

CLOSED-SYSTEM ANALYSIS OF TRITIUM IN PALLADIUM

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COLD FUSION

TECHNICAL NOTE

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A closed-system procedure for the analysis of tritium in palladium has been developed that has a sensitivity and accuracy of 5×10^7 tritium atoms, corresponding to one tritium atom per 10^{13} palladium atoms for a typical 0.1-g palladium sample. The technique involves palladium dissolution in acid, distillation of the tritiated water, and catalytic oxidation of tritium gas to tritiated water, followed by liquid scintillation counting. This technique is not subject to false tritium findings from a variety of chemical factors or environmental influences that may affect the results of open-system analytical procedures. The closed-system procedure has been applied to nearly 100 as-manufactured palladium wire samples of various lots and sizes from two different sources. None of these samples show any tritium contamination within the detection limit of 5×10^7 tritium atoms. By comparison, others, employing an open-system procedure, have reported tritium contamination in as-manufactured palladium 10 000 times larger than the values obtained by this closed system method.

INTRODUCTION

The question of whether or not tritium found in palladium employed in "cold fusion" experiments has actually been generated by nuclear reactions occurring in the deuterium-loaded palladium or was already present as a contaminant in the palladium is central to the reality of cold fusion. Only a few groups have reported on the analysis of tritium in palladium. Yanakura et al.¹ described an analytical method using heavy-ion Rutherford backscattering while degassing the palladium cathodes at 680 K in vacuum. Matsumoto et al.² monitored the beta radiation from palladium immersed in a liquid scintillator. These authors also employed a temperature-programmed desorption technique coupled with mass spectrometry. Murthy et al.³ employed degassing of the palladium at 680 K and catalytic oxidation of the released tritium over heated copper oxide, followed by scintillation counting. Wolf⁴ employed a method of dissolving palladium in acid, followed by alkaline precipitation, centrifuging, and scintillation counting of the resulting solution.

In a previous technical note,⁵ we described the results of tritium analysis in palladium employing an open-system analytical procedure essentially identical to that used earlier by Wolf.⁴ While we found no evidence for tritium contamination in 45 palladium samples, we concluded that this technique cannot be expected to yield reliable results. We found that falsely high tritium counts may be caused by a number of factors; also, an open-system technique is subject to possible contamination from the environment. On the other hand, falsely low tritium readings can result from the escape of tritium gas formed during the acid digestion of the palladium.

We have, therefore, developed a closed-system procedure that involves dissolving the palladium in acid, distilling the water, and catalytically oxidizing the released tritium gas to water, followed by scintillation counting. Thus, the counting is performed on a clear solution, thereby eliminating the possibility of false tritium counts due to color, precipitated particles, and chemical reactions. Since the procedure is performed in a closed system, gases and vapors cannot escape, nor is the procedure subject to contamination from the environment.

The objective of this technical note is to describe the analytical technique and to present the results of analyzing nearly 100 palladium samples of various diameters and cuttings from two manufacturers for possible tritium contamination. Although the manufacturing process of palladium wires and foils makes it highly unlikely that there would be tritium contamination, we decided to address this issue in depth in view of widely and prematurely publicized statements^{4,6} that tritium found in cold fusion studies results from contamination and not from nuclear reactions in the palladium.

EXPERIMENTAL

Materials and Reagents

The following as-manufactured palladium samples were analyzed for their tritium content:

- 1-, 2-, and 4-mm-diam palladium wires of 99.5% purity from Hoover & Strong (Richmond, Virginia); four different batches of 2-mm wires were used
- 0.5- and 1-mm-diam palladium wires of 99.995% purity from Aesar/Johnson-Matthey (Royston, England); two different batches of 1-mm wires were used

- 1-mm-diam palladium wire of 99.5% purity made by Hoover & Strong, supplied by J. O'M. Bockris at Texas A&M University.

Fuel-cell-grade platinum catalyst on silver-plated nickel screen made by E-Tek, Inc., was used as a recombination catalyst.

The aqua regia solution was prepared by mixing 82 ml of concentrated HCl and 18 ml of concentrated HNO₃ (the amounts recommended for palladium in Ref. 7) using concentrated stock solutions (Mallinckrodt, AR grade). The concentrated and diluted solutions of NaOH were prepared from NaOH pellets (Mallinckrodt, AR grade).

Apparatus

A closed system for the distillation apparatus was assembled from KONTES (Vineland, New Jersey) microscale glassware and polypropylene/polytetrafluoroethylene connections. The apparatus is shown in Fig. 1. It comprises the following:

1. a distillation flask that contains 1 to 1.5 ml of aqua regia and the 20 to 200 mg of palladium to be digested
2. a standard water-cooled condenser
3. a glass tube leading from the condenser to the bottom of the receiving flask. The glass tube is surrounded by a glass envelope, sealed at the top but open to the receiving flask. The envelope contains platinum catalyst on two pieces of silver-plated nickel screen of 1 to 2 cm² area each.
4. a receiving flask, containing 1 ml of a 5% aqueous H₂O₂ solution.

