GOLD RECOVERY FROM PRINTED CIRCUIT BOARDS OF MOBILE PHONES SCRAPS USING A LEACHING SOLUTION ALTERNATIVE TO CYANIDE

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Abstract - Currently, the printed circuit boards (PCB) of waste electrical and electronic equipment (WEEE) are attracting interest among researchers for environmental concern reasons and mainly for their content of precious metals such as gold. Thus, this study aims to characterize different types of PCBs from mobile phones in relation to the amount of gold contained, to evaluate alternative leaching agents for the gold (ammonium and sodium thiosulfate) and compare these to commercial stripping (cyanide-based) and then the recovery of gold by the electrometallurgical route. First, the amount of gold was determined. Then, alternative leaching agents were tested under different concentrations and time. A cyanide-based solution was also tested to compare the results. The results showed that the content of gold varied from 142 to 700 g/ton. The cyanide-based solution was able to extract 88% of the gold, while sodium and ammonium thiosulfate extracted 70 and 75% of the gold, respectively. The electrowinning tests showed a 94% recovery of the gold present in thiosulfate solutions.

Keywords: Gold Recovery, Hydrometallurgy, Electrometallurgy, Printed Circuit Boards, Thiosulfate.

INTRODUCTION

Each year, millions of tons of waste electrical and electronic equipment (WEEE) are generated worldwide (Huisman et al., 2008). Most of this scrap (especially from small appliances) continues to be discarded with household waste, causing environmental pollution. Alternatively, it is stored at home or in warehouses waiting for a final destination, resulting in the loss of materials (Gutiérrez et al., 2010; Townsend, 2011; Li et al., 2012). Devices such as mobile phones (increasingly popular and with relatively short life cycles) may be considered important sources of precious metals (Ag, Au and Pd) (Ongondo and Wiliams, 2011; Adie et al., 2016; Zeng, 2016).

Although they are used in small quantities, precious metals have significant economic importance in the production of electric and electronic equipment (EEE). The numbers may be staggering, more than 80% of
the value of a mobile phone, for instance (Hageluken and Corti, 2010). Meanwhile, from an environmental point of view, for some metals, it is estimated that it is currently necessary to remove about three times more soil/rock than a century ago to extract the same amount of ore (Gerst and Graedel, 2008; UNEP, 2013).

Thus, recycling, when properly followed and implemented, can bring innumerable benefits, from both an environmental and an economic perspective. In addition to reducing and/or minimizing the environmental damage inherent in the primary extraction of materials, recycling can be considered as an important secondary source to gain access to valuable materials (Sinha-Khetriwal et al., 2005; Kang and Schoenung, 2005; Williams, 2006; Cui and Zhang, 2008; Mo and Zongguo, 2009; Kasper et al., 2011a).

The presence of elements that are high potential polluters (e.g., toxic metals and flame retardants) on the PCBs of mobile phones make pyrometallurgical recycling in small and medium industries difficult, due to the high cost involved in deploying filters and equipment to avoid air pollution (Hall et al., 2007; Sepúlveda, 2010). Although some studies report that these emissions of pollutants can be greatly reduced in the recovery of PCB precious metals from electronic scrap using pyrolysis processes, these processes can still be considered too complex for application in small recycling industries (Bidini et al., 2015; Hense et al., 2015).

Thus, the extraction of gold by hydrometallurgy, with subsequent recovery of the metal in the solution, emerges as a more viable alternative for industrial application (Lee et al., 2011). Hydrometallurgy, a traditional method for the recovery of gold, consists of metal leaching by cyanide. Thus, currently, products can be found on the market intended for commercial stripping for gold, using cyanide in the formulation, for gold recovery from PCBs and other types of scrap. However, cyanide, being a highly toxic component, must be handled and disposed of to the environment with care and proper treatment (Oh et al., 2003; Yap and Mohamed, 2007; Cui and Zhang, 2008; Konyratbekova et al., 2012).

One of the most promising alternatives to cyanide as a leaching agent for gold is thiosulfate. The interest in thiosulfate arises from the fact that it is a relatively inexpensive product, with low toxicity and with the possibility of application in gold ores that cannot be treated by the conventional cyanidation process (due to the high cost of reagents and/or low gold recovery) (Senanayake, 2005a; Zhang and Senanayake, 2016).

