

Removal of Silver Traces from Palladium by Means of Selective Adsorption

J. Miller & G. Toth

To cite this article: J. Miller & G. Toth (1967) Removal of Silver Traces from Palladium by Means of Selective Adsorption, *Isotopenpraxis Isotopes in Environmental and Health Studies*, 3:1, 19-20, DOI: [10.1080/10256016708622403](https://doi.org/10.1080/10256016708622403)

To link to this article: <http://dx.doi.org/10.1080/10256016708622403>



Published online: 13 Aug 2008.



Submit your article to this journal [↗](#)



Article views: 7



View related articles [↗](#)



Citing articles: 8 View citing articles [↗](#)

[7]. Источники ошибок, дискутированные в этой работе, полностью относятся и к данному определению. Тем же самым способом могут быть получены кривые титрования сложных смесей, причем возможности существенно расширяются при использовании многоканального анализатора импульсов для регистрации гамма-спектров.

3.2. Определение области рН экстракции и кинетика процесса экстракции

Описанный прибор может быть применен также для определения областей рН, в которых происходит полная экстракция

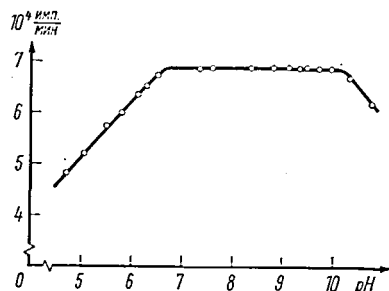


Рис. 4. Область субстехиометрической экстракции скандия теноилтрифторацетоном

для случая субстехиометрического выделения или вообще для исследования зависимостей экстракции на рН раствора. В качестве примера приводится кривая зависимости активности органической фракции от времени при субстехиометрической экстракции Sc (ТТА)₃ хлороформом. рН раствора изменялось прибавлением раствора аммиака. После каждого добавления аммиака измерялось рН раствора. Смесь в течении всего эксперимента попеременно перемешивалась и отстаивалась для разделения фракций. Длительность интервалов перемешивания и отстаивания в описываемом случае приблизительно 1,5 мин. Излом на кривой соответствует времени прибавления NH₄OH, т. е. изменению рН. NH₄OH добавлялось только после установления постоянной активности. Полученная кривая показана на рис. 3.

Дает она информацию как о области полной экстракции Sc(ТТА)₃, так и о времени установления равновесия при разных рН. Кривую зависимости экстрагированного количества Sc на рН, построенную на основании полученной кривой, видно на рис. 4.

Нижний предел области полной экстракции в точности соответствует ранее нами полученным результатам [1]. Разница в величине верхнего предела дана разницей в рН, при которых разлагается Sc(ТТА)₃ и НТТА. Sc(ТТА)₃ разлагается при рН > 10, НТТА уже при рН > 8,5.

Получение описанных зависимостей может быть полностью автоматизировано, если снабдить прибор соответствующими включателями и автоматической записью изменения рН и активности.

3.3. Колориметрические и другие исследования

Кроме приведенных примеров может быть описанный прибор с помощью добавочных оборудований применен для решения ряда аналогичных задач. Относится это главным образом к исследованиям, связанным с процессами в одно-, двух- или более фазовых системах, находящимся в жидком состоянии. Из них, кроме выше приведенных примеров, к наиболее простым принадлежит применение описанного прибора в колориметрии или хелатометрии (используя колориметр с самописцем) и для разного рода титрований.

Автор выражает глубокую благодарность кандидату химических наук М. Крживанеку за ряд ценных указаний, сделанных им как при конструировании прибора, так и при оформлении данной статьи.

Поступила в редакцию 23/VII 1966 г.