Tritium measurements were performed by liquid scintillation counting using a Beckman LS 5000 TD system, as described in our previous technical note.⁵

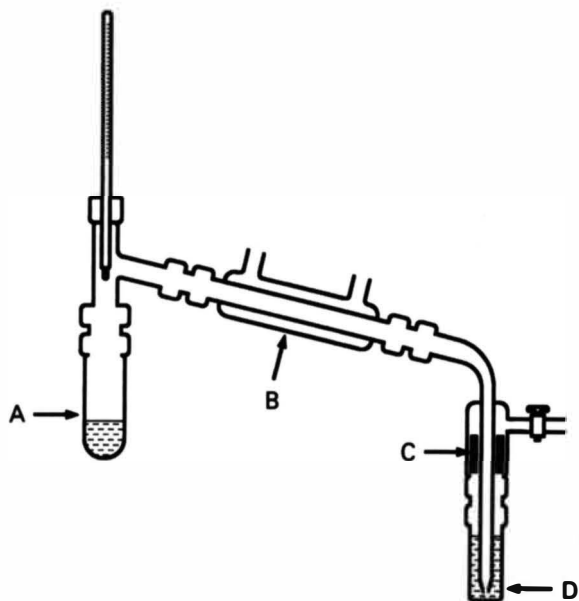


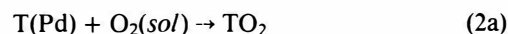
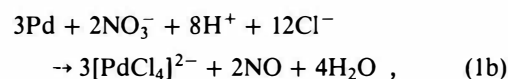
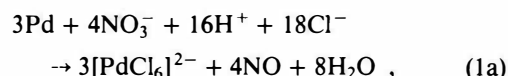
Fig. 1. Distillation apparatus for analysis of tritium in palladium with (A) distillation flask, (B) condenser, (C) catalyst, and (D) receiving flask.

Wet-Chemical Procedure

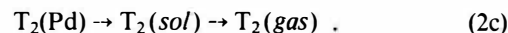
The palladium sample (20 to 200 mg) was placed in the distillation flask and 1 ml of H₂O₂ solution was injected into the receiving flask. With a syringe and septum, 1 to 1.5 ml of aqua regia solution was injected into the distillation flask to dissolve the palladium. The system was then hermetically sealed. After the palladium was dissolved (~ 2 h), the solutions in the flask were neutralized with concentrated NaOH and heated in a mantle to ~110°C until all the water from the flask was distilled. Then 1 ml of deionized water was added to the flask and distilled again, until only dry residue remained. Tritium occluded by the palladium is released into the aqua regia during dissolution of the palladium as gas or is oxidized to T₂O or THO. The T₂ gas recombines on the catalyst to form T₂O, and the T₂O and THO vapors are condensed to the liquid state. This procedure allows us to collect the entire amount of tritium occluded by the palladium in the receiving flask.

The reactions occurring during the distillation/recombination process are as follows:

1. In the distillation flask:



and



2. On the platinum catalyst:



and



3. In the receiving flask:



Following completion of the distillation, the receiving flask was heated to 60°C for 30 min to expel any dissolved T₂ gas for recombination with O₂ on the platinum catalyst. The distillation flask was then removed from the condenser, and the condenser and catalyst were rinsed with deionized water to wash any tritiated water from these parts into the receiving flask. The contents of the flask were diluted to precisely 5 ml in a volumetric flask. One millilitre of this dilute nitric acid solution was added to 10 ml of scintillation cocktail (pseudo cumene/xylene from Beckman), which was then subjected to tritium counting in the Beckman system.

Scintillation Counting

The Beckman system was calibrated employing a procedure described in Ref. 5. To verify that the window settings were correct for tritium, we verified that the liquid scintillation counter measured and displayed the correct energy spectrum for tritium. Such a spectrum is shown in Fig. 2 for a tritium standard supplied by Beckman with the instrument.

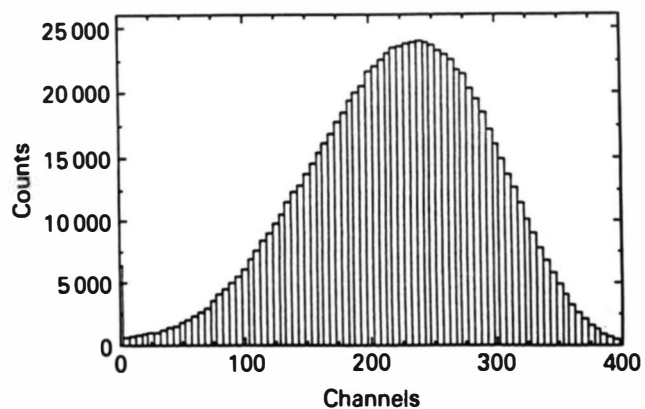


Fig. 2. Energy spectrum of tritium standard.

Verification of Procedure

To verify the correctness and linearity of tritium counting, the tritium activity of standard solutions with known tritium concentrations was determined. The results are plotted in Fig. 3 and show a linear dependency of the tritium counts (dpm/ml values) on the activity of added tritium ($\mu\text{Ci} \times 10^{-6}$), as required.

To establish the efficiency of T_2O transfer from the distillation to the receiving flask, a known amount of T_2O was added to the aqua regia, a distillation carried out, and tritium counting performed, maintaining the identical procedure as in later actual analyses. We obtained 98% distillation efficiency referred to the tritium.

In a second control experiment, the catalytic recombination of gas with O_2 was verified. For this purpose, a 0.1 M aqueous NaOH solution was tritiated by adding various known amounts of T_2O . A glass cell containing this solution and two platinum electrodes was connected to the distillation apparatus, taking the place of the distillation flask. Constant current electrolysis was performed for various lengths of time. The evolving H_2 and T_2 were recombined to H_2O and

