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TECHNICAL REPORT ARLCD-TR-83004

**EXUDATION TEST FOR TNT EXPLOSIVES UNDER CONFINEMENT:
EXUDATION CONTROL AND PROPOSED STANDARDS**

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FEBRUARY 1983



**US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM															
1. REPORT NUMBER Technical Report ARLCD-TR-83004	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER															
4. TITLE (and Subtitle) EXUDATION TEST FOR TNT EXPLOSIVES UNDER CONFINEMENT: EXUDATION CONTROL AND PROPOSED STANDARDS		5. TYPE OF REPORT & PERIOD COVERED															
		6. PERFORMING ORG. REPORT NUMBER															
7. AUTHOR(s) H.W. Voigt		8. CONTRACT OR GRANT NUMBER(s)															
9. PERFORMING ORGANIZATION NAME AND ADDRESS ARRADCOM, LCWSL Energetic Materials Div (DRDAR-LCE) Dover, NJ 07801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS															
11. CONTROLLING OFFICE NAME AND ADDRESS ARRADCOM, TSD STINFO Div (DRDAR-TSS) Dover, NJ 07801		12. REPORT DATE February 1983															
		13. NUMBER OF PAGES 38															
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified															
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE															
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.																	
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)																	
18. SUPPLEMENTARY NOTES																	
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)																	
<table border="0"> <tr> <td>TNT</td> <td>TNT impurities</td> <td>Confined explosives</td> </tr> <tr> <td>Composition B</td> <td>TNT manufacture</td> <td>Exudation test</td> </tr> <tr> <td>Exudation of TNT</td> <td>Selliting of TNT</td> <td>NATO standardization</td> </tr> <tr> <td>Eutectic of TNT</td> <td>Octol</td> <td></td> </tr> <tr> <td>TNT specification</td> <td>TNT additive system</td> <td></td> </tr> </table>			TNT	TNT impurities	Confined explosives	Composition B	TNT manufacture	Exudation test	Exudation of TNT	Selliting of TNT	NATO standardization	Eutectic of TNT	Octol		TNT specification	TNT additive system	
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<p>The process of exudation of confined explosives containing TNT, and the adverse consequences and interactive effects in munitions is described. A 70°C test for prescribed times to measure the exudate from explosives under confinement is described and has been tested, as amended, for reproducibility by the Prins Maurits Laboratorium, The Netherlands. Since exudation values for TNT of a given set point and of various manufacture vary, three</p> <p style="text-align: right;">(cont)</p>																	

20. ABSTRACT (Cont)

descriptive exudation levels are proposed in adding this test to the TNT specification. Since exudation of TNT can be controlled by existing technology with additives (which also reduce cast cracking), more stringent exudation values are proposed to be added now as requirements to end-use specifications for compositions of TNT such as shell fillers. Concurrently, collection and measurement of exudation from inspected munitions under actual hot field conditions must be done to obtain a direct correlation with this test.

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INTRODUCTION

Trinitrotoluene exudation in shell, bombs, and rockets has been a recurring problem since the early 1920's (refs 1, 2, and 3). As confined munitions are heated under hot field and storage conditions (as high as $\sim 70^{\circ}\text{C}$) TNT expands, and a considerable internal pressure is developed. Since the inner wall of munitions is not normally insulated, the exudate is first formed in the layer of TNT nearest the munition wall. The internal pressure squeezes the exudate from the pores to a region of lower pressure, e.g., the nose of the shell, booster cavity, other cavities, or the hot inner wall. The droplets coalesce and dissolve the TNT, cutting channels through the TNT and the main charge becomes more porous and of lower density (ref 4). With increased gun setback forces, any resulting base separation or cracks in the charge can increase the probability of a premature explosion in the gun when the round is fired.

In a recent study which included four standard Comp B-loaded 105 mm projectiles stored at 71°C for 90 days, certain effects occurred as a result of the exudation. The exudate was pushed onto the threads and into each fuze well cup (4.6 to 10.5 grams). Pressure developed and caused the fuze well cup to be displaced outward $1/4$ inch after this storage, with the space beneath full of exudate. Two rounds also had base separation after the storage (fig. 1) (ref 5).

During the manufacture of 2,4,6-trinitrotoluene (TNT) several intermediates and by-products are formed, including dinitrotoluenes (DNT), asymmetrical TNT's, and 1,3,5-trinitrobenzene. The crude TNT analysis depends on the type of process, batch or continuous. Purification of crude TNT by aqueous sodium sulfite treatment (selliting) reduces the amount of the impurities (table 1). Selliting of molten TNT (in lieu of solid granular TNT) introduces two new impurities. These are hexanitrobibenzyl (HNBB) and methyl pentanitrodiphenylmethane (MPDM) (ref 6).

The various impurities form complex eutectic mixtures with 2,4,6-TNT, which have melting points lower than that of pure TNT. When the melting temperatures of the eutectic mixtures are reached or exceeded, partial liquefaction, extraction, and migration of liquid products from consolidated TNT occurs. Confinement of the TNT (or TNT composition), e.g., in steel or aluminum shell or casing, exacerbates this exudation tendency.

A wide range of exudation levels can occur in military TNT's which meet the requirements of U.S. specification MIL-T-248A, requiring a minimum solidification point (set point) of 80.2°C for TNT. An analysis of the exuded material obtained in 1973 from a U.S. continuous process TNT, having a 80.35°C freezing point, is presented in table 2. The exudate adsorbed on filter paper after 68 hours at 71°C was 1.71% of the original cast weight (versus: 0.5% for a WW II batch process TNT standard). A small scale thin-walled version of the test for confined explosives was used to obtain the exudate (ref 7).

Furthermore, there are important nitration and purification differences in TNT as manufactured by various countries. For example, the U.S. uses a continuous process based on the Swedish Nobel-Chemateur process, as modified by Canadian Industries Ltd, but scaled for larger production. An aqueous sodium sulfite treatment of molten TNT is used for the purification step (ref 8).

The British Royal Ordnance Factory (ROF) continuous process also involves multi-stage countercurrent contacting, but mononitration is conducted separately. The ROF system employs a lower temperature for mononitration, 35 to 42° versus 50 to 55°C for the U.S. process. Also 96% sulfuric acid is used, requiring more staging to effect complete nitration. Low temperature continuous sodium sulfite purification of granulated TNT at 67.5°C is used. A higher yield of purer TNT results. Hexanitrobibenzyl and MPDM are not formed. Two-thirds as much sodium sulfite is used compared to the U.S. purification (ref 8).

In West Germany, mononitration is effected by a continuous process, but di- and trinitration are conducted batchwise. Conditions of processing remove the DNT isomers more completely than is possible with the much shorter reaction time used in continuous operation. During purification, solid granular TNT is contacted with aqueous sodium sulfite in a batchwise procedure. The end product yields a TNT of very high purity, i.e., with a solidification point of 80.65 to 80.80°C (ref 8).

Nitration and purification differences in TNT manufactured in various countries cause exudation levels of specification military grades of TNT to vary considerably. The minimum TNT set point requirement is not sufficient to describe an acceptable standard for exudation to allow proper interchange of TNT between countries. An existing exudation test (fig. 2) for confined explosives containing TNT, simulating worst-case hot field conditions, is needed to describe in the specification exudation levels for TNT as manufactured. The test is also important for assessing and specifying exudation control in TNT-based compositions for use in munitions.

The purpose of this report is to describe a proposed test for exudation of confined explosives, to present test results for some TNT explosives, including modified TNT for exudation control, and to draw conclusions for practical standards.

EXPERIMENTAL - EXUDATION TEST FOR CONFINED EXPLOSIVES

Apparatus

This test (fig. 2) simulates the confinement of a closed shell. Also, the length to diameter ratio of the cast explosive approximates that of typical artillery shell fillers. The paper wrap which absorbs the exudate serves as an insulator between the cast specimen and the heavy walled cylinder, which tends to keep the temperature constant. The hermetically sealed feature assures that gaseous exudation products cannot escape.

