

ABSTRACT

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AFATL-TR-72-204

**STUDIES TO DETERMINE
THE
ENVIRONMENTAL EFFECTS
OF
ILLUMINATING FLARE RESIDUE**

**PYROTECHNICS BRANCH
FLAME, INCENDIARY, AND EXPLOSIVES DIVISION**

TECHNICAL REPORT AFATL-TR-72-204

NOVEMBER 1972

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AIR FORCE ARMAMENT LABORATORY

AIR FORCE SYSTEMS COMMAND • UNITED STATES AIR FORCE

EGLIN AIR FORCE BASE, FLORIDA

**Studies to Determine
the
Environmental Effects
of
Illuminating Flare Residue**

**Bobby M. Agerton
Don D. Harrison
John W. Sigler, 2Lt, USAF
Stephen W. Lander, Jr., 2Lt, USAF**

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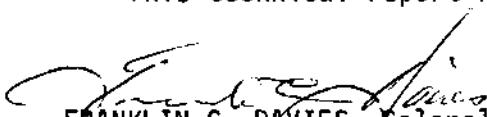
FOREWORD

The USAF project directly related to the information in this report is Project 5066, Armament Development Pollution Control, Task 01, Work Unit 01. This report documents specific studies conducted during the period November 1971 to September 1972.

The authors would like to express their appreciation to Captain Jimmie C. Cornette, Dr. John H. Hunter, 2Lt Vanessa Birdsey, Mrs. Sandra Lefstad, Captain Allen B. Beach, 1Lt Ray Kruzek, and SSgt Terry Collatz for their assistance in this project and in the preparation of this report.

Because of the nature of the experimentation performed, the results were dependent on the exact materials and equipment used; therefore, a notation of sources and manufacturers is provided for reference but is not intended to constitute endorsement of these companies by the United States Air Force.

This technical report has been reviewed and is approved.



FRANKLIN C. DAVIES, Colonel, USAF
Chief, Flame, Incendiary and Explosives Division

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In conjunction with the illuminating flare test and evaluation program on Eglin AFB Reservation, a project was initiated to determine the effect of the flare testing on the flora and fauna on the test areas, as well as selected laboratory species. The results from these tests demonstrate that the residue of illuminating flares has minimal environmental effects except at high concentrations.

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SECTION I

INTRODUCTION

Testing of illuminating flares to determine various illumination characteristics involves outside burning. Air Force Regulation 19-1 requires that the environmental effects of any action be assessed; however, no data were available concerning the environmental consequences of outside testing of flares. Since an ecological impact on the test sites and the surrounding area was considered possible, investigations were conducted to determine the effects of the residue from the combustion process.

Testing of pyrotechnic items is currently carried out at the Pyrotechnics Research Area adjacent to Range 22 of Eglin AFB Main Complex and at Test Area C-52A. The Pyrotechnics Research Area outdoor test facility is adjacent to Choctawhatchee Bay and is 50 to 75 meters from the bay high tide line.

The toxic properties of the illumination flare constituents are fairly well documented with regard to humans. However, little is known concerning the effect of the residue produced on plant and animal life, especially the aquatic ecosystem. LD₅₀ data are available for some species of test organisms. These data (Reference 1), however, deal with massive, one-time injections or orally administered dosages and do not account for effects from exposure associated with soil or aquatic systems. The illumination flare residue is relatively insoluble in water, as is the major constituent, MgO (1.9 mg/l from Reference 1), but it imparts alkalinity to water and also acts as an abrasive.

A series of tests was designed and conducted to determine the actions and effects of illumination flare residues in relation to mammals (white mice), plants (various species), water chemistry (pH, Mg, and Na changes), fish (mosquito-fish, Gambusia affinis Baird and Girard, and bluegill sunfish, Lepomis macrochirus Rafinesque), and leaching in a soil column.

The tests were intended as a survey to determine possible problems in the environment and to indicate where further studies might profitably be directed.

SECTION II

GENERAL MATERIALS AND METHODS

Pyrotechnic residue was obtained from three sources. Samples One and Two were obtained from a contractor's facility bag house and expansion chamber, respectively. The major constituents of these flares before combustion were:

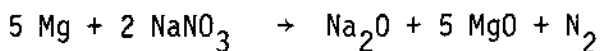
Magnesium (Mg)	58.0%	(Mark 24 or 45 Composition)
Sodium Nitrate (NaNO ₃)	37.5%	
Laminac Binder	4.5%	

Sample Three was collected on polyethylene sheeting at the Eglin Pyrotechnics Research Area outdoor test facility. During a test period when twenty-five LUU-2/B flares were to be burned, a sheet of 6 mil polyethylene 3.04 by 6.1 meters (10 by 20 feet) with an area of 18.6 square meters (200 square feet) was placed on the test site downwind from the flare which was suspended 40 feet above the ground. The wind during this test was generally from the southwest (210 degrees from north) at 5 knots. The center of the polyethylene was 25 meters from a point directly beneath the suspension apparatus.

