❧The Application of Linear Programming and Regression Analysis to Light Duty Liquid Detergent Formulation

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A strategy to help the formulator obtain a good, low cost, light duty liquid detergent formulation which matches or exceeds the properties of a commercially available product in a small number of steps is presented in this paper. The strategy is a sequential procedure, making use of a formulator's prior knowledge of the system, results obtained in previous steps, regression analysis and linear programming. The linear model is improved at each step. In comparison with previously published methods of detergent formulation, a considerably smaller number of steps is required to arrive at a minimum cost formulation which meets the property specifications. The strategy is illustrated by matching the properties of a commercial detergent in less than nine steps using ten components, of which eight can vary.

Product formulation of a multicomponent system is not an easy task for a formulator. In general, the formulator will select from a number of components to be mixed or reacted to obtain a formulation which matches or exceeds one or more required properties or specifications. Minimum cost is also often required. The formulator, based on his/her experience and knowledge of the formulation, can use trial and error methods or the one factor-at-a-time method to obtain a formulation which will match all the required specifications. Statistical methods were developed to handle this problem in a logical manner. Generally, experimental designs and/or regression analysis are first used to find how the properties are related to the components of a multicomponent system. Thereafter, an optimization technique is used to obtain the optimal formulation of the system. The methods to accomplish this are described in various texts (1–6). Usually, however, these methods are used to optimize only one property. When more than one property of a formulation is measured, the formulator must either make some decisions about the relative worth of each property in the form of a desirability function or choose one property, usually cost, to be optimized and place minimum specifications on all other measured properties. Variations of these methods have been applied to detergent formulations (7–9). In one of these variations, Steinle et al. (8) used central composite experimental design, regression analysis and the modified simplex method to optimize a light duty liquid detergent system. They illustrated their method by finding a lowest cost formulation of six components (two fixed) in 30 experiments which met or exceeded five property specifications. Disadvantages of these methods are that, often, more experimental effort is required to reach an optimum formulation than the formulator is prepared to expend. This is especially so when the number of components to be varied is large. Also, most designs are not sequential and hence the formulator must finish all experiments in the design before optimization can be done. In addition, the only prior knowledge of the system that these methods require is the composition range of the components to be varied. However, an experienced formulator often has considerable qualitative knowledge of the system or may obtain it from the component supplier's technical literature. Experimental designs do not take this qualitative knowledge into account.

This paper describes a sequential strategy to help the formulator achieve a formulation with desired specifications and minimum cost in a small number of experiments. The strategy is based on a method used by Kavanagh (10) to find a paint and resin formulation. The aim of the strategy is to use the formulator's knowledge of the system to attempt to reduce the number of experiments which normally would be required to optimize the formulation using an experimental design followed by some optimization technique. It is assumed that each property is a function of certain formulator selected (FS) components in a small concentration range. However, when sufficient experiments have been done, the property model may change. Successive uses of multiple regression analysis and linear programming are applied at each step to obtain a formulation which meets the required specifications at the lowest cost. To start the strategy, a starting formulation is required. This formulation usually is based on the experience of the formulator and/or technical literature. A list of components, on which each property depends, is also required and may be obtained from the formulator's experience and/or technical literature. The model of the properties is improved after each step, as more and more results are obtained. The strategy is terminated when the predicted formulations do not differ significantly in cost or composition.

In this paper, the strategy is applied to a complex, 10-component, light duty liquid detergent formulation (LDLD). Available statistical packages are used to find the property equations and the solution to the linear programming (LP) model at each step. Two starting formulations, from different regions of component space, were used to test the usefulness of the strategy. One was the recommendation of an experienced formulator (M. Hosking, Shell Chemical [Australia] Pty. Ltd., personal communication). The other one was obtained from randomly assigning some reasonable values to the components in the system. The two formulations, obtained after applying the strategy, are similar in composition and cost and point to a small optimum region of component space which has minimum cost and meets all property specifications.

A side benefit of this strategy is that the formulation has to be designed only once. Thereafter the formulation is predicted by the strategy. For example, the correct amount of all bases and linear alkylbenzene sulfonic acid for neutralization is determined by the invariant neutralization constraint and does not have to be solved for at each formulation step.

