Cobalt and Nickel Separation in Nitric Acid Solution by Solvent Extraction Using Cyanex 272 and Versatic 10

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Abstract

Recent trend in extraction of nickel and cobalt from laterite ore through hydrometallurgical route is by using leaching agents that can be effectively regenerated, such as hydrochloric and nitric acid. Despite success stories of nickel laterite leaching in nitric acid, investigations on separation of nickel and cobalt from pregnant leach solution in this leaching media are still poorly reported. Nickel and cobalt are mostly recovered in the forms of intermediate products such as mixed hydroxide precipitate (MHP) and mixed sulphide precipitate (MSP). In this paper, separations of cobalt and nickel in nitrate solution and in pregnant leach solution of nickel laterite ore from Indonesia are discussed. Series of solvent extraction tests using Cyanex 272 and a combination of Versatic 10 and Cyanex 272 were performed under variations of equilibrium pH, extractant concentration, temperature and volume ratio of organic to aqueous phase (O/A ratio). The investigation results demonstrate that Cyanex 272 can be effectively used to separate cobalt and nickel in a solution with low nitrate concentration at the absences of Fe and Mg. The increase of equilibrium pH, extractant concentration, O/A ratio and temperature tends to enhance cobalt extraction and co-extraction of nickel. The best condition for separation of Co and Ni was obtained at equilibrium pH of 5, Cyanex 272 concentration 20% (v/v), temperature 25°C and O/A ratio 1/1. Separation of Co-Ni and Mg from pregnant leach solution can be well performed by 2 stage-SX using Versatic 10 followed by Cyanex 272 and selective stripping of loaded organic from the second stage of the extraction by using 1 molar sulfuric acid.

Keywords: Laterite, Nickel, Cobalt, Solvent extraction

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1. Introduction

Extraction of nickel from low-grade laterite ore of limonitic type is mostly performed through hydrometallurgical route. In addition to nickel, limonite ore typically contains 0.1-0.2% cobalt, 0.5-5% magnesium oxide and 40-50% iron. Leaching of limonite ore in acid solutions such as sulfuric, hydrochloric and nitric acid would result in a pregnant solution that contains nickel, cobalt, iron, magnesium and some other elements in lower concentrations such as chromium, aluminum and manganese. Recent trend in extraction of nickel and cobalt from laterite ore through hydrometallurgical route is by using leaching agents that can be effectively regenerated, such as hydrochloric and nitric acid. One of the processing technologies that is newly introduced is Direct Nickel (D Ni) which has been reported to be successfully extracted nickel and cobalt from laterite ore through leaching in nitric acid and able to recycle more than 95% of this leaching reagent. Despite the success stories of nickel laterite leaching in nitric acid using this technology, investigations on separation of nickel and cobalt in nitrate solution are still very limited. The performances of nickel and cobalt separation in nitrate medium are still poorly reported.

Nickel and cobalt are two elements that have very similar aqueous chemical properties due to their adjacent position in transition metal series in the periodic table. When these elements are both present in aqueous solution, they cannot be easily separated by a simple separation method such as chemical precipitation by pH adjustment. Cobalt and nickel separation in aqueous solution has been performed by oxidative precipitation, selective reduction by hydrogen gas, selective adsorption by ion exchange (IX) resin and solvent extraction. Cobalt can be separated from nickel in a concentrated hydrochloric acid solution in which cobalt is firstly converted to its anion complex of CoCl₄²⁻ and then selectively adsorbed by an anion exchange resin, while nickel is remains in solution as its cation. The drawback of this method is the high hydrochloric acid concentration of up to 9 molar required to form cobalt complex anion. Moreover, if other cations such as ferric, copper and zinc are present in solution, they will also form chloro complexes anions and interfere cobalt separation from nickel. Since the role of phosphorus acid is known, separation of Ni and Co in sulfuric acid solution by solvent extraction (SX) is significantly developed. Pregnant leach solution of nickel laterite ore typically has a high ratio of nickel to cobalt; therefore, separation of nickel and cobalt in this solution by SX requires an extractant that has a high separation factor of Ni and Co (β_{Ni-Co}). One of commercial SX reagents that has a high separation factor of nickel and cobalt (i.e. > 1000) in sulfuric acid solution is Cyanex 272. Cyanex 272 from Cytec Industries Inc. has a main component of bis (2,4,4- trimethylpentyl) phosphinic acid. Separations of nickel and cobalt by solvent extraction using Cyanex 272 in sulfate medium have been done by Hubicki and Hubicka, Tsakaridis, Agatzini and Gotfryd and the extraction mechanism has been reviewed in detail by Flett. Cyanex 272 has been used commercially in more than 13 nickel-cobalt refineries worldwide.

