

ARMY RESEARCH LABORATORY



Liquid Explosive in Pipes

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A simple effective means of testing a liquid explosive in the field was conceived and demonstrated. The central idea was to contain the liquid explosive in plastic pipe and to utilize high speed photography to examine detonation characteristics. Because plastic pipe is a weak confinement, it does not promote detonation of liquid explosive. All the liquid explosives tested were amine-sensitized nitromethane mixtures. Photo diagnostics of the detonations were enhanced by double exposure of the film. Six grams of explosive will reliably initiate 5% mixtures and detonation will propagate through at least a 16-mm passage. This technique proves which mixtures are detonable and bounds their detonation characteristics, such as initiation requirement, critical diameter, velocity and steadiness, and sustainable length of propagation.					
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1. INTRODUCTION

- 1.1 <u>Objective.</u> To use a new means to observe the detonation of sensitized nitromethane.
- 1.2 <u>Central Idea.</u> A good field test of any liquid explosive is filling a long plastic pipe with it and filming the detonation. This means points to the explosive's initiation requirement, detonability, velocity, and critical diameter.
- 1.3 <u>Background.</u> Nitromethane was synthesized in 1872 by Kolbe, but it is so insensitive that not until 1938 did McKittrick, Irvine, and Bergsteinsson report that it could detonate. The liquid detonated in hot confinement or by blasting cap initiation if the liquid was strongly encased. World War II research produced sensitizers (mainly amines) that made nitromethane in weak confinement (e.g., rubber hose, glass test tube, phenolic tube) detonable with a blasting cap. Ericksen and Rowen (1945) listed more than a dozen nitromethane-amine mixtures and their relative explosive power. Audrieth, Ericksen, and Tomlinson (1967) patented these same mixtures; more mixtures were found by Laurence (1966). Mechanical sensitization by inclusion of microbeads of air is also known (Minnick 1967). The present work on liquid explosive concentrated on test development and following in the past vein, used chemically sensitized nitromethane, made more reliably detonable with small (6 g) initiators. In distinction, some large field tests have broken from past research and have used neat or sandy gelled nitromethane initiated by a large (~ 1 kg) booster charge (MIDNIGHT HOUR 1 (1992), ESSEX 1974).

Incidental use of nitromethane in pipes was made earlier. Typically, a center pipe containing gas was collapsed by an exploding jacket of strongly confined, unsensitized nitromethane. In one application, the shocked gas in the center pipe launched a projectile to hypervelocity (Watson et al. 1967; review by Walker 1982). Another collapsing tube apparatus for the explosive driver of a one-shot shock tube used weakly confined, sensitized nitromethane (Bertrand 1967). The present tests may be the first to employ plastic pipe for systematically studying liquid explosive detonation.

2. EXPERIMENT

2.1 <u>Method.</u> The authors' method of testing liquid explosives was initiating mixtures from one end of long plastic plumbing pipe and filming the light from the traveling detonation. Plastic pipe, by being a weak confinement, is a stiff test of innate detonability or sensitizer effectiveness. Detonation does not create hazardous fragments, only plastic particles that are hurled 3 to 4 meters. Plastic pipe is also an abundant, inexpensive material. The liquid quantity (~2 liter), although huge for laboratory scale tests, is well sized for a small test range.

By its very nature, the plumbing pipe technique proves detonability of a liquid explosive weakly confined in a narrow inside diameter (ID) tube and proves the liquid will support detonation over a long path length. These proofs are by inspection, that is, no pipe is found after a successful shot, but a failure or short run will leave whole pipe beyond the detonation stop point. The minimum initiation requirement (weak confinement) can be found merely by reducing the initiator weight and seeing if the liquid still detonates. From the difference of confinement, conceivably some mixtures with small initiators could detonate in metal pipe but not in plastic pipe. Because it uses the worst case (i.e., weak confinement, long path), the method exposes the setup conditions needed for success in many circumstances. The basic findings involve no instrumentation, and thus, the method is well suited to low cost, survey testing of prospective liquid explosives.

With a high speed camera and a double exposure technique, described later, the method will reveal average detonation velocity and steadiness of detonation. With a good camera and filming technique, the detonation velocity of the liquid explosive is found to three significant figures.

