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Acid dissolution of neodymium magnet Nd-Fe-B in different conditions

The separation of rare-earth elements (REE) from a neodymium magnet has been widely studied last year. During the research it was identified that the waste of computer hard disk contains 25.41 % neodymium, 64.09 % iron, and <<1 % boron. To further isolate rare-earth metals, the magnet was acidically dissolved in open and closed systems. In both methods of dissolution, concentrated nitric acid was used. The difference between these methods is the conditions of dissolution of magnet. The magnet was dissolved in a microwave sample preparation system at different temperatures and pressures in a closed system. In the open system, the acid dissolution of the magnet is conducted at room temperature. 0.2 g of the neodymium magnet sample was taken under two conditions, and the dissolution process in the closed system lasted 1 hour, and in the open system 30-40 minutes. The open system is a non-laborious, simple, and cheap method of dissolving the magnet by comparing both systems. Therefore, an open sample preparation system is used for further work. To remove the iron in the magnet, oxalic acid was used and REEs are precipitated as oxalates under both conditions. According to the result of the Inductively coupled plasma mass spectrometry (ICP-MS) method, it was identified that the neodymium and iron contents in the precipitate are 24.66 % and 0.06 %, respectively. This shows that the iron has almost completely passed to the filtrate.

Keywords: acid dissolution, neodymium magnet, rare-earth metals, neodymium, praseodymium, dysprosium, open system, microwave sample preparation system, ICP-MS analysis.

Introduction

Currently, rare-earth metals are widely used in electronic devices and equipment and their production is constantly growing. For example, neodymium magnet NdFeB from computer hard disk HDD, LiH lithium hydride batteries, fluorescent lamps, etc. At this rate of growth of electronic equipment with REE, the amount of electronic waste will increase. As a result, every year a large amount of waste from REE is released into the environment. According to the United Nations Global Monitoring Organization, in 2019, the global volume of electronic wastes reached 53.6 million tons [1]. This creates serious environmental and economic problems. The separation of metals and REE from electronic wastes reduces environmental problems and helps the development of waste-free production. At the same time, it is important to effectively separate REE. The dissolution of neodymium magnets and the separation of rare-earth metals from them are considered in the following works [2-6].

In [7], the demagnetization of a neodymium magnet in the temperature range of 300-400 °C with different holding times in a muffle furnace is considered. The optimal time is 30 minutes at a temperature of 350˚C. The dissolution is carried out with organic acids, such as malic and citric. The best values of the parameters were observed at a temperature of 90 °C at a concentration of 1.0 M and a ratio of 1: 20 for both organic acids. The content of elements was determined by X-ray fluorescence analysis in the calcined and non-calcined form of magnetic powder, which is 66.69 % and 58.5 % iron, 25.19 % and 32.36 % neodymium, respectively.

In [8], rare-earth metals are deposited as oxalates. After thermal decomposition of oxalates, rare-earth metal oxides were obtained. Neodymium was separated by electrochemical method.

For the acid dissolution of the magnet, different acids and mixtures, such as nitric, hydrochloric, sulfuric, hydrogen fluoride, “Aqua regia” are used. In this work, concentrated nitric acid was applied. In concentrated HNO_3 (in small volumes), the magnet alloy was completely dissolved.

The main purpose of this work is to study and determine the optimal method for dissolving a neodymium magnet under various conditions and effectively removing iron — a macronutrient in the magnet. Acid dissolution is used to separate REE from the magnet and, also, it was conducted a comparative analysis with other works.

Experimental

Reagents

The following reagents were used for the experiment: nitric acid HNO_3 (67 %), oxalic acid (purity, 99.6 %), neodymium magnet Nd-Fe-B taken from a computer hard disk.

Apparatus

The following instruments were applied for sample preparation and analysis: ICP-MS inductively coupled plasma mass spectrometry (Agilent 7500a, USA), muffle furnace (SNOL 7,2/1300, Lithuania), analytical scales (Acculab, ALC-210.4, Germany), microwave sample preparation system (Speedwave four “Berghof”, Germany), X-Ray Diffractometer (DRON -4-07, Russia).