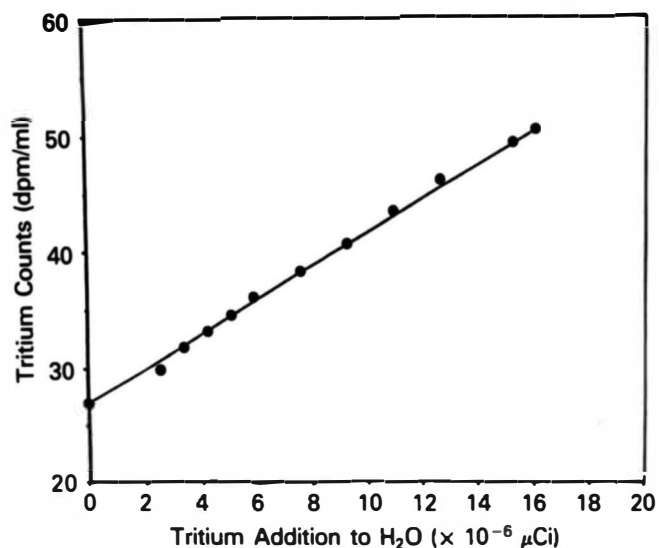
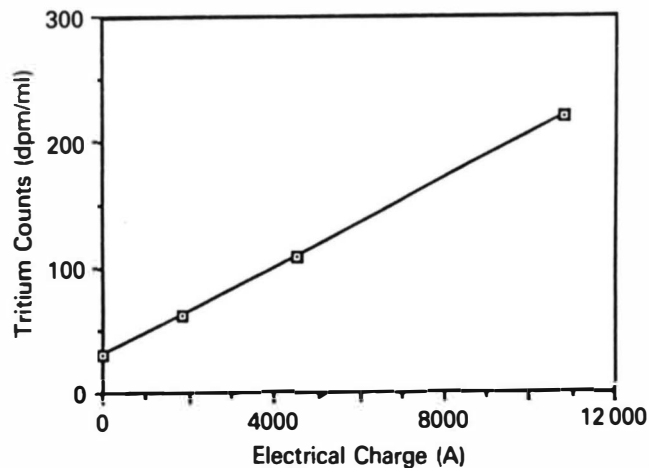
Fig. 3. Calibration curve for H_2O with known tritium contents.

Fig. 4. Dependency of tritium counts on electrical charge when electrolyzing a tritiated NaOH solution.

T_2O , collected in the receiving flask, and analyzed for tritium in the Beckman scintillation counter. Figure 4 shows a linear dependency of the tritium counts in the recombine on the electrical charge passed through the electrolytic cell. This establishes that the catalyst converts tritium gas to T_2O or THO as expected.

RESULTS AND DISCUSSION

The results of tritium analysis on 90 as-manufactured palladium samples and 30 background samples are presented in Table I. The tritium background of the 30 scintillation liquid samples was determined to be 27.1 ± 1.1 dpm/ml, and the overall tritium count average of the 90 palladium samples was 27.2 ± 1.1 dpm/ml, identical to background. Thus, none of the 90 palladium samples^a was found to contain any tritium contamination, in spite of the fact that they were cut from ten different batches of palladium wire of various diameters, supplied by two different manufactures. For each palladium sample, the dpm/ml values shown are averages from at least three measurements.

The sensitivity and accuracy of tritium determination, employing the closed-system analytical procedure is ± 1 dpm/ml, equivalent to 9.28×10^6 tritium atom/ml. For our 5-ml solution volume and for a typical 0.1-g palladium sample, this corresponds to a detection limit of 5×10^7 tritium atoms or ~ 1 tritium atom/ 10^{13} palladium atoms.

CONCLUSIONS

We have developed a closed-system analytical technique for the analysis of tritium in palladium that features high reliability and accuracy. This technique employs a distillation and catalytic gas recombination procedure that leads to the quantitative determination of the tritium content of palladium with an accuracy and sensitivity of 5×10^7 tritium atoms. Application of this technique to 90 palladium wire samples of various lots and sizes, manufactured by two

^aThese measurements have now been extended to >130 palladium samples, and the same results have been obtained on all of these.

TABLE I
Tritium Analysis in Palladium

Palladium Manufacturer	Characteristics of Samples			Tritium Average (dpm/ml)	Standard Deviation
	Wire Diameter (mm)	Number of Cuttings	Number of Samples		
Hoover & Strong	1	8	14	26.9	1.0
Hoover & Strong	2	7	12	27.4	1.4
Hoover & Strong	2	7	12	27.5	0.4
Hoover & Strong	2	8	12	26.8	0.8
Hoover & Strong	2	1	2	27.4	
Hoover & Strong	4	7	12	27.7	1.2
Hoover & Strong ^a	1	1	2	27.7	
Johnson Matthey	0.5	3	6	27.8	1.0
Johnson Matthey	1	4	7	26.8	0.7
Johnson Matthey	1	6	11	26.9	1.5
Total			90	27.2	1.1
Background-scintillation liquid			30	27.1	1.1

^aObtained from Texas A&M University.

different suppliers, yields no tritium contamination in any of these samples.

While our results do evidently not exclude the possibility of rare spot contamination of palladium with tritium, such contamination is exceedingly unlikely both in view of our results and the details of the palladium manufacturing process.

The closed-system analytical procedure described here is not affected by the shortcomings of open-system techniques, such as falsely low or high tritium readings. Application of the closed-system procedure for tritium analysis of palladium prior to use in cold fusion studies is advisable to ascertain that there is no possibility for tritium contamination.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance of Y. Zhang, B. Nkamsi, and X. Du in carrying out the analysis and the receipt of palladium samples from J. O'M. Bockris and S. Guruswamy as well as the financial support from the state of Utah.

REFERENCES

I. M. YANOKURA, M. MINAMI, S. YAMAGATA, S. NAKABAYASHI, M. ARATANI, A. KIRA, and I. TANIHATA,

"An Approach to the Cold Fusion Through Hydrogen Isotopes Analysis by the Heavy Ion Rutherford Scattering," *Chem. Lett.*, **12**, 2197 (1989).

