Investigation of the Processing Parameters on the Chemical Removal of Fouling Lead Layer by Using an Orthogonal Array Design Method

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Abstract: In this research, an orthogonal array design (OAD), OA9 was exploited as a chemometric experimental method for the cleaning of chromium surfaces contamination by using an electrochemical cleaner solution that is established upon the redox reaction principle. The influence of cleaning procedure on the removal of lead fouling layer has been investigated by means of SEM/EDAX and ICP analysis. In order to estimate of the main effect of some experimental parameters, we examined the procedure with a statistical method and the results were quantitatively evaluated by the analysis of variance (ANOVA). The OAD estimation of experimental result provides optimized amount of the parameters including, H₂O₂ 10% (V/V), stirring speed of the mixture 75 rpm and temperature 28°C, to achieve the most cleaned sample, at which the total removal time of 3 g fouling lead layer is predicted only 7 minutes. The prediction has a complete conformity with experimental result achieve at the similar condition that is 8 minutes. Also, the reaction rate constant of the lead bar oxidation at optimum condition was obtained from the ICP spectroscopy data that proposed by Ostwald approximation method. Finally, the results of this study illustrated that the efficiency of the proposed chemometric cleaning method is higher than that our partner sequential experimental technique.

Key words: Taguchi method · Surface cleaning · Lead pollution · Redox reaction · Cleaner solution · Statistical optimization

INTRODUCTION

Chromium coatings are applied on the surface of numerous metals such as iron, cobalt and nickel-base alloys for decorative purposes to enhance resistance and to lower the coefficient of friction. Generally, chromium is oxidized readily in the air and forming a thin nano-scale layer, adherent and transparent coating of Cr₂O₃ [1]. However for this reason some common metals such as aluminum, chromium, zinc and nickel stay bright in the clean inside atmosphere. The most important application of chromium is in the production of steel. However, high-carbon and other grades of ferrochromium alloys are added to steel to improve mechanical properties, increase hardening and enhance corrosion resistance [2-3].

Almost all metallic surfaces products need clean-up procedure to take out surface contaminations that remain after processing or employment [4-9]. However, research on the lead cleaning methods are an interesting subject since lead fouling on the chromium surfaces is a common difficulty in the metallic systems that having interaction with metallic lead in high temperatures and pressures [10-12].

Our recent investigation revealed that H₂O₂/CH₃COOH redox system can provide a low-cost, simple, fast and effective cleaner solution to introduce a novel pickling solution in order to cleaning the lead fouling layer from chromium surfaces and the quality of procedure is influenced by some experimental condition [13].

Nevertheless, in order to approximate the parameters influencing on the procedure, we require using a statistical method for improvement of efficiency of the process. Consequently, an orthogonal array design (OAD) method has been used to evaluate and to optimize the most important
parameters affecting the cleaning quality and increasing the efficiency of the proposed cleaning procedure [14].

Optimization is a significant step in upward a cleaning procedure. That is containing two broad organized methods containing simultaneous and sequential methods [15-16]. In second methods (e.g. simplex optimization), the response surface is consecutively followed up to a most favorable has been achieved [17]. Deficiencies of sequential methods are slow convergence on the complex response surface and difficulty in dealing with the response surface with high dimensionality. However, these methods could be suitable as a means for fine-tuning a separation [18]. On the other hand, the former optimization methods, such as mixture designs [12, 19] and factorial designs, [20-22] do not have these difficulties.

In our study, Taguchi L-9 orthogonal array design as a simultaneous method was employed to display the effect of four procedure parameters that is percent of oxidizing agent (H₂O₂), percent of chelating agent (CH₃COOH), stirring speed of the mixture (revolutions per minute, rpm) and temperature of the cleaner solution on the cleaning qualities. This plan contains nine separate experiments with particular level and a precise combination of parameters. On the other hand, the sequence in which the experiments were carried out was fortuitously to keep away from any kind of personal or subjective bias, which may be conscious or unconscious. This method ensures greater validity of test results [23-27]. For instance, in four parameters at three levels condition, the conventional full factorial design would need 81 experiments. But, in the present design (Taguchi L-9 orthogonal array), the necessary experiments are merely nine [28]. The analyses of the data, assuming no interaction between variables, are containing: (1) identification of the optimum condition, (2) finding of the individual influence of each factor and (3) estimation of the result at the optimum condition [29-30].

