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TECHNICAL REPORT 3911

A RADIOACTIVE TRACER METHOD FOR THE
INSPECTION OF THE DELAY ELEMENT IN
M200 SERIES HAND GRENADE FUZES



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SEPTEMBER 1969

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Technical Report 3911

A RADIOACTIVE TRACER METHOD FOR THE INSPECTION OF
THE DELAY ELEMENT IN M200 SERIES HAND GRENADE FUZES

by

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September 1969

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Feltman Research Laboratories
Picatinny Arsenal
Dover, New Jersey

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ABSTRACT

Incorporation of a radioactive tracer within the delay composition provides a highly reliable method for the inspection of the delay column height in M200 series hand grenade fuzes. The resulting gamma-ray signal from each fuze gives a quantitative measure of total delay charge weight. Based on measurements with plant-size batches of delay composition, levels of radioactivity can be such to accommodate an inspection rate of two units per second, with a single inspection unit, without exceeding permissible external radiation dose levels. The inspection unit itself would consist of a simple, rugged, inexpensive scintillation-counter assembly.

The radioactive tracer element need not be common to the delay composition ingredients but can be added to the mixture as a foreign material in ppm concentration. Thus a wide range of radiation half-lives and gamma-ray energies can be used. The radiological safety factors associated with the handling of bulk quantities of radioactive materials in an ammunition loading plant are discussed briefly.

INTRODUCTION

In the manufacture and quality control of M200 series hand grenade fuzes, the proper length or total weight of the delay charge element is critical. The delay charge is normally loaded into the delay housing (Fig 1) by a series of incremental fillings and compactions performed either manually or automatically. The current method of inspection consists of an x-ray transmission technique in which a fractional length of the total delay column is viewed by a collimated beam of x-rays. A cadmium sulfide detector gives a measure of the relative density of the delay charge. This method, however, does not provide the necessary degree of quality control because it does not provide for the inspection of the entire delay column. A fuze containing one or more missing increments not located in the direct path of the x-ray beam can therefore pass the inspection process. A more reliable method is therefore required, preferably one that includes inspection of the entire delay column or for total charge weight of delay composition.

At the request of the Quality Assurance Division of the Army Munitions Command (Ref 1), a feasibility study was undertaken to perform this inspection by the incorporation of a radioactive tracer within the delay composition. The approach here is based on the simple assumption that if the delay composition is uniformly radioactive, the intensity of a gamma-ray signal from each delay column would be a direct measure of total charge weight. The objective, of course, would be to use the smallest quantity of radioactivity per item necessary for a given inspection rate, to minimize radiation hazards.

This report describes the results of some experiments on the use of the radioactive tracer method for the inspection of the total delay element in M200 series hand grenade fuzes. Type I zirconium-nickel alloy delay composition (Ref 2) having the formulation 60/14/26 BaCrO₄/KClO₄/70-30 Zr-Ni was used for these experiments. Two different radioactive tracers, 27-day chromium-51 and 2.1-year cesium-134, were employed as radioactive tracers. The first was added to the composition in the form of BaCr⁵¹O₄ as a fractional percentage of total barium chromate and the second tracer was included as C_s¹³⁴ClO₄ in ppm concentration of the total mixture. Two batches of delay composition were prepared with each tracer, one being a 2000-gram laboratory run and the other a 50-lb plant batch to simulate actual production quantities. From each batch, one-

hundred M204A2 hand grenade delay elements were prepared by hand loading to various column heights (or total charge weights). A correlation was then observed between column height, radiation signal, and burning time.

Estimation of Required Radioactivity Level
For a Given Inspection Rate

The amount of radioactive tracer required per fuze for any desired inspection rate and accuracy can be estimated from nuclear counting statistics. An arbitrary inspection rate of one unit per second is assumed, with the additional requirement that the charge weight be determined to $\pm 5\%$ (standard error). This accuracy is more than sufficient to detect one missing increment out of the four normally used to fill delay columns.

The standard deviation obtained in N total counts is $\pm\sqrt{N}$ and % error = $\pm\sqrt{N}/N \times 100 = \pm 100/\sqrt{N}$. For a 5% error, $\sqrt{N} = 20$ and $N = 400$. Therefore, 400 counts are required from each radioactive fuze. In one second interval, it is assumed that half the time will be required for counting and the other half for decision making (pass or fail). Therefore, the required count rate will be 800 counts per sec.

The amount (in microcuries) of radioactive element per fuze to give the required count rate of 800 counts per sec is estimated as follows:

$$R = \frac{800}{GFASK}$$

where

R = no. of microcuries of radioactive tracer per fuze.

G = no. of gamma rays emitted per disintegration of each radioactive atom.

F = efficiency of detector for sensing gamma rays from the radioactive tracer.

A = attenuation factor of fuze body housing for gamma rays emitted by the tracer.

S = self-shielding factor of delay column for gamma rays emitted by the tracer.

K = constant for disintegrations per sec per microcurie =
3.7 x 10⁴.

The value for G is obtained from the decay scheme of the particular radioactive atom. F is determined by experiment with known gamma emitters for a particular detector.

The attenuation factor A is calculated from $A = e^{-\mu\rho t}$, where μ is the mass absorption coefficient of the delay column housing in cm²/g, ρ is the density of the housing, and t is the thickness through which the radiation passes. The appropriate value for μ is obtained from the composition of the absorbing material and the specific gamma-ray energy. The self-absorption factor, S, can be estimated from the composition of the delay powder, its physical geometry, and the gamma-ray energy by the method of Rockwell (Ref 3).