EXPERIMENTAL

This strategy was applied to match five properties of a light duty liquid detergent (LDLD) which is sold commercially in Australia. The properties selected to be matched were clear point (CLPT), Geelong soil titration test (GSTT), Ross-Miles flash foam height (RMFH), Ross-miles five-min foam stability (RMFS), and viscosity (VISC). The components selected to prepare the LDLD were linear alkylbenzene sulfonic acid (LAS), alcohol ethoxylate (AEO), alcohol ethoxysulfate (AEOS), diethanolamine (DEA), triethanolamine (TEA), coconut diethanolamide (CDEA), sodium hydroxide (NAOH), sodium chloride (NACL), formalin and water.

All materials used in preparing the LDLD were of commercial or laboratory reagent grade and are listed in Table 1. Raw material costs were bulk supply costs/kg at the beginning of 1984. No attempt was made to take into account cost variation during these years. Published recommendations (11) were followed to prepare the LDLD. Two replicate LDLD's were prepared at each step of the detergent set beginning with the formulation recommended by an experienced formulator.

Five property tests, which were replicated three times, were carried out on each LDLD formulation. All formulations were tested one day after they were prepared. The Ross-Miles foam test was determined with 0.1% detergent concentration in 60 ppm water hardness at 50 C according to ASTM D1173-53. The Ross-Miles flash foam height (RMFH) was the height of the foam after all the detergent solution had delivered from the top pipette, whereas the drop in the foam height after five min (RMFS) was used as a measure of foam stability. Viscosity (VISC) was determined at 25°C according to ASTM D445-74. Clear point (CLPT) was determined by a method recommended by Shell Chemicals Australia Pty. Ltd. (12). A soil titration test (GSTT), used to simulate the dishwashing performance of the LDLD, was carried out according to a method recommended by Shell Chemicals Australia Pty. Ltd. (13). Due to day to day variation of the absolute values, the GSTT results are reported here as a percentage of the control test.

The commercially available LDLD of the control test had the properties listed in Table 2. These proper- ties are the average of nine control tests and are the specifications the strategy attempts to match.

The calculations were carried out on a DecSystem 20 (Digital Equipment Corp.) mainframe computer. The statistical package, MINITAB, (Minitab Inc., Pennsylvania) was used to find the regression coefficients of the property equations, and the package, LPBEST, (Deakin University Computer Center, Victoria, Australia) was used to solve the linear programming model.

THE STRATEGY

A number of improvements to the strategy previously described (10) were made here because a much more complex formulation was investigated. The properties all depend on several components, and the importance of some components was not certain. The strategy was divided into exploration and optimization stages. This division was made to ensure that during the exploration stage, an experimental region of sufficient composition range was covered. Sequential experiments were preferred to some experimental design during this stage so that a good experimental region was found even if starting from a poor experimental region. If the property dependent variables did not change sufficiently, then sufficient composition range was ensured by setting the required property value to a higher/lower figure depending on whether this property was currently below/above the specification value. A stage consists of a number of steps. Each step involves the following sequence: (a) prepare and test the new formulation; (b) obtain the best property equations; (c) transform the property equations to property constraints; (d) calculate the upper and lower bounds for all the components; (e) set up and solve the LP model to obtain a new formulation, and (f) decide whether to stop or continue.

As each property depended on a number of components, rules were formulated to add or reject a component from the model at each step. The components were divided into two types for each property, the formulator-selected (FS) components and the nonformulator-selected (NFS) components. The FS components were the components upon which the property was believed to depend, whereas the NFS components were the components which, it was first thought, would not significantly affect a given property. The same procedures, previously described (10), were used to calculate the property equations in the first two steps. In the latter steps, the property equations were made up of the simplest combination of the FS components that had a coefficient of determination greater than 0.90. However, in some instances, when none of the possible combinations of the components gave a coefficient of determination greater than 0.90, the best linear combination of FS components was chosen to be the property equation.

Bounds constraints were used to prevent violent fluctuation in the amount of components in a formulation, and were not fixed in all formulations. Upper and lower bounds were calculated, in general, by multiplying the component weight fraction in the best formulation to date by 1.5 and 0.5, respectively. Some adjustments were made to these figures, in individual instances, when necessary.