2. O. MATSUMOTO, K. KIMURA, Y. SAITO, H. UYAMA, and T. YAITA, "Detection of Tritium in Cathode Materials After the Electrolysis of D₂SO₄-D₂O Solution," *Denki Kagaku*, **58**, 471 (1990).

3. T. S. MURTHY, T. S. IYENGAR, B. K. SEN, and T. B. JOSEPH, "Tritium Analysis of Samples Obtained from Various Electrolysis Experiments at BARC," *Fusion Technol.*, **18**, 71 (1990).

4. K. L. WOLF, "Cold Fusion and Hot Tritium," Transcript of talk given August 11, 1990, at Stanford University.

5. K. CEDZYNSKA, S. C. BARROWES, H. E. BERGESON, L. C. KNIGHT, and F. G. WILL, "Tritium Analysis in Palladium with an Open-System Analytical Procedure," *Fusion Technol.*, **20**, 108 (1991).

6. R. POOL, "Wolf: My Tritium Was an Impurity," *Sci.*, **248**, 1301 (June 15, 1990).

7. J. W. MELLOR, *A Comprehensive Treatise on Inorganic and Theoretical Chemistry*, Vol. 15, p. 595, Longmans, Green & Company, London (1936).

**Closed-System Analysis of Tritium
in Palladium**

by

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Abstract

We have developed a closed-system procedure for the analysis of tritium in palladium which has a sensitivity and accuracy of 5×10^7 tritium atoms, corresponding to one tritium atom per 10^{13} palladium atoms for a typical 0.1g palladium sample. The technique involves palladium dissolution in acid, distillation of the tritiated water and catalytic oxidation of tritium gas to tritiated water, followed by liquid scintillation counting. This technique is not subject to false tritium findings from a variety of chemical factors or environmental influences which may affect the results of open-system analytical procedures. We have applied the closed-system procedure to nearly 100 as-manufactured palladium wire samples of various lots and sizes from two different sources. None of these samples show any tritium contamination within our detection limit of 5×10^7 tritium atoms. By comparison, others, employing an open-system procedure, have reported tritium contamination in as-manufactured palladium 10,000 times larger than our values.

Introduction

The question of whether or not tritium found in palladium, employed in "cold fusion" experiments, has actually been generated by nuclear reactions occurring in the deuterium-loaded palladium or was already present as a contaminant in the palladium is central to the reality of cold fusion.

There appear to be only few groups that have recently reported on the analysis of tritium in palladium. M. Yanakura et al. (1) described an analytical method using heavy ion Rutherford back-scattering while degassing the Pd cathodes at 680°K in vacuum. O. Matsumoto et al. (2) monitored the beta radiation from Pd, immersed in a liquid scintillator. These authors also employed a temperature-programmed desorption technique, coupled with mass spectrometry. T.S. Murthy et al (3) employed degassing of the palladium at 680°K and catalytic oxidation of the released tritium over heated copper oxide, followed by scintillation counting. K. Wolf (4) employed a method of dissolving palladium in acid, followed by alkaline precipitation, centrifuging and scintillation counting of the resulting solution.

In a previous paper (5), we described the results of tritium analysis in palladium, employing an open-system analytical procedure, essentially identical to that used earlier by Wolf (4). While we found no evidence for tritium contamination in 45

palladium samples, we concluded that this technique cannot be expected to yield reliable results. We found that falsely high tritium counts may be caused by a number of factors; also, an open-system technique is subject to possible contamination from the environment. On the other hand, falsely low tritium readings can result from the escape of tritium gas, formed during the acid digestion of the palladium.

We have, therefore, developed a closed-system procedure which involves dissolving the palladium in acid, distilling the water and catalytically oxidizing the released tritium gas to water, followed by scintillation counting. Thus, the counting is performed on a clear solution, thereby eliminating the possibility of false tritium counts due to color, precipitated particles and chemical reactions. Since the procedure is performed in a closed system, gases and vapors cannot escape, nor is the procedure subject to contamination from the environment.

The objective of this paper is to describe the analytical technique and present the results of analyzing nearly 100 palladium samples of various diameters and cuttings from two manufacturers for possible tritium contamination. Although the manufacturing process of palladium wires and foils makes it highly unlikely that there would be tritium contamination, we decided to address this issue in depth in view of widely and prematurely publicized statements (4,6) that tritium found in "cold fusion" studies results from contamination and not from nuclear reactions in the palladium.

Experimental

Materials and Reagents

The following as-manufactured palladium samples were analyzed for their tritium content:

1. 1, 2 and 4mm diameter Pd wires of purity 99.5% from Hoover & Strong (Richmond, Virginia); four different batches of 2mm wires were used.
2. 0.5 and 1mm diameter Pd wires of purity 99.995% from Aesar/Johnson Matthey (Royston, England); two different batches of 1mm wires were used.
3. 1mm diameter Pd wire of purity 99.5% made by Hoover & Strong, supplied by J. O'M Bockris at Texas A & M University.

Fuel cell grade Pt catalyst on Ag-plated Ni screen (ESN), made by E-Tek, Inc., was used as a recombination catalyst.

The aqua regia solution was prepared by mixing 82 ml of concentrated HCl and 18 ml of concentrated HNO₃ (the amounts recommended for palladium in *Inorganic and Theoretical Chemistry*, p., 595), using concentrated stock solutions (Mallinckrodt, AR grade). The concentrated and diluted solutions of NaOH were prepared from NaOH pellets (Mallinckrodt, AR grade).