Values for G, F, A, S, and the calculated values for R for both chromium-51 and cesium-134 are given in Table 1. Also given are the half-lives and gamma-ray energies for each radioactive tracer. Thus, for an inspection rate of one unit per sec, 0.62 microcurie of chromium-51 and 0.022 microcurie of cesium-134 are required.

EXPERIMENTAL

Delay Composition Blending Procedures

Four batches of delay powder were prepared during this program, as detailed in Table 2. Preparation of these batches was accomplished using conventional wet blending techniques, with ethyl alcohol being used as the mixing medium. All blends were granulated through a 16-mesh screen and then air dried and oven dried at 105°C. Radioactive uniformity was then established for each of the prepared batches using the dry granular material. This was determined by randomly removing 10 one-gram aliquots and ascertaining that the gamma-ray count rate per unit weight agreed to within ± 1 standard deviation. For the two smaller blends it was found that a longer mulling time and a somewhat "soupy" mix was required to bring about a homogeneous distribution of the tagged material throughout the batch of delay powder. No change in the procedure was required in the large scale "plant" preparation of the tagged delay composition.

Two methods were employed to introduce the required amounts of active material into the blends. For the smaller batches a premix of the tagged material and appropriate oxidant was first prepared and, after establishing radioactive uniformity, was then mixed with the fuel and second oxidant. For the larger batches a 1000-gram

premix of the tagged material and appropriate oxidant was prepared. After establishing radioactive uniformity, this premix was mixed with the balance of that oxidant, the fuel and the second oxidant.

Delay Composition Loading Procedures

Loading of the M204A2 hand grenade delay elements was performed in accordance with the drawing requirements (Ref 4) except that no sealant was applied around the M42 primer assembly or over the staking areas. The fully loaded column required 1.333 grams of delay powder pressed as four 333-milligram increments at approximately 20,000 psi consolidating pressure. Increment weights and the number of increments were chosen to give column heights between 70 and 100 percent of maximum. The correspondence between charge weight and percent of maximum column height is indicated in Tables 4-7. To ensure accuracy of composition transfer to the fuze bodies, column heights were measured for all items loaded. One hundred elements were loaded for each of the four delay composition batches. It should be noted that no detonator or igniter assemblies were connected to the fuze bodies.

Determination of Fuze Burning Times

Burning times for all loaded fuzes were determined by an instrumented measurement of the interval between primer initiation and terminal incandescence of the delay column. Counter starting occurs at the moment of firing pin impact against the primer and stops upon photocell response to light output at the termination of delay column burning. Thirty replicate trials were conducted for items loaded to 100 percent of maximum column height, ten replicate trials for items having column heights of 95, 90, 85, 80, and 70 percent of maximum, and twenty replicate trials for items having column heights of 75 percent of maximum. Subsequent to testing, all items were inspected for gas leaks around the primer assembly. All trials were conducted under conditions of room temperature and atmospheric pressure.

Preparation of Radioactive Tracers

Table 3 is a summary of the quantities of tagged ingredients and radioactivity levels used in each of the four batches of delay composition. For these particular experiments, the level of radioactivity per delay fuze was approximately half of that required for an inspection rate of 1 item per second.

Radioactive barium chromate was prepared by stoichiometric precipitation from a 0.1N HCl solution of chromium-51 tagged sodium

chromate by the addition of excess barium chloride solution.

Cesium-134 was introduced into the delay composition by inclusion as a "doping agent" in an aliquot of potassium perchlorate. The indicated quantity of KClO_4 (see Table) was dissolved in hot water and the appropriate amount of radioactive cesium was added as the chloride using a fraction of a ml of aqueous solution. The solution was continuously stirred and heated until most of the water was volatilized off. The resulting precipitate was then treated with methanol and again stirred while the alcohol was evaporated in a stream of air. The cesium-134 "doped" KClO_4 was dried in an oven at 100°C and then tested for radioactive uniformity by counting a number of weighed random aliquots.

Gamma Radioactivity Counting Procedure

All gamma radioactivity measurements on fuzes loaded with tagged delay composition were made with a 3" x 3" sodium iodide well crystal integral line scintillation counting assembly. The detector was operated at 1000 volts from an external power supply, and pulses from the detector were fed to a nuclear counting system consisting of a linear amplifier and a scaler-timer. All gamma rays emitted by the sample above a preset noise threshold level were counted.

DISCUSSION OF RESULTS

The correspondence between both burning time and percent of maximum column height versus radioactivity, as count rate, of the tagged delay composition is shown for items loaded with each of the four batches in Tables 4-7. This same data is also shown graphically in Figures 2-6. The usefulness of having an internal radioactive source to serve as an indicator of loading accuracy and, derivatively, burning time is demonstrated by these plots. Reasonably good linearity is shown for all relationships between counts per minute and percent of maximum column height. The plots of burning time versus counts per minute are generally nonlinear at the higher count rate levels (column heights) due to effects occurring in an obturated system where pressurization of the column occurs. At the lower count rate levels, corresponding to column heights between 70 and 80 percent of maximum, the plots are sufficiently linear to demonstrate a clear and unambiguous relationship between burning time and activity.

Mention should be made of the batch-to-batch lack of reproducibility of burning time. Although not detracting from the demonstrated correspondence between column height and/or burning time with activity necessary for delay fuze manufacture control purposes, some explanation should be supplied for this wide variation in performance.

