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AN IMPACT SENSITIVITY STUDY OF

REACTIVE MATERIALS

by

Carl Edward DeLong

A

THESIS

submitted to the faculty of

THE UNIVERSITY OF MISSOURI-ROLLA

in partial fulfillment of the requirements for the

Degree of

MASTER OF SCIENCE IN CIVIL ENGINEERING

Rolla, Missouri

1969

Approved by

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ABSTRACT

The impact sensitivity of a potassium chlorate, stabilized red phosphorous, Quso, and magnesium oxide mixture in a 34/14/4/2 ratio has been found to be about 18 ± 5 gram-centimeters for a 50% sensitivity to initiation. Chi-square tests at the 95% confidence level have determined that it is not possible to reject the hypothesis that the distribution is either normally distributed or log-normally distributed within the 50% range.

Humidity, aging, and some additives were found to desensitize the mixture. Energy versus drop weight and test height curves show that momentum considerations are crucial over large test ranges in studies of impact sensitivity. Drop weight velocity and energy delivered to the reactants were determined to be the critical parameters.

Sensitivity data for phosphorous and potassium chlorate mixtures and copper chlorotetrazole are presented and interpreted.

A method for appraising the performance of the impact apparatus is included.

ACKNOWLEDGEMENT

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I. INTRODUCTION

A. GENERAL

Impact sensitivity of selected reactive mixtures was determined as part of a larger project on "An Investigation of the Sensitivity and Compatibility of Reactive Materials".[1] Energy level correlations determined by an impact test, a thermal bath initiation test, and an electrostatic initiation test are suggested.

Tests using a modified impact tester were performed in order to determine an energy input value for the 50% or median point. This is the point where 50% of the specimens explode or do not explode. The Bruceton "Up and Down" technique of testing was used. This technique is extensively used in explosives and research sensitivity testing.

Some pyrotechnic compositions have been found to be very sensitive to input energies. Although applications are limited, a knowledge of their sensitivity to initiation is necessary in order to predict how these compositions will react under varying conditions. Factors such as type of input energy, humidity, fuel particle size, aging, etc. can have a definite effect on their behavior. Safety in handling and storing is also a major consideration.

Stabilized red phosphorous and potassium chlorate, when separated, are inert and safe to handle. However, when mixed together they form a highly sensitive reactive mixture which is hazardous to work with, especially in large quantities. The products formed from the reaction are solids which develop a high heat of formation (-2222 Kcal/Mole).

Tests were undertaken to determine the optimum test energy. This included varying the stoichiometry of the mix and noting the effect of additives. Rebound studies were performed so that the impact tester could be correlated with other testers.

B. IMPACT TESTERS

Impact testers have been in use since the early part of this century. Although easy to construct and use, their results are of limited use due to the large number of variables which cannot be controlled and are generally used for comparative purposes. The problem is that there is no definite mechanical sensitivity of an explosive.[2]

Different types of tests such as impact, electrostatic, thermal, etc. frequently do not correlate since each test seems to sample different combinations of the explosives characteristics. Thus, it would seem best not to base a conclusion of sensitivity on the basis of a single type of test.[3] Various investigators have found that theoretical

equations do not apply exactly to impact testing. More-over, it appears that velocity, or possibly the rate of energy absorption are the most important parameters to be considered.[4]

The design and construction of impact apparatus have been extensively reported in the literature. A few of the more important characteristics are included here. The tester must have a firm foundation. The cap and sample holder must be made of hardened steel in order to avoid excessive damage. Renewal of equipment is necessary due to damage and for reproducibility. This is especially true of the caps since they bear the brunt of the explosive force. Lateral movement of the cap with respect to the cup must be minimized. A standard volume of reactant is nec-essary for control purposes.

Standard impact testers have been developed by the Bureau of Mines and by the Picatinny Arsenal. In general, a two kilogram weight is guided by two uprights. An electro-magnet is used to control the weight. The weight, when dropped, impacts with a plunger which rests upon the sample being tested. The sample is centered on a hardened steel anvil.

C. REVIEW OF LITERATURE

1. Impact Literature

Impact sensitivity has been throughly investigated by many researchers. They have developed the theories for impact apparatuses, initiation by impact, and statistical methods of analyzing the results.

Boyars reviews the development of impact sensitivity tests and assesses the significance of the test data.[2] Weingarten presents a method for measuring the performance of impact machines.[5] Both Boyars and Weingarten give extensive bibliographies in their papers. Smith and Richardson provide an approach for determining the force distribution or energy input at the time of initiation.[6] Hollies, Legge, and Morrison suggest that momentum is the most important factor in determining the probability of detonation.[4] However, Churchman and Kersh state that "Drop Test results obtained with various balls and pins are, in reality, quite consistent if total energy is used as the significant variable". They show that when the mass of the ball to the mass of the pin is greater than one, multiple impacts will occur. That is, repeated and separate blows by the ball occur to the pin before the ball reverses direction in a visible bounce. They state that "....the rate of energy application is not one of the contributing variables in multiple impact testing".[7]

Their formula ($E = mH_O + b$) was used to predict initiation probabilities.

$E = mH_O + b$, where: E is the mean critical energy
m is the striker mass or weight
b is the energy intercept
H_O is a parameter of the sensitivity equation
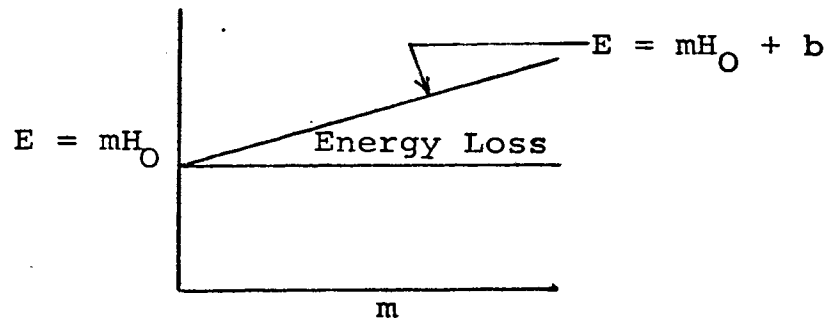
FIGURE 1.

E vs m Sensitivity Equation

Extensive impact sensitivity testing has been undertaken by the Picatinny Arsenal, Dover, New Jersey. Work by Harris, Edelman, and Kaye tested parameters such as fuel particle size, aging, humidity, temperature, and additives.[8] Kristal and Kaye found that the effect of additives cannot be accurately predicted. Further, they found that the oxidizing agents for those mixtures tested were the principal sensitivity contributing agents.[9]

2. Statistical Analysis Literature

Bulfinch presents a description of a unified design-of-experiment procedure for statistically oriented

experiments.[10] He discusses several design guides and compares the "Classical Procedure" with the "Statistical Procedure". Dixon and Mood describe their method of analysis for the Bruceton "Up and Down" technique.[11] An example of this technique is given in Appendix B. Edelman and Prairie in their study found that, for sample sizes of 30,50, and 100, the Bruceton method estimated the mean and standard deviation better than the Probit or One-shot methods.[12] Grant and Van Dolah discuss the application of normal curve methods to explosives testing using the "Up and Down" technique of testing.[13] They show that normal curve procedures can be applied to different explosive testing techniques.

II. MATERIALS

The major proportion of the impact sensitivity tests were performed on a standard mix consisting of potassium chlorate ($KClO_3$)/stablized red phosphorous (P_4)/Quso/magnesium oxide (MgO) in a 34/14/4/2 proportion. The $KClO_3$ and MgO were commercial reagent grade materials. The P_4 was stabilized by a small percentage of alumina (Al_2O_3), a neutralizing agent, to prevent acid formation between the P_4, air, and moisture. The Quso is a micro fine precipitated silica used as an absorbing agent, anti-caking agent, dispersant, and as a binder. Other mixtures with additives included Cab-O-Sil (a silica material similar to Quso), aluminum, Magnesium, Silica Gel, and Pyrex Glass. $KClO_3/P_4$ combinations included 5/2, 34/14, and stoichiometric (10/3) ratios.

A list of materials used in this investigation is tabulated in Table I.

The copper chlorotetrazole (CCT) was received near the end of the investigation. It had been stored in a 50/50 water/methanol mixture for an unknown period of time. The CCT was tested on a larger impact apparatus used in conjunction with this project. It was used for comparative purposes since published values for impact sensitivity were available.

TABLE I.

LIST OF MATERIALS

Materials:	Comments:
Stabilized Red Phosphorous (P_4)	Fuel
Potassium Chlorate ($KCLO_3$)	Oxident
Quso (SiO_2)	Moisture Control
Cab-O-Sil (SiO_2)	Moisture Control
Zinc Oxide (ZnO)	Neutralizer
Magnesium Oxide (MgO)	Neutralizer
Aluminum (Al)	Fuel
Magnesium (Mg)	Fuel
Pyrex Glass	Inert Additive
Silica Gel	Inert Additive
Copper Chlorotetrazole ($Cu(N_4CCl)_2$)	Primary Explosive
Freon 113	Desensitizing Agent

III. EQUIPMENT AND EXPERIMENTAL PROCEDURES

A. EQUIPMENT

A modified impact tester was developed due to the sensitive nature of the mixtures tested. The tester was designed as simply as possible from standard equipment which is readily available. Criteria for constructing impact testers have been extensively reported in the literature.

The apparatus consisted of a cast iron pipe, used as a tube, 36 centimeters long, with calibrated holes, clamped to a ring stand. The pipe had an inner diameter of 18/32 inch, while the outer diameter was 27/32 inch. The holes were drilled at 5 millimeter intervals and were 1/8 inch diameter. The pipe was calibrated to drop a steel ball a specified distance depending upon the height at which the ball was held in place by a pin inserted through one of the holes.

Standard steel ball bearings were used as the drop weight. They varied in diameter from 0.8 centimeters to 1.2 centimeters, and in weight from 3.45 grams to 6.87 grams. A standard ring stand was used to support the tube. The base of the stand was ground smooth and a V-shaped plexiglass spacer was glued to the base for positioning the sample holder.

The sample holder was a one-inch steel cylinder, two-inches in diameter, with a cup in the center. The cup was 1/2 inch diameter and 1/4 inch deep, and it was square bottomed.

Caps of hardened steel fit into the cup and rested upon the specimen. They were 5/16 inch deep and 1/2 inch diameter. The cup and cap were accurately machined to provide a running fit (0.008 inch).

TABLE II.

IMPACT APPARATUS WEIGHTS

Pin	2.45 g
Cap	6.9 g
Sample Holder	400.8 g
Impact Tester	971.5 g
TOTAL	1381.65 g

A plastic tube was clamped to the top of the tube. This tube directed both the ball and cap into a receptacle which prevented their damage or loss. Moreover, it decreased the deposition of solid products on the ball and cap.

B. DISCUSSION

The Impact Tester was constructed as simply as possible for several reasons. Ring stands, clamps, iron pipe, and ball bearings are readily available. Hence,

the apparatus was easily assembled and was very economical. The tester was not designed to be permanent for two reasons. First, as this was a modified type of impact machine, it was subject to change. For example, when initial testing reveals the probability distribution function, new tubes can be inserted with intervals approximating the standard deviation. Secondly, it was felt that a massive base was not necessary due to the small weight of the ball compared to the weight of the sample holder. Rebound studies (see Appendix A) show that there was a slight deviation with different bases.