At the conclusion of the flare test, the material on the sheeting was collected and placed in a drying oven at 49°C for 48 hours. Total weight of the material was approximately 2500 grams after drying. This results in approximately 134.5 g/m² or 5.38 g/m²/flare (12.5 g/ft² or 0.5 g/ft²/flare) at a distance of 25 meters downwind from the burning flare. From these data, the approximate amounts of residue to be used in the various biological and chemical tests described in this report were established. The composition of the flares burned at the Eglin Pyrotechnics Research Area was:

Magnesium (Mg)	61.0%	(LUU-2/B Composition)
Sodium Nitrate (NaNO ₃)	30.0%	
Polymer Binder	9.0%	

The most probable products of combustion from these flares are:



Since magnesium is in excess in the flare composition:



This results in the production of a maximum of 6,771 grams of MgO per flare, assuming 100% conversion or an average of 159,275 grams (352 pounds) per 25 flare test.

Concentrations of illumination flare residue used in the bioassays and chemical studies were selected to approximate the most extreme conditions which might be encountered during normal testing to illustrate the extreme effects of the residue on the environment. Further studies to determine actual concentrations of residue resulting from testing will be conducted and reported in future technical reports.

SECTION III

MAMMALIAN TOXICITY STUDIES

1. METHODS AND MATERIALS

A study was conducted to determine the effects of illumination flare residue (Sample 1) on white mice. The residue was administered by exposure and in drinking water.

Thirty mice were randomly selected to test the effects of the residue. The experiment consisted of three groups, each containing 10 male Swiss-Webster albino mice. The mice were weighed and placed in three separate cages. The cages were supplied with litter (San-i-cel[®]), commercial food (Purina Lab Chow[®]) and water as follows:

CAGE ONE (Control)

1400 gm litter
400 ml H₂O
250 ml food
10 male mice

CAGE TWO (Residue in water)

1400 gm litter
400 ml H₂O (2,500 mg illumination flare residue/l)
250 ml food
10 male mice

CAGE THREE (Residue in litter)

1400 gm litter (with 1 gram of illumination flare residue)
400 ml H₂O
250 ml food
10 male mice

The litter and water were replaced 7 days after the experiment was initiated, and the experiment was terminated at the end of 15 days. It was felt that gross effects would occur during this time period. Weights of the individual mice were again determined at that time. Results of the test are shown in Table I.

The mice in Cage Two ingested the pyrotechnic residue while those in Cage Three were allowed to come in contact with the material through their skin and by inhalation. Autopsies were not performed, but the animals were observed for a period of 30 days after the test termination.

TABLE I. MOUSE TOXICITY STUDY

CAGE NO.	INITIAL WEIGHT, gram	AVERAGE INITIAL WEIGHT, gram	FINAL WEIGHT, gram	AVERAGE FINAL WEIGHT, gram	AVERAGE WEIGHT CHANGE, gram	AVERAGE WEIGHT CHANGE, %
ONE	35	40.4	38	43.1	2.7	6.68
	37		39			
	37		40			
	39		41			
	39		48			
	40		43			
	43		44			
	43		45			
	45		45			
	46		48			
TWO	24	32.5	33	38.1	5.6	17.23
	26		33			
	26		33			
	29		39			
	34		40			
	36		40			
	37		40			
	37		41			
	38		41			
	38		41			
THREE	29	36.4	37	41.6	5.2	14.28
	30		37			
	31		41			
	36		41			
	36		42			
	37		42			
	37		42			
	40		42			
	43		45			
	45		46			

2. RESULTS AND DISCUSSIONS

Visual operations during this experiment indicated that illumination flare residue, in the quantity used, had no detrimental effects on mice. There was a normal increase in weight with the control group, but the mice in both treatments (Cages Two and Three) gained more weight than the control group (Table I). It is not clear whether the increased weight gain was due to the treatment or other factors, and further tests would be required to determine the reason for the variation in weight gain. If it was due to treatment, other observations indicated that it was in no way detrimental.

