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Application of electrodialysis method for high purity metal rhenium obtaining

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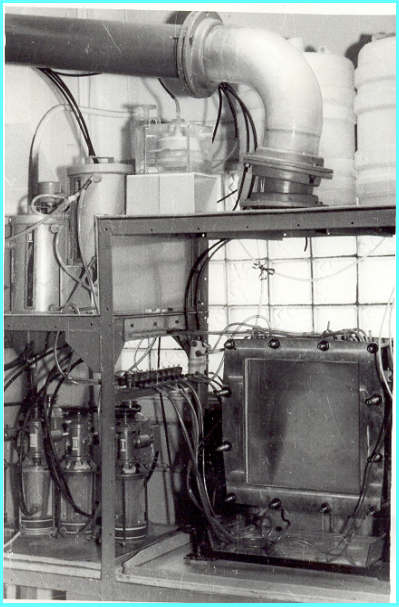
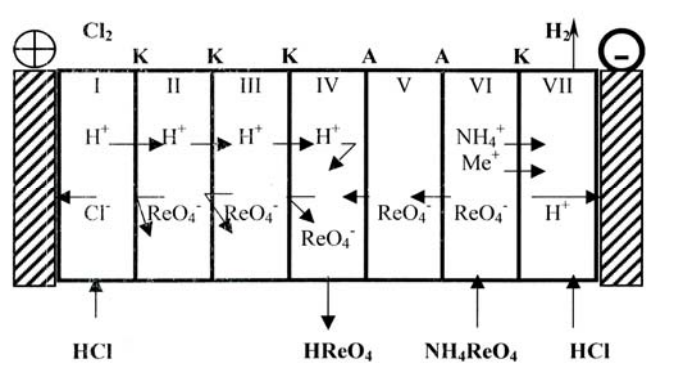


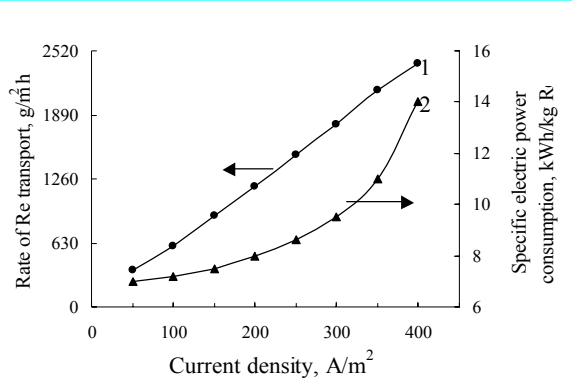
The seven-chamber electrodialyzator and technology of electrodialysis clearings, concentrating and obtaining of rhenium containing products (rhenium acid, perrhenate salts) from industrial rhenium containing solutions are developed.
The device design enables to use the cheap anode materials (graphite, alloys on the basis of lead, etc.), excludes pollution of rhenium acid by anode material that is the important condition for the obtaining solutions of high purity.

The scheme seven-chamber electrodialyzator

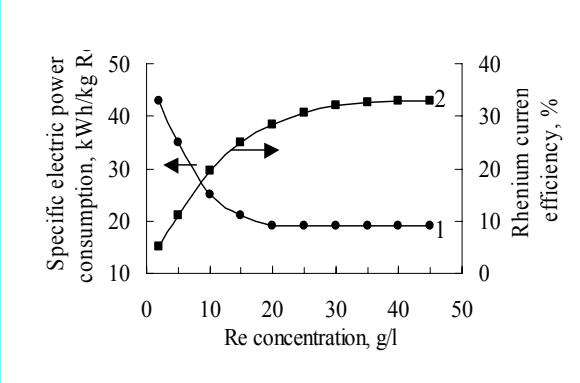


Membranes: K – cation exchanger (MK-40), A – anion exchanger (MA-40)
Chambers: I - anode, II - the first isolating, III - the second isolating, IV - concentrating, V - buffer, VI -desalting, VII - cathodes

