DEVELOPMENT OF A DETERGENT TEST—Part II

A Study of the Controllable Variables of the Test

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A design often found useful in this type of test [i.e., a Confounded Block Design] is described for studying effects on the test of those variables, such as volume or concentration of detergent, quantity of soil and temperature of operation, which can be controlled at different values. Ninety-six tests by the Classical Method would be required to yield the same amount of information as 32 by the method described and, in addition, interaction between the variables can be detected by the latter, but not by the former. Detergent concentration is found to have greater influence than the amount of detergent solution on the number of plates washed, which is found not to be directly proportional to the quantity of the solution, and very accurate temperature control is found unnecessary.

TEST PROCEDURE

THE NEXT stage in the evaluation of the test procedure is to consider the controllable variables and determine how closely and at what levels they ought best to be controlled. The variables selected for the first experiment were :

Temperature of wash liquor—47°, 55°.
Volume of wash liquor—3 litres, 4½ litres.
Quantity of soil per plate—4 g, 8 g.

Each variable was studied by performing tests at the two levels indicated. In order that the conclusions to be drawn from this test would be applicable over a reasonably wide range of practical conditions, it was necessary to use detergents that would cover the desired range of about 4 to 30 plates. Four solutions were used (as before, these were different quantities of the same material) making a total of $2 \times 2 \times 2 \times 4 = 32$ tests.

Only eight tests can conveniently be completed per day, and thus the series will extend over four days, but a previous experiment has shown that the soil can change appreciably from day to day and this effect must be eliminated in some way. The 32 tests must be divided into four groups of eight in such a manner that a large change from one group to the other will not affect the variance calculated for any of the main effects or for any simple (i.e. first order) interaction. The design that will achieve this is known as a "confounded block design."

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The need for this design of experiment frequently arises in detergency testing, for the size of a batch of uniformly soiled material is usually too small to enable all the tests to be carried out on it, and different batches of material will show appreciably different results. The principles of confounding are described in detail by Brownlee[5], where references to earlier works will be found.

In the present example we have three factors at two levels and one—quantity of detergent—at four levels. Suppose the former are denoted by T, V and Q in the order listed at the beginning of the section and the use of the higher level of the variable in any test be designated by t, v or q. The other variable is studied at four levels, but the experiment is simplified if we regard this as a combination of two factors, A and B, and denote the successive levels of detergent quantity by a, –, b and ab. Test abqv, for example, is that using the highest quantity of detergent, plates with 8 g. soil on each, and $4\frac{1}{2}$ litres of water at 47° C. ; and test qt is the use of the second detergent quantity, of 8 g. soil per plate, and 3 litres of water at 55° C.

In designing this experiment with variable wash solution volume, a decision had to be made whether to use four different concentrations of a detergent or four different total quantities. The latter was adopted in order to simplify the preparation of the solutions and the four quantities used were 4, 6, 9 and $13\frac{1}{2}$ g. of stock Nansa solution.

A complete analysis of variance of 32 tests involving the five variables A, B, Q, T, V would yield information concerning five main effects, 10 first order interactions, 10 second order interactions, 5 third order interactions and 1 fourth order—a total of 31 effects each with one degree of freedom. In the adopted block design three of these are to be confounded : i.e. the design will be such that differences between the blocks would contribute to the variance of only three of the interactions and will not affect any of the others. Naturally, we do not wish to lose any information concerning the main effects or their first order interactions, but will choose to sacrifice the results concerning the interactions AQT, BQV and ABTV. Two third order interactions cannot be used without affecting one of first order(which determines our choice of one third order and two second order), and the final selection was made such that each group of tests at the same soil level contained one test with each detergent quantity. If this were not done there might be a risk of insufficient soiled plates being available for a block containing a preponderance of the higher concentrations.

The division of the tests between the four blocks was carried out according to one of the usual methods (see Ref. 5) and then days, operators, and order of testing were allocated by using a table of random numbers giving the design :

Block			*Test Conditions*					*Day*	*Operator*	
I	1	at	bv	abq	bqt	qtv	abtv	3	Z	p.m.
II	a	t	abv	bq	abqt	aqtv	btv	2	Z	a.m.
III	b	abt	v	aq	qt	bqtv	atv	4	X	a.m.
IV	q	aqt	bqt	ab	bt	tv	abqtv	1	X	p.m.

