High-Temperature Cyanide Leaching of Platinum-Group Metals From Automobile Catalysts—Laboratory Tests

By D. P. Desmond, G. B. Atkinson, R. J. Kuczynski, and L. A. Walters
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<tr>
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<tr>
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<td>mL</td>
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<tr>
<td>mm</td>
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HIGH-TEMPERATURE CYANIDE LEACHING OF PLATINUM-GROUP METALS FROM AUTOMOBILE CATALYSTS—LABORATORY TESTS

By D. P. Desmond, G. B. Atkinson, R. J. Kuczynski, and L. A. Walters

ABSTRACT

The U.S. Bureau of Mines investigated leaching automobile catalysts with sodium cyanide (NaCN) solutions at high temperatures to recover platinum-group metals (PGM). The feed was virgin monolith rejects, used monolith, and used pellet catalysts. Leaching with a 5-pct NaCN solution for 1 h at 160°C dissolved over 97 pct of the PGM in the virgin monolith, over 85 pct of the PGM in the used monolith, and over 90 pct of the PGM in the used pellet catalyst. Over 99.8 pct of the dissolved PGM was recovered as a precipitate by heating the solution to 250°C for 1 h in an autoclave. The cyanide complexes were decomposed, and free cyanide was destroyed.

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INTRODUCTION

The primary use of Pt and Rh in North America is in the production of automobile catalysts. In 1989, the amount of platinum-group metals (PGM) used in automobile catalysts, as a percentage of total Western World demand for each metal, was 37 pct for Pt, 6 pct for Pd, and 79 pct for Rh (approximately 1.3 million tr oz of Pt, 200,000 tr oz of Pd, and 260,000 tr oz of Rh) (1). Recovery of PGM from scrap catalysts has been expanding steadily over the past years because of high PGM prices and the increasing number of States requiring scrap yards to remove converters from cars before they are crushed or shredded.

A large percentage of the catalytic converters collected in the United States are decanned, and the catalyst supports are shipped to Japan and Europe for processing. Improved technology for recovering PGM from catalytic converters would benefit the United States by encouraging domestic recycling of critical and strategic PGM.

Both pyrometallurgical and hydrometallurgical processes are currently used to recover PGM from automobile catalysts. Hoffman (2) published a review of current technology for recovering PGM from automobile catalysts. This review describes six processes: substrate dissolution for pellet processing, insoluble substrate for monolith processing, dry chlorination, plasma fusion, Cu collection, and charging crushed catalysts to Cu smelters. Excluding the indirect method of PGM recovery by charging to Cu smelters, estimated recoveries for the other processes ranged from 80 to 96 pct for Pt, from 80 to 96 pct for Pd, and from 65 to 90 pct for Rh. Problems exist with each method. The acid systems are complex and corrosive, and generate large volumes of liquid waste. The melting systems have high electrical energy consumption and major lead emission control problems, and produce a low-grade alloy concentrate. Higher recoveries would be beneficial.

As part of its mission to help ensure a domestic supply of strategic and critical minerals, the U.S. Bureau of Mines is investigating the use of cyanide solutions to dissolve PGM for recovery. Using sodium cyanide (NaCN) offers potential advantages over both acid and melting systems, such as low corrosion, decreased energy requirements, less complicated unit operations, and selectivity for PGM. Cyanide solutions were tested, with limited success, in treating oxidized PGM ores from the Republic of South Africa (3). Previous Bureau research (4) investigated leaching virgin monolith catalysts with cyanide solutions to establish baseline leaching parameters. In that research, two types of monolith were tested: an oxidation type containing Pt and Pd, and a three-way type containing Pt and Rh. The monolith biscuits were crushed to minus 12 plus 30 mesh. To obtain consistently high PGM recovery, it was necessary to have the PGM in elemental form before leaching; therefore, the catalyst samples were roasted in air for 1 h at 550°C, then reduced in flowing H2 for 1 h at 550°C. Rate experiments were conducted to evaluate the effect of NaCN concentrations, promoter concentrations, leaching temperatures, solution pHs, and catalyst pretreatment methods. The best conditions determined for leaching virgin catalysts were 5 pct NaCN, 1 pct promoter, natural pH of 11.8, and catalyst pretreatment by oxidation and reduction. The highest leaching temperature investigated was 80°C. Under these conditions, more than 95 pct of the PGM were extracted. The best conditions determined for processing virgin catalysts were used to treat used catalysts. Less than 75 pct of the PGM were extracted from used catalysts at 80°C.