The metal parts machined from 7075-T6 aluminum consist of (1) a sleeve of 26.3 mm (1.04 in.) inner diameter and 51.7 mm (2.04 in.) outer diameter, by 127.0 mm (5 in.) length, and (2) top and bottom discs each of 50.8 mm (2 in.) diameter by 12.7 mm (0.5 in.) thickness. The two gaskets are cut from Velumoid or equivalent automotive gasket and are 0.5 mm (0.02 in.) thick. It is recommended that the gaskets be impregnated with silicone grease and well rubbed to remove the excess.

The paper wrap and paper discs are prepared as follows: Whatman No. 1 Qualitative (W. and R. Balston Ltd.) or equivalent is cut into sheets 127 mm by 161.9 mm (5 in. by 6 3/8 in.) and discs 25.4 mm (1 in.) diameter. These are placed in a desiccator at room temperature until constant weight is achieved. One sheet and eight discs are then weighed on a suitable balance to 0.001 gram accuracy for each test sampled.

Preparation of the Cast Explosive Specimen

Test samples are prepared separately by melt casting the composition under investigation into a split, cylindrical brass mold (fig. 3), whose dimensions are 25.4 mm (1.0 in.) inside diameter and 152 mm (6 in.) long. Typically the finished dimensions of samples are 127 mm (5 in.) long by 25.39 mm (0.998 in.) diameter.

For safety, a steam bath and air stirrer are used for melting TNT-based explosives. The molten explosive (150 grams for TNT, 200 grams for filled-TNT compositions) is stirred in a stainless steel beaker, until a creamy consistency of the melt is obtained. Melt pour temperature is 82 to 85°C.

Various methods including Sabel's method (ref 9) are suitable for solidifying the TNT in the split mold, provided that a final high-density cast is obtained. The TNT must crystallize from the bottom to the top, thereby supplementing the shrinkage during solidification. This shrinkage is allowed to occur only in the heated upper zone or "riser."

The upper 100 mm of the empty split mold is insulated with an outer wrap of asbestos. A heated inverted cone-shaped former attached to the top of the split mold has also been found satisfactory. After the complete mold has been preheated at approximately 85°C, it is placed onto a thin flat disc of high density polyethylene, on the surface of a flat stainless steel counter. The molten TNT is immediately poured to nearly fill the mold. After 30 minutes or when the explosive cast temperature is 50°C, heating is switched off and the loaded mold is allowed to cool to room temperature.

The cast TNT charge, removed from the split mold, is slid into the upright sleeve of the test apparatus. A line is scribed around the cast at the top of the sleeve to mark the desired height, where the excess riser portion will be severed. The cut is made by machine or using a jeweler's saw (using a directed stream of water as a coolant), to 126.2 mm to 127.0 mm (4.97 in. to 5 in.) length. The rough cut is smoothed, using emery cloth.

The solid cylinder of explosive is weighed on a suitable balance to an accuracy of 0.01 gram. Density of the specimen is obtained by measuring the average diameter by micrometer and the cast length by dial caliper. The density may also be obtained by weighing the specimen under water (except for TNT explosives containing ammonium nitrate) (ref 9).

Sample Confinement, Heat Storage, and Determination of Exudation

The TNT cast specimen is wrapped tightly with two thicknesses of paper (one sheet) and inserted tightly into the aluminum sleeve. Four paper discs followed by a Velumoid gasket are placed at each open end, and an aluminum disc is bolted on each end to seal.

Two sealed cylinders of the cast explosive to be tested along with a sealed cylinder containing a known standard are placed in a steam-heated chamber at 69.3° to 71°C (157° to 160°F). A continuous recording is made of the chamber temperature. (In the Netherlands and in the Federal Republic of Germany 70°C \pm 0.5°C is the selected test temperature, which will be adopted.)

The periods of time for exudation studies of TNT test specimens were 68 and 136 hours and for RDX or HMX filled batch process TNT, 136 hours and 14 days. In a cumulative exudation study, unmodified RDX-filled continuous process TNT required 1 month to obtain approximately complete exudation. The Prins Maurits study on reproducibility (ref 9), as adopted requires a 160 hour period for TNT and 320 hours for compositions containing TNT.

Test cylinders are then removed from the chamber, insulated, cooled gradually to room temperature, and disassembled. For each test, the complete paper wrap is placed in a desiccator at room temperature until constant weight is reached. The paper is weighed on a suitable balance to 0.001 gram accuracy. The gain in weight is recorded as the amount of exudate. The percent exudation value is obtained by dividing the amount of exudate by the original cast weight. This procedure has been used in exudation studies to date because the paper can be more accurately weighed than the cast charge.

The weight of the cast charge is obtained on a suitable balance to an accuracy of 0.01 gram. The loss in weight from the original cast charge weight divided by the original charge weight is used to confirm the above exudation value.

RESULTS

Reproducibility and Test Duration: Netherlands Study

In a study of the reproducibility of the exudation test for confined explosives conducted in the Netherlands (ref 9), the following results are summarized.

For each of the two grades of analyzed TNT studied, when the filter papers were replaced after 20 and 40 hours, the density of TNT decreased, but not reproducibly. Also, the exudation time of 20 hours was not long enough to obtain reproducible values.

Using a continuous testing of TNT for 160 hours, the maximum exudation level was reached with sufficient reproducibility. Four tests of each TNT lot were tested (table 3).

Continuous testing of Octol (75/25 HMX/TNT) for 320 hours was required for the maximum exudation level to be reached with sufficient reliability. The mean exudation of 3 trials for Octol type TL 97/99 was $0.249\% \pm 0.025$. The exudation values were 0.286, 0.231, and 0.229%.

The magnitude of the exudation value affected the amount of scatter of the test results. The mean absolute scatter of the exudation value increased with the exudation level. The absolute mean scatter of the measurements was found to be of the order of magnitude of about 0.02 to 0.07% for exudation values of 0.3 to 2%.

Testing was conducted in an air-heated oven, where the temperature fluctuated over a range of about 1.5 to 2°C. The temperature variation in the exudation mold (about $\pm 0.1^\circ\text{C}$) was determined by inserting a thermocouple between the filter paper and the explosive charge. The heavy wall design of the aluminum test mold accounts for this small temperature variation.

Exudation Levels of Various Types of TNT and TNT-Compositions

This test has been used experimentally over a period of 25 years to evaluate additives to TNT and TNT compositions, with a goal to reduce exudation as well as cast brittleness. In one study (ref 10), a standard lot of World War II-type batch-process TNT (80.4°C set point) was tested in duplicate at 71°C for 68 hours; the percent exudation values were 0.348 and 0.313%. Recently, a synthetic Composition B (60/40/1 RDX/TNT/Wax), which used a current specification continuous process TNT, was tested in duplicate at 73.8°C (165°F) for 5 days (120 hours); the percent exudation values were 1.395 and 1.375%. In the above studies, polymeric additives were then used to modify the standard TNT or TNT composition, and the exudation values were compared to the standard.

Exudation values obtained for unmodified US standard TNT and TNT compositions are presented in table 4. It is apparent that wide differences in exudation occur in specification TNT from various sources. In particular, note that the continuous process TNT, which employs purification by selling of molten TNT, exudes at a higher level than batch process TNT purified by contacting solid granular TNT.

Effect of Additives on Exudation Values

To control exudation effectively, a small quantity of a polymeric formula is added to the TNT-melt or composition to physically bind the low-melting eutectics within the TNT matrix. The types of polymeric additive systems (used in amounts based on the TNT content of the composition) are: a coreactive two-component liquid, polyurethane-producing system (ref 11), a remeltable version of this polyurethane system which contains nitrocellulose (ref 12), and a plasticized cellulose ester system, which also reduce cast brittleness (refs 10, 13). Fine amorphous silica (less reactive with ammonium nitrate) is used to control exudation of ammonium nitrate/TNT (ref 10).

