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# Short Communications

## Szilard-Chalmers Reaction on Antimony

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In order to obtain a Szilard-Chalmers concentration of the $\beta$-active isotopes formed by the action of slow neutrons on antimony, some experiments were carried out with the compounds phenylstibinic acid, $C_6H_5SbO(OH)_2$, and triphenylstibine, $(C_6H_5)_3Sb$. With the first compound the results were negative, presumably owing to the difficulties in the separation of the liberated activity from the bulk. The second compound proved to be suited to the purpose, as could be anticipated from the analogous experiments by Maurer and Ramm[1] on bismuth and lead by means of their tri- and tetraphenyl compounds respectively.

*Experiments with phenylstibinic acid.* An attempt was made to separate the activity by hydrogen sulphide precipitation and also by zinc reduction. The results were negative, and in view of the success with the triphenyl compound the failure appears to have been due to bad separation. In the case of the sulphide, for instance, an organic antimony sulphide is formed under about the same conditions as $Sb_2S_3$.

A very slight concentration was obtained when the solid acid was irradiated, dissolved in chloroform, and the solution extracted with an aqueous solution containing antimony trichloride and tartaric acid. The antimony was finally precipitated as $Sb_2S_3$. The specific activity of the antimony in this compound was 1.5 times that of the original antimony of the acid and contained 1.5 % of the total activity.

It is generally assumed that the phenylstibinic acid dissolves as a colloid in solvents like chloroform. The greater part of the activity may then be retained in the interior of the particles, which seems to be a reasonable explanation of the poor yield.

*Experiments with triphenylstibine.* Triphenylstibine is readily soluble in benzene. When such a solution has been irradiated, it is possible to extract about 50 % of the activity by means of water containing small amounts of antimony and tartaric acid. Some activity may also be obtained in a second extraction. The trisulphide is finally precipitated. Two typical experiments are given in Table 1 ($\gamma$-radiation measured).

*Table 1. Two typical concentrations of active antimony.*

| | $(C_6H_5)_3Sb$ irradiated | $Sb_2S_3$ obtained | Spec.act. of Sb increased | Yield % |
|---|---|---|---|---|
| I (first. extr.) | 10 g | 24 mg | 102 t. | 51 |
| (sec. extr.) | | — | — | 9 |
| II (first extr.) | 24 g | 35 mg | 135 t. | 41 |
| (sec. extr.) | | — | — | 6 |

The product contains both activities formed (Sb $^{122}$ and Sb $^{124}$), but it has not been established whether the ratio is the same as that in which they are formed by the nuclear reactions, a matter which may be of some interest.

By the use of less carrier it should be possible to attain higher concentrations.

*Preparation of antimony compounds.** The phenylstibinic acid was prepared according to Schmidt [2], and the triphenylstibine by the method given in *Org. Syntheses* [3].

*Szilard-Chalmers reaction by means of* $(C_6H_5)_3Sb$. The compound is dissolved in benzene (1 g in 5 ml), and the solution clarified if necessary by means of charcoal, which is filtered off. The solution is irradiated with slow neutrons in the cyclotron.

After the irradiation a sample of the solution is saved for comparative activity measurements. The main part of it is mixed with the same volume of an aqueous solution containing 2 % tartaric acid and 0.05 % potassium antimonyl tartrate, and the solutions are shaken together for 15 minutes. (It seems to be of importance for the yield to use an adequate amount of aqueous solution and to shake long enough.) After separation the solutions are filtered clear, and the organic solution is ready for a new irradiation. From the aqueous solution $Sb_2S_3$ is precipitated with $H_2S$, washed and dried. The sulphide prepared in this simple manner may not have the correct composition, but that is of little importance in the present experiments.

1. Maurer, W., and Ramm, W. *Z. Physik* **119** (1942) 602.
2. Schmidt, H. *Ann.* **421** (1920) 188.
3. *Org. Syntheses*, Coll. vol. I, 2nd ed., New York (1946).

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# A New „Daylight-device" for Titrations

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The work in the analytical laboratory would be greatly facilitated if the micro titrations which have to be carried out in broad daylight in order to obtain accurate readings (*e. g.* iodometric titrations) could be performed with the same precision by artificial light.

The so-called »daylight-bulbs» which so far have been on the market, do not produce light of such a quality that very slight colour changes near the end-point of the titration can be read with the precision necessary in micro titrations.

We have succeeded in constructing an artificial »daylight device» which satisfies all our demands.

The light-source is a helical, high-voltage fluorescent lighting tube which with a current of 25 mA will produce light having a luminous flux of about 100 lumen. The nature of the light justifies the term: daylight of a colour temperature of about 5000° K. The tube will stand about 8000 lighthours.

A leak transformer having a primary voltage of 220 V and a secondary no load voltage of 1500 V has been used for the operation of the above fluorescent lighting tube. The drop in voltage between the electrodes of the tube will amount to about 300 V at 25 mA. It is therefore sufficient to use a transformer having a no load voltage of 6—700 V if such a transformer is available. The consumption will in the latter case be 6—8 W.

The helical light-source has a total diameter of 6.5 cm. The transformer and the electrodes of the tube are insulated mounted in a lattice case (height 14.5 cm, length