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**TOXICITY AND STABILITY OF IRON-ARSENIC ALLOY**

**Abstract:** Iron-arsenic alloy is a waste-form of a novel gold recovery technology from gold-arsenic-bearing concentrates. This study investigates toxicity and solubility of Fe-As alloy in aqueous solutions. Results of the TCLP-like test, to evaluate the potential toxicity of a solid Fe-As alloy, suggest that the alloy is not stable in acidic conditions (pH 2.88), and releases As in concentrations exceeding the TC threshold level by more than four times. Calcium arsenate dust toxicity has been also evaluated for comparison. It was found to release 40 times more As than iron-arsenic alloy. Concentrations of As in the SPLP-like test leachates were well within the regulatory limit, thus demonstrating the relative stability of the Fe-As alloy in normal meteoric waters (pH 5). Long-term leaching tests in the environmentally-relevant pH range of 5 to 9 at 22°C demonstrate that Fe-As alloy is very susceptible to acidic pH conditions. Arsenic solubilization was high under acidic conditions (at pH5 and pH6), which suggests that in the long term it can be mobilized within the environment if exposed to meteoric waters, and certainly to acid rain. However, at the neutral and alkaline pH conditions typical of monofills it appears to be stable over the long term.

**Key words:** gold recovery; Fe-As alloy; arsenic; toxicity, stability.

**Introduction**

As mobility from waste materials is of considerable environmental significance, particularly in relation to their mobilisation into water supplies [1]. Following increasing body of published research proving As toxicity [2], the WHO reduced drinking water limit for As to 10 µg/L, albeit some countries have more stringent standards. Kazakhstan has not brought regulatory limits for As in drinking water to this concentration yet due to issues related to detection limit of analytical equipments currently used in laboratories, and still operates at MCL of 50 µg/L.

As often accompanies non-ferrous and precious metals in sulfidic ores and it is the main concern in mining and processing of the refractory As-bearing gold ores. Earlier, we proposed a novel single stage method for gold recovery from double refractory gold-arsenic-bearing carbonaceous concentrates, based on melting of the concentrate with Fe-rich waste slag and lead oxide in reducing atmosphere, the so called direct reductive melting (DRM) process [3]. Processing locks As into a relatively benign (compared to alternative methods) iron-arsenic alloy and concentrates gold into lead bullion. The method has been published as a patent with the Patent Office of the Republic of Kazakhstan [4]. The Fe-As alloy, consisting of mixed iron arsenides, obtained under optimal conditions for As recovery has been characterised using EPMA (Electron probe microanalysis), QEMSCAN (Quantitative Evaluation of Minerals by Scanning Electron Microscopy), powder X-ray and neutron diffraction [3]. Thermal behaviour of the alloy has also been studied [5].

Removal of As from gold-arsenic-bearing refractory materials into solid waste, such as slag and speiss, is known as a potentially safe disposal option. It allows one to avoid the transition of As to the gas phase, and to avoid subsequent troublesome gas collection and solidification/stabilisation processes. Calcium sulfite and sulfate were added to suppress sublimation of As during smelting of refractory gold-arsenic bearing concentrates [6], this incorporated As into the slag. However, the process consumed large quantities of reagents. In addition, a certain amount of As still passed into the gas phase, and the leaching behaviour of the slag is unknown. There has been an attempt to investigate the leaching behaviour of the slag, obtained by incorporation of arsenates into typical waste slags of lead and copper smelting [7]. The product of ferric arsenate and lead furnace slag, containing 20.7% As, leached out 0.01% of the As after 8000 hours of static water leaching. A

product, containing 23.5% As, that was obtained by slagging of copper slag and calcium arsenate, leached a similar amount of As. In another study, As was converted into speiss in the course of gold recovery from double refractory gold-arsenic-bearing concentrates [8] by melting with metallic iron and lead sulfate. Once again, the leaching behaviour of the obtained speiss material has not been studied.

Furthermore, to date no single study has reported on the leaching behaviour of iron-arsenic alloys with high As content (up to 40%), such as those obtained from DRM of the Bakyrchik concentrates. The purpose of this study is to characterize the toxicity and stability of the iron-arsenic alloy. The objectives of the research are:

- Evaluate toxicity of the iron-arsenic alloy.
- Examine the stability of the iron-arsenic alloy.
- Study long-term leaching behaviour of the iron-arsenic alloy under various pH conditions.