Notable exudation control is obtainable by employing additive systems to TNT, Comp B, and Octol, based on a stabilizing plasticizer with either low-nitrogen nitrocellulose or cellulose propionate. In Table 5, it is shown that TNT and Octol, based on batch process TNT, were modified to reduce exudation to acceptable levels. In Table 6, using continuous process TNT as the matrix for 60/40 RDX/TNT, a plasticized cellulose ester replaced the wax of Comp B to prevent the otherwise excessive exudation. It is noted that current RDX/TNT formulas of the CW3 type employing 80/20 mononitrotoluene (MNT)/polyvinylnitrate (PVN) increased the exudation level. The MNT plasticizer content adversely affects exudation of TNT. It has also been found that hexanitrostilbene (HNS) (which is useful to impart fine random crystallization of solidified TNT) has no effect on the exudation level.

Cumulative Exudation Tests

The exudation test at 71°C was extended for a 60/40 RDX/TNT composition and for two modified compositions until exudation was essentially completed (fig. 4). The TNT used was a U.S. continuous process specification grade. For the unmodified composition, exudation (1% by weight) was essentially completed in 4 weeks. It was necessary to change the paper containing the adsorbed exudate each week since about 0.3 to 0.35% by weight exudate saturated the paper (fig. 5 and 6).

For the 0.7% D3 additive system based on plasticized modified nitrocellulose, exudation was essentially completed in 8 weeks, slightly below 0.2% by weight exudate. The test was ended at 16 weeks (fig. 7).

For the 1% thermoplastic polyurethane additive (Estane 5702), the exudation curve during the first 8 weeks was similar to that of the D3 additive system. At 8 weeks, an unexpected delayed sharp rise in exudation to 0.6% occurred, not previously detected with the 5 day test. This is attributed to the heat sensitivity of the elastomer with eventual depolymerization at the 71°C test temperature. The TNT cast turned a deep dark-brown color throughout, indicating decomposition.

A cumulative exudation test for 28 days was also conducted on a 66/34 RDX/TNT in which batch process TNT, lot KNK 11-271, was used. Exudation of the unmodified composition, of 0.32%, was essentially completed in 14 days time. This composition modified with 1.1% of a remeltable, polyurethane-producing system had a maximum level of 0.08% exudation in 14 days (ref 12).

DISCUSSION

An exudation test in use for unconfined explosives consists of inducing exudation from the bottom surface of an unconfined solid cylinder of TNT. This "Spot Test" from the Federal Republic of Germany (ref 14) has been studied in Canada (ref 15) and in the Netherlands (ref 16). The test, found to be less reproducible than the test for confined TNT explosives, is more suitable for preliminary laboratory testing and qualitative evaluation of TNT exudation. In

this test a small cast (21 mm diameter x 40 mm height) is placed upright on a filter paper. A 200 gram weight is placed on top of the cast. The assembly is stored for 20 hours at 70°C in an oven. Exudation is determined by measuring the diameter of the resulting spot on the filter paper and also by the loss in weight of the TNT.

In contrast, the Exudation Test for TNT Explosives Under Confinement, as described in this report, was designed to simulate munitions and hot field conditions. The test, along with descriptive levels of exudation, is a necessary addition to the TNT specification for several reasons. As noted, minimum TNT set point of the specification does not insure an acceptable exudation standard for military TNT of various manufacture. More important are the adverse consequences of excessive exudation on the quality of the explosive cast and on the functioning of the munition.

It is important now to correlate exudation values from this test with exudate collected from production lots of TNT compositions loaded in shell, subjected to actual hot field conditions or 70° storage. Sealed shell, sampled and marked, can have the empty fuze well modified by fitting a collector, which may contain adsorbent material to trap the exudate.

There is also no direct correlation of the initial physical quality of the cast shell fillers when inspected with the eventual condition in the field, as influenced by exudation. The cast filler of a shell released to the field or for storage is inspected (by x-ray or other means) and accepted by analyzing the density profiles, pipes, and cracks. These shell should later be sampled, inspected, and compared again after undergoing long-term, warm-field conditions or simulated conditions with a 70°C skin temperature induced.

Within the limits of the inherent properties of TNT, certain interacting improvements to TNT or to composite TNT are being made by additions to the TNT melt matrix. For example, a high-purity TNT is more brittle and subject to cracks than a TNT having more eutectic liquids, which function as plasticizers. However, an exudation control additive comprising plasticized cellulose ester (to physically bind the exudate) introduces additional plasticizer to reduce cast brittleness of either type of TNT (refs 10 and 13).

Concurrently, well-known adverse properties of TNT may be dealt with in a practical manner, by means of a component in the additive system to the TNT melt matrix. For example, the difference of the coefficients of expansion between cast TNT and the steel shell is known. Further, the crystals of TNT have a coefficient of longitudinal expansion along the shortest axis of about 4 times greater than along the longest one. This results in cracks, parallel with the long axis on solidification. The resulting spaces are particularly disadvantageous in the rear part of the shell. The solidification contraction of TNT is another crack-tendency property of TNT. To offset these inherent properties of TNT, Back et al of Sweden have added 0.5% of hexanitrostilbene (HNS) to molten TNT just above its melting point to achieve a solidified TNT of fine random crystalline structure (ref 17). Certain slightly soluble polymeric elastomers added to the TNT melt also make for fine random TNT crystallization. These polymers also increase compressive strength of the TNT-based cast (ref 18).

In munitions, TNT finds use mainly in composite explosives such as RDX/TNT and HMX/TNT. The nitramine suspended in the TNT matrix is slightly soluble in the TNT. Thus, the composite exudate will have some nitramine content. However, the amount of exudation will vary roughly with the amount of TNT present in the composite.

To reduce the hazard of prematures associated with composite TNT, an additional improvement is suggested by Velicky (ref 19). The nitramine suspended in the TNT matrix is precoated by a polymeric protectant, rather than dissolving the polymer in the TNT matrix. In this manner, based on closed bomb studies, the transition from surface burning to deconsolidated burning is delayed, to allow a higher pressure for this transition.

In summary, exudation control, and other desired improvements to TNT and to composite TNT are obtained simultaneously by means of balanced additive systems.

CONCLUSIONS

1. The "Exudation Test for TNT Explosives Under Confinement" is recommended for specification testing.
2. An advisory description of the tested exudation levels should be added to the specification for manufactured TNT as follows:

Type A - Less than 0.4% exudation in 160 hours (6.7 days).

Type B - 0.4% to 0.8% exudation in 160 hours.

Type C - More than 0.8% exudation in 160 hours.

3. For TNT or its compositions destined for use as shell or rocket fillers in munitions, stringent maximum exudation limits are recommended, with appropriate modification of TNT used as required to control exudation. The added requirements recommended for end-use specifications to include TNT and compositions of RDX/TNT, HMX/TNT, and TNT-containing ammonium nitrate or aluminum, all modified as required, are as follows:

Type A - Less than 0.2% exudation in 320 hours (13.3 days):
satisfactory-low exudation.

Type B - 0.2% - 0.4% exudation in 320 hours: satisfactory-moderate
exudation.

Type C - More than 0.4% exudation in 320 hours: excessive exudation.

4. To obtain a direct correlation of the exudation test with actual or simulated hot field conditions, it is recommended that sampled munitions be modified internally to collect and measure exudation from TNT-based cast shell fillers. To obtain interactive effects on cast quality, inspection is recommended before and after the field test by X-raying or by Compton light scattering, to observe relative changes in density profile, cavities, and porosity of the shell filler.