This report describes further research with cyanide leaching conducted at higher temperatures. Initial tests at higher temperatures showed that adding the promoter had no benefit; therefore, the promoter was removed from the system. Although higher temperatures increased cyanide consumption, the added cost of the reagent may be offset by improved extraction of the valuable PGM. Several methods were tested for recovering PGM from the cyanide leaching solution, including cementation, C adsorption, ion resins, electrowinning, and thermal decomposition of the cyanide complexes.

SAMPLE DESCRIPTION

Three types of automobile catalyst were tested: virgin monolith, used monolith, and used pellet. The virgin monolith was a reject from a manufacturer’s production line and was a mixture of three-way and oxidation-type monolith catalysts. Used catalysts were obtained from a commercial decanner and consisted of grab samples from several lots to obtain a representative sample. Oxidation catalysts are made with Pt and Pd; three-way catalysts contain Rh in addition to Pt or Pd. There is no standard catalyst formulation, and both types of catalyst can contain other metals and compounds. Metals are added to promote the catalytic reactions, and compounds are added...
to stabilize the gamma-alumina wash coat. Table 1 shows
the amount of PGM in the virgin and used catalyst
samples.

<table>
<thead>
<tr>
<th>Catalyst type</th>
<th>Pt</th>
<th>Pd</th>
<th>Rh</th>
</tr>
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<tbody>
<tr>
<td>Virgin monolith</td>
<td>55.0</td>
<td>8.5</td>
<td>3.0</td>
</tr>
<tr>
<td>Used monolith</td>
<td>24.0</td>
<td>8.4</td>
<td>1.5</td>
</tr>
<tr>
<td>Used pellet</td>
<td>8.9</td>
<td>3.2</td>
<td>.1</td>
</tr>
</tbody>
</table>

Monolith catalysts consist of a honeycomb structure of
cordierite coated with gamma-alumina and impregnated
with PGM. The cordierite base provides support, while
the gamma-alumina provides the large surface area
necessary for good catalyst operation. Pellet catalysts were
approximately 3-mm-diam by 4-mm-long cylinders of
gamma-alumina impregnated with PGM. The monolith
catalysts were crushed in stages to minus 12 mesh.
Crushing eliminated the large pores in the monolith
structure, which trap air bubbles during leaching and
solution during washing. Pellet catalysts were not crushed.

During its useful life, an automobile catalyst undergoes
many chemical and physical changes caused by impurities
and extreme temperature cycles. The major contaminants
are Pb from tetraethyl Pb used as a gasoline additive, and
C from inefficient engine operation. The used monolith
sample contained 0.85 pct Pb and 0.63 pct C, and the used
pellet sample contained 1.1 pct Pb and 1.1 pct C. Sulfur
contamination was also present in the used catalysts, pri-
marily as sulfates, and ranged between 0.03 and 0.15 pct S.
Some used catalyst samples tested had been operated at
temperatures high enough to melt the ceramic support.
Reaction of PGM with impurities, such as Pb and sulfate,
could form compounds that are refractory to cyanide
solutions. High-temperature excursions, as evidenced by
melted ceramic, caused sintering, which decreased the
surface area and masked the PGM. Sintering would
prevent the cyanide solution from dissolving PGM by
limiting solution to solid contact. All of these problems
would decrease leaching of PGM from used catalysts
compared with virgin catalysts.