Effect of current density on rhenium transport to concentrating chamber (1) and specific electric power consumption (2)



Effect of rhenium content in initial solution of ammonia perrhenate on specific electric power consumption (1) and current efficiency (2)



Effect of current density on the rate of rhenium acid concentrating (experiment time is 60 minutes) in the process of electrodialysis of ammonia perrhenate solution

Current density, A/m^2	Re concentration in chambers, g/L		$HReO_4$ concentration in concentration chamber, g/L	Rhenium current efficiency, %	Specific electric power consumption, kWh/kg Re
	First barrier Chamber	second barrier chamber			
50	0.165	0.833	43.60	68.6	7.0
100	0.330	1.676	86.97	68.4	7.2
150	0.502	2.450	134.06	68.8	7.6
200	0.530	3.325	177.69	69.1	8.0
250	0.837	4.032	213.86	67.9	9.0
300	1.025	5.100	273.15	70.1	10.1
400	1.300	6.550	337.48	67.7	14.4

Effect of temperature on rhenium transport for electrodialysis of potassium perrhenate solution (experiment time is 60 minutes)

Current density, A/m^2	Solution temperature, °C	Rhenium transport to electrodialysis chambers, g/m^2 .h			Rhenium current efficiency, %
		first barrier chamber	second barrier chamber	Concentration chamber	
250	30	0.020	0.053	114.0	6.3
250	40	0.020	0.056	169.0	9.4
250	50	0.022	0.058	389.0	22.4
350	30	0.029	0.084	415.0	16.8
350	40	0.029	0.084	468.0	18.8
350	50	0.029	0.084	630.0	25.2
400	30	0.035	0.104	428.0	15.0
400	40	0.036	0.111	477.0	16.8
400	50	0.037	0.112	640.0	22.4

Effect of flow rate of initial solution of ammonia perrhenate on the rate of rhenium transport to concentrating chamber (current density is 300 A/m^2, temperature of solutions 50 ^{o}C, reaction time of experiment is 60 minutes)

Flow rate of initial solution in demineralization chamber volume of chamber/min e	$HReO_4$ concentration in concentration chamber, g/L	Re concentration in catholyte, g/L	Rate of Re transport to chamber of concentrating, g/m^2 .h	Rhenium current efficiency, %	Specific electric power consumption, kWh/kg Re
0.20	272.1	0.004	2568	70.0	10.10
0.37	273.4	0.004	2575	70.3	9.90
0.48	276.2	0.004	2580	70.5	9.75
0.67	278.7	0.004	2586	71.0	9.40
0.83	280.4	0.007	2595	71.3	9.20
0.98	283.7	0.008	2600	71.7	9.00
1.10	284.4	0.010	2605	71.9	8.80
1.20	288.1	0.016	2610	72.2	8.70

It was identified that the optimum conditions of the process of rhenium acid concentration by electrodialysis of ammonia and potassium perrhenate solutions are the following: 300 A/m^2 current density, 50^{o}C solution temperature in electrodialyzer's chambers, 35-40 g/L rhenium concentration in initial solution, 0.67-0.83 volume of chamber/min flow rate of initial solution in a demineralization chamber. Concentration of rhenium acid in these conditions is 300 g/L and higher. However, higher concentration of produced rhenium acid is inadvisable because the selectivity and mechanical strength of ionite membranes are decreased.

The transformation of rhenium in rhenium acid from the initial quantity rhenium was 99 %. The basic rhenium losses (0,2 %) with anode and cathodes chambers waste solutions occur. The outcome on a current of rhenium has made 60 %, the expense of the electric power on 1 kg Re - 6 кVt/h.

