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# DOUBLE-BASE BINDER IMPROVEMENT

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A DIVISION OF LOCKHEED AIRCRAFT CORPORATION
REDLANDS, CALIFORNIA

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AIR FORCE ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

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A Division of Lockheed Aircraft Corporation
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# FOREWORD

This is the second Quarterly Report issued under Contract No. F04611-67-C-0078, "Double-Base Binder Improvement," covering the period 1 June through 31 August 1967 and submitted September 1967. This contract is assigned to Lockheed Propulsion Company, Redlands, California, and is monitored by Lt. William Anders, Air Force Rocket Propulsion Laboratory, Edwards, California.

Technical effort under this contract has been performed by the following: Dr. W. E. Baumgartner, (Program Manager); Dr. G. E. Myers, (principal technical investigator); E. E. Larkin, and Waldemar Koehler.

This report is unclassified in its entirety.

Publication of this report does not constitute Air Force approval of the report's findings and conclusions. It is published only for the exchange and stimulation of ideas.

William Ebelke
Colonel, USAF
Chief, Propellant Division

## ABSTRACT

An investigation is underway into the physico-chemical factors controlling the mechanical behavior of slurry cast and base grain composite double-base propellants, the primary objective being to improve the mechanical properties of slurry cast systems at least up to those of analogous base grain systems. Studies during the second quarter involved establishment and testing of tritium autoradiographic procedures for investigating inhomogeneities in these systems; synthesis of tritiated ingredients; preparation of casting powder and base grain propellant by NOS and of special ball powders by Olin; observations of glass transitions in NC gels and plasticizers; construction of microscope-strain apparatus.

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# SECTION I

## INTRODUCTION AND SUMMARY

In this program, an investigation is being conducted into physico-chemical factors controlling the mechanical behavior of slurry cast and base grain composite double-base propellants (CMDB), the primary objective being to improve the mechanical behavior of slurry cast systems to the point where they at least equal those of similar base grain propellants.

The base grain process has been used almost exclusively whenever nitroglycerin serves as a plasticizer and generally (though not always) produces propellants of superior mechanical properties relative to those from the slurry cast process. The latter process, however, possesses very distinct processing advantages over the base grain because of its comparative simplicity, safety, and versatility.

While extensive investigations have been made into the general problem of CMDB propellant mechanical behavior, our understanding still remains comparatively poor, in both a fundamental and an applied sense. Considering their wide usage, in fact, nitrocellulose (NC) gels are perhaps the least understood of the major thermoplastic systems. Nevertheless, the CMDB propellants, and particularly those prepared by the slurry cast process, appear at present to offer the greatest potential as a vehicle for high energy ingredients. Thus, a significant improvement in slurry cast propellant mechanical behavior is much to be desired.

Because of the complexity of the CMDB system, any program expecting to achieve significant improvements in mechanical behavior within a reasonable period must be highly selective in its choice of variables for study and in the depth to which it can delve among those variables. In the present program, specific tasks have been selected, each dealing with a critical facet of the overall program. Under each task, the particular phenomena which are believed to be controlling will be singled out for thorough study.

The major program tasks are concerned with:

- Detection of various types of binder micro-inhomogeneity, determination of consequent limitations upon physical properties, and study of means to overcome such deficiencies.
- Differentiation among the various factors controlling the separate processes occurring during combined NC solution/gelation/cross-linking to provide the basis for development of an optimized binder system.
- Establishment of the inherent binder-solids adhesional character in the optimized binder system and determination of means to overcome deficiencies to yield an optimized propellant.

Efforts during this quarter included:

- Establishment of tritium autoradiographic procedures and conduct of preliminary experiments for the purpose of determining binder/propellant inhomogeneities.
- Synthesis of tritium-labeled double-base ingredients--EGDN, TMETN, NC.
- Preparation of casting powder and base grain propellant at the Naval Ordnance Station to be employed in a comparison of mechanical behavior of base grain and slurry cast propellants at LPC. Procurement of special ball powder samples from Olin.
- Initiation of studies of low temperature transitions in CMDB binders/propellants.
- Design and construction of an apparatus permitting microscopic observation of thin specimens during straining.