Understanding the long-term stability of iron-arsenic alloy is crucial for prediction of the long-term behaviour of As in such alloys, and in weathered products following its disposal, as well as to evaluate its pollution load and associated environmental hazards.

### Experimental

The toxicity of the iron-arsenic alloy has been assessed by the «Toxicity Characteristics Leaching Procedure» (TCLP) – the United States Environmental Protection Agency (USEPA) regulatory method for classifying wastes as hazardous based on toxicity [9]. The gold extraction process, previously employed at Bakyrchik, removed As in the form of calcium arsenate dust. The toxicity of the calcium arsenate dust from the Bakyrchik minesite was also evaluated to compare to that of iron-arsenic alloy.

The stability of the iron-arsenic alloy has been studied by conducting short-term and long-term leaching tests. The short-term leaching test resembled a standard «Synthetic Precipitation Leaching Procedure» [10]. The major difference between the SPLP and the TCLP tests is in the extraction fluid used.

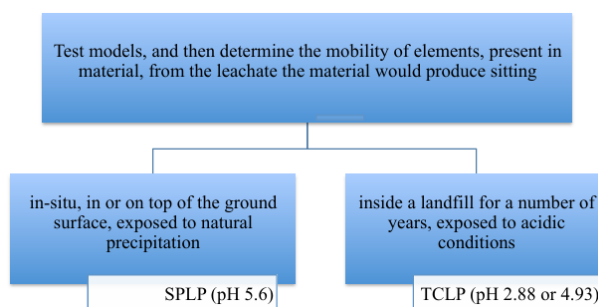


Figure 1 – Difference between SPLP and TCLP tests

The SPLP extraction fluid reflects the acidity of precipitation in the area where the sample is located. Therefore, in the short-term leaching test, pH 5 has been chosen to simulate a pH typical of normal rainwater. Short-term SPLP-like leaching test comprised five consecutive contacts of Fe-As alloy with deionized water at pH 5 and 22°C in polycarbonate Erlenmeyer flasks, stoppered to prevent evaporation (Table 1). After each 24-hour contact, the slurry was filtered using a glass microfibre filter. The filtrate was analysed for iron and arsenic, while the wet solid was placed in contact again with fresh extraction water and so on. The regulated temperatures, and the shaking process for the short-term leaching test, as well as for long-term leaching test were controlled using an Adolf Kuhner ISF-1-W incubator-shaker. The pH adjustment was controlled using analytical grade 0.01M NH<sub>4</sub>OH and 0.01M HNO<sub>3</sub> solutions. Deionised water (DW) with the resistivity of >18 MΩ/cm was used throughout the entire experiment.

Since simple conduct of commonly accepted leaching tests (such as TCLP and SPLP) is insufficient for realistic assessment of environmental impact of the waste material, the long-term leaching behaviour of the Fe-As alloy in environmentally-relevant pH conditions has also been studied (Figure 2).

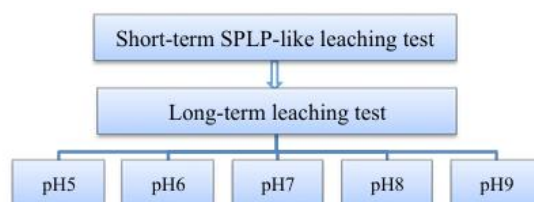


Figure 2 – Experimental design

Wet solid residue after the five-stage short-term experiment was used to evaluate long-term stability and leaching behaviour of the iron-arsenic alloy. A mixture with a S:L ratio of 1:40 was equilibrated for 12 weeks. Over this time a 2 mL aliquot of the slurry was collected daily using 0.1 µm syringe filters in the first week, and weekly using 0.02 µm syringe filters in the following 11 weeks. The experiment was conducted without the addition of any fresh extractant. The pH was adjusted many times a day as required. At the end of the test the leachates were filtered and the residues were washed three times with the same volume of water as the volume of the filtrates. The temperature of the water used for washing was the same as the leaching temperature. The solid residues were then allowed to dry for a week at room temperature, and then analysed by XRD.

Details of the tests are presented in Table 1. Fe and As content in aliquots was determined using ICP-MS.