One of the drawbacks of using Cyanex 272 for separation of nickel and cobalt in pregnant leach solution of laterite ore is its low selectivity to iron and magnesium. Separation of Ni and Co can be effectively performed in a high purity solution which has very low concentration of magnesium and iron. In order to tackle this problem, two stage extraction of Co-Ni using Versatic 10 followed by Cyanex 272 has been studied. Tanlega reported that Ni and Co can be separated from Mg in sulfate solution at recovery level of 99.9% by two-stage extraction using 20% (v/v) of Versatic 10 with 5% (v/v) Tributyl Phosphate (TBP) as modifier at equilibrium pH of 7 and temperature 40°C. Co-extracted Mg was reported to be easily removed by a single stage scrubbing of the loaded organic using Co-sulfate solution, while co-extracted Fe(II) was removed by conducting selective stripping. As has been earlier mentioned, cobalt and nickel separations are mostly done in sulfate medium. By the developments of leaching processes in other medium such as nitric acid, investigations on cobalt and nickel separation in this media are urgently required. Hutton-Ashkenny et.al. have conducted solvent extraction test works in nitrate-based solution by using Versatic 10 extractant and TBP as modifier. The best condition found to obtain the highest cobalt and nickel recoveries of higher than 90% with co-extracted Mg of 2.6% is obtained at equilibrium pH of 5.5, O/A ratio 1/1, Versatic 10 concentration 0.8M, TBP 0.18M at 40°C. In this research work, separation of Ni and Co in nitrate solutions which contains only Ni and Co and Ni, Co and Mg as well as in pregnant leach solution of nickel laterite ore in nitric acid solution which has been subjected to iron removal is studied. The extractant used are Cyanex 272 and a combination of Versatic 10 and Cyanex 272. A critical factor such as the effect of nitrate concentration to the effectiveness of nickel and cobalt separation is elucidated.
2. Materials and Method

2.1. Materials

Initially, SX test was performed by using pregnant leach solution (PLS) that was generated by leaching of nickel limonite ore from Halmahera Island, Indonesia in 8M nitric acid at 95°C for 8h. The PLS contains (g/l): 1.13 Ni, 0.14 Co, 30.52 Fe, 3.47 Mg and 2.14 Al. The SX test was done by using Cyanex 272 to evaluate the extraction behaviors of Ni, Co, Fe, Mg and Al by Cyanex 272 in nitrate medium. After the extraction behaviors of Ni, Co, Fe, Mg and Al in PLS by Cyanex 272 was elucidated, three types of aqueous feed solutions were prepared. The first solution was a nitrate solution which contains only Ni and Co, while the second one was the nitrate solution which contains Ni, Co and Mg. The nitrate solutions were prepared by dissolving Ni(NO₃)₂·6H₂O, Co(NO₃)₂·6H₂O, and Mg(NO₃)₂·6H₂O powders in distilled water. All precursor materials are of analytical grade. The concentrations of Ni, Co and Mg refer to the concentrations of these metals in PLS that was generated from the leaching of limonite ore as previously described. The third type of the feed solution was PLS that was already subjected to iron removal. After solid-liquid separation, the PLS was subjected to iron precipitation at pH 2 by using NaOH as a neutralizing agent. The solution has 1.02 g/l Ni, 0.12 g/l Co and 3.4 g/l Mg. The organic extractants used were Cyanex 272 and Versatic 10 that were received from Cytec Inc. Kerosene and Tributyl Phosphate (TBP) were used as diluent and modifier, respectively. The extractant was mixed with diluent and modifier at certain volume percentages according to the desired experimental variations.