An electromagnetic apparatus developed for the project did not function properly with the smaller weights. Hence, the pin arrangement was devised. Tubes, initially made from plastic and glass, were easily damaged. Thereafter, a cast iron pipe was used. The pin arrangement allowed the ball and cap to be directed into a plastic tube at the top of the cast iron pipe. The explosive force of the reaction shot the ball and cap through the tubes into a receptacle which prevented damage to these components.
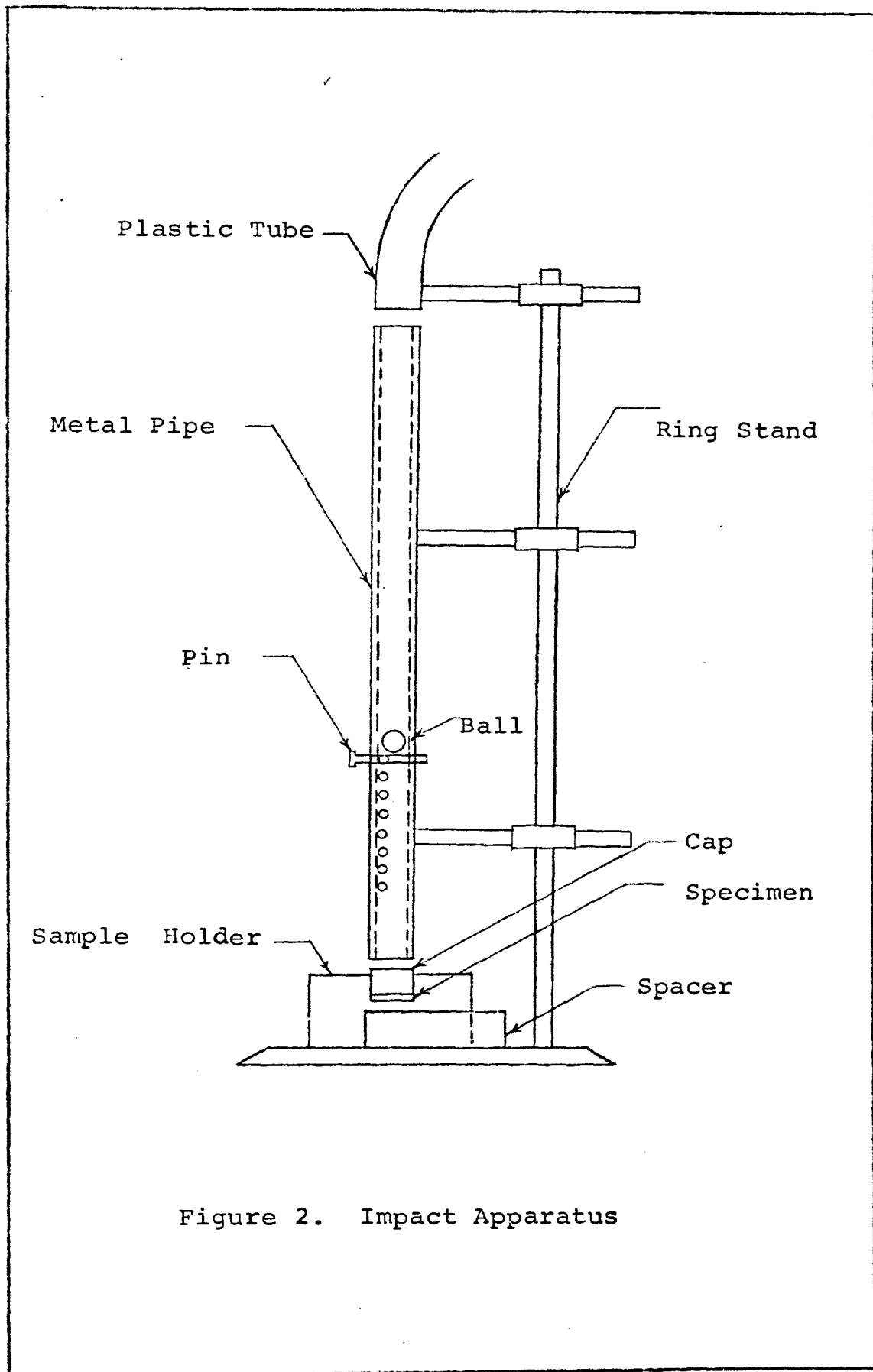
Figure 2. Impact Apparatus

C. TESTING

1. Sample Preparation

Handling, mixing and testing were performed by the author, so that conditions would not vary. The ingredients were weighed on a Mettler H6T Balance which gives a four decimal place accuracy. After weighing, the $KCLO_3$ was ground in a mortar. Freon 113, a desensitizing agent, was added to the $KCLO_3$. Quso and MgO were added next and throughly mixed with the $KCLO_3$. The stabilized red phosphorous was added last and blended with the other ingredients. The mixture was placed into an explosion proof oven set at 52° centigrade.

Drying time varied with the amount of freon. In general, one hour and thirty minutes was necessary for drying the mix thoroughly. However, in no case was the drying time less than three hours and usually the mix was allowed to dry overnight. The explosive mix was then removed from the oven and placed in a desiccator and allowed to cool to room temperature.

2. Test Procedure

A small spatula was used to remove the specimen from the sample and place it in to a calibrated scoop with a -1/2 to + 1 milligram deviation. The specimen was placed into the sample holder and spread to a constant depth.

A small tamper was used to press the mix in order to obtain a fairly constant density. The chlorate tended to clump into round particles which would absorb the energy of impact so that care was necessary to avoid this problem. The cap was then placed on top of the specimen by the use of tweezers.

The mix was tested as soon as possible after being removed from the desiccator so as to decrease the effect of humidity. Care was taken so that the loaded sample holders were not jarred. The specimen was then placed on the impact tester and positioned by means of a V-shaped spacer glued to the stand. The pin was pulled and the ball dropped onto the cap. The results were recorded as either a GO or as a NO GO (reaction or no reaction). There was no difficulty in determining if a reaction had occured. Humidity and temperature were controlled during the testing period.

If the sample did not explode, the used mix was collected into a receptacle for further disposal. If a reaction did occur, the sample holder, cap, and ball were cleaned. A cotton swab and water were used since the products of the reaction are water soluble. The smoke generated from the reaction was pulled from the test booth by a fan which was vented to the outside of the building.

D.   PROBLEMS

When the explosive mixture dried, it was filled throughout by pockets of air due to the method that the freon took to evaporate from the mix. The particles of $KCLO_3$ coagulated into small circular shapes, which were coated very lightly with the phosphorous. Thus, it was difficult to obtain an intimate mixture between the components of the mix. Moreover, the clumped particles separated from the mix while handling thereby changing the ratio of the components. These particles would absorb the energy of impact thereby requiring higher energy levels for initiating the mixture. Care had to be exercised so that this problem was minimized.

Although the freon desensitizes the mix, it does not insensitize it. Extreme care must be used when working with these mixtures due to their unpredictable nature. Less than five grams of standard mix can shatter a standard laboratory mortar.

The equipment rapidly became worn and deformed. A constant supply of caps was necessary for adequate operations. Indentations and nicks had to be filed so that smooth surfaces were used for testing. The cup and caps were accurately machined in order to prevent lateral movement of the cap with respect to the cup, so as to obtain reproducible results.

Enough lateral space had to be available for the ball to free fall after the pin was removed. This created two problems. First, the ball leaned against the side of the tube, thereby slightly changing the drop height. Secondly, friction occurred between the ball and the tube. It was noted, during the rebound studies, that this friction resisted any rotational energy being imparted to the ball. Thus, the ball would fall freely.

E.   SAFETY

Special precautions had to be considered when working with the standard mix since energies as low as seven gram-centimeters, approximately 0.7×10^{-3} joules, have caused reactions. Values as low as 1.35×10^{-3} joules initiated a reaction when using the electrostatic discharger test. Since it was difficult to determine the actual energy imparted to the standard mix due to system losses by the electrostatic tester, these values are undoubtably higher than the actual energy needed for initiation.

Amicone, et al., reported that twenty to thirty thousand volts potential can be expected to develop on a human being depending upon certain conditions.[14] Brown, et al., reported that a charge of 0.015 joules is a reasonable value that a person can generate when he is charged to 10,000 volts.[15] This figure is more than

nine times the amount of energy required to initiate a 50%,or median point,reaction of the standard mix. Moreover, it is approximately three times the amount of energy required for initiation by the electrostatic tester used in conjunction with this project. Static conductive linoleum, equipment and wearing apparel are necessary in order to reduce this electrical hazard.

Safety wearing apparel included goggles, face shields, ear muffs, conductive gloves, aprons, boots and heel protectors. Non-synthetic clothes were worn during testing.

One-half inch plexiglass shields were used as a front cover for the work tables and test booths. Arm holes cut in the lower corners of the plexiglass enabled the operator to be protected from the explosive, but yet facilitated handling, mixing and testing. Conductive linoleum covered the working area and was also used as a floor covering. This enabled the operator to always remain at the same potential as the explosive. The linolium and all test apparatus and equipment was grounded by solid copper wire.

The ball was inserted into the tube before the sample holder was placed into position. The pin was pulled from the tube by using tongs so that the hands would be behind the plexiglass shield when the reaction occurred. Care was exercised in handling the loaded sample holders so

that they were not jarred.

    The force of the reaction propelled both the ball and cap into a plastic tube attached to the top of the impact tester. The tube directed them into a receptacle so that they were not damaged or lost. This also prevented potential injuries to personnel.

IV.   RESULTS OF TESTING

A summary of the results obtained from the small impact test is presented in Table III. A plot of relative humidity versus the energy level is given in figure 3. Figures 4, 5, and 6 give the relationships between the energy level, the drop weight, and the test height.

TABLE III.

TESTING RESULTS

TEST NO.	SAMPLE MIXTURE (PARTS) $KCLO_3$	P_4	CAB-O-SIL	ENERGY (g-cm)	HEIGHT (cm)	BALL WEIGHT (g)	SPECIMEN SIZE (mg)	TEMP (°C)	REL HUM	NO.IN TEST
1 A	34	14	1	---(a)		3.81	4-5	---	---	20
2 A	34	14	1	16.8	4.42	3.81	4-5	---	---	30
3 A	5	2		10.1	2.85	3.53	16-17	---	---	20
4 A	5	2		11.3	3.20	3.53	10	---	---	20
5 A	5	2		12.9	3.65	3.53	20	---	---	20

	SAMPLE MIXTURE (PARTS) $KCLO_3$	P_4	QUSO	M_gO	ENERGY (g-cm)	HEIGHT (cm)	BALL WEIGHT (g)	SPECIMEN SIZE (mg)	TEMP (°C)	REL HUM	NO.IN TEST
1	34	14	4	2	19.1	5.40	3.53	20	---	---	20
2	34	14	4	2	15.1	2.20	6.87	20	---	---	20
3	34	14	4	2	15.7	4.45	3.53	20	---	---	20
4	34	14	4	2	13.0	3.70	3.53	25	---	---	20
5	34	14	4	2	7.8(b)	2.26	3.47	25	---	---	35
6	34	14	4	2	6.1(b)	1.77	3.47	25	24	---	35
7	34	14	4	2	15.3	2.23	6.87	9	22.2	---	35
8	34	14	4	2	16.2	2.36	6.87	15	21.2	---	35
9	34	14	4	2	14.0	2.04	6.87	4.5	21.8	---	35

(a) Test run
(b) Unconfined

TABLE III (continued)