Apart from changes of the variables being studied, the experiment was carried out in the same manner as the previous one[6] and the results were as

TABLE IX

Original Data, Experiment 2

Date	15		16		17		19	
Operator	X		Z		Z		X	
Soil quantity	4 gr.	8 gr.	8 gr.	4 gr.	4 gr.	8 gr.	8 gr.	4 gr.
4 g. Nansa	7t	6	6tv	13t	8tv	10t	6v	9
6 g. Nansa	13tv	9t	7v	15	11v	12	12tv	14t
9 g. Nansa	18v	11v	12	15tv	17	15tv	13t	16v
13½ g. Nansa	17	12tv	16t	20v	25t	13v	16	22tv

Statistical Analysis

For the statistical analysis we transform the results to values of 100 × (logarithm of plates −1) and rearrange as Table X.

TABLE X

	4 g. soil per plate				8 g. soil per plate			
	3 litres		4½ litres		3 litres		4½ litres	
	47°	55°	47°	55°	47°	55°	47°	55°
4 g.	−5	11	−15	−10	−22	0	−22	−22
6 g.	18	15	4	11	8	−5	−15	8
9 g.	23	26	20	18	8	11	4	18
13½ g.	23	40	30	34	20	20	11	8
Total	59	92	39	53	14	26	−22	12

The simplest method of performing an analysis of variance for a confounded block experiment is first to proceed by ignoring the block differences. The result is the same as that of a complete five-factor experiment, except that in the present instance we shall revert to considering the detergent quantity as a single variable at four levels.

TABLE XI

ANALYSIS OF VARIANCE, EXPERIMENT 2

Source of Variance	d.f.	S.S.	M.S.
Between detergent amount (D)	3	5189	1730 ***
Between soil amounts (S)	1	1418	1418 ***
Between temperatures (T)	1	270	270 **
Between volumes (V)	1	371	371 **
Detergent amounts × soil amount	3	39	13
Detergent × temperature	3	67	22
Detergent × volume	3	136	45
Soil amount × temperature	1	0	0
Soil amount × volume	1	3	3
Temperature × volume	1	1	1
D × Q × T	3	108	36 C
D × Q × V	3	100	33 C
D × T × V	3	386	129 C
Q × T × V	1	52	52
D × Q × T × V	3	142	47
Total	31	8282	—

The interactions marked C were those which were confounded with days which means that operator and age effects will have contributed to these terms. We now correct for this by calculating the S.S. for the days, which is 286, the day totals being 30, 68, 94 and 81, i.e. $(30^2 + 68^2 + 94^2 + 81^2)/8 - (30 + 68 + 94 + 81)^2/32 = 286$.

For estimate of error variance we combine the second and third order interactions and deduct the days effect to give a value of $502/10 = 50{\cdot}2$. Neglecting the day-to-day variations would have given a residual variance of $788/13 = 60{\cdot}7$, and thus the block design has given a slight improvement. Nevertheless, the residual variance is greater than the variance of all but one of the first order interactions above. This suggests that one of the interactions chosen for confounding with days is itself significant, or alternatively that the days effect (which includes that of changing the operator) interacts with one of the other variables.

This high error, however, will not invalidate any of the tests of significance which may be applied to the data but will simply cause some loss of precision. Pooling the smallest first order interaction terms with the residual of 502/10 gives an error variance of $612/19 = 32{\cdot}2$, and compared with this the remaining interaction of samples × volumes is not significant. Finally, we use the F test to examine the significance of the main effects and find those marked ** to be significant (0·01 level) and those marked *** to be highly significant (0·001 level).

These effects will now be examined from the practical viewpoint and, for clearness, the results will be expressed as plates and compared as ratios instead of using the logarithms of plates and the differences between logarithms.

VOLUME OF WASH SOLUTION

One of the problems in the design of the experiment was to decide whether detergent quantity or detergent concentration would prove to be the more satisfactory variable. The former was chosen but the data now show the latter to be better as the volume effect is then more consistent. The means of four tests provide the data which is expressed below in the alternative forms :

Detergent Quantity gms.	4	6	9	13½
In 3 litres, mean plates = In 4½ litres, mean plates =	9½ 6¾	12½ 10¾	15 14¼	18½ 16¾
Ratio =	·72	·86	·95	·91

Detergent Concentration g/l	·89	1⅓	2	3	4½
In 3 litres, mean plates = In 4½ litres, mean plates =	— 6¾	9½ 10¾	12½ 14¼	15 16¾	18½ —
Ratio =	—	1·14	1·14	1·12	—

The first table shows that dissolving a certain amount of detergent in 50 per cent more water reduces the number of plates by between 5 per cent and 28 per cent. The second table shows that the use of 50 per cent more detergent solution at constant concentration washes 12–14 per cent more plates. The latter is the more consistent effect showing that detergent concentration is the better selection for the variable. This conclusion will be a surprise to persons unfamiliar with the subject who may expect 50 per cent more solution to wash something approaching 50 per cent more plates.