The aim of this investigation was to verify how the different parameters influence on the removal of deposited lead layer from chromium surfaces. Consequently, an experimental design method was employed to examine the influences of these parameters and the results have been examined by Scanning electron microscopy (SEM), Energy dispersive X-ray microanalysis (EDAX) and Inductively coupled plasma spectroscopy (ICP) analysis techniques. Finally, on the basis of the ANOVA results, optimum condition for the lead cleaning of polluted surface has been proposed and the performance of investigated chemometric method has been compared with our pervious sequential experimental method.

Experimental Part
Materials and Apparatus: Aqueous hydrogen peroxide solution (H₂O₂) 35% (V/V) and acetic acid (CH₃COOH) 100% (V/V) were obtained from Fluka (Tehran, Iran). ASTM B 32 standard lead bar (with 99% purity) was prepared from local industrial Zanjan zinc-lead Co. (Tehran, Iran).

Optimization of Lead Dissolving Procedure: An experimental design method was employed to optimization experimental parameters for the dissolving procedure [16-17]. The variables including percent of oxidizing agent...
(H\textsubscript{2}O\textsubscript{2}), percent of chelating agent (CH\textsubscript{3}COOH),
stirring speed of the mixture (rpm) and temperature of the
cleaner solution were changed as shown in Tables 1 and 2. However optimum conditions which proposed by
ANOVA method were employed for further lead bar oxidation studied.

Kinetic Measurements: In order to inspect the kinetic of
the oxidation reaction of metallic lead bar with cleaner
solution at optimum conditions (proposed by ANOVA
method), lead ion concentration as a function of time at
aqueous solution was followed by ICP spectroscopy
technique.

Treatment of Real Sample: To estimate the usefulness of
the proposed method for treatment of a polluted chromium
surface a steel plate sample that electroplated with a
specified thickness of chromium layer was used. Then,
about 3 g of metallic lead as a pollution was deposited on
the chromium surface in order to exploit as a real sample.
However the dirty metallic surface was treated by
cleaner solution at the ANOVA proposed optimum
condition and the efficiency of the method was precisely
evaluated.

RESULTS AND DISCUSSION

Dissolving Results and Optimization Strategy: In this
research the parameters are composed of percent of
oxidizing agent (H\textsubscript{2}O\textsubscript{2}), percent of chelating agent
(CH\textsubscript{3}COOH), stirring speed of the mixture (rpm) and
temperature of the cleaner solution. The tested factors
and the levels are given in Tables 1 and 2. Also,
dissolving time of 3 g lead bar is presented in the last
column of Table 2.

The average values of the coefficients of variation of
the factors at each level were calculated according to
assignment of the experiment (Table 3) [17-19]. For
instance, to calculate the average value of dissolving time
of 3 g lead bar for the effect of the percent of oxidizing
agent (H\textsubscript{2}O\textsubscript{2}) at level 1, dissolving time of the nine trials in
which percent of oxidizing agent (H\textsubscript{2}O\textsubscript{2}) was set at
level 1 (Trials 1, 2 and 3) were pooled and divided by
the numbers of the values (three values). The average
value of the three levels of a parameter displays how
dissolving time will change when the level of the
parameter is altered.

The analysis of variance (ANOVA) for the results
dissolving time is shown in Table 4. In this inquiry, the
influence of percent of oxidizing agent (H\textsubscript{2}O\textsubscript{2}) on the
dissolving time at three diverse levels (5, 7.5 and 10% V/V)
was studied. It was recognized that percent of oxidizing
agent is an important factor for the control of the
dissolving time of lead bar. Also, it was appear that
temperature of the cleaner solution is an important
parameter that could influence the dissolving time of lead
bar. Conversely, our finding confirmed that percent of
chelating agent (CH\textsubscript{3}COOH) during the dissolving
process is not a considerable parameter for the control of
dissolving time of lead bar.

