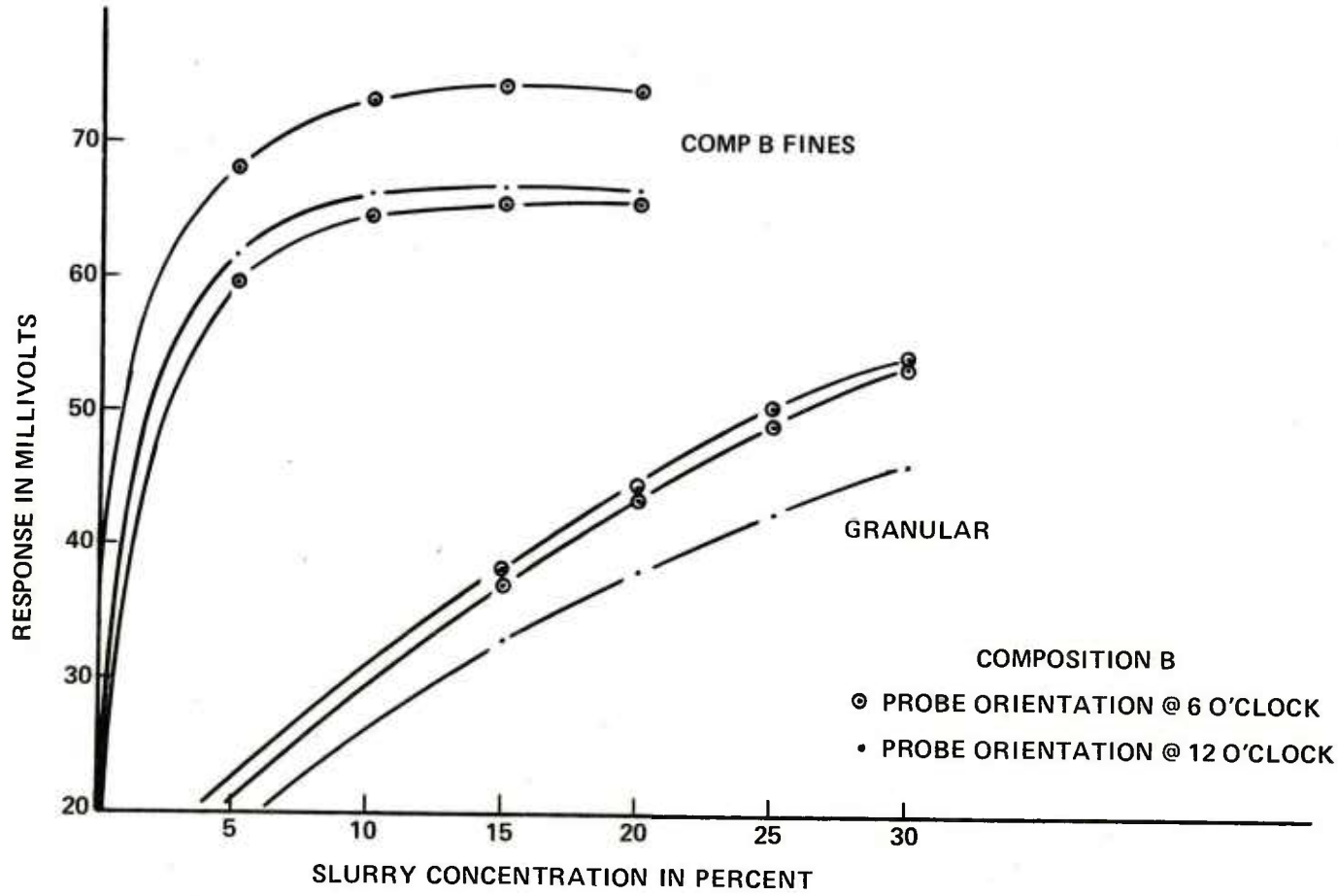

Fig 8 Effect of particle size on probe output

