Investigation of Tetracene Decomposition Variation Using Taguchi Methodology

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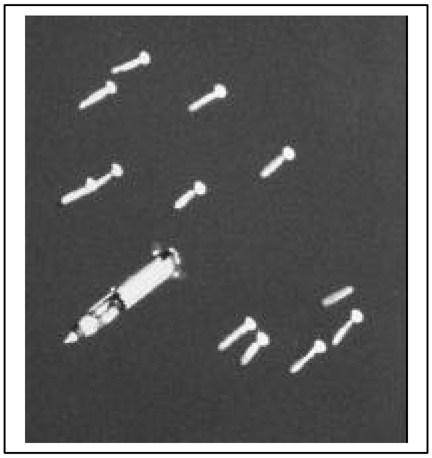
Introduction

Over the past decade, it has become increasingly apparent that it is essential for military customers to maintain close working relationships with production facilities and contractors to ensure quality performance. The Armament Research, Development, and Engineering Center (ARDEC) has had a long history of working closely with the Lake City Army Ammunition Plant, the main producer of small caliber ammunition for the U.S. Military. Recently, this plant experienced difficulties in the production and testing of Tetracene, an initiating explosive used in ammunition primers. To resolve this problem, government and contractor personnel worked together to investigate and model the manufacturing process of Tetracene. As a part of this modeling process, test matrices were developed using the Taguchi Method of Quality Engineering. These modeling efforts resulted in a better understanding of the factors effecting the production, performance, and testing for Tetracene, ultimately allowing the plant to avoid production delays.



Tetracene is a compound used in ammunition primers to increase and provide stability in the sensitivity of ignition. The original military specifications (Mil-Spec) require Tetracene to be manufactured with a “melting point” of between 128-132 degrees Celsius. Tetracene does not actually have a “melting point” but rather a decomposition point. (Decomposition point- is the point in which the compound is separated into its constituents through chemical reaction). It is this decomposition point that was of concern. The origin of this Mil-Spec requirement is not known for certain, except that it was instituted in the early 1960’s. The Mil-Spec requires periodic testing of Tetracene samples to insure that they meet the standard.

During one of these tests, the decomposition point results suddenly began a downward trend into the range of 122-125 degrees Celsius. This problem threatened to shut down production of all 5.56 mm and 7.62 mm ammunition at the Lake City Army Ammunition Plant (LCAAP). The Small Caliber Ammunition Branch of the U.S. Army Armament Research, Development & Engineering

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Center (ARDEC) at Picatinny Arsenal was tasked with the investigation and modeling of this Tetracene decomposition problem.

We will discuss our model, a design of experiments used to test the Tetracene production, the analysis of Taguchi methods applied to this design, and the conclusions found by the modelers. These efforts resulted in a better understanding of the factors effecting the production, performance, and test methodology for Tetracene, ultimately allowing the plant to avoid production delays in the manufacturing of small caliber ammunition. Changes were made to the Military-Specification as a result of this design test.

Production and Testing Procedures for Tetracene

Production

Tetracene is created in a manner that greatly resembles a large cooking type production. Most of the work is accomplished by hand by employees that have been producing Tetracene for years. The method has changed little over the past several decades, and due to the explosive nature of the compound, complete automation with electronic devices is difficult. Tetracene is formed in a three step method:

1. Mix a sodium nitrite solution with water.
2. Mix an amino guanidine bicarbonate (AGB) solution with water. Slowly add sulfuric acid at the rate of 50cc per minute to produce amino guanidine sulfate (AGS).
3. Mix the sodium nitrite solution (from Step 1) with the AGS (from Step 2). The mixing is done by a machine in a large closed room. A large spatula-like arm gently stirs (or agitates) the mix. The reaction is held to a temperature range of 95-101 degrees Celsius for five hours, at which time the reaction is assumed to be complete. During the five hour time period, Tetracene is precipitated from the mixture. This precipitate is filtered out and washed, then collected in conductive containers.