The dissolution of gold with a thiosulfate solution occurs by forming a gold and thiosulfate complex, \( \text{Au(S}_2\text{O}_3\text{)}^2^- \). The complex is soluble and very stable in neutral and alkaline medium. However, only the maintenance of adequate concentrations of thiosulfate, ammonium, copper and oxygen, in suitable potential and pH conditions, enables efficient extraction of the gold (Jeffrey, 2001; Jeffrey and Breuer, 2002; Grosse et al., 2003; Senanayake, 2005b; Arslan and Sayiner, 2007; Ha et al., 2014).

The leaching reaction with thiosulfate is very slow. Therefore, for the process to occur at reasonable speeds, the presence of copper (II) ions and ammonium is required. The cupric ions (Cu\(^{2+}\)) act as the catalyst for the reaction. The ammonium has the role of stabilizing the copper in cupric form (Cu\(^{2+}\)), influencing the gold dissolution rates (Jeffrey, 2001; Jeffrey and Breuer, 2002; Grosse et al., 2003; Senanayake, 2005b; Arslan and Sayiner, 2007; Ha et al., 2014; Oraby et al., 2014).

Thiosulfate is a metastable anion, which tends to suffer chemical decomposition in aqueous solutions, depending on the pH and the potential of the solutions. In the case of the gold leaching process using thiosulfate, copper oxidation by thiosulfate deserves special attention because it is a thermodynamically favored reaction. However, in alkaline solutions, oxidation of copper by thiosulfate is much slower (Zhang, 2004).

Most published studies on gold leaching with thiosulfate focus on gold ores. However, in recent years some studies have begun to be conducted on the recovery of gold from electronic scrap (Ha et al., 2010; Tripathi et al., 2012). Thus, this study aims to characterize different types of PCBs in relation to the amount of gold contained, to evaluate alternative leaching agents (ammonium and sodium thiosulfate) for the extraction of the gold present in the PCBs of mobile phones, and to compare this to commercial stripping (cyanide-based). Furthermore, a gold recovery process from thiosulfate solutions by the electrometallurgical route was performed.

**MATERIALS AND METHODS**

**Experimental procedure**

In this work, sodium thiosulfate (Na\(_2\text{S}_2\text{O}_3\)) and ammonium thiosulfate ((NH\(_4\))\(_2\text{S}_2\text{O}_3\)) were tested as alternatives to cyanide in the leaching of gold from the PCBs of mobile phones. The work involved several steps that are shown in the flowchart in Figure 1.
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From a literature review (leaching of gold ores, mostly), it was found that there are two research lines. Some authors worked with lower concentrations of thiosulfate and ammonium for a leaching time of 4 hours, whereas others worked with higher concentrations of thiosulfate and ammonium and a leaching time of 24 hours (O'Malley, 2002; Zhang, 2004; Xu et al., 2015; Aylmore et al., 2014; Lampinen et al., 2015). Thus, for this study it was decided to test the two hypotheses, to check which approach to the leaching of gold from the PCBs is better. Thus, tests were performed with lower concentrations of thiosulfate and ammonium for the 4-hour leaching time and tests with higher concentrations of thiosulfate and ammonium for 24 hours. The sodium and ammonium thiosulfate were tested under different conditions of concentration, time, pH and temperature, as described below.

**Printed circuit boards**

The PCBs used in this study were obtained from defective or obsolete mobile phones, collected in stores with technical assistance offered by multiple brands. PCBs from mobile phones manufactured in the years 2001-2005 were used.

The leaching tests were performed with unbroken PCBs (without grinding), manually removed from the mobile phone devices.

The use of unbroken PCBs is justified by previous studies. These show that mechanical processing is not an efficient way to recover precious metals from PCBs, due to the high loss rate of these metals during processing. In addition, milling allows the release of other metals (nickel, copper, etc.) present on the PCB and increases their contact area with the leaching solution, thus competing with the desired reactions of the gold leaching (Wu et al., 2008; Kasper et al., 2011b; Hou et al., 2013). Moreover, morphological studies showed that 90% of the gold (in the gold-nickel alloy) is found in the external layers (Ha et al., 2014).