In some circumstances, no feasible solution was obtained for the early LP models. This was because the value of a component predicted by the property constraint could be much bigger/smaller than the upper/lower bound of that component. In order to solve this problem, the LP model was loosened by removing the property constraint which caused this problem, and the value of a particular component was set to its upper or lower bound as appropriate. In some other circumstances, no feasible solution was obtained because some property constraints used the same component to explain their variation and have an equality sign. This problem was solved by arranging properties to have an order of preference. For this LDLD system, the property preference was RMFH > GSTT > CLPT > VISC > RMFS.

The aim of the strategy was to match or exceed the properties of the commercial LDLD listed in Table 2. However, a viscosity in the range 150 to 500 cSt, and also a clear point less than 5°C, was considered acceptable. The percent active matter was set in the range of 10 to 12%.

Below we show how one typical LP was set up in a step. The only component chosen not to be varied was the preservative, formalin. Water was added as a diluent and was not included in any model of the LDLD system. The fourth LP of the set in which the first formulation was suggested by an experienced formulator is as follows:

$$1.38\text{LAS} = 7.17 \quad [1]$$

$$10.92\text{AEOS} + 23.00\text{CDEA} \geqslant 76.56 \quad [2]$$

$$220.9\text{NAOH} = 6.95 \quad [3]$$

$$25.49\text{NAOH} + 9.49\text{NACL} \leqslant 15.13 \quad [4]$$

$$\text{LAS} \geqslant 3.89 \quad [5]$$

$$\text{AEO} \geqslant 0.07 \quad [6]$$

$$\text{AEOS} \geqslant 3.33 \quad [7]$$

$$\text{CDEA} \geqslant 0.95 \quad [8]$$

$$\text{DEA} \geqslant 0.23 \quad [9]$$

$$\text{TEA} \geqslant 0.28 \quad [10]$$

$$\text{NAOH} \geqslant 0.19 \quad [11]$$

$$\text{NACL} \geqslant 0.56 \quad [12]$$

$$\text{LAS} \leqslant 6.48 \quad [13]$$

$$\text{AEO} \leqslant 1.67 \quad [14]$$

$$\text{AEOS} \leqslant 5.55 \quad [15]$$

$$\text{CDEA} \leqslant 1.58 \quad [16]$$

$$\text{DEA} \leqslant 1.83 \quad [17]$$

$$\text{TEA} \leqslant 1.28 \quad [18]$$

$$\text{NAOH} \leqslant 0.48 \quad [19]$$

$$\text{NACL} \leqslant 0.94 \quad [20]$$

$$\text{LAS} - 2.79\text{DEA} - 2.03\text{TEA} - 0.17\text{CDEA} - 6.95\text{NAOH} = 0 \quad [21]$$

$$\text{LAS} + \text{AEO} + 0.25\text{AEOS} + 0.86\text{CDEA} + 0.97\text{DEA} + \text{TEA} + 0.53\text{NAOH} \geqslant 10.0 \quad [22]$$

$$\text{LAS} + \text{AEO} + 0.25\text{AEOS} + 0.86\text{CDEA} + 0.97\text{DEA} + \text{TEA} + 0.53\text{NAOH} \leqslant 12.0 \quad [23]$$

The objective equation which is to be minimized is

$$\text{Cost} = 1.35\text{LAS} + 1.71\text{AEO} + 0.68\text{AEOS} + 1.20\text{CDEA} + 0.92\text{DEA} + 1.59\text{TEA} + 0.465\text{NAOH} + 0.117\text{NACL} \quad [25]$$

The cost is in Australian dollars/100 kg, and the coefficients are the cost/kg of the raw materials.

Constraints 1, 2, 3 and 4 were the RMFH constraint, the GSTT constraint, the VISC constraint and the CLPT constraint, respectively. All the property equations were obtained by assuming that the property was a linear function of the FS components in the LDLD system. Table 3 shows the list of FS components. They are listed in the order of preference and were suggested by an experienced detergent formulator (M. Hosking, Shell Chemical [Australia] Pty. Ltd., personal communication). RMFH and VISC constraints have only one component in them, whereas GSTT and CLPT constraints have two components in each. This was because none of the single component property models for GSTT and CLPT had a coefficient of determination greater than 0.90, and thus, one more FS component was added. Taking the CLPT constraint as an example, the respective coefficients of determination of CLPT to NAOH, NACL and NAOH and NACL together were 0.85, 0.52 and 0.99, we clearly see that adding the NACL component to the property model which already has the NAOH component will increase the coefficient of determination from 0.85 to 0.99.

