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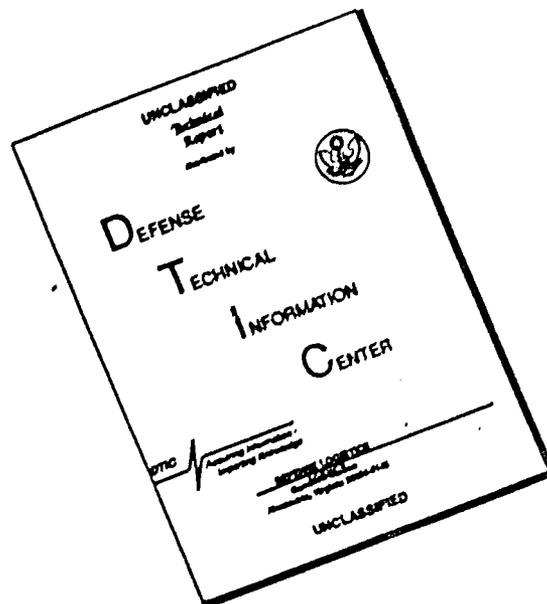
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RESEARCH ON HIGH-DENSITY LIQUID-PROPELLANT GELS

(U)

by
G. W. Burdette
D. H. Couch
D. R. Poole

Propulsion Development Department

ABSTRACT. The effects of introducing powdered, high-density metals into liquid-propellant fuels are reported, and future plans are briefly mentioned. The thixotropic gels formed by the addition of lead and tungsten powders to unsymmetrical dimethylhydrazine are within viscosity limits required for use in liquid-propellant engines. These gels were found to be insensitive to shock and were thermally stable at 74°C. Attempts to gel hydrazine with several metal powders resulted in decomposition of the hydrazine. (CONFIDENTIAL)

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FOREWORD

This study of gelled liquid propellants was done at the U. S. Naval Ordnance Test Station (NOTS) under Bureau of Naval Weapons Task Assignment RMMP-24-080/216-1/F009-06-002 during the period 1 July 1961 to 1 January 1962.

The status of high-density, gelled, liquid-propellant work and methods for the preparation and evaluation on a laboratory scale of various types of gels are discussed. Future plans are briefly mentioned.

This report has not been technically reviewed and does not necessarily reflect the official views of this Station.

Released by
CHARLES J. THELEN, Head,
Propellants Div.
8 June 1962

Under authority of
JAMES T. BARTLING, Head,
Propulsion Development Dept.

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INTRODUCTION

Liquid propellants offer a number of attractive features including high performance, variable thrust, on-off operation, and hypergolic ignition. They have relatively low densities, however, and some of them present safety hazards when spilled.

The low density of liquid systems can be improved by the use of high-density additives. Theoretical calculations have shown that the addition of a dense material to a propellant system can increase performance in terms of burnt velocity of a rocket (Ref. 1). Maximum benefit is achieved when the dense material is programmed into the rocket chamber in decreasing amounts with time. These dense additives are generally solids that must be handled as finely divided powders, and some means of preparing a stable suspension of the dense additive in the liquid propellant is needed.

In addition to better performance, gels offer a safety advantage. In case of spills, the material tends to remain where it falls and does not flow freely as a liquid would. Gelled propellants could be lost from a ruptured tank in two ways: if the rupture is in the lower part of the tank, the force needed to cause the gel to flow could result from either the vapor pressure of the propellant or the force of gravity or both; and propellant loss could occur through evaporation.

One method of incorporating dense metal additives in liquid propellants is to form a fluid slurry of the particles in the fuel. This method has the disadvantage that the particles tend to settle out and the slurry must be stirred continually, or an additional gelling agent must be added to prevent the settling action.

If the metal particles are very small (1 micron or less), a stable gel suitable for liquid-propellant applications may be formed without the use of an additional gelling agent. The preparation of the gel is simplified, and better performance is to be expected without an inert gelling agent.

