Experimental Methods:

Tetrakis(thiourea)dichlorotellurium(II) dihydrate was prepared by the method of Foss et al.[8]

Tetrakis(thiourea)dichlorotellurium(II) dihydrate and sodium hydro-tris(methimazolyl)borate were mixed in methanol in a 1:2 ratio. The solution was stirred for 3 hrs and the solid produced collected and extracted with $CHCl_3$. The product obtained from solvent evaporation was essentially pure. However, the material can be recrystallized from $CHCl_3$/hexane by vapor diffusion from which the X-ray quality crystals were obtained (yield 65%).

Anal. Calcd for $C_{24}H_{32}N_{12}S_6B_2Te$ C, 34.75; H, 3.86; N, 20.26. Found: C, 34.35; H, 3.81; N, 19.45. ^{1}H NMR (400 MHz, d^6 dmso @ 20^{o}C) δ 7.29, s (broad), 1H, CH; δ 6.79, s (broad), 1H, CH; δ 3.51 s, 3H, NMe. IR (KBr cm^{-1}) 2509 (ν_{B-H}), 738 ($\nu_{C=S}$). MS (FAB) m/z 833.03 (molecular ion). Crystal data: yellow prism (0.30 × 0.10 × 0.08 mm), $C_{24}H_{32}B_2N_{12}S_6Te_1$. $2CHCl_3$, M = 1068.93, Monoclinic, Space group $P2_1/a$ (No. 14), a = 15.1347(2) Å, b = 19.4830(3) Å, c = 15.2672(3) Å, β = 105.6840(10) º, V = 4334.21(12) Å^3, Z = 4, D_c = 1.638 g cm^{-3}, T = 150 K, μ (Mo kα) = 1.383 mm^{-1}, 32215 reflections measured, 9553 unique reflections (R_{int} = 0.0628), R_1 (all data) = 0.0665, wR_2 = 0.0952, 476 parameters.

Bismuth trichloride and sodium hydrotris-(pyrazolyl)borate were mixed in cold acetone (0^{o}C) in a 1:3 ratio and stirred for 3 hrs in a salt/ice bath. The solution was rapidly filtered to remove the sodium chloride and bismuth solids. The acetone was removed slowly in aliquots under a stream of N_2 at 0^{o}C, after which the sample was returned to the freezer (–25^{o}C). A white powder formed amongst which a small number of X-ray quality crystals were found (yield 18%).

Anal. Calcd for $C_{21}H_{24}N_{14}B_2BiCl$: C, 34.15; H, 3.28; N, 26.55. Found: C, 35.47; H, 3.19; N, 27.21. Crystal data: colourless fragment (0.20 × 0.15 × 0.10), $C_{21}H_{24}B_2Bi_1Cl_1N_{14}$, M = 738.59, Orthorhombic, space group Pbca (No. 61) a = 15.716(7) Å, b = 37.40(10) Å, c = 9.236(2) Å, V = 5428(2) Å^3, Z = 8, D_c = 1.807 g cm^{-3}, T = 291 K, μ (Mo kα) = 6.623, absorption correction DIFABS (T_{max} = 1.000, T_{min} = 0.798), 6043 reflections measured, 5366 unique reflections (R_{int} = 0.101), R_1 (all data) = 0.0489, GOF = 1.188.