Two factors can be considered. The first is that 70-30 zirconium-nickel alloy of different lots was used in the program, material from one lot having been used for the small-scale batches and material from another lot having been used for the large-scale batches. The second factor contributing to batch-to-batch irreproducibility of burning time is the batch size itself. It has been shown in other work, involving the preparation of tungsten-barium chromate-potassium perchlorate delay compositions, that the large-scale batches generally have faster burning rates than those obtained for identically formulated smaller scale batches. It has been suggested that for the large-scale batches the longer blending times and consequently greater amount of work being done on the constituent materials results in a shift in particle size distribution of these ingredients, notably the fuel, with an attending speed-up in burning rate for the final blend.

The most important observation from this data is that with each of the four batches of tagged delay composition, there was no significant change in measured burning time of loaded fuzes, until at least 20% of the column height (or charge weight) was missing. The count rate, however, decreased essentially linearly with column height. Therefore, in an automatic inspection process, with radioactive delay composition, the pass or fail criterion will depend on a measured difference in count rate of 20%. If we double the inspection safety factor, this pass or fail criterion can be set at 10% count rate difference. Thus, for an inspection speed of one unit per second, wherein a fully loaded delay column gives a count rate of 800 per second, a count rate of 720 per second or less will cause rejection.

A secondary advantage of this technique is that by virtue of the radioactivity of the mixture the uniformity of composition can be easily checked before loading, by the random counting of a number of aliquots. Thus, loading of fuzes need not be undertaken until such uniformity is first established.

RADIOLOGICAL SAFETY CONSIDERATIONS

The successful implementation of a radioactive tracer method for the inspection of M200 series hand grenade fuzes will be very much dependent on the degree of radiological safety practices to be imposed on a contractor-operated loading plant. External radiation levels obtained from the mixing operation of 30-lb batches of delay composition, for both chromium-51 and cesium-134 tagged mixes, are shown in Table 8. In this operation, where the total radioactive material is concentrated in one location, the highest radiation levels will be encountered. These measured levels are well within the accepted minimum permissible value of 2.5 millirems per hour for radiation workers and are sufficiently low so that the radioactivity concentration can be safely doubled to allow for an inspection rate of two units per second. These levels are also of the same order of magnitude as obtained from the use of x-ray and nuclear inspection gages. Thus, with respect to external radiation, the radiotracer method presents no special safety problem.

Another matter which must also be considered is the problem associated with exposure to loose radioactive delay powder. This would entail hazards from breathing of radioactive dusts and accidental internal ingestion of loose material deposited on clothes, skin, and hands. Such internal contamination could be compounded in the event of an explosion or fire involving appreciable quantities of even slightly radioactive loose material.

The above hazards can be adequately controlled by adherence to good housekeeping, wearing of gloves and dust masks, and strict control of the inventory, storage, and transport of radioactive material. There are numerous industrial processes involving other toxic materials where such procedures are common practice. It is anticipated, however, that many ammunition loading plant managers will be reluctant to adopt such practices because of the possible slowing down of production rates. Another consideration is the fear of radiation and the lack of knowledge about its properties that still prevails among plant personnel. The use of radioactive materials in an industrial process would also require appropriate licensing and periodic inspection by both Federal and State Regulation agencies. All of these factors will hinder the wholesale acceptance of a radioactive tracer method for an industrial inspection process. Nevertheless, the use of this particular approach should certainly be considered when other techniques fail to give the required degree of reliability.

REFERENCES

1. Ltr dated 14 March 1967 from Hdqts, US Army MUCOM to CO, Picatinny Arsenal, ATTN: SMUPA-D; signed by Wesley J. Thomas, Director of Quality Assurance.
2. Specification MIL-C-13739, November 1954, covering zirconium-nickel alloy delay compositions of three types for use in delay elements of fuzes.
3. Theodore Rockwell III, Editor, Reactor Shielding Design Manual, McGraw-Hill Book Co. Inc., 1956.
4. Drawing No. 7548570, February 1968. Fuze, grenade, Hand, M204A2, M206A2S. Fuze, grenade, Hand, Practice, M205A2 assembly.

TABLE 1

Factors for estimation of amount of tracer per fuze
for an inspection rate of one unit per second

| <u>Radioactive Tracer</u> | <u>Half- Life</u> | <u>E_{γ}, Mev</u> | <u>G</u> | <u>F^b</u> | <u>S</u> | <u>A</u> | <u>R</u> |
|-------------------------------|-----------------------|-------------------------------------|----------|-------------------------|----------|----------|----------|
| Chromium-51 | 27.8 d | 0.32 | 0.09 | 0.5 | 0.92 | 0.85 | 0.62 |
| Cesium-134 | 2.1 y | 0.75 ^a | 2.26 | 0.5 | 0.95 | 0.90 | 0.022 |

^a Average of 6 gamma rays.

^b Assuming that each fuze is counted between two 2" x 2" sodium iodide scintillation crystals.