TEST NO.	SAMPLE MIXTURE (PARTS)				ENERGY (g-cm)	HEIGHT (cm)	BALL WEIGHT (g)	SPECIMEN SIZE (mg)	TEMP (°C)	REL HUM	NO. IN TEST
	$KClO_3$	P_4	QUSO	M_gO							
10	34	14	4	2	22.9[c]	3.33	6.87	15	23.2	---	35
11	34	14	4	2	20.9[c]	6.03	3.47	15	21	49.5	35
12	34	14	4	2	35.3[d]	5.14	6.87	30	26	Inc.	35
13	10	3	-	-	38.0[e]	5.54	6.87	20	27.8	---	28
14	10	3 + 5% Al	-	-	41.7[f]	6.07	6.87	20	24	---	21
15	34	14 + 5% Al	4	2	54.2[g]	7.89	6.87	20	28	Inc.	35
16	34	14 + 5% Mg	4	2	61.2[h]	8.90	6.87	20	22.5	Nor.	36
17	34	14 + 46% Silica gel	4	2	60.7[i]	8.83	6.87	20	24	Nor.	35
18	34	14 + 46% Silica gel	4	2	57.3[i]	8.34	6.87	20	23.5	Nor.	34
19	34	14			56.2[j]	8.19	6.87	20	25	Nor.	16

(c) Aged 7 days
(d) Raining
(e) Stoichiometric mix
(f) Stoichiometric mix with 5% Al added
(g) 5% Al added
(h) 5% Mg added
(i) 46% Silica gel added
(j) Accident test discontinued

TABLE III (continued)

TEST NO.	SAMPLE MIXTURE (PARTS)				ENERGY (g-cm)	HEIGHT (cm)	BALL WEIGHT (g)	SPECIMEN SIZE (mg)	TEMP (°C)	REL HUM	NO.IN TEST
	$KCLO_3$	P_4	QUSO	M_gO							
20	34	14	4	2	$70.7^{(k)}$	10.3	6.87	20	26	Nor.	10
	+ 46% Pyrex glass										
21	34	14	4	2	46.6	6.79	6.87	23-24	24.5	45.6/ 48.5	36
22	34	14	4	2	29.4	4.28	6.87	23-24	25	35/39	48
23	34	14	4	2	18.1	2.64	6.87	23-24	26.6	28.5/ 32	75
24	34	14	4	2	20.2	4.50	4.47	23-24	24	30/39	75
25	34	14	4	2	$---^{(l)}$	---	6.87	23-24	24.8/ 25.8	27/38	120
26	34	14	4	2	$23.2^{(m)}$	3.38	6.87	23-24	23/24	24/26	75
27	CCT				$940^{(n)}$	38.0	24.76	11	25.6	30	26
28	CCT				$940^{(n)}$	38.0	24.76	11	23.3	46	25

(k) Discontinued test 46% Pyrex glass added
(l) Probit or rundown test-20 tests at 6 levels
(m) Mix aged for 30 days
(n) Copper Chlorotetrazole

Composition of standard mix

$KCLO_3$	P_4	QUSO	MgO
34	14	4	2
62.96%	25.93%	7.41%	3.70%

Inc. indicates increasing
All tests were confined except No. 5
and 6 Rel. Hum. and Temp. not marked
were at ambient conditions

Combined 50% point energy of 290 test values
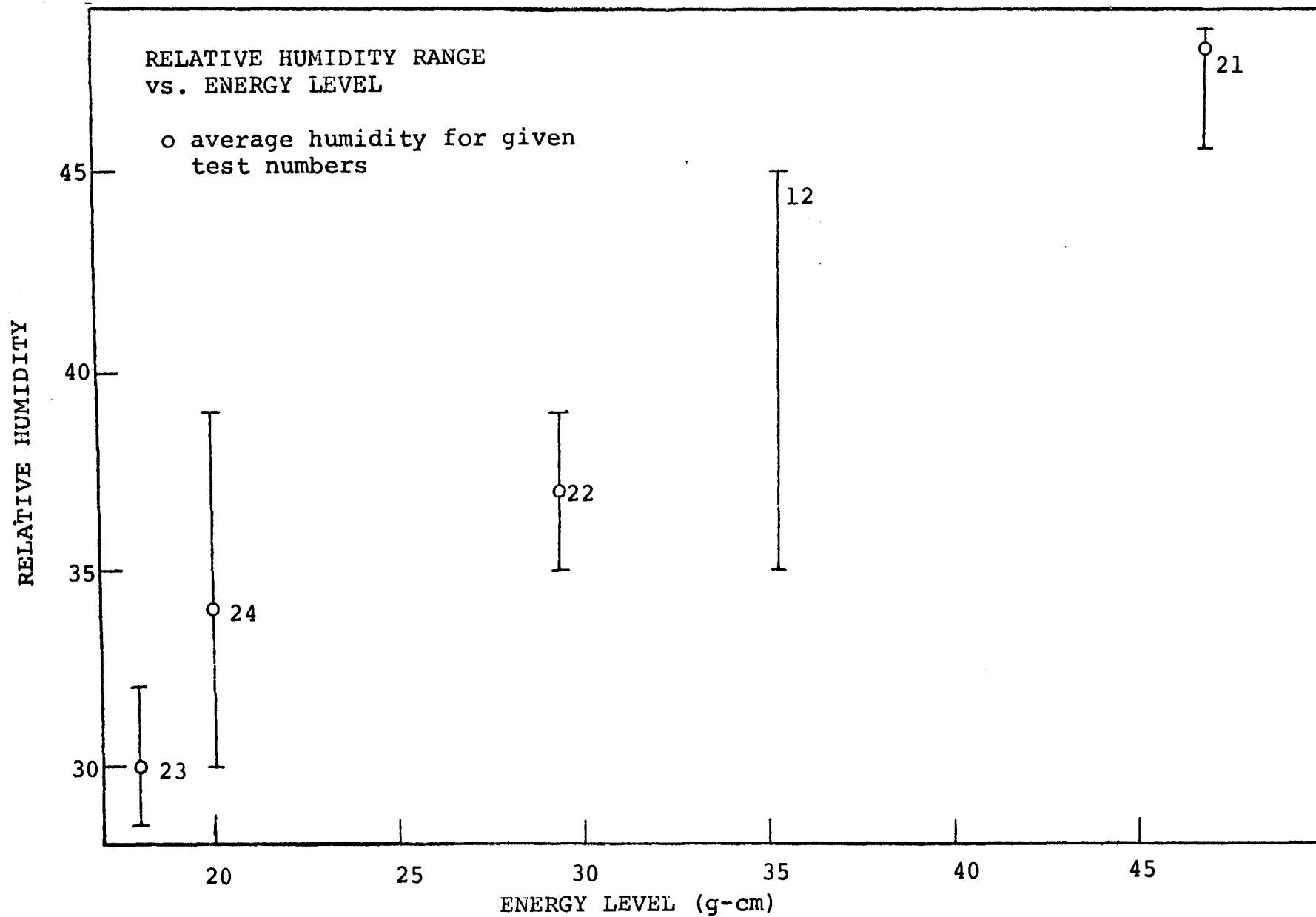
$\bar{x}$ = 17.6 g-cm

