PdX®

Palladium API Screening Fluorescent Detection Kit

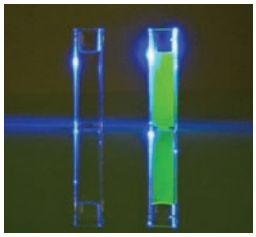
Catalog Number K007-F1

ARBOR ASSAYS

KIT INSERT

FEATURES

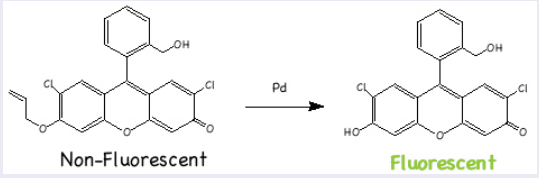
- 30 minute assay to detect Pd contamination. HTS compatible
- Works with APIs in HCl, NMP, MeCN, DMF, Ethanol and Toluene
- Works on any fluorescent plate reader



INTRODUCTION

In recent years, many new synthetic transformations have been developed that use palladium (Pd) compounds for the catalysis of carbon-carbon and carbon-heteroatom coupling reactions such as the Buchwald-Hartwig, Heck, Sonogashira, Suzuki-Miyaura, and Tsuji-Trost transformations. These reactions have found increased popularity for pharmaceutical processes as they utilize a wide-range of functional groups to build complicated molecules. However palladium-catalyzed reactions present a problem in that the palladium can often be retained in the isolated API product. Current FDA and EAEMP regulations limit all platinum group (Pt, Pd, Ir, Rh, Ru, Os) metal contamination (as a group) to less than 5-10 ppm. The standard methods of quantifying palladium in APIs are atomic absorption analysis, x-ray fluorescence, or inductively-coupled plasma mass spectroscopy (ICP-MS). These expensive instruments dictate a highly trained scientist to operate, suffer from cross-contamination of the probe and require scrupulous clean up methodology.

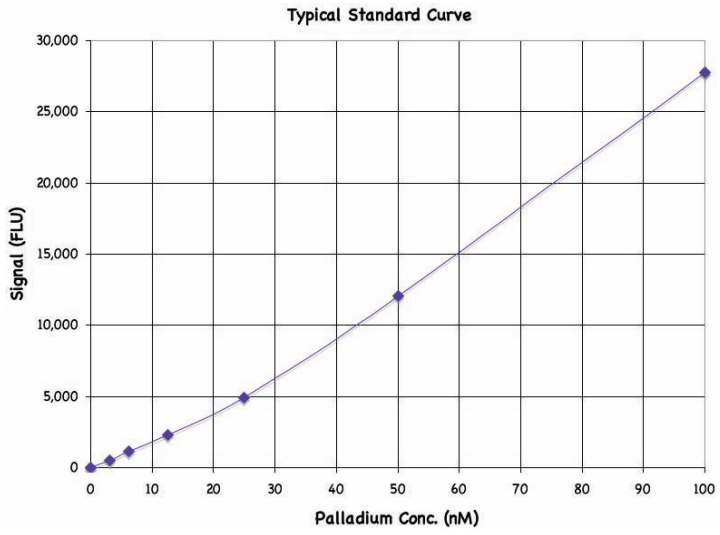
The PdX® Palladium API Fluorescent Detection Kit is designed to allow rapid screening of methods for Pd removal from solutions of APIs in acetonitrile, DMF, NMP, Ethanol, dilute HCl and Toluene. Standards or diluted samples are pipetted into a microtiter plate along with supplied Detection Reagent and supplied Sodium Borohydride reducing agent. After 30 minutes the intensity of the generated fluorescence is read at 525 nm in a microtiter plate reader. **Some API samples may bind the Pd catalyst so strongly that acid microwave digestion will be required.**



SAMPLE TYPES

Various diverse palladium catalysts in oxidation states (0) and (II) have been tested in a variety of organic solvents including aceonitrile, DMF, NMP, Ethanol, dilute Hydrochloric Acid and Toluene.

TYPICAL DATA



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SAMPLE PREPARATION

Toluene samples at 20 mg/mL should be diluted 1:20 into DMF before being diluted in Sample Diluent. All samples should be at 1 mg/mL before being diluted 1:30 in provided Sample Diluent. Acid digests should be diluted in Sample Diluent at least 100 fold prior to being added to the assay.

ASSAY PROTOCOL

- Pipet 100 µL of samples or standards into duplicate wells in the black microtiter plate.
- Add 25 µL of the PdX® Palladium Detection Reagent to each well.
- Add 25 µL of the Sodium Borohydride Reagent to each well.
- Incubate at room temperature for 30 minutes.
- Read fluorescence intensity at 525 nm with excitation at 495nm.

KIT INSERT

SENSITIVITY

Sensitivity was determined as 0.082 nM.

LINEARITY

Linearity was determined by taking two palladium catalyst samples in toluene, diluted 1:20, and then diluted in Sample Diluent, one with a low diluted Pd level of 7.6 nM and one with a higher diluted level of 59.5 nM and mixing them in the ratios given below:

Low Pd Sample %	High Pd Sample %	Expected Conc.	Observed Conc.	% Recovery
80%	20%	18.0	17.2	95.7%
60%	40%	28.4	27.5	97.0%
40%	60%	38.7	34.0	87.8%
20%	80%	49.1	44.2	90.0%
			Mean Recovery	**92.6%**

INTRA ASSAY PRECISION

Three different palladium catalyst samples were diluted with Sample Diluent and run in replicates of n=20 in an assay. The mean and standard deviation of the calculated Pd concentrations were:

Sample	Pd Conc. (nM)	Standard Deviation (nM)	%CV
1	16.6	0.7	4.1
2	56.0	2.9	5.2
3	83.0	4.6	5.5

INTER ASSAY PRECISION

Three different palladium catalyst samples were diluted with Sample Diluent and run in duplicates in twenty assays over two days by four operators. The mean and standard deviation of the calculated Pd concentrations were:

Sample	Pd Conc. (nM)	Standard Deviation (nM)	%CV
1	16.5	1.7	10.4
2	48.3	4.8	9.9
3	76.4	11.4	14.9

INTERFERANTS

To assess the effect of colored API's on the measured palladium concentration, we took three FD&C food color preparations representing red, yellow and blue colored materials. These were added at 1:800 and 1:3200 dilutions to palladium catalyst solutions in Sample Diluent. Interference was only seen when either red or blue colored materials were present in the neat solutions at concentrations equivalent to optical densities of ~120 at 460-450 nm and ~60 at 500-540 nm.

For details concerning this kit or to order any of our products please contact us:
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