TABLE 2

Blending information for batches of type I delay composition

| <u>Blend No.</u> | <u>Emitting Species</u> | <u>Batch Size</u> | <u>Method of Preparation</u> |
|------------------|-------------------------|-------------------|------------------------------|
| 1 | Chromium-51 | 2000 grams | Lancaster Mixer |
| 2 | Chromium-51 | 30 pounds | Simpson Mixer |
| 3 | Cesium-134 | 2000 grams | Lancaster Mixer |
| 4 | Cesium-134 | 30 pounds | Simpson Mixer |

TABLE 3

Summary of radioactive tracer concentration data used for each of four batches

| <u>Radioactive Tracer</u> | <u>Batch Size</u> | <u>Tagged Component</u> | <u>Total Quan. of Tagged Component</u> | <u>Total Radioactivity in Batch, Microcuries</u> | <u>Microcuries per Fuze</u> |
|-------------------------------|-------------------|--------------------------------|--|--|---------------------------------|
| Chromium-51 | 2000 g | BaCrO ₄ | 10 g | 400 | 0.3 |
| Chromium-51 | 30 lb | BaCrO ₄ | 25 g | 2724 | 0.3 |
| Cesium-134 | 2000 g | KClO ₄ ^a | 28 g | 14 | 0.005 |
| Cesium-134 | 30 lb | KClO ₄ ^a | 100 g | 100 | 0.01 |

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^a Radioactive cesium incorporated as an impurity in parts per million concentration.

TABLE 4
 Summary of data for delay fuzes tagged with chromium-51;
 2000-gram batch

| <u>No. of Fuzes</u> | <u>Charge Weight, g</u> | <u>% Max Column Height</u> | <u>Relative Count Rate^a</u> | <u>Mean Burning Time, sec^a</u> |
|---------------------|-------------------------|--------------------------------|--|---|
| 30 | 1.333 | 100 | 1.000 ± 0.017 | 1.11 ± 0.15 |
| 10 | 1.265 | 95 | 0.982 ± 0.021 | 1.11 ± 0.12 |
| 10 | 1.199 | 90 | 0.920 ± 0.018 | 1.11 ± 0.13 |
| 10 | 1.132 | 85 | 0.887 ± 0.011 | 1.08 ± 0.07 |
| 10 | 1.066 | 80 | 0.853 ± 0.022 | 0.95 ± 0.15 |
| 20 | 0.999 | 75 | 0.814 ± 0.020 | 0.84 ± 0.15 |
| 10 | 0.932 | 70 | 0.771 ± 0.015 | 0.76 ± 0.12 |

^a values denote 2 σ or 95% confidence levels.

TABLE 5
 Summary of data for delay fuzes tagged with chromium-51;
 30-lb batch

| <u>No. of Fuzes</u> | <u>Charge Weight, g</u> | <u>% Max Column Height</u> | <u>Relative Count Rate^a</u> | <u>Mean Burning Time, sec ^a</u> |
|---------------------|-------------------------|--------------------------------|--|--|
| 30 | 1.333 | 100 | 1.000 ± 0.010 | 0.59 ± 0.17 |
| 10 | 1.265 | 95 | 0.956 ± 0.011 | 0.60 ± 0.14 |
| 10 | 1.199 | 90 | 0.913 ± 0.011 | 0.60 ± 0.17 |
| 10 | 1.132 | 85 | 0.882 ± 0.011 | 0.57 ± 0.12 |
| 10 | 1.066 | 80 | 0.835 ± 0.010 | 0.56 ± 0.16 |
| 20 | 0.999 | 75 | 0.780 ± 0.015 | 0.47 ± 0.14 |
| 10 | 0.932 | 70 | 0.728 ± 0.013 | 0.44 ± 0.08 |

^a values denote 2σ or 95% confidence levels.

TABLE 6

Summary of data for delay fuzes tagged with cesium-134;
2000-gram batch

| <u>No. of Fuzes</u> | <u>Charge Weight, g</u> | <u>Column Height</u> | <u>Relative Count Rate</u> | <u>Mean Burning Time, sec</u> |
|---------------------|-------------------------|----------------------|--------------------------------|-----------------------------------|
| 29 | 1.333 | 100 | 1.000 ± 0.012 | 1.75 ± 0.20 |
| 10 | 1.265 | 95 | 0.952 ± 0.010 | 1.70 ± 0.26 |
| 10 | 1.199 | 90 | 0.914 ± 0.011 | 1.64 ± 0.16 |
| 10 | 1.132 | 85 | 0.866 ± 0.005 | 1.58 ± 0.11 |
| 10 | 1.066 | 80 | 0.813 ± 0.010 | 1.44 ± 0.24 |
| 20 | 0.999 | 75 | 0.765 ± 0.009 | 1.38 ± 0.24 |
| 10 | 0.932 | 70 | 0.718 ± 0.009 | 1.35 ± 0.10 |

a± values denote 2σ or 95% confidence levels.

TABLE 7
 Summary of data for delay fuzes tagged with Cesium-134;
 30-lb batch

| <u>No. of Fuzes</u> | <u>Charge Weight, g</u> | <u>% Max Column Height</u> | <u>Count Rate^a</u> | <u>Mean Burning Time, seca</u> |
|---------------------|-------------------------|--------------------------------|-------------------------------|------------------------------------|
| 30 | 1.333 | 100 | 1.000 ± 0.040 | 0.95 ± 0.22 |
| 10 | 1.265 | 95 | 0.948 ± 0.020 | 0.94 ± 0.30 |
| 10 | 1.199 | 90 | 0.892 ± 0.011 | 0.88 ± 0.25 |
| 10 | 1.132 | 85 | 0.852 ± 0.015 | 0.91 ± 0.30 |
| 10 | 1.066 | 80 | 0.795 ± 0.008 | 0.93 ± 0.19 |
| 20 | 0.999 | 75 | 0.760 ± 0.013 | 0.86 ± 0.24 |
| 10 | 0.932 | 70 | 0.716 ± 0.010 | 0.74 ± 0.09 |

^a ± values denote 2 σ or 95% confidence levels.