Testing

The purpose of taking a decomposition point reading is that it serves as an indicator of the purity of the compound. An important part of determining the factors affecting the decomposition point is the actual testing process itself. A Mettler testing device is used to capture the temperature in which the compound decomposes. Tetracene is a decomposing compound that undergoes an exothermic reaction from solid straight to liquid. Because of this characteristic, it has been very difficult to measure the exact decomposition point.

The Mettler decomposition testing device electronically measures the decomposition point using a computer controlled rate of change. The operator simply inserts a capillary tube with a sample of Tetracene into a the machine and sets the rate of change and the initial temperature. The computer senses when the Tetracene begins to decompose. The Mettler device replaced the previously used Vanderkamp device. The Vanderkamp device was found to be less reliable during the design analysis.

It was the Vanderkamp device that originally was used in the finding that the decomposition point had changed to the 122-125 degree range. The Mettler device confirmed this range of temperature for the decomposition point for the samples of Tetracene.

We developed a model, an experimental design test on the manufacturing process of the Tetracene. Many “applied statistics” options can be used. Due to a combination of budgetary and time constraints, we selected a Taguchi Design model.

Design Methodology

Taguchi Theory

The design factors developed by the Japanese engineer, Genichi Taguchi, are used to identify factors that control the “melting point." Taguchi focuses on the quality control of designing engineering products and production processes. Taguchi sees the loss a faulty product imparts to society (after shipment) in terms of monetary loss, dissatisfaction, loss of time, and hazards to the environment [4]. *Which of these, if any, does the Tetracene potential loss function appear to cover?*

Taguchi identifies three phases of the design process (system design, parameter design, and tolerance design) where statistical methods can be used in controlling the engineering process [1]. The first step, systems design, is not considered since Tetracene has already been developed and used for decades. There were also no available alternatives for the use of the Tetracene. Parameter design is the focus of this modeling effort. We identified the factors that control the design process by reducing fluctuation in variation, and determined the nominal target value for the quality characteristic being tested. This will ensure quality in the engineering and production process. We focus on the nominal target as it will be used in actual production cycles. The last phase is tolerance design where range specifications are set based upon economics and a loss function. Although this is not a one of the primary concerns, we will provide some insights into the specification limits on the decomposition point of the Tetracene.

Since the loss function is the heart of the Taguchi focus, let’s explore it in a little more detail. Taguchi uses a unique way to illustrate the loss incurred by a firm and society when the production process is not operating efficiently and the process does not mitigate the variation in the production process. Most engineering processes have specification limits that give a range for values for the process characteristic for which any value in this range is acceptable. According to Ryan [6], “Implicit in this view is the idea that all values within this range are equally good." Taguchi disagrees with this assessment and instead promotes the use of a specific nominal target value in the control of quality characteristics, such as the decomposition point.

The nominal target value helps focus the quality of the product as opposed to the product just needing to meet a specification range. Thus, as the quality characteristic varies from the target value the firm and society experiences a loss. The *square of the difference* is referred to as the *mean square deviation* (*MSD*). By focusing on the target value and reducing the variation in the design process, the ammunition plant can reduce its loss because the production goal of the ammunition plant is met. This reduction in the expected loss (attributed to the reduction of the variance) is the main reason why Taguchi stresses that all manufacturing firms should strive to reduce the variance for all process [6].

The loss function is an excellent tool to determine the magnitude of the quality characteristic from the target value and to determine the range of the tolerance level [5]. Loss to the Army occurs when the Tetracene cannot be used, resulting in the 5.56 MM and 7.62 MM small caliber production lines being shut down. The loss function is generally known and as a result can be modeled [6].

Application to the Design Model

Now that Taguchi’s approach has been described, we focus on the statistical computations and concepts used in parameter design. In order to minimize the sensitivity of a process, Taguchi has established orthogonal arrays for his design of experiments. The use of orthogonal arrays is similar to the concept used in more classical factorial design of experiments. There are several differences in both designs but both have the goal of identifying which factors control the experiment and what levels should be used to met the characteristic desired.