SECTION IV

PLANT TOXICITY STUDIES

Plant toxicity data were obtained from three experiments: (1) cucumber seed germination and root development, (2) illumination flare residue applied to foliage, and (3) plant growth in soil containing illumination flare residue.

1. EXPERIMENT ONE

a. Methods and Materials

Several preliminary experiments were conducted to determine concentration ranges to consider in this experiment. This experiment was then set up to determine the effects of illumination flare residue on the germination and initial development of cucumber seeds. Thirty-gram samples of soil, each containing 50, 100, 250, 500, or 1000 mg/kg of illumination flare residue (Sample One) were placed in petri dishes. For comparison purposes, reagent grade MgO was added to separate soils in various concentrations. Distilled water (12 ml) was then added and a piece of Whatman #3 filter paper was placed on the soil surface. Five cucumber (*Cucumis sativus* var. Long Green) seeds were placed on the filter paper and allowed to germinate for 72 hours in the dark at 26°C. The root lengths were measured to determine if any inhibition had occurred. Experiments were conducted in triplicate.

b. Results

<u>Treatment</u> (illumination flare residue/soil)	<u>Root Length (cm) after 3 days</u>			
	<u>Average of each Petri dish</u>			<u>Average</u>
Control	5.4	5.1	6.4	5.6
50 mg/kg	4.1	4.3	4.1	4.2
100 mg/kg	4.5	3.9	3.9	4.1
250 mg/kg	1.7	1.9	1.9	1.8
500 mg/kg	1.4	1.3	1.3	1.3
1000 mg/kg	0.9	0.8	0.6	0.8

Slight inhibition of root development occurred at levels as low as 50 mg/kg and soil with 1000 mg/kg almost completely inhibited germination. There was also a large increase in inhibition from 100 mg/kg to 250 mg/kg. These results, however, were not different from the effects of the reagent grade MgO used in this experiment. The pyrotechnic residue used in this experiment had no effects on cucumber development greater than the reagent grade MgO.

2. EXPERIMENT TWO

a. Methods and Materials

Several species of plants received foliar application of illumination flare residue (Sample One) to determine if dusts or fall-out from illumination flare tests would injure vegetation. The foliage was wet with a small hand sprayer to facilitate sticking and then approximately 1 gram of the illumination flare residue was dusted onto a portion of the foliage. The following plant species were used:

<u>SCIENTIFIC NAME</u>	<u>COMMON NAME</u>
<u>Paspalum notatum</u>	bahia grass
<u>Pueraria thunbergiana</u>	kudzu
<u>Portulaca oleracea</u>	portulaca
<u>Manihot utilissima</u>	cassava
<u>Melia azederach</u>	chinaberry
<u>Prunus caroliniana</u>	cherry laurel
<u>Musa sp.</u>	banana
<u>Oryza sativa</u>	rice
<u>Utricularia sp.</u>	bladderwort
<u>Cynodon dactylon</u>	bermuda grass
<u>Pinus elliotii</u>	slash pine

Observations were made periodically for 30 days after treatment to determine if any damage had occurred.

b. Results

No visible damage had occurred to any plants 30 days after foliar treatment.

3. EXPERIMENT THREE

a. Methods and Materials

An experiment was initiated to determine if residue from illumination flare tests visibly affected the growth of several plant species. Since the

flare residue does not leach readily (see Section IV) and would therefore remain predominantly on the soil surface, the residue was applied in terms of units per area rather than units per volume of soil. Seeds of the following species were planted in a soil consisting of a 7:3:1 ratio of sandy loam, peat moss, and perlite with 5 pounds of dolomite lime and 1 pound of super phosphate added per cubic yard of soil mix.

<u>SCIENTIFIC NAME</u>	<u>VARIETY</u>	<u>COMMON NAME</u>
<u>Oryza sativa</u>	IR-8	rice
<u>Latua sativa</u>	Grand Rapid	lettuce
<u>Zea mays</u>	Coker 71	corn
<u>Cucumis sativus</u>	Long Green	cucumbers
<u>Lycopersicon esculentum</u>	Homestead	tomatoes

Illumination flare residue (Sample I) was added to the soil samples on the surface at rates of 500 lb/acre and 1000 lb/acre after seeds were planted. Plants were grown in a glass greenhouse with 45% shade. Pots were watered daily from the top to allow the residue to leach downward. Visual observations only were made for 60 days.

b. Results

No visible difference was observed between the control plants and those receiving either concentration of illumination flare residue. Lettuce plants died after approximately 30 days as a result of disease, but there was no effect from the treatment.