For this study, eight different types of mobile phone PCBs were used, as seen in Figure 2.

All tests were performed under agitation. In each test a single unbroken PCB was digested and the tests were performed in triplicate.
Characterization of PCBs in relation to gold quantity

Initially, each type of PCB was characterized by digestion of gold with aqua regia (3 HCl + 1 HNO₃) and posterior determination of the concentration by atomic absorption spectroscopy. This procedure was aimed to obtain the total amount of gold contained in the PCBs.

The parameters used in this characterization were: temperature 60±(2)°C, 2 hours of digestion and solid/liquid ratio 1/25. These tests were realized in a closed reactor to prevent environmental contamination.

Gold leaching tests

After characterization, leaching tests with commercial stripping (cyanide-based) and alternative leaching agents (sodium and ammonium thiosulfate) under different conditions were performed.

Leaching tests with commercial stripping

The commercial stripper used here for chemical stripping of gold and alloys of nickel involved simple immersion. The product is provided in the form of a yellow liquid containing cyanide that removes a layer of up to 1 mm gold per minute at room temperature (Galva, 2011).

The leaching tests were carried out using the parameters described in the user manual of the product. The parameters used in gold leaching of mobile phone PCBs with commercial stripping were: temperature 25±2(°C), 2 hours of extraction, pH 12.5 and solid/liquid ratio 1/25.

All tests were performed in a closed reactor. The PCB samples were suspended from a nylon wire to prevent them from being in contact with the walls of the reactor. For economic and environmental reasons, only PCBs of types A, B and C, shown in Figure 2, were tested.
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**Leaching tests with sodium and ammonium thiosulfate**

In this work, different concentrations of thiosulfate, ammonium hydroxide and copper (II) sulfate were tested. Sodium hydroxide (NaOH) and sulfuric acid (H\textsubscript{2}SO\textsubscript{4}) were used to adjust the pH of the system during the tests. All tests were performed in an open reactor (due to requiring the presence of oxygen in the process). As before, the PCB samples were suspended from a nylon wire to prevent contact with the walls of the reactor.

The parameters used in gold leaching from mobile phone PCBs with thiosulfate are described in Tables 1 and 2.

**Leaching tests for 4 hours**

The parameters used in the leaching tests of gold with sodium thiosulfate and ammonium thiosulfate for 4 hours are shown in Table 1.

**Leaching tests for 24 hours**

The parameters used in the leaching tests of gold with sodium thiosulfate and ammonium thiosulfate for 24 hours are shown in Table 2.

**Electrowinning process**

The solutions used in the electrowinning tests were obtained by leaching ten different PCBs from mobile phones in a solution containing 15 mM copper sulfate, 0.12 M thiosulfate and 0.2 M ammonia. After leaching, the solutions were analyzed in relation to their gold and copper content and then submitted to the electrowinning process.

In each test, a single solution was first subjected to a potential of -500 mV vs. Ag/AgCl for the gold recovery. Subsequently, the cathode and the anode were changed to the copper electrowinning using a potential of -700 mV vs. Ag/AgCl. These potentials were defined in our previous work (Kasper et al., 2016).

The electrochemical reactor used in this work consisted of a 150 mL Pyrex glass. Two electrodes of graphite were used as the cathode and the anode, and an electrode of Ag/AgCl (in saturated KCl) was used as the reference electrode. Both the cathode and the anode were made of two cylindrical graphite bars with an effective area of 14.15 cm\textsuperscript{2}.

The electrowinning experiments were controlled with a potentiostat / galvanostat Autolab PGSTAT302N that was connected to a PC (Software GPES 4.9).

Electrowinning tests with solutions containing commercial stripper were not performed because the process with cyanide solution is well known and established.

**Chemical analysis**

The amounts of gold extracted in the characterization tests (digestion) and leaching tests of the mobile phone PCBs were determined by atomic absorption spectroscopy and inductively coupled plasma atomic emission spectroscopy, using a spectrometer ICP OES OPTIMA 2000DV (PerkinElmer) and a Spectrophotometer PerkinElmer, model Analyst 100.

**RESULTS AND DISCUSSION**

**Characterization of PCBs**

Table 3 shows the amounts of gold based on the results obtained in the characterization tests for each type of PCB sample.