**EQUIPMENT AND PROCEDURE**

The primary reagent used to dissolve PGM was
reagent-grade NaCN. The use of cyanide requires that
trained personnel be employed and safety precautions be
observed. The pH of solutions must be maintained above
11 to retard formation of poisonous hydrocyanic gas
(HCN). Good hygiene must be practiced to prevent
contact of cyanide with the skin (5). Leaching tests were
conducted in a 250-mL autoclave lined with Teflon™
fluorocarbon polymer. Twenty-gram samples of catalyst
were leached with 100 mL of solution both with and
without agitation for 1 h at different temperatures. Solids
and liquids were separated after leaching by vacuum
filtration with a Buchner funnel. The residue was dis-
placement washed with 400 mL of water.

PGM extraction was calculated using analysis of the
PGM content of the leaching solution and leach residue.
The calculated head was always within 10 pct and usually
within 5 pct of an independent head analysis. The dis-
crepancy in the mass balance is due to the sample's
inhomogeneity and difficulties in analyzing the low PGM
concentrations. Solutions were analyzed with a sequential
inductively coupled plasma (ICP) spectrometer. Amounts
of PGM in solid samples were determined by fire assaying
the solids, dissolving the doré bead, and analyzing the
solution by sequential ICP (6).

Cementation, C adsorption, and ion resin were tested
for ability to recover PGM by placing 100 mL of pregnant
leaching solution in a beaker with excess recovery reagent
and stirring. Vacuum filtration with a Buchner funnel was
used to separate the solids and liquids. Solution analyses
were used to calculate recoveries. Tests to evaluate
recovery of PGM by electrowinning were conducted in a
100-mL cell containing a porous graphite cathode and a
stainless steel anode. High agitation was achieved by
pumping. The solution was periodically sampled, and
comparison with the feed solution was used to calculate
the recovery. Recovery by thermal decomposition was
tested by placing pregnant solution in an autoclave and
heating to temperatures between 200° and 275° C with no
agitation. The residue was collected on filter paper but
was present in too small a quantity to analyze accurately.
Solution analyses for PGM both before and after thermal
treatment were used to calculate recoveries.

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Reference to specific products does not imply endorsement by the
U.S. Bureau of Mines.
CATALYST LEACHING

The effect of leaching automobile catalysts with a NaCN solution at temperatures greater than 80° C was tested. Twenty-gram samples of virgin monolith were leached at temperatures between 100° and 200° C with 100 mL of a 5-pct NaCN solution for 1 h. The 5-pct NaCN solution supplied 5 g of NaCN, which ensured cyanide in excess of the less than 0.05 g of NaCN required to complex the PGM. Experiments conducted at 80° C had shown no further leaching after 1 h (4). Therefore, 1-h retention at temperature was considered acceptable to ensure maximum leaching. Tests were conducted with and without agitation. After the first series of tests, no change in results was noticed with or without agitation. Therefore, agitation was discontinued and all reported results are for tests without agitation. Tests required approximately 2 h to reach temperature and 2 h to cool sufficiently for filtering.

Figure 1 shows the results from individual tests on leaching virgin monolith at temperatures between 100° and 200° C. Best leaching results were obtained at 160° C; at this temperature over 97 pct Pt and Pd and 90 pct Rh were dissolved from virgin catalyst. Above 160° C the amount of Pd in solution quickly decreased and only 8 pct was solubilized at 175° C. Extraction of Pt and Rh decreased more slowly than Pd above 160° C, indicating that the Pt- and Rh-cyanide complexes are more stable than the Pd-cyanide complexes at higher temperatures. The concentration of free cyanide decreased from 26,500 to 50 ppm after 1 h at 160° C because of thermal hydrolysis of free cyanide at high temperatures.

The relationship between metal-cyanide complexes, free cyanide, and temperature can be explained using Pd as an example. As initially prepared, the leach solution contained a significant excess of cyanide, but the rate of soluble Pd-cyanide complex formation was slow. As the temperature was increased, the rate of Pd-cyanide formation increased but free cyanide was being destroyed. At 160° C, the rate of Pd-cyanide formation was maximized versus cyanide destruction. Above 160° C, in addition to free-cyanide destruction, the rate of Pd-cyanide complex destruction is significant. This resulted in loss of Pd from the solution and destruction of the liberated cyanide. These factors combined to create a balance between Pd-cyanide in the solution and thermal destruction of the Pd-cyanide complex and free cyanide after 1 h at about 160° C.

