

TO W. Miringoff

AT Burlington

DATE August 21, 1975

FROM W.C. Champion

AT Burlington

COPY TO J.W. Poarch
R.F. Panter

SUBJECT PLEX

File

The attached article from the Journal of Quality Technology may be of interest. The way we've been approaching plant problems now has legitimacy - it has a name, and it's been published in a scientific journal!



W.C. Champion

WCC/gs

Attachment

PLant EXperimentation (PLEX)

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Unexplained process variability is likened to a ghost wandering freely through the plant spooking the process. Plant experimentation is discussed as a tool for investigating this behavior, alleviating it, and providing information for process improvement. Two different plant experimentation approaches are discussed and compared: PLEX (PLant EXperimentation) and EVOP (EVolutionary Operation). The PLEX approach is illustrated using a recent case study of a plant process. The results aided in reducing variability, increasing capacity, and improving overall profitability in the plant.

Introduction

TRACKING down the causes of extreme plant process variability (as in Figure 1) can sometimes be comparable to stalking a ghost. This appears so, when by passive observation, none of the meas-



FIGURE 1. Yields in 1st Reactor of Intermediate Step Before Plant Experimentation Studies.

ured variables correlates with the elusive and capricious variability in yields or quality. For example, a great deal of time and effort can go into the recording of data on variables (e.g., temperature, pressure, feed rates, catalyst concentration, etc.) to determine if they correlate with the responses of interest (e.g.,

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reaction rates, product purity, yield, etc.). When this effort does not show any fruitful results, it may be jokingly said that a ghost is walking through the plant and it is he who is upsetting the process. This is the same as conceding process behavior to the whims and ways of one or more unidentified critical variables.

An approach for combating the above behavior is to go on the offensive with a program of controlled tampering to find out what can cause things to happen. In this way, one may be able to beat the ghost at his own game and severely limit his activity in the plant. It is this offensive approach or plant experimentation that is the topic of this paper.

Approaches to Plant Experimentation

A guideline for a plant experimentation program can be borrowed from a quote by G. E. P. Box [3], "To find out what happens to a system when you interfere with it you have to interfere with it (not just passively observe it)." Plant experimentation is a form of controlled interference to learn more about the process without upsetting it. This can be done in different ways, two of which are discussed in the following paragraphs.

The first approach will be referred to as the PLEX approach for PLant EXperimentation. It involves executing one or more sets of experiments in a limited amount of time in order to get process improvement information on selected sets of variables. The plant usually returns to its original operating conditions after the experiments until the results of PLEX can be fully digested, and incorporated into the operating procedure, if beneficial. It involves "getting in" and "getting out" of the plant as quickly as possible when there is technical supervision available to

closely monitor the process, take samples, and make corrective action at the slightest warning of upset. Bacon [1] referred to a similar approach as "one-shot experimentation" when a single plan, rather than a series of sequential experimental plans, is executed. An example of the application of the PLEX approach will be given later in the paper.

The second and most widely publicized plant experimentation approach is EVOP (EVolutionary Operation). This approach, originally developed by Box [2, 5, 6, 7], has the basic philosophy "that a process should be operated so as to produce not only a product but also information on how to improve the product [6]." It is a continuous investigative approach carried out mainly by the operating staff. It can become the standard way of operating the plant since it usually involves only small changes in the process variables thus keeping risk of upset at a minimum.

PLEX may be one or a series of short duration one-shot experimental designs under the watchful eye of both technical and operating supervision thus providing more incentive to broaden the ranges on the variable levels. In contrast, EVOP is an experimental design approach with small changes in the variable levels and is an approach that goes on continuously with little or no additional technical supervision. Both have the purpose of improving the process but their execution differs as shown here.

found knowledge (e.g., technical and engineering departments) and new profitability (e.g., operating and sales departments). Also, since EVOP has been criticized by some for being too slow in returning information, PLEX is an attempt to speed up information-gathering by concentrating on a sequence of short duration studies with the variable levels changed over wider more informative ranges than those felt safe for EVOP. This is possible in PLEX by adding extra technical supervision for surveillance to minimize risk of upset over the wider variable settings. From our experience certain ingredients are necessary for successful plant experimentation whether it be PLEX or EVOP. These include:

- (1) a willing and cooperative operating staff;
- (2) good interaction between technical and operating personnel;
- (3) one or more crusaders who are strong believers and can keep the studies active;
- (4) a definite need or reason for tampering with process conditions.