Material preparation. Demagnetization

Neodymium magnet Nd-Fe-B from the computer hard disk is provided by the company “Taza Alemdik” LLP (Kazakhstan), which is engaged in the disposal of electronic waste. Several stages of sample preparation were carried out:

1) Removing the magnet from the hard disk drive (HDD);
2) Cleaning the nickel shell of the magnet;
3) Demagnetization of the sample in the muffle furnace;
4) Grinding the sample to a homogeneous mass.

After removing the nickel shell, the sample is ready for demagnetization. Demagnetization was carried out by heating at 750 °C in a muffle furnace (SNOL 7,2/1300, Lithuania) for 1 hour. The demagnetized sample is ground to a homogeneous mass.

Acid dissolution of a neodymium magnet in a closed system

The sample was decomposed in a closed system in an autoclave (Speedwave four “Berghof”, Germany). Concentrated nitric acid (acid concentration of 15 M) is added to the sample. The decomposition process in HNO_3 must be carried out until the ending of the NO_2 release. Then the samples were placed in an autoclave at different temperatures and pressures (at 100 °C, $p = 3$ MPa, $\tau = 10$ min; 160 °C, $p = 2$ MPa, $\tau = 10$ min and cooled at 50 °C, $p = 2$ MPa, $\tau = 10$ min). After dissolution, the samples were cooled at room temperature and diluted with distilled water, and semi-quantitative analysis was performed using ICP-MS to determine the chemical composition of the sample. The total time for dissolving the magnet in the autoclave is 1 hour.

Acid dissolution of a neodymium magnet in an opened system

The 0.2 g magnet sample was dissolved in concentrated nitric acid at room temperature. When the magnet is dissolved, NO_2 is released, so the process must be carried out until the gas is released. After that, the magnet solution is diluted with distilled water (NO is released). Semi-quantitative analysis was performed using the ICP-MS method. The total time to dissolve the magnet is 30-40 minutes.

Precipitation of rare-earth metals and iron removal

In both methods, a saturated solution of oxalic acid was added to the resulting neodymium magnet solution. REE is deposited as oxalates. The precipitate was filtered through a paper filter “blue ribbon filter” and after 2 hours the filtrate was checked for the presence of Fe^{3+}, Fe^{2+} cations. Both forms of iron ions were present in the filtrate. The precipitate was then calcined in a platinum crucible at 650 °C for 40 minutes. The resulting precipitate was dissolved in concentrated nitric acid, and a pale lilac solution was obtained. The composition of the resulting solution was analyzed by inductively coupled plasma mass spectrometry. The operation parameters of the ICP-MS 7500 are as follows: the carrier gas flow rate is 0.82 L/min, the plasma gas flow rate is 0.17 L/min, the signal integration time is 0.1 s, and the high-frequency signal power is 1450 W. Figure 1 shows the scheme of dissolution in the open and closed systems and the removal of iron in a neodymium magnet sample.