Литература

- [1] F. Kukula, M. Křivánek, в книге: Proceedings of the Analytical Chemical Conference, Budapest 1966, p. 408.
- [2] F. Kukula, B. Mudrová, M. Křivánek, в печати.
- [3] J. Růžička, J. Starý, Atomic Energy Review 2 (1964), No. 4, 3.
- [4] T. Braun, J. Tölgyessy, Talanta 11 (1964) 1277.
- [5] H. Spitz, Microchim. Acta (1960) 789.
- [6] T. Braun, E. Körös, в книге: Proceedings of the IAEA Symposium on Radiochemical Methods of Analysis, Vol. II, Vienna 1965, p. 213.
- [7] F. Kukula, M. Křivánek, Chem. Zvesti 20 (1966) 188.
- [8] A. Galik, Talanta 13 (1966) 109.
- [9] A. Galik, M. Křížek, Talanta 13 (1966) 589.
- [10] F. Kukula, Патентное заявление ЧССР PV-7201-65, 1965.

Removal of Silver Traces from Palladium by Means of Selective Adsorption

by J. Miller, G. Toth

(Report from National Atomic Energy Commission, Institute of Isotopes)¹⁾

A highly effective method has been developed for the separation of silver traces from palladium based on the selective adsorption of the former on platinum surface. Authors separated 97 per cent of silver traces labelled with Ag¹¹¹ from palladium with the initial silver-palladium weight ratio being 10⁻⁸. The silver separated proved to be chemically free from palladium. Influence of the preliminary treatment of platinum on its adsorption capacity has been studied.

I. Introduction

It is rather difficult to remove silver traces from palladium and so to produce palladium compounds of high purity especially because of the very similar chemical and electrochemical properties of these two elements. This similarity of the chemical properties renders difficult the separation, for instance, of carrier-free Ag¹¹¹ isotope produced by neutron irradiation of palladium target.

Electrochemical separation of small amounts of silver from palladium can be attained only after insertion of the latter into a complex, failing which the palladium might be deposited prior

to silver in accordance to the more positive normal potential of the former.

It is a well-known phenomenon that ions present in micro-concentration may show different behaviour in many aspects from that of the same ions in macroconcentration [1-3]. Such differences have been observed in the case of silver which make possible to separate silver from palladium with high selectivity even if their weight ratio is as small as 10⁻⁶ - 10⁻⁸.

2. Experimental

Disks made of a 99.99 per cent platinum (diameter 20 mm, thickness 0.3 mm) welded together with a piece of 0.5 mm diameter platinum wire are taken as adsorbents. Preliminary anodic or cathodic polarization is applied to secure the reproducibility

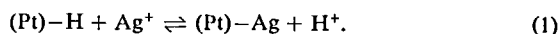
¹⁾ Address: P. B. 77, Budapest 114/Hungary.

of the surface properties. The solution containing the silver to be adsorbed is stirred with constant revolution per minute.

The adequate silver ion concentration is adjusted with reagent grade Merck silver nitrate. The amount adsorbed on the surface is determined from the distribution of the activity of the Ag^{111} used for labelling. The activity measurements are carried out using a $\text{Na}(\text{Tl})$ well-type scintillation crystal. The proper pH value is set with nitric acid and/or sodium hydroxide.

3. Adsorption of silver on platinum surface

On the basis of previous experimental data, it seems probable that the concept „adsorption“, in the given case, covers an exchange reaction between the hydrogen atoms bound to the platinum surface and the silver ions being in the solution according to the scheme as follows [4, 5]:



The hydrogen becomes to be deposited on platinum surface during the cathodic polarization of the latter. The adsorption capacity of the surface is 1 or 2 orders of magnitude decreased and fails to be reproducible if such a preliminary cathodic polarization is omitted.

We investigated the dependence of the preliminary treatment of the adsorbent on the adsorbed quantity so as to find the optimum conditions of the separation. Furthermore, the effect of silver-ion concentration on the extent of adsorption has been investigated in order to determine the upper limit of the separation efficiency. Significant differences due to the preliminary treatment and surface conditions in the adsorbed quantities were observed. The adsorbed amounts are decreased to a few per cent of the maximum if the adsorption is performed after the anodic polarization of the platinum.

