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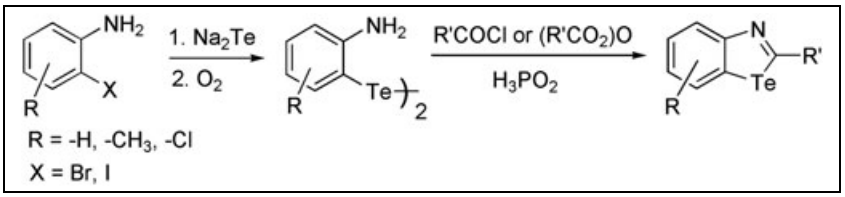

Benzo-1,3-tellurazoles carrying alkyl or aryl substituents in position 2 were prepared in a facile two-step sequence, starting with readily available 2-haloanilines. This approach relies on the preparation of bis (2-aminophenyl) ditellurides by nucleophilic halide displacement from 2-haloanilines with sodium telluride in *N*-methylpyrrolidone. Subsequent reductive cyclization with carboxylic acid halides or carboxylic anhydrides furnished benzo-1,3-tellurazoles in good yields.

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INTRODUCTION

Benzo-1,3-tellurazoles constitute highly light- and air-stable compounds, which have found interest in applications as diverse as the preparation of telluracarbocyanine dyes [1], potential anthelmintics [2], and organic polymers [3]. Several methods for their preparation have been described, nearly all of which rely on displacing a suitable leaving group by tellurium in the presence of a nitrogen-containing substituent in ortho position (Scheme 1). These include treatment of 2-nitrophenylboronic acid with tellurium tetrachloride [4], the reaction of 2-(*N*-trimethylsilylamino)phenylmagnesium bromides with elemental tellurium [5], reaction of 2-lithionitrobenzene with benzyltellurocyanate [6], and reaction of 2-acetamidophenylmercury chlorides with tellurium tetrachloride [7]. All of these methods have disadvantages, such as relatively inaccessible starting materials, highly toxic intermediates, or tedious procedures. Consequently, benzo-1,3-tellurazoles have remained relatively unexplored.

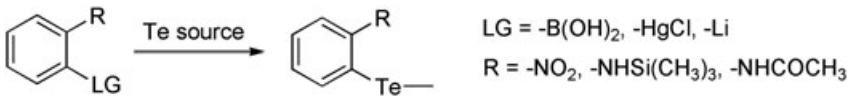
We now report the facile preparation of benzo-1,3-tellurazoles in two steps, starting with readily available 2-haloanilines. These were found to undergo nucleophilic displacement of halogen by tellurium when treated with sodium telluride in NMP, to yield bis(2-aminophenyl) ditellurides. Cyclization to the target compounds was achieved in one additional step, by treatment of the resulting bis(2-aminophenyl) ditellurides **1–3** with benzoyl chlorides or aliphatic acid anhydrides and subsequent reductive cyclization in a one-pot procedure (Scheme 2).

RESULTS AND DISCUSSION

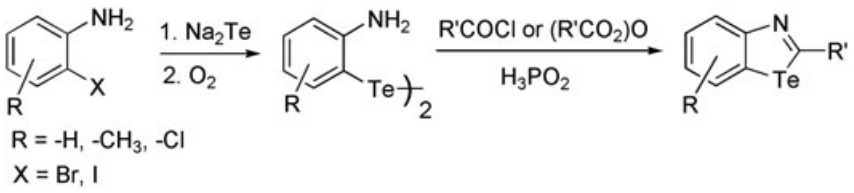
The displacement of halogen from halobenzenes by Te^{2-} was reported before, but only for relatively electron-poor aromatic substrates [8]. The fact that electron-rich haloanilines suffer nucleophilic attack is remarkable and attests to the high nucleophilicity of the Te^{2-} anion. Indeed, this reaction proceeds well only within a narrow range of conditions and cannot be adapted to selenium analogues. Both the reported reaction temperature and time constitute a compromise between the fairly vigorous conditions needed for halogen displacement and product decomposition. Other solvents, such as DMSO, HMPA, DMF, and sulfolane, did not produce satisfactory results. An increase of the reaction temperature or extension of the reaction time much beyond 4.5 h resulted in drastically diminished yields for the corresponding bis(2-aminophenyl) ditellurides **1–3**, presumably due to carbon–tellurium bond fission. The reaction provided the highest yield, when a threefold molar excess of sodium hydride was used. Initially, aryl tellurolates were generated, which underwent rapid air oxidation to the corresponding ditellurides. Remarkably, 4-chloro-2-iodoaniline produced the corresponding ditelluride **3** in rather modest yield, whereas 2-bromo-4-chloraniline and 2-iodo-4-fluoroaniline did not generate ditellurides in synthetically useful quantities.