Table 1 – Details of the leaching tests

Parameter	TCLP-like test	SPLP-like test	Long-term test
Particle size, mm	< 2	< 2	< 2
Agitation speed, rpm	30	30	30
Extractant (pH)	DW (pH2.9)	DW (pH5)	DW, pH5-9
Temperature, °C	22	22	22, 50, 75
Extraction time	five 24-hour-contacts		up to 16 weeks
Solid:liquid (S:L) ratio, g:ml	1:20	1:20	1:40
Filter pore size, µm	0.7	0.7	0.1 and 0.02

## Results

The result of the TCLP-like test is shown in Table 2. The leachability of the iron-arsenic alloy was compared to that of calcium arsenate dust.

Table 2 – TCLP-like (pH 2.88) test results for Fe-As alloy and calcium arsenate dust

Sample (As content)	Leached As, ppm ± SD	Multiple of TC limit
Iron-arsenic alloy (up to 30% As)	24.45±0.06	4.8
Calcium arsenate dust (3% As)	888.15±0.08	177.6

Results of the short-term SPLP-like test of the Fe-As alloy are shown in Figure. 3. The potential long-term leachability of As from iron-arsenic alloy is depicted in Figure. 4. The long-term leaching test was run for 84 days.

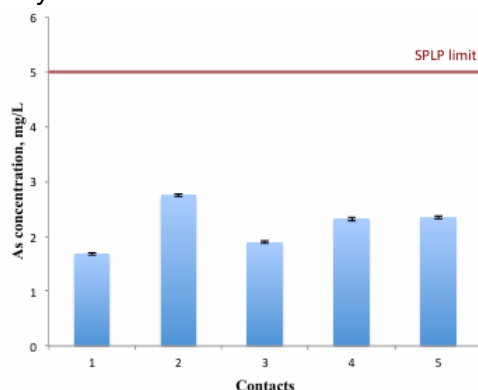


Figure 3 – Short-term SPLP-like test of Fe-As alloy over successive contacts

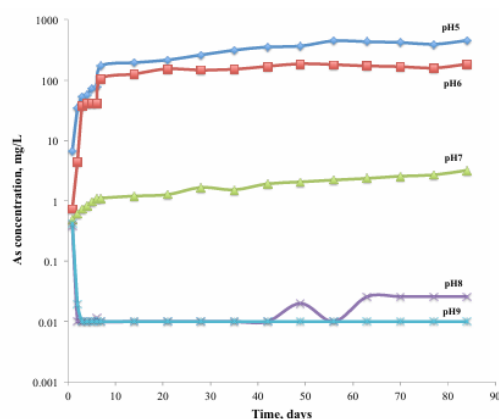


Figure 4 – Leaching of arsenic from Fe-As alloy as a function of time and pH at 22 °C

## Discussion

The TCLP-like test held at pH 2.9 leached 24.45 mg/L As from the Fe-As alloy, exceeding As toxicity characteristic limit of 5 mg/L. Based on the TCLP-like test, the alloy shall be deemed toxic. Some countries have less stringent requirements for disposal of As-containing residues, taking into account the fact that it is not xenobiotic to the environment, and does not bioaccumulate in the body. According to the UK's Waste Management Paper of the Department of the Environment (1980), the regulatory limit for As-bearing wastes co-deposited in a landfill is 10 mg/L of As in leachate, whereas in hydrologically secure sites, which excludes any risk for groundwater quality, it is 25 mg/L [11]. From this less emotive and reasonably pragmatic perspective Fe-As alloy would have passed the required limit for disposal of waste in secure to groundwater monofills.

Calcium arsenate dust released 888 mg/L As. This is close to the As concentrations (900-1400 mg/L) reported for calcium arsenate compounds subjected to the TCLP [12]. The Fe-As alloy appears to be considerably less soluble, releasing almost 40 times less As compared to the calcium arsenate dust. It is clear that Fe-As alloy is by far a better solution for safe disposal of As than previous option – calcium arsenate dust.

