Kinetics of the reaction between Pb-Sb complex sulphide concentrates and water vapour

by F. Zhu*, Y. Hua†, and Y. Meng‡

Synopsis
The kinetics on the separation of antimony from lead by oxidative volatilization roasting of Pb-Sb complex sulphide concentrates with water vapour is investigated in this paper. The experimental results show that the reaction rate of the separation process is controlled by chemical reaction and internal diffusion simultaneously when the temperature <1073 K and by internal diffusion when the temperature >1073 K. The apparent activation energies were calculated to be 85.92 kJ/mol for the chemical reaction and 10.27 kJ/mol for the internal diffusion, respectively. X-ray diffraction (XRD) analysis of the products demonstrates that the reaction of the Pb-Sb complex sulphide concentrates with water vapour can promote the Pb-Sb separation, because steam can decompose the Pb-Sb complex sulphide concentrates and the complex intermediate products into simple sulphides of lead, antimony and iron. In contrast, in nitrogen atmosphere, Pb-Sb complex sulphide concentrates can be decomposed into simple sulphides to a limited extent. The remaining Pb-Sb complex sulphide concentrates hinders volatilization of Sb₂S₃. In air atmosphere, the Pb-Sb complex sulphide concentrates is transformed into stable Pb₄Sb₃FeO₁₃, making volatilization of antimony more difficult. The condensed product consists of Sb₂O₃ with a small quantity of PbS and S by XRD analysis. This result indicates that only in steam atmosphere Sb₂S₃ can be selectively oxidized to Sb₂O₃ while PbS cannot be oxidized. As a result, the Pb-Sb separation will be promoted.

Keywords: Kinetics, reaction, Pb-Sb complex sulphide concentrates, water vapour

Introduction
Pb-Sb complex sulphide concentrates (major phase is jamesonite (FePb₄Sb₆S₁₄) and PbS, ZnS, SiO₂ and FeS are minor phases), mainly distributed in the Guangxi Zhuang autonomous region, are important resources of lead and antimony in China. Presently they are treated by pyrometallurgical process, as shown in Figure 1. Although it has some advantages, its major disadvantages are inferior Pb-Sb separation, low direct recoveries of Pb (68%) and Sb (40%), and meaningless recycling of Pb and Sb in the smelting process. The existing pyrometallurgical flowsheet usually produces low-quality antimony or even lead-antimony alloy. The Pb-Sb separation is a key problem in treating this kind of Pb-Sb complex sulphide concentrates. Moreover, the problem with this process is that the sulphur dioxide generated during fluid bed roasting can not be efficiently recovered due to its low concentration. In the present investigation, the lead-antimony separation technology for the Pb-Sb complex sulphide concentrates by oxidative volatilization roasting with water vapour is proved to be highly effective. The direct recoveries of Pb and Sb are >94% and >93%, respectively. The emission of sulphur-bearing gases during the volatilization roasting process of the Pb-Sb complex sulphide concentrates will be considerably reduced. However, the kinetics and mechanism of oxidation of the Pb-Sb complex sulphide concentrates with water vapour have not been studied. It is worthwhile to study the kinetics and mechanism of oxidation of the Pb-Sb complex sulphide concentrates with water vapour.

Experimental
The schematic diagram of experimental apparatus is illustrated in Figure 2. The Pb-Sb complex sulphide concentrates are obtained from the Guangxi Huaxi Group Corporation Ltd. (Guangxi, China). The chemical composition of Pb-Sb complex sulphide concentrates is given in Table I, which indicates that the major components of the concentrates are Pb, Sb, S, Fe, Zn and SiO₂, app. 85%. It is indicated that the major phase observed in the concentrates is jamesonite (Pb₄Sb₆FeS₁₄), and that PbS,
Kinetics of the reaction between Pb-Sb complex sulphide concentrates and water

ZnS, SiO₂ and FeS as minor phases are also observed by XRD analysis of the concentrate, as shown in Figure 3.