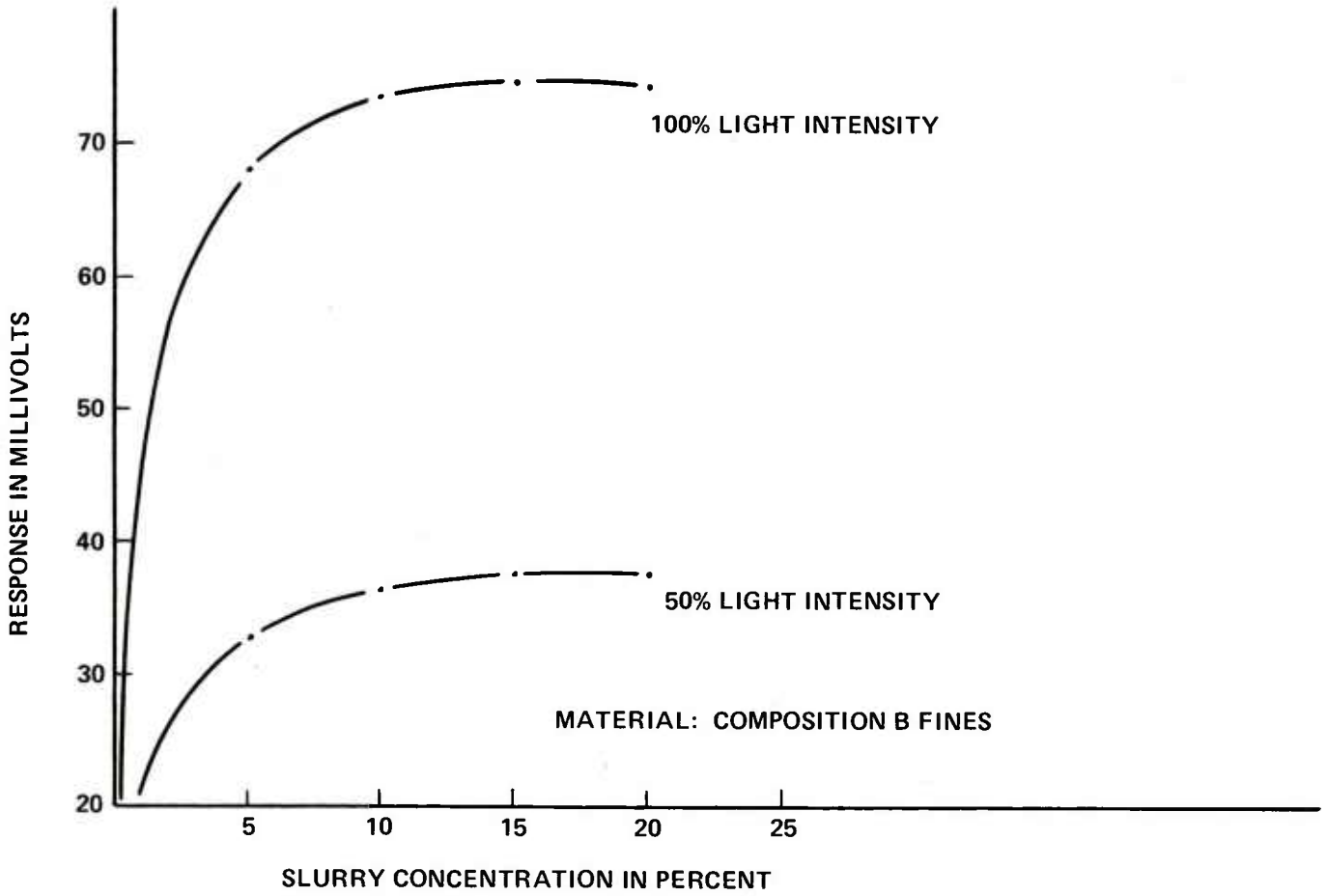

Fig 9 Effect of light intensity on probe response

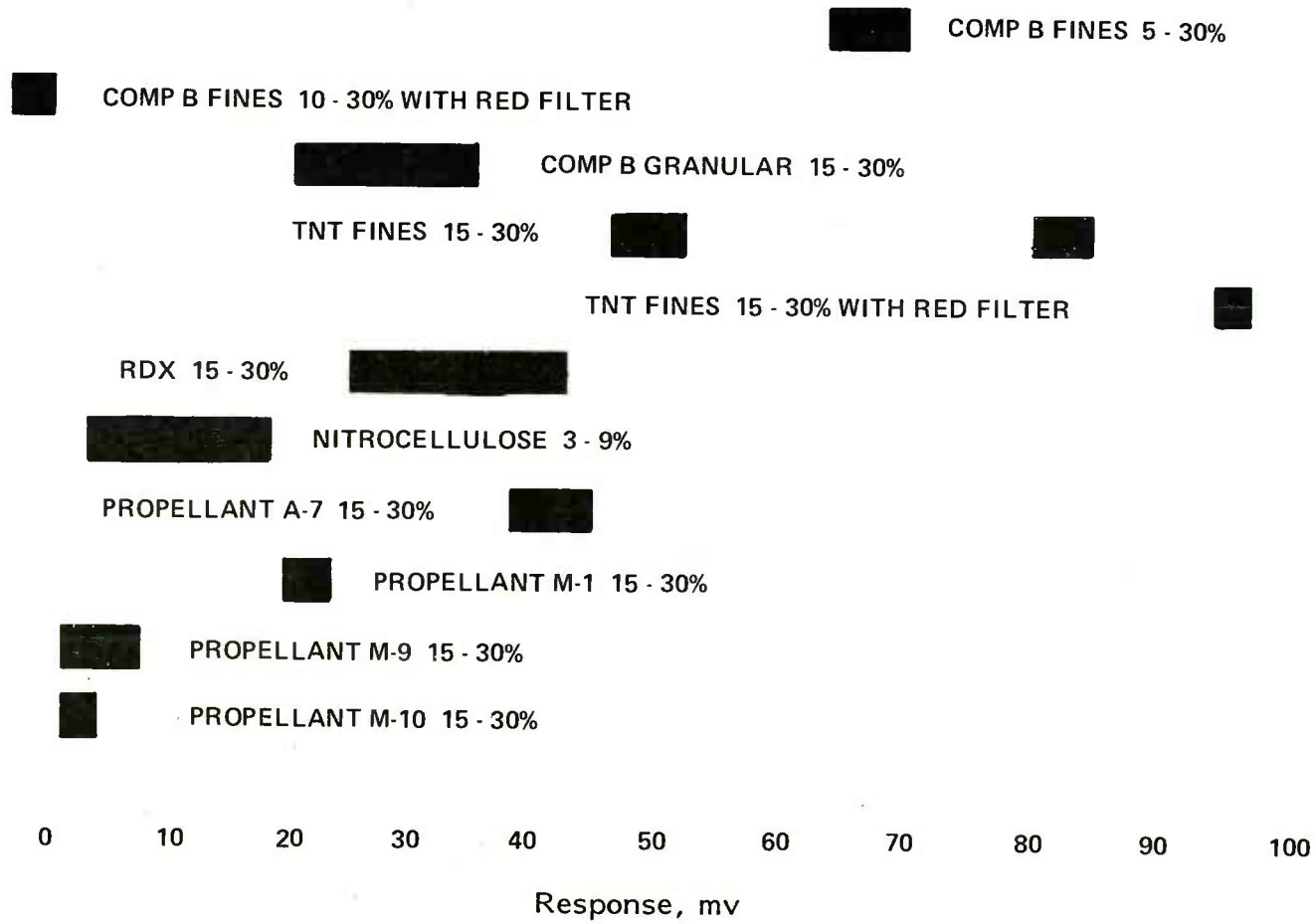