Apparatus

A closed system for the distillation apparatus was assembled from KONTES (Vineland, New Jersey) microscale glassware and polypropylene/PTFE connections. The apparatus is shown in Figure 1. It comprises the following: (A) A distillation flask which contains 1 to 1.5 ml aqua regia and the 20 to 200 mg Pd to be digested. (B) A standard water-cooled condenser. (C) A glass tube leading from the condenser to the bottom of the receiving flask; the glass tube is surrounded by a glass envelope, sealed at the top, but open to the receiving flask. The envelope contains Pt catalyst on two pieces of Ag-plated Ni screen of 1 to 2 cm² area each. (D) A receiving flask, containing 1 ml of a 5% aqueous H₂O₂ solution.

Tritium measurements were performed by liquid scintillation counting (LSC), using a Beckman LS 5000 TD system, as described in our previous paper (5).

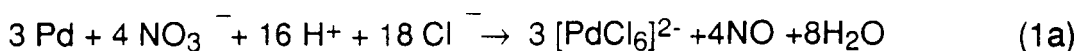
Wet-Chemical Procedure

The Pd sample (20-200 mg) was placed in the distillation flask, 1 ml H₂O₂ solution injected into the receiving flask and, using a syringe and septum, 1 to 1.5 ml of aqua regia solution was injected into the distillation flask to dissolve the Pd. The system was then hermetically sealed. After the Pd was dissolved (approximately 2 hours), the solutions in the flask were neutralized with concentrated NaOH and heated in a mantle to approximately 110°C until all the water from the flask was distilled. Then 1 ml of deionized water was added to the flask and distilled again until only dry residue remained. Tritium, occluded by the Pd, is released into the aqua regia during dissolution of the Pd as gas or is oxidized to T₂O or THO. The T₂ gas recombines on the catalyst to form T₂O, and the T₂O and THO vapors are condensed to the liquid

state. This procedure allows us to collect the entire amount of T, occluded by the Pd, in the receiving flask.

The reactions occurring during the distillation/recombination process are as follows.

1. In the distillation flask:



2. On the Pt catalyst:



3. In the receiving flask:



Following completion of the distillation, the receiving flask was heated to 60°C for 30 minutes to expel any dissolved T₂ gas for recombination with O₂ on the Pt catalyst. The distillation flask was then removed from the condenser and the condenser and catalyst rinsed with deionized water to wash any tritiated water from these parts into the receiving flask. The contents of the flask were diluted to precisely 5 ml in a volumetric flask. One ml of this dilute nitric acid solution was added to 10ml of scintillation cocktail (pseudo cumene/xylene from Beckman), which was then subjected to tritium counting in the Beckman system.

Scintillation Counting

The Beckman was calibrated, employing a procedure described in an earlier paper (5). To verify that the window settings were correct for tritium, we verified that

the Liquid Scintillation Counter measured and displayed the correct energy spectrum for tritium. Such a spectrum is shown in Figure 2 for a tritium standard, supplied by Beckman with the instrument.

Verification of Procedure

To verify the correctness and linearity of tritium counting, the tritium activity of standard solutions with known tritium concentrations was determined. The results are plotted in Figure 3 and show a linear dependency of the tritium counts (DPM/ml values) on the activity of added tritium ($\mu\text{Ci} \times 10^{-6}$), as required.

To establish the efficiency of T_2O transfer from the distillation to the receiving flask, a known amount of T_2O was added to the aqua regia, a distillation carried out and tritium counting performed, maintaining the identical procedure as in later actual analyses. We obtained 98% distillation efficiency, referred to the T.

In a second control experiment, the catalytic recombination of gas with O_2 was verified. For this purpose, a 0.1 molar aqueous NaOH solution was tritiated by adding various known amounts of T_2O . A glass cell, containing this solution and two Pt electrodes was connected to the distillation apparatus, taking the place of the distillation flask. Constant current electrolysis was performed for various lengths of time, the evolving H_2 and T_2 recombined to H_2O and T_2O , collected in the receiving flask and analyzed for tritium in the Beckman scintillation counter. Figure 4 shows a linear dependency of the tritium counts in the recombine on the electrical charge passed through the electrolytic cell. This establishes that the catalyst converts T gas to T_2O or THO as expected.

Results and Discussion

The results of tritium analysis on 90 as-manufactured palladium samples and 30 background samples are presented in Table 1. The tritium background of the 30 scintillation liquid samples was determined as 27.1 ± 1.1 DPM/ml, and the overall tritium count average of the 90 palladium samples was 27.2 ± 1.1 DPM/ml, identical to back-ground. Thus, none of the 90 Pd samples* was found to contain any tritium contamination, in spite of the fact that they were cut from ten different batches of Pd

* These measurements have now been extended to over 130 Pd samples, and the same results have been obtained on all of these.

wire of various diameters, supplied by two different manufactures. For each Pd sample, the DPM/ml values shown are averages from at least three measurements.

The sensitivity and accuracy of tritium determination, employing the closed-system analytical procedure is ± 1 DPM/ml, equivalent to 9.28×10^6 tritium atoms per ml. For our 5 ml solution volume and for a typical 0.1g Pd sample, this corresponds to a detection limit of 5×10^7 tritium atoms or approximately 1 tritium atom per 10^{13} Pd atoms.