Finally, the influence of stirring speed of the mixture
(rpm) on the dissolving time of lead bar was examined at
diverse values: 25, 50 and 75 rpm. The results
illustrated that stirring speed of the mixture can be
considered as a significant factor on the dissolving time
of lead bar.

Evaluation of the ANOVA results of this
investigation show that (at 90% confidence level), with
omitting percent of chelating agent (CH\textsubscript{3}COOH), all
variables (percent of oxidizing agent (H\textsubscript{2}O\textsubscript{2}), stirring speed
of the mixture (rpm) and temperature of the cleaner
solution) have considerable effects on the dissolving time
of lead bar during the process.

The consequences of ANOVA disclosed that the
excellent optimum conditions for the producing of
cleaning solution are 10% V/V of oxidizing agent (H\textsubscript{2}O\textsubscript{2}),
75 rpm stirring speed of the mixture and 28°C temperature
of the cleaner solution.

As a common rule, the maximum performance
(in our study, the shortest time for dissolving of lead
bar) will be obtained by means of the subsequent
equation [17]:

<table>
<thead>
<tr>
<th>Process parameter</th>
<th>Symbol</th>
<th>Level 1</th>
<th>Level 2</th>
<th>Level 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxidizing agent</td>
<td>O</td>
<td>5</td>
<td>7.5</td>
<td>10</td>
</tr>
<tr>
<td>Chelating agent</td>
<td>C</td>
<td>10</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>Stirring speed of the mixture (rpm)</td>
<td>R</td>
<td>25</td>
<td>50</td>
<td>75</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>T</td>
<td>15</td>
<td>21</td>
<td>28</td>
</tr>
</tbody>
</table>

Table 1: The various parameters selected and their respective levels in the present experimental design.
Table 2: Taguchi L-9 orthogonal array design and dissolving time of 3 g lead bar.

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Oxidizing Agent (%)</th>
<th>Chelating Agent (%)</th>
<th>Stirring speed of the mixture (rpm)</th>
<th>Temperature (°C)</th>
<th>Dissolving Time (minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>10</td>
<td>25</td>
<td>15</td>
<td>72</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>20</td>
<td>50</td>
<td>21</td>
<td>66</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>30</td>
<td>75</td>
<td>28</td>
<td>41</td>
</tr>
<tr>
<td>4</td>
<td>7.5</td>
<td>10</td>
<td>50</td>
<td>28</td>
<td>31</td>
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<tr>
<td>5</td>
<td>7.5</td>
<td>20</td>
<td>75</td>
<td>15</td>
<td>39</td>
</tr>
<tr>
<td>6</td>
<td>7.5</td>
<td>30</td>
<td>25</td>
<td>21</td>
<td>34</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>10</td>
<td>75</td>
<td>21</td>
<td>19</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>20</td>
<td>25</td>
<td>28</td>
<td>14</td>
</tr>
<tr>
<td>9</td>
<td>10</td>
<td>30</td>
<td>50</td>
<td>15</td>
<td>38</td>
</tr>
</tbody>
</table>

Table 3: Results of the main effects for each variable on the dissolving time of 3 g lead bar

<table>
<thead>
<tr>
<th>Process parameter</th>
<th>Symbol</th>
<th>Level 1</th>
<th>Level 2</th>
<th>Level 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxidizing agent</td>
<td>O</td>
<td>59.7</td>
<td>34.7</td>
<td>23.7</td>
</tr>
<tr>
<td>Chelating agent</td>
<td>C</td>
<td>40.7</td>
<td>39.7</td>
<td>37.7</td>
</tr>
<tr>
<td>Stirring speed</td>
<td>R</td>
<td>40.0</td>
<td>45.0</td>
<td>33.0</td>
</tr>
<tr>
<td>Temperature</td>
<td>T</td>
<td>49.7</td>
<td>39.7</td>
<td>28.7</td>
</tr>
</tbody>
</table>