The last point is very important and the prime motivator for experimenting on a large scale process. It is perhaps human nature to resist tampering with a plant process for the sake of information, especially if the result could be a serious upset with considerable downtime and lost productivity. There has to be an emphasized NEED for information leading to benefits that far exceed the cost of the effort.

Comparison of PLEX and EVOP

FACTOR	PLEX	EVOP
1. Purpose	Process Improvement and information gathering	Process Improvement and information gathering
2. Responsibility	Shared by Participating Departments	Operating Department
3. Procedure	Series of One-Shot Plans	Continuous Investigative Procedure
4. Size of Variable Changes	Usually Wider than EVOP	Usually Small
5. Risk of Upset	Kept Manageable by Extra Surveillance	Small
6. Urgency	Usually urgent	Not necessarily

Some of the drawbacks to successful EVOP application were described in a review paper on EVOP by Hahn and Dershowitz [7]. It was their finding and ours "that there is a strong reluctance by the operating department to tamper with a process, especially if they do not know if their efforts are going to be recognized and lauded in the case of a success or criticized in the event of an upset." PLEX programs attempt to share the responsibility among the departments who benefit from any new-

Some examples of need include higher capacity to meet increasing sales demand, improved quality to remain competitive, controlling extreme or ghost-like variability, and reductions in pollutant levels. The emphasis or push usually must come from the plant manager's office or higher in order to overcome any resistance to information gathering via plant experimentation. In addition to the input from management, a believer or crusader is a good catalyst for selling the benefits of using plant experi-

mentation to meet the need and helping to set up the program. Last but not least, cooperation and good interaction among the personnel involved are absolutely essential ingredients.

A case history is described below where the ingredients discussed above were essentially present and were the major factors for a successful outcome. However, it should be pointed out that although the PLEX approach was the tool applied, *it was the people who made the program go.*

A Case History

The plant of interest here was a large continuous chemical operation with three separate reaction steps referred to here as: initial, intermediate and final. Although the plant had recently been expanded, it was not giving the additional 25% capacity that was expected. The most serious problem was erratic yields in the first stage reactor of the intermediate step (see Figure 1). This was the principal capacity limiting component of the process since the erratic or "ghost-like" behavior forced a reduction in production rates to avoid off-spec quality. The second problem was to increase the yield and capacity of the final reaction step assuming that the first problem could be solved. It should be emphasized that there was a strong and motivating need to get valuable process improvement information. Successful economics were not possible without it.

PLEX in the Intermediate Reaction Step

It was strongly felt by a number of technical personnel assigned to the problem that an additional reactor would need to be added to the intermediate reaction step. This increase in the number of reactor stages would provide additional residence time for reaction completion. It was realized however, that although this might give higher capacity at a higher cost, it would not necessarily explain why there was severe variability in the reactor yields.

It was decided then, prior to any further capital expenditure, that all the variables would be listed and reviewed. These included (1) temperature, (2) pressure, (3) agitation, (4) catalyst concentration, and (5) residence time. The first four variables were chosen initially to be studied in the plant using a four variable eight run fractional factorial design (2^{4-1}) [4]. As discussed in more detail in the next section, factorial and fractional factorial designs were chosen because of their efficiency in screening the variables and keeping the number of runs to a minimum. The initial operating condition in this

design corresponded to running at the low level of each of the four variables. A downward effect on yield was expected but not to the severe degree that actually occurred (see April 17 in Figure 1). The remaining experiments were shelved. It appeared obvious that the process was too near a precipice to risk venturing out in many directions using a designed set of experiments over the ranges being considered.