Because the exploration stage was finished in the fourth step and the optimization stage was entered, the original specifications of the properties were used. With these specifications, the property equations were transformed to the property constraints. The RMFS constraint was dropped from the LP models after the third step because of the poor experimental accuracy and the difficulty in obtaining a model equation for it.

Constraints 5 to 12 and 13 to 20 were the lower and upper bounds of the components, respectively. The

TABLE 1

Raw Materials of LDLD

Material	Abbreviation	Percent by mass	Cost (A$/kg)
Linear alkybenzene sulfonic acid	LAS	94.0	1.35
Alcohol ethoxylate	AEO	100	1.71
Alcohol ethoxysulfate	AEOS	25	0.68
Coconut diethanolamide	CDEA	79	1.20
Diethanolamine	DEA	98.5	0.92
Triethanolamine	TEA	85	1.59
Sodium hydroxide	NAOH	97.0	0.465
Sodium chloride	NACL	99.9	0.117
Formalin	-	36.0	0.47
Water	-	-	0.0

TABLE 2

Properties of the Commercially Available LDLD

Property	Values
Ross-Miles flash foam height	10.3 cm
Ross-Miles 5-min foam height drop	1.0 cm
Geelong soil titration test	10.2 g
Viscosity (at 25 C)	214.3 cSt
Clear point	0°C
pH	6.6
Active matter	12.3%

TABLE 3

List of Formulator-Selected Components

Property	Formulator-selected components (in order of importance)
RMFH	LAS, AEOS, CDEA
RMFS	AEOS, CDEA
GSTT	AEOS, CDEA
VISC	NACL, NAOH, CDEA, DEA, TEA
CLPT	TEA, NACL, DEA, NAOH

upper and lower bounds of the components which were not used in any property models were obtained by multiplying the corresponding highest component weight fraction by 1.5 and the corresponding lowest component weight fraction by 0.5, respectively. On the other hand, the upper and lower bounds of the components, which were used in the property models, were obtained by multiplying the component weight, in the best formulation to date for that particular property, by 1.25 and 0.75, respectively. The smaller variation used for the components which were used in the property models was to prevent too great a fluctation in case of a poor model. Constraints 21 to 23 were the invariant constraints and were unchanged for all formulations. Constraint 21, the neutralization constraint, ensures that the LDLD formulations obtained are in the neutral region. Con-

TABLE 4

The LDLD Formulations for Which the First Formulation was Recommended by an Experienced Formulator

Component/ property	1	2	3	4	5	6	7
LAS	5.18	6.15	6.88	6.99	5.20	6.10	6.10
AEO	1.11	0.56	0.28	0.14	1.32	0.0	0.0
AEOS	4.44	5.38	5.67	7.12	3.68	2.45	2.03
CDEA	1.26	0.63	1.89	0.95	1.58	2.13	2.19
DEA	0.54	0.81	0.45	1.22	0.78	1.21	1.42
TEA	0.85	0.56	0.65	0.67	0.28	0.14	0.0
NAOH	0.25	0.38	0.57	0.30	0.32	0.30	0.25
NACl	0.50	0.75	0.58	0.25	0.56	0.28	0.28
Formalin	0.20	0.20	0.20	0.20	0.20	0.20	0.20
Water	85.67	84.58	82.83	82.16	86.08	87.19	87.53
RMFH (cm)	10.5	11.7	12.8	13.1	10.7	11.7	11.8
RMFS (cm)	1.0	0.8	1.1	1.0	1.0	1.0	1.0
GSTT (%)	99.0	98.5	130.0	121.9	100.9	103.5	102.5
VISC (cSt)	94.5	260.3	740.5	38.4	300.0	190.0	274.6
CLPT (C)	0.5	7.0	9.7	0.2	4.5	0.4	-0.1
pH	7.0	6.9	6.9	7.3	7.0	6.8	6.7
Active matter (%)	10.7	11.1	12.5	12.3	10.8	10.7	10.6
Cost (A$/100 kg)	15.54	15.67	17.77	18.11	15.15	14.06	13.79