The testing diameter limit, now set by plastic pipe availability, could be lowered by a change (e.g., an extension of clear, flexible, vinyl tubing) that would retain the long path feature of the method. Smaller plastic pipe is easily bent (an unused feature) after mild heating with a propane torch. This fact permits suitability tests of liquid explosives for conceivable applications demanding propagation of the detonation wave through curved, narrow passages. These particular tests were set up with straight, level pipe.

2.2 Tests.

2.2.1 Makeup. Nitromethane was sensitized eight ways, and the mixtures were tested for detonability by the pipe method of Section 2.1. The combinations of kind and percent sensitizer in various sized pipes are summarized in Table 1.

Table 1. Sensitizers Employed in Pipe Tests

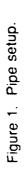
Sensitizer ^a (% volume)	Pipe Inside Diameter							
	12	16	17	18	21	26	35	40
DE	3%							
DETA	3%	3%	3%		3%	3%	3%	3%
ETH	3%	3%		7%				
ED	5%							
НМТ	<3%	••						
MOR	3%							
Neat NM		0%						
PYR	8%							
TETA	5%			7%				

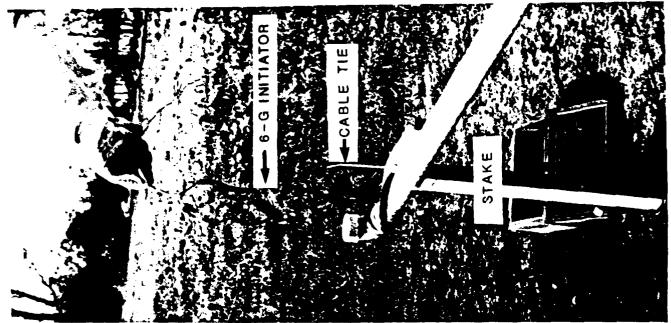
a. DE = diethylamine, DETA = diethylenetriamine, ETH = ethanolamine, ED = ethylenediamine, HMT = hexamethylenetetramine, MOR = morpholine, NM = nitromethane, PYR = pyridine, TETA = triethylenetetramine.

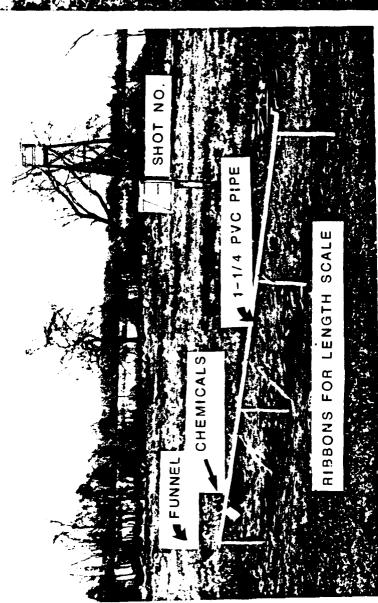
All mixtures were tested in the narrowest pipe available (12 mm inside diameter) in order to find a least upper bound for the critical diameter. Critical diameter is the explosive size below which a steady-state detonation will not propagate. Those tests fixing upon 3% DETA were attempts to learn about wall interaction effects on a mixture's detonation velocity.

2.2.2 Setup. The tests summarized in Table 1 were made at Range 8, Spesutie Island, APG, MD, in winter 1990-1991. Next to the control trailer, a high speed carnera was aimed downrange at an area near a firing line switching enclosure. In the test area, a line was run square to and approximately dividing the carnera's line-of-sight. This squaring was done once. For each test, wood stakes were hammered in line, and the plastic pipe was tied on the ends. A plastic cable tie was looped through a crosswise hole near the top of each stake to loosely confine the pipe. By sighting across a spirit level, a caller identified the stakes that needed to be pulled or hammered to level the pipe. End-to-end straightness was judged by eye. When the pipe was up, the ends were daubed with cleaner and plastic solvent. An elbow went on the initiating end, and another elbow, sometimes with a short standpipe, went at the far end. Bright surveying tape was tied at pipe midpoint and 1 meter on either side for a length scale. (The pipe itself served better on film.) The mounted pipe, shown in Figure 1, was then filmed, for a purpose of double exposure, and the film rewound and reloaded.