2.2. Methods

As has been earlier mentioned, SX test was firstly done by contacting PLS that majorly contains Ni, Co, Fe, Mg and Al with Cyanex 272 at 25°C, O/A ratio 1/1 in a 250 mL beaker glass. The Cyanex 272 concentration was 20% (v/v) and 5% (v/v) TBP was used as a modifier. The extraction behavior of Ni, Co, Fe, Mg and Al was investigated under variations of equilibrium pH. After the selectivity of Cyanex 272 to Co, Ni, Fe, Mg and Al was known, series of SX test works were carried out under variations of equilibrium pH, extractant concentration, temperature and O/A ratio by using nitrate solutions that contains only Co and Ni and Co, Ni and Mg. SX test by using PLS that has been subjected to iron removal by Cyanex 272 was done at the best condition obtained by the previous test works using nitrate solution.

Solution pH adjustments were performed by introducing drop-wise of NaOH or HNO₃ using a pipette until desired equilibrium pH was attained. In order to make the change of solution volume negligible, concentrated NaOH and HNO₃ of 10M, respectively, were used. Until equilibrium state is achieved, the pH values would change due to a release of H⁺ ions by the extractant during metals extraction. Equilibrium state was attained after approximately 3 minute. The SX tests were conducted at room temperature (i.e. 25 ± 0.5°C), unless it is stated being done at higher temperature. Variations of O/A ratios were made by varying organic volume at a constant volume of aqueous solution. After the equilibrium state is achieved, the solution was kept for several minutes to convince that no more pH alterations take place. Separation of aqueous and organic phase was then carried out by using a separator funnel, based on the specific gravity difference between the organic and the aqueous solution.

Metal concentrations in aqueous solution after extraction finished were measured by Atomic Absorption Spectrophotometer (AAS). The percentages of extracted metals (％E) in organic phase are determined from the difference between its initial weight in aqueous phase (w) and its final weight at equilibrium state (w₁), following this equation:

\[ %E = \frac{w - w_1}{w} \times 100\% = \left(1 - \frac{w_1}{w}\right) \times 100\% \]  

(1)

The ratio of metal concentration in organic phase and metal concentration in aqueous phase at equilibrium state during extraction is defined as distribution coefficient (D).
\[
D = \frac{[A]_{\text{org,eq}}}{[A]_{\text{aq,eq}}} \times 100\%
\]  

(2)

in which \([A]_{\text{org,eq}}\) and \([A]_{\text{aq,eq}}\) are the concentrations of metal A in organic and aqueous phases, respectively at equilibrium state. The effectiveness of cobalt and nickel separation is quantitatively measured by the value of separation factor of Co and Ni (\(\beta_{\text{Co-Ni}}\)) which is defined as:

\[
\beta_{\text{Co-Ni}} = \frac{D_{\text{Co}}}{D_{\text{Ni}}}
\]

(3)

in which \(D_{\text{Co}}\) and \(D_{\text{Ni}}\) are distribution coefficients of cobalt and nickel, respectively.

3. Results and Discussion

3.1. Results of Single Stage Extraction in PLS and Nitrate Solution Using Cyanex 272

In this section, the effect of equilibrium pH on extraction behavior of Co, Ni, Fe, Mg and Al by Cyanex 272 from PLS of Halmahera ore in 8M nitric acid solution is discussed. After the extraction behavior of the metals from PLS was understood, series of SX investigations using Cyanex 272 were conducted in nitrate solutions that contains only Ni and Co and Ni-Co-Mg to study the effects of extractant concentration and temperature on the distribution coefficients of cobalt (\(D_{\text{Co}}\)) and nickel (\(D_{\text{Ni}}\)) and separation factor of Co and Ni (\(\beta_{\text{Co-Ni}}\)). Profiles of extracted metals from PLS by Cyanex 272 as a function of equilibrium pH are depicted in Fig. 1. It was found that pH50 (i.e. pH at which 50% of dissolved metal in aqueous solution is extracted to organic phase) for Ni \(\approx 5.4\), Co \(\approx 4.2\), Mg \(\approx 4.0\) and Al \(\approx 3.4\), while pH50 for Fe cannot be determined since 100% of Fe extraction already occurred at pH 2. The experimental results indicate that Co and Ni in nitrate medium can be separated by Cyanex 272 with the pH50 difference of \(>1\), while Mg and Co separation is very difficult due to a very close pH50 of these metals. Similarly, separation of Fe and Al from Co and Ni in nitrate medium by Cyanex 272 cannot be effectively performed because the extractant tends to extract 100% Fe and Al prior to the extraction of Co and Ni takes place. At pH 5, extracted Co was 91%, while co-extracted Ni was 26.7%. At higher pH, co-extracted Ni was increased. Based on the experimental results, Fe and Al removal must be carried out prior to separation of Ni and Co in nitrate medium using Cyanex 272. Magnesium removal prior to extraction of Co and Mg is not commonly recommended due to alkaline pH range required for this metal precipitation by pH adjustments.

