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Copper 1,14:7,8-Diethenotetrapyrido-[2,1,6-de:2',1',6'-gh:2'',1'',6'''-na][1,3,5,8,10,12]hexaazacyclotetradecine Trifluoroacetate

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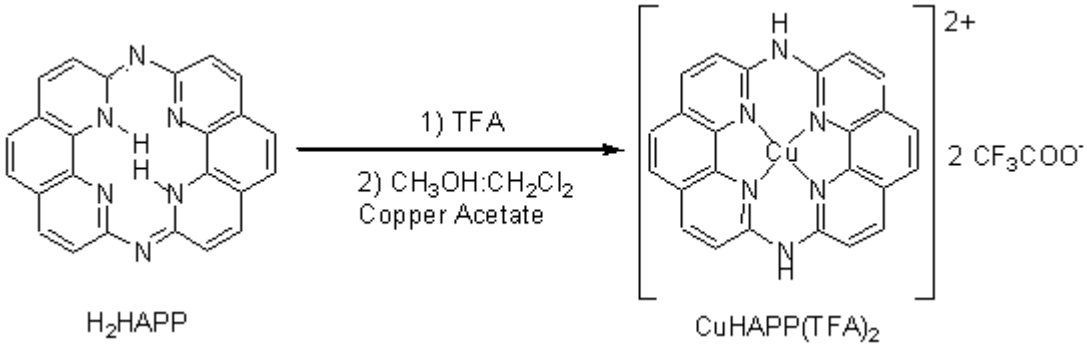

1,14:7,8-Diethenotetrapyrido-[2,1,6-de:2,1,6-gh:2,1,6-na][1,3,5,8,10,12] hexaazacyclotetradecine (H_2HAPP) was synthesized according to Ogawas method[1]. 20 mg H_2HAPP was dissolved in 0.5 mL trifluoroacetic acid, then 2mL ether was added, and the white precipitate [$H_4HAPP(TFA)_2$] was filtered off and washed with diethyl ether for several times. The mixture of this white precipitate and 50 mg copper acetate was refluxed in CH3OH:CH2Cl2 (V:V, 1:1) for 1 hour, the solvent evaporated, and the orange precipitate was washed with methanol. The product was purified by diffusing diethyl ether into a TFA solution at 4°C. Brown-yellow needle crystals of $CuHAPP(TFA)_2$ were obtained, 23 mg. Yield: 65.7 %.

NMR : no NMR signal could be observed because Cu^{++} ion is paramagnetic.

IR (KBr): 2716 (br), 1666 (s), 1642 (s), 1619 (m), 1597 (s), 1565 (s), 1505 (m), 1467 (s), 1421 (m), 1382 (m), 1369 (m), 1360 (m), 1291 (s), 1256 (m), 1198 (s), 1169 (s), 1151 (s), 1129 (s), 1106 (m), 866 (m), 828 (m), 800 (m), 749 (w), 717 (m), 672 (m), 642 (w), 606 (m), 576 (m), 519 (w), 486 (m).

UV-Vis (in $CH_3OH:CH_2Cl_2$, V:V, 1:1): 329.5, 281.0, 224.5 (spectra is pH sensitive).

FAB-MS ($[M-1]^+$): 448.

Anal. Calc. for $C_{28}H_{14}N_6O_4F_6Cu$ (675.5): C 49.74, H 2.07, N 12.43 %; Found: C 49.70, H 2.32, N 12.15 %.

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Reference

1. Ogawa, S.; Yamaguchi, T.; Gotoh, N. *J. Chem. Soc., Perkin Trans. I* **1974**, 976.

Sample Availability: Available from the authors and from MDPI.