There was a regrouping of thoughts in the subsequent week with the conclusion that the process should be run at the high operability levels of each of the first four variables to avoid upsets. The literature and lab data indicated that this was a desirable direction. Also, an initial appreciation of the reaction mechanism suggested increased pressure and agitation would improve the reaction rate. A scheme was used to increase agitation and pressure together in steps (see Figure 2) while maintaining temperature

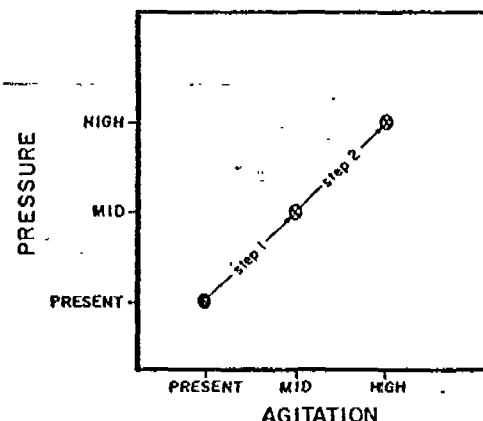


FIGURE 2. Plant Experimentation Using *a priori* Knowledge.

and catalyst concentration at their practical limits. This zooming in or moving in the direction thought *a priori* to be optimal gave dramatic results. This is easily seen when control charts "before" (Figure 1) and "after" (Figure 3) are compared. The reduced variation as shown in Figure 3 opened the way for increased throughput without fear of upset. Also, the way was now clear for further experimentation.

Before leaving the study of this reaction step, plant experimentation essentially yielded the following:

- (1) original objective of 25% increase in capacity plus an additional 11% with a total pretax realization of 2 million dollars;

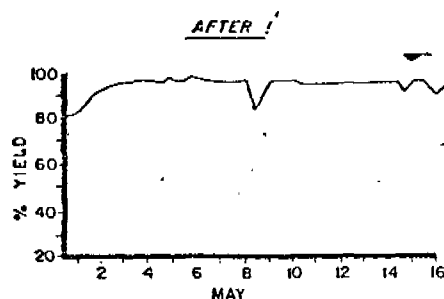


FIGURE 3. Yields in 1st Reactor of Intermediate Step After Plant Experimentation Studies.

- (2) data for mathematical modeling which later explained the underlying mechanism;
- (3) a harness on the variability or ghost in this important intermediate step.

PLEX Approach in the Final Reaction Step

Although it was felt that the ghost had been limited in his freedom to wander through the intermediate reaction step, he was still having his own way in the third and final step.

The dominant symptom in the final reaction step was inconsistent reaction yields. Unlike the previous step, it was felt that the reaction was not prone to serious upset and therefore a more systematic plan of experimentation could be used. Therefore, a designed program of experiments seemed ideal for investigation of this reaction step. However, it was preferred that this be done in a series of one-shot experimental designs (PLEX) rather than in a continuous EVOP. The reasons were: (a) analytical analyses on process samples were laborious and generally took more than a week for processing, (b) urgency, and (c) round-the-clock technical supervision was felt to be necessary to aid in rigorous data recording and sampling. Hence, it was felt that "getting in" and "getting out" over a period of several days would not strain the manpower requirements and production scheduling and also give the analytical lab enough time to digest the samples. Also, it was felt that a short duration technically supervised program of experiments would initially yield the best possible data. EVOP could follow later once the routine of monitoring the variables, changing their levels, and taking samples was established.

Discussions revealed there were at least eight variables that required investigation. Research reports and plant operating records provided information on possible ranges over which these variables could be changed without serious risk of upset.

Manageability and caution in studying these variables were also exercised by breaking the variables up into groups of three or four. The groups were selected on the basis of: (1) estimated importance or priority, and (2) likelihood of detecting interactions between certain pairs of variables. Group sizes of three or four variables kept the number of runs small while still providing an opportunity to measure the effects and interactions of variables. Also, with caution in mind, a study involving too many variables might lead to unstable or upsetting operating conditions. To satisfy the above requirements, factorial designs [4] seemed most suitable. They kept the number of runs small while providing an opportunity to measure the effects and interactions related to the variables.