Strategies for the conversion of bis(2-aminophenyl) ditellurides to benzo-1,3-tellurazoles **4–11** have been reviewed recently [9]; methods reported here are optimized for simplicity. The use of acid halides was found to be well suited for the preparation of 2-arylbenzotellurazoles, whereas acid anhydrides were best suited for the synthesis of 2-alkylbenzotellurazoles such as **10**. The synthesis of 2-arylbenzotellurazoles is facilitated by the fact that 2-arylbenzotellurazolium hydrochlorides are poorly soluble in the media employed for their synthesis, allowing most by-products to be removed by filtration.

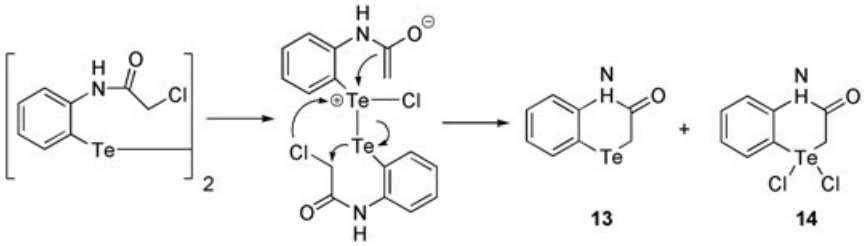
Scheme 1



Scheme 2



Scheme 3



The behavior of bis(2-chloroacetamidophenyl) ditelluride was investigated in further detail. It was observed that solutions of bis(2-chloroacetamidophenyl) ditelluride are unstable and precipitate a mixture of elemental tellurium and a colorless solid upon standing, both in the presence and absence of light. A well-formed crystal, when isolated and submitted to X-ray crystallography, identified this solid as 1,1-dichloro-2*H*-1,4-benzotellurazin-3(4*H*)-one **14**. It is plausible that this product results from disproportionation via an enolate intermediate (Scheme 3, Fig. 1).

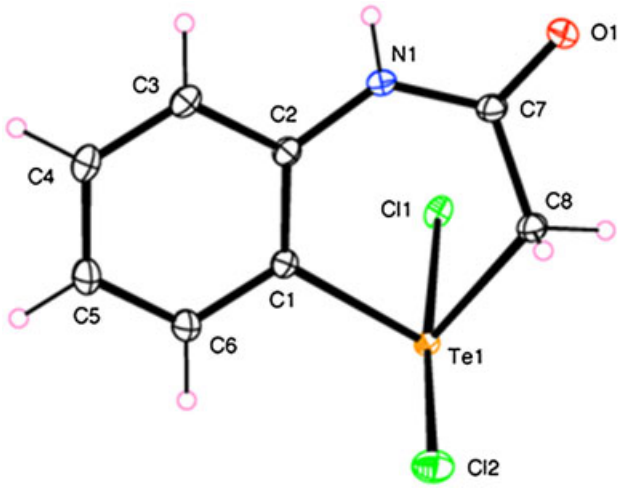


Figure 1. ORTEP plot of 1,1-dichloro-2*H*-1,4-benzotellurazin-3(4*H*)-one. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Because of its low yield, however, this reaction is poorly suited for the preparation of 2*H*-1,4-benzotellurazin-3(4*H*)-one **13**. The synthesis of this compound was reported previously [10], but no experimental details were provided. Consequently, they are reported in the following section. At δ 1.5 ppm, the methylene carbon of this compound is remarkably shielded. Product yields are summarized in Table 1.