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18. H.W. Voigt, "Castable TNT Composition Containing a Broad Spectrum Preformed Thermoplastic Polyurethane Elastomer Additive," U.S. Patent 4,284,442, 18 August 1981.
19. Rudolph Velicky and J. Hershkowitz, "Anomalous Burning Rate Characteristics of Composition B and TNT," Seventh International Detonation Symposium, Annapolis, MD, June 1981.

Table 1. Impurities present in TNT prepared by continuous nitration and purification^a

Compound	Approximate maximum nominal concentration (%) ^b	
	Crude	Finished
2,4,5-Trinitrotoluene	2.50	0.30
2,3,4-Trinitrotoluene	1.75	0.20
2,3,6-Trinitrotoluene	0.50	0.05
2,3,5-Trinitrotoluene ^c	0.05	0.05
2,6-Dinitrotoluene	0.25	0.25
2,4-Dinitrotoluene	0.50	0.50
2,3-Dinitrotoluene	0.05	0.05
2,5-Dinitrotoluene	0.10	0.10
3,4-Dinitrotoluene	0.10	0.10
3,5-Dinitrotoluene	0.01	0.01
1,3-Dinitrobenzene	0.02	0.02
1,3,5-Trinitrobenzene	0.15	0.10
2,4,6-Trinitrobenzyl alcohol	0.25	0.25
2,4,6-Trinitrobenzaldehyde	0.25	0.25
2,4,6-Trinitrobenzoic acid	0.50	0.05
α -Nitrato-2,4,6-trinitrotoluene	0.10	0.10
Tetranitromethane	0.10	none
2,2'-Dicarboxy-3,3',5,5'-tetranitroazoxybenzene (white compound)	0.35	0.05
2,2',4,4',6,6'-Hexanitrobibenzyl (HNBB)	none	0.40
3-Methyl-2',4,4',6,6'-pentanitrodiphenyl- methane (MPDM)	none	0.40
3,3',5,5'-Tetranitroazoxybenzene	none	0.01

^aData from Radford Army Ammunition Plant, Radford, VA.

^bConcentrations listed are the maximum possible. Actual values will be much lower.

^cThe 3,4,5-isomer has also been identified in crude TNT to the extent of 0.006%.

Table 2. Typical composition of a TNT exudate (main components)

	<u>Percent by weight</u>
2,4 DNT	7.65
2,4,5-TNT	0.67
2,6-DNT	0.98
2,5-DNT	0.19
2,3,4-TNT	1.99
HNBB	0.1-0.5
MPDM	0.1-0.5
2-MNT	0.05
TNB	0.01
2,4,6-TNT	88.19

NOTE: 1. Analysis conducted by gas chromatography.

2. Exudate collected on filter paper from small-scale version of exudation test for confined explosives. In 68 hrs at 71°C, 1.71% by weight of the original continuous process TNT cast, exuded. Under similar conditions, 0.5% by weight of a batch process TNT cast, exuded.

Table 3. Continuous exudation experiments* of 160 hours for TNT

<u>Mixture no.</u>	<u>Initial density grams/mL</u>	<u>Exudation %</u>	<u>Final density grams/mL</u>	<u>Mean exudation %</u>
1	1.612	0.731	1.585	0.713 ± 0.036
	1.613	0.766	1.585	
	1.622	0.692	1.587	
	1.620	0.663	1.586	
2	1.626	0.481	1.597	0.472 ± 0.014
	1.609	0.491	1.586	
	1.606	0.452	1.581	
	1.613	0.463	1.585	

* Performed in air-heated oven (data from ref 9).

Table 4. Amount of exudation from TNT of various processes, including RDX and HMX-filled TNT

	Percent exudation hours at 71°C		
	<u>68</u>	<u>120</u>	<u>136</u>
TNT, Continuous Process, Radford Lot 6-0671 Set Point, 80.37°C	0.670		0.939
TNT, Batch Process, KNK Lot 12-076 Set Point, 80.4°C	0.348 0.313		0.67
TNT, Joliet Lot JA-206	0.547		
TNT, SRI Process, unsellited, Lot TN-4 washed, neutralized	1.08		
TNT, Continuous Process Volunteer Lot 77A 014-407	1.68 (72 hours)		
Cyclotol 60/40 RDX/TNT Lot Rad 6-0671			0.661
Comp B 60/40 RDX, Hol 77G870-001/TNT, Vol 77A014-407 + 1% wax (made from Comp B4 Type 2 Grade A Lot Hol 77G660-001+1% Petrolite wax)		0.662	
Octol 75/25 HMX/TNT, Batch Process, Lot KNK 12-076			0.130
	<u>Hours at 65°C</u>		
	<u>24</u>		
Crude TNT, no purification treatment, washed (U.S. continuous process, Radford Army Ammunition Plant)	5.22		

Table 5. Exudation control by modification of batch process TNT and Octol (ref 10)

	<u>% Exudation, 136 hours, 71°C</u>		
TNT, Lot KNK 12-076	0.670		
TNT + 1.2% plasticized cellulose propionate ^{a,c}	0.066		
TNT + 1.8% plasticized nitrocellulose, RS 1000 sec viscosity ^{b,c}	0.095		
TNT + 0.3% amorphous silica	0.127		
	<u>% Exudation at 71°C</u>		
	<u>6 days</u>	<u>14 days</u>	<u>15 days</u>
Octol	0.130		
Octol + 0.3% plasticized cellulose propionate ^{a,c}			0.058
Octol + 0.45% plasticized nitrocellulose ^{b,c}		0.070	

^a Plasticized cellulose propionate (1.2%) comprises: 0.6 part cellulose propionate (EAP-482-20) powder, 0.25 part epoxy plasticizer, 0.1 part epoxy resin (Epon 828) and 0.25 part acetylated castor oil (USP 3,706,609)

^b Plasticized nitrocellulose (1.8%) comprises: a coprecipitate powder of 0.6 part nitrocellulose (12.1% nitrogen, 1000 RS seconds) and 0.2 part phenoxy resin, 0.5 part epoxy plasticizer, and 0.5 part acetylated castor oil (USP 3,706,609)

^c Cellulose propionate powder is manufactured by Eastman Chemical Products Co.; nitrocellulose is from Hercules, Inc.; epoxy plasticizer (Estynox 308) and acetylated castor oil are from NL Industries, Baker Castor Oil Div.; and Epon 838 is a trademark of Shell Oil Co.

Table 6. Five day exudation values of RDX/TNT compositions^a

	<u>69.2°C (157°F)</u> <u>% exudation</u>
Comp B: 60/40 RDX/TNT + 1% wax ^a	0.599
60/40 RDX/TNT + 1% plasticized cellulose propionate ^b	0.164
Comp B: 60/40 RDX/TNT+ 1% wax ^a	0.510
CW3 Variant: 60/40 RDX/TNT + 0.3% X28M (80/20 MNT/PVN) + 1% beeswax + 0.4% PETN	0.732
	<u>75°C (167°F)</u> <u>% exudation</u>
Comp B: 60/40 RDX/TNT + 1% wax ^a	0.634
CW3: 60/40 RDX/TNT + 0.2% HNS + 1% beeswax + 0.3% X28M (80/20 MNT/PVN)	0.872

^aTNT used was VOL Lot 77A-014-407, a US continuous process specification TNT; RDX used was Class A HOL 77G870-001; wax was Petrolite B2 #11385, ES 670.

^bPlasticized cellulose propionate composition added was 0.6% cellulose propionate, 0.25% epoxy plasticizer, 0.25% acetylated castor oil, and 0.1% Epon 828 epoxy resin.