PLEX-1

Overall, three PLEX designs were performed on the final reaction step which is schematically shown in Figure 4. In the first step of designed experiments

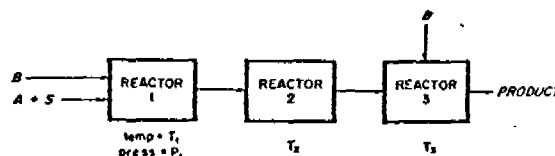


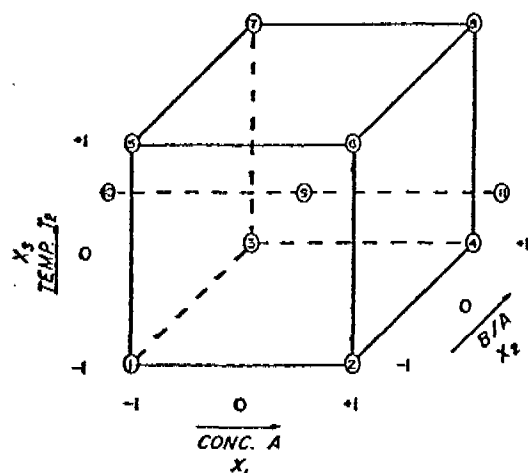
FIGURE 4. Configuration of Final Reaction Step.

(PLEX-1), three variables were chosen: x_1 —concentration of reactant A in solvent S, x_2 —ratio of reactant B to reactant A, and x_3 —temperature T_2 in second reactor. The designed experiments (Table 1) were performed in random order over an elapsed period of ten days with eight hours at each operating condition. (There were some interruptions due to slowdowns in other steps in the plant.) Actual levels of the variables are coded here and the yields are disguised for the purpose of anonymity. The notation -1 is for the low level of the variable, 0 for the mid level, and +1 for the high level. The complete set of experiments represented a 2^3 factorial design with repeats and center point. Also, two runs at extreme levels of concentration of A were performed with -2 denoting the lowest level and +2 the highest level. Extreme levels of the other variables were not tried because of risk of upset.

During the experimentation there was difficulty in running all 3 variables on target, and hence the final configuration of experiments was somewhat distorted from the original cube. Therefore, the data were analyzed using the actual settings instead of

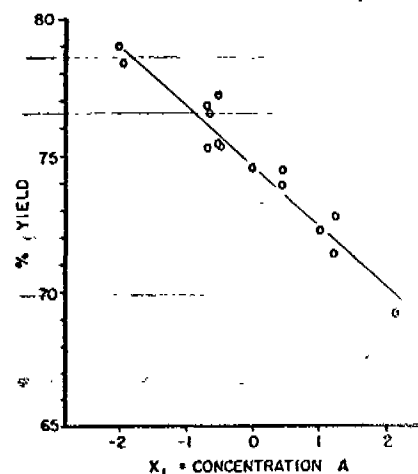
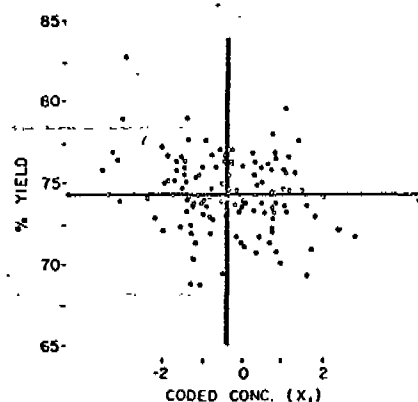
TABLE 1. Statistically Designed Plant Experimentation (PLEX-1)

Run #	x_1	x_2	x_3	Yield
1	-1	-1	-1	75.4
2	+1	-1	-1	73.9
3	-1	+1	-1	76.8
4	+1	+1	-1	72.8
5	-1	-1	+1	75.3, 75.3
6	+1	-1	+1	71.4
7	-1	+1	+1	76.5, 77.2
8	+1	+1	+1	72.3
9	0	0	0	74.4, 74.5
10	-2	0	0	79.0, 78.4
11	+2	0	0	69.2



the target values in Table 1. Both in this and subsequent PLEX-2 design, the departures from target were not so serious that reasonable measures of the variable effects could not be found using the target values. Since the analysis of factorially designed data is often insensitive to small and moderate departures in conditions, the target values could have been used with little or no loss in the data interpretation. The actual values were only used for the sake of mathematical rigor.