TABLE 5

The LDLD Formulations for which the First Formulation was a Random One

Component/ property	1	2	3	4	5	6	7	8	9
LAS	4.00	5.36	7.97	8.33	5.58	5.58	6.23	6.35	6.27
AEO	3.50	1.75	0.88	0.44	1.29	0.77	0.22	0.11	0.0
AEOS	1.58	2.16	2.66	2.89	1.70	1.28	1.48	1.18	0.94
CDEA	1.18	1.37	0.75	0.38	1.72	2.28	1.80	2.16	2.46
DEA	0.50	0.75	1.13	0.89	0.67	0.98	1.24	0.90	0.90
TEA	0.50	0.25	0.46	0.13	0.38	0.29	0.29	0.30	0.30
NAOH	0.20	0.36	0.54	0.79	0.38	0.27	0.27	0.41	0.39
NACL	0.50	0.75	0.88	0.56	0.38	0.29	0.41	0.30	0.30
Formalin	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
Water	87.84	87.05	84.53	85.39	87.70	88.06	87.86	88.09	88.24
RMFH (cm)	9.4	10.0	12.7	12.7	10.5	9.7	11.6	11.3	11.5
RMFS (cm)	1.0	0.8	1.2	0.9	1.0	0.9	1.0	1.0	1.0
GSTT (%)	87.7	106.7	112.6	110.6	102.8	108.6	102.1	100.0	103.0
VISC (cSt)	80.7	126.6	191.1	396.3	257.2	291.0	446.4	350.3	448.6
CLPT (C)	0.5	25.8	27.8	17.4	1.1	0.9	0.2	0.7	1.9
pH	6.9	6.6	7.0	6.9	6.8	6.7	6.8	6.8	6.7
Active matter (%)	10.4	10.9	13.0	11.9	10.6	10.5	10.7	10.6	10.5
Cost (A$/100 kg)	15.37	14.78	17.20	15.97	14.50	14.07	13.82	13.78	13.67

straints 22 and 23, the active matter constraints, ensure that the LDLD formulations obtained are between 10 and 12% active matter.

This LP model was solved by the LPBEST computer program, and the fifth formulation of the first set was obtained.

Each step thus consists of building up and solving an LP model to obtain a new minimum cost formulation, and producing and testing this formulation. This procedure is repeated until a formulation with the desired properties is obtained or until a model, of sufficient accuracy to predict that the desired properties are unobtainable with the selected components, is obtained.

Starting with a formulation suggested by an experienced formulator, six LP models and seven LDLD formulations were used to show that the seventh formulation was a good low cost formulation and satisfied all the specifications. Similarly, eight LP models and nine LDLD formulations were used to achieve the same result as before when starting with a random formulation. All the formulations and properties of these formulations are listed in Tables 4 and 5. The models of the properties are summarized in Table 6.

TABLE 6

Property Equations

Property equation	R^2
Starting with a recommended formulation	
1. RMFH = 3.68 + 1.33LAS	0.98
2. GSTT = 27.44 + 9.98AEOS + 24.20CDEA	0.96
3. VISC = -550 + 139CDEA + 1800NAOH	0.92
4. CLPT = -9.28 + 23.88NAOH + 9.54NACL	0.98
Starting with a random formulation	
1. RMFH = 5.65 + 0.87LAS	0.89
2. GSTT = 11.76 + 30.28AEOS + 24.66CDEA	0.90
3. VISC = -2173 + 529CDEA + 286DEA + 787NAOH + 2671NACL	1.00
4. CLPT = -7.66 - 32.25TEA + 54.64NACL	0.95
Combined data	
1. RMFH = 4.29 + 0.93LAS + 0.26AEOS + 0.44CDEA	0.91
2. GSTT = -24.45 + 13.08LAS + 10.16AEO + 5.00AEOS + 17.52CDEA	0.91
3. VISC = -3329 + 380LAS + 210AEO + 487CDEA + 96TEA - 630NAOH + 1360NACL	0.98
4. CLPT = -26.15 + 2.97LAS - 7.68TEA + 34.78NACL	0.79

DISCUSSION

Tables 4 and 5 show that all components vary significantly and cover an experimental region that should be considered large by an experienced formulator. Starting with two formulations from different points in this region, the strategy produced two formulations which are similar in composition and have similar properties and costs. However, it does not appear from Tables 4 and 5 that the two formulations will converge, except possibly, very slowly. This is because only linear models were used and the costs of the components do not vary much, and also because of the possible shape of the optimum region. To investigate this region further, two more formulations were made. Formulation one in Table 7 was found by combining the experimental data from both starting points, finding the best linear models, by stepwise regression, for each property and using linear programming to find the minimum cost formulation to meet the required specifications. Formulation two in Table 7 was found by averaging the component weights in the two optimum formulations, taking into account the neutralization constraint. The properties and costs of these formulations do not differ significantly from the best formulations found after starting from two different points in the original experimental region.

