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Studies on Preparation of High Purity Sulfur. IV. Effect of the Distillation with Adsorbent and Silver on the Removal of the Impurities in Sulfur[1)]

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Synopsis. The distillation was shown to be effective in removing arsenic, selenium, tellurium, carbon, and common elements in sulfur. However, there was the disadvantage that trace amounts of common elements were not reduced further by the repetition of the distillation. This could be carried out by the zone refining.

In the course of studies of the preparation of high-purity sulfur, the removals of trace amounts of the main impurities, such as arsenic, selenium, tellurium, and carbon, have been separately presented in previous papers.[2-4)]

In order to remove the impurities simultaneously, a distillation of sulfur, using an adsorbent and silver, was proposed. In the distillation, the removal of the impurities was ascertained. Simultaneously, the ignition residue of the distilled sulfur and the common elements in the ignition residue were investigated.

The distillation was shown to be effective in removing various impurities in sulfur. At the same time it was found that trace amounts of the common elements remained in the distilled sulfur.

The zone refining of sulfur[5-8)] was carried out parallel with the distillation. The experimental results suggest that trace amounts of the common elements in the distilled sulfur can be further reduced by the technique.

Experimental

Distillation of Sulfur. The distilling apparatus and the silver supported on a pumiceous substance were the same as have been described in a previous paper.[3)] The column of the distilling flask, 50 cm long, was filled with the silver. In the flask, 50 g of a sulfur sample and 5 g of activated carbon (24—40 mesh) were then taken. The column was heated at 600 °C, the flask was heated below 500 °C, and the sulfur was distilled in a flow of inert gas.

Zone Refining. Sixty grams of sulfur were placed in a glass tube (15 mmϕ) and heated at 140 °C for several hours. After the atmosphere of the tube had then been replaced with nitrogen, the tube (30 cm long) was sealed. It was kept horizontal, heated to melt the sulfur, and then cooled slowly. A horizontal zone-refining apparatus was used. The conditions of the zone refining were as follows: zone width, 3 cm; speed of zoning, 1 cm per hour; pass of zone, 10 times.

Determination of the Impurities in Sulfur. The arsenic, selenium, and tellurium were separately determined by the spectrophotometric methods described in previous papers.[2,3)]

The common elements in the ignition residue of sulfur were determined by the spectrographic method. A sulfur sample was mixed with graphite powder in a proportion of 10: 1 in a platinum crucible and then heated to the melting point. Then, the graphite powder remaining in the crucible was heated at 650 °C for 3 hr; it was afterward transferred into a capsule and shaken sufficiently with the mixer mill. A weighed portion of this was used as the sample for measurements. The intensities of the spectral lines of the elements, such as Co, Si, Fe, Pb, Cu, Mg, Ca, Al, Cd, B, Bi, Ag, and Sn, were simultaneously measured by using a direct-reading emission spectrometer (Shimadzu, Ebert Grating Spectrometer GEM-340).

Results and Discussion

The distillation with an adsorbent and silver was compared with the distillations without them with respect to the removal of various impurities. The former distillation was more effective than the latter, as is shown in Table 1.

Arsenic was adsorbed to the activated carbon, and it remained in the flask. While sulfur vapor passed through the column, selenium and tellurium were caught in it, because they reacted rapidly with the heated silver in it. The removal rates of arsenic, selenium, and tellurium by the distillation were about 99, 95, and 95% respectively. The repetition of the

Table 1. Contents of the impurities in raw and distilled sulfurs

	Impurities, ppm																
	Co	Si	Fe	Pb	Cu	Mg	Ca	Al	Cd	B	Bi	Ag	Sn	As	Se	Te	Ignition residue
Raw sulfur	0.36	>10	>10	1.1	0.96	1.2	3.4	>10	0.40	0.80	0.52	*	0.28	43	130	56	290
Sulfur distilled with adsorbent and silver	*	0.56	0.16	*	0.04	*	0.04	0.12	*	*	0.04	*	*	0.4	6.5	2.8	2.4
Sulfur distilled without ones	0.04	0.72	1.0	*	0.12	*	0.24	0.24	*	*	0.08	*	0.04	26	117	45	43
Sulfur redistilled without ones	*	0.72	0.56	*	0.12	0.04	0.04	0.04	*	*	0.04	*	0.04	16	105	36	3.7

* No detection.

Table 2. Contents of the impurities in fractions of sulfur traversed by molten zones

Distance in zone lengths (cm)	Impurities, ppm													Distance in zone lengths (cm)	Impurities, ppm		
	Co	Si	Fe	Pb	Cu	Mg	Ca	Al	Cd	B	Bi	Ag	Sn		As	Se	Te
0—10	0.59	1.50	2.66	0.43	0.26	0.13	0.68	0.98	0.01	1.94	0.21	0.18	0.40	0— 6	0.9	79	20
10—20	0.02	0.42	0.81	0.05	0.26	0.10	0.20	0.14	0.01	*	*	*	*	6—12	1.3	79	20
20—30	0.02	0.33	0.50	0.04	0.10	0.09	0.20	0.13	0.01	*	*	*	*	12—18	1.9	79	20
														18—24	1.9	79	20
														24—30	3.2	79	20

* No detection.

distillation was effective in removing trace amounts of the impurities.

Although it is not shown in Table 1, most of the carbonaceous impurity was removed, and the halogen was removed somewhat. The carbonaceous impurity was decomposed rapidly and separated from the sulfur in the process, by which it was brought in contact with the heated silver in the column.

The common elements were effectively removed, too. Their contents in the sulfur were all reduced to the range of $n \times 10^{-1}$—10^{-2} ppm. However, there was the disadvantage that trace amounts of the common elements remaining in the distilled sulfur were not reduced further by the repetition of the distillation.

Table 2 shows the contents of various impurities in the fraction of sulfur traversed by molten zones. The results of the experiment showed that the contents of most of the common elements were reduced, and suggested that trace amounts of the impurities in sulfur could be reduced by zone refining. At the same time, it was found that the contents of arsenic, selenium, and tellurium could not be expected to be reduced, for the impurities in sulfur had unfavorable coefficients.

By the distillation, such impurities as arsenic, selenium, tellurium, and carbon were effectively removed, and the common elements were reduced to trace amounts. The further reduction of the common elements could be carried out by the zone refining.

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