# SECTION II

# DISCUSSION

## 1. BINDER - PROPELLANT INHOMOGENEITIES

Past investigations have demonstrated the existence of relatively gross inhomogeneities in some CMDB binders and propellants. Definitive studies are lacking, however, with regard to the existence of more subtle inhomogeneities, the extent of their influence upon mechanical behavior, and their dependence upon ingredient structure and form. This program is attempting to resolve these questions with the use of tritium autoradiography, which is capable of greater specificity, spatial resolution, and sensitivity than can be obtained by applying more conventional techniques to such systems (e.g. x-ray radiography, electron or optical microscopy). The weak $\beta$-rays of tritium, for example, in principle permit localizations of labeled ingredients to be determined within a resolution of one micron.

### a. Autoradiographic Experiments

Autoradiographic procedures involve the following general steps:

(1) Sample preparation, including introduction of tritium-labeled ingredients.

(2) Preparation of thin sections of the system of interest.

(3) Application of radiation-sensitive photographic emulsions or films.

(4) Storage/exposure of composite sample, followed by development and microscopic examination.

These steps and efforts taken to date to implement them are discussed in greater detail in the following:

- Sample preparation: This necessitates first the synthesis of labeled materials, e.g., tritiated plasticizers, NC, crosslinkers, and efforts in this direction are discussed in detail in Section 1b below. Incorporation of the tritiated ingredients into binder/propellants is straightforward. We have constructed equipment for use in making the necessary 5 to 10 g mixes under vacuum and with careful temperature controls.

- Preparation of thin sections: Microscopic examination of the final autoradiograph requires the use of relatively flat and transparent sections of the system of interest. Thus far these specimens have been prepared by sandwiching a small amount (~10 mg) of the uncured mix between two microscope slides. After cure the cover slide is removed, leaving an approximately 0.010 inch thick

section. Ultimately the more desirable procedure will be to microtome sections from bulk (5 to 10 g) samples and cement these to a microscope slide; brief experiments have demonstrated the feasibility of this operation.

- Application of radiation sensitive photographic emulsion: This coating must adhere very closely to the section because of the very short path length of tritium β rays in air. Of the applicable procedures here, experiments have demonstrated the superiority of coating with molten (40°C) NTB-2 Kodak Nuclear Track Emulsion, using a simple dipping technique. After dipping, the samples are allowed to drain and dry in the dark before storing in dry, light-tight boxes.

- Storage/exposure, etc., of emulsion-coated sample: Wherever the tritium-labeled ingredient is concentrated in the sample, the resultant β rays will cause darkening of the immediately adjacent film, and this darkening may be correlated with any optically observable structure within the sample. Since the average path length of tritium β-rays is about 1 micron in organic matter, resolution is of that order and only the topmost layer of sample actually affects the photographic emulsion.

  Controversy exists as to whether the emulsion-coated sample can be stored (exposed) at ambient temperature or should be exposed at lower temperatures. Both may well prove desirable for this study but as yet only low temperature (4°C) has been employed to minimize possible chemical interactions between sample and emulsion.

  Exposure time obviously depends upon tritium activity level and the extent of ingredient localization, i.e., upon microactivity levels. Following guidelines in the biological literature, initial experiments here were performed with PNC, TEGDN, tritiated ethylene glycol mixtures at various activity levels. These were exposed for varying times at 4°C and subsequent development and examination indicated that ten days with an overall activity of 5 μc/g mix resulted in detectable localized darkening of the film.

  After the samples are stored for a sufficient length of time, the slides were developed using standard photographic techniques. The developer is Kodak Dektol (1:1) and the sample is fixed in Kodak Acid Fixer. Development is performed with the emulsion still coating the sample so that subsequent microscopic examination of the composite will permit correlation between the radiograph and any optically observable structure.

b. Synthesis of Tritium-Labeled Ingredients

Initial materials selected here are nitrocellulose and the plasticizers EGDN and TMETN. Ultimately it is expected that other plasticizers, as well as crosslinkers and catalysts, will be prepared.