Experiments were carried out in a typical tubular furnace, where the corundum crucible containing the sample (10 g) was placed in the centre of a tube reactor (6.8 cm i.d. and 100 cm long). The even temperature zone is 10 cm long. In experiments the apparatus was sealed, flushed with high purity argon (>99.99%) for 30 min. to eliminate the oxygen in the reaction system and then heated in an argon atmosphere. Temperature was measured by a Pt-Rd thermo-couple (error ±1°C). After reaching the desired temperatures, the crucible was quickly put into the even temperature zone from the left of the furnace and water vapour was introduced into the furnace tube from the right. The gaseous reaction products were carried away from the hot zone by water vapour and then condensed on the wall of the furnace tube. After completion of the reaction, the reactor was pulled out from the hot zone to the cold zone and cooled under the flow of high purity nitrogen (>99.99%). The weight of the roast thus obtained was determined by electronic balance.

The flow rate of water vapour was controlled by a steam rotor flow meter. The sulphur-bearing gas produced during reaction process was determined by standard iodine solution. The volatile species and roast were collected for XRD analysis.

Results and discussion

Theory of kinetics

The reaction between the Pb-Sb complex sulphide concentrates and water vapour is represented by the following:

\[
\begin{align*}
\text{FePb}_2\text{Sb}_6\text{S}_1\text{s}(s) + 9\text{H}_2\text{O}(g) &= 3\text{Sb}_2\text{O}_4(g) + \\
4\text{PbS}(s) + \text{FeS}(s) + 9\text{H}_2\text{S}(g)
\end{align*}
\]

According to the law of additive reaction times⁷⁻¹⁴, when the process was controlled by chemical reaction, internal diffusion and the external diffusion simultaneously, the kinetics for the gas-solid reaction can be expressed as:

\[
t = \frac{d_b}{b}\left(\frac{F_P V_P}{A_P}\right)\left(\frac{F_P V_P}{A_P}\right)\left(\frac{F_P V_P}{A_P}\right) + \\
+ \frac{d_b}{6bD_c}\left(\frac{F_P V_P}{A_P}\right)^2 + \\
+ \frac{d_b}{b}\left(\frac{F_P V_P}{A_P}\right)^X
\]

Table I

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Pb</th>
<th>Sb</th>
<th>S</th>
<th>Fe</th>
<th>Zn</th>
<th>Cu</th>
<th>As</th>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
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<tbody>
<tr>
<td>Wt Pct</td>
<td>23.73</td>
<td>19.70</td>
<td>23.21</td>
<td>11.00</td>
<td>5.85</td>
<td>0.30</td>
<td>1.20</td>
<td>1.30</td>
<td>2.50</td>
<td>1.00</td>
</tr>
</tbody>
</table>

Figure 1—Pyrometallurgical flow chart for Pb-Sb complex sulphide concentrates

Figure 2—Schematic diagram of experimental apparatus
where $X$ is the fractional conversion of the solid reactant. $X$ can be expressed as follows:

$$X = 1 - \frac{Sb\% W}{Sb_0\% W_0}$$  \[7\]

Where $Sb\%$ is the Sb content of the roast (%); $Sb_0\%$ is the Sb content of the Pb-Sb complex sulphide ores (%); $W$ is the weight of the roast (g); $W_0$ is the weight of the Pb-Sb complex sulphide ores (g); $t$ is time (min); $\sigma$ is the generalized gas-solid reaction modulus; $Sh^*$ is the modified Sherwood number; $A_P$ is the external surface area of the individual pellet (cm$^2$); $F_P$ is the shape factor for the pellet; $V_P$ is the volume of the individual pellet (cm$^3$); $D_e$ is the effective diffusion coefficient of gaseous H$_2$O through the product layer (cm$^2$.min$^{-1}$); $k_g$ is the gas phase mass transfer coefficient (cm.min$^{-1}$); $b$ is the stoichiometric coefficient; $k$ is the chemical reaction rate constant (cm.min$^{-1}$); $C_{H_2O,g}$ is the concentration of H$_2$O in the gas phase (mol.cm$^{-3}$); $M_g$ is the molecular weight of the solid reactant; $\rho_b$ is the density of the solid reactant (g.cm$^{-3}$); $R_0$ is the initial radius of the grain (cm).