The results indicated that each type of PCB contains a different amount of gold, which varies according to the complexity and the number of functions displayed by the devices to which they belonged. In the case of the PCBs used in this study, the amounts of gold varied from 142 g/t to over 700 g/t.

![Table 1. Parameters used in the gold leaching tests with thiosulfate for 4 hours](image)

![Table 2. Parameters used in the gold leaching tests with thiosulfate for 24 hours](image)
The results of this study also highlighted that, except for the board type C, the others types of PCBs contained greater amounts of gold than those cited in the literature, at 300-350 g/t (Huisman et al., 2007; Chancerel et al., 2009; Hageluken and Corti, 2010). This disparity between the amounts of gold extracted in this work and the data cited in the literature can be explained by the use of different types of PCBs and/or the year of manufacture of the PCBs tested (it is known that the amount of gold used in PCBs has varied over the years) (Hageluken and Corti, 2010; Polak and Drápalová, 2012).

Finally, the results of the characterization tests show that the PCBs of mobile phones contain appreciable amounts of gold in their composition, since the average levels found in ores are only 5-10 g/t of metal. This makes recycling interesting economically.

**Results of gold leaching tests**

**Leaching tests with commercial stripping**

Cyanide has been used for more than 100 years in gold leaching of ores, and is currently also used in the leaching of gold from secondary sources, due to its high efficiency and relatively low cost (Trindade and Barbosa Filho, 2002; Syed, 2012).

The results of this study show that 2 hours of extraction with commercial stripping obtained an average of 88% extraction of the gold contained in the PCBs and an average standard deviation of 3.5%.

This result was considered satisfactory, due to the practicality of the process (without heating and a relatively short leaching time) and the possibility of recovery of gold present in the solution by techniques such as cementation or electrometallurgy.

However, this commercial product has cyanide in its formulation, which has a high degree of toxicity by inhalation, ingestion and skin contact and a high capacity for environmental contamination, because contact with acids releases toxic gases (Galva, 2011). For this reason, in the present study, the replacement of this cyanide-based product was investigated.

**Leaching tests with sodium thiosulfate and ammonium thiosulfate**

**Leaching tests for a duration of 4 hours**

According to literature reports (Abbruzzese, et al., 1995; Breuer and Jeffrey, 2000; Chandra and Jeffrey, 2004; Zhang and Nicol, 2005; Ha et al. 2010; Ha et al. 2014), the main function of the use of copper ions in the leaching process with thiosulfate is to accelerate the rate of chemical reaction. In view of this, all the tests with thiosulfate for a duration of 4 hours included copper (II) ions (in the form of copper sulfate).

**Sodium thiosulfate**

The results (average and standard deviation) of the leaching tests for a duration of 4 hours, using sodium thiosulfate as a gold leaching agent, are shown in Figures 3(a) and 3(b).

In Figure 3(a), it is observed that most of the solutions containing 0.3 M ammonium resulted in a percentage of extraction higher than 50%. The best results were obtained in solutions containing 0.15 M thiosulfate and 15 mM copper sulfate, with a percentage greater than 62% of extraction and a standard deviation of 4.5%.

In Figure 3(b), it can be seen that the best results obtained in tests with 0.2 M ammonium were obtained with the use of solutions containing 0.12 M of thiosulfate and 20 mM of copper sulfate. With these concentrations of reagents, it was possible to extract almost 70% of the gold contained in the PCB, at a standard deviation of 5.4%.

For solutions containing 0.3 M of ammonium, the gold extraction was similar using solutions containing 0.12, 0.15 or 0.20 M of thiosulfate. With just 0.10 M, an extraction rate below 50% occurred. For the solution containing 0.2 M ammonium, the maximum leach was achieved using solutions containing 0.12 M of thiosulfate.

**Ammonium thiosulfate**

The results of the 4-hour tests using ammonium thiosulfate as a gold leaching agent are presented in Figures 4(a) and 4(b). The figures show that, in the case of ammonium thiosulfate, the best results were obtained using the lower thiosulfate concentrations tested, i.e., 0.1 to 0.12 M. In these cases, the extraction exceeded 50%.