The applicability of the TCLP for classification of metallurgical wastes has been the subject of much controversy. Some authors have found that As levels leached by the TCLP test exceeded those leached by six months of continual column leaching [13]. The TCLP is especially inadequate when the lowest solubility of the waste constituent matches the pH of the TCLP leaching solution. It has been suggested that sulphate would be a more suitable leaching agent for As-containing non-ferrous wastes [14]. Hence, for mineral processing and metallurgical wastes, which are generally disposed of in monofills with neutral to alkaline pHs, the SPLP seems to be more appropriate. According to the USEPA "The SPLP is a method of choice when evaluating fate and transport of metals in a properly engineered waste land disposal facility from which municipal solid is excluded". It can be seen from the Figure 3, that the Fe-As alloy leached between 1.67 to 2.75 mg/L As during the test. This is less than the SPLP regulatory limit of 5 mg/L for As [15]. The SPLP solution pH represented the acidity of meteoric precipitation in the Bakyrchik area. According to the routine industrial monitoring results the pH of surface and ground waters in the vicinity of the Bakyrchik Mine site is circumneutral [16], suggesting that compared to the TCLP-like test the SPLP-like test is more representative of the conditions, to which the As residue would be exposed. The results of the SPLP-like test confirms that the Fe-As alloy is relatively stable in the short term under natural weathering conditions.

According to the long-term leaching test (Figure 4), Fe-As alloy solubility appears to be very sensitive to the pH of the extractant: the lower the pH of the solution the greater the leaching of As. The concentration of As in solution increases significantly in the first week of the test at pH 5, pH 6 and pH 7. In contrast, at pH 8 and 9 it decreases, with As concentration below detection limit in most of the measured aliquots. The highest leachability of As is observed at pH 5, ranging from an initial value of 6.71 to 447 mg/L upon reaching equilibrium. The As concentration at pH 7 increased moderately in the first week from 0.49 to 1.11 mg/L, then continued with a very small but steady increase of As up to 3.20 mg/L determined on the final day of the test. The final concentration of As

for each run plotted as a function of pH is shown in Figure 5, where the strong dependence of equilibrium concentration on pH is clear.

Information regarding the stability of iron-arsenic alloys is scarce. The only relevant study is of Harris and Monette [17] on weathering of speisses (chemically iron arsenides) in laboratory conditions. In their experiment, some speiss samples remained relatively unaffected, but others significantly decomposed upon weathering. The complete breakdown of speiss was accompanied by steady increase of dissolved As content in leachate. However, after reaching a certain value As concentration in leachate started to decline gradually. The product of complete breakdown of speiss was ferrihydrite. Decrease of As content in leachate was attributed to adsorption of As by ferrihydrite. It is known that over time ferrihydrite converts to goethite, hematite or the mix of both [18]. However, if ferrihydrite contains adsorbed species, like arsenates [19,20], the transformation can be substantially inhibited. From these considerations, it appears that speiss materials can be buried and allowed to weather in monofills with impermeable liner.

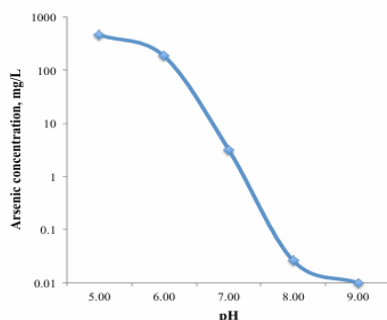


Figure 5 – Solubility of Fe-As alloy as a function of pH at 22 °C

In addition, The Fe-As alloy is a solid dense monolithic material, with less free surface area for permeability and leaching compared to amorphous precipitates and dust wastes. Hence, given the monolithic structure of the Fe-As alloy, and the fact that mineral processing wastes are not subjected to crushing prior to their disposal, if we had done a leaching test designed for assessment of monolithic wastes (such as Dutch Tank Leach Test NEN 7345 [21]), which do not require the particle size reduction employed here, much less As would have been released from the alloy.

### Conclusion

The results of this study suggests that the iron-arsenic alloy, if disposed of in neutral to alkaline conditions, would be a good medium for As immobilisation. The Fe-As alloy is a compact monolithic, dense material, which can accommodate up to 40% As. Furthermore, it doesn't require any troublesome and/or costly stabilisation/solidification pre-treatments prior to disposal. These advantages, together the high degree of gold extraction, make iron-arsenic alloy an attractive waste-form for removal of As from double refractory Au-As-bearing concentrates.