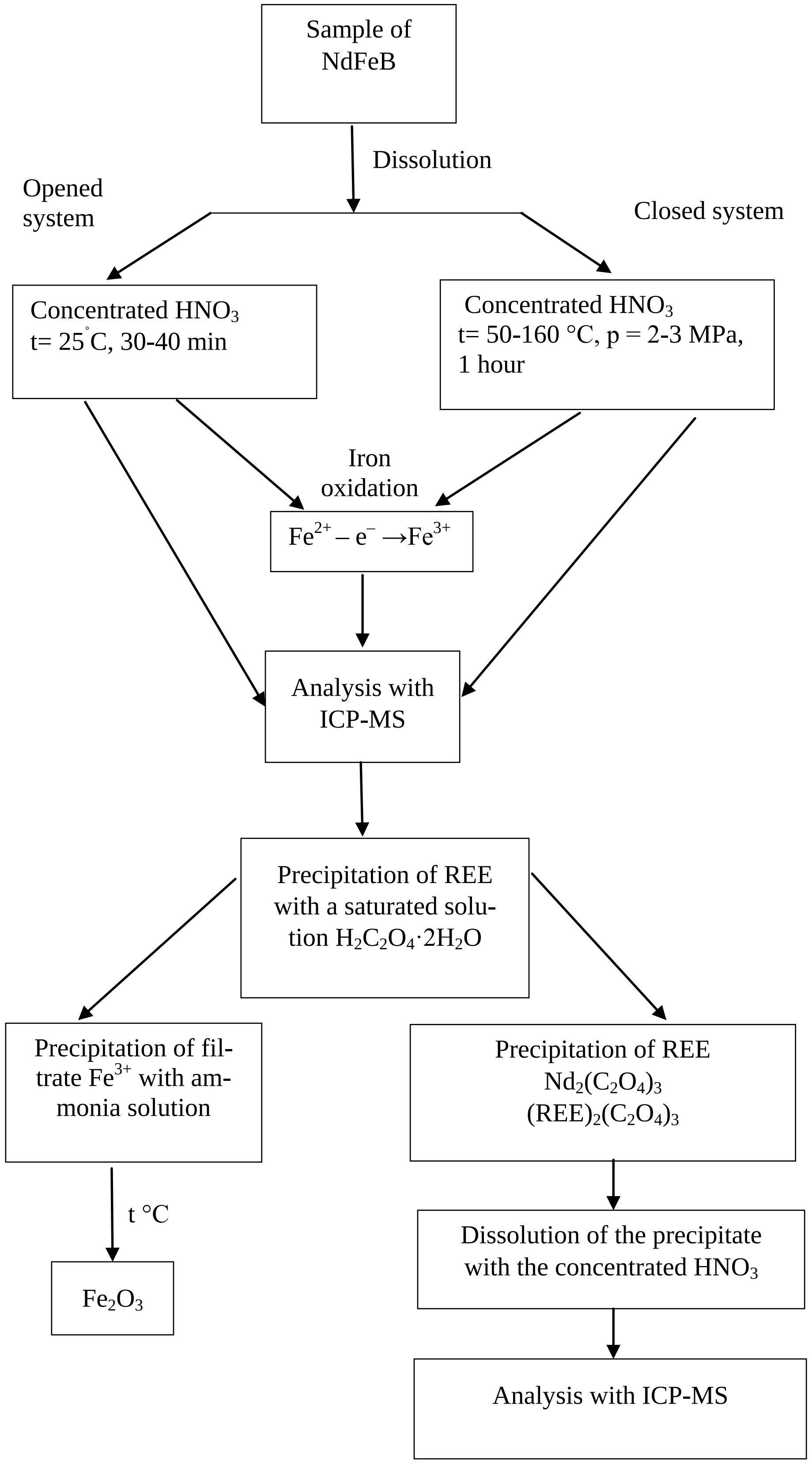

Figure 1. Scheme of acid dissolution and removal of iron in a neodymium magnet sample

Results and Discussion

In this paper, the dissolution of a neodymium magnet have been carried out in an open and closed system. The neodymium magnet was dissolved in nitric acid (Eq. (1)). Then a semi-quantitative analysis was performed using the ICP-MS method. The result showed that for further analysis is necessary to select elements with concentration values are significantly higher than those of other elements: iron, neodymium, praseodymium, dysprosium, and nickel. The concentration of boron is insignificant, but the neodymium magnet contains boron, and therefore, in the literature the formula of the neodymium magnet is written in the form of NdFeB.

Table 1

Advantages and disadvantages of open and closed dissolution systems

Open system of dissolution		Closed system of dissolution	
Advantages	Disadvantages	Advantages	Disadvantages
– simple; – safety; – not time-consuming; – non-energy-intensive; – cheap	– non-modern	– modern	– high temperature; – energy-intensive; – complex equipment; – explosive; – limited sample weight; – time-consuming; – restrictions on the choice of acid for dissolution

Table 1 describes the comparative characteristics of open and closed dissolution systems. It is illustrated that an open system is more efficient than a closed one, since they have many advantages. Therefore, an open system for dissolving a neodymium magnet was chosen for further research. A closed system has many disadvantages and limitations, such as sample weight limitations and restrictions on the use of different acids. In a closed system, an explosion can occur at high temperatures, even at low pressures. Therefore, this method is more complex than the open system.

Nevertheless, in a closed system, the neodymium magnet decomposes faster than in an open system, because in a closed system, the neodymium magnet solution is heated in an autoclave to 160 °C. This contributes to the rapid dissolution of the magnet.

In this paper, we considered a sample of a single neodymium magnet, with an average weight of 3.86 g, and 0.2 g of the sample was taken from this magnet for analysis. The magnet contains several rare-earth metals. However, the concentration of neodymium, praseodymium, and dysprosium is much higher than that of other rare-earth metals.