This report contains the results of a continuing study of the preparation and properties of gelled propellants in which the metal particles act as gelling agents.

DISCUSSION

Since gelled liquid-propellant systems offer advantages both in safety and performance, the preparation and determination of physical properties of such systems is very important. Unfortunately, however, the preparation of gels is still more an art than a science, and information on systems of interest for propellants is practically nonexistent.

Thixotropic, pseudoplastic, and plastic gels appear to be the most satisfactory types for use with liquid-propellant systems.

Thixotropic gels are those whose consistency depends on the duration and rate of shear. Under static conditions, these gels exhibit a weak structure and are nonflowing semisolid bodies. If, however, the yield point (a critical value of stress that must be exceeded before flow begins) is exceeded and the gels are sheared at a constant rate, the structure will be progressively broken down, and the viscosity will decrease with time.

The structure of true thixotropic materials breaks down completely under the influence of high shearing action (such as injection through an orifice), and they behave like true Newtonian liquids. As soon as the shearing action has ceased, however, the structure begins to reform. The rate of breakdown and reformation of the structure of a gel is a characteristic of that gel, and the time required for the transition may vary with different gels from a fraction of a second to many hours.

Pseudoplastic gels exhibit no yield point, and the flow curves for these materials indicate that the ratio of shear stress to the rate of shear (apparent viscosity) falls progressively with increasing shear rate. The flow curves become linear, indicating constant viscosity, at very high rates of shear.

Plastic gels behave like pseudoplastic gels with the exception that they do have a finite yield point.

The three gels listed in Table 1 are considered to have properties that would permit them to be used in liquid-propellant engines. The

TABLE 1. RHEOLOGICAL PROPERTIES OF HIGH-DENSITY GELS

* The carrier liquid was UDMH, temperature was 25°C, and shear rate was 30,000 sec⁻¹.

Gelling agent	Agent wt., %	Particle size, μ	Carrier liquid wt., %	Apparent viscosity, cp	Yield point, dynes/cm ²
Tungsten	84.0 ^a	1.0	5.7	50.7	3,200
Tungsten	85.0	2.0	15.0	50.0	2,700
Lead (spherical)	91.0	2.5	9.0	23.4	400
None	100.0	0.509

^aIncludes 0.3% SiO₂.