Fig. 1. Profiles of extracted metals from PLS by Cyanex 272 as a function of equilibrium pH at extractant concentration of 20% (v/v), 5% TBP

After the selectivity of Cyanex 272 to Fe, Al and Mg was elucidated, series of investigations were then carried out by using nitrate solutions that contain only Ni and Co and Ni, Co and Mg. For divalent metal cations (\(M^{2+}\)) such as Co, Ni, and Mg, metals extraction by organic extractant takes place through the following reaction:
\[ M^{2+} + (2+x)HA = MA_x \cdot xHA + 2H^+ \]  
\[ (4) \]
For Reaction (4), correlations between \( D \), \( K_E \), \( x \), and \( pH \) can be formulated as follows:

\[ K_E = \frac{[MA_x \cdot xHA \cdot [H^+]^2}{[M^{2+} \cdot [HA]^{(2+x)}} \]  
\[ (5) \]
\[ D_M = \frac{[HA]^{(2+x)}}{[H^+]^2} \]  
\[ (6) \]

In which \( KE \) is equilibrium constant for the extraction reaction, \([HA]\) is organic extractant concentration, and \([MA_x \cdot xHA]\) is concentration of organo-metallic complex forms during extraction. For Cyanex 272, P(0)OH is a functional group which actively reacts with metal cations by releasing \( H^+ \) ions. In logarithmic form, Equation (6), can be expressed as:

\[ \log DM - 2pH = \log KE + (2+x) \log [HA] \]  
\[ (7) \]

In which \( pH \approx -\log [H^+] \).

For extraction of divalent metal cations such as Ni, Co and Mg, at a constant extractant concentration, a plot of \( \log DM \) vs. equilibrium \( pH \) would result in a linear correlation with a slope value of 2. Profiles of \( \log DM \) vs. equilibrium \( pH \) from the SX testwork data in nitrate solutions that contain only Ni and Co and Ni, Co and Mg are presented in Fig. 2.a and Fig. 2.b, respectively. The slopes of linear regressions obtained for Co extraction in nitrate solutions that contain only Co and Ni and Co, Ni and Mg were 1.5 and 1.4, respectively. While, the slope of linear regressions for Ni extraction in solutions that contain only Co and Ni and Co, Ni and Mg were 1.58 and 1.08, respectively. These lower linear slopes (i.e. < 2) are associated with protonation of the extractant. At higher hydrogen ion concentration, the extractant tends to directly react with \( H^+ \) to form complex cation of \( H_2A^+ \) according to the following reaction:

\[ A^- + H^+ \leftrightarrow HA + H^+ \leftrightarrow H_2A^+ \]  
\[ (8) \]

If the extractant is stable in its complex cation of \( H_2A^+ \), there would be no organo-metallic complex formations and metals extraction does not take place.

SX tests under variations of extractant concentrations were carried out at equilibrium \( pH \) of 5, O/A ratio 1/1 at 25°C. The purpose of the test was to obtain extractant concentration that gives the highest separation factor of Co-Ni (\( \beta_{Co-Ni} \)). Extractant concentrations (v/v) were varied at 5%, 10%, 15%, 20% and 25%. The aqueous feed solutions were nitrate solutions that contain only Co and Ni and Co, Ni and Mg as has been previously described. Profiles of extracted metals from nitrate solutions that contain only Co and Ni and Co, Ni and Mg as a function of extractant concentration are presented in Fig. 3.a and 3.b, respectively. As expected, extracted metals increase by increasing extractant concentration. The best condition for extraction in Co-Ni solution was obtained at 20% of Cyanex 272 with extracted Co of 99.7% and co-extracted Ni 8.9%. For Co-Ni-Mg-nitrate solution, the best condition was also obtained at extractant concentration of 20% with extracted Co of 98.8% and co-extracted Ni 9.2%.

