Simple apparatus for cleaning laboratory ware used in trace analysis

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A simple cleaning apparatus made of glass and teflon (PTFE) is described and characterized. It is suitable for an efficient and rapid cleaning of small test tubes, volumetric flasks, crucibles, *etc.* made of glass, quartz, PTFE, glassy carbon. The cleaning effect arises from a continuous contact of hot vapours and condensates of the cleaning liquids (hydrochloric acid, nitric acid, water) with the inside of the air-cooled laboratory ware. The cleaning efficiency was tested by cleaning intentionally contaminated quartz test tubes and volumetric flasks.

Описан и охарактеризован простой моющий аппарат из стекла и политетрафторэтилена (ПТФЭ). Он годится для эффективного и быстрого мытья небольших пробирок, волюмометрических колб, тиглей и т. п., изготовленных из стекла, кварца, ПТФЭ или стекловидного углерода. Моющий эффект возникает благодаря продолжительному контакту горячих паров и конденсатов моющих жидкостей (соляной кислоты, азотной кислоты, воды) с внутренней поверхностью воздухом охлаждаемой лабораторной посуды. Эффективность мытья была проверена посредством очистки сильно загрязненных кварцевых пробирок и волюмометрических колб.

The accuracy of results in trace analysis can be seriously influenced by contamination of the sample during the analytical procedure. The most significant contamination sources are reagents, airborne dust, containers, laboratory ware, and the analyst [1—3].

The contamination caused by the laboratory ware and containers is due to the desorption or leaching of elements from the surface of these vessels when being in contact with the sample and reagent solutions. On the other hand, the vessel surface can give rise to losses of trace elements from the solution owing to adsorption and/or ionic exchange phenomena [4].

To minimize risks of contamination and losses the labware has to be made of high-purity materials with negligible adsorption properties (*e.g.* quartz, PTFE, glassy carbon, *etc.*) and it should be carefully cleaned before use. The cleaning of vessels in trace analysis is an inevitable but laborious and costly procedure [3]. To make it easier the steaming of vessels with hot vapours of acids

or water has been recommended [1]. For this purpose an apparatus described in [1] can be used. Such a cleaning procedure, though time-consuming, guarantees both the proper cleaning of vessels and a significant reduction of their adsorption properties.

This paper presents a description of a simple device for cleaning laboratory vessels used in trace analysis. The cleaning effect of the apparatus is due to the continuous contact of hot vapours and condensates of cleaning liquids with the inner surface of the air-cooled vessels.

Experimental

Twice distilled and deionized water was used in the experiments. The solutions and the treated vessels were handled on a horizontal-laminar-flow clean bench.

The solutions were analyzed on the AAS 3 atomic absorption spectrometer equipped with the EA-3 graphite furnace atomizer using pyrolytically coated graphite tubes (all from Zeiss, Jena). Sample volumes of 50 mm^3 were injected. The temperature programs used were those recommended by the manufacturer.

Cleaning apparatus

The cleaning apparatus consists of the outer borosilicate glass vessel (common laboratory beakers can be used) and the inner PTFE disks connected with PTFE supports (Fig. 1). The cleaning liquid in the bottom of the device is heated on a hot plate. The purpose of the two lowest disks is to trap the liquid particulates in the vapour stream formed during bubble rupture of the boiling liquid. The vapour stream passes through the PTFE tubes into the vessels to be cleaned. Due to the air-cooling of the vessels, most of the vapour condensates on the inner surface of the vessels, the hot condensate washes the impurities off and it flows back through the centre tube into the bottom of the apparatus. The rest of vapours is retained in the condenser.

The packings made from PTFE tubings of suitable diameters direct the vapour stream and prevent the vapours to escape from the device. The size of the apparatus, the number and diameters of the holes in the upper part should be accommodated to the size and number of the vessels to be cleaned. When using H_2O or HCl as a cleaning liquid, water-cooled condenser should be used instead of the air-cooled one.

After having a vessel cleaned it can be simply replaced by another one without interrupting the cleaning of the other vessels.

Results and discussion

The cleaning effect of the apparatus is due i) to the contact of hot vapours with the inner surface of the cleaned vessels, depressing its adsorption proper-

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ties; ii) to the intensive condensation of vapours in the air-cooled vessels enabling a continuous washing off the impurities from the inner surface with the hot condensate. Since the droplets-separator prevents the particles of cleaning liquid to pass into the vessels their inside is steamed with a continuous flow of pure vapour.