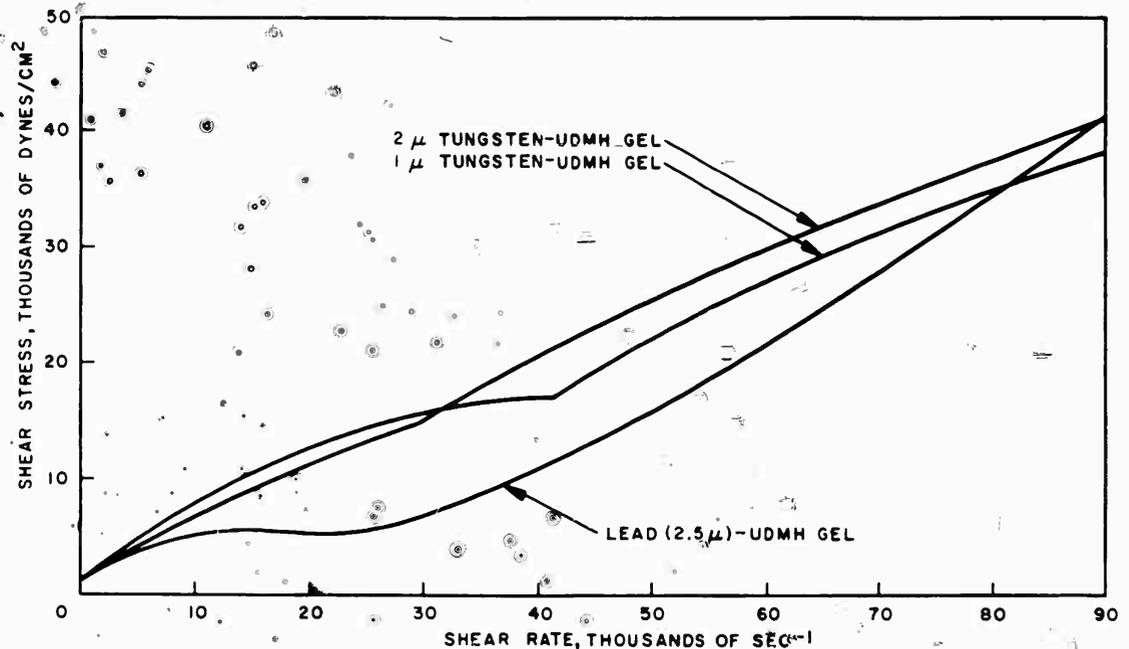


FIG. 1. Shear Rate-Shear Stress Curves for Some High Density Gels at 25°C.

injectors would probably need to be altered slightly because of the higher viscosity, but the changes would not be difficult.

The unusual shapes of the curves in Fig. 1 are not easily explained. Whatever the cause (excluding the possibility of measurement error), it appears that over a certain range of shear rate the apparent viscosity is less than at higher or lower shear rates. This could be of importance in injector design.

The gels mentioned in this report are assumed to be thixotropic, although it has not been proved that their shear thinning (reduction in apparent viscosity) is a function of time as well as shear rate. This is probably irrelative in any case, because the gel-fluid and the fluid-gel transformations appear to be extremely rapid, which is a very desirable feature of the gels since they may be sheared only for a short time in the injector, and in case of a spill they would "set up" rapidly to form a pile rather than to flow freely.

One problem encountered in the expulsion of gels from tanks using direct nitrogen or air pressure is a coring or plug flow of the gel only above the outlet hole, resulting in a large portion of the gel remaining around the walls of the tank. This problem can be overcome by using a piston or a collapsible bag to expel the gel.

GELLING AGENTS

Several high-density, finely divided metals were considered as gelling agents for various rocket fuels. These metals must be nonreactive with the fuels to be gelled and also be of small enough particle size to form suitable gels.

Particle shapes may play an important part in the gel formation. It is generally believed that thixotropy is more pronounced with asymmetrical particles (Ref. 2), but thixotropic gels have also been made using spherical particles (Ref. 3).

For this study, tungsten was obtained in angular and spherical form, and lead and iron were obtained in spherical form. Gels prepared with spherical lead particles were compared to gels prepared with angular tungsten particles of the same size, and it was found that at a given shear rate the viscosity of the lead gel was approximately one-half that of the tungsten gel (Table 1). The density of the lead gel was slightly greater than that of the tungsten gel.

The gels were prepared with metal powders in the 1 to 2-micron range. The carrier liquid was unsymmetrical dimethylhydrazine (UDMH) in all cases. Attempts to gel hydrazine resulted in its decomposition (Table 2).

TABLE 2. RESULTS OF SEARCH FOR HIGH-DENSITY GELLING AGENTS

Gelling agent	Source	Particle		Observation of gelling agent in	
		Size, μ	Shape	UDMH	N ₂ H ₄
Tungsten powder	Wah Chang Corp., New York	1.0	angular	gel	decomposition
Tungsten powder	Sylvania Electric Products Inc., New York	2.0	angular	gel	decomposition
Tungsten powder	Linde Co. Div. of Union Carbide Corp.	0.03	spherical	gel	decomposition
Lead powder	Metalead Products Corp., Palo Alto, Calif.	2.5	spherical	gel	decomposition
Iron carbonyl powder	General Aniline and Film Corp., N. Y.	3.0	spherical	gel	decomposition
Zirconium	Carborundum Co., Niagara Falls, N. Y.	3.5	angular	vigorous decomposition ^a
Zirconium	Carborundum Co., Niagara Falls, N. Y.	2.0	angular	gel
Zirconium	Carborundum Co., Niagara Falls, N. Y.	20.0	angular	compatible

^a The zirconium evidently catalyzed the decomposition of the hydrazine, which ignited shortly after mixing.