(1) EGDN

Labeled EGDN (25 g) has been prepared at sufficiently high activity (170 μc/g) by nitration of tritiated ethylene glycol. This was carried out by stepwise addition of the glycol to a two-phase mixture of $HNO_3/H_2SO_4$ and $CH_2Cl_2$ kept below 20°C. After two hours at ambient temperature the acid layer was drawn off and discarded and the $CH_2Cl_2$ layer washed with $H_2O$ and $NaHCO_3$. Subsequent drying, filtration, and vacuum removal of $CH_2Cl_2$ gives the EGDN in almost 100 percent yield. The purity of the high level EGDN ($H^3$) was verified by $CH_2Cl_2$ elution from an alumina column, using liquid scintillation counting of the fractions.

(2) TMETN

Three grams of high level (585 μc/g) trimethylolethane (TME) have been synthesized by a combined aldol condensation/Cannizzaro reaction between propionaldehyde and tritiated formaldehyde (Ref. 1).

$$CH_3\,CH_2\,CHO + 3\,HCHO \xrightarrow[NaOH]{aq} CH_3\text{-}C(CH_2OH)_3 + HCOOH$$

The literature procedure was followed closely except that greater incorporation of tritium was found if, instead of adding the propionaldehyde slowly to the mixed cold/labeled formaldehyde, one mole of labeled formaldehyde was first reacted per mole of propionaldehyde and the remaining two moles of formaldehyde subsequently reacted. Based upon the overall theoretical yield of tritiated TME, a 33 percent yield was actually obtained. Although this high level material has not yet been nitrated, successful nitrations of cold and low level TME have been conducted using the same procedure employed for the EGDN.

(3) NC

One intent of this program is to study the differences in structure of base brain and slurry cast propellants by incorporating labeled NC into casting powder and PNC and performing autoradiographic observations upon propellants prepared from them. For this purpose the labeled NC must not differ significantly in behavior from NC normally employed in preparing casting powder and PNC.

Initial experiments have centered upon labeling NC by means of low degrees of reaction with tritiated acetic anhydride. Since the latter is available at quite high activities, it should be possible to prepare sufficiently active NC without reacting more than 1 to 2 percent of the NC residual hydroxyls. This should constitute negligible modification of the NC structure and properties as long as the acetylation conditions were mild enough to preclude other structural changes, e.g., oxidation, denitration.

Successful incorporation of tritiated acetic anhydride has been accomplished in preliminary experiments under heterogeneous (Ref. 2) and homogeneous conditions using fibrous NC (12.6 percent N). Results of one series of experiments in the heterogeneous system are shown in Table I.

## TABLE I

## HETEROGENEOUS ACETYLATION OF NC

| Reaction System | Volume of Added Acetic Anhydride[a] | Total Activity in Reaction Mixture | Tritium Activity in Product | % of Added Activity Incorporated | % Residual Hydroxyls Acetylated[b] |
|---|---|---|---|---|---|
| 2.5 g NC; 60 ml $CH_2Cl_2$; 100 µl 70% $HClO_4$ | 250 µl | 117 µ curie | 1.3 µc/g | 3.6% | 2% |
| | 500 " | 234 " | 10.0 " | 10.7% | 12% |
| Variable acetic anhydride; Reflux 2 hours. | 750 " | 351 " | 23 " | 16.4% | 26% |

(a) 0.47 µc/µl.

(b) Based upon % activity incorporated

Because of competition for the anhydride between the NC hydroxyls and the water present from the NC and $CHIO_4$, the percent acetylation is not a linear function of the added amount of anhydride. Nevertheless, it is obvious that a labeled NC of quite small degrees of acetylation can be prepared with this procedure by proper control of water/anhydride/NC ratios and of anhydride specific activity.

The heterogeneous systems suffer from one drawback, however, namely the probability that the acetate groups are not uniformly attached but are concentrated upon NC molecules in the outer or amorphous regions of the fibers. Experiments have therefore been extended to homogeneous systems, i.e., those in which the NC is dissolved. Acetone was first employed as solvent but this system produced negligible reaction with the NC at varying (but low) levels of acetic anhydride and $HClO_4$, ambient or reflux temperatures, anhydrous or containing moisture from the NC and 70 percent $HClO_4$.

With the thought that this absence of NC reaction might be the consequence of competition with the enol form of acetone, NC solvents were sought in which such enol formation could not occur. In dimethoxyethane, for example--a suprisingly good NC solvent--initial experiments similar to those of Table I did indeed yield incorporation of tritiated acetic anhydride into NC. At present, therefore, this appears to be the system of choice for the NC labeling.