Let

$$f_1 = \frac{1}{k_1} = \frac{d_B}{b k C_{H_2O}} (\frac{F_P V_P}{A_P}) (\text{min})$$  \[8\]

$$f_2 = \frac{1}{k_2} = \frac{d_B}{6 b D_e C_{H_2O}} (\frac{F_P V_P}{A_P})^2 (\text{min})$$  \[9\]

$$f_3 = \frac{1}{k_3} = \frac{d_B}{b k F_P C_{H_2O}} V_P (\text{min})$$  \[10\]

where $k_1$ (min$^{-1}$), $k_2$ (min$^{-1}$) and $k_3$ (min$^{-1}$) are apparent rate constants for the chemical reaction, internal diffusion and external diffusion, respectively. Substituting Equations [8], [9] and [10] into Equation [2], one has

$$t = f_1 [1 - (1 - X)^{1/3}] + f_2 [1 - 3(1 - X)^{2/3} + 2(1 - X)] + f_3 X$$  \[11\]

A multi-variable least-squares linear regression method was used to find the values of the time constants of the three reaction steps, using the package ORIGIN 7.5, and fitting Equation [11] (as given above) to the data.

For the fitting procedure, the independent variables ($X_1$ to $X_3$) and model parameters ($\beta_1$ to $\beta_3$) were as follows

$$\beta_1 = f_1 X_1 = [1 - (1 - X)^{1/3}]$$

$$\beta_2 = f_2 X_2 = [1 - 3(1 - X)^{2/3} + 2(1 - X)]$$

$$\beta_3 = f_3 X_3 = X$$  \[12\]

**Effect of steam flow rate on the overall reaction rate**

The relationship between steam flow rate and overall reaction rate is given by

$$Q_{H2O} = \beta_1 + \beta_2 + \beta_3$$  \[13\]

where $Q_{H2O}$ is the steam flow rate (ml/min) at 1023 K.
Kinetics of the reaction between Pb-Sb complex sulphide concentrates and water

Figure 4—Effect of steam flow rate on the overall reaction rate at 1023 K

Table II

<table>
<thead>
<tr>
<th>Flow rate (ml/min)</th>
<th>( f_1 )</th>
<th>( f_2 )</th>
<th>( f_3 )</th>
<th>correlation coefficient</th>
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<td>567</td>
<td>15.16</td>
<td>171.83</td>
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<td>869</td>
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<td>0.9982</td>
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<td>1408</td>
<td>15.23</td>
<td>166.58</td>
<td>56.25</td>
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<tr>
<td>1723</td>
<td>15.22</td>
<td>162.70</td>
<td>38.37</td>
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<tr>
<td>2000</td>
<td>15.19</td>
<td>159.82</td>
<td>28.77</td>
<td>0.9886</td>
</tr>
<tr>
<td>2500</td>
<td>15.19</td>
<td>156.37</td>
<td>4.47</td>
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<td>3500</td>
<td>15.21</td>
<td>151.96</td>
<td>2.40</td>
<td>0.9742</td>
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rate for 30 minutes and 60 minutes at 1023 K is shown in Figure 4. From Figure 4, the volatilization rate of antimony is kept fixed when the steam flow rate is above 2500 ml/min. It is demonstrated that the external diffusion resistance \((f_3)\) of \(\text{H}_2\text{O}\) in the gas phase is very small.

The effect of steam flow rate in the range 567 to 3500 ml/min on the overall reaction rate at 1023 K is shown in Figure 5. It is clear that the reaction rate is enhanced with increasing steam flow rate. Substitution of the data obtained from Figure 5 into Equation [11] and making a regression analysis, the values of \(f_1\), \(f_2\) and \(f_3\) can be obtained at different steam flow rates, as shown in Table II.