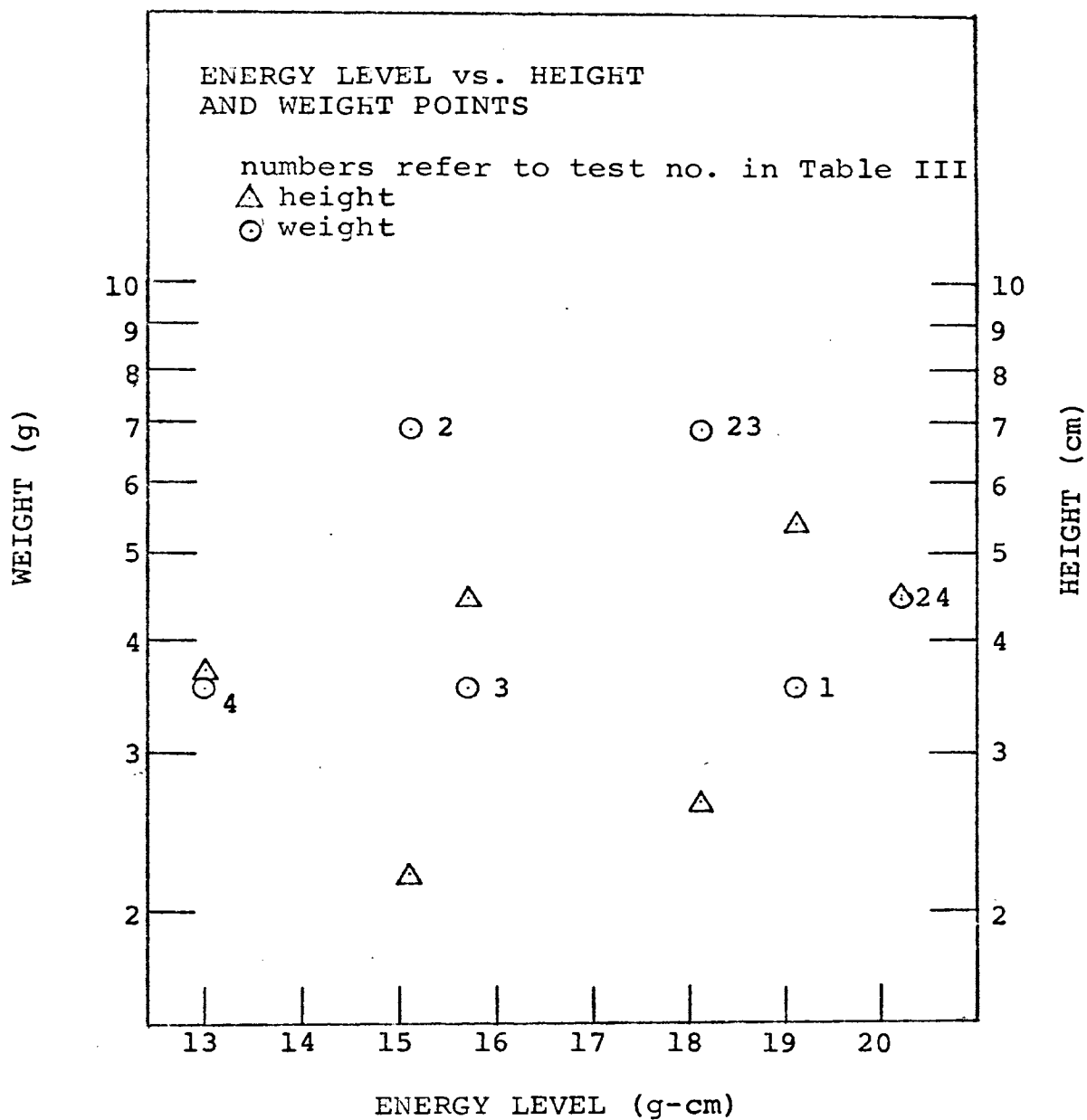Figure 3. Humidity vs. Energy

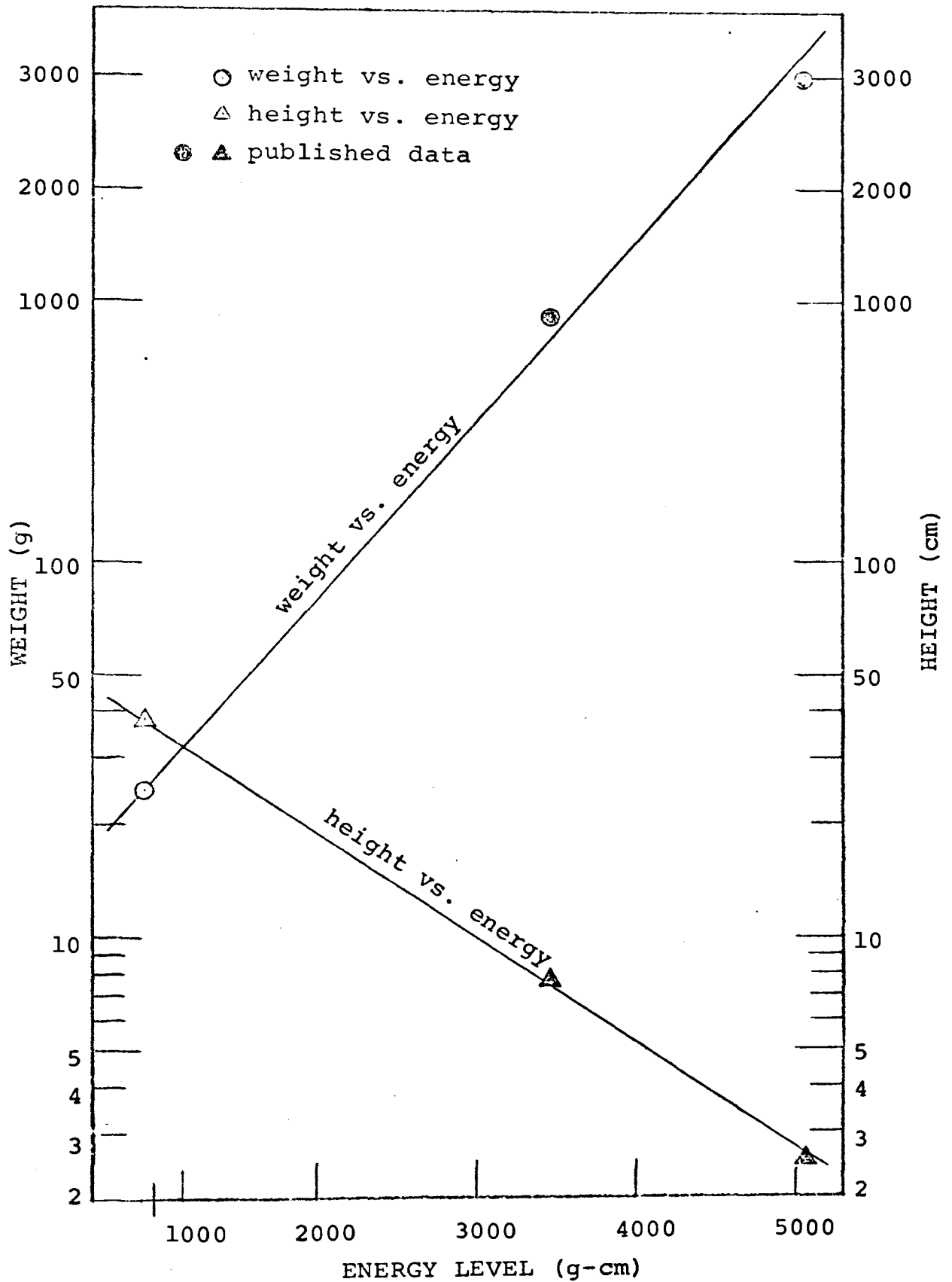Figure 4. Energy vs. Height and Weight - Small Impact Test
(semilog plot)

Figure 5.   Energy vs. Height and Weight of Copper
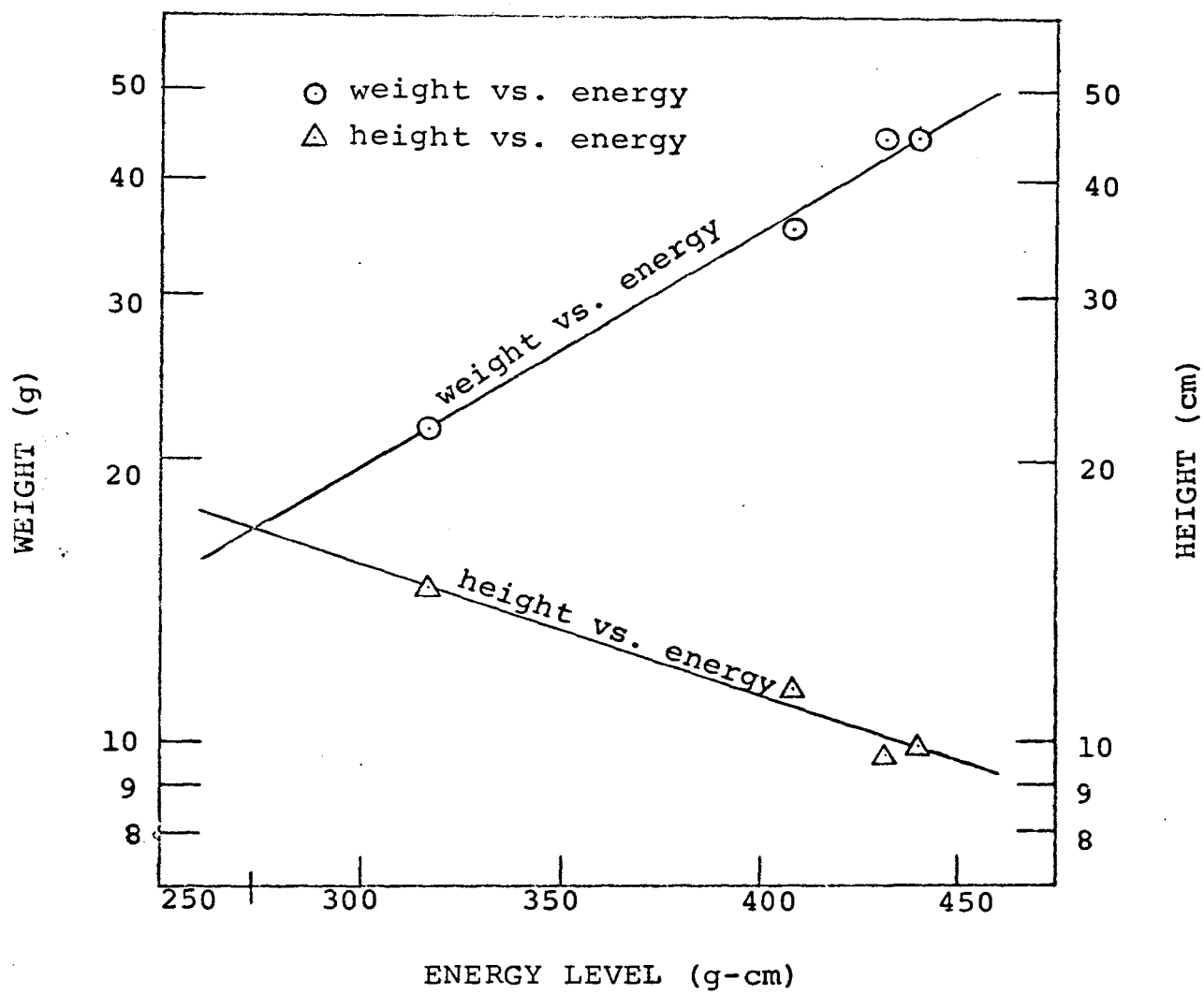Chlorotetrazole - Large Impact Test

Figure 6.   Energy vs. Height and Weight of the Standard Mix -
Large Impact Test

V.   DISCUSSION OF RESULTS

A.   GENERAL

Tests were conducted in order to determine the effects of confinement, aging, humidity, number of specimens in a test, reproducibility, specimen weight, additives, and stoichiometric changes.

Tests 5 and 6, which were unconfined, had 50% energy points less than one-half of the confined values. Some partial reactions were observed at the lowest energy levels.

Tests 10 and 11 were made on material that had been stored under ambient conditions for one week. Test 26 used material that had been aged for 30 days. Comparison showed a slight desensitization due to storage.

As time progressed, it became apparent that humidity was having an effect on the energy required for initiation. This is shown in figure 3. As the relative humidity increased the energy level also increased in an approximately linear manner within this range of values. Tests 21 and 22 were performed to show this effect.

It was noted that consecutive series of go's or no go's were occurring during the earlier tests. Therefore, it was decided to increase the number of specimens in each test. Accordingly, they were increased from 20 to 35 and then to 75. This requirement was especially necessary when additives were used. Estimation of the mean became erratic and it could not be predicted. The increased number was

also necessary for reproducibility. Weighted energy means
comparing tests 1, 2, 3, and 4 with tests 23 and 24 in com-
binations (see Table IV) showed no significant differences.
The weighted means showed that the lower ball weights tended
to give slightly higher energy values although the differ-
ences were not significant. The scatter about the average
50% point energy value (18 g-cm) within the standard devia-
tion (±5 g-cm) is presented in figure 4. This is discussed
in more detail under section B on statistical analysis.

Null Hypothesis Tests were made on tests 7, 8, and 9
which varied the specimen weight from 4.5 to 15 milligrams.
Energy levels (gram-centimeters) were tested for comparing
9 with 7 and 8 with 7. For a 95% confidence interval, the
analysis showed that tests 9 and 7 were significantly
different while 8 and 7 were not. Test 12 was made with a
30 milligram specimen size. The higher 50% energy value
indicates a cushioning, energy absorbing effect of large
specimen sizes. This procedure excluded the use of very
small or large specimen sizes. Thereafter, tests were con-
ducted with sizes of 20 or 23-24 milligrams. The larger
specimen sizes also caused extensive damage to the caps.

Tests 14 through 20 were not performed on the stan-
dard mix. These tests show the desensitizing effect of the
additives. The addition of pyrex glass and silica gel
caused an increase in the 50% initiation energy due to
the cushioning effect of the large particle size used in
the relatively small sample weights. Other tests used in

conjunction with this project gave lower energy values when these inert materials were added to larger specimen sizes.

Small changes in the stoichiometry of the standard mix were not undertaken due to the large effect of the additives. However, it can be noticed that fuel-rich mixtures are more sensitive by comparing tests 2A, 3A, 4A, 5A, and 13. The 50% point energy level of test 13, the stoichiometric mix, is about double that of the standard mix.

A probit or rundown test (No. 25) was performed in order to check the energy level and distribution of the standard mix. However, this test gave erratic results. Test runs which used material straight from the oven tended to be more sensitive and erratic than material which had been cooled to room temperature. Tests were performed at ambient temperature for consistency.

It is not clear that the variation in ball weight has an effect upon the energy level within the small test range used for the standard mix (Figure 4). However, Figure 5 which compares energy with the drop weight and height shows that there is a definite effect for the copper chlorotetrazole. The lower energy was determined from the large impact apparatus used in conjunction with this project. The two higher energy levels were taken from published data.[16] For comparative purposes it would be preferable to test at the cross over point.

Figure 6 is a similar comparison for the standard mix using the large impact tester. However, notice that this

trend does not continue into the lower energy values as
determined by the small impact tester (Figure 4). The
large impact apparatus used 230 milligram specimens and a
44.6 g ball weight compared to the 20 to 24 milligram speci-
mens and 3.47 to 6.87 g ball weight used in the small im-
pact test. This confirms the energy absorbing effect of
the larger specimen sizes. Further, it also indicates that
momentum is one of the controlling parameters in these
tests.

In comparing the trends of figures 5 and 6, it would
seem that momentum considerations would be crucial. The
lower energy levels of figure 4 can not be extended and com-
pared to the trends of figures 5 and 6 since the testing
parameters are different. Momentum has a definite effect
over large test ranges. The total energy required appears
to be a function of the specimen weight, the ball weight,
the relative humidity, and other variables.

B. STATISTICAL ANALYSIS

The results of several tests were plotted on probabili-
ty paper. Normal and log-normal plots were made on tests
23 and 24, 23 and 24 combined, and with tests 7, 8, 9, 11,
23, and 24 combined. Plots were made with energy levels
and heights on the GO's, NO-GO's, and total observations.
These plots showed that within the testing ranges the re-
sults obtained from the standard mix were both normally
and log-normally distributed. Hence, Chi-square tests of
these plots were calculated. The theoretical points were

determined from the straight lines drawn through the actual points. These tests determined that it is not possible to reject the hypothesis that the distribution is either normally or log-normally distributed over the test range. The log-normal plots showed the least amount of deviation, especially near the extreme percentages. Therefore, it is suggested that the testing results of this mixture are log-normally distributed over the test range. However, the normal plots showed that the normal distribution extended through two standard deviations. This allowed normal methods for calculating heights and energies. Standard deviations were also calculated. The results show that the testing interval of 5 millimeters is less than one standard deviation for both the lighter (4.5 gram) and the heavier (6.87 gram) balls. This fact would account for some of the longer consecutive series of go's and no go's encountered during testing.

