



# Removal of impurities from crude lead with high impurities by vacuum distillation and its analysis



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## ABSTRACT

A novel technology was developed to remove impurities from crude lead with high impurities by vacuum distillation. Refined lead (purity over 99%) was achieved through vacuum distillation using crude lead (92.88%) with high impurities as input material under a dynamic vacuum of 5–15 Pa. The results show that vacuum distillation should be taken to obtain lead from crude lead matrix, in which low-volatile impurities namely Cu, Sn, Ag and partial Sb are concentrated in residue at the first fraction stage of 1373 K. Volatilized lead containing high-volatile impurities such as Zn and As is distilled to remove these impurities at the second fraction stage of 973 K. But the property of impurity Bi is similar to lead that can not be separated from crude lead. The separation coefficient ( $\beta_i$ ) and the activity coefficient ( $\gamma_i$ ) of impurity elements are calculated according to the experiments to fill the inadequate data of the thermodynamics.

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## 1. Introduction

Crude lead produced by the traditional pyrometallurgy processes usually needs to refine that can be extensively used in lead-acid battery industry, industrial chemicals, anticorrosive materials in the field of metallurgical devices, protective materials in the area of the devices of atomic industry and X-ray instrument, etc. Fire refining process and electrolytic process are normally applied for refining of crude lead. At present, fire refining has been used in more lead smelters, accounting for about 70% of the world's refined lead production, only some smelters use electrolytic process in Canada, Peru, Japan and China [1–4]. The refining of crude lead is aimed to remove the impurities, obtaining the refined lead (purity over 99%), and to recover copper, silver and bismuth and other precious metals in crude lead. Cu, Sn, Ag, Zn, As, Sb and Bi are the common impurities in crude lead. Although both fire refining process and electrolytic process could obtain naturally the requirements to the quality of refined lead, there are some serious problems in conventional refining processes of crude lead. The fire refining process has some disadvantages such as procedure

complex, lower direct rate of lead, bad state of operation, evident environmental pollution, etc [5–7]. The electrolytic process has some disadvantages such as long production period, large investment, high energy consumption, low economic profits, etc [8–10]. Vacuum metallurgy has many advantages such as short flow, low pollution, low energy consumption, etc [11–13] and can eliminate the disadvantages of traditional refining processes. Vacuum distillation has been studied and successfully used in separation of various elements from binary alloys, crude nickel and crude indium [14–20]. The investigation of the refining of crude lead by vacuum distillation has not been reported.

Herein, this paper investigated systemically the performance for the refining of crude lead with high impurities via vacuum distillation to provide a simple, green, efficient and referential way for the removal of Cu, Sn, Ag, Zn, As and Sb from crude lead.

## 2. Method

### 2.1. Saturated vapor pressure

The impurities can be removed from crude lead by vacuum distillation, mainly depending on the different property of impurity elements contained when vaporizing and condensing. The difference in saturated vapor pressure of impurities in the pure state is the basic principle of crude metal vacuum distillation at different temperatures. According to Van Laar Equation [13], the relationship

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between saturated vapor pressure of impurities and temperature can be shown in Eq. (1), and the evaporation constants  $A$ ,  $B$ ,  $C$  and  $D$  for different impurities are shown in Table 1 [13].

$$\lg p^* = AT^{-1} + B\lg T + CT + D \quad (1)$$

where  $p^*$  is the saturated vapor pressure of pure substances in Pascal, Pa;  $T$  is the absolute temperature in Kelvin, K.

Using the values of  $A$ ,  $B$ ,  $C$  and  $D$  in Table 1, the saturated vapor pressure can be calculated from Eq. (1), as shown in Fig. 1. The comparison of the saturated vapor pressure of the impurities in crude lead at the same temperature could be used as a rough guide for determining which should exhibit preferential volatilization. One with a higher vapor pressure can be expected to volatilize preferentially. Others with lower vapor pressure can be remained in residual after vacuum distillation.

Fig. 1 showed that the saturated vapor pressures of As, Zn and Sb were much higher than that of Pb at 873–1073 K. And at 823 K, As was started to sublime. These mean As, Zn and Sb are prior to volatilize into vapor phase from crude lead during distillation. On the contrary, the saturated vapor pressures of Cu, Sn and Ag were much lower than that of Pb at 1273–1523 K, which means Cu, Sn and Ag would concentrate in residue and Pb would volatilize. So a two-stage vacuum distillation process should be adopted to obtain the refined lead.