Linear regressions of \( \log D_{Co} \) and \( \log D_{Ni} \) data versus log concentration of extractant in Co-Ni solution demonstrate slope values of 4.6 and 3.3 for Co and Ni, respectively. This indicates that for Co extraction, one mole of cobalt ions would react with 5 mole of extractant molecule, while, one mole of Ni would react with 4 mole of extractant molecule during extraction according to the following reactions:

\[ Co^{2+} + 5HA = CoA_2 \cdot 3HA + 2H^+ \]  
\[ (9) \]
\[ Ni^{2+} + 4HA = NiA_2 \cdot 2HA + 2H^+ \]  
\[ (10) \]

SX tests under variations of temperatures were performed using Co-Ni nitrate solution at Cyanex 272 concentration of 20%, O/A 1/1 to evaluate whether the extraction reaction is endothermic or exothermic. The analysis was made by using the data of \( \log D \) from SX tests at various temperatures. Correlation between \( \log D \) versus \((I/T)\) follows the following prominent Van’t Hoff Equation:

\[ \log D = -\frac{\Delta H}{2.303RT} + K * \]
in which D is metal distribution coefficient, R is a constant of ideal gas (8.314 J/mole.K), T is absolute temperature (K), $\Delta H$ is reaction enthalpy (J/mole), $K^*$ is a constant that is obtained from the sum of $K_1$ and $K_2$. The constants of $K_1$ and $K_2$ are the values of $\frac{\Delta S}{R}$ and $\log \left( \frac{|HA|^{2+x}}{|H^+|^x} \right)$, respectively, in which $\Delta S$ is the entropy of the reaction. The SX tests were carried out at constants equilibrium pHs and extractant concentration. At this condition, profiles of log D of divalent metals versus (1/T) should be linear with a slope $-\frac{\Delta H}{2.303R}$. As can be seen in Fig. 4, profile of log D versus (1/T) for Co and Ni extraction in nitrate solution that contains only Co and Ni is linear with slope values of -2898 and -3660, respectively. These values are in correlation with the values of enthalpies ($\Delta H$) for Co and Ni extractions of 51.66 kJ/mole and 70.08 kJ/mole, respectively. The positive values of enthalpies indicate that Co and Ni extraction in nitrate media are endothermic reactions. This implies that extraction of both metals would be enhanced by the increase of temperature. The experimental result shows that temperature gives stronger effect to Ni extraction than to Co extraction. Higher extraction of Ni at higher temperatures results in lowering the value of $\beta_{\text{Co-Ni}}$.

Profiles of $\beta_{\text{Ni-Co}}$ as a function of equilibrium pH and extractant concentration in solutions that contain only Co and Ni and Co, Ni and Mg are presented in Fig. 5a and 5b, respectively. It can be seen that the presence of Mg which was added as Mg(NO$_3$)$_2\cdot6$H$_2$O powder reduces $\beta_{\text{Co-Ni}}$. This is in association with lowering Co extraction at the presence of...
Mg and at higher nitrate ion concentration by the addition of Mg(NO₃)₂.6H₂O. Profiles of β_{Co-Ni} in Fig. 5.a and 5.b demonstrate that the best condition for Ni and Co separation in nitrate solutions which is indicated by the highest β_{Co-Ni} is obtained at equilibrium pH 5 and Cyanex 272 concentration of 20% (v/v).

To predict the number of stage for multiple extractions of Co and Ni in nitrate solution by Cyanex 272 required to obtain the highest Co extraction (i.e. Co ≥ 99.9%) at the lowest co-extracted Ni, McCabe-Thiele was constructed. The diagram was constructed based on the SX test results at equilibrium pH 5, extractant concentration 20% (v/v), and temperature 25°C under variations of O/A ratios. For multistage extraction, the concentration of metal in aqueous solution after nth stage of extraction (Cₙ) can be determined by the following equation:

\[ C_n = C_i \left(\frac{1}{1+D_0}\right)^n \]  

in which Cᵢ is initial concentration of the metal in aqueous solution and D is distribution coefficient. The number of extraction stage for the highest Co extraction at the lowest co-extracted Ni at a certain O/A ratio can be determined by using the above equation. The calculation result shows that in nitrate solution that contains only Co and Ni, the highest Co extraction of 99.99% and the lowest co-extracted Ni of 19.29% can be obtained by two-stage extraction at O/A 1/1.