Combining tests 23 and 24 gave a calculated mean of 18.65 ± 5.5 gram-centimeters. Values from a normal plot and a log-normal plot are 17.8 ± 4.5 g-cm and 16.5 ± 5.0 g-cm, respectively. Results calculated from 290 tests gave a calculated mean of 17.6 ± 4.8 g-cm, a normal median of 16.2 ± 4.6 g-cm and a log-normal median of 15.3 ± 4.7 g-cm. A weighted mean consisting of tests 1, 2, 3, 4, 23, and 24 gave a value of 17.9 g-cm. Therefore, the 50% energy level is about 18 ± 5 gram-centimeters.

A table of mean heights, energies, and standard deviations is tabulated in Table IV.

TABLE IV.

LIST OF HEIGHTS, ENERGIES, AND STANDARD DEVIATIONS

INCLUDING T-TEST AND CHI-SQUARE TESTS

TEST NO	NO IN TEST	BALL WEIGHT (g)	50% HEIGHT (cm)	SIGMA	50% ENERGY (g-cm)	SIGMA	CHI^2 TEST	T-TEST
23	75	6.8728	2.640 [a]	.758	18.144	---		.173
			2.583 [b]	1.419				
			2.42 [c]	.720			1.55<11.1	
			2.24 [d]	.830			2.40<11.1	
24	75	4.4720	4.500 [a]	.852	20.124			.196
			4.507 [b]	2.176				
			4.27 [c]	.857			.89<11.1	
			4.14 [d]	.933			1.33<11.1	
23 &24	150				18.65 [e]	5.45		
					17.8 [c]	4.79	3.61<11.1	
					16.5 [d]	5.02	2.66<11.1	
--	290				17.6 [f]	4.76		
					16.2 [c]	4.65	1.27<12.6	
					15.3 [d]	4.74	3.18<12.6	

(a) Calculation by normal curve methods
(b) Calculation by Dixon-Mood technique
(c) Plot on normal probability paper
(d) Plot on log-normal probability paper
(e) Test 23 & 24 combined and grouped by energy levels
(f) Tests 7, 8, 9, 12, 23, and 24 combined and grouped by energy levels

TABLE IV (continued)

TEST NO	NO IN TEST	BALL WEIGHT (g)	50% HEIGHT (cm)	SIGMA	50% ENERGY (g-cm)	SIGMA	CHI^2 TEST	T-TEST
1,3, 4&24	135	3.53 & 4.47			18.3 [g]			
2&23	95	6.87			17.5 [h]			
--	230				17.95 [i]			

(g) Weighted mean of lower ball weights
(h) Weighted mean of 6.87 ball
(i) Total weighted mean of tests 1, 2, 3, 4, 23, and 24

VI. CONCLUSIONS

Under laboratory conditions (78 - 80°F and 25 - 35% relative humidity), the energy value for a 50% initiation sensitivity was found to be about 18 ±5 gram-centimeters for the standard mix. Testing results show a log-normal distribution. However, the results obtained from the standard mix are also normally distributed about the mean. As the specimen size increased, the sensitivity of the mixture decreased. For specimens greater than or equal to 30 milligrams, a dampening effect was observed such that larger amounts of energy were required for initiation.

Fuel rich specimens of the $KClO_3/P_4$ mixture were found to be more sensitive than the stoichiometric mixture. Additives, such as aluminum, magnesium, silica gel, and pyrex glass desensitized the mixture. Aging and humidity also increased initiation energies.

Energy versus drop weight and test height curves suggest that momentum considerations are important over large energy ranges. That is, the drop weight velocity and the rate of energy absorption are important variables. For the smaller weights, the velocity is higher and the energy is lower, as the impact velocity increases the kinetic energy required for reaction decreases. There is a critical velocity necessary to fire 50% of the specimens depending on the drop weight and energy absorbed. There is possibly more than one impact necessary for a reaction to

occur although this is unlikely for the standard mix. The intersections of the curves in figures 5 and 6 represent the weight and height values most desirable for reproducibility and comparability of different reactive materials by this test method.

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VITA

Carl Edward DeLong was born on July 9, 1938, in Hazel Park, Michigan, where he received his primary and secondary education. He has received his college education from the University of Detroit, in Detroit, Michigan, and from Wayne State University, in Detroit, Michigan.

After working in industry for two years, he returned to college at the University of Missouri-Rolla and received his Bachelor of Science Degree in Civil Engineering on May 28, 1967.

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APPENDIX A

Rebound Performance of Impact Tester

Herein are presented rebound versus drop height relationships for various bases. Four different bases were tested. They were: (1) the actual test booth, (2) a massive steel base, (3) a wood table, and (4) a formica covered wood cabinet.

The sample holder was inverted and a drilled glass tube was used. Each plotted point is an average of 20 determinations.

Figures 7 and 8 compare an unused ball with a used ball of the same weight and with a lighter ball. They show that within the actual testing range (0.5 centimeters to 8.0 centimeters) that the rebound values are not significantly different. Figures 9 to 12 compare drop height with rebound height for the four bases.

The coefficient of restitution (e^2) is defined as the ratio of rebound height to drop height. Ideally, the deviation from the theoretical value should be a straight line. Thus, the greater the deviation, the lower the efficiency of the apparatus. The impact tests on the standard mix have been on the lower part of the curves, from 0.5 to 5.0 centimeters.

$$V_{ai} - V_{bi} = e(V_{af} - V_{bf})$$

$$\tfrac{1}{2}M_a V_{ai}^2 + \tfrac{1}{2}M_b V_{bi}^2 = \tfrac{1}{2}M_a V_{af}^2 + \tfrac{1}{2}M_b V_{bf}^2$$

$$M_a g H_i = \tfrac{1}{2}M_a V_{ai}^2 \quad \text{and} \quad M_a g H_f = \tfrac{1}{2}M_a g V_{af}^2$$

where

$$V_{ai} = \sqrt{2gH_i} \quad \text{and} \quad V_{af} = \sqrt{2gH_f}$$

$$e = \frac{V_{ai}}{V_{af}} = \frac{\sqrt{2gH_i}}{\sqrt{2gH_f}} = \frac{\sqrt{H_i}}{\sqrt{H_f}}$$