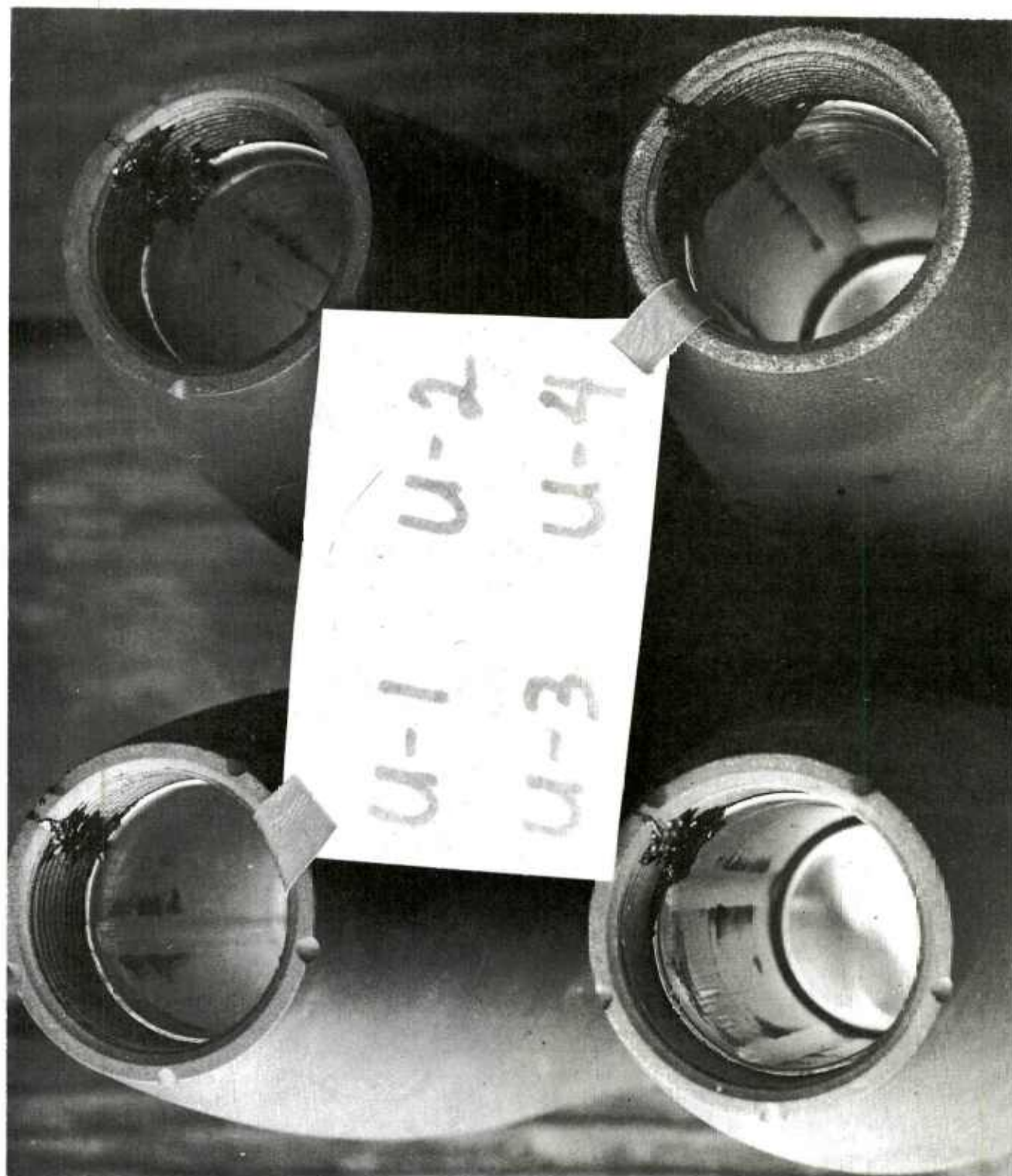


Figure 1. Projectiles loaded with standard Comp B after 90 days storage at 71°C (160°F)