From Table II, it can be seen that the external diffusion resistance \((f_3)\) of \(\text{H}_2\text{O}\) in gas phase is much smaller than the internal diffusion resistance \((f_2)\) and chemical reaction resistance \((f_1)\) when the flow rate is above 2500 ml/min. Therefore, Equation [11] may be simplified as

\[
t = f_1\left[1 - (1 - X)^{1/3}\right] + f_2\left[1 - 3(1 - X)^{2/3} + 2(1 - X)\right]
\]

**Effect of temperature on the overall reaction rate**

In order to eliminate the effect of external diffusion on the overall reaction rate, the flow rate of steam was controlled at 2500 ml/min. The effect of temperature on the overall reaction rate is shown in Figure 6. It is demonstrated that the conversion \((X)\) appreciably increases with raising reaction temperature. Substituting the data obtained from Figure 6 into Equation [13] and making a regression analysis, the values of \(f_1\) and \(f_2\) can be calculated at different temperatures, as shown in Table III. It is obvious that the chemical reaction resistance was decreased with the increase of temperature. At a lower temperature \((T < 1073 \text{ K})\), the process is controlled by chemical reaction and internal diffusion simultaneously\(^9\)-\(^12\), due to the values of \(\sigma^2\) being in the range of \(0.1-10\). Both the chemical reaction rate and internal diffusion rate can be accelerated by increasing the reaction temperature. When \(T > 1073 \text{ K}\) the values of \(\sigma^2\) is greater than 10, the chemical reaction resistance is smaller than that of internal diffusion. Under this condition, the process is mainly controlled by internal diffusion.

The values of \(f_1\) and \(f_2\) obtained previously were found to be in agreement with Equation [13] when \(1 - (1 - X)^{1/3} + f_2/f_1\left[1 - 3(1 - X)^{2/3} + 2(1 - X)\right]\) was plotted against \(t\) at different temperatures(Figure 7). It is clear from Figure 7 that straight lines are observed when temperature \(\leq 1023 \text{ K}\). This indicates that the volatilization rate of \(\text{Sb}_2\text{S}_3\) is in good

Figure 6—Effect of temperature on the overall reaction rate for \(Q_{\text{H}_2\text{O}}=2500 \text{ ml/min}\)

Table III

<table>
<thead>
<tr>
<th>Temp. (K)</th>
<th>( f_1 )</th>
<th>( f_2 )</th>
<th>( \sigma^2(f_2/f_1) )</th>
<th>Correlation coefficient</th>
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<tr>
<td>773</td>
<td>592.12</td>
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<tr>
<td>1023</td>
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<td>170.50</td>
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<tr>
<td>1073</td>
<td>13.28</td>
<td>143.65</td>
<td>10.82</td>
<td>0.9940</td>
</tr>
<tr>
<td>1173</td>
<td>9.17</td>
<td>137.13</td>
<td>14.95</td>
<td>0.9773</td>
</tr>
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</table>
agreement with Equation [13]. When the temperature > 1023 K and the fractional conversion \( X < 0.95 \), approximate linear relationships are obtained. However, when \( X > 0.95 \), the experimental results evidently deviate from the linear relationships. It may be explained by the existence of a small quantity of Pb-Sb complex compounds. Comparing the volatilization of antimony from the residual Pb-Sb complex compounds and single compounds, it is evident that the volatilizations of antimony from the Pb-Sb complex compounds become more difficult, causing deviations from experimental results. According to the values of \( f_2 \) and \( f_1 \), the values of \( k_1 \) and \( k_2 \) can also be calculated. Plotting \( \ln k_1 \) or \( \ln k_2 \) against \( 1/T \) (as shown in Figure 8) gives the temperature dependence of \( k_1 \) or \( k_2 \) as follows:

\[
\ln k_1 = -10334.4/T + 6.801 \quad [14]
\]

\[
\ln k_2 = -1235.1/T \pm 3.887 \quad [15]
\]

The apparent activation energies were calculated to be 85.92 kJ/mol for the chemical reaction and 10.27 kJ/mol for the internal diffusion, respectively.

**Effect of atmosphere on the overall reaction rate**

The effect of atmosphere on the overall reaction rate is shown in Figure 9 for \( t = 150 \) min at 1023 K. It is seen that the conversion(\( X \)) in the steam atmosphere is much greater than those in the air or nitrogen atmosphere.

In steam atmosphere, we consider the reaction:

\[
\text{Reaction 16}
\]

\[
\text{Reaction 17}
\]

Reaction [17] has been discussed in detail in our previous work published in Journal of Materials Science & Technology (Reference [15]).

