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TECHNICAL MEMORANDUM 1101

ESTIMATION  
OF THE  
MOISTURE CONTENT OF M61 IGNITER

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PICATINNY ARSENAL  
DOVER, NEW JERSEY

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Picatinny Arsenal Technical Memorandum No. 1101

ESTIMATION OF THE MOISTURE CONTENT OF M61 IGNITER

By

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November 1962

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## TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1
CONCLUSIONS	1
RECOMMENDATIONS	1
INTRODUCTION	2
RESULTS AND DISCUSSION	3
DESCRIPTION OF METHOD	
A. Specimen	5
B. Special Apparatus	5
C. Special Materials	6
D. Procedure	6
REFERENCES	10
TABLES	
I. Effects of Various Factors on Recovery of Water and Estimation of Moisture Content of Composition	11
FIGURES	
1. Distillation Receiver	12
DISTRIBUTION LIST	

#### ABSTRACT

A procedure has been developed for determining the moisture content of M61 igniter composition. A 5g specimen is boiled for 2 hours with 100 ml. of a water immiscible solvent such as trichlorethylene, and the moisture is collected in a return-solvent type of distillation receiver. The accumulated moisture and solvent are washed into a beaker with methanol and titrated using the Karl Fischer method. A blank determination is performed concurrently and the moisture content is calculated from the net titration. Recoveries of weighed amounts of water are quantitative. Moisture contents of samples of igniter composition are in the range of 0.6 - 0.8%. The procedure also appears applicable to the measurement of small percentages of moisture in many other types of mixtures containing compounds that react with Karl Fischer reagent.

#### CONCLUSIONS

The moisture content of M61 igniter composition may be determined by separating the water from the sample by distillation in an immiscible solvent and titrating the collected water by the Karl Fischer Method.

#### RECOMMENDATIONS

1. The procedure described in this report should be adopted for the determination of the percentage of moisture in M61 igniter composition.
2. An investigation should be made of the applicability of this procedure to the determination of moisture in other compositions where satisfactory methods are not presently available.
3. When the scope of the method has been established it should be included in an appropriate "Military Standard" specification.

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## INTRODUCTION

M61 Igniter Composition serves as a charge for electric igniters and is manufactured according to the formula:

<u>Ingredient</u>	<u>%</u>
Potassium Chlorate	40 ± 2
Lead Thiocyanate	32 ± 2
Activated Charcoal	18 ± 1.5
Lacquer (Egyptian Lacquer Co., (Newark, N.J.))	3-1/3
Butyl Acetate	6-2/3
	} ± 1

The Quality Assurance Division, Picatinny Arsenal, requested that a method for the determination of the moisture content of this composition be developed which would be suitable for incorporation into a specification. Such a method must, needless to say, be expeditious, be reproducible within acceptable limits, require no expensive or highly specialized instrumentation, and be applicable to relatively small specimens since the sensitivity of the material limits its preparation to 50-gram batches, of which a maximum of 10 grams is available for analysis.

The high percentage of butyl acetate and other lacquer solvents ruled out methods based on loss of weight when heated under various conditions (1). The method of distilling in a water immiscible solvent and reading the volume of water collected in a graduated trap was equally unsuited because the limited sample and small percentage of water did not yield a directly measurable volume (2). Moisture determination by the desiccation method (3) would have been slow and subject to interference from the lacquer solvents present.

The Karl Fischer method, since its publication in 1935, has become the most popular procedure for estimating small quantities of water, and has been applied in diverse modifications of technique to a large number of situations, many of which are described in a monograph by Mitchell and Smith (4).