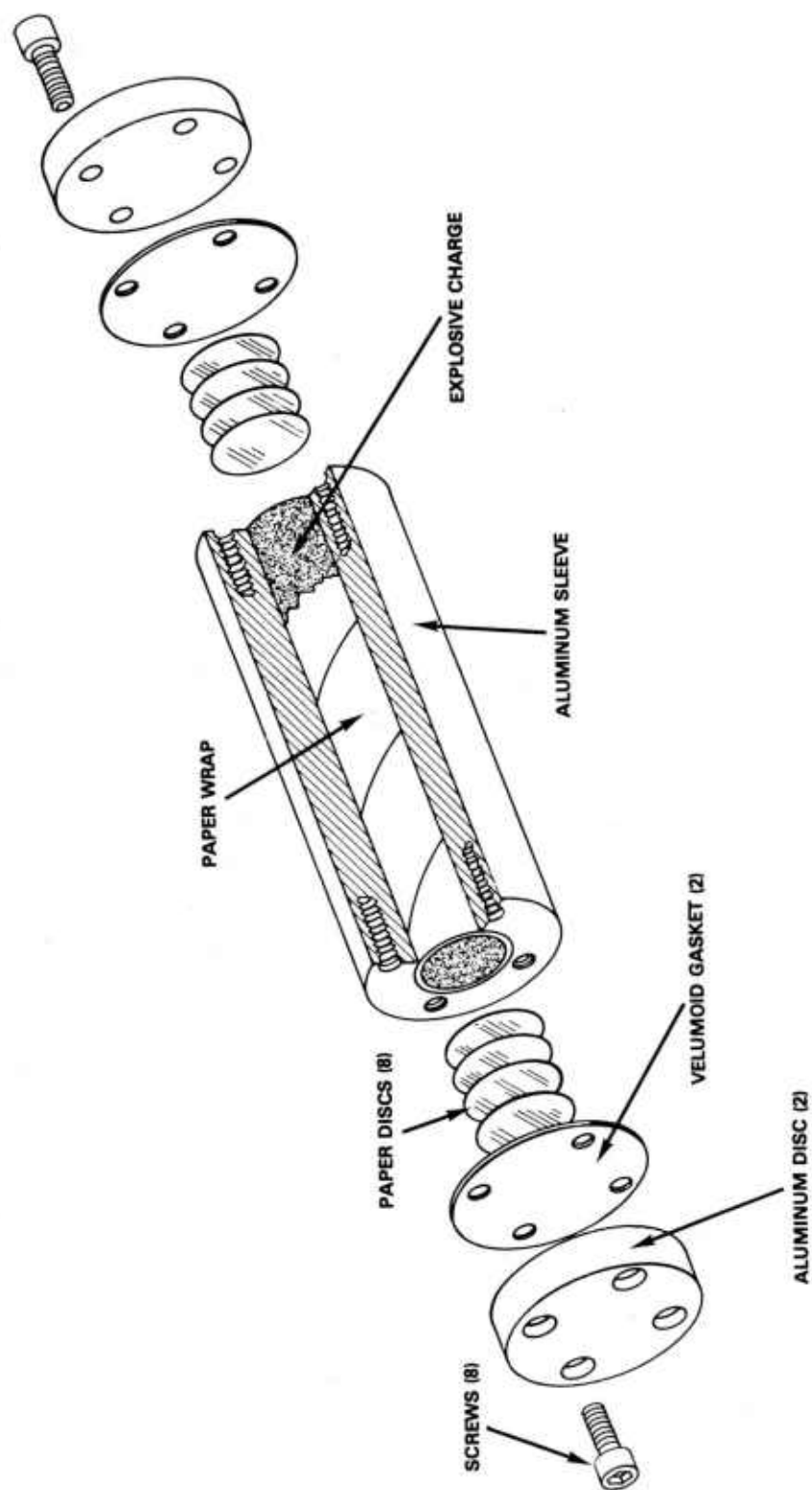


Figure 2. Exudation test assembly

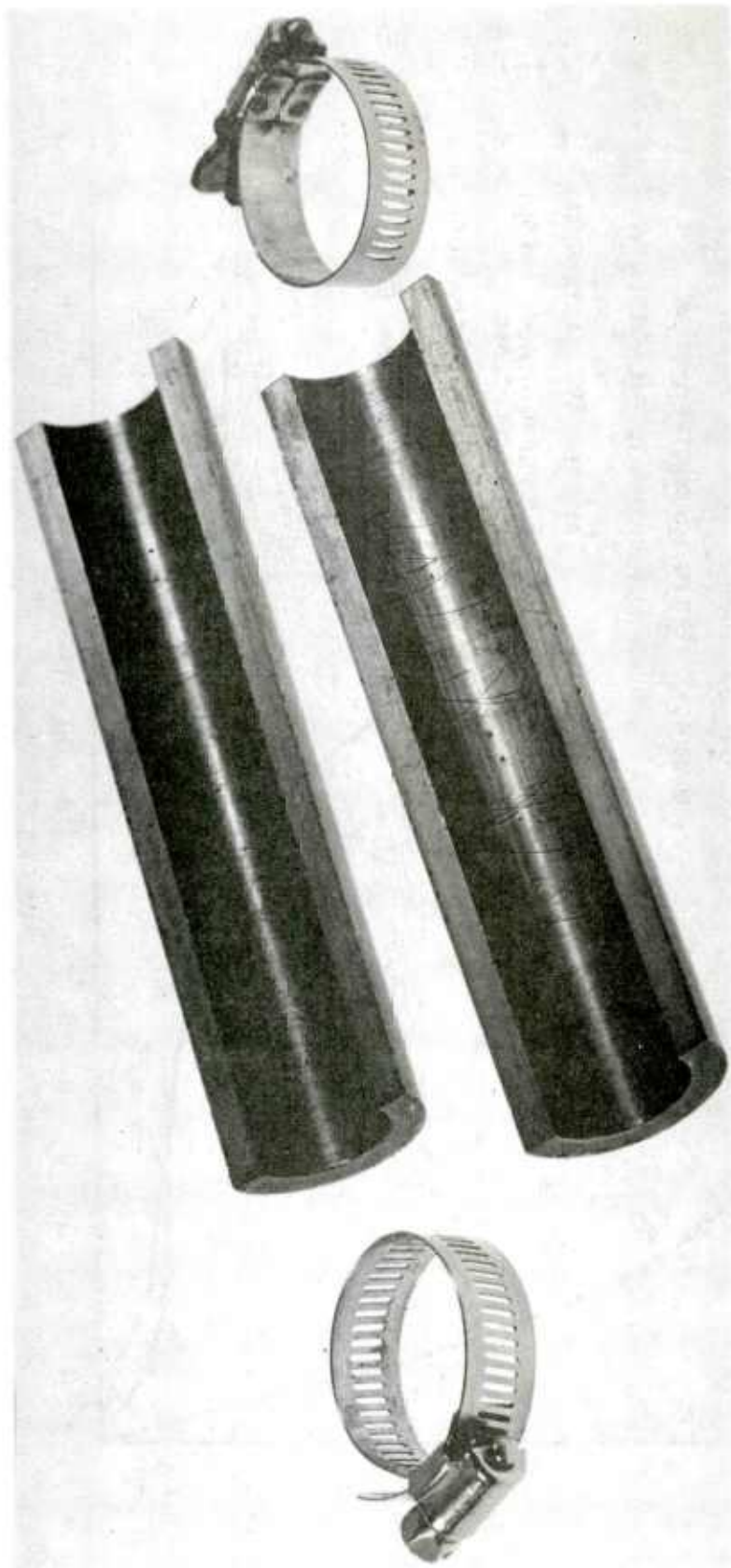


Figure 3. Split mold for preparing casts for exudation test

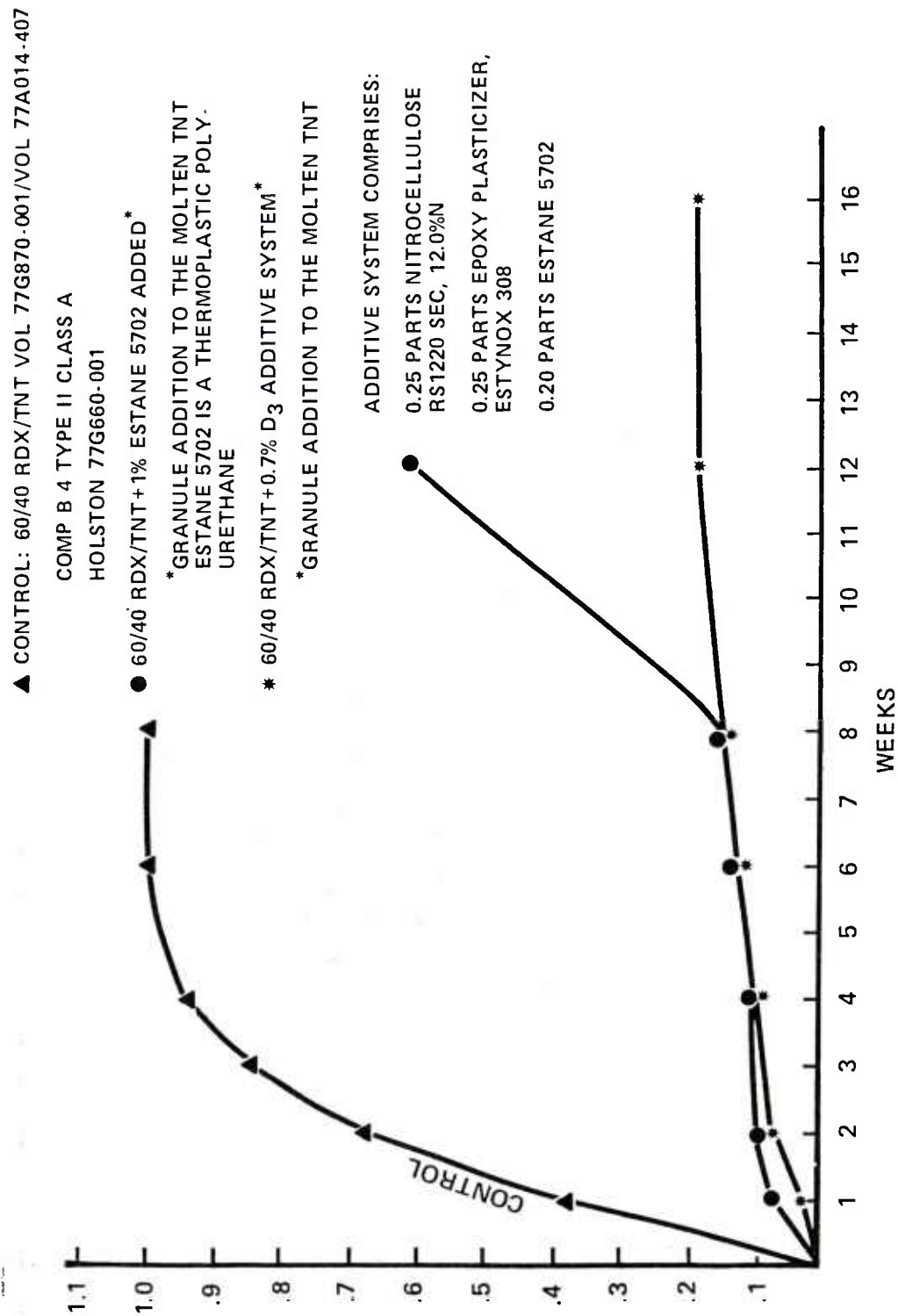


Figure 4. Cumulative exudation at 71°C for 60/40 RDX/continuous process TNT and modifications

