5% Pd/C – Precise but Vague

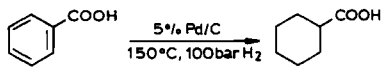
For liquid phase batch hydrogenations catalysed by supported platinum group metals (pgms), the most frequently used combination of metal and support is 5% palladium/carbon (Pd/C) paste (1). Catalytic hydrogenation is very widely used in industrial organic chemistry and includes reactions as diverse as carbon-carbon multiple bond reduction; aromatic ring saturation; reduction of carbonyl groups to alcohols or hydrocarbons; reduction of nitro and nitroso compounds, imines and nitriles to amines; reductive aminations/alkylations; hydrogenolysis, for example, hydrodehalogenation, removal of protecting groups; etc.

For some pgm catalysed hydrogenations, Pd would not necessarily be the metal of choice. For example, the reduction of aliphatic aldehydes to primary alcohols is best performed with a ruthenium catalyst (2).

However, in practice, about three quarters of all liquid phase batch hydrogenations which use a heterogeneous pgm catalyst are in fact performed with 5% Pd/C paste. Some of the variables exploited in the manufacture of any given 5% Pd/C paste include: source of the activated carbon support; pretreatment (if any) of the support; the nature of the soluble Pd salt used to impregnate the support; the nature and form of the reducing agent used to produce the Pd metal crystallites on the support; pH and temperature of both the Pd salt impregnation and reduction steps; rate and order of addition of the reagents; efficiency of the final washing/filtration step; etc.

Clearly a vast number of combinations of the above is possible, all capable of yielding an end product that could accurately be described as '5% Pd/C paste' with each being assigned some unique identification code by the manufacturer.

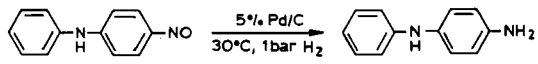
One of the key parameters that can be controlled is the location of the Pd on the support. At one extreme the Pd can be entirely located at the surface of the individual carbon particle: eggshell metal distribution. At the other extreme the Pd can be evenly distributed throughout the particle: uniform distribution. Somewhere between these two extremes is the intermediate distribution. It does not follow that catalysts with eggshell distributions are invariably the most active. For example:



Pd distribution	Eggshell	Intermediate	Uniform
% Conversion after 1 hour	10	60	85

At higher pressures, catalysts with the same total metal loading but deeper metal locations tend to be more active than those with eggshell distributions because of their higher metal dispersions.

In some extreme cases, certain 5% Pd/C paste formulations have no catalytic activity whatsoever and others can show a wide variation in reaction rate. For the reaction below, performed in an alkaline methanol solvent, the following relative reaction rates have been observed:



Types of 5% Pd/C paste catalyst	1	2	3	4	5
Relative reaction rate	0	38	10	7	4

To achieve reproducible results, it is absolutely essential therefore to ensure that the same catalyst formulation, identified by the manufacturer's code, be used throughout any evaluation programme.

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References

1 D. E. Grove, *Platinum Metals Rev.*, 2002, 46, (1), 48
2 See for example, "The Catalyst Technical Handbook", Johnson Matthey, 2001

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