QUANTITY OF SOIL

The table below shows the mean numbers of plates for the different soil quantities.

Detergent quantity	4 g.	6 g.	9 g.	13½ g.
4 g. soil per plate 8 g. soil per plate	9¼ 7	13¼ 10	16½ 12¾	21 14¼
Ratio	1·32	1·33	1·30	1·48

With half as much soil per plate, somewhere between 30 per cent and 50 per cent more plates can be washed. This result, too, will surprise those viewing the detergency operation as principally an interaction between soil and the detergent solution.

Temperature of Wash Solution

The main results here show an increase of between 7 per cent and 32 per cent in the numbers of plates washed at 55° with 47°.

Detergent quantity	4 g.	6 g.	9 g.	13½ g.
47° mean plates	7	11¼	14	16½
55° mean plates	9¼	12	15¼	18¾
Ratio	1·32	1·07	1·09	1·14

The data are very variable, but sufficient to show that temperature is not a highly critical factor, for the change is of the order of 1–2 per cent per degree.

Summary

The second experiment has illustrated how the effects and the interactions of a number of factors can be simultaneously assessed in a single investigation. Only 32 tests—four days' work—were required to investigate three main variables over a range of four concentrations, and all 32 results provide information on each factor. With the classical approach of changing one variable at a time 32 tests would have to be made for each factor, giving a total of 96. Not only is the amount of work reduced by our factorial design, but two further advantages are possessed over the classical method. The first is that interactions can be detected : thus the starting temperature may have been an important variable when 4½ litres of wash solution were used, but of comparatively less importance with only 3 litres of solution. The classical design would not have detected any such effect, whereas the experiment above has provided adequate evidence regarding its non-existence. Secondly, the conclusions drawn from the former type of experiment would apply only to one set of conditions, whereas the effects revealed by the above work are more likely to be applicable to whatever working conditions for the detergency test that are finally decided upon.

The above experiment has been described exactly as it was carried out during a course of a series of investigations into the dishwashing test, and before it was made the writers had no prior knowledge of the nature of the soil quantity or solution volume effects. Consequently, it was expected that an experiment of such a wide scope, performed on such a small scale, would provide only a rough indication of the nature of the main variables, and leave many doubtful points to be cleared up later. The experiment has shown that 32 tests are sufficient to distinguish the main effects of the four variables from the variance due to error, and has also shown that detergent concentration is a better variable than detergency quantity, and the pilot experiment has thus served its purposes. But it has done much more than this and, exceeding all expectations, has established all the main effects at significance levels all better than 0·01, and shown all interactions to be non-significant. The largest interaction variation has not even reached a significance of p = 0·2, and as no effects are left as "probably significant" the investigation of the importance of the variables is complete.

REFERENCES

[5] K. A. Brownlee, "Industrial Experimentation," H.M.S.O. 1949 (a), pp. 154–161.
[6] W. B. Smith and A. Taylor, *J.S.C.C.*, VI, pp. 96–107.

WHITE OILS

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The early history of white oil manufacture is outlined, examples are given of its current wide use and reference made to the most recent spectroscope technique for determining its stability.

THERE ARE many formulations of the cosmetic industry which contain white oil; that is, hydrocarbon oil of petroleum origin, and it is this raw material which is the subject of this talk. White oils are, however, comparatively new entries into an art or science which has, at times, claimed the attention of priests, and even politicians, and of which there are relics dating back to 5,000 years ago. The petroleum refining industry is by contrast in its infancy.

Research shows that there are very few aids to beauty to-day which have not had their counterpart in earlier centuries, but apparently it was not until the early 1900's that cosmetic compounding became really scientific. Strangely, or perhaps one of the reasons for the development, was the introduction of white mineral oils about the same time. To-day, of course, the cosmetic industry of this country holds a very important part in the national economy. It engages a large number of people, directly and indirectly, in its manufacture and distribution, and it has a rapidly increasing output both on the home and on export markets.

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