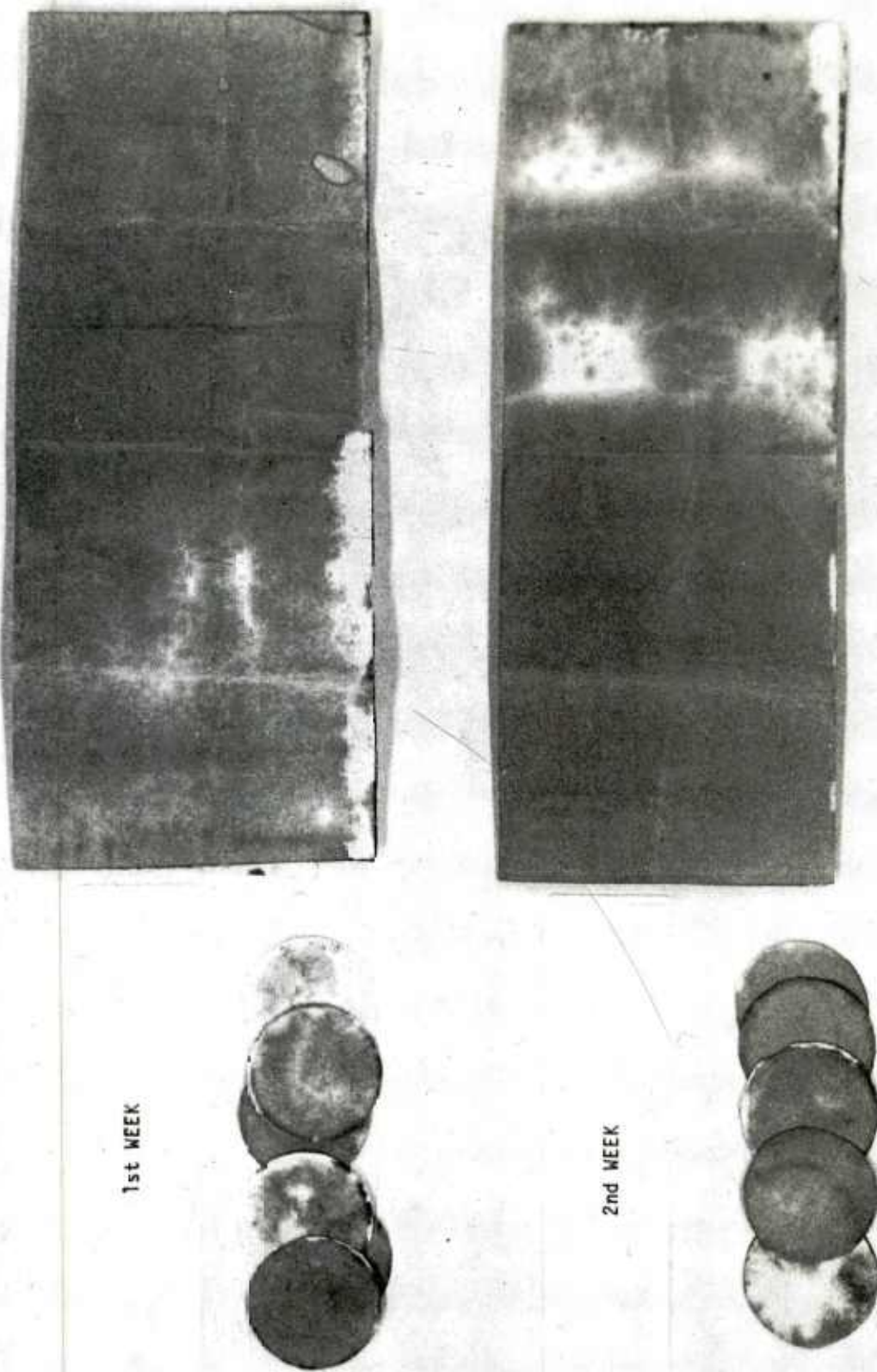


Figure 5. Exudation collected on paper wrap during long term 71°C exudation tests -
Control: 60/40 RDX/TNT (Ho1 77G 660-001) - 1st and 2nd week

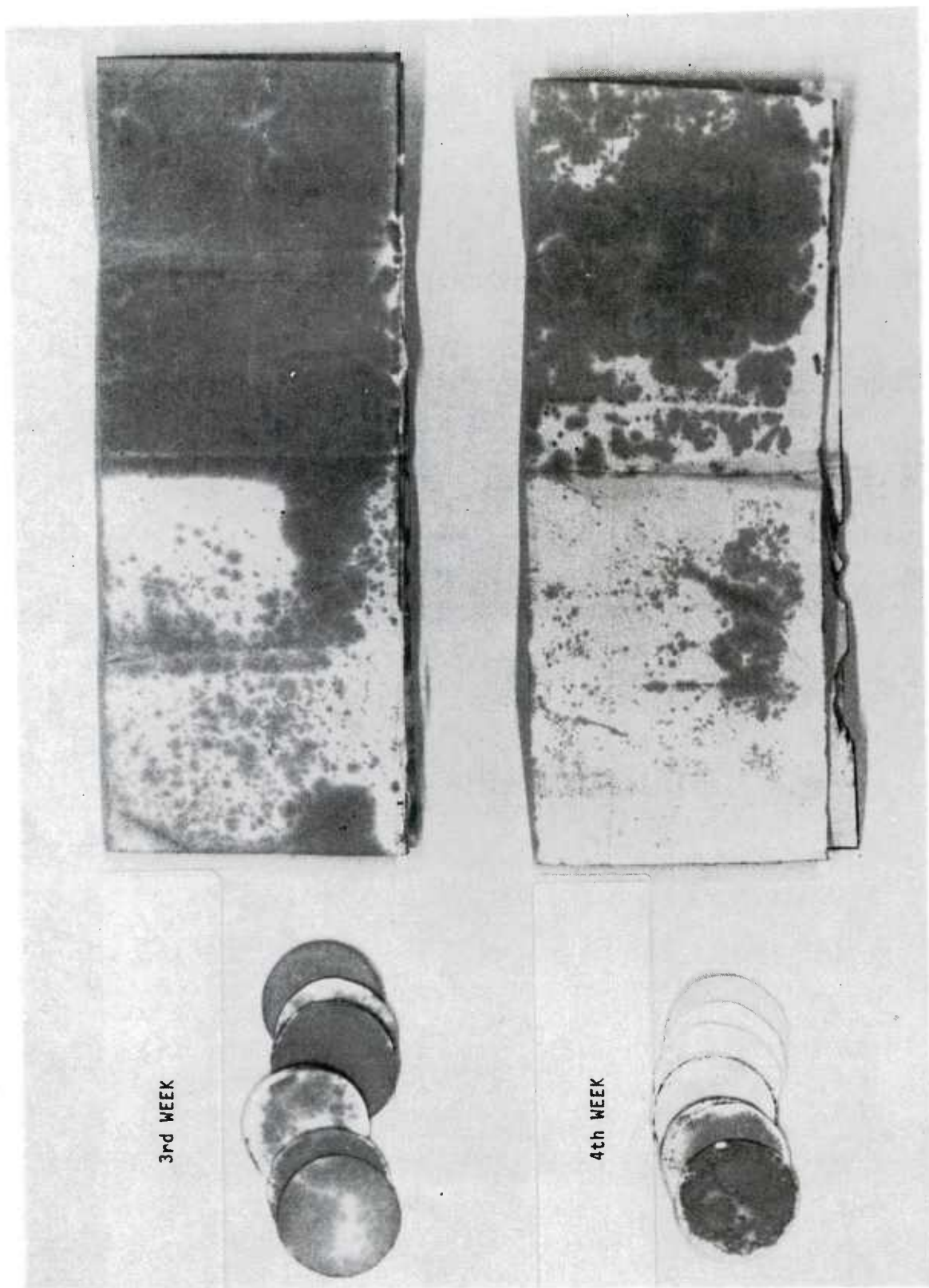


Figure 6. Exudation collected on paper wrap during long term 71°C exudation tests -
Control: 60/40 RDX/TNT (Ho1 77G 660-001) - 3rd and 4th week