## 2. MECHANICAL BEHAVIOR

### a. Propellant for Macroscopic Studies

- To provide the requisite background data, mechanical property behavior will be measured upon base grain propellant and slurry cast propellant, the latter with various types of NC. This comparison will be made in two formulations, identical except for the use of a "good" plasticizer in one and a "bad" plasticizer in the other. Details of composition and testing were presented in the previous report (Ref. 3).

  The casting powder and the base grain propellant are being prepared by the Naval Ordnance Station (NOS). Casting powder containing 5.1 percent TMETN and 0.6 percent TEGDN has been successfully prepared. However, some difficulties have been encountered by NOS in obtaining good propellant grains from that powder with the desired casting solvent compositions. With the approximately 1/1 TMETN /TEGDN casting solvent, for example, unexpectedly large swelling occurred, necessitating the application of significant pressures during cure; this in turn eliminated the propellant containers submitted by LPC and necessitated the use instead of NOS hardware. With the 9/1 TMETN/TEGDN casting solvent, however, insufficient swelling of the powder occurred to yield a cohesive grain, and it was therefore agreed to substitute a 4/1 TMETN/TEGDN casting solvent. (It is perhaps noteworthy that slurry cast processing of these formulations presents no serious problems.)

- As a consequence of the above, the shipment of propellant, PNC, and HMX from NOS has been delayed and this in turn has caused postponement of slurry cast propellant preparation at LPC.

- As part of the slurry cast physical property comparison, Olin has prepared ten pounds of a ball powder impregnated with approximately 10 percent of a 9/1 TMETN/TEGDN mixture. Five pounds of this impregnated powder were further enzyme-treated by Olin, a process discovered by them to be effective in removing skins. Both these powders have now been received by LPC and will be used in both the "good" and "poor" plasticizer propellant formulations.

b. Low Temperature Transitions

- Low temperature transition measurements in NC gels and propellants have been initiated, using the Differential Scanning Calorimeter (DSC).

  Gel samples were prepared by adding hand-mixed plasticizer/PNC to the DSC sample pans and curing at 60°C. Samples were cooled to -100°C at approximately 5 to 10°C per minute and then scanned upwards at 40°C/minute, using an empty pan as the reference.

  Initial results are shown in Figures 1 and 2 for TEGDN/NC and TMETN/NC respectively. The relatively abrupt changes seen in heat capacity are typical of glass transitions. No other transitions were observed between -100°C and +25°C. Although several studies of glass transitions in NC gels have been reported, (Ref. 4), no data are available for these particular systems for direct comparison. Taken literally, these results lead to the surprising conclusion that it is the plasticizer instead of the NC which possesses the glass transition in this region and thereby limits the low temperature physical capability. Obviously, further experimentation is needed. In particular, it will be of interest to determine whether cooling rates more pertinent for bulk samples will actually yield glass formation in the plasticizers and gels.

c. Microscope-Strain Apparatus

In order to observe the failure process in binders (intermicellar?) and propellants (dewetting?) an apparatus has been constructed which will permit microscopic observations upon thin films while the films are being strained. The apparatus is illustrated in Figures 3 and 4 and consists of a movable stage, Teflon baseplate with sample grips and attached pulley for stress application, plus a metal thermostat chamber. Thus, semi-quantitative values of stress/strain will be obtained as a function of temperature, with simultaneous visual observation, and/or photographic recording.