In air atmosphere, the process can be described by the following reactions:

\[
\text{Reaction 18}
\]

\[
\text{Reaction 19}
\]

The FePb\(_5\)Sb\(_3\)O\(_{13}\) produced in Reaction 18 was enhanced with increasing air flow rate. Therefore, the conversion(\( X \)) was decreased with increasing air flow rate. The XRD pattern of the roast in the different atmospheres for 2000 ml/min flow rate at 1023 K is shown in Figure 10. In the steam atmosphere, the roast obtained is composed of PbS with small quantities of FeS, SiO\(_2\), and ZnS, and no Sb-containing species is found in the roast, making good a good Pb-Sb separation result. In the air atmosphere, quite differently, the roast consists of Pb\(_4\)Sb\(_3\)FeO\(_{13}\), FeS, Sb, and PbSO\(_4\).

In the nitrogen atmosphere, the roast contains complex sulphides (Pb\(_5\)Sb\(_3\)FeS\(_4\), PbSb\(_2\)S\(_3\), FeS, and PbS. The needle crystal Sb\(_2\)S\(_3\) is observed in the dust and tube wall. These complex compounds cannot be decomposed further into...
Kinetics of the reaction between Pb-Sb complex sulphide concentrates and water

single compounds; consequently, the Pb-Sb separation will be inhibited. It is suggested that the oxygen partial pressure of the system is a significant factor for Pb-Sb separation. The XRD pattern of condensed product in the steam atmosphere at 1023 K is shown in Figure 11. The condensed product obtained is composed mainly of Sb$_2$O$_3$ with small quantities of PbS and sulphur. The primary reason for different Pb-Sb separation results is that steam can decompose the Pb-Sb complex sulphide concentrates and the complex intermediate products into simple sulphides of lead, antimony and iron. At the same temperature, the saturated vapour pressure of the Sb$_2$S$_3$ is higher than that of PbS$^{17,18}$. The detailed discussion about the volatilization of Sb$_2$S$_3$ was published in *Mineral Processing and Extractive Metallurgy*.$^{18}$ Therefore, Sb$_2$S$_3$ will be preferentially volatilized into vapour phase and PbS enriched in the roast. Besides Sb$_2$S$_3$ in the vapour phase can be selectively oxidized by steam into Sb$_2$O$_3^{15}$. As a result, the partial pressure of Sb$_2$S$_3$ in the vapour phase will be reduced and the volatilization of Sb$_2$S$_3$ will be promoted. In contrast, in nitrogen atmosphere, Pb-Sb complex sulphide concentrates can be decomposed into single sulphides to some extent only. The remaining Pb-Sb complex sulphide concentrates hinders to volatilization of Sb$_2$S$_3$, although it is easily volatile. In air atmosphere, the Pb-Sb complex sulphide concentrates are transformed into stable Pb$_4$Sb$_3$FeO$_{13}$, making volatilization of antimony more difficult. The condensed product consists of Sb$_2$O$_3$ with a small quantity of PbS and S, as shown in Figure 11. This result indicates that Sb$_2$S$_3$ can be selectively oxidized to Sb$_2$O$_3$ by steam while PbS cannot be oxidized. In this case, the Pb-Sb separation achieves the anticipated result.
Kinetics of the reaction between Pb-Sb complex sulphide concentrates and water

Conclusion
The kinetics of the separation of antimony from lead for Pb-Sb complex sulphide concentrates by oxidation and volatilization roasting with water vapour is investigated. When the temperature <1073 K, the process rate of separation of antimony from lead is controlled by chemical reaction and internal diffusion. However, when the temperature >1023 K, the process rate is mainly controlled by internal diffusion. The apparent activation energies were calculated to be 85.92 kJ/mol for the chemical reaction and 10.27 kJ/mol for the internal diffusion, respectively.