Table 4: ANOVA table for the dissolving of lead bar with redox procedure by OA9 (3^3) matrix with the dissolving time of 3 g lead bar as the response.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Code</th>
<th>DOF</th>
<th>S</th>
<th>V</th>
<th>DOF</th>
<th>S'</th>
<th>F'</th>
<th>P'</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxidizing agent</td>
<td>O</td>
<td>2</td>
<td>2042</td>
<td>1021</td>
<td>2</td>
<td>2042</td>
<td>145.8</td>
<td>69</td>
</tr>
<tr>
<td>Chelating agent</td>
<td>C</td>
<td>2</td>
<td>14</td>
<td>7</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Stirring speed</td>
<td>R</td>
<td>2</td>
<td>218</td>
<td>109</td>
<td>2</td>
<td>218</td>
<td>15.6</td>
<td>7</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>T</td>
<td>2</td>
<td>662</td>
<td>331</td>
<td>2</td>
<td>662</td>
<td>47.3</td>
<td>22.1</td>
</tr>
<tr>
<td>Error</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>14</td>
<td>-</td>
<td>1.9</td>
</tr>
</tbody>
</table>

CI = \( Y_{opt} \pm \frac{F_\alpha(f_1, f_2)W_e}{n_e} \) \tag{3} 

where \( F_\alpha(f_1, f_2) \) is variance ratio for degree of freedom (DOF) \( f_1 \) and \( f_2 \) at the level of significance \( \alpha \) (in this work, \( \alpha = 90\% \)) and the confidence level is (1- \( \alpha \)), \( f_1 = \) DOF of average (which constantly equals 1), \( f_2 = \) DOF of the error term, \( n_e = \) number of equivalent replications and given by \( n_e = \) number of trials/ DOF of average (always 1)+DOF of all factors results used in the estimation. Statistical calculations for determining the result and CI at optimum condition revealed that the dissolving time of lead bar with the shortest time will be 6.8 ± 0.3 minutes.

In the next step of our inquiry, 3 g lead bar was treated at obtained optimum condition (10% of oxidizing agent, 75 rpm stirring speed of the mixture and 28°C
temperature of the cleaner solution). The results of experiment revealed that dissolving time of 3 g lead bar at the predicted optimum condition is 8 minutes. The cleaning solution at this condition was employed for the kinetic studies of lead bar oxidation.

**Kinetic of Lead Reaction:** The reaction rate was studied to achieve more information from the mechanisms of the oxidation reaction. Our last investigation [13] illustrated that in the present method, successive reaction was occurred in the cleaner solution:

\[
Pb^{2+} (aq) + 2e^- \rightarrow Pb(s) \quad \text{First stage} \quad (4)
\]

\[
2CH_3COOH (aq) \rightarrow 2CH_3COO^- (aq) + 2H^+ (aq) \quad \text{Second stage} \quad (5)
\]

\[
Pb^{2+}(aq) + 2CH_3COO^- (aq) \rightarrow Pb(CH_3COO)_2(s) \quad \text{Total reaction} \quad (6)
\]

For the oxidation reaction of lead bar, equation (4) is the rate-determination step of the reaction. However, lead ions in aqueous solution will form lead acetate complexes with acetate ions (reaction 6). Consequently, the overall kinetic equation for the reaction rate could be resulting from the reaction rate of this equation (6). The reaction rate could be concluded by applying the next equation:

\[
R = \frac{dx}{dt} = K(Pb^{2+})^\alpha (CH_3COO^- - x)^\beta \quad (7)
\]

However, in this study oxidizing agent (H2O2) and chelating agent (CH3COOH) were employed at high concentrations and it was presume that, their concentrations not change during the reaction. Therefore, the reaction rate (equation 7) can be simplified by using the Ostwald approximation method [33-35] and is shown as follow:

\[
R = K(CH_3COO^-)^\beta (Pb^{2+})^\alpha \quad (8)
\]

If it is assumed that \( K(CH_3COO^-)^\beta \equiv K^* \), the reaction rate is defined as:

\[
R = K'(Pb^{2+})^\alpha \quad (9)
\]