TABLE 8

External radiation measurements
for 30-pound delay compositions

| <u>Location</u> | <u>Millirems/hr^a</u> | |
|---|---------------------------------|-------------------------|
| | <u>⁵¹Cr</u> | <u>¹³⁴Cs</u> |
| Against mixing vessel | 0.1 | 1.0 |
| 12 inches from mixing vessel | b | 0.2 |
| Over open mixing vessel (at lip) | 0.2 | 0.6 |
| Delay composition 30 pounds (at surface) | 0.6 | 1.0 |

^a Measurements were made with a Victoreen Survey Meter, Thyac II, Model 489.

The values are adjusted for the quantities required for a one unit per second inspection speed.

The accepted maximum permissible radiation level is 2.5 millirems per hr.

^b Background.

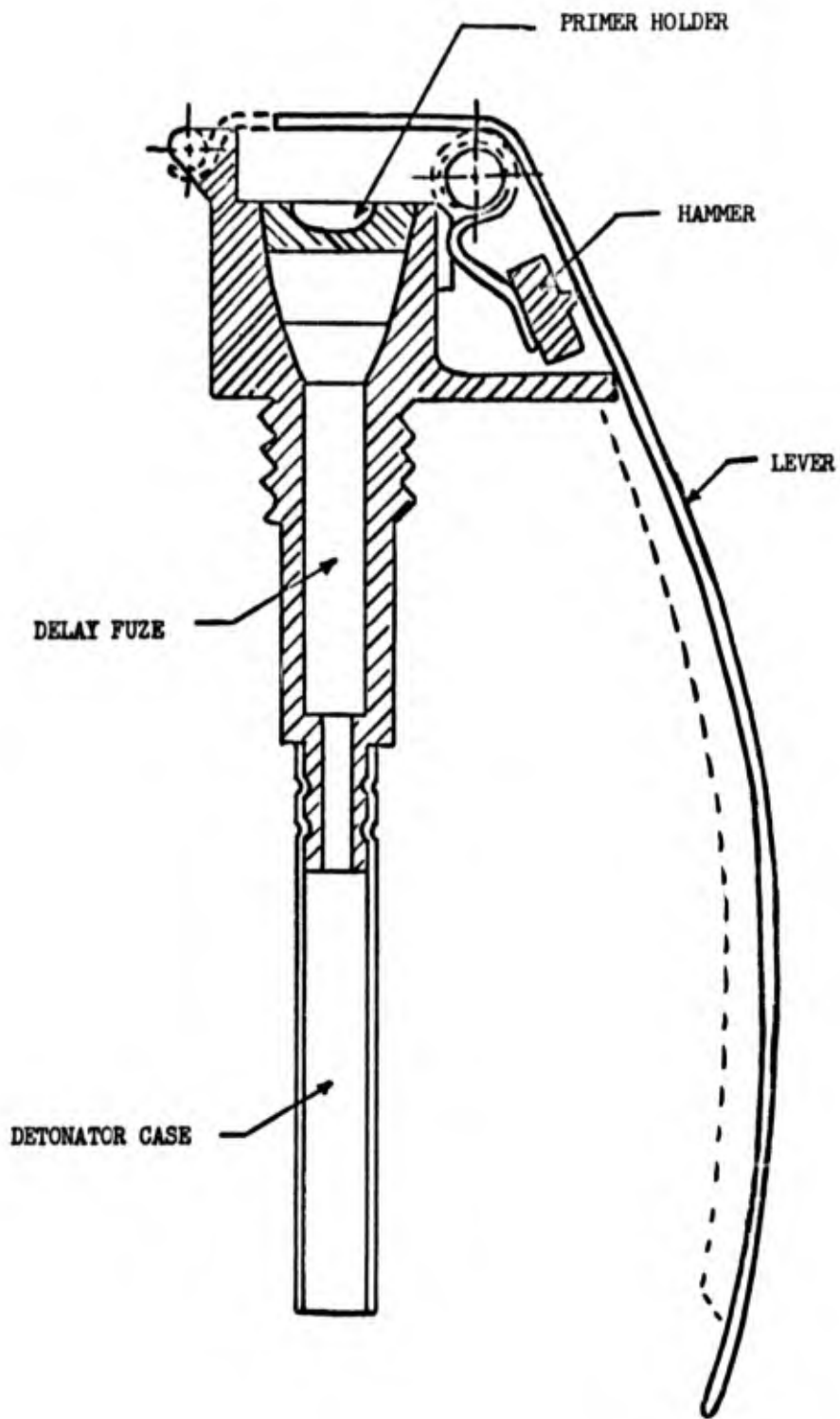


Fig 1 Grenade fuze
(magnified 2 x)

