

INVESTIGATION OF CONTAMINATION CAUSED BY RUBBER COMPACTOR IN ARSINE PURIFIED IN GAS CENTRIFUGE

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In the process of studying the application of gas centrifuges for deep purification of arsine from impurities sulphur was found. Estimation of quantitative content of sulphur in pure arsine showed the value $\sim 10^{-4}$ wt. %. The sources of sulphur was stated to be rubber compactors forming gas centrifuge complex and made from rubber on the basis of butadiene-nitrile caoutchouc using sulfur cure. Using rubber compactors made from rubber produced from fluoroelastomers, not undergoing to sulfur cure one can provide sulphur content in pure product less than 10^{-5} wt. %.

One of the problems of substance deep purification in gas centrifuges as well as many other methods of high-clean substances obtaining is a contamination of substance purified by impurities delivered from structural material of processing equipment. Studying the possibilities of gas centrifuges application for arsine deep purification (AsH_3) [1] sources of Freon and toluene occurrence in purified substance were determined. Freon and toluene content in pure product may reach $\sim 10^{-4}$ %. The given paper is the continuation of investigations [1]. In this paper we investigate in details influence of rubber compactors, being the part of gas centrifuges complex, on arsine purification process.

Arsine purification was carrying out at purification plant consisting of two cascades of gas centrifuges: «upper» and «lower». Arsine purification from «heavy» admixtures occurs on the «upper» cascade (molecular weight is more than arsine molecular weight), on the «lower» cascade – from «light» impurities (molecular weight is less than arsine molecular weight). The plant is completed with gas centrifuges, specially designed for high-clean substances obtained and equipped with isolated path for pumping out gas release products of structural material – evacuation system of behind rotor area.

Effective testing of impurities content in arsine was carried out with MI-1201B mass-spectrometer, for this purpose recording of mass-spectra of installation feeding flux (F), heavy and light fraction of «upper» cascade (H_1 , L_1), heavy and light fraction of «lower» cascade (H_2 , L_2) were systematically carried out.

The analysis of «light» part of mass-spectra (components with molecular weight less than arsine molecular weight) of fluxes F and H_2 of the purification plant (fig. 1) made it possible to determine the following differences in them: firstly, presence of ionic peak with $M=31$ AMU, which was identified as conforming to phosphine (PH_3), occurring in initial arsine; secondly, presence of double ionic peak $M=32$ AMU in spectrum of flux H_2 . According to mass defect ionic peak from the side of «light» masses on $M=32$ AMU in spectrum of flux H_2 was identified as conforming to sulfur isotope ^{32}S . Estimation of quantitative content of sulfur in pure arsine gives the value $\sim 10^{-4}$ wt. %. Another sulfur compounds at susceptibility level of mass-spectrometer ($10^{-4}\dots 10^{-5}$ %) in purified product in the range of molecular mass from 10 to 200 AMU were not stated.

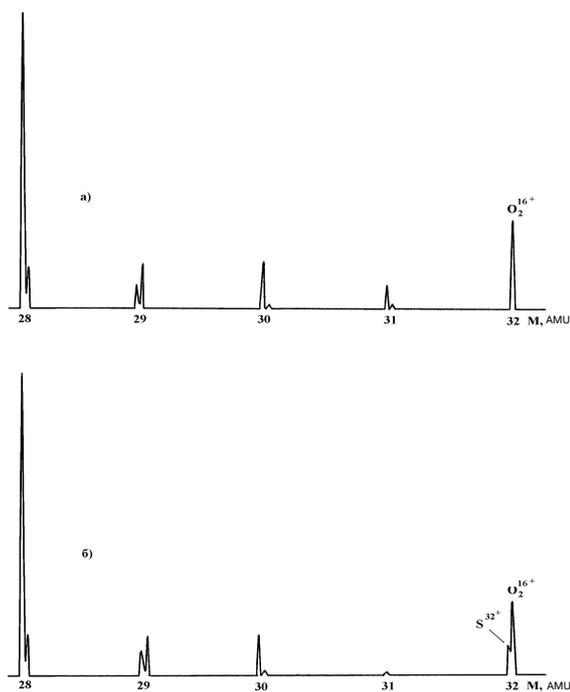


Fig. 1. Mass-spectra of purification plant fluxes: a) F , b) H_2

Thus, according to mass-spectra (fig. 1), it is clear that in process of purification arsine is released from impurities being in feeding (PH_3), but at the same time it is contaminated with sulfur.

Sulfur is a limiting element for arsine used in solid-state microelectronics. Its content in pure product should not be more than 10^{-5} wt. %. Therefore it is necessary to determine a source of gas release and to find a method to decrease its content in pure arsine just by the order.

The most probable sources of arsine contamination by sulfur are general mechanical rubber goods used in gas centrifuges as compactors. Among all constructional materials these ones contain the most amount of sulfur.