The sensitizer was a small percent of the volume of mixture required to fill the pipe. For different pipes, the total mixture volume was scaled from the ID-ratio squared, using ~700 ml for 12-mm ID, 20-ft pipe. A slight overage of mixture, when poured into the elbow, while jostling the pipe, left a small remainder to verify that air was not left trapped in the pipe. When no more gurgling was heard or seen at the ends, the ties were cinched. The explosive handler placed and taped in the elbow an initiator package consisting of an explosive pellet (5-gram of foil wrapped PBX) glued to a low voltage detonator connected to the replaceable firing line. When all was ready, a sequence timer was started from a countdown, and it started the high speed camera slightly before energizing the firing line. After the shot, when the area was declared safe, the test party inspected ground zero. Normally, all pipe was gone and the stakes were splintered. Their holes were reused, which accelerated the next setup by removing pipe alignment except for leveling.







A few passing remarks are in order here. The smallest pipe sagged so much that it was laid on slats over the wooden stakes. Half-inch pipe only needed an extra stake for support, and anything larger was rigid so that four stakes were enough. Early tests began with a transition piece (a) to keep the initiator explosion back from the straight section where the detonation velocity would be measured, and (b) to turn the detonation wave less abruptly than 90°. The transition piece consisted of a 45° elbow, one foot pipe, and another 45° elbow. Film did not show a detonation wave decelerating from the initiating explosion, so the transition piece was dropped for a plain elbow. A standpipe, used with light pipe, ensured that the sometimes unsupported end of pipe would not sag and drop the liquid level in the open elbow. A low level there puts the liquid out of touch with the booster, and a failure results.

2.2.3 .pes. These tests used opaque plastic plumbing pipe made of either PVC (polyvinyl chloride) or CPVC (the co-polymer) resin in a variety of diameters. The PVC pipe is white and comes in the greatest range of sizes, from so-called 1/2-inch to at least 6-inch diameter, in both thin and thick wall, for drain and cold water delivery, respectively; the CPVC is tan, is used for hot water service, and only comes in so-called 1/2- and 3/4-inch pipe diameters. The color of each type's fittings matches its pipe, and the fittings are not interchangeable and no adapters are made; therefore, a system cannot be of intermixed type. Both types are sold in standard lengths of 20 feet, and that became standard test length, too. In one bravura test, two lengths were coupled to see if 40 feet could be handled and shot. It could, awkwardly, but no advantage was found compared to using just one length.

Both pipe types are useful to our method: the PVC because its great range of diameter allows the finding of the maximum detonation velocity, which is a property of the mixture¹; the CPVC because its smaller inside diameter (12 mm) bounds the least diameter through which a detonation will propagate. About the smallest reported limit for weakly confined liquid explosive is 6 mm, in (5% weight) ethylenediamine or morpholine-sensitized nitromethane (Audrieth 1967). These particular tests aimed at finding both upper speed and lower diameter, but examination of results shows they were more complete in the latter goal. The method of these tests remains quite valid for both aims, nevertheless.

¹Once wall influence on the detonation wave is eliminated, no increase in speed will occur even for still larger pipe.

2.2.4 Camera. A rotating prism type of high speed camera was placed ~200 feet away to have a true length, broadside view of the pipe. The positions of the camera and pipe were reused test to test. A zoom lens was used to fill most of the 16-mm film frame with the 20-ft pipe length. The easily visible, standard pipe length and frame filling gave good spatial resolution of the head light of the detonation. The film was Eastman Ektachrome high speed daylight 7251, (ASA 400) in 16-mm x 100-ft rolls. It is a good film for general tests and is widely used at APG. Camera speed (and lead time) were continually raised to try to obtain more frames of the event. The authors' camera (Redlake Lab HYCAM) was dependable to 5,000 pictures/second, which gave six frames of the event. The start and finish frames are ignored, leaving four frames for analysis, a number that is sufficient to find average velocity and plot progress (constancy or steadiness) of the detonation along the pipe. Another plaguing problem was that the timing light system often failed, though checked beforehand. This failure caused most shots to be unusable for velocity measurement (steadiness can still be plotted).

Film reading was troublesome. At the head of the film, where it is still accelerating to the dialed-in camera speed, the exposure is correct to show the pipe and background. One of these frames was used to set the length scale in the projected view. However, at full speed, the frames darkened and nothing was seen until the booster explosion gave light. Next, the light from the traveling detonation wave was seen, but the pipe itself was hardly discernible. Because the film reader felt disoriented, a new procedure was entered to restore a sense of location of the detonation in the scene.