Figure 2 shows results of tests of 20-g samples of used monolith leached with 100 mL of 5-pct NaCN solution for 1 h at temperatures between 100° and 200° C. The curves show behavior similar to that shown in leaching virgin monolith, but the amount of PGM leached was less. Best results were obtained by leaching at 160° C; recoveries of Pt, Pd, and Rh were 88, 80, and 75 pct, respectively.

Based on the results from leaching monolith catalysts, 20 g...
of used pellets was leached for 1 h at 160°C with 100 mL of 5-pct NaCN solution. Tests consistently produced leaching extractions for Pt, Pd, and Rh of 95, 95, and 90 pct, respectively.

Tests were conducted to determine the effect of NaCN concentration on leaching virgin and used monolith catalysts. Twenty-gram samples of catalyst were leached for 1 h at 160°C with solutions having NaCN concentrations between 0.5 and 5 pct. Figure 3 shows the percentage of PGM leached from virgin monolith, and figure 4 shows the percentage leached from used monolith at different NaCN concentrations. These results indicate that comparable results are obtained from leaching virgin and used monolith with solutions having NaCN concentrations between 1 and 5 pct. NaCN concentrations below 1 pct tended to decrease the percentage of PGM in solution. Selection of NaOH concentration would be a tradeoff between reagent cost and product extraction.

**CATALYST PRETREATMENT**

Several pretreatment methods were tested to enhance leaching of PGM from used catalysts. Carbon contamination was considered a potential problem because C might adsorb cyanide complexes. This would be similar to problems with premature Au removal from solution encountered with C in Au ores in which Au extractions are decreased during cyanidation by adsorption of cyanide complexes. Carbon was removed from used catalysts by heating at 550°C in air for 1 h. Analyses showed that C concentration generally decreased to less than 0.1 pct. Cyanide leaching of samples both with and without heat treatment in air gave similar extractions of PGM. This showed that C contamination was not responsible for decreases in PGM extraction.

Results from leaching tests conducted on virgin catalysts at 80°C showed that extractions were enhanced if the PGM were in elemental form. Used catalyst samples were roasted in air at 550°C for 1 h, followed by treatment in H₂ atmosphere at 550°C for 1 h. This pretreatment did not enhance the extraction of PGM from used catalysts during high-temperature leaches.

Chemical and physical changes during the catalyst life, such as formation of chemical compounds and sintering of the metals and gamma-alumina wash coat, cause masking of the PGM. To expose more PGM to the NaCN leaching solutions, samples of used catalyst were pretreated by leaching with 1.25N NaOH solution at 80°C for 1 h to decrease Pb contamination and dissolve a portion of the
gamma-alumina wash coat. The NaOH solution did not dissolve PGM. Approximately 30 pct of the Pb and less than 2 pct of the alumina dissolved. Twenty-gram samples of used monolith treated with NaOH were leached in an autoclave with 100 mL of a 5-pct NaCN solution at 160° C for 1 h. Values obtained by averaging the results of five tests conducted under the same NaOH pretreatment and NaCN leaching conditions showed the amounts of PGM leached were 89 pct Pt, 82 pct Pd, and 80 pct Rh. These results are slightly better than the 88, 80, and 75 pct Pt, Pd, and Rh, respectively, leached from untreated used monolith. Pretreatment with 2.5N NaOH was also tested. No improvement was observed in extraction of PGM compared with pretreatment at 1.25N, but more alumina was dissolved, which could cause processing problems.