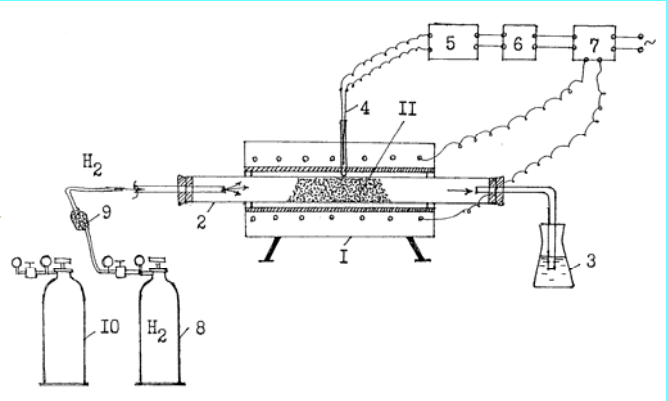
From obtained rhenium acid by an ammonia solution neutralization the ammonium perrhenate salt besieges, weights, containing. %: Re - 69,2; K $<1\cdot10^{-3}$; Na - $1\cdot10^{-4}$; S - $2\cdot10^{-3}$; P $<1\cdot10^{-4}$; Si $<1\cdot10^{-4}$; Mg $<1\cdot10^{-4}$; Mn $<1\cdot10^{-4}$; Al $<1\cdot10^{-4}$; Fe $<5\cdot10^{-4}$; Ca $<1\cdot10^{-3}$; Mo $<5\cdot10^{-4}$; Cu $<5\cdot10^{-5}$ and corresponding ammonium perrhenate of mark AP-0.

For higher purity metal rhenium obtaining the initial ammonium perrhenate of mark AP-0 was cleared from present impurity industrially on industrial installation with use of specially cleared water. Produced rhenium acid contained, weight %: Re - 27,5; K - $4\cdot10^{-5}$; Na - $8\cdot10^{-5}$; Fe - $2\cdot10^{-4}$; SO_4^{2-}-0,01; Cl^--0,06; mechanical impurities were not determined.

The ammonium perrhenate besieged from this acid by ammonia contained, mass. %: Re-69,2; K $<2\cdot10^{-4}$; Na - $6\cdot10^{-5}$; S $<2\cdot10^{-4}$; P $<1\cdot10^{-3}$; Si $<4\cdot10^{-5}$; Mg - $8\cdot10^{-6}$; Mn - $4\cdot10^{-6}$; Al - $1\cdot10^{-4}$; Fe - $5\cdot10^{-6}$; Ca - $3\cdot10^{-4}$; Mo - $1\cdot10^{-4}$; Ni $<1\cdot10^{-4}$; Cu $<5\cdot10^{-5}$. The maintenance in ammonium perrhenate of such impurity as potassium, sodium, magnesium, manganese, iron were on 1-2 order lower than in initial salt.

11

Installation for restoration ammonium perrhenate



1 - the tubular furnace; 2 - a quartz pipe; 3 - an absorbing bottle; 4 - the thermocouple; 5 - device KSP; 6 - the thermo relay; 7 - a pressure regulator; 8 - a cylinder with hydrogen; 9 - a trap with silica gel; 10 - a cylinder with inert gas; 11 – ammonium perrhenate