The results of PLEX-1 exceeded everyone's expectations. A single variable (concentration of A) explained 94% of the variation in yield. (See Figure 5a.) A comparison of these PLEX results in Figure 5a versus earlier results obtained from the plant production records (Figure 5b) shows a dramatic difference in the quality of the data. A combination of technical supervision to assist in adjusting the

FIGURE 5a. Plot of Yield Versus Concentration A (x_1) in PLEX-1.FIGURE 5b. Prior Production Data on x_1 .

variables and sampling, plus the use of improved analytical methods, put the data in focus and made it less ghostlike.

A simple prediction equation was developed to relate yield to the concentration A. A least squares fit (i.e., the line that minimized the sum of squares of distances between the observed and predicted values) gave the following prediction equation yield

$$\text{Yield} = 74.6 - 2.2 x_1$$

This equation provided a statistically adequate fit to the data leaving only 6% of the variation to be easily explained by the error variation, as checked by the repeats. No effects were found due to the other two variables over the ranges studied.

PLEX-2

A second design (PLEX-2) was set up within two weeks of PLEX-1 to further study temperature T_2

and reactant ratio B/A to confirm their unimportance. In addition, two other variables were added: x_4 —temperature T_3 , and x_5 —the amount of reactant B entering the third reactor. A 2^{4+1} fractional factorial design [4] was applied studying 4 variables in eight runs, plus center point, and three repeats (Table 2). This design was chosen because it allowed

TABLE 2. Statistically Designed Plant Experimentation (PLEX-2)

Run #	Order Of Running	x_2	x_3	x_4	x_5	% Yield
1	5, 11	-1	-1	-1	-1	71.0, 72.6
2	2, 12	-1	+1	-1	+1	71.4, 73.4
3	7	+1	-1	-1	+1	72.9
4	8	+1	+1	-1	-1	73.3
5	4	-1	-1	+1	+1	71.3
6	9	-1	+1	+1	-1	73.7
7	6	+1	-1	+1	-1	73.2
8	3	+1	+1	+1	+1	71.4
9	1, 10	0	0	0	0	70.4, 71.8

VARIABLE

- x_2 - B/A Ratio In Reactor 1
- x_3 - Temperature T_2
- x_4 - Temperature T_3
- x_5 - Amount B Entering Reactor 3

the plant to screen four variables in a reasonably small number of runs. Also, interactions among the variables were not expected, and hence their independent effects were not sought by using a full 2^4 factorial design.

The target values of the variables were not obtained in all runs. A statistical analysis of the data using the actual values showed no significant effect due to any of the planned variables, but it did show a significant effect from a "lurking" or "ghostlike" variable. As seen in Figure 6, there was an approximate 2% upward shift in yield after the fifth experiment in sequence. This shift occurred simultaneously with a change in the purity of the feed material A. Theoretically, the observed change in purity could cause a 2% change in yield, and hence it was strongly

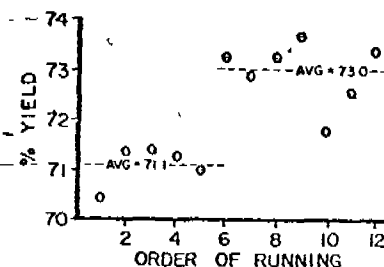


FIGURE 6. Plot of Yield Versus Run Order in PLEX-2

suspected as being the lurking variable. Because all four planned variables were found to be nonsignificant over the ranges studied, cost savings could be realized by setting them at their economic optimum.

Although 3 and 4 experiments were duplicated in each of PLEX-1 and PLEX-2 for the primary purpose of estimating the process error variation, the repeats were also intended to flag any unusual shifts in the data, as seen previously. Actual selection of repeat conditions was normally left to the technical supervisor's opinion as to which conditions either required additional data or were convenient to rerun.

