Bromination of sodium p-toluenesulphinate; p-toluenesulphonyl (tosyl) bromide

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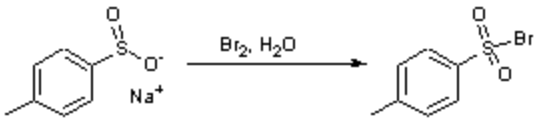

Chemicals Used

p-toluenesulfinic acid sodium salt (TsNa), 30.4g, 171mmol; distilled water, 1000mL; bromine,29g, 9.35mL, 188mmol carbon tetrachloride (can still buy from Aldrich)

Procedure

TsNa is dissolved in water and filtered to obtain a clear solution. Bromine is added dropwise over about 30min with rapid stirring and venting to allow for build-up of bromine gas. A yellow precipitate of crude TsBr is observed. Mixture is stirred for a further 30min following complete addition of the bromine. Filter off the crude TsBr and wash well with water. Dry thoroughly in vacuo overnight. Typical crude yield of dry powdery solid, 63-69%. Recrystallisation from carbon tetrachloride First dissolve in minimum hot CCl_4 and hot filter to remove any insoluble oily solid. Allow to cool and filter off crystals of TsBr. Wash with very cold CCl_4 (-20^{o}C) Do not air dry for more than a few seconds or crystals will go off. Final overall yield approx 50%

Author's Comments

This is a quick, easy and cheap way to make gram quantities of TsBr. There does not seem to be a ready alternative to the use of CCl_4 for the recrystallisation step which is a pity as the compound is rather soluble in this solvent. The product does not withstand drying and is best kept moist with CCl_4. This makes assessing yields a bit tricky as well as when using in a reaction. Removal of excess solvent just prior to the reaction under a stream of nitrogen is a reasonable compromise, but be careful as it really does go off if dried. Purity is best determined by TLC and melting point since other methods of analysis (ie NMR, IR) do not readily distinguish between the product and the possible impurities. Does not mass spec well. Where slight impurities do exist, these have not interfered with radical additions in my experience - reference 2.

Data

MPt: 92-93^{o}C ^{1}H NMR: 7.81 (2H, d, J=8.4Hz, H2&H6) 7.40 (2H, d, J=8.3Hz H3&H5), 2.42 (3H, s). ^{13}C NMR: 147.2, 130.6, 127.0, 126.3, 22.3.

Lead Reference

Abbott, D.J. et al.(1976): J. Am. Chem. Soc. Perkin Trans.1, 492-498. Miller, B. and Kalnins, M.V.(1967):Tetrahedron, 23, 1145-1152.

Other References

1. Caddick S.; Shering C.L.; Wadman S.N. Tetrahedron, 56, 465-473, 2000. 2. Caddick, S.; Hamza, D.; Wadman, S.N. Tetrahedron Lett. 1999, 40, 7285-7288.

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