4. DISCUSSION

Even when illumination flare residue falls directly on vegetation, it does not appear to be extremely harmful. The largest portion of the residue is MgO, and this is probably used by the plant after it enters the soil system. Magnesium is one of the required plant nutrients and is a component of the dolomitic limestone commonly used in agricultural operations.

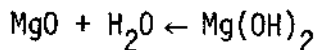
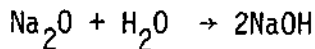
The only effect observed in these studies was inhibition of initial root growth after the germination of cucumber seeds in soil that contained the illumination flare residue. This residue could conceivably affect the seed germination of native plant species around a pyrotechnic test area if sufficient amounts accumulated. However, it is expected that any effect would be limited to a small area close to a highly used test site.

SECTION V

FISH BIOASSAY STUDY

The limited reference material available concerning the effects of illumination flare residue applies chiefly to terrestrial animals ("Environmental Statement" compiled in October 1971 by Captain Jimmy C. Cornette from Navy OP 2793). Therefore, the effect of the residue on two fish species was investigated.

The chemistry of the illumination flare residue and the reactions which occur in combination with water are fairly straightforward. Upon combination with water (humid air is sufficient), Na_2O and MgO convert as follows:



Magnesium and sodium hydroxides impart alkalinity to water if present in high concentrations. Sodium compounds, NaOH and particularly Na_2O , combine readily with water (Reference 1). Due to the lack of pertinent information concerning aquatic effects, a study utilizing fish as a bioassay organism was designed and implemented. It should be stressed at this time that these tests were a survey and did not provide detailed information concerning the effects of the illumination flare residue on fish.

1. WATER CHEMISTRY INVESTIGATION

Because pH increase was known to be the main effect of the materials incorporated in the illumination flare residue, varying concentrations of the residue were added to four types of water, and the increase in pH was measured.

Quantities of the illumination flare residue were added to water from the following sources:

1. Distilled water (from the laboratory Barnstead still)
2. Weekly Pond (fresh water)
3. Choctawhatchee Bay (salt water)
4. Tap water (aerated for 5 days)

a. Methods and Materials

A beaker containing 1 liter of the water to be tested was placed on a magnetic stirrer (speed six, Sargent Stir Plate®) and constantly stirred with a Teflon® stir bar. Amounts of the illumination flare residue (Sample One) were added to the water to obtain concentrations of from 1.0 mg/liter to 1000 mg/liter of the residue. After 5 minutes of stirring at each concentration, pH determinations were made with a standard laboratory unit. The results are shown in Table II.

b. Discussion

The pH of all four types of water tested was increased by the addition of as little as 0.01 gram (10.0 mg/liter) of the illumination flare residue.

A smaller initial increase in pH and a lower, or equal, increase in pH at the higher concentrations were noted in the Choctawhatchee Bay samples compared to the other types. This effect is probably due to the greater buffering capacity of sea water compared with that of fresh water. Because of the lower buffering capacity of the distilled water and tap water, it follows that the pH of the Weekly Pond water would increase more than that of sea water and less than or equal to that of tap water. These effects probably account for a large percentage of the variability in the results of the Fish Bioassay Section as discussed below.

2. METHODS AND MATERIALS

Fish were obtained from three sources. Mosquitofish (Gambusia affinis Baird and Girard) were obtained from ponds on Eglin AFB Reservation, chiefly from Anderson Pond. Bluegill sunfish (Lepomis macrochirus Rafinesque) were obtained from the Holt Fish Hatchery, Holt, Florida, and the Jackson Guard Station on Eglin AFB Reservation.

All fish were transported from the field to the laboratory in 50 gallon plastic containers and placed in 20 gallon Instant Oceans®, aquaria equipped with a two air stone filter-flow aeration system. The tanks were filled with either tap water (aerated for 5 to 10 days) or water from Weekly Pond. Variations in water type used will be discussed later.

Fish were placed in glass battery jars which had previously been filled with 10 liters of the test water to be used. An air stone system attached to a series of Silent Giant® air pumps was placed in each jar.

With the exceptions noted on the tables, five male and five female mosquitofish of approximately equal size were used for each treatment involving Gambusia. Six bluegill of approximately equal size, without regard to sex, were used for each treatment in the Lepomis tests. During Test Two with the Lepomis, all fish were weighed and marked, but only limited data were obtained due to the premature termination of the test (see Table III).