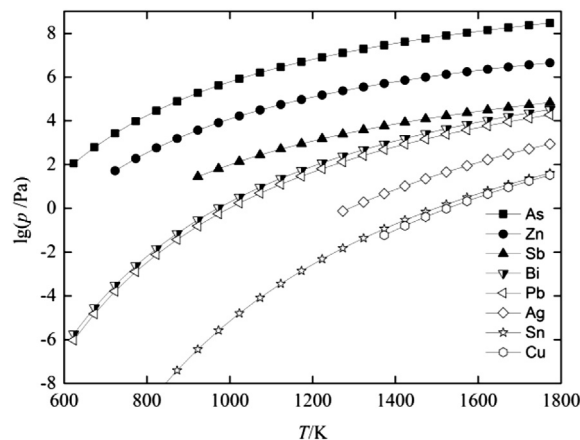
## 2.2. Experimental

The schematic details of the vacuum distillation furnace were shown in Fig. 2 describing the refining process of crude lead using stainless steel condenser comprising of water cooled tubes to collect the distillate. The evaporator was made up of an isostatic fine grain high density graphite crucible in the cylindrical shape of dimensions (50 mm OD  $\times$  40 mm ID  $\times$  80 mm Ht). The crude lead with high impurities (1 kg) to be distilled was kept in the graphite crucible and placed inside the distillation section of the vacuum furnace, which was then evacuated to a dynamic vacuum of 5–15 Pa by means of appropriate combination of mechanism and diffusion pump. Furnace pressure was measured using a stand McLeod Gauge which could be accurate to within 1 pct at pressures below 300 Pa. The experiment was conducted in two stages. In the first stage, namely the high temperature fraction stage, the distillation experiment was conducted at a comparatively higher soaking temperature of 1373 K to remove low-volatile impurities. As the temperature in the furnace reduced to room temperature, the lead in the condenser was used in the second stage, i.e. the low temperature fraction stage, the soaking distillation temperature was maintained at 973 K to remove high-volatile impurities. After each distillation, the samples for analysis were taken from the condensate and the residue by random selection and mixing at five to six places in order to obtain an average impurity concentration. The flame atomic absorption spectrometry (FAAS) and chemical

**Table 1**  
Values of evaporation constants of  $A$ ,  $B$ ,  $C$  and  $D$  for different elements [13].

Element	$A$	$B$	$C$	$D$	$T/K$
Pb	-10130	-0.985	—	13.28	600–2013
Cu	-17520	-1.21	—	15.33	1356–2840
Sn	-15500	—	—	10.355	505–2473
Ag	-14400	-0.85	—	13.825	1233–2468
Zn	-6620	-1.255	—	14.465	692–1773
As	-6160	—	—	11.945	873–1773
Sb	-6500	—	—	8.495	904–1860
Bi	-10400	-1.26	—	14.47	544–1837

— stands for the value that does not exist.



**Fig. 1.**  $\lg p$ - $T$  diagram of main impurities in crude lead with each element being in the pure state.

analysis were applied to the analysis of input material and the distilled lead.

## 3. Results and discussion

### 3.1. Results of experimental

The vacuum distillation results for common impurities in crude lead in high concentration have been studied. The impurity content in the raw material and the vacuum distilled lead were given in Table 2. The values in ppm showed the complied average analysis of a large number of samples of around 20 experiments. Impurities C and N were not shown in the analysis results, since the ferrous elements including C and N could not be analyzed precisely by FAAS and chemical analysis and C and N were not usually existed in the crude lead with high impurities.

It was observed that most of the impurities, except Bi, had been removed in varying degrees. Low-volatile impurities such as Cu, Sn and Ag were easily removed, and the removal rate of Cu was very close to 100% during first fraction stage. The impurities such as Zn and As were found difficult to be removed from crude lead matrix and volatilized into vapor phase along with lead at the high temperature. The reason was the differences of the vapor pressures. In addition, first distillation of high-volatile Zn and As followed by distillation of lead at the higher temperature of 1373 K was carried out in preliminary experiment, the removal results of Zn and As were given in Table 3. It showed that high-volatile impurities such as Zn and As have not been removed from crude matrix at the lower temperature of 973 K. So the lead in the condenser obtained from the process of high temperature distillation was distilled again at the low temperature to remove Zn and As. But the high-volatile impurity namely Sb was partially removed and concentrated in residue during first fractional distillation experiment. This phenomenon was contrary to the theory of saturated vapor pressure in Fig. 1. This can be attributed to the effect of the separation coefficient ( $\beta_i$ ) and activity coefficient ( $\gamma_i$ ) of Sb that will be calculated later on. High-volatile impurities such as Zn and As were considerably removed during second fraction stage. The impurity Bi was hardly separated at both the high temperature distillation and the low temperature distillation. This can be put down to the similar property of Bi and Pb. Fig. 1 revealed that the pressure of Bi was close to that of Pb at any temperature. Furthermore, the separation coefficient ( $\beta_i$ ) and activity coefficient ( $\gamma_i$ ) of Bi were calculated hereafter to account for this argument.

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