The cost of all formulations, as shown in Tables 4, 5 and 7, are seen to converge to the same cost. There is obviously no reason to predict further formulations from either starting point. The cost of the formulation predicted by combining the two sets of results is very slightly lower than the others in the region and is the local cost minimum as it is predicted from the linear program with the property constraints removed and component bounds set to the values in the two optimum formulations. It appears that the optimum region for this LDLD system is rather broad and flat.

In all the best formulations, we see that the values of RMFH are better than the specified value. Thus, it may be possible to find a lower active matter formulation which will satisfy all the specifications. Two additional formulations were made to test this assumption. Formulation three in Table 7 was obtained using the same LP as in formulation one but with the lower active matter constraint removed. Similarly, formulation four was obtained as in formulation three but with the lower bound of LAS removed. Within experimental error, the properties of these formulations satisfy the specifications but the GSTT is borderline for formulation four.

TABLE 7

The Formulation and Properties of Four Formulations in the Optimum Experimental Region

Component/ property	1	2	3	4
LAS	6.27	6.19	6.10	5.86
AEO	0.0	0.0	0.0	0.0
AEOS	0.94	1.49	0.94	0.94
CDEA	2.29	2.31	2.28	2.46
DEA	1.42	1.16	1.08	0.98
TEA	0.0	0.10	0.0	0.0
NAOH	0.28	0.34	0.39	0.39
NACL	0.24	0.26	0.24	0.24
Formalin	0.20	0.20	0.20	0.20
Water	88.36	87.95	88.77	88.93
RMFH (cm)	11.6	11.8	11.5	10.9
RMFS (cm)	1.0	0.9	1.0	1.0
GSTT (%)	100.0	102.0	102.2	98.9
VISC (cSt)	311.9	304.9	215.5	282.3
CLPT (C)	0.6	0.9	0.5	1.6
Active Matter (%)	10.5	10.6	10.0	9.9
pH	6.6	6.7	7.0	6.8
Cost (A$/100 kg)	13.41	13.65	12.91	12.71

The goodness of the property models can be tested by the lack of fit statistical test provided that genuine replicates of samples are prepared and tested (4,6). Thus, we can apply this test to the set of data of which the first formulation is recommended by an experienced formulator, and the set of combined data. The results of the test show that there is no evidence of lack of fit for the property models of RMFH and GSTT. However, there is a significant lack of fit for the property models of VISC and CLPT. Consequently, the linear statistical model is adequate for RMFH and GSTT but not for VISC and CLPT. Similar conclusions are found by Steinle et al. (8) despite the fact that the two systems are not very similar and are in a different region of component space.

The property models found from different starting points are not exactly the same. Further, the property models found by MINITAB stepwise regression using FENTER = FREMOVE = 3.0 (14) from the set of combined data are also different. These property models are shown in Table 6. Although the models found from different starting points are satisfactory within their own set of data, as shown by their large coefficient of determination, the models of the first set do not predict very well the properties of the second set and vice versa. This is to be expected, considering the lack of fit of the viscosity and clear point models and the different regions of component space covered by the two sets of data. However, both these models predict very well the properties of the four additional formulations. The experimental results of these four formulations nearly all fall within the 95% prediction interval estimates of the properties. Hence, these property models are adequate within their own set of data and predict well in the small optimum region.

In comparison with some experimental design followed by some optimization technique, the strategy suggested has found a small optimum region of component space with much less experimental work. However, the property models found with this strategy are less meaningful in that not all significant components are in the property equations and the property equations vary, depending on the region of component space covered in reaching the optimum region. However, we believe this strategy has some value where sufficient qualitative knowledge can be aquired to make a list of formulator selected components for each property and when the much larger experimental effort required for a more rigorous experimental design cannot be justified.

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