TABLE II. WATER pH INCREASES EFFECTED BY ILLUMINATION FLARE RESIDUE (SAMPLE THREE)

CONCENTRATION, mg/ℓ	TAP WATER	DISTILLED	WEEKLY POND	CHOCTAWHATCHEE BAY
00.00	8.4	5.4	7.4	8.0
1.00	8.4	5.6	7.5	8.0
10.00	8.4	5.6	7.5	8.1
25.00	8.5	5.8	7.7	8.1
50.00	8.5	6.0	8.0	8.2
100.00	8.6	6.4	8.8	8.3
200.00	8.8	8.7	9.3	8.5
300.00	---	9.4	9.6	8.7
500.00	9.1	9.7	9.9	8.9
1000.00	9.6	10.0	10.0	9.6

Readings were taken after a 5 minute period on a magnetic stirring plate.

During the tests, the fish were fed 0.1 gram of commercial fish food (Purina Fish Food®) in the morning. If the fish did not eat during the morning feeding, the evening feeding was omitted for all test fish. Seven tests were conducted with Gambusia and two with Lepomis. Due to the wide variations in test procedures, each test and its results will be described separately. Because of the method of introducing the illumination flare residue and the fact that the tests dealt with an aquatic system, TL_x data are presented rather than LD_{50} or LD_{100} data. All conditions and results of the tests are presented in Tables III to V.

3. RESULTS

In concentrations of 100 mg/ℓ and above, the illumination flare residue appeared to be toxic to the Bluegill Sunfish. Lower concentrations seemed to have little detrimental effect (Table III).

Gambusia were not affected detrimentally by the illumination flare residue when tested in water from Weekly Pond, but seemed extremely susceptible in tap water (Table IV).

As this study was intended to be a survey, replications of individual tests were not to be performed. However, the results obtained in the Gambusia bioassays in tap water were so difficult to interpret that they were repeated (Table V). At the end of three replications, it appeared that the illumination flare residue was not toxic to Gambusia in the concentrations used.

TABLE III. BLUEGILL SUNFISH BIOASSAY TESTS

TEST NUMBER	HOLDING TANK WATER TYPE	TEST JAR PARAMETERS/ACCLIMATION TIME	TEST MATERIAL TYPE/CONC.	TL ₅₀	TL _x	COMMENTS
ONE	Tap	pH 8.1 DO --- % Sat. 1 hr.	SAMPLE ONE ^a 50 mg/ℓ 100 mg/ℓ 500 mg/ℓ	>2 days 41 hr. 18 hr.	TL ₁₆ 144 hr. TL ₆₆ 66 hr. TL ₁₀₀ 52 hr.	All fish that died showed gill damage. Filaments were torn. Heavy mucous was present and all deaths observed were preceded by flashing.
TWO	Weekly Pond	pH 8.1 DO 6.4-8.0 % Sat. 16-20.5 8 days	SAMPLE THREE ^b 100 mg/ℓ 125 mg/ℓ 150 mg/ℓ 200 mg/ℓ 300 mg/ℓ			The fish were weighed and placed in the tank. Observations were made during a 12 hour period of the day. Additional observations were made after 24 hours. No distress was evident in any tank. After 48 hours all the fish were found dead with the exception of the controls. The fish had been dead for 12-18 hours and were partially decayed. These data yield an approximate TL ₁₀₀ of 36-42 hours.

^aSample I: Residue from bag house at contractor's facility.
^bSample III: Residue from Eglin Pyrotechnics Research Area.

TABLE IV. GAMBUSIA BIOASSAY TESTS

TEST NUMBER	TEST JAR ¹ WATER TYPE	TEMPERATURE OF TEST, C	TEST MATERIAL ² TYPE/CONC.	TL ₅₀	ADDITIONAL TL _x	COMMENTS
ONE	Tap ³	18°	SAMPLE ONE 10 mg/l 100 mg/l 1000 mg/l	10 hr. 5 hr. 10 hr.	TL ₈₀ 30 hr. TL ₁₀₀ 30 hr. TL ₁₀₀ 30 hr.	Moderate to severe distress before all deaths.
TWO	SEE TABLE V					
THREE	Weekly Pond	17°	SAMPLE ONE 0.1 mg/l 1.0 mg/l 10.0 mg/l 50.0 mg/l	No mortality at any concentration. Experiment terminated at end of 13 days.		
FOUR ⁴	Weekly Pond	18°	SAMPLE THREE 50 mg/l 100 mg/l 125 mg/l	> 24 days > 24 days > 24 days	TL ₃₃ ⁴ 7 days TL ₁₆ 9 days TL ₁₆ 4 days	No aeration for 2-1/2 hours before test started. Additional subsequent loss of power at 24 days prompted termination of experiment.