hence

$$e^2 = \frac{H_i}{H_f}$$

i = initial   H = height

f = final or rebound   V = velocity

a = ball   m = mass

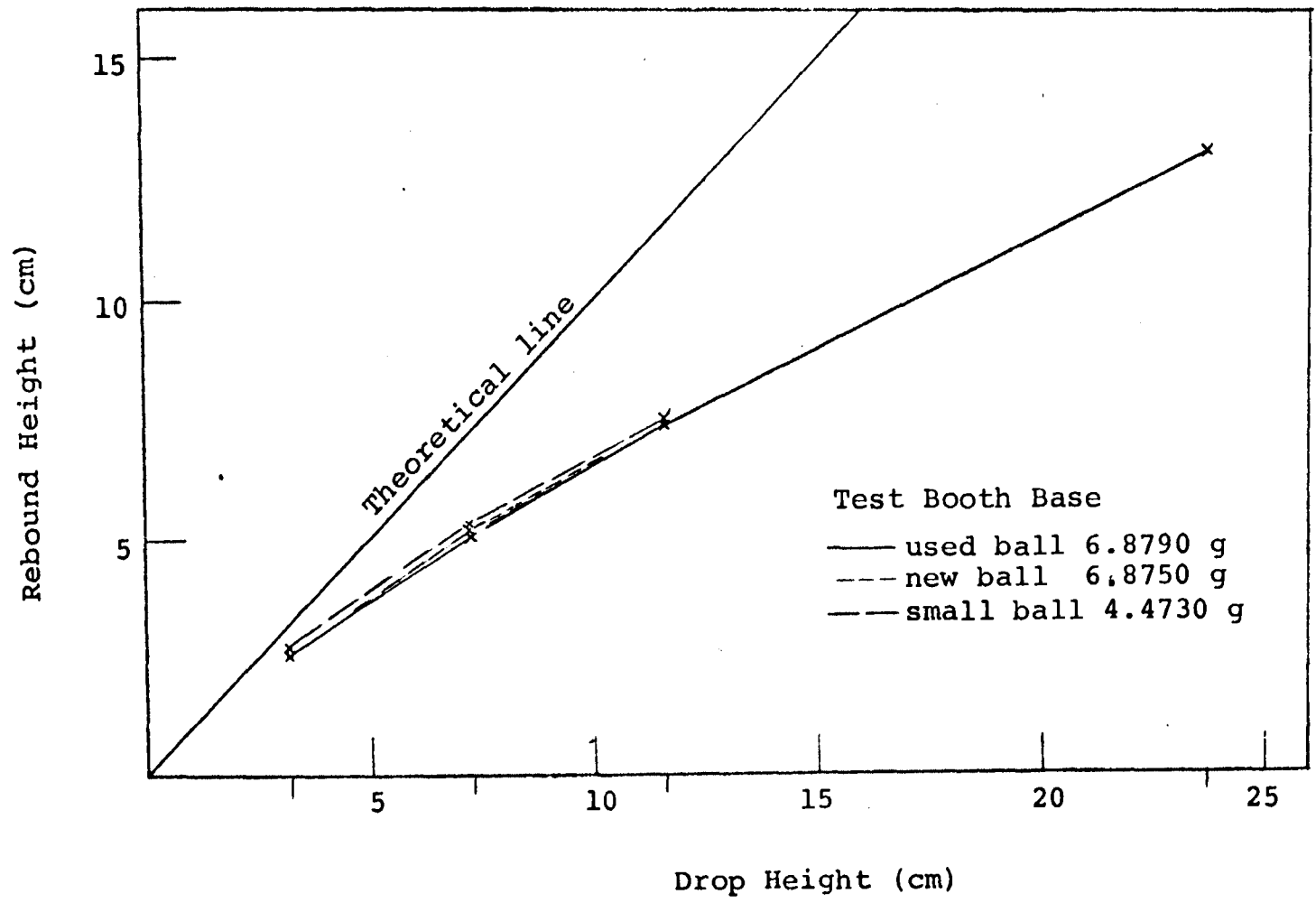b = sample holder   e = coefficient of resti-
                                   tution squared
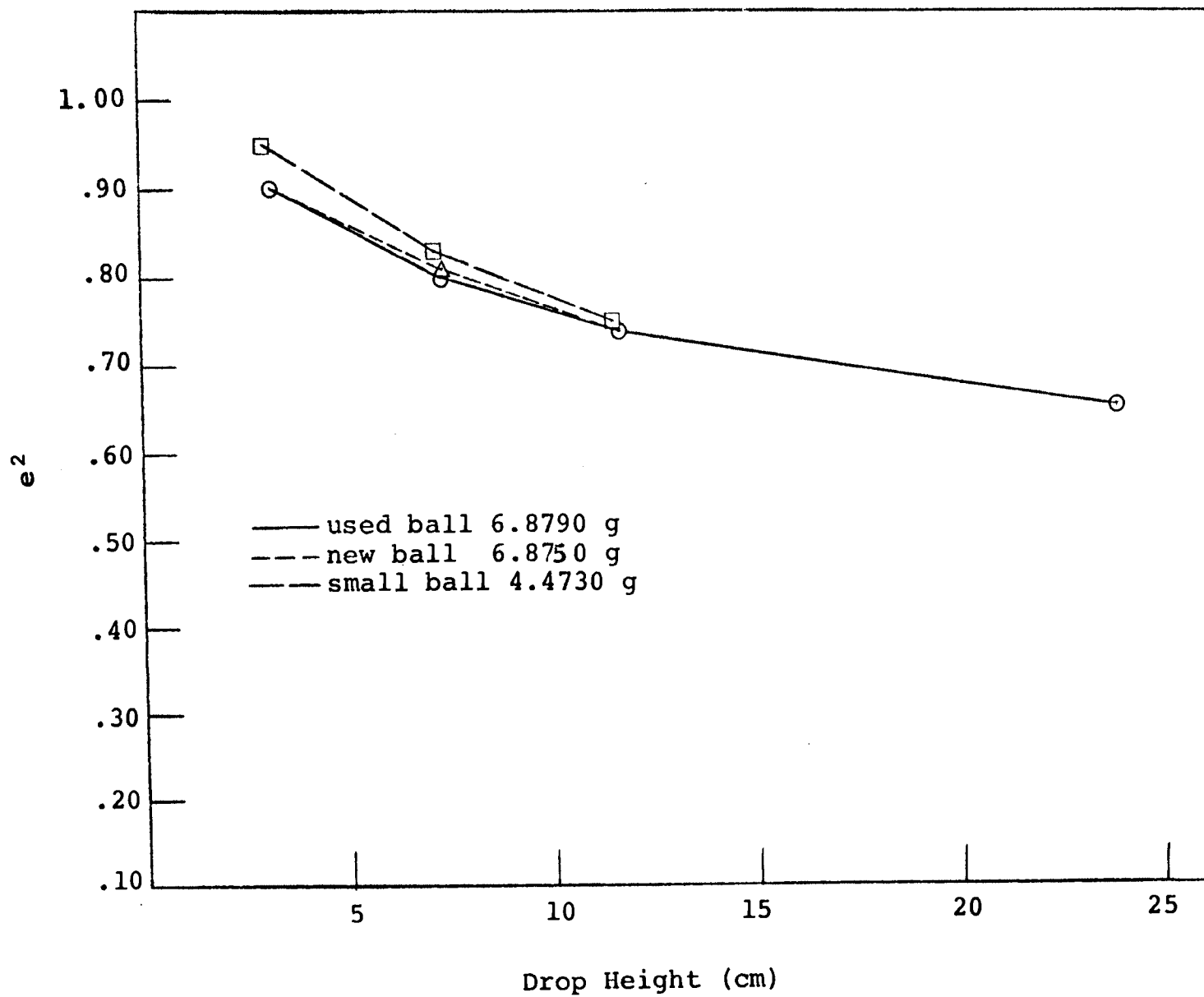
Figure 7.   Rebound vs. Drop Height for Different Balls
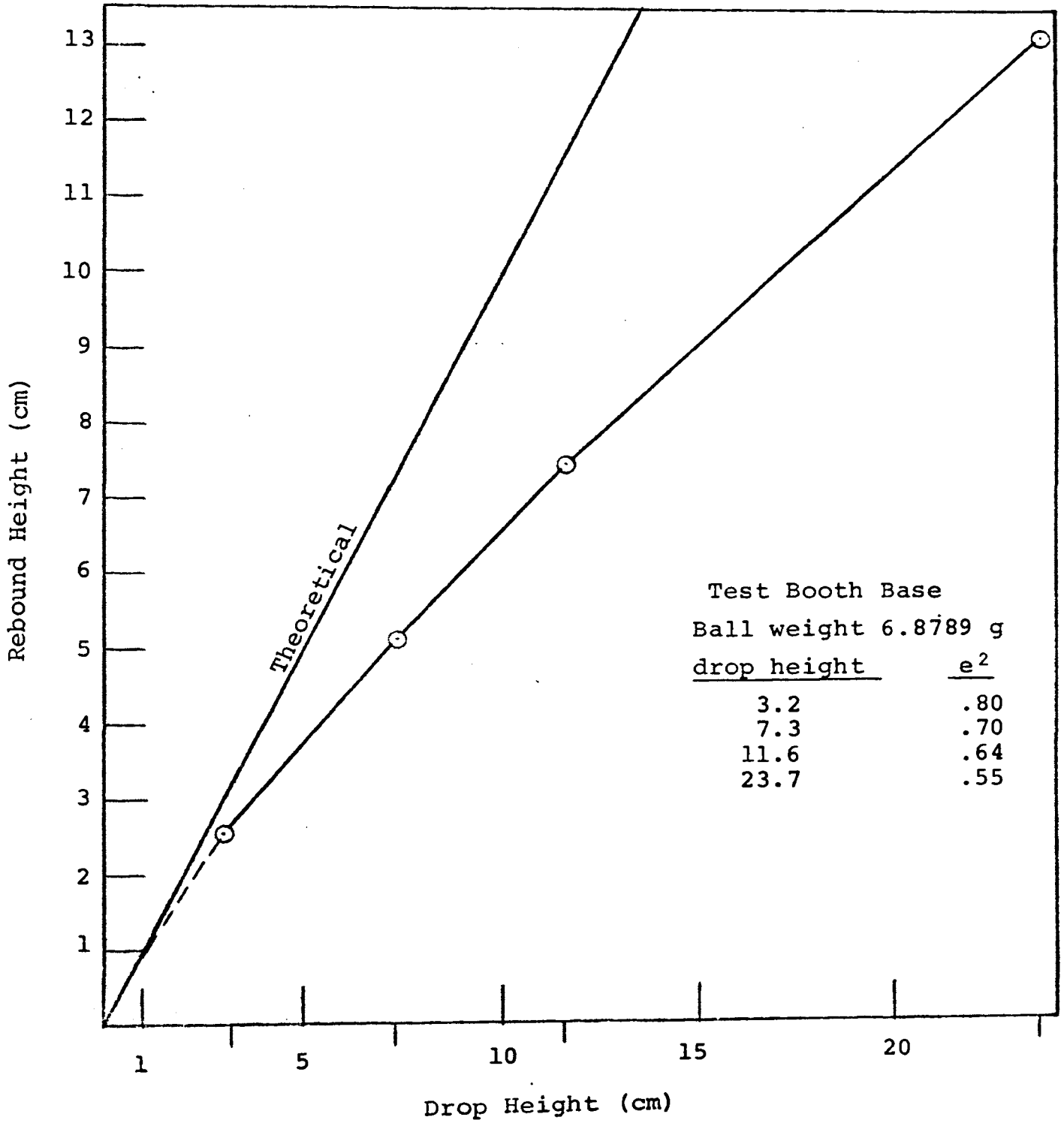
Figure 8. e^2 vs. Drop Height

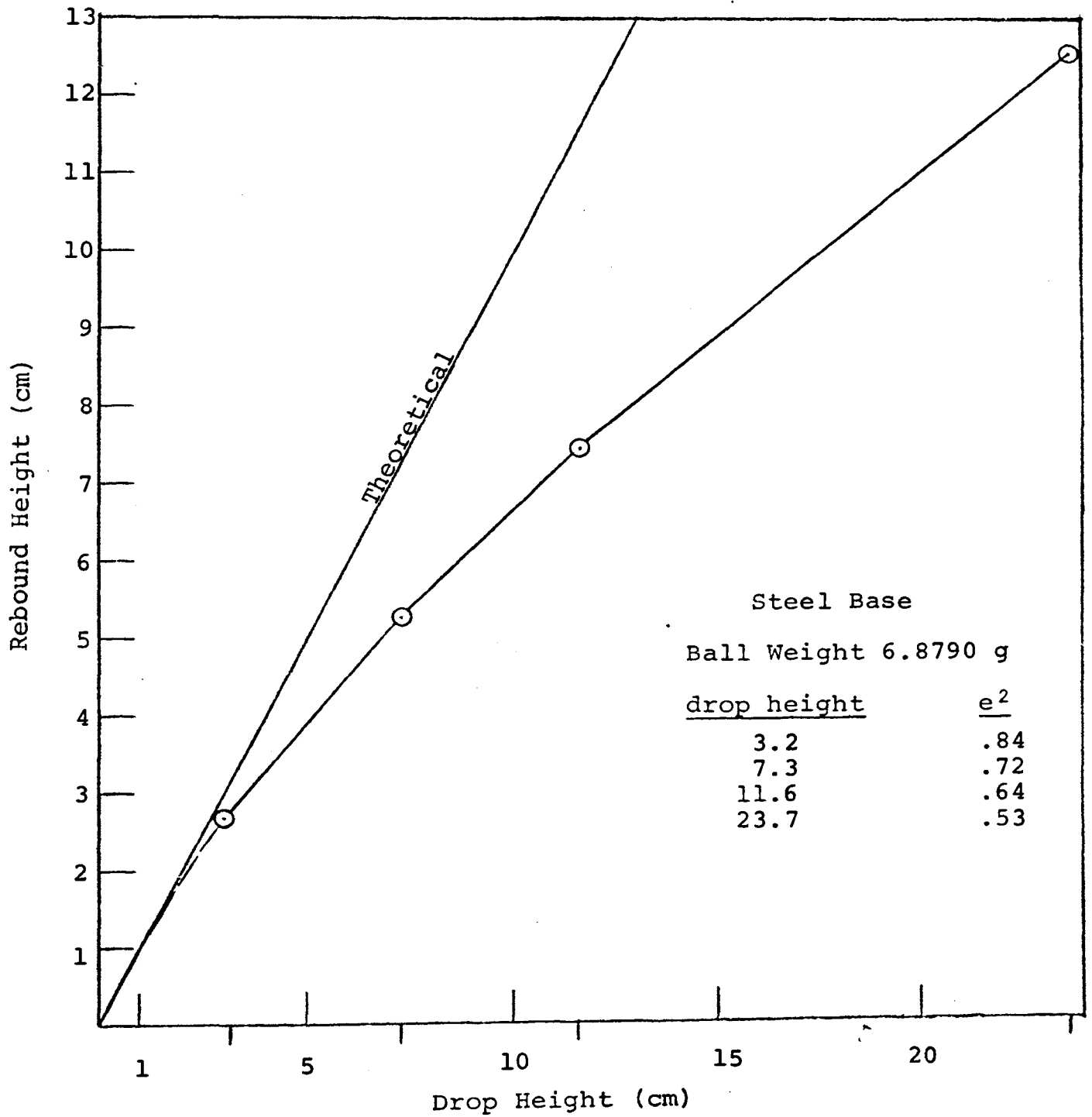Figure 9. Rebound vs. Drop Height - Test Booth Base