The mechanisms for treating Pb-Sb complex sulphide concentrates have been investigated. XRD analysis of the products in steam atmosphere demonstrates that the reaction of Pb-Sb complex sulphide ores with steam can promote the Pb-Sb separation. The direct recoveries of Pb in roast and Sb in dust, which were examined by chemical analysis, are >94% and >93% for Pb-Sb complex sulphide concentrates by oxidative volatilization roasting with water vapour, respectively. Comparing the direct recoveries of 68% Pb and 40% Sb in the existing process, the Pb-Sb separation result is better than that of the existing process. In contrast, in a nitrogen atmosphere, the jamesonite (FePb₅Sb₃O₁₃) can be decomposed into single sulphides to some extent only. The remaining jamesonite hinders to volatilization of Sb₂S₃, although it is easily volatile. In air atmosphere, the jamesonite (FePb₅Sb₃S₁₄) is transformed into stable FePb₅Sb₃O₁₃, making volatilization of antimony more difficult.

Although hydrogen sulphide may be formed in the process, it is easily removed by the Claus process from the gas phase. When water vapour instead of air is used for the volatilization roasting of antimony-bearing concentrates and concentrates, the emission of sulphur-bearing gases will be reduced.

Acknowledgement
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References
The winners of the Nedbank Capital Green Mining Awards, which seek to acknowledge and celebrate the contribution that responsible mining makes to the economic development of Southern Africa, were announced at a ceremony at Nedbank’s head office in Sandton.

The winners are: Anglo Coal for its Isibonelo Wetlands project in the Environmental Category; Palabora Mining Company for the Palabora Foundation in the Socio-Economic Category and Anglo Platinum for the company’s sustainability programme in the Sustainability Category. No award was made in the Limited Resource Category.

Mark Tyler, head of Mining & Resources at Nedbank Capital says: ‘Nominations received for this year’s award were encouraging, bearing in mind this is the first official year where nominations were called for. No award was granted in the Limited Resource Category, as nominations did not satisfy all of the award criteria. This demonstrated that stringent judging played a significant role in determining the winners.

‘Sustainability is a philosophy, something that all mining companies should be striving for. Yet it is a complex and multi-faceted issue to measure, and therefore genuine success is difficult to determine. The bar for demonstrating sustainability is constantly being raised, whilst at the same time the ground upon which the principles of the philosophy stand, is constantly moving. It is against this background that the winners were chosen.’

Anglo Coal’s Isibonelo Wetlands Project is innovative and extends beyond compliance. Anglo Coal and its partners pioneered ‘off-site’ wetland rehabilitation in South Africa as mitigation action for on-site wetland loss. Anglo Coal recognized that due to its Isibonelo opencast operations, a portion of the Steenkoolspruit wetlands would be destroyed. As a mitigation measure for this loss, the company committed to rehabilitate a wetland area elsewhere in the Upper Oliphants River Catchment. They created a precedent, which other mining companies are following.

Palabora Mining Company’s Palabora Foundation, winner of the Socio-Economic Award, was recognized for its significant contribution in terms of social and economic upliftment of the communities surrounding the mine. Noteworthy characteristics of the Foundation’s work include, amongst others, the benefits of the programme, especially in the fields of mathematics, science and environmental education, educator development, schools governance programmes, skills development training, SMME programmes and healthcare. While the concept of mines forming trusts to benefit communities that surround them is not unique, Palabora’s win demonstrates a clear intent to continuously ‘push the boundaries’ to ensure the benefits are sustained way beyond the life of the mine.

Anglo Platinum, winner in the Sustainability Category, has made a significant effort in converging economics, the environment and society for the benefit of present and future generations with its Sustainable Development Programme. The programme is strategic, has executive buy-in and is mainstreaming sustainability at Anglo Platinum’s operations. The programme comprises a range of projects, including the Anglo Platinum Converting Project (ACP). It is acknowledged for its innovative approach to reducing sulphur emissions to well below the legal limit and, in doing so, setting a new standard for the platinum industry, and for working with the community in resolving key issues. In particular, RPM Union Section’s Community Environmental Outreach Programme has fostered an appreciation for the environment among learners and encourages them to make a positive contribution to the environment. The project has demonstrated a fruitful partnership between the environmental and corporate social responsibility departments of the mine, whereby a shared vision has reaped tangible benefits.

Other finalists for the Awards included:

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<td>Kumba</td>
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<td>Kgalagadi Charcoal and Firewood</td>
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<td>Project</td>
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