A lower solution-to-solid ratio would help decrease cyanide requirements. Because stoichiometric requirements accounted for less than 5 pct of the cyanide, most cyanide that did not form stable metal-cyanide complexes was destroyed during processing at 160° C. Figure 3 shows the importance of having a NaCN concentration above 1 pct. Although the 0.5-pct NaCN solution was strong enough to provide excess stoichiometric cyanide requirement, it appears the cyanide was not available at high temperatures. By decreasing the solution-to-solid ratio, a strong cyanide solution can be provided while decreasing the cyanide consumption per pound of catalyst processed. Table 2 shows the results from leaching used monolith catalyst both with and without NaOH pretreatment with a 1-pct NaCN solution at 160° C for 1 h at different solution-to-solid ratios. The best results were obtained with a 2:1 solution-to-solid ratio on a sample pretreated with 1.25N NaOH. The results from this experiment compare favorably with those obtained for a catalyst pretreated with 1.25N NaOH and leached with a 5:1 solution-to-solid ratio. By decreasing the NaCN concentration from 5 to 1 pct and decreasing the solution-to-solid ratio from 5:1 to 2:1, the amount of NaCN required for each autoclave leach decreases from 500 lb/st of catalyst to 40 lb/st of catalyst. At the 1990 price of $1/lb for NaCN, this represents a savings of $460/st of used catalyst treated for each autoclave leach. Decreasing NaCN concentration from 5 to 1 pct had no noticeable effect on leaching used catalyst.

Table 2.—Results from leaching used monolith catalyst with 1-pct NaCN solution at 160° C for 1 h

<table>
<thead>
<tr>
<th>Catalyst pretreatment</th>
<th>Solution-to-solid ratio</th>
<th>Pct leached</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Pt</td>
</tr>
<tr>
<td>None</td>
<td>5:1</td>
<td>88</td>
</tr>
<tr>
<td>1.25N NaOH</td>
<td>5:1</td>
<td>89</td>
</tr>
<tr>
<td>None</td>
<td>2:1</td>
<td>84</td>
</tr>
<tr>
<td>1.25N NaOH</td>
<td>2:1</td>
<td>88</td>
</tr>
<tr>
<td>None</td>
<td>1:1</td>
<td>85</td>
</tr>
<tr>
<td>1.25N NaOH</td>
<td>1:1</td>
<td>79</td>
</tr>
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</table>

PLATINUM-GROUP METALS RECOVERY

Methods were tested to recover the PGM from the pregnant leach solutions. When evaluating PGM recovery methods, the concentration of PGM in pregnant leaching solution must be considered. A typical pregnant leaching solution might contain 500 ppm of PGM with a potential value of $10/L. Economic considerations make it desirable that recovery of a PGM concentrate be fast and complete. Methods tested included cementation, carbon adsorption, ion resins, electrowinning, and thermal decomposition of the cyanide complexes.

Cementation tests were conducted with powdered Zn (Merrilite) or Al. One-hundred-milliliter samples of catalyst leach solution containing 550 ppm Pt, 100 ppm Rh, and 50 ppm of free cyanide were treated with five Al or Zn additions of 1, 1, 2, 4, and 8 g each, made at 1-h intervals. Solution samples were taken after 1 h and before each addition. Tests were conducted at 30°, 45°, and 60° C. The best results obtained were 50-pct removal of Pt and 10-pct removal of Rh with Zn powder at 60° C. Al powder was not effective for removing Pt or Rh. Because of very poor removal of PGM, no further tests with cementation were conducted.

Loading PGM on activated C was evaluated as a possible recovery method because of its wide use in the gold industry. Samples of leaching solution were contacted with activated C for 1 h in a stirred beaker using 10 times the recommended amount of C. More than 99 pct of the Pt and Pd was removed from solution, but less than 15 pct of the Rh was removed for all types of C tested. Contacting activated C with solutions containing high concentrations of PGM resulted in maximum loadings of 70 tr oz PGM per short ton of C. Testing of activated C was discontinued because of the incomplete removal of Rh and the relatively low loading of PGM.