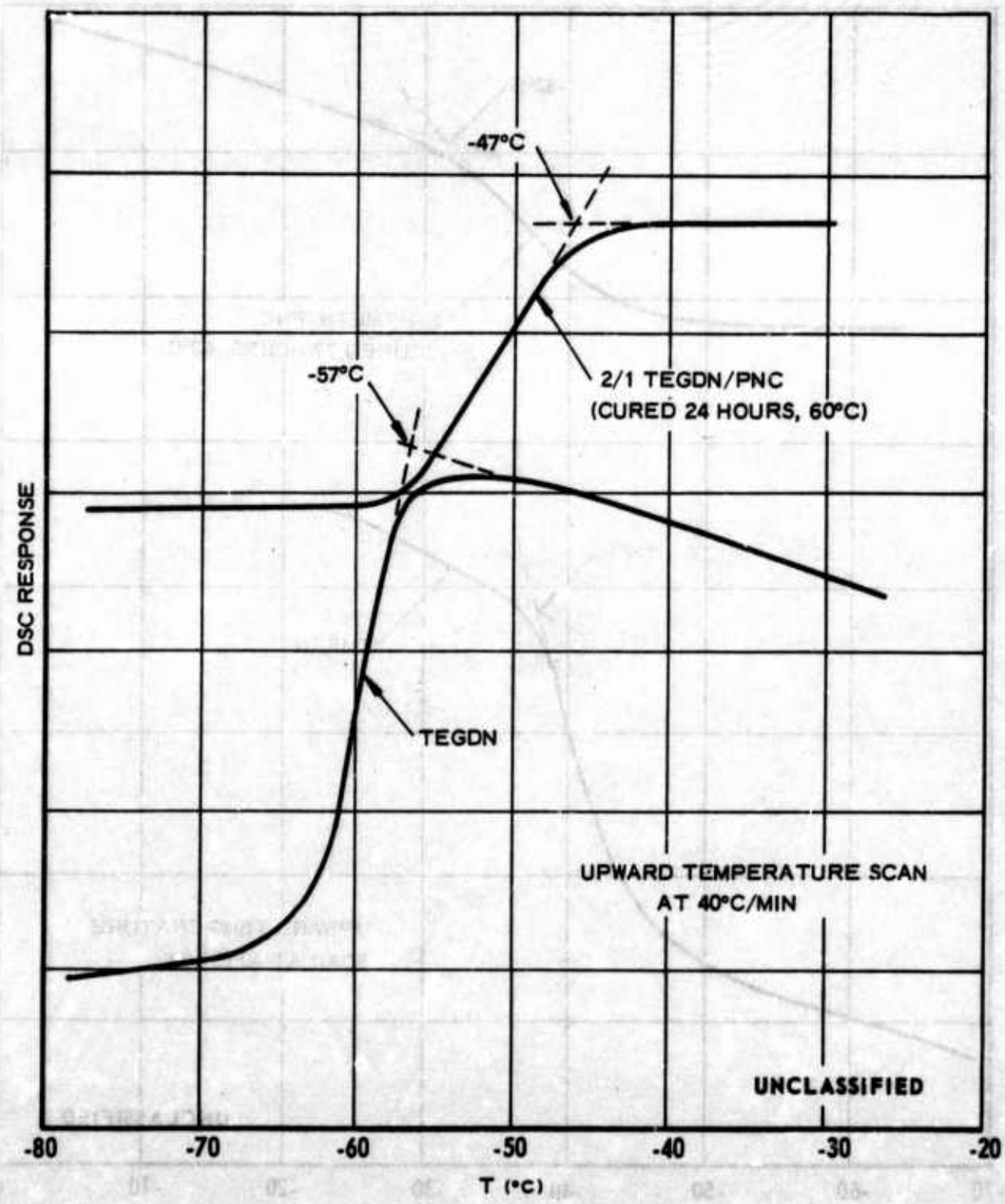


Figure 1 Glass Transition by DSC for TEGDN-NC

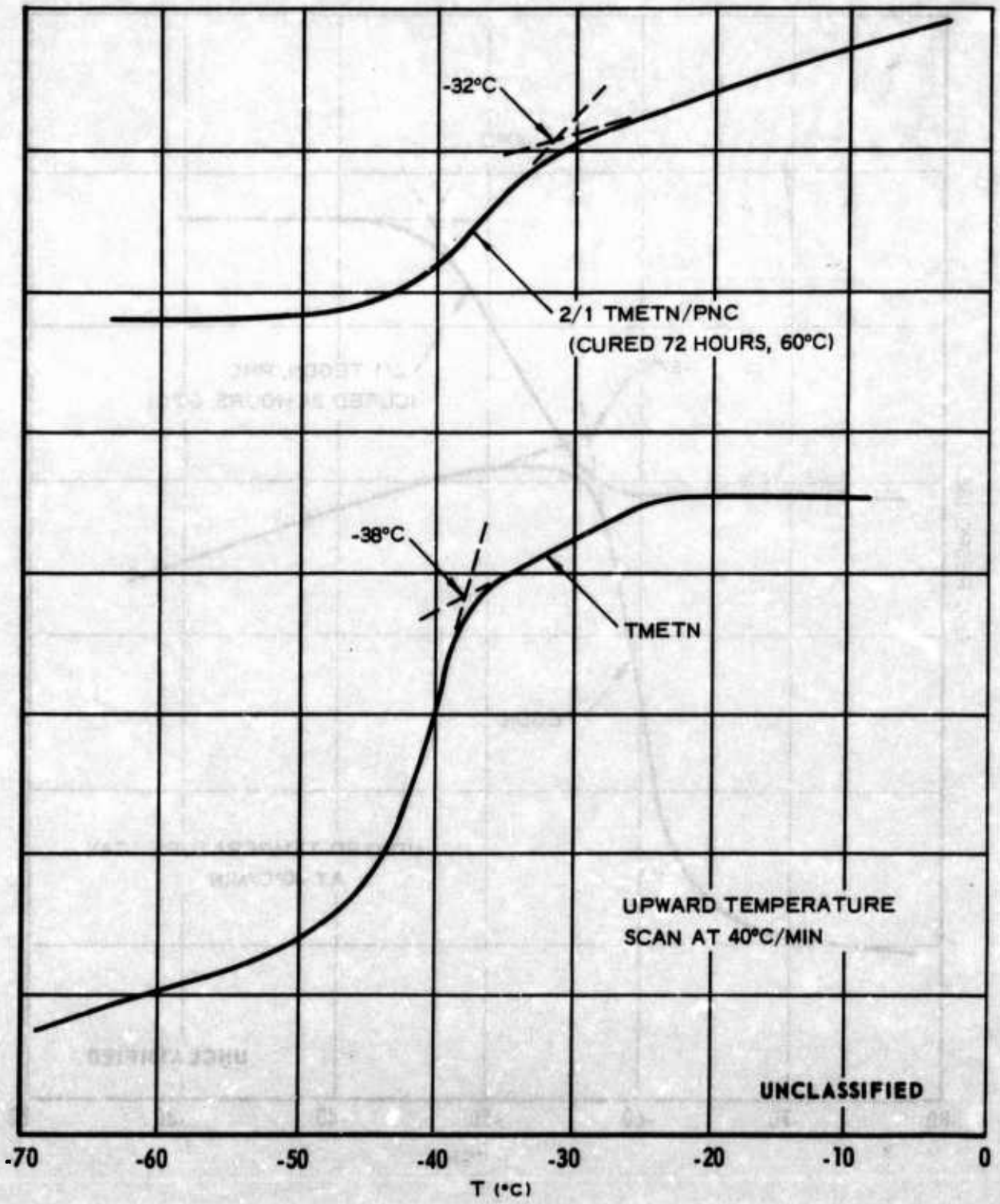


Figure 2 Glass Transition by DSC for TMETN-NC

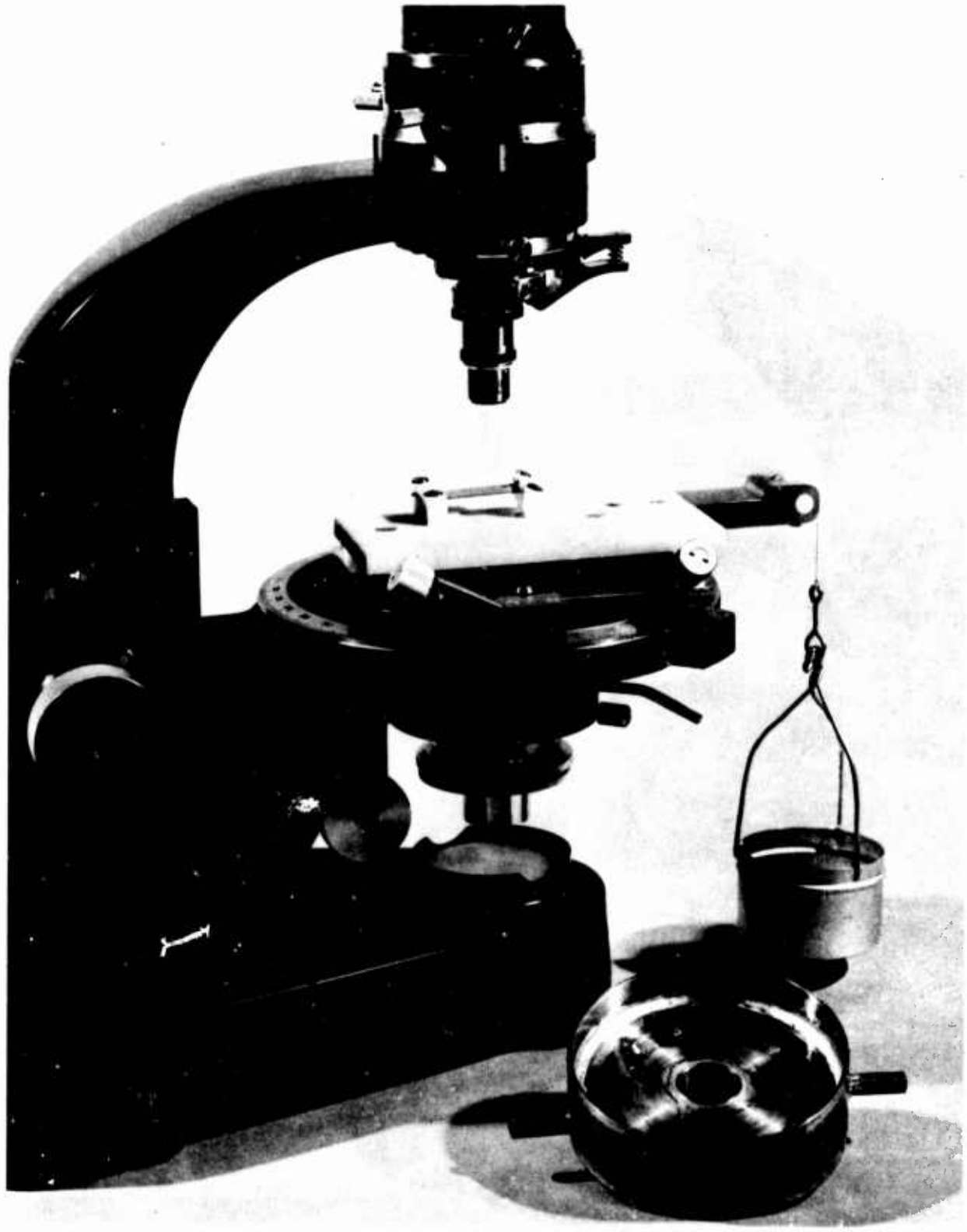

Figure 3 Microscope-Strain Apparatus, Disassembled

Figure 4 Microscope-Strain Apparatus

## 3. FUTURE WORK

- Experiments are continuing in tritium autoradiography. Emphasis now will be upon the examination of sections cut from bulk specimens (5 to 10 g mixes).

- Efforts toward synthesis of labeled ingredients are being held at a low level pending the degree of success obtained in the autoradiographic studies. Positive results in the latter will be followed by a resumption of NC labeling experiments and by synthesis of tritiated crosslinker (polyester caprolactone) and catalyst. The labeled NC will be incorporated by NOS into PNC and casting powder.

- Mechanical and physical property data will be obtained upon base grain and slurry cast propellants containing "good" and "bad" plasticizer compositions. These will be correlated with observed (autoradiographic, microscopic) propellant and binder structures and inhomogeneities and with NC structure and type.

- The study of low temperature transitions in binder/propellant will continue, covering the variables of composition and NC and plasticizer structure. Where desirable, these data will be supplemented by determinations of viscoelastic behavior using a microshear device at varying frequency and temperature (Ref. 5).

- Upon completion of a detailed review of recent literature, which is now in process, that phase of the program dealing with binder/propellant crosslinking will be initiated.

# GLOSSARY

| | |
|---|---|
| DSC | Differential Scanning Calorimeter |
| EGDN | Ethylene glycol dinitrate |
| NC | Nitrocellulose |
| PNC | Plastisol nitrocellulose |
| TEGDN | Triethylene glycol dinitrate |
| TME | Trimethylo lethane |
| TMETN | Trimethylohethane trinitrate |

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5. (a) D. O. Miles, Journal of Applied Physics, 33, 1422 (1962)
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