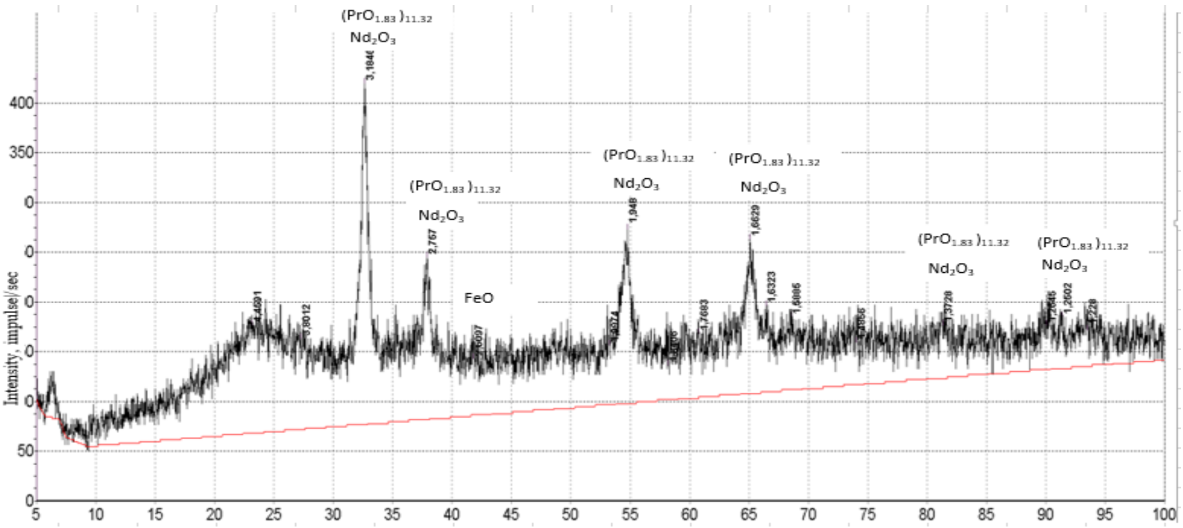

Figure 2. Composition of precipitate REE with X-Ray phase analysis

Table 2 demonstrates the comparative results of the analysis of the precipitate of REE after dissolution with nitric acid and the addition of oxalic acid (element concentrations in %). As can be seen from Table 2, the concentration of iron after acid dissolution was 2 times greater than that of neodymium. Therefore, it is necessary to remove the Fe in the sample. It is important to note the possibility of separating REE from iron ions by precipitation in the form of oxalates (Eq. (2)), and this helps to get rid of iron for further REE isolation. Iron (III) passes into solution, and Fe^{2+} in an acidic medium is oxidized to Fe^{3+} (Eq. (6)). Therefore, almost all of the iron passes into the filtrate. The composition of the precipitate is only 0.06 % Fe (Table 2). This proves that the iron was almost completely removed. Figure 2 indicates the content of REE sediment in X-Ray phase analysis. This shows the main composition of the precipitate of REE consists of neodymium oxide (praseodymium oxide together) and iron oxide in a small amount. Reflexes of neodymium and praseodymium oxides are the same.

Table 2

Results of the analysis of acid dissolution of neodymium magnet and REE precipitate

Element	Acid dissolution of magnet (opened and closed systems), HNO_3, %	Precipitate of REE, HNO_3, % (after removing macro- and microelements)
Nd	**25.41**	**24.66**
Pr	6.28	5.96
Dy	2.53	2.4
Fe	**64.09**	**0.06**
Ni	1.2	0
B	2.6×10^{-5}	2.6×10^{-5}
Other elements	0.5	-

It should be recalled that Table 2 illustrates the concentrations of elements after the decomposition of 0.2 g of the magnet. The analysis was conducted in an open and closed system 3 times, the table shows the average values of the concentrations of elements. The concentrations of neodymium, praseodymium, and dysprosium were almost the same in open and closed systems. After acid dissolution of the magnet, the concentration of iron was 2.5 times greater than that of neodymium. And after the addition of oxalic acid, the iron content was negligible, and nickel was also present in negligible amounts or absent. The boron content was low and the concentration was the same in both conditions. The result of the analysis with ICP-MS pointed out the concentration of REE in the precipitate which almost did not change after dissolution with nitric acid, as in the initial solution.