¹Tests One and Two had a jar acclimation time of 1 hr. 45 minutes, Test Three had a jar acclimation time of 68 hours, and Test Four had a jar acclimation time of 16 days.

²Sample One: Residue from contractor's facility, bag house, Sample Two: Residue from contractor's facility, expansion chamber, and Sample Three:

³Material collected on polyethylene sheeting at Eglin Pyrotechnic Research Area.

⁴Tap water had been aerated from 2 to 4 days.

⁴Only six fish were used in each treatment of Test Four.

TABLE V. RESULTS OF TEST TWO, GAMBUSIA

TEST TWO/ REPLICATES ¹	TEST JAR WATER TYPE	TEMPERATURE OF TEST, C	TEST MATERIAL TYPE/CONC.	TL ₅₀	ADDITIONAL TL _x	COMMENTS
A	Tap	18°	SAMPLE TWO 10 mg/l 100 mg/l 1000 mg/l	3 hr. 7 hr. 7 hr.	TL ₉₀ 21 hr. TL ₁₀₀ 19 hr. TL ₁₀₀ 19 hr.	Sample Two material is approximately twice as dense as Sample One.
² B	Tap	18°	SAMPLE TWO 10 mg/l 100 mg/l 1000 mg/l	0 0 0	TL ₁₀ 76 hr.	
³ C	Tap	18°	SAMPLE TWO 10 mg/l 100 mg/l 1000 mg/l	0 76 hr.	TL ₁₀ 76 hr.	
^{3,4} D	Tap	18°	SAMPLE TWO 10 mg/l 100 mg/l 1000 mg/l			

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¹In the initial test, A, 10 mg/l appeared to be more toxic than 1000 mg/l. The test was then replicated three times.

²Replicate B was run with 10 fish in each test jar. Two jars of each level, 10, 100, and 1000 mg/l were observed for 80 hours. These jars were 1 liter and were not aerated during the test.

³Tests C and D were replicates of each other using 10 liters of water and aeration. Statistically meaningful deaths occurred only in the 1000 mg/l test (TL₅₀ 76 hours) in Test C above. During the course of the test, one of the pregnant Gambusia gave birth to 3 young at between 80 and 90 hours into the test. These young were observed dead at 96 hours.

⁴Fish selected without regard to sex.

4. DISCUSSION

During the fish bioassay study, results were obtained over a moderately wide range of toxicity. It must be remembered that these tests were conducted under laboratory conditions as static bioassays involving only two species of fish. However, the results seem to indicate that in concentrations of 100 mg/l or greater, the illumination flare residue would prove to have detrimental effects during 1-2 day exposures. Concentrations up to 50 mg/l would probably have little permanent effect during longer exposures.

Mortality data (Tables III to V), indicate that within the controlled conditions (as outlined in the tables) of these tests, the fish were affected depending on the type of water used. This evidence is augmented by the findings during the water chemistry investigation, which indicate that the limited buffering capacity of tap water affects the pH increase upon addition of even minute amounts of the illumination flare residue.

The effect of this residue on the aquatic ecosystem is then largely dependent not only on the type of water that it is deposited in but also on the chemistry of that water. The chemical effect is, of course, a direct result of the amount of the material added.

SECTION VI

SOIL LEACHING STUDY

To determine the effects of illumination flare residue in the soil, (and the mechanism of these effects) a laboratory experiment was designed and implemented to duplicate a portion of the soil column.

1. METHODS AND MATERIALS

Soil samples were obtained from an area south of the Eglin Environmental Research Facility from a soil type closely resembling that of the flare test areas. Several samples were collected from the natural soil column with a 2 inch core borer to a depth of 48 inches in 6 inch increments. All of the samples from each increment were thoroughly mixed in a dry soil blender, and the soil was added to an aluminum tube 4 inches in diameter and 56 inches high (Figure 1). The soil was added to the tube in small increments and packed to approximate natural conditions. Each level of the natural soil column was thereby represented within the study apparatus.

