Deposition of osmium tetroxide on reactive surfaces

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The recent study of the chemistry of element 108, hassium [1], leads to the conclusion, that it forms a volatile oxide, as expected for a member of group 8 of the periodic table [2]. So far, no chemical reaction of this oxide is known. To learn more about the chemical behaviour of hassium, one would like to investigate the chemistry of hassium oxide, the only known compound of hassium. Presumably, it is chemically similar to OsO_4 and RuO_4, which have an acidic character and are able to form salts with alkaline materials.

For that reason, a **C**ontinuously Working **A**rrangement For **C**lust**er**l**e**ss Transport Of **I**n-**s**i**t**u Produced Volatile **O**xides, *CALLISTO*, was developed and successfully used to deposit the volatile OsO_4 on metallic sodium surfaces [3]. Although these surfaces are very efficient for the deposition process, the quality of the α-spectra deteriorates with time. This could be explained by the obvious fact, that the sodium surface is covered with an increasing oxide layer, which doesn't interfere significantly with the deposition process, but disturbs considerably the detection process. Consequently, other deposition materials are required.

It is well known, that OsO_4 reacts with olefins. Mostly this reaction is done in solution. For purposes of α-detection, a solid olefin with the ability to form a smooth layer would be required.

Fortunately, cis-1,4-polybutadiene is such an substance. It contains double bonds and because of its polymeric character one can easily produce reproducible layers of it. (Fig. 1)

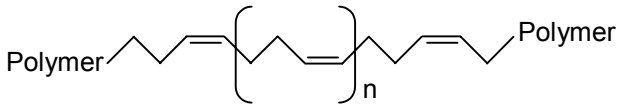

Fig. 1: cis-1,4-polybutadiene

The yield of the reaction of the solid polymer with the gaseous OsO_4 is shown in Fig. 2.

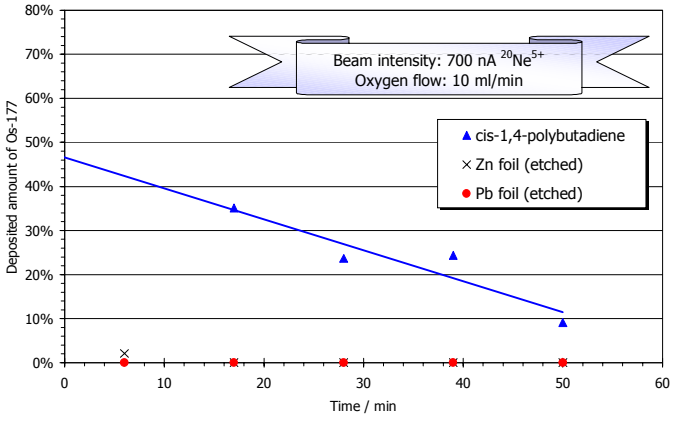

Fig. 2: Deposition of osmium tetroxide on cis-1,4-polybutadiene

Unfortunately, this relatively low yield shows, that this method is not adequate for experiments with hassium oxide. As shown, cis-1,4-polybutadiene is more reactive than etched surfaces of zinc and lead, on which almost nothing is deposited.

This leads to the implication, that alternative materials for the deposition of OsO_4 are needed.

If alkaline materials are suitable for our purposes, an alkaline surface would be most efficient. Unfortunately, it is hardly possible to reproducibly prepare thin layers of an alkali hydroxide without a substrate. Nevertheless, is it possible to coat an inert material with a smooth layer of alkali hydroxide. We choosed at first graphite as inert substrate and coated it with a thin layer of KOH, using the solubility of KOH in C_2H_5OH and preparing the layer from an ethanolic solution. The results for 2 different amounts of helium as transport gas are shown in Fig. 3.

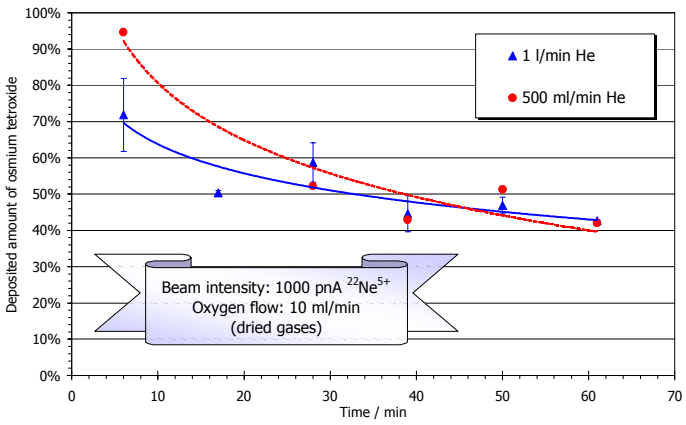

Fig. 3: Deposition of OsO_4 on graphite, coated with KOH

We used our recently developed gas drying-unit to dry the transport gases, remaining less than 0.5 ppm humidity in the gas flow. Surprisingly, the yield of the deposited OsO_4 decreases significantly with time. This behaviour is relatively unexpected, because the macroscopic amount of hydroxide cannot be fully neutralised with the microscopic amount of osmium tetroxide.

In a recent experiment, we found evidence for the important role of catalytic amounts of water. If we add some additional water vapour to the transport gas, the deposition process benefits greatly. This could explain, why an alkaline surface would be less reactive after a certain time of contact with the dried gas. This has to be studied in a forthcoming experiment.

We want to thank Dr. M. Watson, MPI Mainz, for the preparation of cis-1,4-polybutadiene.

[1] http://www.gsi.de/presse/Hassium.html

[2] V. Pershina *et al.*, J. Chem. Phys. **115** (2001), p.792

[3] A. v. Zweidorf, GSI Scientific Report 2000-1, p.171