Table 3 shows the comparative composition of a neodymium magnet dissolved under different conditions in comparison with the works of other authors. According to the work [6], the content of the main elements (neodymium, iron, and boron) in the composition of magnet waste, three groups can be distinguished: waste with a low content of REE (REE < 20 %), waste with an average content of REE (REE about 20-30 %), and waste with a high content of REE (REE > 30 %). Table 3 designates the content of magnet waste with average concentrations of REE and it can be seen nitric acid and "aqua regia" are often used to dissolve the magnet.

Table 3

Comparative contents of main elements (%) of neodymium magnet NdFeB in this work and other data [6]

№	Condition of magnet dissolution	ω (Nd), %	ω (Fe), %	ω (B), %	References
1	A) Acid dissolution of neodymium magnet (in opened and closed systems) 67 % HNO_3	25.41	64.09	<< 1	In this work
	B) 67 % HNO_3, precipitation with $H_2C_2O_4 \cdot 2H_2O$	24.66	0.06	<<1	
2	Dissolution in "aqua regia" and 0.5M HNO_3	25.38	61.09	1.00	[9]
3	Dissolution in $HCl:HNO_3:H_2O$ (1:1:1) and precipitation with $H_2C_2O_4 \cdot 2H_2O/Na_2S \cdot 3H_2O$	26.1	63.5	0.73	[10]
4	Dissolution in 0.1M H_2SO_4 50 °C/"aqua regia", and precipitation with $H_2C_2O_4 \cdot 2H_2O$	19.40	66.30	0.96	[11]
5	Dissolution in 65 % HNO_3 at 80 °C, 72 hour; and precipitated iron (II) with 35 % H_2O_2 at 40 °C	25.95	58.16	1.00	[12]

The chemical reactions of all processes are shown below:

$$NdFeB + 2HNO_3 + 2H_2O \rightarrow Nd^{3+} + NO_3^{-} + Fe^{3+} (Fe^{2+}) + BO_3^{3-} + NO_2 + 6H^{+} \quad (1)$$

$$2Nd^{3+} + 3H_2C_2O_4 \rightarrow Nd_2(C_2O_4)_3 + 6H^{+} \quad (2)$$

$$Nd_2(C_2O_4)_3{\cdot}nH_2O \rightarrow Nd_2O_3 + 3CO_2 + 3CO + nH_2O \quad (3)$$

$$(REE)_2(C_2O_4)_3{\cdot}nH_2O \rightarrow (REE)_2O_3 + 3CO_2 + 3CO + nH_2O \quad (4)$$

here, REE — rare-earth elements

$$Fe^{2+} - e^{-} \rightarrow Fe^{3+} \quad (5)$$

Eq. (3) and (4) illustrate the decomposition of oxalates. The decomposition products were oxides of rare-earth metals and iron (II) oxide. In the initial solution of the magnet, iron Fe^{2+} is oxidized to Fe^{3+} (Eq. (5)). Most of the iron passes into the filtrate. The effective use of the filtrate can be described as follows.

For waste-free production, it is to use a filtrate separated from the REE precipitate. In the filtrate, iron ions Fe^{3+} are present in large quantities, so it is necessary to isolate iron in an effective way. For this purpose, an ion exchange reaction with an ammonia solution was carried out:

$$Fe^{3+}+3NH_4OH \rightarrow Fe(OH)_3+3NH_4^{+} \quad (6)$$

The resulting iron (III) hydroxide precipitate was subjected to thermal decomposition and iron (III) oxide was formed at 500 °C (Eq. (7)) (thermogravimetric analysis (TGA)).

$$2Fe(OH)_3 \rightarrow Fe_2O_3 + 3H_2O \quad (7)$$

The further task of this work is to effectively isolate rare-earth metals such as neodymium, dysprosium, and praseodymium from the matrix by extraction and sorption methods, and to consider the possibility of isolating other metals contained in significant amounts (iron, nickel, etc.).