**Table 3. Results of the characterization of PCBs**

| Types of PCB | Amounts of gold calculated (g/t of PCB) |
|--------------|----------------------------------------|
| A            | 508.7 ± 21.7                           |
| B            | 641.4 ± 29.8                           |
| C            | 142.6 ± 14.1                           |
| D            | 702.3 ± 25.5                           |
| E            | 412.4 ± 14.6                           |
| F            | 421.0 ± 13.3                           |
| G            | 560.9 ± 18.9                           |
| H            | 523.5 ± 27.2                           |
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Figure 4(a) shows that, in solutions containing 0.3 M ammonium, the best results were obtained for solutions containing 0.10 M of thiosulfate and 20 mM of copper sulfate, with an extraction percentage greater than 58% and a standard deviation of 5.1%. Moving further, Figure 4(b) maps the best results obtained in the 4-hour duration tests that were carried out using solutions containing 0.12 M of thiosulfate, 0.2 M of ammonium and 20 mM of copper sulfate. With these concentrations of reagents it was possible to extract almost 75% of the gold contained in the PCB, with a standard deviation of 4.0%.

With regard to copper sulfate concentrations, no dependent relationship between these concentrations and the leaching rates could be observed.

Leaching tests for a duration of 24 hours

Sodium thiosulfate

The results obtained in leaching tests with a duration of 24 hours are presented in Figures 5 (a) and (b). These results show that leaching with sodium thiosulfate gave the best results when solutions containing 2 M thiosulfate, 2.5 M ammonium and 20 mM copper sulfate were used. Using these concentrations of reagents, it was possible to extract 51% of the gold of the mobile phone PCBs, with a standard deviation of 4.3%.

Figure 5 (a) shows that the leaching reaches its maximum in 2 M thiosulfate and then reduces again when the thiosulfate concentration increases beyond 3M.
In the graph shown in Figure 5 (b), it is observed that the leaching reached the maximum value for a concentration of 0.5 M of thiosulfate, remained stable in the concentrations of 1 M and 2 M of thiosulfate, but was reduced in a concentration of 3 M of thiosulfate.

These results indicate that an increase in the concentration of thiosulfate does not represent an increase in the gold leaching rate. Literature data (Jeffrey, 2001; Tripathi et al., 2012) specify that higher concentrations of thiosulfate causes an upsurge in the concentration of its degradation products, such as sulfate, tetrathionate, and trithionate. Since the stability of the gold thiosulfate complex depends on the thiosulfate concentration in the solution, it is expected that the gold leaching increases with an increase in the concentration of thiosulfate until an optimal composition of the solution is reached. Therefore, the thiosulfate concentration must be controlled in order to maintain a proper ratio between the concentrations of thiosulfate and ammonium, so that the copper can perform as a catalyst involving the variation between the cuprous and cupric states.

It is also observed that solutions containing 1 M ammonium thiosulfate and 2.5 M ammonium (without copper sulfate), and solutions containing 0.5 M ammonium thiosulfate and 1.5 M ammonium (with and without copper) resulted in an extraction of 48%. Considering the lower consumption of reagents this may also be seen as a good result.

**Ammonium thiosulfate**

Because of the high cost of ammonium thiosulfate (approximately eight times the cost of sodium thiosulfate) and the results obtained in leaching tests with sodium thiosulfate, leaching tests using 3 M ammonium thiosulfate were not conducted. Thus, in this work the maximum ammonium thiosulfate concentration tested was 2 M.

The results obtained in 24-hour leaching tests using ammonium thiosulfate as a gold leaching agent are presented in Figures 6(a) and 6(b). The best results were obtained using solutions containing 2 M thiosulfate and 2.5 M ammonium (regardless of the addition or not of copper ions). Using these concentrations of reagents, it was possible to extract about 51% of the gold from the mobile phone PCBs, with a standard deviation of 3.2%.

In general, it can be said that, under the conditions tested, the results were inferior to those obtained when using sodium thiosulfate, since, with the exception of solutions containing 2 M thiosulfate and 2.5 M ammonium, the other solutions tested resulted in low rates of gold leaching. In addition, the higher cost of ammonium thiosulfate than sodium thiosulfate should be taken into consideration.