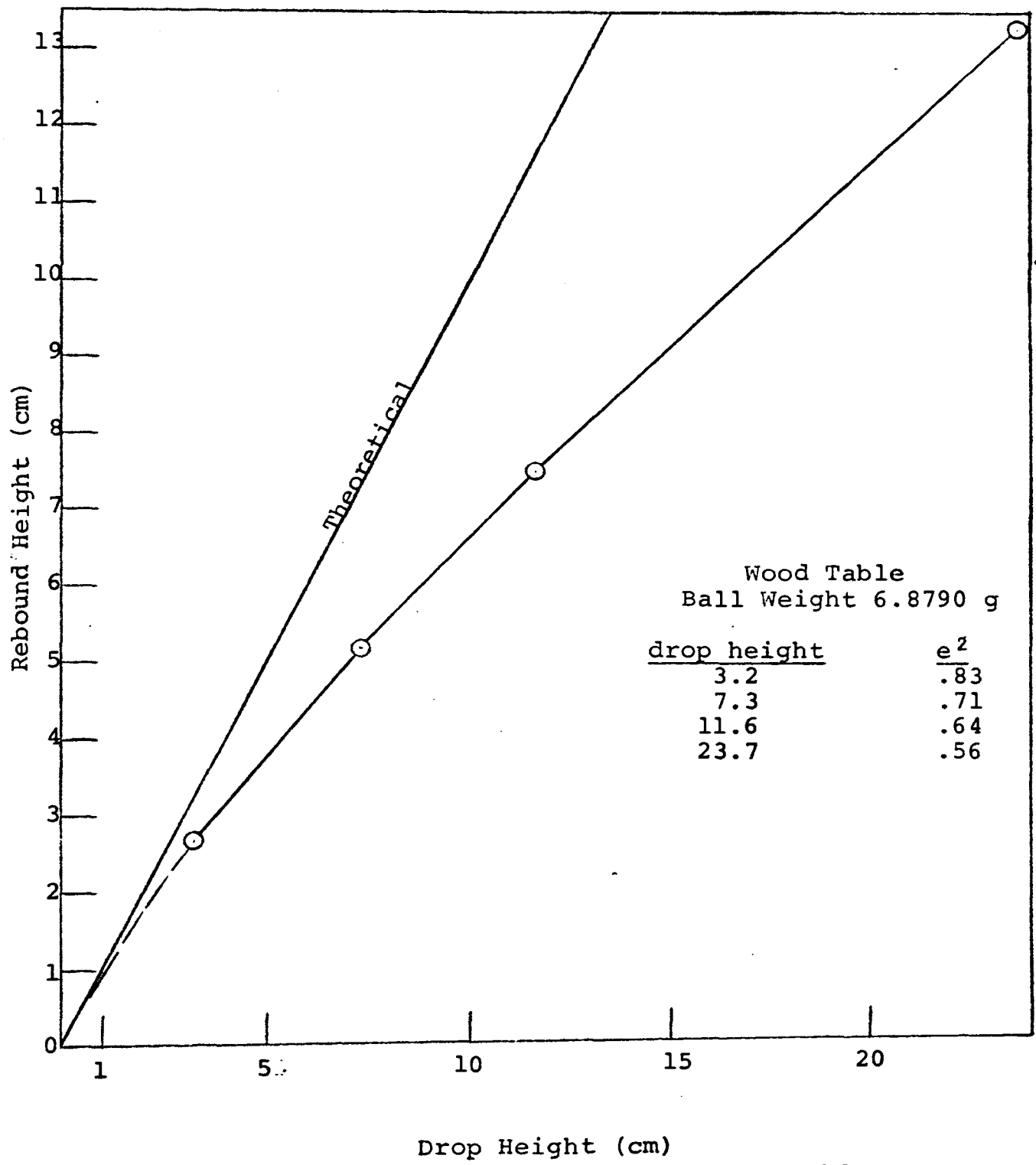Figure 10. Rebound vs. Drop Height - Massive Steel Base

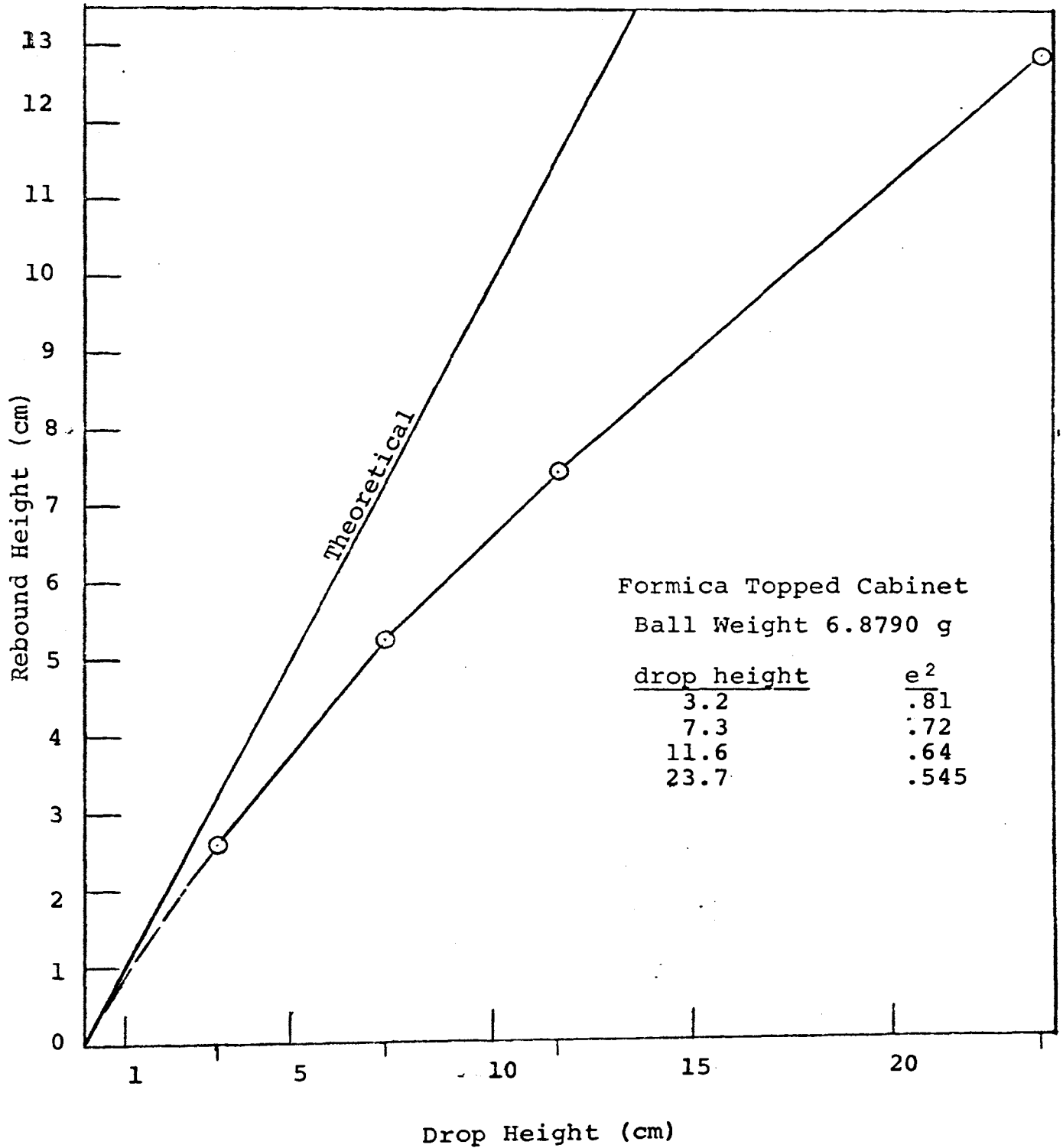Figure 11. Rebound vs. Drop Height - Wood Table Base

Figure 12. Rebound vs. Drop Height - Formica Topped Cabinet

APPENDIX B
Method of Analysis

Herein are included calculations and figures pertaining to Test 24. Figure 13 is a graph of the actual test which includes the actual testing conditions. Figure 14 is a normal probability plot of Test 24 and Figure 15 is a log-normal plot.

Calculations of the sample mean were performed by the Dixon-Mood method[1] and by normal curve methods[2]. Also included are a chi-square test[3] for determining the distribution of the function and a t-test for determining the 95% confidence interval of the mean.
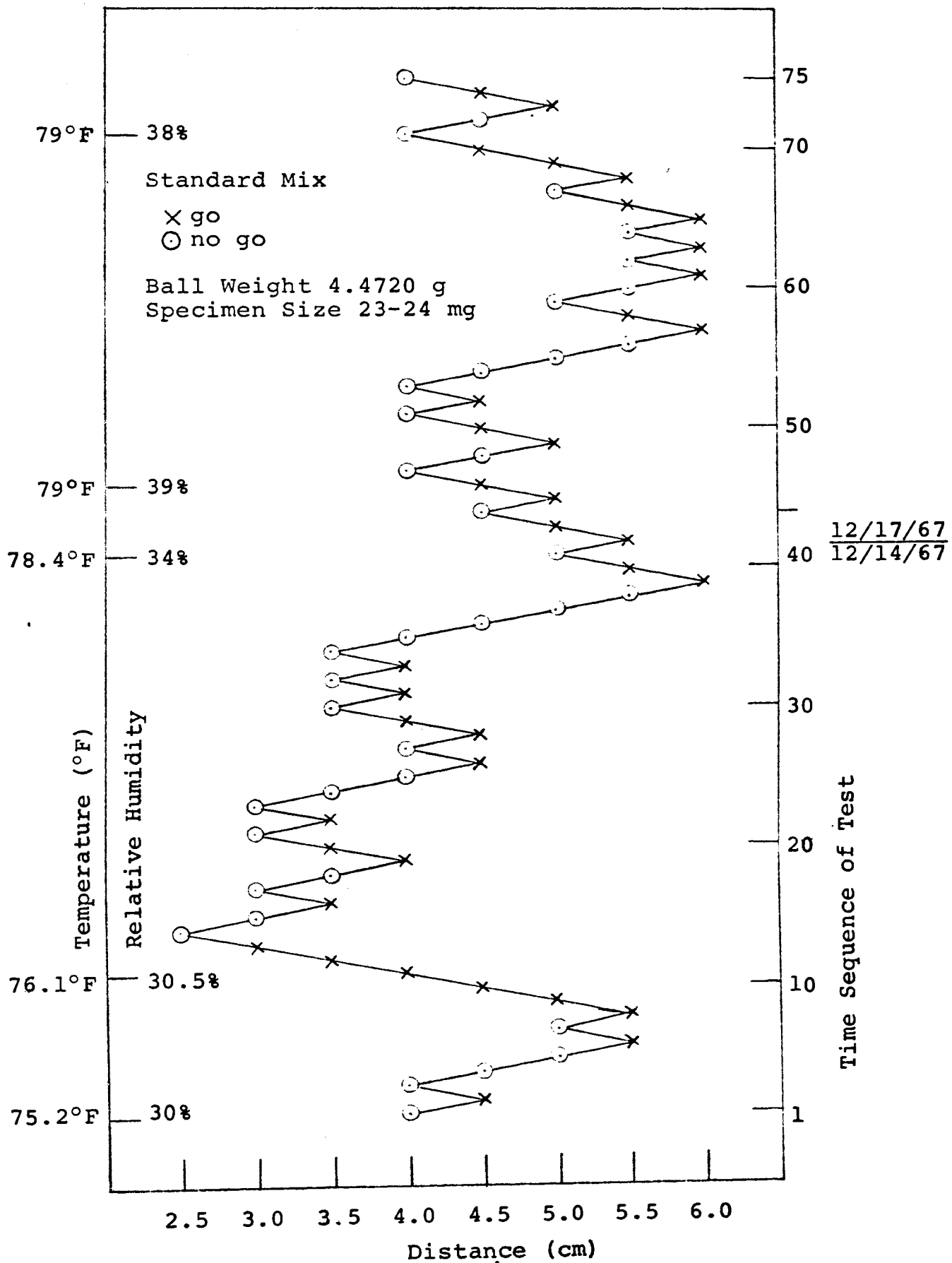
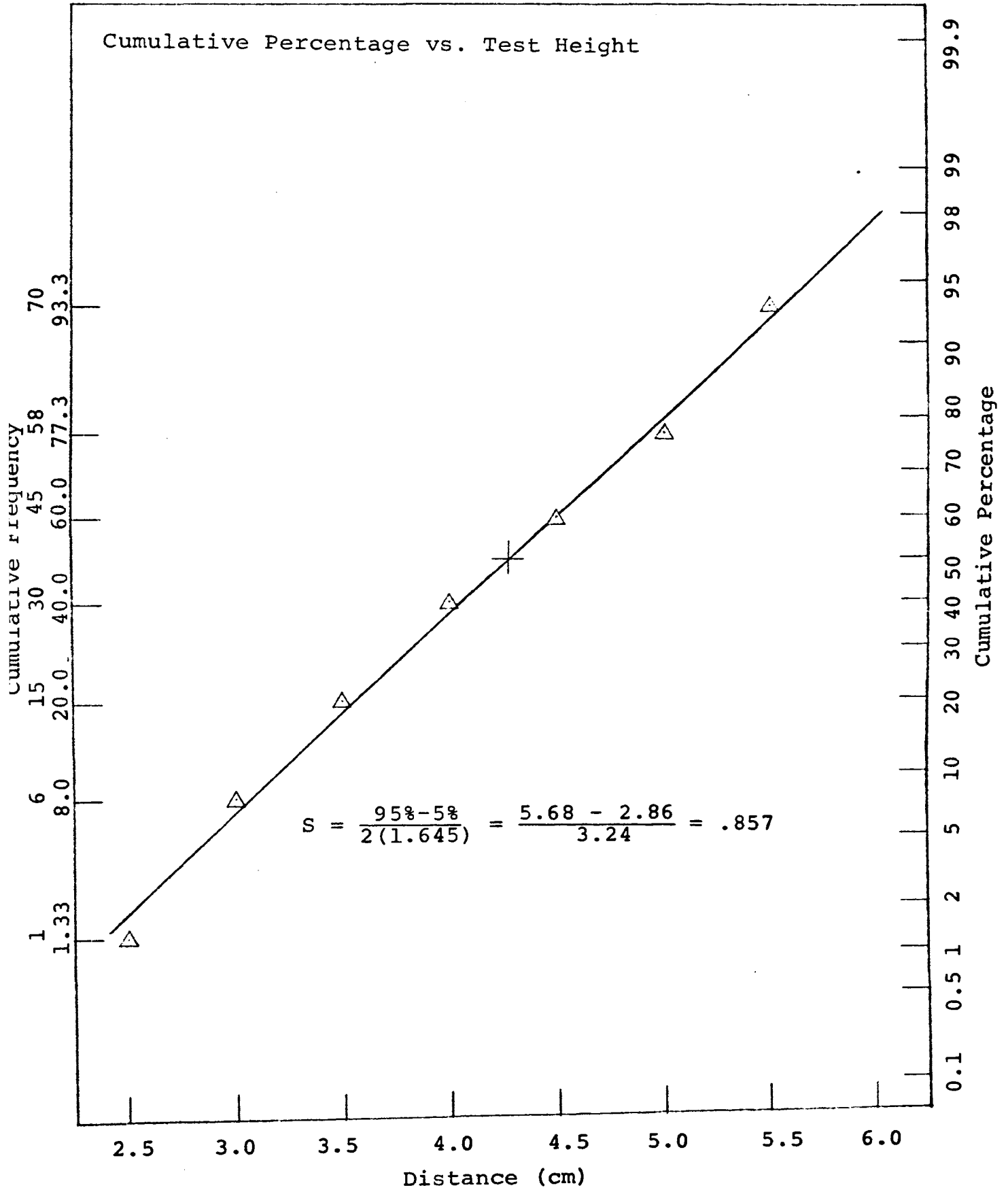Figure 13. Impact Test 24 - Time Sequence vs. Test Height