EXPERIMENTAL

4-Chloro-2-iodoaniline was prepared according to the literature [11]. NMP and THF were dried over molecular sieves. All other chemicals were purchased in reagent-grade purity and used as received. Tellurium was used as 200 mesh powder. Chromatographic purifications were carried out using Acros Organics (Geel, Belgium) activated aluminum oxide, neutral, 50–200 micron. Melting points were recorded using a Mel-Temp EM-6 (Bibby Scientific US, Burlington, NJ) apparatus. nmr spectra were recorded on a JEOL (JEOL USA, Peabody, MA) Eclipse 300-MHz spectrometer. MS was performed on a Finnigan (Thermo Finnigan LLC, San Jose, CA) Mat GCQ mass spectrometer equipped with a direct probe inlet. Only the most abundant masses are reported for isotope clusters containing tellurium. ir spectra were recorded on a PerkinElmer (Perkin Elmer Inc., Waltham, MA) Spectrum 100 FTIR spectrometer. Elemental analyses were performed on an Exeter (Exeter Analytical Inc., North Chelmsford, MA) CE-440 Elemental Analyzer. Due the possibility for the generation of trace amounts of highly toxic hydrogen telluride at various stages of the following syntheses, "all operations should be carried out under a well-vented hood."

Bis(2-aminophenyl) ditelluride (1) and bis(2-amino-4-methylphenyl) ditelluride (2). A 100-mL three-necked flask fitted with mechanical stirring and a nitrogen purge line was

charged with 1.0 g (7.84 mmol) of tellurium powder, 0.94 g, (23.51 mmol) of 60% sodium hydride suspension in mineral oil (the oil was not removed prior to use) and 10 mL of anhydrous NMP. The flask was purged with nitrogen and immersed in a Wood's metal bath kept at 185 ± 2. The mixture was stirred vigorously for 10 min, during which time the tellurium dissolved. Following the dropwise addition of 1.35 g (7.84 mmol) of 2-bromoaniline and 1.46 g (7.84 mmol) of 2-bromo-4-methylaniline, respectively, over approximately 1 min, the mixture was heated and stirred for an additional 4.5 h. It was then allowed to cool to RT, diluted by adding 80 mL of water, and buffered by the addition of 1.3 g (24.3 mmol) of ammonium chloride. A brisk stream of air was forced through this solution for 2 h to oxidize the sodium aryl tellurolate to the ditelluride, as well as to convert any unreacted sodium telluride to elemental tellurium. A color change to reddish was noticed during this time, and a mixture of tellurium and the target compound precipitated. Subsequently, this mixture was collected by vacuum filtration, washed with copious amounts of water, and finally with 5 mL of petroleum ether to remove any mineral oil introduced with sodium hydride. The remaining solid was extracted with hot acetone in 20-mL portions until no further red ditelluride dissolved; the combined extracts were filtered and concentrated on a rotary evaporator. The crude product was purified by crystallization from ethanol.

Bis(2-aminophenyl) ditelluride (1). Yield 1.05 g, 61%, mp 104–105°C; ir (potassium bromide): 3302, 3398 (NH); ^{1}H nmr ($CDCl_3$): δ 4.17 (NH), 6.81–7.70 (ArH); ^{13}C nmr ($CDCl_3$): δ 95.0, 113.7, 119.1, 131.7, 143.3, 150.7; m/z (30 eV EI): 92.0 (C_6H_6N, 100%), 184.1 ($C_{12}H_{12}N_2$, 83%), 221.9 (C_6H_6NTe, 35%), 346.8 ($C_6H_5NTe_2$, 14%). *Anal*. Calcd for $C_{12}H_{12}N_2Te_2$: C, 32.80; H, 2.75; N, 6.38. Found: C, 32.72; H, 3.01; N, 6.18.

Bis(2-amino-4-methylphenyl) ditelluride (2). Yield 1.15 g, 63%, mp 116–117°C; ir (potassium bromide): 3158, 3357 (NH); ^{1}H nmr ($CDCl_3$): δ 2.18 (CH_3), 4.06 (NH), 6.66–7.48 (ArH); ^{13}C nmr ($CDCl_3$): δ 20.1, 95.3, 113.7, 128.4, 132.4, 143.3, 148.2; *m/z* (30 eV EI): 106.1 (C_7H_8N, 100%), 212.2 ($C_{14}H_{16}N_2$, 56%), 235.9 (C_7H_8NTe, 27%), 360.8 (C_7H_7NTe, 28%). *Anal*. Calcd for $C_{14}H_{16}N_2Te_2$: C, 35.97; H,3.45; N,5.99. Found: C, 35.67; H, 3.31; N, 5.94.