12

The maintenance of impurity in metal rhenium *

The maintenance, weights. %	Tests metal rhenium					
	1	2	3	4	5	6
K	$9\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$
Na	$2,5\cdot10^{-3}$	$3\cdot10^{-4}$	$3\cdot10^{-4}$	$7\cdot10^{-4}$	$1\cdot10^{-3}$	$3\cdot10^{-4}$
Ca	$5\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$	$<1\cdot10^{-3}$
Si	$4,2\cdot10^{-4}$	$3,2\cdot10^{-4}$	$2,2\cdot10^{-4}$	$1,8\cdot10^{-4}$	$2,8\cdot10^{-4}$	$2,4\cdot10^{-4}$
Al	$4,3\cdot10^{-4}$	$1,8\cdot10^{-4}$	$1,3\cdot10^{-4}$	$1\cdot10^{-4}$	$5\cdot10^{-5}$	$8\cdot10^{-5}$
Fe	$6\cdot10^{-5}$	$8\cdot10^{-5}$	$5\cdot10^{-5}$	$5\cdot10^{-5}$	$6\cdot10^{-5}$	$5\cdot10^{-5}$
Mg	$7\cdot10^{-4}$	$1,8\cdot10^{-4}$	$1\cdot10^{-4}$	$1,5\cdot10^{-4}$	$1,7\cdot10^{-4}$	$2\cdot10^{-4}$
Ni	$1\cdot10^{-4}$	$1\cdot10^{-4}$	$1\cdot10^{-4}$	$1\cdot10^{-4}$	$1\cdot10^{-4}$	$1\cdot10^{-4}$
Mo	$<1\cdot10^{-4}$	$<1\cdot10^{-4}$	$<1\cdot10^{-4}$	$<1\cdot10^{-4}$	$<1\cdot10^{-4}$	$<1\cdot10^{-4}$
Cu	$6\cdot10^{-5}$	$2\cdot10^{-5}$	$2\cdot10^{-5}$	$2\cdot10^{-5}$	$3\cdot10^{-5}$	$1\cdot10^{-5}$
Re не менее	99,9806	99,9967	99,9970	99,9969	99,9962	99,9969

*1 test metal rhenium is obtained from ammonium perrhenate of mark AP-0;
2-5 tests metal rhenium are obtained from the cleared ammonium perrhenate;

CONCLUSION

The optimum conditions of electrodialysis rhenium acid concentration process conducting from the ammonium perrhenate and potassium solutions are: the density of a current - 300 A/m^2, temperature of solutions in electrodialyzators chambers - 50 ºC, rhenium concentration in an initial solution - 35-40 g/dm^3, speed of an initial solution channel in the desalting chamber – 0.67 -0.83 volumes/minutes. Under these conditions the 300 g/dm^3 and above of rhenium acid concentration is reached. However, the more concentrated rhenium acid is inexpedient, as selectivity and mechanical ionic membranes durability thus decreases.

The transformation of rhenium in rhenium acid from the initial quantity rhenium was 99 %. The basic rhenium losses (0,2 %) with anode and cathodes chambers waste solutions occur. The outcome on a current of rhenium has made 60 %, the expense of the electric power on 1 kg Re - 6 κVt/h.

From obtained rhenium acid by an ammonia solution neutralization the ammonium perrhenate salt besieges corresponding ammonium perrhenate of mark AP-0. Metal rhenium produced from marketable AP-0 grade ammonium perrhenate by method of high-temperature reduction with gaseous hydrogen at 800-900º C contained no more than 99,981 % of the basic metal.

Initial ammonium perrhenate purification was carried out on industrial electrodialysis apparatus with using specially purified water for increase of metal rhenium purity in industrial conditions. Produced rhenium acid contained, weight %: Re - 27,5; K - $4\cdot10^{-5}$; Na - $8\cdot10^{-5}$; Fe - $2\cdot10^{-4}$; SO_4^{2-}-0,01; Cl^--0,06; mechanical impurities were not determined.

Ammonium perrhenate, deposited from this acid by ammonia salt contained, weight %: Re-69,2; K $<2\cdot10^{-4}$; Na - $6\cdot10^{-5}$; S $<2\cdot10^{-4}$; P $<1\cdot10^{-3}$; Si $<4\cdot10^{-5}$; Mg - $8\cdot10^{-6}$; Mn - $4\cdot10^{-6}$; Al - $1\cdot10^{-4}$; Fe - $5\cdot10^{-6}$; Ca - $3\cdot10^{-4}$; Mo - $1\cdot10^{-4}$; Ni $<1\cdot10^{-4}$; Cu $<5\cdot10^{-5}$, that on 1-2 degree (order) lower than content of such impurities as K, Na, Mg, Mn, Fe in initial ammonium perrhenate.

Metal rhenium powder, produced by high-temperature reduction with gaseous hydrogen of ammonium perrhenate, preliminary purified by electrodialysis method, exceeds on degree (order) by basic metal content the rhenium, obtained by reduction of AP-0 grade ammonium perrhenate.