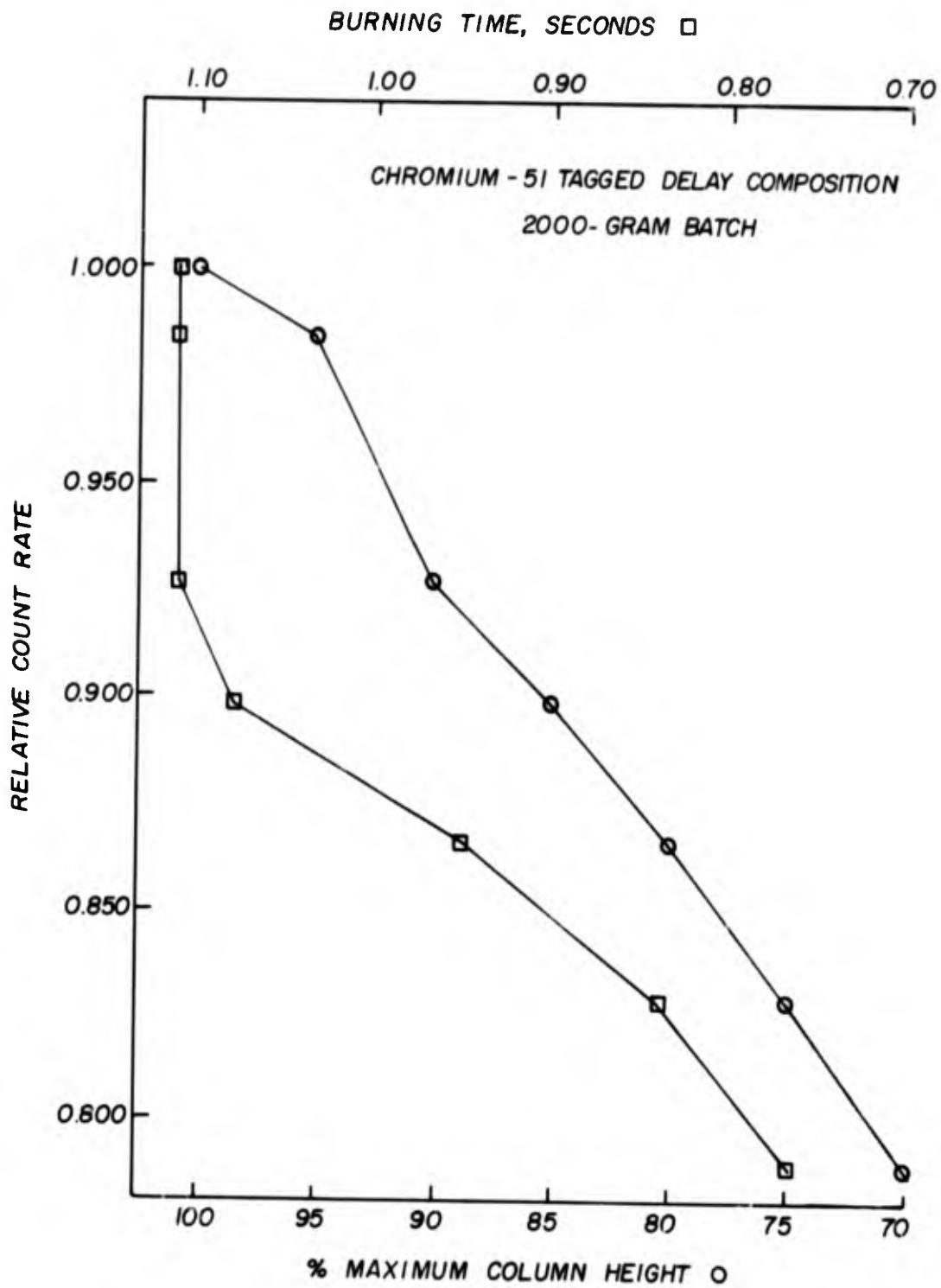


Fig 2 Column height and burning time vs relative count rate for 2000-gram batch of chromium-51 tagged delay composition