The 0.03-micron tungsten-UDMH gel (Table 2) was prepared from a small complementary sample; recently, a large enough sample was obtained to permit a more thorough evaluation.

MICROSCOPIC EXAMINATION OF METAL POWDERS

Examination of the 1-micron tungsten powder produced by the Wah Chang Corp. and the 2-micron tungsten powder produced by Sylvania Electric Products Inc. through a 1,000-power microscope showed the particles to be very irregular with many jagged corners. The 2.5-micron lead powder produced by the Metalead Products Corp. appeared spherical and smooth under the same magnification. Electron photomicrographs of the 0.03-micron tungsten produced by the Linde Co. Division of Union Carbide Corp. showed the particles to be spherical.

METHODS OF GEL PREPARATION

The gels listed in Table 3 were prepared by slowly adding UDMH to the metal powders until a gel of desired consistency was obtained. Stirring by hand was sufficient for preparing homogeneous gels in several-hundred-gram quantities. Prolonged mixing by hand or vigorous mechanical shearing (Waring Blendor) caused no noticeable change in the physical properties of the gels. No noticeable exothermic reactions occurred upon mixing the gelling agents and carrier liquids in the above-mentioned quantities. These gels behaved as thixotropes and flowed quite readily when shaken or agitated. After standing, a very slight settling of the suspended materials occurred, which had no significant effect on the rheological properties.

TABLE 3. COMPOSITIONS OF HIGH-DENSITY GELS

The carrier liquid was UDMH.

Gelling agent	Particle size, μ	Agent wt. %	Carrier liquid wt. %	Density of gel
Tungsten.....	1.0	84.0 ^a	15.7	4.2
Tungsten.....	2.0	85.0	15.0	4.0
Tungsten (spherical) ^b	0.03	45.0	55.0
Lead (spherical).....	2.5	91.0	9.0	5.1
Iron (spherical) ^b	3.0	91.5	8.5	4.3

^a Includes 0.3% SiO₂. No obvious changes in rheological or physical properties were noticed after adding this material.

^b Have not been fully evaluated.

VISCOMETRIC MEASUREMENTS AND APPARATUS

The accurate measurement of flow properties of gels is necessary for selecting those suitable for use in liquid-propellant engines.