Conclusions

The comparative acid dissolution of the NdFeB neodymium magnet alloy under open and closed conditions have been considered. To remove iron, REE was precipitated in the form of oxalates, in an acidic medium, iron (II) passed into iron (III). Therefore, iron in the composition of the REE precipitate is contained in a small amount, which contributes to the effective separation of REE from the magnet alloy. This method is simple and can be effectively used in production, in which a large amount of neodymium magnet can be dissolved, given the waste-free technology. The scheme of dissolving a neodymium magnet in an open system is very simple, not labor-intensive, and safer than in a closed system. It can be concluded that the open system of dissolution of neodymium magnet is effective in production for the separation of not only REE, but also other metals, such as iron, nickel, etc. Therefore, it is more effective to perform the dissolution in open conditions.

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А.Г. Исмаилова, Г.Ж. Аканова, Д.Х. Камысбаев

Әртүрлі жағдайда Nd-Fe-B неодимді магнитті қышқылдық еріту

Соңғы уақытта неодимді магниттен сирекжер элементтерін (СЖЭ) бөліп алу кеңінен зерттелуде. Қалдық магниттің құрамында 25,41 % неодим, 64,09 % темір және <<1 % бор бар. Сирекжер металдарын әрі қарай бөлу үшін магнитті ашық және жабық жүйеде қышқылдық еріту жүргізілді. Екі жағдайда даерітуге концентрлі азот қышқылы қолданылды. Екі әдістің айырмашылығы магнитті еріту жағдайларында. Жабық жүйеде магнитті еріту микротолқынды үлгі дайындау жүйесінде әртүрлі температура мен қысымда жүргізілді. Ашық жүйеде магнитті қышқылдық еріту бөлме температурасында жасалды. Екі жағдайда да 0,2 г неодимді магнит үлгісі алынды және жабық жүйедегі еріту процесі 1 сағатқа, ал ашық жүйеде 30-40 минутқа созылды. Жабық жүйемен салыстырғанда ашық жүйе көп еңбекті қажет етпейтін, қарапайым және арзан әдіс болып табылады. Сондықтан, алдағы жұмыстарға ашық жүйеде үлгі дайындау қолданылады. Магниттің құрамындағы темірді жою үшін қымыздық қышқылы қолданылды және екі жағдайда да СЖЭ оксалат күйінде тұнбаға түсірілді. ICP-MS әдісінің нәтижесі бойынша, тұнбаның құрамындағы неодим мен темір сәйкесінше 24.66 % және 0,06 % болды. Бұл темірдің толықтай дерлік фильтратқа өткендігін көрсетеді.

Кілт сөздер: қышқылдық еріту, неодимді магнит, сирекжер металдары, неодим, празеодим, диспрозий, ашық жүйе, микротолқынды үлгі дайындау жүйесі, ICP-MS талдауы.

А.Г. Исмаилова, Г.Ж. Аканова, Д.Х. Камысбаев

Кислотное растворение неодимового магнита Nd-Fe-B в различных условиях

В последнее время выделение редкоземельных элементов (РЗЭ) из неодимового магнита широко исследуется. Авторами статьи показано, что отход магнита содержит 25,41 % неодима, 64,09 % железа и <<1 % бора. Для дальнейшего выделения редкоземельных металлов проведено кислотное растворение магнита в открытой и закрытой системах. В обоих способах растворения использована концентрированная азотная кислота. Разница между этими методами заключается в условиях проведения растворения магнита. В закрытой системе растворение магнита проводили в микроволновой системе пробоподготовки при разных температурах и давлениях, в открытой системе — при комнатной температуре. В обоих условиях были взято 0,2 г образца неодимового магнита, и процесс растворения в закрытой системе длился 1 ч, а в открытой системе — 30-40 мин. По сравнению с закрытой системой, открытая система является нетрудоемким, простым и дешевым методом для растворения магнита. Поэтому для дальнейшей работы выбрана открытая система пробоподготовки. Для удаления железа в составе магнита применяли щавелевую кислоту и осаждали РЗЭ в виде оксалатов в обоих условиях. Результаты использования метода ICP-MS показали, что после осаждения содержание неодима и железа составляет 24,66 и 0,06 % соответственно. Это указывает на то, что железо почти полностью перешло в фильтрат.

Ключевые слова: кислотное растворение, неодимовый магнит, редкоземельные металлы, неодим, празеодим, диспрозий, открытая система, микроволновая система пробоподготовки, ICP-MS анализ.

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