Biotage® MP-Borohydride

Reducing Agent

Key Facts

Stoichometric | Shelf Life | Capacity (mmol/g) | BSE/TSE | Scalable | Particle Size (µm) | Thermally & Mechanically Stable | Good Laboratory Practice | Bulk Density (g/L)

Specifications

Chemical Name:	Macroporous triethylammonium methylpolystyrene triacetoxyborohydride
Resin Type:	Macroporous polystyrene
Application:	Reduction of carbonyl compounds, azides and oximes; reductive reduction of conjugated enones to unsaturated alcohols.
Typical Conditions for Aldehyde and Ketone Reduction:	1.0 mmol of carbonyl compound in ethanol or methanol and 0.5 mmol of MP-Borohydride stirred at room temperature for 2–12 h, depending on the nature of the carbonyl compound. Product isolated by filtration to remove the resin.
Typical Conditions for Reductive Amination:	1.2 mmol of carbonyl compound, 1.0 mmol of primary or secondary amine in ethanol and 0.1 mL of acetic acid stirred for 4 h at room temperature, followed by 1.5 mmol of MP-Borohydride with gentle agitation overnight at room temperature. Product isolated by filtration to remove the resin.
Compatible Solvents:	THF (2.9 mL/g), DCM (3.4 mL/g), MeOH (3.4 mL/g), DMF (2.9 mL/g).
Storage:	We recommend storage in a closed container at 5 °C. MP-Borohydride is stable at room temperature for at least 12 months.

Biotage® MP-Borohydride is a macroporous, solid-supported borohydride that is a solid-supported equivalent of tetraalkylammonium borohydride. The bound borohydride is a versatile reducing agent[1,2,3] used for the reduction of carbonyl compounds and imines, and the reductive amination of aldehydes and ketones. The resin, in conjunction with some transition metal salts, can also be used for a number of other important reductive applications,[4,5,6] such as reduction of oximes, azides, and alkyl halides. The reduced products are isolated by simple filtration from the resin.

MP-BH_4 may be handled under bench conditions, and dispensed using standard laboratory equipment (spatulas, Argoscoops). Reactions may be run using microwave or traditional heating technologies and these can be agitated mechanically (overhead stirring, shaker, blood rotatory wheel) or by gently magnetic stirring.

In addition to crystallization and flash chromatography, reductive amination products can be purified by catch-and-release of the amines with MP-TsOH.[7] In the case of reductive amination using an excess of primary amine, PS-Benzaldehyde can be used to scavenge the excess starting primary amine from the product secondary amine.

MP-BH_4 and transition metal auxiliaries can be used in reductive applications [6,7,8] such as reduction of oximes, azides, and alkyl halides. With titanium isopropoxide, it can be used in reductive amination under neutral conditions (Figure 1b)[9]. This facilitates reaction with more difficult substrates (enolizable carbonyls, acetophenones, sterically hindered substrates or acid sensitive groups).

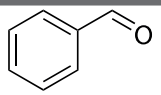
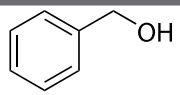
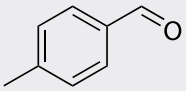
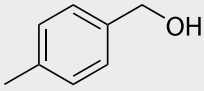
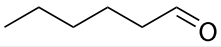
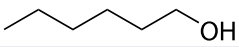
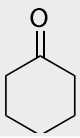
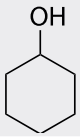
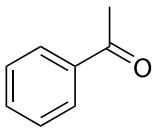
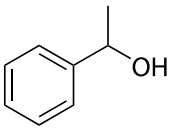
Entry	Carbonyl Compound	Reduced Product	Time (h)	% Yield (isolated)	% Purity (GC)
1			3	89	98
2			3	85	100
3			3	80	98
4			8	85	96
5			14	-	6

Table 1. Reduction of carbonyl compounds with MP-Borohydride.