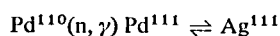
On the contrary, it is noticed in case of previously reduced adsorbents that the silver ions can be quantitatively adsorbed from the solution until the coverage of the geometrical surface reaches 0.26×10^{-6} g of adsorbed silver/cm² on the average (Fig. 1).

We assume the connection between the preliminary treatment and the adsorbed quantity is caused by the rise or removal of a surface oxide layer.

According to Fig. 2, increasing hydrogen ion concentration has no decreasing effect on the adsorption capacity up to about 1 molarity.

4. Separation of silver traces labelled with Ag^{111} from neutron irradiated palladium

The labelling has been carried out making use of the nuclear reaction



induced by thermal neutron irradiation and resulting homogeneously dispersed Ag^{111} in the volume of the target. Irradiated palladium metal (0.1 g) was dissolved in aqua regia and the obtained solution was evaporated to dryness. After driving off tracer amounts of hydrochloric acid the residue was dissolved in 10^{-3} M nitric acid. Preliminary reduced platinum adsorbent having proper adsorption capacity was immersed into the solu-

tion and after stirring for 30 min 95–97 per cent of the Ag^{111} were found to be adsorbed.

In case if the separation of Ag^{111} is desirable the procedure includes rinsing of platinum with redistilled water until the removed water shows any traces of palladium chloride. The desorption proved to be completed after having rinsed the adsorbent with a small volume of 9 M nitric acid.

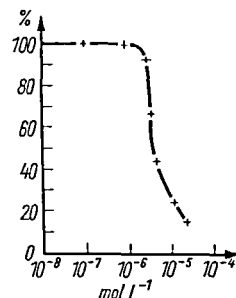


Fig. 1. Adsorbed percentage plotted against initial silver-ion concentration in case of previously reduced adsorbents

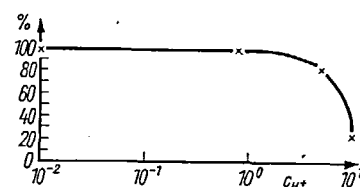


Fig. 2. Extent of adsorption of carrier-free Ag^{111} plotted against hydrogen-ion concentration in case of previously reduced adsorbents

5. Conclusions

1. The procedure based on the selective adsorption method described here is simpler and more effective for the removal of silver traces from palladium than any other one reported previously [6–9].

2. The separation of carrier-free Ag^{111} (produced by neutron irradiation) from the bulk of the palladium target material is of high importance among other practical applications of this method. The separation can be carried out even if the palladium-silver weight ratio exceeds 10^8 .

3. As it follows from point 2 with regards of the production of Ag^{111} the critical impurity in the palladium target is silver because of its effect to decrease the specific activity of the isotope produced. Thus the method can be very useful for purifying palladium targets before irradiation.

4. The authors expect the method to be applicable for the removal of different traces from other macrocomponents as well.

Received July 18, 1966

References

- [1] H. M. Eiland, M. Kahn, J. phys. Chem. 65 (1961) 1317.
- [2] M. Kahn, A. C. Wahl, J. chem. Phys. 21 (1953) 1185.
- [3] V. M. Doctor, Endocrinology 64 (1959) 455.
- [4] J. Molnár, Magyar Kémiai Folyóirat 68 (1962) 498.
- [5] G. Toth, Magyar Kémiai Folyóirat 70 (1964) 361.
- [6] D. N. Sunderman, W. W. Meinke, Anal. Chem. 29 (1957) 1578.
- [7] J. C. Griess, L. B. Rogers, J. electrochem. Soc. 95 (1949) 3129.
- [8] G. Rauser, P. F. Hahn, J. Amer. chem. Soc. 74 (1952) 2398.
- [9] G. K. Schweitzer, D. K. Nehls, J. Amer. chem. Soc. 74 (1952) 6186.