The essence of the new procedure was to double expose the film--the scene, then the event. The scene of the finished pipe setup was exposed at 1,000 pictures/second, daylight f/stop. The film was run out, reels removed, and, in the darkened trailer, rewound emulsion side in on its out-of-the-can reel. The rewind restores the original head of the film to its position outside the roll, feeding off from the bottom. The roll was rethreaded through the camera, and the camera speed and the f/stop were changed to expose only for the fast, bright detonation. Usually, these values were 5,000 pictures/second, f/5.6. The second camera run was of the shot, after which, the film was given normal development. The resulting look of the event was the normally exposed scene with a small, bright spot moving down the pipe with a comet-like plastic spray cone behind it, but no pipe destruction after the detonation passage. The glaring, spatially extended detonation light seen in singly exposed film is reduced to a small spot, and the position of the front is more accurately read.

The two superimposed images were aligned except for one test. Potential causes of picture misalignment are: use of a long focal length lens, camera vibration, accidental reversal of the reel during rewind, and accidental movement of the camera during film reloading. Misalignment (or miswinding) does not seriously harm film reading. The picture shows two pipes or a detonation not riding on the pipe or moving in the wrong direction.

A problem with the camera, besides timing light failure, was that the light cannot be turned off. The double exposure causes two sets of timing pulses, and that confuses camera speed determination.

3. RESULTS

The different mixtures of sensitized nitromethane were tested for detonability by the plumbing pipe method. Table 2 is a summary of shot conditions and results.

4. DISCUSSION

Table 1 is an organizing table that shows what sensitizers were used in what pipe;
Table 2 lists the results in the order they were obtained. Table 1 shows that mixtures were preferably tested in the narrowest pipe. That was done to obtain a small upper bound to the critical diameter. By Table 2, most mixtures in that restriction did detonate, showing that the mixtures' critical diameter is below 12 mm. Thus a pipe of inside diameter some amount below (above) 12 mm will stop (not stop) detonation. Otherwise, Table 1 shows that 3% DETA was tested in nearly all pipes. This theme was an attempt to measure detonation velocity with diameter restriction. No superiority is implied by the selection of 3% DETA. It was chosen because a low presence of sensitizer in the narrowest pipe gave detonation and the sensitizer was in ample stock. Since the mixture did detonate in 12-mm pipe, Table 2 not surprisingly records it detonated in all larger pipe. Due to camera timing light failure, just one velocity datum was collected for the runs in the mixture: 6240 m/s in 12-mm pipe. The expectation is for velocity to rise in larger pipe and level off to a final velocity which is the true detonation velocity of the mixture. The exceptional reason to test in larger pipes is to assure that the true detonation velocity of the mixture has been found.

Table 2. Summary of Pipe Tests

Shot	Pipe ⁸	Inside Diameter (mm)	Sensitizer ^d (% volume)	Result (6-g initiator)
1	1/2 PVCb	18	7% ETH	Detonation
2	1/2 PVCb	18	7% TETA	Detonation
3	1/2 PVC ^C	16	5% TETA	Detonation
4	1/2 CPVC	12	5% TETA	Partial [®]
5	1/2 CPVC	12	8% PYR	Partial ^{e,f}
6	1/2 CPVC	12	5% ED	Detonation
7	1/2 CPVC	12	3% DE	Detonation
8	1/2 CPVC	12	3% MOR	Detonation
9	1/2 CPVC	12	3% DETA	Detonation ^g
10	1/2 PVC ^C	16	3% ETH	No Test ^h
11	1/2 CPVC	12	3% ETH	Detonation
12	1/2 CPVC	12	<3% HMT	Detonation ^I
13	3/4 PVC	21	3% DETA	Detonation
14	1 PVC	26	3% DETA	Detonation
15	3/4 PVC	21	3% DETA	Detonation
16	1-1/2 PVC	40	3% DETA	Detonation
17	1 PVC	26	3% DETA	Detonation
18	3/4 PVC	21	3% DETA	Detonation
19	1/2 PVC ^C	16	Neat NM	Failed
20	1/2 PVC ^C	16	Neat NM	Failed
21	1/2 PVC ^C	16	3% DETA	Detonation
22	1/2 PVC ^C	16	3% DETA	Detonation
23	1-1/4 PVC	35	3% DETA	Detonation
24	3/4 CPVC	17	3% DETA	Detonation

