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2*H*-1,4-Benzotellurazin-3(4*H*)-one and its Derivatives

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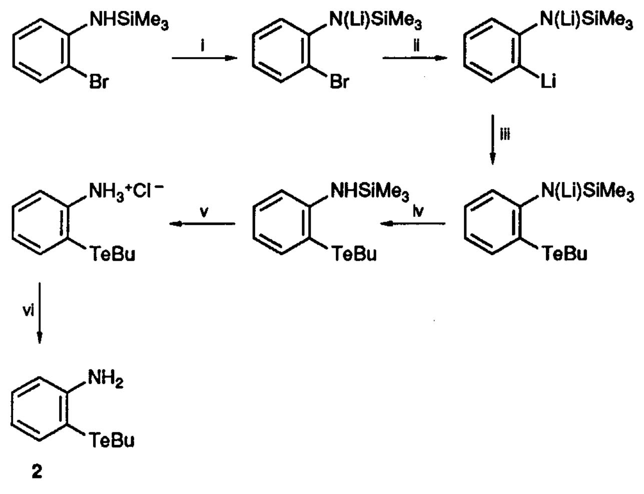
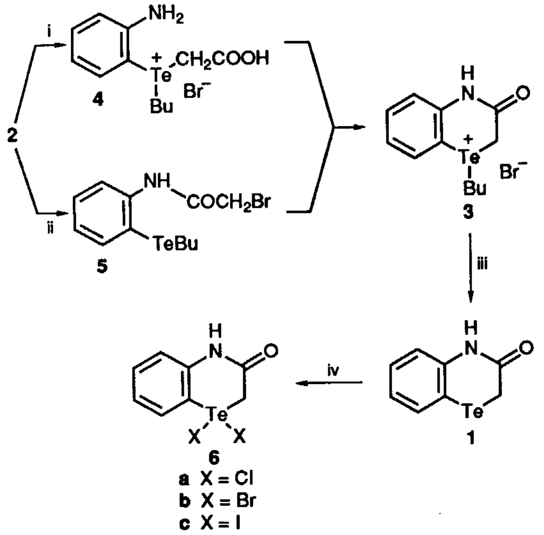
A novel tellurium-containing heterocycle, 2*H*-1,4-benzotellurazin-3(4*H*)-one **1** has been prepared by treatment of butyl 2-aminophenyl telluride **2** with either bromoacetic acid or bromoacetyl chloride and subsequent elimination of butyl bromide from the tellurazinium compound **3** so formed.

While the synthesis and reactions of phenotellurazines[1–6] (dibenzo derivatives of 1,4-tellurazine[7,8]) have been amply described, no data on any of its monobenzo derivatives have been reported. Interest in these compounds is in particular aroused by the numerous useful applications that have been found for 1,4-benzothiazines.[9] The present work describes the preparation of the first example of a 1,4-benzotellurazine derivative, 2*H*-1,4-benzotellurazin-3(4*H*)-one **1** starting from butyl 2-aminophenyl telluride **2**.



2

Scheme 1 *Reagents and conditions*: i, MeLi and Li (excess); diethyl ether, 10°C; ii, BuBr (5–10 mol%), 6–8°C; iii, Te then BuBr (excess), 34°C, 1 h; iv, H_2O, 0°C; v, MeOH, HCl, r.t.; vi, H_2O, KOH, 50°C, 0.5 h



Scheme 2 *Reagents and conditions*: i, $BrCH_2COOH$, MeOH, r.t.; ii, $BrCH_2COCl$, Py, $CHCl_3$, 60°C; iii, DMF, 120°C; iv, X_2, CH_3COOH, r.t.

Because of the great reducing potential of tellurolate anions which react with α-halogenocarbonyl compounds by single-electron transfer rather than as nucleophilic reagents,[10] the general synthetic method for 2*H*-1,4-benzothiazin-3(4*H*)-ones, *i.e.* coupling of *o*-aminothiophenols with halogenoacetic acids under basic conditions, cannot be adapted to the preparation of the tellurium analogue **1**. Therefore, a reaction scheme was developed that involved butyl 2-aminophenyl telluride **2** as the starting material (Scheme 1).†

Treatment of **2** with an equimolar amount of bromoacetic acid gives rise to 1-butyl-3,4-dihydro-3-oxo-2*H*-1,4-benzotellurazinium bromide **3** through the spontaneous cyclization of the intermediate telluronium salt **4**. That *Te*- rather than *N*-alkylation occurs in the initial stage of the reaction is supported by the fact that butyl 2-aminophenyl telluride **2**, on treatment with methyl bromoacetate under the same reaction conditions, affords a readily isolable telluronium salt (the methyl ester of **4**) in 60% yield. As for many other alkyltelluronium salts,[11] butyl bromide is smoothly eliminated when a solution of **3** in DMF is heated, giving **1** in 90% yield (Scheme 2).‡

When bromoacetyl chloride is employed instead of bromoacetic acid, the reaction with butyl 2-aminophenyl telluride **2** proceeds, most probably through an *N*-acylation step with formation of anilide **5** as the intermediate. This approach to **3** is similar to that employed in the preparation of 2*H*-1,4-benzothiazin-3(4*H*)-ones using a cyclization reaction of alkyl 2-(α-halogenoacetamido)phenyl sulfides.[12] However, in this case attempts to isolate the sulfonium salts analogous to **3** were unsuccessful.

As expected, heterocycle **1** containing a dicoordinated tellurium centre readily undergoes oxidative addition reactions when treated with halogens or sulfuryl chloride. The products of these reactions are the tellurazines **6a–c**.

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† Characterisation data for **2**: b.p. 162–165 °C/2 mmHg; ^{1}H NMR δ_H (300 MHz, $[^2H_6]$DMSO), 0.88 (t, *J* 7 Hz, Me, 3H), 1.28–1.44 (m, CH_2, 2H), 1.60–1.76 (m, CH_2, 2H), 2.77 (t, *J* 7 Hz, CH_2, 2H), 5.18 (s, NH_2, 2H), 6.40–7.62 (m, ArH, 4H).

‡ **3**: m.p. 158–161 °C (decomp.); ^{1}H NMR δ_H (300 MHz, $[^2H_6]$DMSO) 0.80 (t, Me, 3H), 1.23–1.37 (m, CH_2, 2H), 1.43–1.82 (m, CH_2, 2H), 2.18–3.02 (m, CH_2, 2H), 3.37 (d, *J* 4.3 Hz, CH_2, 1H), 3.94 (d, *J* 4.3 Hz, CH_2, 1H), 7.08–9.06 (m, ArH, 4H), 10.62 (s, NH, 1H); IR ν/cm^{-1} 3388, 3188 (NH), 1667 (CO).

1: m.p. 207–209 °C; ^{1}H NMR δ_H (300 MHz, $[^2H_6]$DMSO) 3.31 (s, CH_2, 2H), 6.87–7.65 (m, ArH, 4H), 10.12 (s, NH, 1H); IR ν/cm^{-1} 3263, 3162 (NH), 1636 (CO); *m/z* 261 (M^+, 51%), 132 (M – Te, 53%), 91 (M – C_2H_3O – Te, 100%) (Calc. M^+ 261).

6a: m.p. 255–260 °C; IR ν/cm^{-1} 3183, 3097, 3040 (NH), 1667 (CO).

6b: m.p. 285–295 °C; IR ν/cm^{-1} 3189, 3102, 3055 (NH), 1669 (CO).

6c: m.p. 275–280 °C; IR ν/cm^{-1} 3192, 3105 (NH), 1670 (CO).