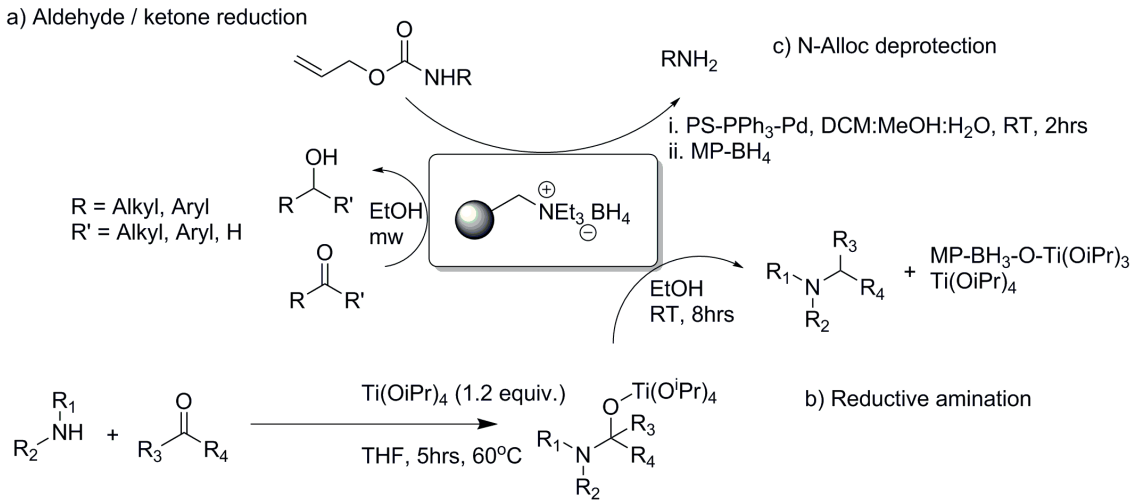


Figure 1 a–c. MP-Borohydride (center) and typical applications, 1a) Aldehyde / ketone reduction, 1b) Reductive amination, 1c) N-Alloc Deprotection

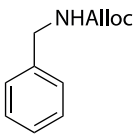
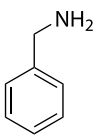
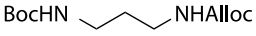
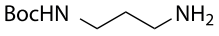
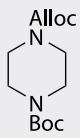
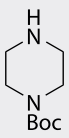
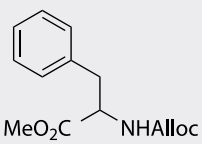
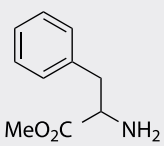
Substrate	Product Amine	Yield	% Purity	Substrate	Product Amine	Yield	% Purity
		85	98			79	97
		82	97			90	99

Table 2. Deprotection of N-Alloc Groups.

Capacity and Stability

The borohydride content of the resin was determined by measuring hydrogen evolution after addition of 1 M HCl. The resin has been found to be stable at 5 °C for at least 12 months.Unlike other commercially available solid-supported borohydride reagents, MP-Borohydride is relatively odorless.

Representative Procedures

Reduction of Aldehydes (Table 1, Entry 1)

A mixture of benzaldehyde (0.1 g, 1.0 mmol) and MP-Borohydride (2.6 mmol/g, 0.2 g, 0.5 mmol) in MeOH (5 mL) was stirred at room temperature for 3 h. The resin was filtered and the resin washed with DCM (2 x 3 mL). The combined solution was concentrated to afford benzyl alcohol in 89% yield and 98% GC purity.

Reduction of Ketones (Table 1, Entry 4)

A mixture of cyclohexanone (0.1 g, 1.0 mmol) and MP-Borohydride (2.6 mmol/g, 0.3 g, 0.78 mmol) in absolute MeOH (5 mL) was stirred at room temperature for 8 h. The resin was filtered and washed with DCM (2 x 3 mL). The combined solution was concentrated to afford cyclohexanol in 85% yield and 96% GC purity.

Ordering Information

Part Number	Quantity
800512	3 g
800401	10 g
800402	25 g
800403	100 g
800404	1000 g

References

1. Gibson, H. W.; Bailey, F. C. *J. Chem. Soc. Chem. Commun.* 1977, 815.
2. Habermann, J.; Ley, S. V.; Scott, J. S. *J. Chem. Soc. Perkin Trans.* 1, 1999, 1253.
3. Ley, S. V.; Schucht, O.; Thomas, A. W.; Murray, J. P. *J. Chem. Soc. Perkin Trans.* 1, 1999, 1251.
4. Yoon, N. M.; Park, K. B.; Gyoung, Y. S. *Tetrahedron Lett.* 1983, 24, 5367.
5. Kabalka, G. W.; Wadgaonkar, P. P.; Chatla, N. *Synth. Commun.* 1990, 20, 293.
6. Yoon, N. M.; Choi, J.; Ahn, J. H. *J. Org. Chem.* 1994, 59, 3490.
7. Part Numbers: 800478, 3 g; 800479, 25 g; 800480, 100 g; 800481, 1000 g.

bsi. ISO 9001 Quality Management FM 31206 ISO 14001 Environmental Management EMS 640981

Biotage holds certification for both ISO9001 Quality Management and ISO14001 Environmental Management.

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