Fig 10 Window of reflectivity of tested energetics

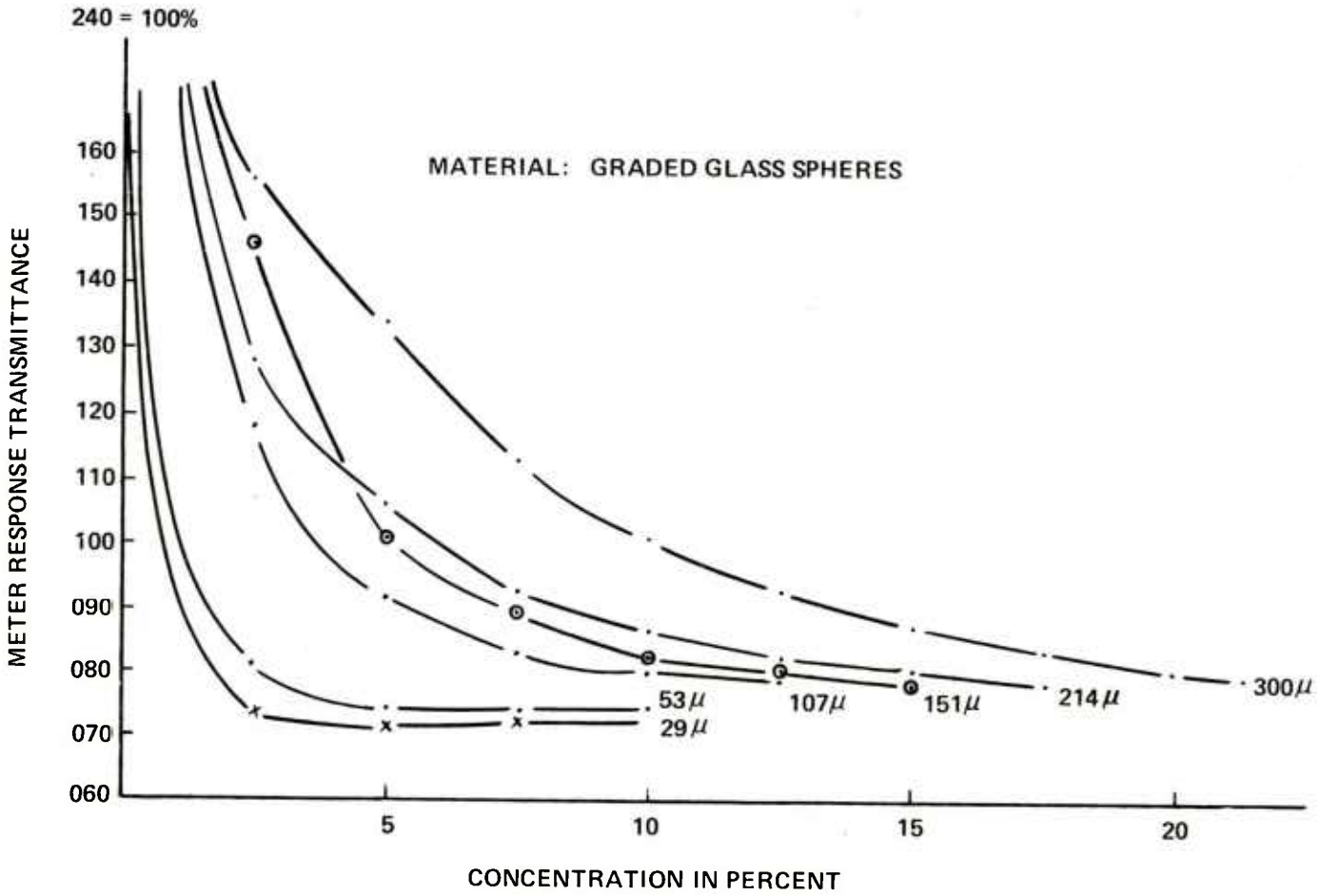

Fig 11 Effect of particle size change on transmittance

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