Initial attempts to apply the Karl Fischer Method to the estimation of the moisture content of the M61 igniter composition resulted in failure, apparently because of massive interference on the part of one or more of the components. It was found, however, that the moisture could be separated from the interfering components by distilling the mixture in a water-immiscible solvent and collecting the water in a suitable type of return-solvent distillation receiver, as in the "distillation" method for the moisture content of propellants(2). The Karl Fischer titration solvent (usually methanol) was used to wash the accumulated moisture together with some distillation solvent into the titration beaker. Titration was accomplished by the dead-stop back-titration method in the usual manner. The distillation solvent (usually trichlorethylene) was saturated with water before use. A blank determination, in which an equal quantity of distillation solvent was subjected to a like treatment was also performed, and the resulting titration subtracted from that of the specimen. Because the blank titration is rather high and related to the volumes of distillation and titration solvents, these volumes were closely controlled. The procedure finally developed is described under the heading "DESCRIPTION OF METHOD". Essentially quantitative recoveries were obtained when weighed amounts of water were subjected to the procedure, and acceptable precision resulted when the procedure was applied to a sample of the igniter composition.

#### RESULTS AND DISCUSSION

Table I presents the results of tests to measure both moisture contents of samples of the igniter composition and percentage recoveries of added water. Variations were made in volume of solvent used for distillation, time of distillation, size of sample, and weight of water added. As shown, the tests were performed in three successive groups, A, B, and C.

In Test 1, Group A, the moisture content of a sample was found to be 0.59%, while in Test 2, in the absence of sample, a practically quantitative recovery - 98.2% - of added water was made. In Test 3 the two were performed simultaneously, the water recovery being 103.7% and the moisture content of the sample 0.64%. That the results of Test 3 should show substantial agreement with those of Tests 1 and 2 lends confidence to the proposition that this method may be used to measure the moisture content of the system, whether added as such or as a component of the composition under study.

In calculating the results of this group of tests a run in which no sample was taken nor water added was used as the blank run for Test No. 1 in which 5 grams of sample were used and for Test No. 2, to which 44.0 mg. of water were added. For Test No. 3, in which 5 grams of sample were used and 47.6 mg of water added also, Test No. 1 was used as the blank for water recovery and Test No. 2, adjusted for the difference in weight of water added, as the blank for sample moisture.

Having established generally that moisture might be measured in this manner, it was desired to optimize the details of technique. Group B, Tests 4-7, constitutes a study of the effects on the measured moisture content resulting from variation of the volume of solvent, the time of distillation, and the weight of sample. While this group of tests represents a half-replicate of a  $2^3$  factorial design and may be analyzed as such if desired, it is sufficient to note by inspection that the percentage of moisture does not appear greatly affected by any of the three factors studied. It was concluded, therefore, (a) that 100 ml of solvent should be employed rather than 200 in order to lessen the task of pipetting solvent and to minimize the blank titration, which was found to be greatly dependent on the volume of solvent used, (b) that 2 hours of distillation should surely be sufficient to recover all distillable moisture from the system, and (c) that 5-gram specimens should be taken. (It may be noted that the sample used for Group B was not the same as that for Group A).

Group C constitutes a more extensive study of the recovery of water in the absence of sample, using those details of technique established by the tests of Group B. Increasing amounts of water were added up to a maximum which represents three or four times the amount of moisture ordinarily found in a 5-gram specimen. In each case the recovery closely approximates 100%. The general recovery calculated by the regression method, assuming that 0 mg. of water are recovered when none is added, is 106% (5). This is an altogether satisfactory approach to the ideal value of 100% and demonstrates the validity of the method over the range of moisture content of interest.

It would be expected that the method should have a broad general application, not only to igniter compositions but also to completely dissimilar substances. Its use in situations where presently available methods are not altogether satisfactory will merit study.

## Description of Method

### Specimen

The specimen shall consist of approximately 5 gm of the mixture weighed to within 0.2 mg. In the event that the sample has begun to harden before weighing, it should be rubbed gently through a 20-mesh screen with a rubber stopper.