Taguchi’s design incorporates both controllable and uncontrollable factors. The uncontrollable factors are referred to as noise factors. In our design set up, the different employees making the Tetracene, the climate, manufacturing imperfections, product deterioration, and day to day variations are examples of noise factors. No specific noise factors were set for our design, instead an umbrella approach covering all these variations was used by repeating the matrix creating Batch 1 and Batch 2 samples.

Control factors are the variables that can be controlled in the production process. The selection of these variables is critical to ensure that the correct factors that dominate the process can be identified. These factors, as in factorial design, are assigned two levels. The purpose of the experiment is to determine not only which factors control the design (even with the noise levels exerting their influence) but also which of the two assigned levels will minimize the variance in the production process while driving the quality characteristic toward the nominal target value. The parameters identified as potential controllable factors in the process of manufacturing Tetracene were lot variations in amino guanidine bicarbonate, sodium nitrite and sulfuric acid; water; agitation of the Tetracene mix; sodium nitrite concentration; and temperature control of the mix. Two variation levels were chosen for each variable. The variables were fit into one of Taguchi's orthogonal arrays that allows for seven parameters or variables, with a total of eight experimental runs. We repeat the matrix to allow for noise variation, thus performing sixteen runs.

TEST MATRIX FOR TETRACENE INVESTIGATION

CONTROLLABLE FACTORS		LEVEL 1	LEVEL 2
A	AMINO GUANIDINE BICARBONATE	LOT 1	LOT 2
B	SODIUM NITRITE	PRODUCTION	REAGENT
C	SULFURIC ACID	REAGENT	PRODUCTION
D	WATER	DEIONIZED	DISTILLED
E	AGITATION OF MIX	WITH	WITHOUT
F	SODIUM NITRITE CONCENTRATION	EXCESS	STARVED
G	TEMPERATURE CONTROL	CONTROLLED (95-101 DEG C)	UNCONTROLLED
NOISE FACTORS	B1 & B2		

Classical statistical models used in factorial design processes use the average response to calculate the main effects as well as the analysis of variance (ANOVA) to identify if the controlled variables were significant in the process. In contrast, Taguchi uses a signal to noise ratio (S/N) to measure the main effects of the experimental design. The signal to noise ratio is the change in the quality characteristic, the decomposition point of Tetracene. Therefore, the S/N measures the sensitivity of the quality characteristic being investigated in a controlled manner, to external influencing factors not under control [5].

A high S/N as the measure of performance statistic is better than a low ratio because a high S/N implies that the signal is stronger than the noise factors. It also implies that the quality characteristic has minimum variance. In order to understand these two concepts, we must look at how the S/N is calculated.

$$S/N = -10\ LOG_{10}\ (MSD) \tag{1}$$

The MSD is the square of the response measure minus the target value.

$$MSD = (Y-T)^2 \tag{2}$$

In order for the MSD to be small, the response measured from the design of experiments must be close to the target value. Thus indicating a small variance from the target value. The small variance also indicates that the noise factors had a minimal effect on the response measure of the quality characteristic. MSD is consistent with Taguchi's objective of reducing variation around the target value [5]. The logarithmic transformation of the MSD into the signal to noise ratio is used to maximize the measured responses with the smallest MSD, making the parameters that control the design easier to identify.

Another advantage in using Taguchi design is the number of design points needed to run the experimental design. In our design, we identified seven control factors that will vary over two levels. In a 2^k design, we would need 2^7 or 128 experiments for each run. We would need 384 for three runs. Even if we used a half factorial design (2^{k-1}), we would need 64 design points from each run. With the Taguchi design, we need only 8 design points or 24 responses for the orthogonal array matrix. This has obvious advantages such as time and costs.