![Fig. 4. Profiles of Log DCo and Log DNi versus (1/T) in nitrate solution that contains only Co and Ni; Fig. 5. Profiles of ECo-Ni: (a) as a function of equilibrium pH at extractant concentration of 20% (v/v), 25°C and O/A 1/1; (b) as a function of extractant concentration at 25°C, equilibrium pH 5 and O/A 1/1.](image)

3.2. Results of Two Stage SX of PLS By Versatic 10 Followed by Cyanex 272

Single stage SX of PLS that has been subjected to Fe removal by Cyanex 272 at pH 5, extractant concentration 20% at 25°C and O/A 1/1, resulted in Co extraction of 99.25% and co-extracted Ni of 67.59%. For reducing co-extracted Ni from PLS, two-stage extraction by using Versatic 10 followed by Cyanex 272 was performed. Refer to the results of SX test in sulfate medium, Versatic 10 is expected to separate Co and Ni from Mg. Co and Ni recovery by Versatic 10 was also aimed at reducing nitrate anion concentration in the feed solution of the second stage extraction using Cyanex 272 for Co-Ni separation. The SX test using Versatic 10 was carried out at equilibrium pH of 6.95, extractant concentration 20% (v/v), O/A 1/1 and temperature 40°C. In contrast to the result in sulfate medium, SX test using Versatic 10 using PLS in nitrate medium exhibits low selectivity to magnesium with co-extracted Mg of 88.8% at extracted Co and Ni of 97.95% and 92.03%, respectively.

The next step done was stripping of loaded Versatic 10 by using 1 molar nitric acid. Cobalt stripping percentage of 99.79% was obtained, while stripped nickel and magnesium were 75.3% and 62.04%, respectively. The solution from stripping of loaded Versatic 10 was then contacted with Cyanex 272 at pH 5, extractant concentration 20% (v/v), O/A 1/1 at 25°C. Extracted cobalt of 99.73% was obtained, while co-extracted nickel and magnesium were 53.62% and 77.09%, respectively. This experimental result demonstrates that two-stage extraction of Co and Ni by Versatic 10 and Cyanex 272 does not give significant improvement on separation of Co and Ni in nitrate-based PLS. The reduce of co-extracted Ni was only 13.97% in comparison to the single stage extraction by Cyanex 272 that give co-extracted
Ni of 67.59%. High level of co-extracted Ni can be solved by selective stripping. Stripping of loaded Cyanex 272 from the second stage extraction was performed by using 1 molar sulfuric acid. The stripping test using 1 molar sulfuric acid resulted in stripped Co and Mg of 99.99% and 99.99%, respectively, while stripped Ni was only 1.48%. Separation of Co from Mg can be easily done by conducting selective precipitation of Co as its hydroxide at pH range where Mg$^{2+}$ still remains in solution. Extracted Ni in organic phase can be stripped later by more concentrated sulfuric acid of about 2 molar and recovered as its hydroxide or pure metal by electrowinning.

4. Conclusions

The investigation results demonstrated that Cyanex 272 has excellent performance for separation of cobalt from nickel in artificial nitrate solution which contains only Co and Ni cations and has the highest $\beta_{\text{Co-Ni}}$ of 6364.95. The increase of equilibrium pH, concentration of Cyanex 272, O/A phase ratio and temperature tend to increase extracted Co and co-extraction of Ni. The optimum condition found for separation of cobalt from nickel in artificial nitrate solution which contains only Co and Ni cations are at equilibrium pH of 5, Cyanex 272 concentration of 20% (v/v), at room temperature and O/A ratio 1/1, which resulted in Co extraction of 99.08% and co-extracted Ni 10.16%. Separation of Co and Ni from pregnant leach solution of laterite ore from Halmahera Island by direct SX using Cyanex 272 did not take place selectively. Separation of Co and Ni in pregnant leach solution can be well performed by 2 stage-SX using Versatic 10 followed by Cyanex 272 and selective stripping of loaded organic from the second stage of the extraction by using 1M H$_2$SO$_4$. By using this method, Co and Mg stripping percentages of 99.99%, respectively were obtained, while Ni stripping percentage was only 1.48%.

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