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Microdetection of Silver with p-Dimethylaminobenzilidene-rhodanine by the "Resin Spot Test" Technique

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Mikronachweis von Silber mit p-Dimethylaminobenzilidenrhodanin nach der Tüpfelmethode mit Hilfe von Ionenaustauschern

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A sensitive and selective method for the microdetection of silver has been worked out, which can be applied to solutions of various kinds. The well-known "resin spot test" technique is applied using p-dimethylaminobenzilidene-rhodanine as reagent. Among 11 resins tested only the hydroxide form of Amberlite IR-45 gave satisfactory results. The limits of detection, of concentration, of dilution and the exponent of sensitivity (pD) are as follows: 6.04×10^{-8} (g), 2.08 $\times 10^{-6}$ (g/ml), 0.48×10^{6} (ml/g), 5.68. Interferences of foreign ions were examined and results are given in Table 1. Pd(II) and Pt(IV) react in the same way as silver; limits of detection are 3.0×10^{-6} g for Pd and 1.45×10^{-6} g for Pt. The tolerance limits for oxidizing and complexing agents are listed in Table 2.

Table 1. Selectivity of silver detection (3 µg of silver) in the presence of diverse ions

Tolerance limit (µg)	Ion added
≥30[a]	Hg(I), Hg(II), Cd, K, Na, Ba, Ca, Mg, Zn, Al, Sn(II), Mn(II), Co(II), Pb, Sr, Cu(II), Fe(III)[b], Ni, Cr(III)[b], NH_4^+, Cl^-[b], NO_3^-, SO_4^{2-}, PO_4^{3-}, AsO_3^{3-}, MoO_4^{2-}, $[Fe(CN)_6]^{3-}$, $[Fe(CN)_6]^{4-}$, CrO_4^{2-}, AsO_4^{3-}
27	Cu(I)[b]
24	Br^-[b]
15	CNS^-[b]
9	Fe(II)[b]
3	Sb(III)[b], Bi[b], Au(III), I^-[b]

[a] Most ions tested do not interfere even if the limiting proportion of 1:10 is exceeded
[b] These ions lower the sensitivity of the test with formation of brown-red colour in the resin beads

Table 2. Selectivity of silver detection (3 µg of silver) in the presence of oxidizing and complexing agents

Tolerance limit (µg)	Substance added
>5000	H_2O_2
1500	KCN
600	diethylenetriamine pentaacetic acid
500	ethylenediamine tetraacetic acid 1,6-diaminohexane tetraacetic acid 1,2-diaminocyclohexane tetraacetic acid
400	$K_2S_2O_8$
350	chloramine T ($C_7H_7ClNNaO_2S$)[a]
250	nitrilotriacetic acid iminodiacetic acid
100	Na_2O_2 aminoacetic acid $K_2Cr_2O_7$
50	$KMnO_4$
30	KNO_2
6	KIO_3

[a] This substance lowers the sensitivity of the test with formation of brown-red colour in the resin beads

Procedure. One drop of the test solution is mixed with 1 drop of 5% KCN solution on the spot plate. Several grains of the ion-exchanger [Amberlite IR-45(OH)] are added and occasionally mixed with the solution for about 10 min. The supernatant is then drawn off by a piece of filter paper. One drop of 2 M HNO_3 and 3 drops of reagent solution (saturated in acetone) are added. A positive test for silver is indicated by a red-brown to dark red-violet colour forming on the resin beads. The intensity of the colour depends on the amount of silver and the reaction time. In the presence of very small amounts of silver as well as in the presence of foreign substances, it is necessary to observe the colour formation after 20 min. A control test is run without silver solution; the grains become yellow-orange in the case.

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