Taguchi's design is not without its faults. This methodology has received criticism because of the limited number of interactions that can be measured between the control variables. For our design, we can only measure two interaction effects. Furthermore, the design team must have an indication of which factors will interact in order to properly place them in the design matrix. For our design, interaction can only be determined between the following control variables:

1. Amino Guanidine Bicarbonate and Sodium Nitrite
2. Sodium Nitrite and water

TABLE 1: TAGUCHI MATRIX: Seven Parameter Design-Eight Runs

L(8)	A	B	C	D	E	F	G
1	1	1	1	1	1	1	1
2	1	1	1	2	2	2	2
3	1	2	2	1	1	2	2
4	1	2	2	2	2	1	1
5	2	1	2	1	2	1	2
6	2	1	2	2	1	2	1
7	2	2	1	1	2	2	1
8	2	2	1	2	1	1	2

Results

We evaluated both the Batch 1 and the Batch 2 data collected on the decomposition of Tetracene, shown in Table 2. The results, using the Taguchi method with the signal-to-noise ratio, identified the controlling variables to be agitation (Factor E) and temperature control (Factor G), see Tables 3-6.

In Table 3, we calculate the row totals, row averages, row averages squared, the row variance, total squared/number of observations, sensitivity in decibels, and signal to noise ratios. These will be used in other calculations to determine the most significant variables.

Table 2. Batch Results

	Batch 1				Batch 2	
1	128.3	128	128.2	124.8	124.7	124.6
2	127.1	126.9	127.1	127.1	126.9	127
3	126.5	126.6	126.5	124.5	124.3	124.4
4	126.5	126.7	126.7	126	126	126
5	127.2	127.4	127.3	127.1	126.6	127.2
6	128.0	127.9	127.8	124.0	124.1	124.03
7	126.5	126.1	126.1	124.9	124.8	124.83
8	125	124.9	125.0	123.8	123.8	123.8

Table 3. Data Layout

Total	YBAR	YBAR^2	S^2	S_m	S_mD_b	ND_b
758.6	126.43	15985.39	3.618	95912.33	49.818	36.451
762.1	127.016	16133.23	0.0097	96799.4	49.858	62.224
752.8	125.467	15741.88	1.371	94451.31	49.752	40.601
757.9	126.317	15955.9	0.1256	95735.4	49.810	51.037
762.8	127.133	16162.88	0.0787	96977.31	49.867	53.127
758	126.333	15960.11	2.9547	95760.67	49.812	37.325
753.2	125.533	15758.62	0.61066	94551.71	49.757	44.117
746.3	124.383	15471.21	0.409667	92827.28	49.677	45.771
6051.7	1008.617	127169.2	9.1783	763015.4	398.3523	370.655

Next, we calculate the response table utilizing the following rules:

(1) Sum each variable (A through G) at Level 1 and Level 2. Each variable is at each level 24 times. Find the absolute difference and rank order them.
(2) Take the average of each variable at each level. Find the absolute difference and rank order them.
(3) Total the signal to noise ratios for each variable at each level. Compute the absolute difference between level 1 and 2 and rank order them.
(4) Compute the average signal to noise ratio for each variable at each level. Compute the absolute difference and rank order them.
(5) Use the ranking to determine which variables are the controlling factors.

The variables that control the process are those that have an influence on the signal to noise ration. If there is a large difference in S/N when the variables are at different levels, this shows a strong influence. Table 4 (subtable 4.4) identifies the effect each variable had on the S/N by calculating the difference between levels. Subtable 4.4 also displays the ranking for each variable with respect to S/N. Of significance are the variables ranked #1 and #2. These ranked variables, #1 and #2, represent variables E and G, Agitation and Temperature Control. As stated earlier, it is desirable to maximize the S/N ratio to achieve the best process performance. To maximize the S/N of the variables E and G, they should both be set at Level 2. This would indicate that there is no agitation and no temperature control (remembering the definitions of Level 2 for these variables) in the manufacturing process. The other variables of the matrix do not have a significant effect on the S/N and thus, are considered to have no effect on the decomposition of the Tetracene compound.

