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IMPROVED BROMINATION OF 2C-H TO 2C-B HBR

WRITTEN BY EUPHORIA

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This method is based on the one found in Pihkal #20 for the synthesis of 2C-B. However, some tips and tricks has been added, and the isolated and purified salt is the hydrobromide instead of the hydrochloride made by Shulgin.

The hydrobromide salt of 2C-B is relatively water-soluble, as opposed to the hydrochloride, which is nearly insoluble. This makes the insufflation of this salt (if desired) much less painful than with the more common hydrochloride. There is still a nasty burn, but not at all as sharp. Due to the higher molecular weight of this salt, 29mg of 2C-B hydrobromide is equivalent to 25mg of 2C-B hydrochloride.

EXPERIMENTAL:

In a 100ml beaker with good magnetic stirring, 24.8g 2,5-Dimethoxyphenethylamine (2C-H) was dissolved in 40ml glacial acetic acid, which caused heat evolution and the deposition of a clear crystalline mass of 2C-H acetate, which upon continued stirring redissolved to form a clear solution. Elemental bromine was chilled to -5 (as to minimize evaporation), and 7ml (22g) was pipetted up and added to 40ml of glacial acetic acid, and this solution was added in one portion to the stirred 2C-H solution, and the beaker covered with aluminum foil.

After a few minutes, the dark brown solution begun to crystallize with the evolution of heat and hydrobromic acid gas (hence the AI foil cover). When the suspension had solidified to a solid crystalline mass, the stirring was turned off and the beaker with its contents was allowed to stand until it had returned to room temperature. The light brown crystalline mass was crushed against the sides of the beaker with a spatula to give a thick light brown paste, which was transferred to a fritted glass funnel and filtered at the pump and washed with 20ml glacial acetic acid. The filtrate was poured out of the filter flask and set aside. The precipitate in the funnel was then washed with another 20ml glacial acetic acid and 2x20ml dry ether (this filtrate was also set aside), to give a light tan powder of 2C-B.HBr, which was air dried and sifted through a metal mesh to make it finely powdered. It was then boiled for 15 min in 200ml dry ether, filtered and washed with 50ml ether and air dried to give 36.42g 2C-B Hydrobromide as a white powder. No significant amounts of 2C-B.HBr could be recovered from the ether washes.

The black filtrate set aside earlier was diluted with 50ml ether and put in the freezer overnight, making it solidify almost completely, and after thawing it in the fridge, it was filtered and washed with a little ether to give 1.91g tan 2C-B.HBr after air drying. The other filtrate saved earlier (a light amber in color) was subjected to the same treatment, yielding 0.56g tan 2C-B.HBr. Both crops were combined, and recrystallized from a boiling mixture of 60ml acetonitrile and 6ml methanol. When the solution had cooled somewhat, 30ml dry ether was added, and the salt was allowed to slowly precipitate in the fridge overnight. The solution was then suction filtered and the precipitate washed with a little ether to yield 1.55g 2C-B.HBr as very fine, off-white needles.

The combined crops together weighed 37.9g, representing a total yield of 81% from 2C-H freebase.

NOTES:

The most important part in the purification of the salt is to thoroughly remove all the dark brown bromine and bromination byproducts directly in the intitial washing of the precipitated 2C-B.HBr, which is ensured by breaking up the light brown crystalline mass after the bromination so that no lumps at all are present, which could otherwise trap impurities. Then this mass has to be carefully washed, first with two acetic acid washes, followed by two ether washes to wash out dirty acetic acid and to facilitate drying of the powder. Even if you are doing an acid/base extraction of the hydrobromide followed by precipitation of the hydrochloride salt, this careful purification is absolutely necessary to ensure nice white crystals, unless you are planning to vacuum distill the 2C-B freebase before crystallization. Most people skip that step, and I feel that vacuum distillation of the 2C-H freebase followed by this cleaning procedure is a fully adequate alternative to obtain a product of good quality. Not vacuum distilling the 2C-B freebase also avoids having gaseous hallucinogens around. To really perfect the color and appearance of the product, it is dried, finely powdered and boiled in ether to remove the last traces of colored impurities. The ether does not dissolve any of the product, and is afterwards yellow-colored, indicating that it has taken up unwanted stuff from our product.

The reason the washings are collected in two separate portions is that by doing so, more 2C-B.HBr can be recovered from these solutions. If both acetic and etheral washes are combined, the solution forms two layers and much less salt is precipitated on cooling.

The recrystallization of the crude 2C-B.HBr is not optimized, better results may be obtained by using an isopropanol/ether recrystallization instead of this using acetonitrile:

ALTERNATIVE RECRYSTALLIZATION

1.55g off-white 2C-B.HBr was suspended in 25ml isopropanol in a 75ml beaker with magnetic stirring, and was heated until all the salt had dissolved^{*}, then removed from the heat, and 15ml ether added to the light amber solution. A suspension of white crystals slowly appeared, and after the solution had returned to room temperature it was put in the fridge for a few hours to ensure complete crystallization, the precipitate filtered, washed with 2x10ml ether and sucked free of solvents. After air drying overnight, the resulting fluffy white powder weighed 1.40g (90% recovery).

* As everything dissolved already at ~40-50 C, using 25ml IPA was unnecessary. Better aim for 10ml IPA and 5ml ether per gram 2C-B.HBr to minimize the required solvent volume when working with larger amounts. Using the freezer after the fridge may also allow to cut down on the ether, as most of the salt will crystallize anyway.