Regarding the influence of the addition of copper on the leaching rates, it was observed that, unlike literature data (Jeffrey, 2001; Tripathi, 2012), in the 24-hour leaching tests no significant difference was found between the tests without copper (II) ions and tests with 10 and 20 mM copper (II). However, in at least one case in the literature, the addition of copper was also deemed unnecessary. In their work, Feng and van Deventer (2010) did not add copper sulfate to the leaching ores containing copper (chalcopyrite, for example) by thiosulfate, but they found that rapid formation of the copper(II)-ammonium complex resulted from adding the ammonium.
The results are consistent with those reported in the literature for gold ores.

**Gold and copper electrowinning**

The chemical analyses indicated that the solution used in the electrowinning tests contained 45.1 ppm of gold and 1130 ppm of copper.

For this step of the work, the analyzed parameters were: fraction of gold and copper recovered, electrical efficiency, productivity and energy consumption.

Figure 7 shows the fractions of gold and copper recovered. The gold fraction recovered was equivalent to 0.94, at a potential of -500mV vs. Ag/AgCl. The copper fraction recovered was equivalent to 0.95, to a potential of -700mV vs. Ag/AgCl.

Figure 8 shows the values calculated for electrical efficiency, productivity and specific energy consumption.

**Figure 6.** Comparison of leaching with ammonium thiosulfate (a) in 2.5 M ammonium (b) in 1.5 M ammonium.

**Figure 7.** Fractions of gold and copper recovered.

**Figure 8.** Comparative of (a) electrical efficiency, (b) productivity and (c) specific energy consumption.
As can be observed, the electrowinning of gold from solutions containing copper, especially when the amount of copper is much greater than the amount of gold, leads to a low electrical efficiency. In this case, it is observed that the electrical efficiency reached 2.2%. However, according to the literature (O'Malley; 2002), this low current efficiency is usual in the gold industry, which normally operates at current efficiencies below 10%.

The productivity falls over time, because in this case the system is operating at a current density limit. The current density decreases over time because it is proportional to the concentration, and this decreases with time. Thus, the speed of gold recovery will decrease over time, which leads to a reduction in productivity. It is also observed that energy consumption increases with time. This fact is quite logical, since over time the amount of gold ions in solution to be recovered decreases and the secondary reactions take on more and more significance in the system.

The results obtained to evaluate the efficiency of the electrowinning process may be considered quite satisfactory. Among the factors that contributed to these results are the low amount of gold present in the solution and the possibility of the occurrence of other metals besides copper in solution. Regarding the presence of other metals in solution, in addition to copper, it should be noted that mobile phone PCBs are composed of a large quantity of metals that may, eventually, have been leached by thiosulfate.

CONCLUSIONS

In this work, the extraction of the gold present in obsolete and/or damaged mobile phone PCBs was studied. The results showed that the PCBs of scapped mobile phones contain appreciable amounts of gold in their composition. The gold content present in the PCBs depends on the type of PCB used in each device, and may vary between 140 and 700 g of gold per tonne of PCB. The gold content found in these PCBs is much higher than the average levels found in gold ore, which is currently 5-10 g of gold per tonne of ore.

In leaching tests with commercial (cyanide-based) stripping, it was possible to extract 88% of the gold present in PCBs. The advantage of using cyanide is the simplicity of the process, while its disadvantage is the high toxicity and the need for greater control in the treatment of effluents.

In the sodium thiosulfate and ammonium thiosulfate tests, it was possible to extract 70 and 75% of the gold contained in the PCB, respectively, using solutions containing 0.12 M of sodium or ammonium thiosulfate, 0.2 M of ammonium and 20 mM of copper sulfate in 4 hours of leaching, at a temperature of 30(2°C and pH 10. The use of alternative leaching agents (sodium thiosulfate and ammonium thiosulfate) has as its major advantages the low toxicity and rates of extraction of gold close to those when using cyanide. The disadvantage of the process is the great complexity of the reactions involved, where some points of the process are not fully understood, and the need for tight control of all the process variables.

In real solutions containing approximately 45 ppm of gold and 1130 ppm of copper, the fraction of gold recovered reached 0.94 (i.e., 94% of the gold present in the solution), while the copper fraction recovered reached 0.95 (i.e., 95% of the copper present in the solution), using electrode potentials of -500 and -700 mV, respectively. The current efficiency achieved for gold in the experiments with real solutions was low, less than 3%.

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