### Special Apparatus

1. Allihn condensers, 300mm jacket with standard taper 24/40 inner-member joint, such as Kimax no. 18260, protected by drying tubes containing a suitable desiccant.
2. Automatic burettes-two, 25 ml, such as Kimax no. 17138F.
3. Boiling flasks, 250 ml, flat-bottomed, with standard taper 24/40 joint, such as Kimax no. 25055.
4. Dead-stop indication circuit consisting of platinum electrodes in series with microammeter and source of dc voltage variable from 0 to  $1\frac{1}{2}$  volts.
5. Distillation receiver, similar to Figure 1, of borosilicate glass. This item may be conveniently fabricated by attaching a standard taper No. 2 stop-cock such as Kimax no. 41004, to a crank-case dilution receiver ASTM standard D-322 such as Kimax no. 22009, and drawing-out and curving the outward stem to serve as a draw-off spigot, as shown in the figure.
6. Electric hot plate, adjustable, 300 watts per heater, such as Labline no. 5000-2, with triethylene glycol baths on each heater, and with its power supply interconnected with the flow of cooling water to the condensers.
7. Oven, 70°C, suitably heated.
8. Tall-form beakers, 300 ml. such as Kimax no. 14030.

9. Volumetric flasks, 50 ml, with standard taper stopper, such as Kimax no. 28015.

NOTE: The boiling flasks, tall form beakers and volumetric flasks are dried at 70°C and are removed from the oven and cooled to room temperature just prior to use.

#### Special Materials

1. Karl Fischer reagent, stabilized, Fisher Scientific Company No. SO-K-3 or equivalent, stored in an automatic buret the air-inlet of which is protected by a drying tube containing a suitable desiccant.

2. Methanol, anhydrous.

3. Sodium tartrate dihydrate containing 15.66  $\pm$  0.05% of water as determined by the loss of weight when dried at 150°C for 3 hours.

4. Trichlorethylene, Dupont Triclene or equivalent, prepared not longer than 48 hours previous to use by shaking with water in a separatory funnel at least two minutes and left to stand in contact with water at least overnight.

5. Water-in-methanol standard solution, 1 ml of water per 100 ml. of anhydrous methanol, stored in an automatic buret, the air inlet of which is protected by a drying tube containing a suitable desiccant.

#### Procedure

The specimen is weighed and transferred to the dried boiling-flask which is immediately re-stoppered. Another such flask without sample is subjected to the same subsequent treatment and serves as a blank. Just previous to use the condenser and receiver are rinsed with anhydrous methanol which is discarded. (A small residue of methanol in the condenser and receiver does no harm). The stopper is now removed from the flask, the receiver (its stopcock closed) is inserted in its place, and a 100-ml portion of water-saturated trichlorethylene pipetted into the receiver so that column A (Fig. 1) remains substantially filled. The assembly is now placed in the bath and the condenser inserted into the receiver. (In the event that these operations are interrupted, the receiver should be stoppered). The temperature of the bath is adjusted so that the reflux from the condenser is about 2 drops per second and the distillation continued for 2 hours, at the end of which time the bath is allowed to cool.

When condensate no longer drips from the receiver into the flask the contents of the receiver are drawn off through the stopcock into a dried 50-ml volumetric flask. The drying tube is removed and the condenser and receiver washed with a few 5-ml portions of anhydrous methanol which are drained into the volumetric flask. The boiling-flask and receiver are now detached from the condenser and removed from the bath and the receiver further washed with successive portions of methanol which should be sufficient in volume to substantially fill the capillary E (Fig. 1) without causing it to overflow, until the volumetric flask is filled to its mark. The flask is stoppered and mixed by shaking and may be set aside until titrated.

The volumetric flask is emptied into a dried 300-ml "tall-form" beaker containing a magnetic stirring bar, and the flask washed out by filling it once again to the mark with anhydrous methanol, shaking, and pouring it into the first portion. The beaker is quickly mounted on the Karl Fischer titrating stand, covered, the polarized electrodes inserted, stirring commenced, and Karl Fischer reagent added, 1 ml at a time, until the titrated solution remains a distinct red-brown color (not light tan) for at least a minute. The solution is now back-titrated with standard water-in-methanol solution, which is added in a steady stream until the brown color is replaced by yellow, then drop wise until the microammeter reading commences to fall (other indications of a dead-stop end-point, such as an electronray tube with suitable circuitry, may alternatively be used). The percentage of water is calculated as follows:

$$\text{Percent Water} = \frac{[R(V_a - V_b) - (S_a - S_b)]}{10 W} F$$

where:

F = mg. of water per ml. of standard water-in-methanol solution as determined below.