Table 1
Tritium Analysis in Palladium

<u>PALLADIUM MANUFACTURER</u>	<u>CHARACTERISTICS OF SAMPLES</u>			<u>TRITIUM AVERAGE</u>	<u>STANDARD DEVIATION</u>
	Wire Diameter (mm)	No. of Cuttings	No. Of Samples	DPM/ml	
Hoover & Strong	1	8	14	26.9	1.0
Hoover & Strong	2	7	12	27.4	1.4
Hoover & Strong	2	7	12	27.5	0.4
Hoover & Strong	2	8	12	26.8	0.8
Hoover & Strong	2	1	2	27.4	
Hoover & Strong	4	7	12	27.7	1.2
Hoover & Strong*	1	1	2	27.7	
Johnson Matthey	0.5	3	6	27.8	1.0
Johnson Matthey	1	4	7	26.8	0.7
Johnson Matthey	1	6	11	26.9	1.5
<hr/>					
Total			90	27.2	1.1
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Background - Scintillation Liquid			30	27.1	1.1

* Obtained from Texas A & M University

Conclusions

We have developed a closed-system analytical technique for the analysis of tritium in palladium which features high reliability and accuracy. This technique employs a distillation and catalytic gas recombination procedure which leads to the quantitative determination of the tritium content of palladium with an accuracy and sensitivity of 5×10^7 T atoms. Application of this technique to 90 palladium wire samples of various lots and sizes, manufactured by two different suppliers, yields no tritium contamination in any of these samples.

While our results do evidently not exclude the possibility of rare spot contamination of Pd with tritium, such contamination is exceedingly unlikely both in view of our results and the details of the Pd manufacturing process.

The closed-system analytical procedure, described here, is not affected by the shortcomings of open-system techniques, such as falsely low or high tritium readings. Application of the closed-system procedure for tritium analysis of Pd prior to use in "cold fusion" studies is advisable to ascertain that there is no possibility for tritium contamination.

Acknowledgements

The authors gratefully acknowledge the assistance of Y. Zhang, B. Nkamsi and X. Du in carrying out the analysis, the receipt of palladium samples from Drs. J. O'M. Bockris and S. Guruswamy as well as the financial support from the State of Utah.

References

1. M. Yanakura, M. Minami, S. Yamagata, S. Nakabayashi, M. Aratani, A. Kira, I. Tanihata, "An Approach To The Cold Fusion Through Hydrogen Isotopes Analysis By the Heavy Ion Rutherford Scattering," Chemistry Letters, 2197 (1989).
2. O. Matsumoto, K. Kimura, Y. Saito, H. Uyama and T. Yaita, "Detection of Tritium in Cathode Materials after the electrolysis of D_2SO_4 - D_2O Solution", Denki Kagaku, 58, 471 (1990)

3. T.S. Murthy, T.S. Iyengar, B.K. Sen, and T.B. Joseph, "Tritium Analysis of Samples Obtained From Various Electrolysis Experiments At BARC," *Fusion Technology*, 18, 71 (1990).
4. K.L. Wolf, "Cold Fusion And Hot Tritium, " transcript of talk given August 11, 1990, at Stanford University.
5. K. Cedzynska, S. C. Barrowes, H.. E. Bergeson, L. C. Knight and F. G. Will, "Tritium Analysis in Palladium With an Open-System Analytical Procedure" to appear in "Fusion Technology", Aug. 1991.
6. R. Pool, "Wolf: My Tritium was an Impurity", *Science*, 248, 1301; 15 June 1990

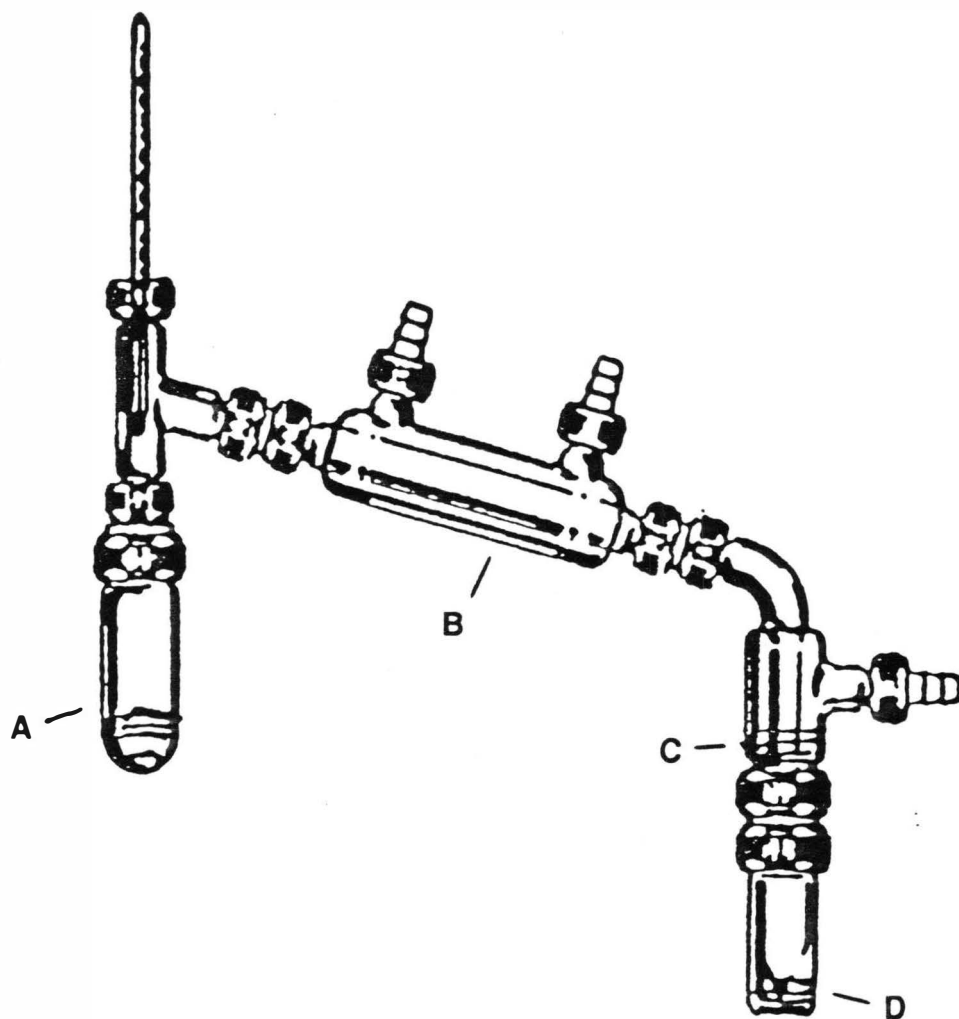


Figure 1: Distillation apparatus for analysis of tritium in palladium with distillation flask A, condenser B, catalyst C, receiving flask D.