To check this hypothesis the interaction of arsine with samples of two types of rubber compactors: made on basis of butadiene-nitrile rubbers (rubber IRP-9024) and made on basis of fluoroelastomers (rubber IRP-1345) was investigated in the conditions close to those in the purification plant. Rubber compactors made of rubber on basis of butadiene-nitrile rubbers were

included in gas centrifuges complex of the purifying plant. The compactors made on basis of fluoroelastomers are generally used in gas centrifuges intended for operation with chemically active compounds. As rubber samples standard spacers of gas centrifuges were used. Before making experiments the samples were hung out and put into hermetic reservoir which had been vacuumized and filled with arsine. The samples were kept in arsine medium during twenty-four hours and then mass-spectra were recorded in certain time intervals. After samples removal from the reservoir rubber samples were weighted again.

The results of rubber samples weighing before and after interaction with arsine are given in tables 1 and 2.

Table 1. The results of ИРП-9024 rubber samples weighing before and after interaction with arsine during 5 days

№	Samples weigh, mg		Weigh changes, ΔG , mg	$\frac{\Delta G}{G_1} \cdot 100$, %
	Before the experiment, G_1	After the experiment, G_2		
1	406,7	407,4	0,7	0,17
2	534,8	535,5	0,8	0,15
3	1345,1	1346,2	1,1	0,08
4	1817,0	1818,3	1,3	0,07
5	5736,4	5738,8	2,4	0,04

Table 2. The results of ИРП-1345 rubber samples before and after interaction with arsine during 9 days

№	Samples weigh, mg		Weigh changes, ΔG , mg	$\frac{\Delta G}{G_1} \cdot 100$, %
	Before the experiment, G_1	After the experiment, G_2		
1	2992,2	2992,0	0	0
2	2876,6	2876,4	0,2	0,007
3	3039,0	3038,6	0,1	0,002
4	2912,8	2912,8	0	0

From table 1 it is seen that after interaction of ИРП-9024 rubber samples with arsine the expected weigh reduction does not occur but, on the contrary, their weigh have become 0,04...0,17 % more due to sorption of arsine on the surface ИРП-1345 rubber samples weigh after the interaction with arsine (table 2) has not practically changed.

Dependence of sulfur concentration in arsine on contact time with the samples of different types of rubber is shown in fig. 2.

The obtained results (fig. 2) indicate obviously that rubber compactors of the purification plant (ИРП-9024)

are really the main sources of arsine contamination by sulfur: just in a day after filling the reservoir with the samples with arsine sulfur concentration in arsine amounted $2,1 \cdot 10^{-4}$ wt. % and further it increased till it reached $\sim 10^{-3}$ wt. % in 5 days. Sulfur concentration for rubber ИРП-1345 was increasing only during the first 48 hours and after that reached the «saturation» threshold and it was not practically changed in the course of time.

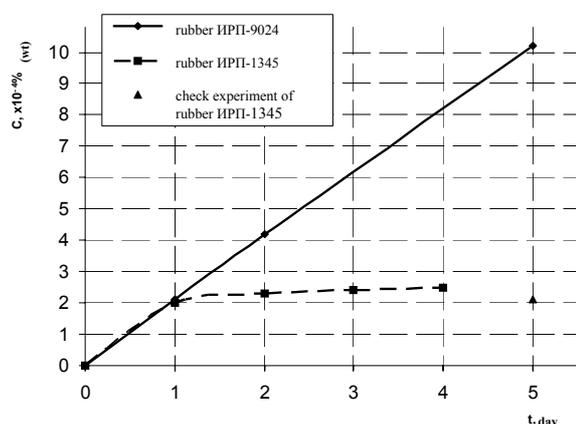


Fig. 2. Dependence of sulfur concentration in arsine on contact time with rubber samples

An additional experiment was carried out with rubber ИРП-1345. The reservoir with rubber samples was pumped out to pressure less than 10 Pa, then it was filled with arsine again. The concentration of sulfur in arsine was determined after 5 days of exposure. It amounted $2,1 \cdot 10^{-4}$ wt. %. Thus, it is obvious that the samples of ИРП-1345 rubber contaminate arsine with sulfur significantly less than another investigated rubber samples. All these correlate well with the fact that rubber on the basis of butadiene-nitrile rubbers undergoes sulfur cure (heating with elemental sulfur at temperature 140...160 °C) when producing [2, 3] to attain the necessary service properties unlike rubber on the basis of fluoroelastomers. It greatly increases sulfur content in it.

Thus, it is stated that in the process of arsine purification in gas centrifuges rubber compactors can contaminate purified product by sulfur. To provide sulfur content in high-clean arsine less than 10^{-5} wt. % it is necessary to eliminate application of elements made of rubber subjected to sulfur cure in gas centrifuges construction. One of the variants of a valid replacement may be compactors made on the basis of fluoroelastomers.

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