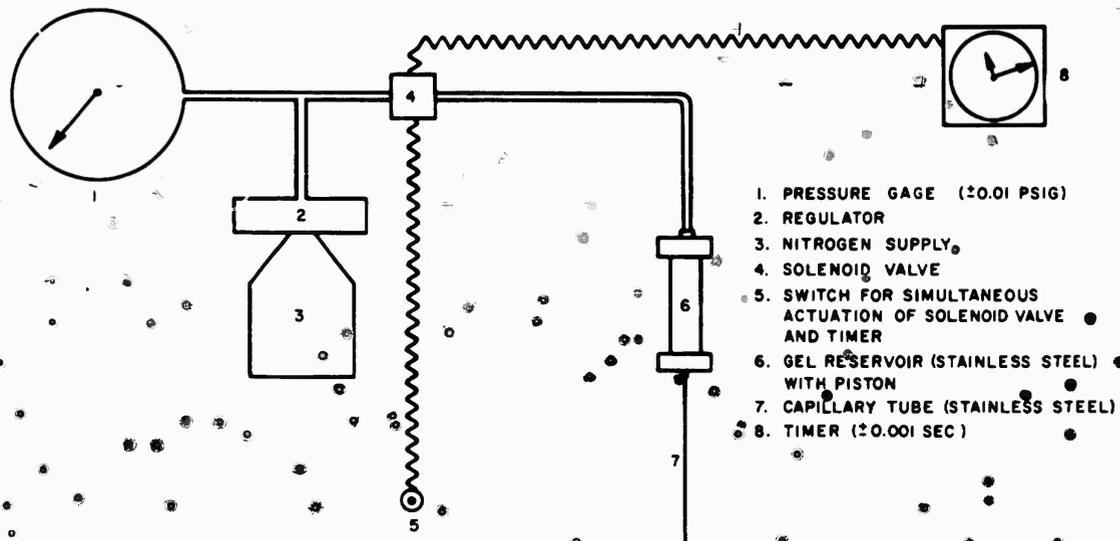


FIG. 2. Capillary Tube Viscometer.

A capillary-tube viscometer (Fig. 2), which is rather easily built and is capable of providing reliable quantitative rheological data, was built to measure flow properties of the gels. Capillary-tube viscometers are obviously to be preferred when the data are to be used for pipe flow systems inherent in liquid-propellant engines.

The essential features of the viscometer are a means for the control and measurement of the pressure difference across a capillary tube of known dimensions together with a method for determining the rate of flow through the tube.

The gel reservoir consisted of a stainless steel cylinder of approximately 100-milliliter capacity that was fitted at both ends with 1/4- to 1-inch flare fittings. A stainless steel capillary tube of known length and bore diameter was inserted through a hole drilled in a 1/4-inch flare cap and was silver-soldered in place. When the cap was screwed onto the lower 1/4-inch flare fitting, the capillary tube extended up into the gel reservoir. This eliminated any gel flow through the flare fitting, which, because of its irregular shape, could alter flow properties.

Nitrogen from a high-pressure cylinder was used to force the gel from the reservoir through the capillary. Regulators and a large ballast tank were used to control and maintain a constant pressure, and a three-way solenoid valve was used to ensure rapid application and release of this pressure. An electric timer coupled to the solenoid valve was used to measure accurately the time the pressure was applied to the gel. The rate of flow was measured by weighing a sample collected over a measured time.

The viscometer was calibrated with Newtonian fluids.

The fundamental equation (Poiseuille's Law) for flow through a capillary (Ref. 4) is

$$Q = \frac{\pi(\Delta P)R^4}{8LN}$$

where

Q = flow rate, cm³/sec
 R = capillary radius, cm
 L = capillary length, cm
 ΔP = pressure across capillary, dynes/cm²
 N = fluid viscosity, poise

Although apparent viscosity may be calculated directly from this equation, the mean shear rate (D) and the shear stress (S) are calculated and the apparent viscosity is obtained from the ratio of S to D because the apparent viscosity of non-Newtonian fluids is dependent on the rate at which the gel is sheared.

$$N = \frac{S}{D}$$

where

$$S = \frac{R(\Delta P)}{2L}$$

$$D = \frac{4Q}{\pi R^3}$$

Shear stress-shear rate curves for several gels are shown in Fig. 1. It may be observed that the measurements extend over a wide range of shear rates. The gels that were studied exhibited shear thinning to a degree that indicates they are suitable for expulsion through liquid-propellant injectors. Apparent viscosities at given flow rates for these gels are shown in Table 1.

These gels all exhibit a yield point (stress that must be exceeded before flow will begin) that is desirable in that the gels will not flow under static conditions. Yield points for the gels were determined by the method of Agranat and Shirokov (Ref. 5), which utilizes a cone immersed under its own weight into the test gel to a depth that is determined by the yield point of the gel, the vertex angle of the cone, and the cone weight (Fig. 3). The depth of immersion must not be greater than the height of the cone. For a given gel this can be controlled by varying the density of the cone or the angle of the vertex.