 $^{^{\}rm a.}$ Pipe 6.1 m (20 ft) length; exception pipe 3 was 12.2 m. Designations are conventional; not actual inch dimensions.

b. Thin wall pipe. c. Thick wall pipe.

d. DE = diethylamine, DETA = diethylenetriamine, ETH = ethanolamine, ED = ethylenediamine, HMT = hexamethylenetetramine, MOR = morpholine, NM = nitromethane, PYR = pyridine, TETA = triethylenetetramine

e. Detonation for 2/3 of pipe length. f. 6190 m/s

g. 6240 m/s h. Pipe leaked dry. i. 6640 m/s

The initiator weight used was small (5.5 g) but not minimal. The mixtures, though mostly cap sensitive according to the literature, were not so initiated. Instead, the initiator size was a compromise between accommodating a reliable load in an elbow fitting and not causing an overdriven detonation that might not decelerate within a short distance on the pipe.

The key variables in nitromethane explosive testing are the percent sensitizer and inside diameter of the pipe. Detonation will either not occur or will stop itself as both variables diminish. As the information is more useful, the program makeup inclined toward using minimum combinations that might cause detonation failure. In fact, failure was only produced in neat nitromethane (Shots 19 and 20), while triethylenetetramine and pyridine-sensitized nitromethane failed 2/3 of the way down the pipe (Shots 4 and 5). Neat nitromethane was the "mixture" in the only repeated test. It was contained in a favorably large pipe and moderately strongly initiated as usual and neither time did it detonate. Those two tests confirmed the importance of sensitizing nitromethane to obtain a liquid explosive. The partial failures were likewise important for they identified the probable critical diameter as 12 mm for those mixtures. As far as the authors know, more amine sensitizer does not reduce the critical diameter of the mixture. However, the partial failures of Shots 4 and 5 show that this question could be tested by the pipe method i.e.,add more sensitizer and see if the detonation travels the entire pipe length.

Three mixtures in 12-mm ID plastic pipe gave a detonation speed of 6300 ± 300 m/s. For comparison, the fastest ethylenediamine mixture is 6600 m/s at 1 to 2% sensitizer (Ericksen and Rowen 1945). They found that velocity will decrease 10% between 1 and 15% sensitizer present; it will be zero (no detonation) above approximately 40% sensitizer (Laurence 1966). These tests were unconcerned with the upper sensitizer limit. The other effect of adding sensitizer above an efficacious minimum amount is to slightly raise the mixture's sensitivity to initiation. Choosing the best sensitizers makes this property unimportant and avoids the loss of potency because of the reduction of the explosive fraction, the nitromethane. As a practical matter then, 5% sensitizer would be a first choice.

5. SUMMARY

A method of testing liquid explosives by filling and exploding plastic plumbing pipe was conceived and demonstrated. The method worked well; tests were simple to set up and inexpensive. By its nature, the method succeeds without use of instrumentation, in revealing quantities important to explosive testing such as detonability of prospective mixtures without the aid of strong confinement, initiation requirement, bound to critical diameter, and propagation over long path. Installation of a high speed framing camera yields detonation velocity and steadiness. A double exposure technique was described and tested and resulted in improved film reading of the event.

These tests tried eight known sensitizers, a small fraction of those in the literature.

A three percent solution of these sensitizers was enough for a detonable mixture in 16-mm ID pipe: diethylamine, diethylenetetramine, ethanolamine, ethylenediamine, hexamethylenetetramine, morpholine, triethylenetetramine. Two sensitizers, 8% pyridine and 5% triethylenetetramine, allowed detonations that stopped on long, narrow (12-mm ID) pipe. Such partial failure cannot be detected in laboratory test tube experiments of mixtures. This unexpected result demonstrated a feature of the method.

The importance of sensitizers was confirmed by two non-detonation results with neat nitromethane. This result was partly unexpected because the tests were run with contraconditions of moderate initiator weight but weak confinement. Other agencies have accepted the risk, costly in large quantity tests, of non-detonation of neat nitromethane. These agencies use large booster charges to compensate for the insensitivity of neat nitromethane.

For practical use as a bulk liquid explosive, a preferred combination would be 5% (by volume) amine sensitizer in nitromethane and 6 grams explosive initiator. Overall, the plastic plumbing pipe method is a workable means for testing liquid explosives.

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