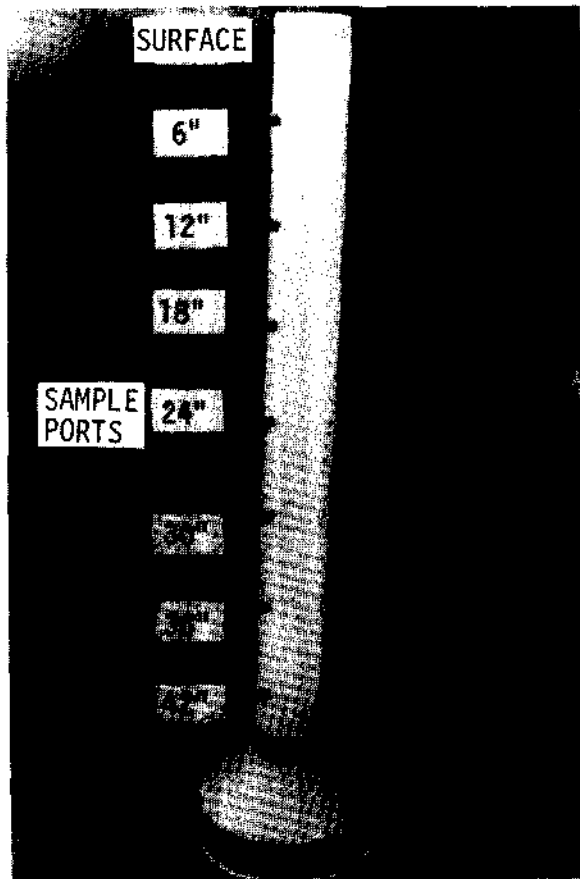


Figure 1. Soil Leaching Tube Test

Distilled water was then poured through the column to approximate packing due to rainfall and allowed to dry. Sufficient illumination flare residue (Sample Three) was then applied to equal 4,000 pounds per acre on the surface. This rate is equal to high applications of agricultural lime, which would probably result in similar reactions.

A uniform amount of distilled water (205 ml) was then sprinkled evenly on the top of the column daily for 54 days to simulate 1 inch of rainfall per day.

Subsamples of the initial mixes and the soil in the column were taken at 0, 20, and 54 days and analyzed for magnesium and sodium content by atomic absorption spectroscopy. Determinations of pH were made at 0 days and 54 days only. A replicate of the samples analyzed for Mg and Na were sent to the University of Florida Soils Department for independent analysis.

2. ANALYTICAL TECHNIQUES FOR SOIL pH

Before pH determinations were made, all soil samples were dried at 50°C. for 24 hours. Two separate methods were used to determine pH. Literature reviews (Reference 2) indicate that use of distilled water introduces variability.

METHOD ONE consisted of the dilution of 20 grams of premixed soil with 80 ml of distilled water.

METHOD TWO consisted of the dilution of 50 grams of premixed soil with 100 ml of 0.01 M CaCl_2 . This method was employed following the recommendations of Smiley and Cook (Reference 2).

All pH measurements were taken on a standard laboratory pH unit. Samples were stirred for 2 minutes and allowed to equilibrate before the final reading was recorded. Results of the pH determinations of the original control soil and the subsamples from the soil leaching column are shown in Table VI.

3. ANALYTICAL TECHNIQUES FOR MAGNESIUM AND SODIUM DETERMINATIONS IN SOIL

To determine the actual concentrations of magnesium and sodium present in the soil samples collected from the laboratory soil leaching study, soil extractions were made, and analyses were performed to determine magnesium and sodium as mg/kg of soil. Five grams of soil were extracted (Reference 3) with 100 milliliters of one normal ammonium chloride (1N NH_4Cl) for 6 hours in 250 ml Nalgene bottles with mechanical shaking. The solutions were filtered through Whatman #1 filter paper to remove suspended soil particles from the extract. Before analysis, each sample was diluted as necessary to be within the working range of the instrument (0.1 to 3 ppm).

TABLE VI. SOIL pH VALUES OF LEACHING EXPERIMENT		
SOIL COLUMN DEPTH, inches	SOIL pH VALUE	
	BEFORE ADDITION OF PYROTECHNIC RESIDUE	54 DAYS AFTER ADDITION OF PYROTECHNIC RESIDUE
Surface	5.7	8.6
6	5.7	6.5
12	4.9	5.1
18	5.1	5.5
24	5.3	5.4
30	5.8	5.4
36	5.8	5.5
42	6.1	5.8

Analysis for magnesium was performed by aspirating samples into a Jarrel-Ash Model 82-500 Atomic Absorption Spectrophotometer on the absorbance mode with a tri-flame burner. Operating conditions were: wavelength, 2851 Angstroms; lamp current, 10 milliamperes; fuel, hydrogen at 10 SCFH flow; oxidant, compressed air at 15 SCFH flow, chart recorder range, 0 to 10 millivolts; average sample aspiration time, 5 seconds.