The cleaning efficiency of the apparatus can be demonstrated by cleaning of intentionally contaminated $4\ cm^3$ quartz test tubes: The test tubes were filled with a solution containing $0.1\ \mu g/cm^3$ of Mg and Cd and they were left to stand
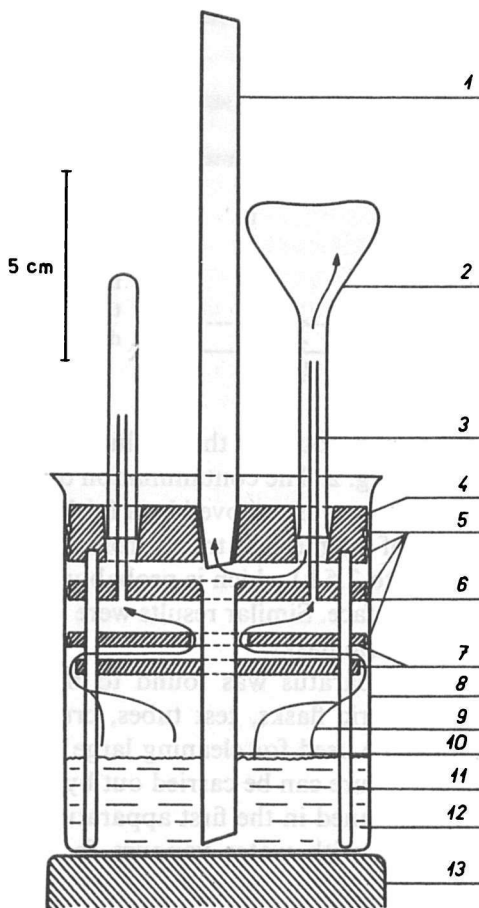

Fig. 1. Cleaning apparatus.
1. Air-condenser; 2. vessels to be cleaned; 3. PTFE tubes for the vapour flow; 4. upper disk with conical holes for the vessels and condenser; 5. packing; 6. vapour distributor; 7. drop-separator; 8. outer vessel; 9. vapour flow; 10. back-flow tube; 11. support; 12. cleaning liquid; 13. hot plate.

for 2 h. After having been rinsed three times with water they were put into the cleaning apparatus. The cleaning liquid was anal. grade 65 % nitric acid. After cleaning the test tubes were rinsed twice with water, filled with water and after standing for 10 min the resulting solution was analyzed.
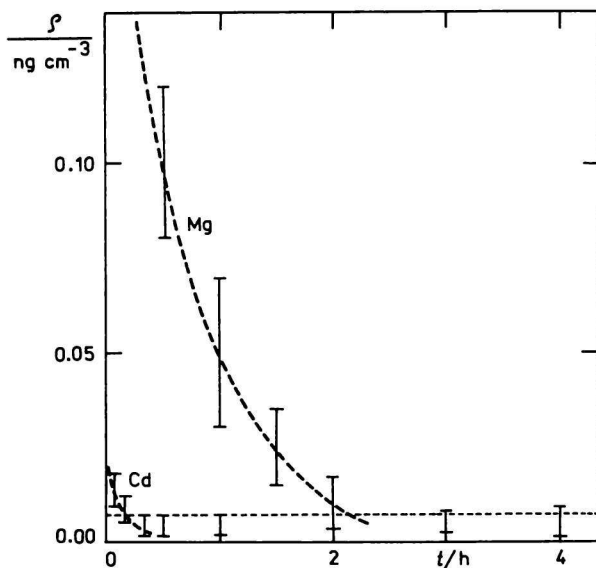

Fig. 2. Dependence of water contamination by the test tube surface on the cleaning period. Dashed line: detection limit for Mg and Cd $(7 \, pg \, cm^{-3})$.

The magnesium and cadmium found in these solutions after various cleaning periods are demonstrated in Fig. 2. The contamination due to the desorption of cadmium from the vessel surface was removed by a fairly short steaming period (10—15 min). The removal of magnesium traces from the inner part of the test tubes took longer times (1.5 to 2.5 h) which is probably due to the high affinity of Mg^{2+} ions to the quartz surface. Similar results were obtained when cleaning contaminated quartz volumetric flasks.

The described cleaning apparatus was found to be suitable for cleaning laboratory vessels as volumetric flasks, test tubes, crucibles, and small containers. It, however, cannot be used for cleaning large beakers, dishes, *etc.*

An efficient cleaning procedure can be carried out by using two devices: The vessel is rinsed with water, cleaned in the first apparatus containing HNO_3 for 2 h and finally it is steamed with water vapours for 1—2 h in the second apparatus.

The main advantage of the apparatus over that described in [1] is the higher cleaning efficiency as a consequence of the continuous condensate formation in the inside of the air-cooled vessels and shorter cleaning periods. Moreover, the manipulation with the cleaned vessels is simpler, since the cleaning process

should not be interrupted and the apparatus should not be opened when replacing the vessels. Finally, the apparatus can be manufactured quite easily and at low costs.

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