$V_s$  and  $V_b$  = ml. of Karl Fischer reagent added to specimen and blank respectively.

$R$  = ml. of standard water-in-methanol solution per ml. of Karl Fischer reagent as determined below.

$S_s$  and  $S_b$  = ml. of standard water-in-methanol solution for back-titration of specimen and blank respectively.

$W$  = grams of specimen.

To determine  $R$ , a 100-ml portion of anhydrous methanol in a dried 300-ml "tall form" beaker is "neutralized" by titration with Karl Fischer reagent and back-titration with water-in-methanol solution in the manner already described. (These volumes need not be recorded). An accurately measured 5-ml. volume ( $V_r$ ) of Karl Fischer reagent is added to the neutralized methanol and titrated back to the endpoint with standard water-in-methanol solution.  $R$  is then calculated as follows:

$$R = \frac{S_r}{V_r}$$

where:

$V_r$  = ml. of Karl Fischer reagent added (5, as directed).

$S_r$  = ml. of standard water-in-methanol solution required to neutralize  $V_r$ .

To determine  $F$ , an accurately weighed 0.6  $\pm$  0.2 gram portion of sodium tartrate dihydrate is added to a 100-ml volume of anhydrous methanol which has previously been neutralized as described in the foregoing paragraph. When dissolved, the solution is titrated with Karl Fischer reagent and back-titrated with standard water-in-methanol solution in the same manner as the methanol solution resulting from distillation of the sample.  $F$  is calculated in the following manner:

$$F = \frac{156.6 W_f}{V_f R - S_f}$$

where:

$W_f$  = grams of sodium tartrate dihydrate.

$V_f$  = ml of Karl Fischer reagent added to sodium tartrate dihydrate solution.

$S_f$  = ml of standard water-in-methanol solution for back-titration of sodium tartrate dihydrate solution.