Table 4. Response Tables

4.1 Totals (DEG C)	A	B	C	D	E	F	G
Level 1	3031.4	3041.4	3020.2	3027.4	3015.7	3025.6	3027.7
Level 2	3020.3	3010.2	3031.5	3024.3	3036	3026.1	3024
Difference	11.1	31.3	11.3	3.1	21.7	.5	3.7
Ranking	#4	#1	#3	#6	#2	#7	#5
4.2 Averages							
Level 1	126.308	126.729	125.842	126.142	125.654	126.067	126.154
Level 2	125.845	125.425	126.313	126.013	126.5	126.088	126
Difference	.4625	1.304	.4708	.1291	.8458	.0208	.1541
Ranking	#4	#1	#3	#6	#2	#7	#5
4.3 Totals (ndB)							
Level 1	190.313	189.127	188.562	174.2967	160.147	186.385	168.93
Level 2	180.3417	181.527	182.092	196.358	210.507	184.269	201.724
Difference	9.97	7.57	6.46	22.06	50.36	2.17	32.794
Ranking	#4	#5	#6	#3	#1	#7	#2
4.4 Averages (ndB)							
Level 1	47.578	47.282	47.14	43.57	40.03668	46.5962	42.2325
Level 2	45.08	45.383	45.5232	49.0895	52.6229	46.0674	50.4312
Difference	2.4935	1.9000	1.6184	5.5151	12.5893	.5297	8.1983
Ranking	#4	#5	#6	#3	#1	#7	#2

Table 5: Signal-to-Noise Ratio: High/Low Table

FACTOR	LEVEL	HIGH	LEVEL	LOW	DIFFERENCE
E	2	52.6265	1	40.0372	12.5893
G	2	50.4310	1	42.2327	8.1983
D	2	49.0894	1	43.5743	5.5151
A	1	47.5786	2	45.0851	2.4935
B	1	47.2821	2	45.3816	1.9005
C	1	47.1410	2	45.5227	1.6184
F	1	46.5967	2	46.0670	0.5297

Conclusion

The Taguchi Design Model was used to evaluate the manufacturing process for Tetracene. Because both the temperature of the mix and the agitation are controllable, and were controlled, they could not have had an effect on the decomposition results. In addition, the variables that could have changed: the chemical compounds, the water, the sodium nitrite concentration, are not found by the Taguchi analysis to have a positive effect on decomposition. Therefore, the manufacturing process is deemed acceptable because we are adequately controlling the two variables that effect the decomposition the most.

When we examine the quality/performance of the ammunition versus the decomposition result of Tetracene through various tests, we find that a change in the decomposition point (to the levels that we found) has had no effect on the ammunition performance either near or far term. This determination was made by testing ammunition manufactured with Tetracene that did not meet the decomposition specification against a lot of ammunition manufactured prior to any difficulties that had been encountered. Side by side tests as well as accelerated aging tests show no difference in ammunition performance.

The final stage was to examine the actual decomposition test method itself to determine if that is a factor in the changing results. The test method was found to be suspect, the temperature at which the test began and the rate of temperature rise both had an effect on the decomposition result. Therefore, to resolve this issue, the following changes were made to the Mil-Spec that governs Tetracene:

1. Use the Mettler testing apparatus instead of the Vanderkamp. The Mettler performs the same decomposition test, but is less operator dependent.
2. Revise specification limits for Tetracene. The limits were changed from 128-130 degrees Celsius to 124-130 degrees Celsius. This new limit was derived from testing of thirty different production batches of Tetracene using the Mettler testing apparatus.
3. The test method was altered to require a start temperature of 120 degrees Celsius. There has been no previous instruction as to what temperature the test must begin.

Since the specification revision has fallen within the new limits for the production and testing of the Tetracene, no further difficulties have been encountered. The results of this model and its analysis show that the manufacturing of Tetracene was following a procedure that both satisfies requirements and results in “good” ammunition. Based on this design analysis, the Lake City Ammunition Depot continues to produce the chemical compound, Tetracene.

Exercises

1. Consider a catapult and the following problem identification. Predict the distance a projectile travels being “thrown” or “shot” from the catapult. Our catapult appears as in Figure 1.