The yield point is calculated using the following formula:

$$\theta = K(G/h^2)$$

where

θ = yield point
 G = force by action of cone immersing
 h = immersion depth
 K = constant depending on α
 α = 1/2 vertical angle of the cone

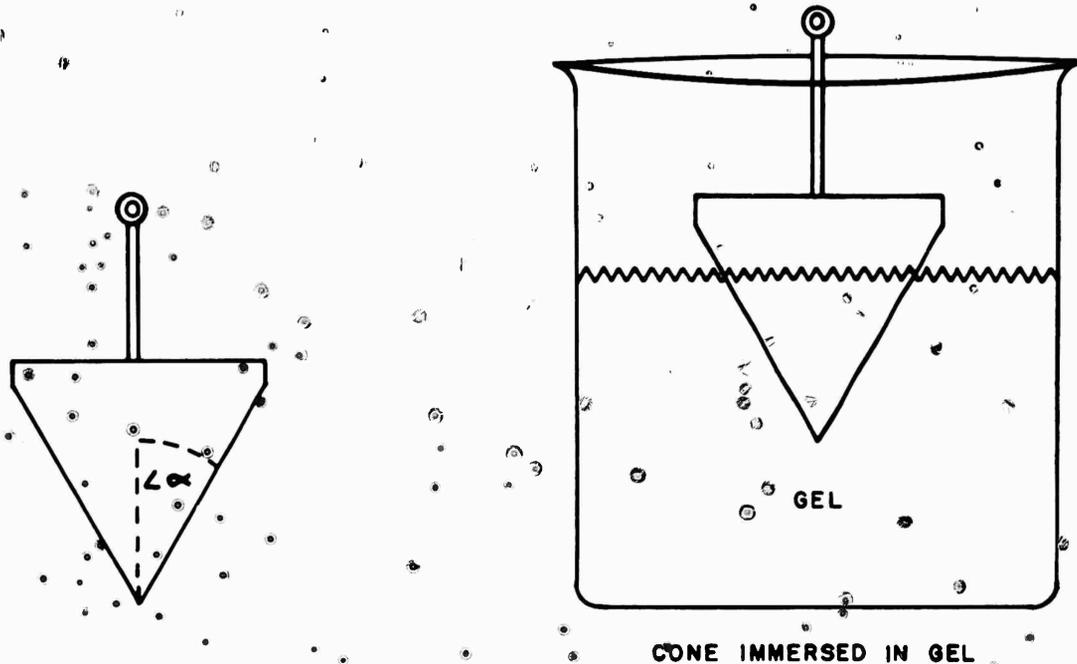


FIG. 3. Yield Point Apparatus.

Yield point values are given in Table 1.

THERMAL STABILITY OF GELS

Thermal stability tests were performed at both low and high temperatures on the gels listed in Table 1 to determine whether or not decomposition, detonation, or various other changes in gel structure might occur. Samples of the gels sealed in glass ampules for a 5-month period exhibited no noticeable changes in physical properties, and no pressurization of the ampules occurred as a result of decomposition. Samples stored at -10°C for a 2-month period showed no noticeable change in appearance or properties.

High-temperature studies were performed by placing one 10-milliliter sample of each of the gels in a thermal stability apparatus (Fig. 4). These gels were subjected to a maximum temperature of 74°C for a period of several hours, as shown in Fig. 5. Any pressure rise that might have occurred under isothermal conditions could be attributed to decomposition of the gelled propellant. No decomposition was evident in the various gels.