The reaction rate is simplified to pseudo first-order. By using the assumption that the \( \alpha \) (reaction order) value is equal to 1, the rate expression of reaction is simplified to:

\[
-ln(Pb^{2+}) = kt \quad (10)
\]

In the pseudo first-order’s approach, the reaction rate constant \( k' \), where \(-\ln(Pb^{2+})\) is the lead ion concentration during the reaction time \( t \). A straight line through the data points was obtained by linear regression. Consequently the rate constant \( k' \) was obtained from the slope and the validity of estimation \( (\alpha = 1) \) was checked on the data of linear regression \( (r) \).

However, Figure 1 and Table 5 show the experimental values of \( (Pb^{2+}) \) and \(-\ln(Pb^{2+})\) at different time of the reaction. The values of \(-\ln(Pb^{2+})\) were plotted versus the values of time \( t \). A straight line through the data point was achieved by linear regression. The current graph obeys the least- squares line:

\[
y = -0.0065t + 4.53 \quad (11)
\]

The number of data points, \( n \), was equal to 14, time, \( t \), ranged from 30 to 420 seconds. The rate constant \( k' \) was achieved from the slope of graph, \( 6.5 \times 10^{-3} \text{ (S)}^{-1} \). From the linear regression, it was found that the value of \( r = 0.998 \) and hence, the assumption \( \alpha = 1 \) is acceptable.

**Lead Cleaning from Real Sample:** As mentioned in the experimental section, to evaluate the efficiency of the suggested chemometric process for cleaning of a dirty surface a chromium plate with about 200×10 mm2 dimension that is covered with a thin layer (nearly 3 g) of metallic lead was treated with the cleaner solution at the optimum conditions (10% of oxidizing agent (H2O2), 75 rpm stirring speed of the mixture and 28°C temperature). In order to lead cleaning, the dirty sample was treated according to the experimental procedure (section 2.2) and the chemical removal of lead fouling was initiated and after about 8 minutes, reducing rate of realizing gas bubbles revealed the ending of the cleaning process. Moreover, to avoid biological pollution [36], the lead acetate generated during the procedure was removed from the waste cleaner solution by Nonliving Green Algae Cladophora Fascicularis as a biological method [37]. However, our primary evolutions has been shown that the procedure has proper efficiency for cleaning of a dirty surfaces, Nevertheless for more evaluation of the method all surfaces were examined by the SEM and EDAX analysis.
Fig. 1: The experimental result of oxidation of lead bar measured by ICP spectroscopy. (a) values of \([\text{Pb}^{2+}]\) (mol/L) as a function of time, (b) values of \(-\ln [\text{Pb}^{2+}]\) as a function of time.

Fig. 2: SEM images of (a-b) cross-section of real sample (stainless-steel plate coated with a 20 µm layer of chromium fouling with about 3g of metallic lead) before treatment with cleaner solution, (c) cross-section of the sample after cleaning at optimum condition and (d) surface of the sample after cleaning at optimum condition.

**Results of SEM/EDAX Analysis:** SEM photographs of different samples comprising, impure and cleaned chromium surfaces were illustrated in Figure 2. The photographic results demonstrated that on the dirty samples (Figure 2a-b) three layers containing lead fouling, chromium electroplated and stainless-steel body can be clearly observed. Also, (Figure 2c-d) presents that in the treated surfaces of samples, stainless-steel plate has only the chromium layer without any corrosion and no lead fouling streak was observed on the surface of the plate.
Table 5: The experimental values of $-\ln [\text{Pb}^{2+}]$ as a function of time, measured by ICP spectroscopy.