Ion exchange and ion reduction resins were screened for ability to recover PGM. Each type of resin was added in stages to 100 mL of pregnant leaching solution; the final addition resulted in an excess of at least 30 times the theoretical requirement. Solution analyses showed that after 24 h of contact, the best reduction resin removed 95 pct of the Pt and 50 pct of the Rh. The best ion exchange resin gave better results: it removed 99 pct of
the Pt and 80 pct of the Rh. Resins rapidly removed PGM from solution, but incomplete removal was a drawback and resin testing was terminated.

Electrolysis was tested for ability to recover PGM directly from leaching solutions, since the concentrations of PGM in solution are relatively high. Electrolysis removed PGM from solution, but deposition rates steadily decreased as the concentration of PGM decreased, and many metal impurities were codeposited. After several days of electrolysis, current efficiency of less than 1 pct was calculated and only 50 pct of the PGM was removed. Because of the long time requirement and low amount of PGM removed, electrolysis was not selected as the recovery method.

Thermal decomposition of cyanide complexes was also tested as a recovery method. Figures 1 and 2 show that, at higher temperatures, the amount of metal in solution decreased from a maximum at 160° C, suggesting that the metal-cyanide complexes formed at lower temperatures were unstable at higher temperatures and decomposed to precipitate the metal from solution. Tests were conducted in which the pregnant leaching solution was reheated in a pressure vessel to even higher temperatures to remove PGM from solution. Table 3 shows the results of tests in which pregnant leaching solutions from virgin catalysts were heated to temperatures above 200° C for 1 h. More than 99.8 pct of the PGM was removed by heating the solution to 250° C or higher for 1 h. Additional tests showed that Pt was the most difficult of the PGM to remove from solution, indicating that Pt-cyanide is more thermally stable than Pd-cyanide and Rh-cyanide. Another important benefit of thermal decomposition for PGM recovery is the almost complete destruction of total cyanide during thermal treatment. Cyanide determinations on solutions after all PGM were removed to less than 1 ppm showed that total cyanide concentrations were less than 0.2 ppm. Although the solution is safe for disposal from a cyanide and heavy-metal standpoint, the total amount of dissolved solids, primarily Na₂CO₃, and pH are too high for recycling or dumping into the sewer. The precipitated product was a fine black powder containing mostly metallic PGM. PGM with a potential value of $15,000 can be recovered from each ton of catalyst using less than $100 in reagents, primarily NaCN.

<table>
<thead>
<tr>
<th>Temp, °C</th>
<th>Reactor pressure, psig</th>
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<tr>
<td>Head</td>
<td>NAp</td>
<td>Pt</td>
</tr>
<tr>
<td>200</td>
<td>210</td>
<td>146</td>
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<td>600</td>
<td>.2</td>
</tr>
<tr>
<td>275</td>
<td>900</td>
<td>.4</td>
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</table>

NAp Not applicable.

**SUMMARY AND CONCLUSIONS**

Extraction of PGM from automobile catalysts by leaching with NaCN solutions at high temperatures in an autoclave was tested. Leaching virgin auto catalyst with a 5-pct NaCN solution at 160° C for 1 h at a solution-to-solid ratio of 5:1 removed over 97 pct of the PGM. Leaching used catalyst under the same conditions dissolved 85 pct of the PGM from the monolith-type and over 90 pct of the PGM from the pellet-type catalyst. No negative effect was found from leaching without agitation. The decrease in leachability shown between virgin and used monolith catalyst was due to contamination by exhaust byproducts and physical alteration of the catalyst substrate impeding good solution-to-solid contact. Preleaching used monolith with 1.25N NaOH produced an average 3-pct improvement in overall leaching results. However, a ±5-pct mass balance accuracy makes determination of absolute effectiveness difficult. Experiments showed that decreasing NaCN concentration to 1 pct and solution-to-solid ratio to 2:1 produced similar recoveries and decreased NaCN requirements for each autoclave leach from 500 to 40 lb per ton of catalyst.

The best method tested for recovering PGM from the leaching solution was thermal decomposition of all cyanide complexes. Over 99.8 pct of the PGM contained in the solutions was recovered as a precipitate by treating the solutions at 250° C for 1 h while decreasing the total cyanide concentration to less than 0.2 ppm.
REFERENCES