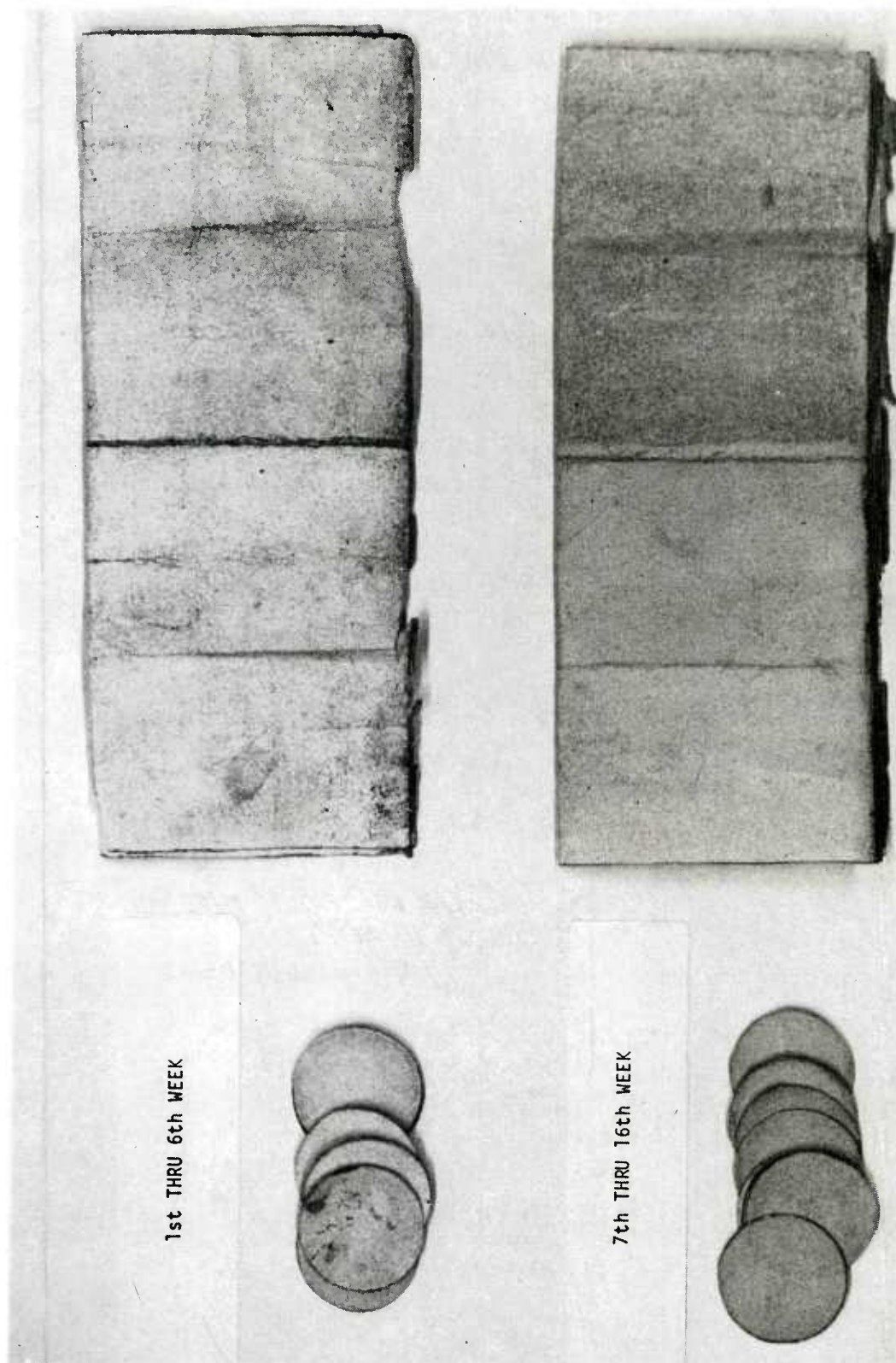


Figure 7. Exudation collected on paper wrap during long-term 71°C exudation tests -
Control: 60/40 RDX/TNT + 0.7% D3 additive system

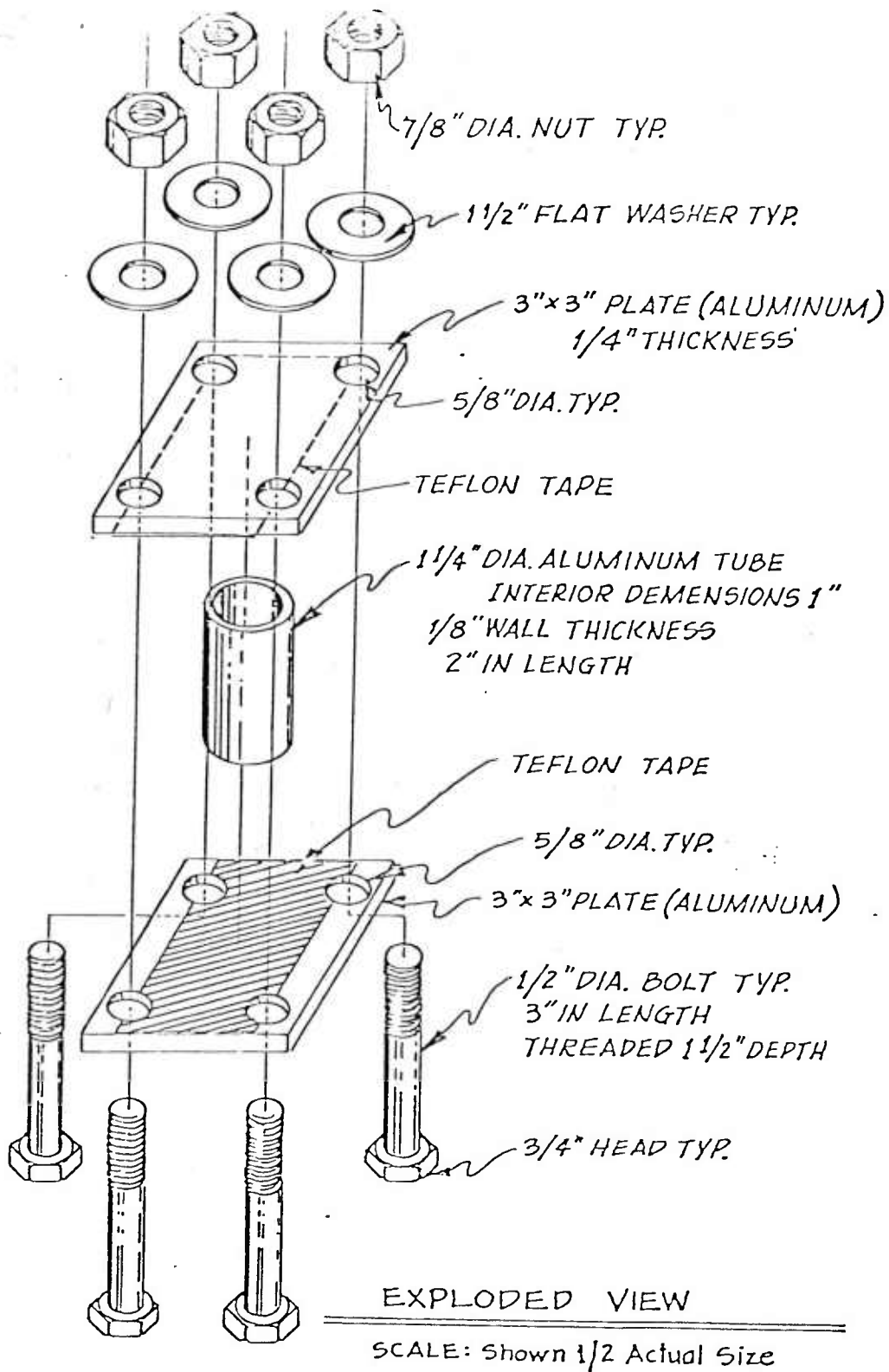


Figure 8. Exudate test fixture: small-scale adaptation

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