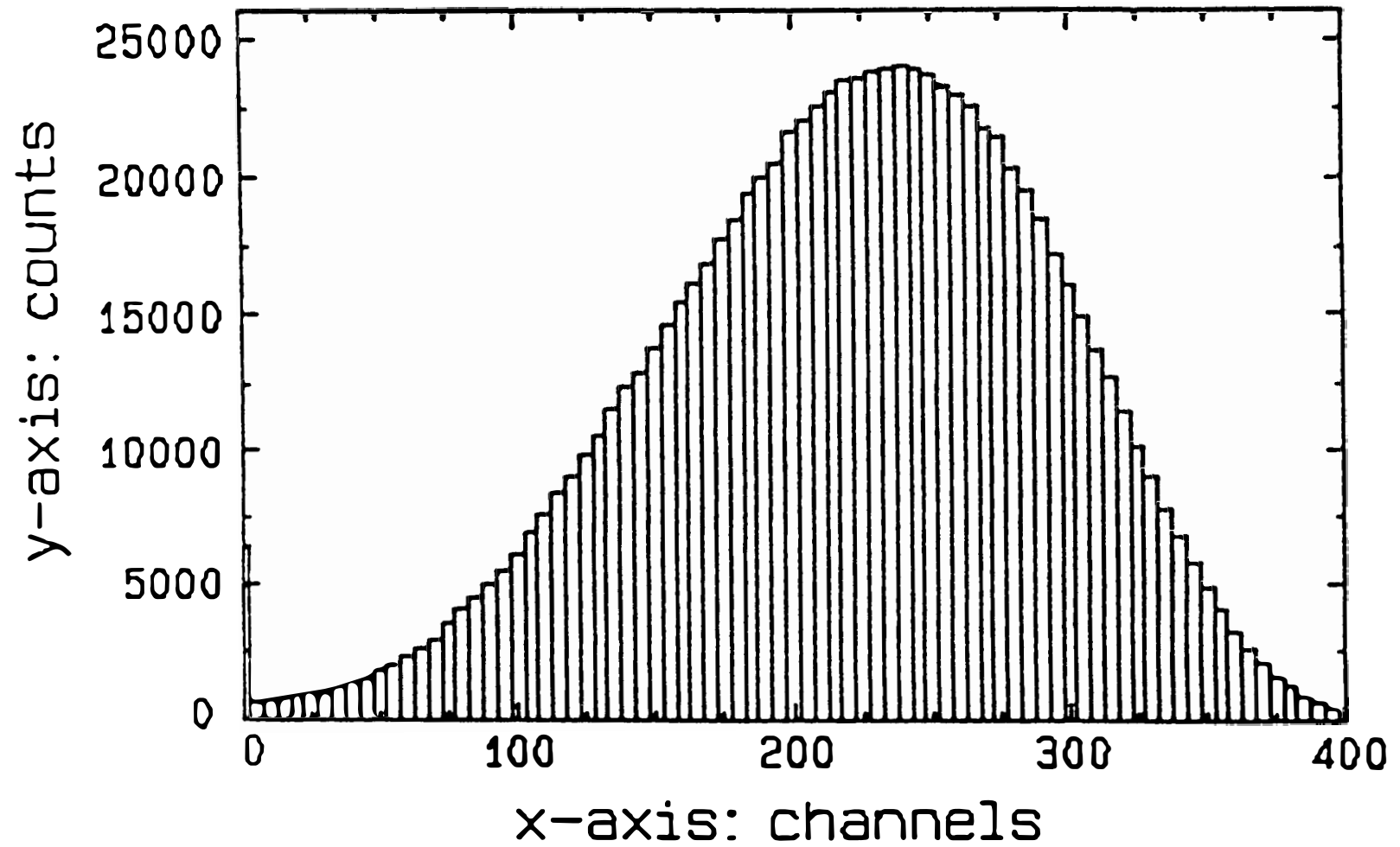


Figure 2: Energy Spectrum of Tritium Standard.