DETONATION SENSITIVITY OF GELS

Five 1-milliliter samples of the gels listed in Table 3 were subjected to the blast of M-36 detonators to see whether a detonation would

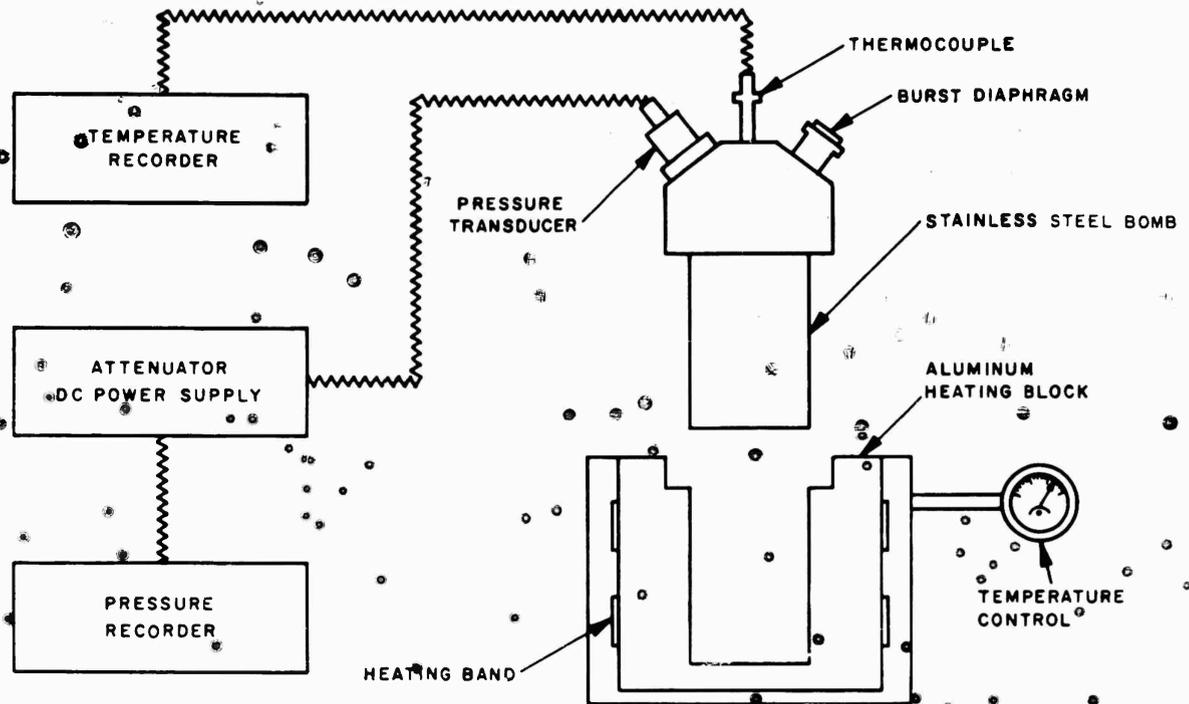


FIG. 4. Thermal Stability Apparatus.

occur or propagate. Each gel sample was placed in a glass tube 1.2 centimeters in inside diameter and 7 centimeters long, which was, in turn, placed in a standard half-inch iron pipe 9 centimeters long. Pipe caps were screwed onto each end of the pipe, with the detonator lead wires protruding through a small hole in the top cap. The detonator was placed at the surface of the gel and then detonated. Detonation of a propellant sample is evidenced by the fragmentation of the half-inch pipe. Very slight damage to the pipe indicated no detonation. This simple qualitative test is one method used at NOTS for screening new propellants or various propellant mixtures to see whether they are safe to use in engine tests.

FUTURE WORK

The rheological properties of various new high-density gels will be determined, including 0.03-micron tungsten-UDMH gels. Work will also be carried out on systems containing high-energy metal additives, such as boron, aluminum, aluminum hydride, and magnesium.