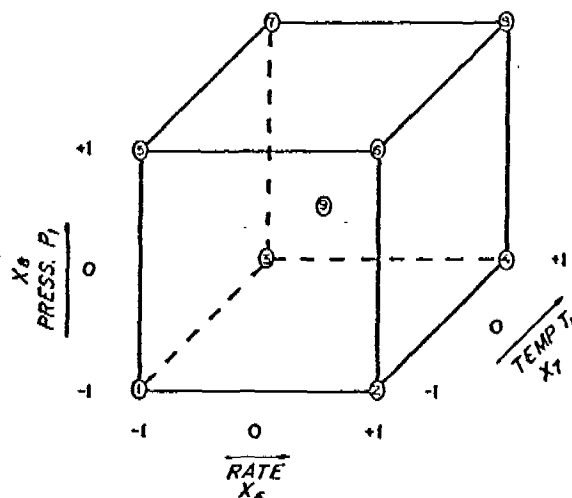
PLEX-3

Once the important yield relationships were established, priorities shifted to other parts of the plant, and it was not until another seven months that a third designed set of experiments (PLEX-3) was performed on this reaction step. Here, the purpose was to determine if extra cooling requirements were needed in order to meet the demand for increased throughput. Information on the effect of changing temperature T_1 was of primary importance. Therefore, PLEX-3 was set up to study throughput rate (x_6) of A and S combined, temperature T_1 (x_7) and also pressure P_1 (x_8) in reactor 1. The plant experiment was a 2^3 factorial design for 3 variables in eight runs plus repeats and center point (Table 3).

To show that he was still very much present, the ghost played havoc with the pressure instrument thus making the low pressure readings, at best, approximate. However, high pressure runs were still judged to be meaningful and showed the main effects of temperature T_1 and throughput rate to have negligible effects over the range of the variables studied. There was evidence of a slight temperature-pressure interaction which could not be confirmed because of difficulty in running low pressure runs. In a subsequent one variable study, where only temperature was varied, a small but significant effect was found due to temperature when varied over a 50% wider range.

TABLE 3. Statistically Designed Plant Experimentation (PLEX-3)

Run #	x_6	x_7	x_8	% yield
1	-1	-1	-1	74.7, 73.0
2	+1	-1	-1	73.2
3	-1	+1	-1	72.8
4	+1	+1	-1	71.6, 72.6
5	-1	-1	+1	72.9, 71.4
6	+1	-1	+1	71.6
7	-1	+1	+1	73.8
8	+1	+1	+1	72.8
9	0	0	0	72.2



The important results of the PLEX studies in this reaction step were listed as follows:

- (1) identification of the important yield and capacity limiting variable;
- (2) reduction in reactant *B* level;
- (3) information for future cooling requirements;
- (4) information on a total of eight variables;
- (5) direction for new yield and capacity improvements with a potential savings of an additional 1.5 million pretax dollars yearly;
- (6) confidence in running reaction step.

In essence, the PLEX studies provided valuable process improvement information plus considerable cost savings, and new levels of profitability.

Discussion

Two approaches to plant experimentation have been discussed with specific examples on the PLEX approach. The second approach, EVOP, has not been used as yet in the plant discussed here. However, it should be pointed out that during the course of the studies on the third reaction step, people referred to the PLEX program as an EVOP program because it involved designed experiments in a plant. But there is a difference, mainly in that a PLEX study is a series of one-shot efforts for information gathering under technical supervision, whereas EVOP is a continuous operating program evolving toward an optimum. Some may argue the difference is of no consequence as long as information is gathered with no upset in production. Both approaches have this goal and hence do not differ in purpose. However, they do differ in execution and type of supervision. Also, in PLEX, the variables are usually changed over wider ranges of conditions, but this is done under the alert eye of the technical supervision, so that warnings of upset are quickly detected and corrected. In EVOP, the changes in variables are usually smaller with less risk of upset. However, with smaller changes, EVOP would normally require more data and longer time to establish the significant variables. In choosing between EVOP and PLEX, one must carefully assess risk, urgency of information, manpower and other important production factors.

Finally, success in the case history was due to a cooperative effort of people from plant operating staff, plant technical staff, analytical, R&D, and engineering personnel. No single administrator, engineer, chemist or statistician can claim but a small part of the total effort. Therefore, duality of authorship is not to be interpreted as duality of effort. Rather, it was a presumption on the part of the authors to think that other people would be interested in learning about a successful application of plant experimentation.

Epilogue

The question may be asked "Did you catch the ghost?" The following vague answer will be left with the reader. Shortly after a number of the process questions were resolved and the process was improved substantially, a power failure occurred. The power failure was traced to a racoon which had caused its own demise by coming too close to an important power supply. Initially, there was some conjecture that the racoon was our mischievous

ghost which had made his last "mortal" move. However, we know better since the ghost still comes to visit us periodically, but considerably less frequently.

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