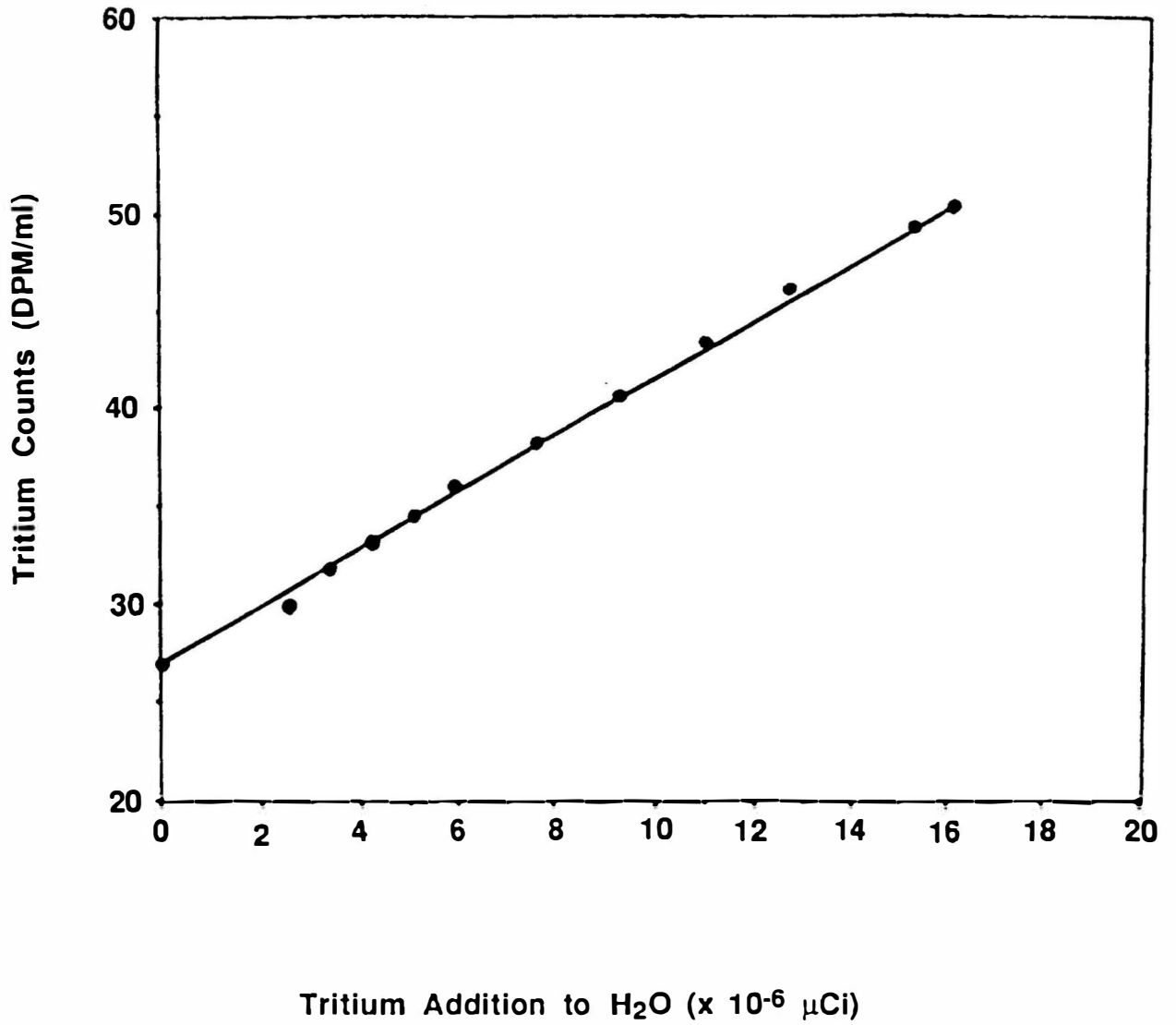


Figure 3: Calibration curve for H₂O with known tritium contents.

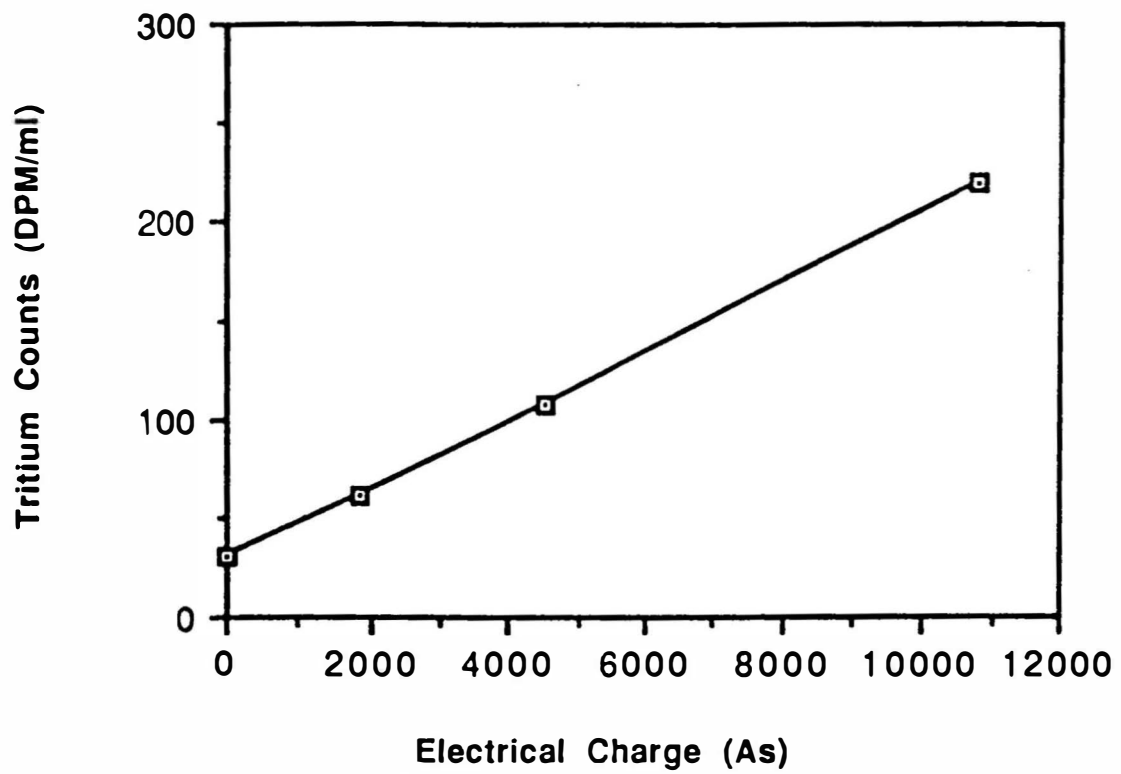


Figure 4: Dependency of tritium counts on electrical charge when electrolyzing a tritiated NaOH solution.