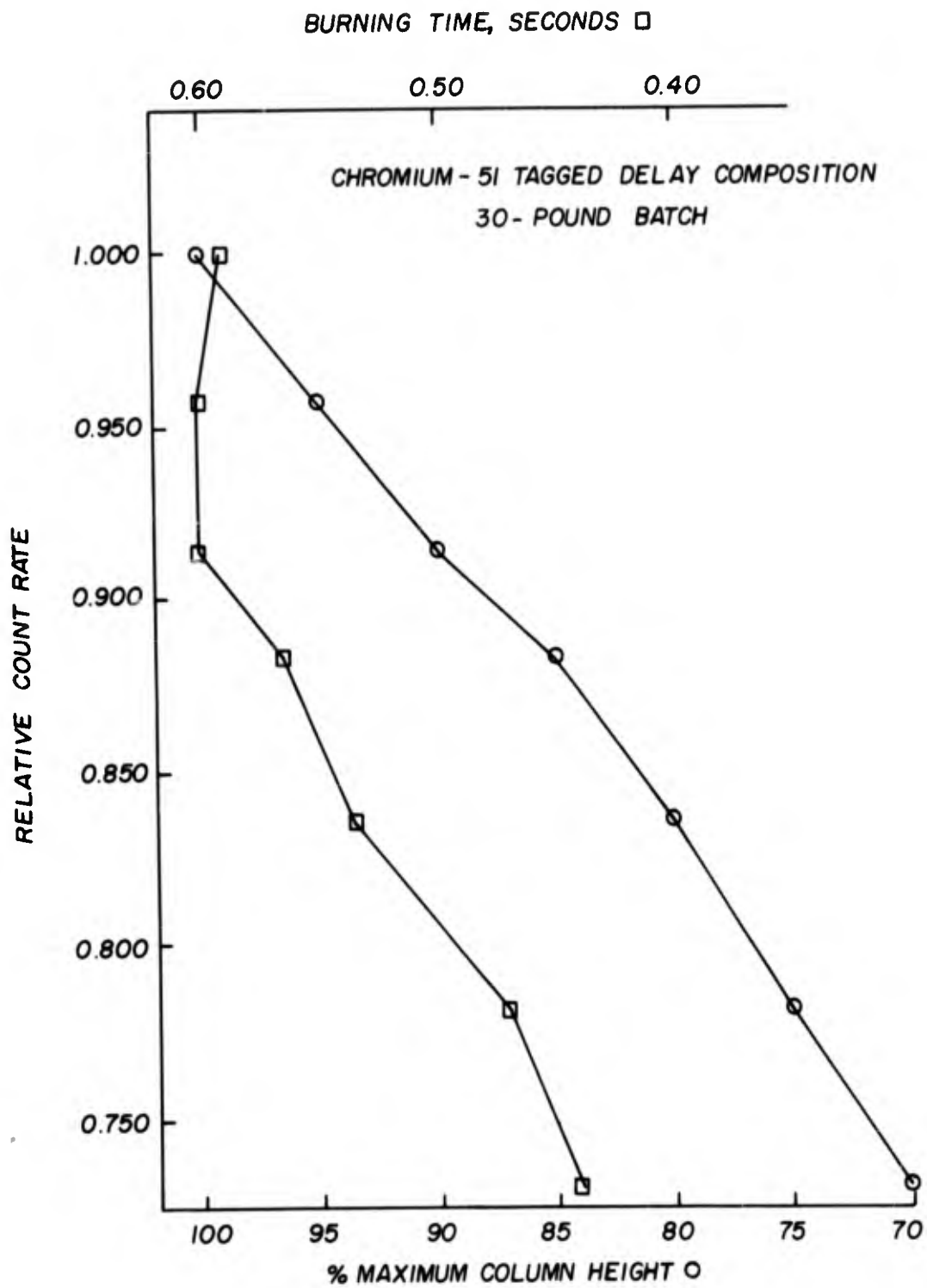


Fig 3 Column height and burning time vs relative count rate for 30-pound batch of chromium-51 tagged delay composition

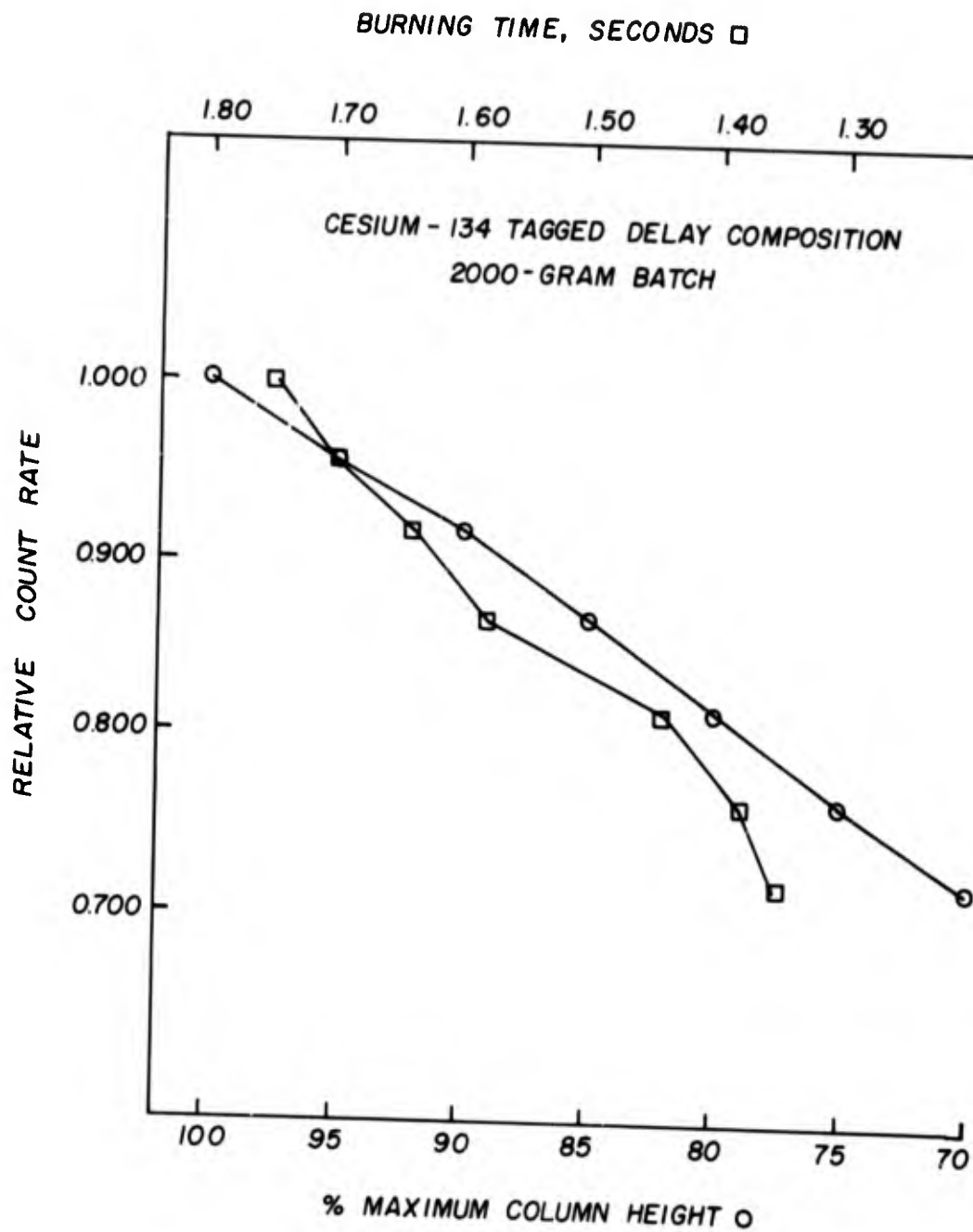


Fig 4 Column height and burning time vs relative count rate for 2000-gram batch of cesium-134 tagged delay composition