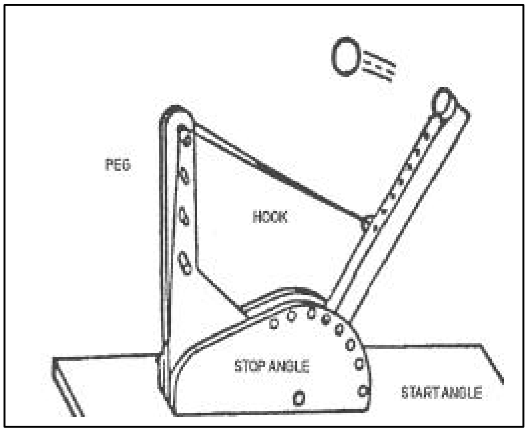

Figure 1. Air Academy Catapult

The possible factors and levels are:
- Peg position for the rubber band (4 positions).
- Hook position for the rubber band (5 positions).
- Angle of the firing arm (approximately 80° to 190°).
- Stop angle for the firing arm (6 positions are available).
- Ball type to be used (2 types--rubber and whiffel).

a. Determine the total number of possible permutations of these variables.

b. Let’s limit our selves to the top two levels of the peg, the top 2 positions below the cup for the hook, integer angles 155° and 170° only, positions 2 and 3 for the stop angle, and the two balls. How many permutations do we have now?

c. Use a L_4 Tachugi Design Matrix and the following collected data to find the significant variables that effect the distance the “ball” is thrown from the catapult.

L_4 Design

Run	A	B	A*B
1	1	1	1
2	1	2	2
3	2	1	2
4	2	2	1

Assume: Signal Factors are Pull back angles of 155° and 170°.
Noise: Ball type (R=rubber and W=whiffel)

Control Factors: STOP (Positions 1,2) & Cup Location (Positions 1,2) where 1 is low position and 2 is high position.

			Distance (in.)	Distance (in.)	Distance (in.)	Distance (in.)
	Control Variable 1	Control Variable 2	Signal & Noise 155°	Signal & Noise 155°	Signal & Noise 170°	Signal & Noise 170°
RUN	STOP	CUP	R	W	R	W
1	1	1	53	52	108	109
2	1	2	35	36	85	87
3	2	1	45	55	95	105
4	2	2	35	36	85	82

d. Using only your significant variables (STOP or CUP), build a linear regression model to predict the distance the "ball" travels as a function of your significant variables. Your signal and noise variables should be included in your model.

e. Using your model, determine the other settings to "shoot" a rubber ball 100 inches.

2. Consider a new army missile system, where we want to predict the distance the missile travels as a function of its significant variables. Let's define the following limited experimental variables:
D= distance in miles that the missile travels
C= charge of the missile warhead (2 charges, heavy (H=2) and light(L=1))
W = weight or mass of the missile (2 missile mass types 500lb (1) and 1000lb (2))
A = angle of fire (we will fire experiments at 45° and 60°)
E = elevation of missile tube (110 mils and 170 mils)

Let's let the angle and elevation be the signal and noise variables, as we control the charge and weight of the missile. The following data is collected:

			Distance (miles)	Distance (miles)	Distance (miles)	Distance (miles)
	Control Variable 1	Control Variable 2	Signal & Noise 110 mils	Signal & Noise 110 mils	Signal & Noise 170 mils	Signal & Noise 170 mils
RUN	Charge	Mass	45°	60°	45°	60°
1	1	1	33	42	108	109
2	1	2	35	36	85	88
3	2	1	45	52	95	103
4	2	2	35	37	85	82

(a) Determine the significant variables for this experiment, using the L4 Taguchi design procedure.

(b) Using your significant variables and the signal & noise variables, build a linear regression model to predict the distance. Use your model to determine the levels and factors for a missile to travel a distance of 95 miles.

3. Relate the Taguchi Design Models to the Factorial design models in statistics. How are they similar and how are they different?

4. Describe a scenario that you feel could benefit using a design model approach. List some assumptions to the model and some variables that would be used.

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