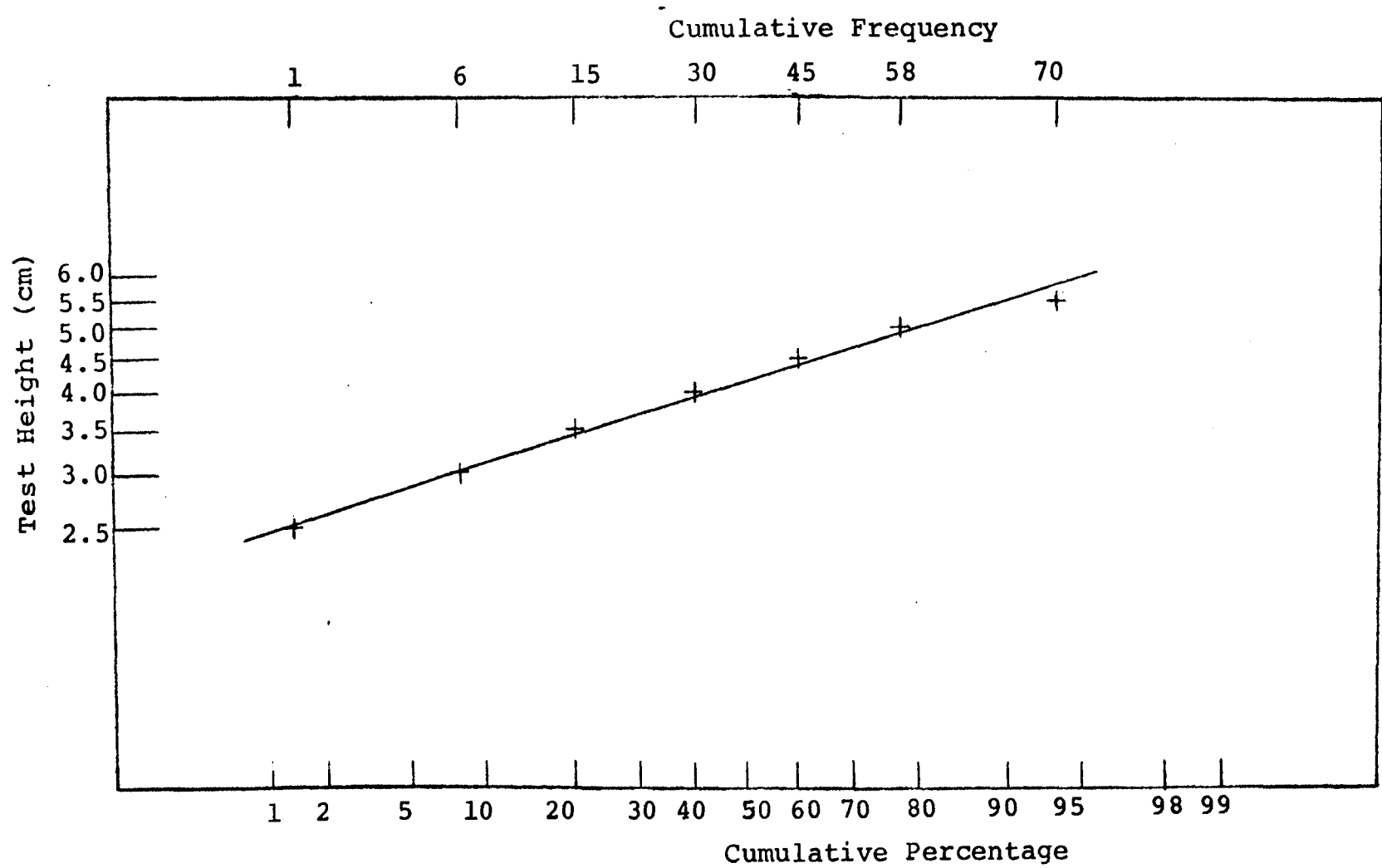Figure 14. Frequency - Normal Probability Plot - Test 24

Figure 15. Log-Normal Probability Plot - Test 24

STATISTICAL METHOD OF ANALYSIS

This method of analysis is taken from Dixon and Mood[1]. The test conditions and actual data points are given in Figure 13.

D_i	TF_i	F_i NoGo's	F_i Go's	N_i	i	iN_i	i^2N_i
2.5	1	1	1	--	-	-	-
3.0	5	4	1	N0	0	0	0
3.5	9	5	4	N1	1	4	4
4.0	15	10	5	N2	2	10	20
4.5	15	6	9	N3	3	27	81
5.0	13	7	6	N4	4	24	96
5.5	12	5	7	N5	5	35	175
6.0	5	0	5	N6	6	30	180
	75	38	37			130	557

$$D_i = \text{drop height}$$
$$T = \text{total}$$
$$F = \text{frequency}$$

The frequency of the go's is used for the analysis since it is the less frequent event.

$$\bar{x} = d + \sigma \left(\frac{A}{N} \pm \frac{1}{2}\right)$$

$\bar{x}$ = median

d = lowest drop height for less frequent event.

σ = test interval

N = total frequency for less frequent event

$A = iN_i$

$B = i^2N_i$

s = standard deviation

\+ is used for no go's

\- is used for go's

$$s = 1.620d \left(\frac{NB-A^2}{N^2} + .029\right)$$

$$\bar{x} = 3.0 + .5 \ (\frac{130}{37} - .5)$$

$$\bar{x} = 3.0 + .5 \ (3.0135)$$

$$\bar{x} = 4.5067$$

$$s = 1.620 \ (.5) \ \left(\frac{37(557) - (130)^2}{(37)^2} + .029 \right)$$

$$s = 2.2181$$

This method is taken from standard statistics texts[2].

d_i	TF_i	CUMULATIVE F	CUMULATIVE %	$d_i F_i$
2.5	1	1	1.33	2.5
3.0	5	6	8.00	15.0
3.5	9	15	20.00	31.5
4.0	15	30	40.00	60.0
4.5	15	45	60.00	67.5
5.0	13	58	77.33	65.0
5.5	12	70	99.33	66.0
6.0	5	75	100.00	30.0
	75			337.5

$$x = \frac{d_i t_i}{T} = \frac{337.5}{75} = 4.500$$

d_i	$d_i - \overline{d}$	$(d - \overline{d})^2$	f	$f(d_i - \overline{d})^2$
2.5	2.00	4.00	1	4.00
3.0	1.50	2.25	5	11.25
3.5	1.00	1.00	9	9.00
4.0	.50	.25	15	3.75
4.5	.00	.00	15	.00
5.0	.50	.25	13	3.25
5.5	1.00	1.00	12	12.00
6.0	1.50	2.25	5	11.25
		11.00		54.50

$$s^2 = \sum_{i=1}^{n} \frac{f(d_i - \overline{d})^2}{n-1}$$

$$s^2 = \frac{54.50}{75-1} = .7365$$

$$s = .858$$

$$u = \overline{x} \pm t_{n-1}; \quad \alpha/2 \; \frac{s}{\sqrt{n}}$$

$$u = 4.500 \pm 1.994 \left(\frac{.858}{\sqrt{75}}\right)$$

$$u = 4.500 \pm 1.994 \left(\frac{.858}{8.66}\right)$$

$$u = 4.500 \pm .1961$$

CHI-SQUARE TEST (3)

Normal Probability Plot

Distance	Cumulative Observed %	Cumulative Theoretical %	$(\dfrac{0_i - e_i}{e_i})^2$
2.5	1.33	1.95	.1971
3.0	8.00	6.90	.1753
3.5	20.00	18.20	.1780
4.0	40.00	37.50	.1666
4.5	60.00	60.60	.0059
5.0	77.33	80.30	.1098
5.5	93.33	92.40	.0093
6.0	100.00	97.80	.0494
			.8914

$$k - 1 - c = 8 - 1 - 2 = 5$$

$$x_{.05} (5) = 11.1$$

$$.89 < 11.1$$

Log Probability Plot

Distance	Cumulative Observed %	Cumulative Theoretical %	$(\dfrac{0_i - e_i}{e_i})^2$
2.5	1.33	1.10	.0480
3.0	8.00	7.40	.0486
3.5	20.00	22.20	.2180
4.0	40.00	43.60	.2972
4.5	60.00	64.00	.2500
5.0	77.33	80.00	.0891
5.5	93.33	90.00	.1232
6.0	100.00	95.10	.2524
			1.3265

$$1.33 < 11.1$$

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      duction to Statistical Analysis, Sensitivity Experi-
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