<table>
<thead>
<tr>
<th>No</th>
<th>Time (S)</th>
<th>$[\text{Pb}^{2+}]$ (mol/L)</th>
<th>$-\ln [\text{Pb}^{2+}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30</td>
<td>0.0013</td>
<td>6.64</td>
</tr>
<tr>
<td>2</td>
<td>60</td>
<td>0.0018</td>
<td>6.30</td>
</tr>
<tr>
<td>3</td>
<td>90</td>
<td>0.0022</td>
<td>6.14</td>
</tr>
<tr>
<td>4</td>
<td>120</td>
<td>0.0026</td>
<td>5.96</td>
</tr>
<tr>
<td>5</td>
<td>150</td>
<td>0.0030</td>
<td>5.80</td>
</tr>
<tr>
<td>6</td>
<td>180</td>
<td>0.0036</td>
<td>5.60</td>
</tr>
<tr>
<td>7</td>
<td>210</td>
<td>0.0043</td>
<td>5.45</td>
</tr>
<tr>
<td>8</td>
<td>240</td>
<td>0.0050</td>
<td>5.30</td>
</tr>
<tr>
<td>9</td>
<td>270</td>
<td>0.0060</td>
<td>5.10</td>
</tr>
<tr>
<td>10</td>
<td>300</td>
<td>0.0072</td>
<td>4.90</td>
</tr>
<tr>
<td>11</td>
<td>330</td>
<td>0.0085</td>
<td>4.80</td>
</tr>
<tr>
<td>12</td>
<td>360</td>
<td>0.0105</td>
<td>4.60</td>
</tr>
<tr>
<td>13</td>
<td>390</td>
<td>0.0120</td>
<td>4.40</td>
</tr>
<tr>
<td>14</td>
<td>420</td>
<td>0.0140</td>
<td>4.30</td>
</tr>
</tbody>
</table>

Comparison with Sequential Experimental Method:
Recent chemometric cleaning procedure has been compared with our previous sequential experimental method for removal of fouling lead layer from chromium surfaces in terms of accuracy, rapidity, cost and performance. The present statistical cleaning method is clearly faster than its sequential partner. For example, the experimental results of our pervious sequential experimental method, showed that at the optimum condition (acetic acid concentration 28% (V/V), hydrogen peroxide 8% (V/V) and temperature 35°C), 15-minutes time is needed for complete removal of 3g fouling lead layer from chromium surface [13]. However, the statistical results showed that chromium surfaces can optimally be cleaned merely by controlling of hydrogen peroxide concentration, stirring speed of the mixture and temperature of the cleaner solution and percent of chelating agent (CH$_3$COOH) is not a significant parameter during the removal process. Also, the statistical results showed that under stirring speed of 75 rpm, the most cleaned sample was achieved by using cleaning solution with 10% (V/V) of hydrogen peroxide as oxidizing agent and temperature of 28°C within only 8 minutes. Comparison of the results confirms that in the moderate experimental condition, lower percent of chelating agent (10 respect to 28%), lower cleaning solution temperature (28 respect to 35°C) and higher stirring speed of cleaner solution (75 respect to 50 rpm), the efficiency of the proposed statistical chemometric method is higher and the total removal time of fouling lead layer is considerably lower (8 minutes respect to 15 minutes) than our previous sequential experimental method.

CONCLUSION

In this study, we have investigated a simple, fast and controllable method for cleaning of deposited metallic lead layer by using a redox reaction system. The variables were examined in order to increase the efficiency of the proposed method as potential industrial application. The OAD evaluation of experimental data provides optimized amount of the parameters, H$_2$O$_2$ 10% (V/V), stirring speed of 75 rpm and temperature of 28°C, to obtain the most
cleaned sample, at which the total removal time of 3 g of
the fouling lead layer is about 8 minutes. The experiments
proved that by using this method, chromium surface
could be completely cleaned at optimum conditions.
SEM/EDAX photographs of completely cleaned samples
showed that the treated surfaces were uniform and
without any surface corrosion. Also by using ICP
spectroscopy experimental data, the kinetic parameters of
redox reactions of lead bar dissolving were obtained by
Ostwald approximation method. The results showed that
the rate constant \( k^p \) was obtained by least-squares line
is equal to \( 6.5\times10^{-3} \text{ (S)}^{-1} \), that confirmed the pseudo first-order reaction approximation. Finally; the results of this
investigation confirmed that the simultaneous proposed
chemometric method is considerably rapid, accurate and
simple than our pervious sequential experimental
methods.

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