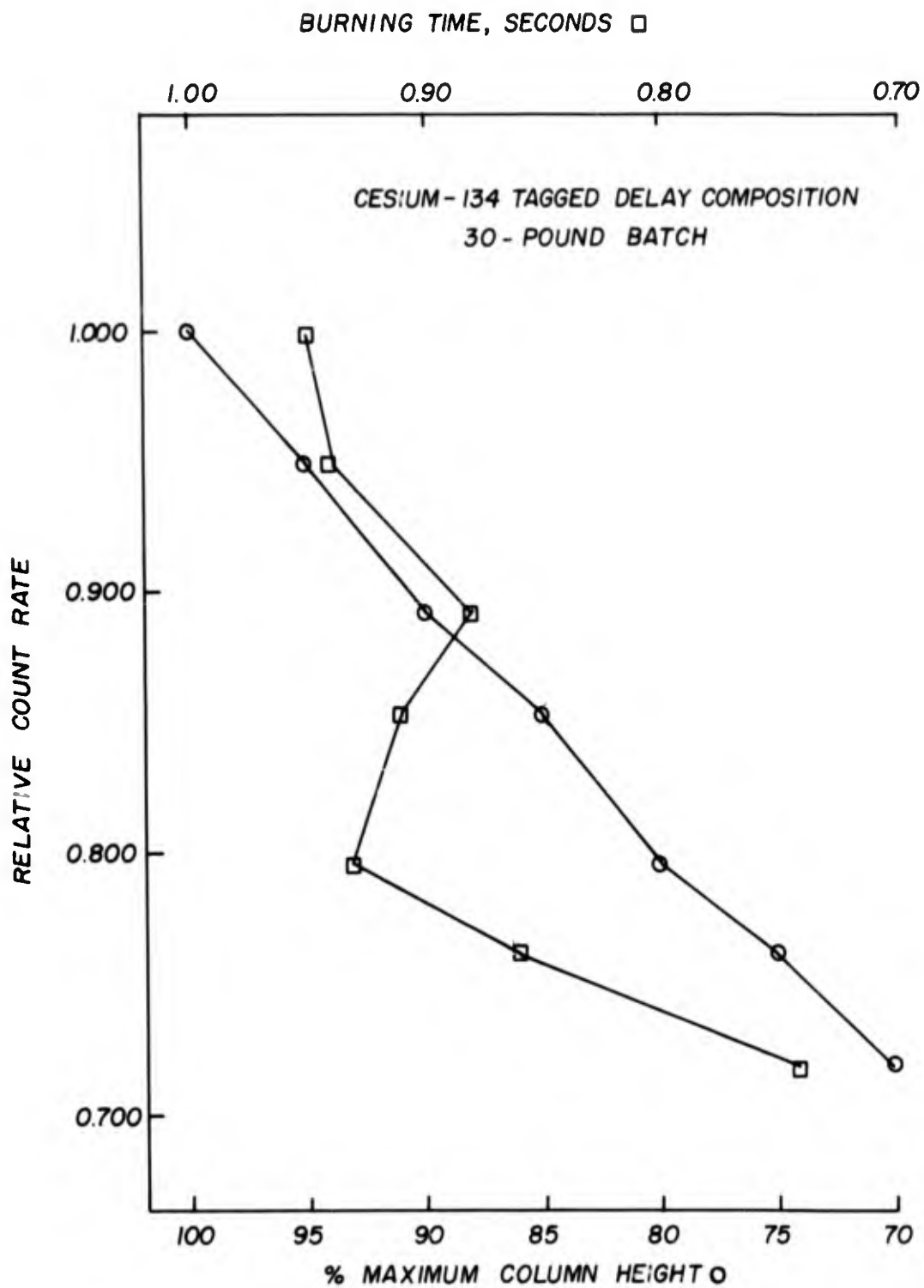


Fig 5 Column height and burning time vs relative count rate for 30-pound batch of cesium-134 tagged delay composition

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| 13. ABSTRACT Incorporation of a radioactive tracer within the delay composition provides a highly reliable method for the inspection of the delay column height in M200 series hand grenade fuzes. The resulting gamma-ray signal from each fuze gives a quantitative measure of total delay charge weight. Based on measurements with plant-size batches of delay composition, levels of radioactivity can be such to accommodate an inspection rate of two units per second, with a single inspection unit, without exceeding permissible external radiation dose levels. The inspection unit itself would consist of a simple, rugged, inexpensive scintillation-counter assembly. The radioactive tracer element need not be common to the delay composition ingredients but can be added to the mixture as a foreign material in ppm concentration. Thus a wide range of radiation half-lives and gamma-ray energies can be used. The radiological safety factors associated with the handling of bulk quantities of radioactive materials in an ammunition loading plant are discussed briefly. | | | |

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