Investigations will be conducted on the gelation of various oxidizers. Efforts will be directed toward minimizing or eliminating syneresis (gel

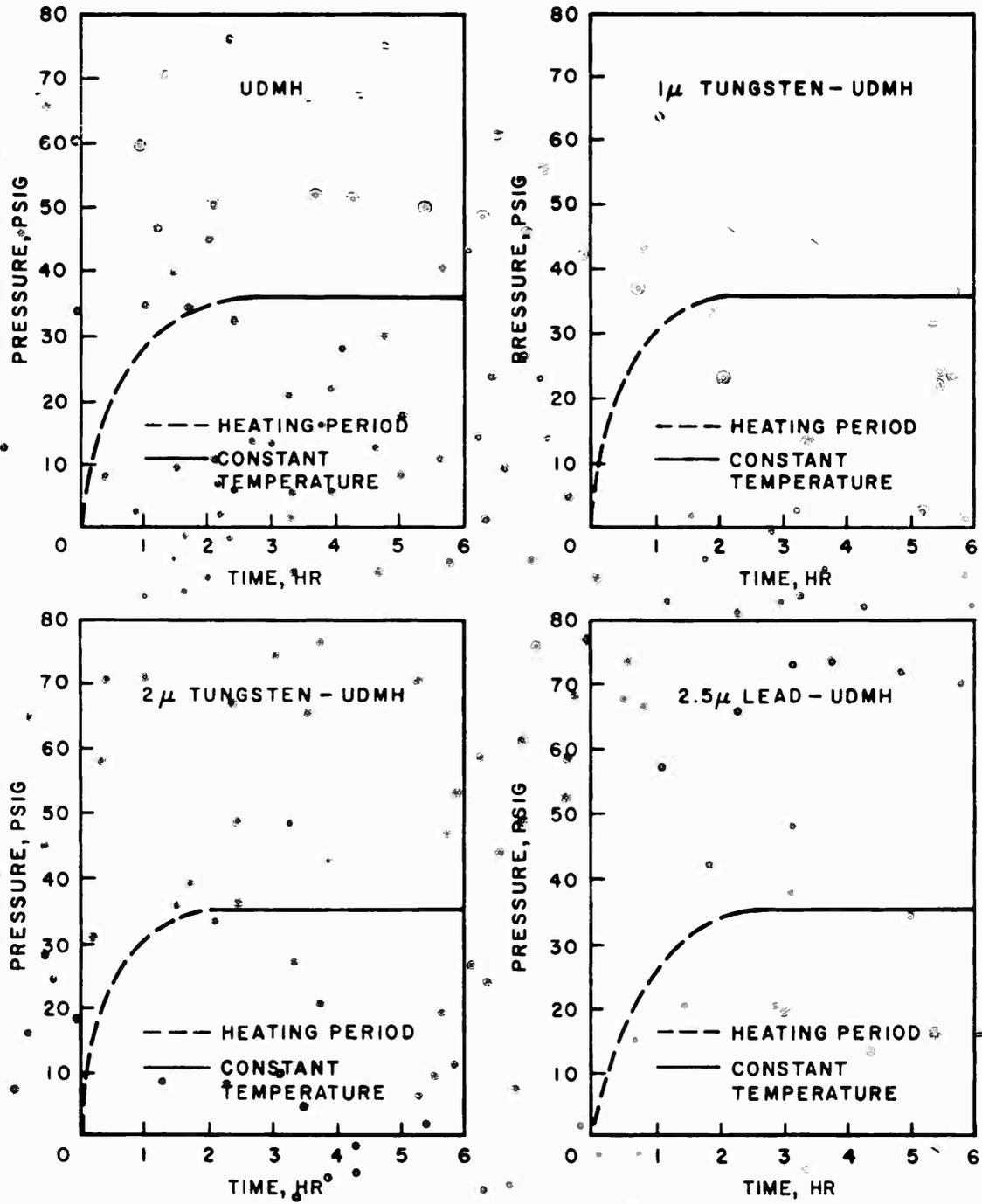


FIG. 5. Thermal Stability Curves for Some High-Density Fuel Gels at 74°C.

shrinkage) and will be continued to determine the effect of particle shape and size of various gellants on gelation.

Plans are being made to use present and future gels in small-scale static-engine tests (300-pound chamber pressure, 200-pound-thrust liquid-propellant test engine).

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