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### **ТЕМІР-МЫШЬЯК ҚОРЫТПАСЫНЫҢ УЫТТЫЛЫҒЫ ЖӘНЕ ТҰРАҚТЫЛЫҒЫ**

*Темір-мышьяк қорытпасы алтынды табанды алтын-мышьякты концентраттардан бөліп алудың жаңа технологиясы – тікелей тотықсыздандыра балқытудың қалдық өнімі болып табылады. Бұл мақалада сулы ерітінділердегі Fe-As қорытпасының уыттылығы мен тұрақтылығын зерттеу нәтижелері берілген. Fe-As қорытпасының әлеуетті уыттылығын бағалау үшін «Шаймалау арқылы уыттылықты анықтау процедурасы» пайдаланылды. Сынақ нәтижелері қорытпаның қышқыл ортада (рН 2,88) тұрақсыз екенін және уыттылықтың шекті деңгейінен төрт еседен жоғары концентрацияларда As шайылатынын көрсетті. Салыстыру үшін кальций арсенаты шаңының уыттылығы да осы әдіспен тексерілді. Шаңның темір-мышьяк қорытпасынан 40 есе көп мышьяк бөлетіні анықталды. Fe-As қатты қорытпасының тұрақтылығын бағалау үшін «Жасанды (атмосфералық) жауын-шашынмен шаймалау процедурасы» қолданылды. Бұл кезде филтраттардағы As концентрациясы стандартты шектерден аспады, яғни атмосфералық жауын-шашынға тән рН 5 ерітінділерде Fe-As қорытпасы салыстырмалы түрде тұрақты болып келеді. 22°С температурада рН 5- 9 аралығында ұзақ уақыт шаймалау сынақтары Fe-As қорытпасының қышқыл рН жағдайларға өте сезімтал екенін көрсетеді. Шаймалау кезінде мышьяқтың қорытпадан сулы ерітіндіге өтуі рН5 және рН6 жағдайында жоғары болды, бұл қорытпаның қоршаған ортада ұзақ уақыт бойы жауын-шашынның және, әлбетте, қышқыл*

жаңбырдың әсеріне ұшыраса құрамындағы мышьяқтың ерітіндіге шайылуы мүмкін екендігін білдіреді. Дегенмен, жүргізілген эксперименттер қорытпаның монополигондарға тән бейтарап және сілтілі рН жағдайында ұзақ уақыт бойы тұрақтылығын сақтайтынын дәлелдеді.

**Түйін сөздер:** алтынды бөліп алу; Fe-As қорытпасы; мышьяк; уыттылық, тұрақтылық.

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## ТОКСИЧНОСТЬ И СТАБИЛЬНОСТЬ ЖЕЛЕЗО-МЫШЬЯКОВОГО СПЛАВА

Железо-мышьяковый сплав является отходом новой технологии извлечения золота из упорных золото-мышьяковых концентратов – прямой восстановительной плавки. В данной статье представлены результаты исследования токсичности и стабильности Fe-As сплава в водных растворах. «Процедура определения характеристик токсичности выщелачиванием» (ПХТВ) была использована для оценки потенциальной токсичности твердого Fe-As сплава. Результаты ПХТВ-подобного теста позволяют предположить, что сплав нестабилен в кислой среде (pH 2,88) и выделяет As в концентрациях, превышающих пороговый уровень токсичности более чем в четыре раза. Для сравнения, также, была оценена токсичность пыли арсената кальция. Было обнаружено, что пыль выделяет в 40 раз больше мышьяка, чем железо-мышьяковый сплав. Для оценки стабильности твердого Fe-As сплава была применена «Процедура выщелачивания искусственными (атмосферными) осадками» (ПВИО). Концентрация As в фильтрах ПВИО-подобного теста находилась в пределах нормы, что свидетельствует об относительной стабильности Fe-As сплава в растворах с pH 5, подобных атмосферным осадкам. Длительные испытания на выщелачивание в экологически безопасном диапазоне pH от 5 до 9 при температуре 22 оС показывают, что Fe-As сплава характеризуется высокой чувствительностью к кислым условиям среды. Растворимость мышьяка была высокой при pH5 и pH6, что позволяет предположить, что в долгосрочной перспективе сплав может проявлять подвижность в окружающей среде при воздействии атмосферных осадков и, конечно же, кислотных дождей. Однако в нейтральных и щелочных условиях pH, типичных для монополигонов, сплав сохраняет стабильность в течение длительного времени.

**Ключевые слова:** извлечение золота; Fe-As сплав; мышьяк; токсичность, стабильность.

### Авторлар туралы мәліметтер

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