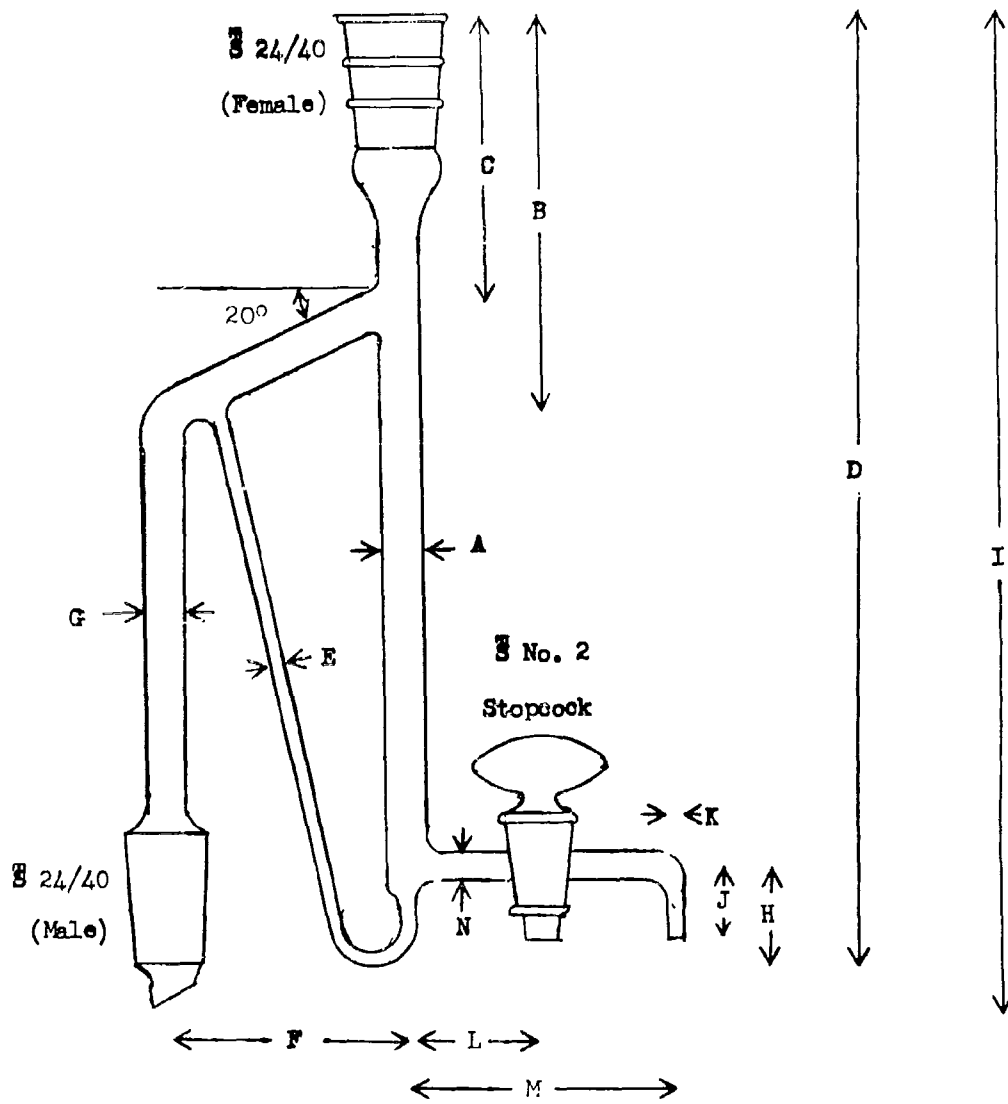
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2. Ibid., Method No. 102.1.2.
3. Ibid., Method No. 101.3.1.
4. J. Mitchell, Jr. and D.M. Smith, "Aquametry" Interscience Publishers, Inc., New York, 1948.
5. C.A. Bennett and N.L. Franklin, "Statistical Analysis in Chemistry and the Chemical Industry", sec. 6.25, John Wiley & Sons, Inc., New York, 1954.

TABLE I  
EFFECTS OF VARIOUS FACTORS ON RECOVERY OF WATER AND MOISTURE CONTENT OF COMPOSITION

Test Group	Test No.	Sample No.	Sample Used, g.	Water Added, mg.	Solvent Used, ml.	Distillation time, hr.	Sample Moisture, %	Water Recovery, %
A	1	I	5	0	200	3	0.59	-
	2	-	0	44.0	200	3	-	98.4
	3	I	5	47.6	200	3	0.64	103.7
B	4	II	5	0	200	3	0.74	-
	5	II	5	0	100	1	0.73	-
	6	II	10	0	100	3	0.74	-
	7	II	10	0	200	1	0.69	-
C	8	-	0	46.6	100	2	-	104.7
	9	-	0	84.8	100	2	-	96.9
	10	-	0	123.6	100	2	-	110.4

Fig. 1 - DISTILLATION RECEIVER



A...10.7-11.3 mm	D...280-300 mm	H...22-27 mm	L...35-45 mm
B...122-127 mm	E...4-5 mm I. D.	I...300-320 mm	M...75-85 mm
C...87-92 mm	F...65-75 mm	J...20-25 mm	N...7-9 mm O. D.
G...11-15 mm I. D.	K...1.0-1.4 mm		

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A procedure has been developed for determining the moisture content of M61 igniter composition. A 5g specimen is boiled for 2 hours with 100 ml. of a water-immiscible solvent such as trichlorethylene, and the moisture is collected in a return-solvent type of distillation receiver. The accumulated moisture and solvent are washed into a beaker with methanol and titrated by the Karl Fischer method. A

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2. Water-Determination

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- III. Karl Fischer method

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