Analysis for sodium was performed by aspirating samples into the spectrophotometer while operating on the flame emission mode using the HETCO burner. Operating conditions were: wavelength, 5890 Angstroms; fuel, hydrogen at 10 SCFH flow; oxidant, compressed air at 15 SCFH flow; chart recorder range 0 to 10 millivolts; average sample aspiration time, 5 seconds.

A standard curve was established for both elements from which the concentrations of the unknown samples were read. Standards were prepared by the dilution of stock solutions of 1000 mg/l of Mg or Na atomic absorption standards (HARLECO). The data were plotted as peak height (percent absorption) versus concentration. The observed values of Mg and Na concentrations in the soil samples are given in Table VII.

4. DISCUSSION

The results of the analyses for magnesium and sodium in the soil column leaching study (Table VII) show that the illumination flare residue leaches through the soil to a depth of only 12 inches. The analysis for magnesium showed extremely high concentration levels (500 and 450 mg/kg) in the first

SOIL COLUMN DEPTH, inches	BEFORE ADDITION OF PYROTECHNIC RESIDUE		20 DAYS AFTER ADDITION OF PYROTECHNIC RESIDUE		54 DAYS AFTER ADDITION OF PYROTECHNIC RESIDUE	
	Mg,mg/kg	Na,mg/kg	Mg,mg/kg	Na,mg/kg	Mg,mg/kg	Na,mg/kg
Surface	5	20	500	26	450	25
6	2	28	2	26	30	23
12	1	23	1	24	2	28
18	1	27	1	30	1	25
24	1	23	1	30	1	25
30	1	27	1	28	1	28
36	1	23	1	24	1	21
42	1	21	1	28	1	23

level, which was to be expected since grains of the white illumination flare residue were visible in the soil sample before extraction. A significant amount leached into the second level (6 inches) after 54 days. Below 12 inches, there was no increase in the magnesium concentration over that of the control soil. The sodium concentration in the soil, however, after the illumination flare residue had been added, remained approximately the same as the control soil. Data obtained from the University of Florida Soils Department indicated the same trends. Unpublished results from an experiment conducted by Harrison, Lander, and Sigler ("Residual Levels of Sodium and Magnesium in Soil from Two Pyrotechnic Tests Areas on Eglin AFB, Florida") indicate that the flare residue collected at Eglin AFB had no sodium present, while that collected at the contractor's facility did. The difference is apparently due to the method of collecting the residue. At Eglin AFB, the flares were burned in an open area, and the residue was collected on polyethylene sheeting. The wind dispersed the light material which could have contained the sodium. At the contractor's facility all the residue was collected within the test chamber.

The analytical technique was limited to approximately 90% accuracy due to mixing, weighing, and extracting procedures. The atomic absorption instrumentation data itself were reproducible to 0.01 ppm.

SECTION VII

CONCLUSION

Illumination flare residue appears to have a very low toxicity to mice, plants, and fish. Concentrations above 100 mg/ℓ of illumination flare residue would likely have detrimental effects on indigenous fish populations during a short term (1 to 2 day) exposure. Lower concentrations (10 to 50 mg/ℓ) appear to be relatively innocuous over longer periods (10 to 20 days) of exposure. Mice were not affected by ingestion or skin contact and inhalation of the residue at relatively high concentrations. Plants were not affected detrimentally at concentrations of 1000 lb/acre in the soil or by having the residue applied directly to the foliage. Germination of the cucumber seeds were slightly affected at concentrations above 50 mg/kg in a petri dish bioassay method. The concentrations required to cause any of the above effects, however, are not likely to occur as a result of pyrotechnic testing even after several years of testing over the same site.

Calculations from the data in this study indicate that the pH in a 4 hectare (10 acres) pond with an average depth of 3 meters (10 feet) would be increased less than 0.1 unit if all the residue from 100 flares (15 pounds is the approximate composition weight/flare or residue weight/100 flares) fell into the pond and was evenly distributed. The concentration of pyrotechnic residue for this hypothetical case would be 2.27 mg/ℓ.

The results from these studies indicate that the effects of illumination flare residue are very minimal and are not particularly dangerous to the environment in the concentrations used in these studies, which were selected to represent the high range that would be found on a pyrotechnic test area.

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