Bis(4-chloro-2-aminophenyl) ditelluride (3). The crude product, prepared as discussed previously from 4-chloro-2-iodoaniline at a reaction temperature of 175°C, had to be further purified by chromatography (dichloromethane : alumina) before being crystallized from ethyl acetate : methyl-*tert*-butyl ether (1: 3). Yield, 0.42 g, 21%; mp 125–127°C; ir (potassium bromide): 3320, 3411 (NH); ^{1}H nmr ($CDCl_3$): δ 4.12 (NH), 6.63–7.61 (ArH); ^{13}C nmr ($CDCl_3$): δ 95.1, 114.3, 122.6, 131.6, 142.0, 149.1; *m/z* (30 eV EI): 125.9 (C_5H_5NCl, 100%), 253.9 (C_6H_5ClNTe, 70%), 380.8 ($M^+ - Te$, 6%). *Anal*. Calcd for $C_{12}H_{10}Cl_2N_2Te_2$: C, 28.35; H, 1.98; N, 5.51. Found: C, 28.31; H, 1.80; N, 5.84.

General procedure for preparation of 2-arylbenzotellurazoles. A 25-mL round-bottom flask equipped with magnetic stirring and reflux condenser was charged with the corresponding ditelluride (**1**, **2**, or **3**, 0.7 mmol), dissolved in 2 mL of THF. The mixture was stirred for 1 min until all ditelluride had gone into solution, 1.5 mmol of the corresponding benzoyl chloride added, and stirring continued for 5 min. This was followed by an addition of 2 mL of 95% ethanol, 330 mg (2.5 mmol) of 50% aq hypophosphorous acid, and 1.5 mL of 36% hydrochloric acid. The mixture was subsequently heated to reflux for 15 min. A gradual color change from orange to greenish was observed. Subsequently, the mixture was chilled in a freezer for 1 h, and precipitated solids were collected by filtration. These consisted mainly of the tellurazolium hydrochlorides in addition to trace quantities of elemental tellurium. The filter cake was placed in a beaker; 10 mL of 5% aq ammonia solution and 10 mL of DCM were added. The beaker was covered, set aside for several hours and occasionally stirred to complete phase partitioning. The organic phase was collected, and traces of extremely fine elemental tellurium were removed by centrifugation (filtration proved ineffective). After solvent removal, products were recrystallized from petroleum ether. All were isolated as pale yellow needles.

2-Phenylbenzotellurazole (4, from 1 and benzoyl chloride). Yield 267 mg, 62%, mp 100–101°C; ^{1}H nmr ($CDCl_3$): δ 7.15–8.22 (ArH); ^{13}C nmr ($CDCl_3$): δ 125.1, 126.6, 127.2, 128.6, 129.2, 131.1, 131.7, 134.4, 141.1, 162.2, 173.4; *m/z* (30 eV EI): 179.2 ($M^+ - Te$, 41%), 206.0 (C_6H_4Te, 55%), 308.8 (M^+, 100%). *Anal*. Calcd for $C_{13}H_9NTe$: C, 50.89; H, 2.96; N, 4.57. Found: C, 50.83; H, 2.76; N, 4.58.

2-(4-Chlorophenyl)benzotellurazole (5, from 1 and 4-chlorobenzoyl chloride). Yield 280 mg, 60%, mp 124–125°C; ^{1}H nmr ($CDCl_3$): δ 7.15–8.22 (ArH); ^{13}C nmr ($CDCl_3$): δ 125.3, 126.7, 127.3, 129.4, 129.7, 131.7, 134.7, 137.0, 139.7, 162.0,

Table 1

Yields of compounds prepared.

Compound	Formula	Yield (%)
Bis(2-aminophenyl) ditelluride (**1**)	$C_{12}H_{12}N_2Te_2$	61
Bis(2-amino-4-methylphenyl) ditelluride (**2**)	$C_{14}H_{16}N_2Te_2$	63
Bis(4-chloro-2-aminopenyl) ditelluride (**3**)	$C_{12}H_{10}Cl_2N_2Te_2$	21
2-Phenylbenzotellurazole (**4**)	$C_{13}H_9NTe$	62
2-(4-Chlorophenyl)benzotellurazole (**5**)	$C_{13}H_8ClNTe$	60
2-(4-Methoxyphenyl)benzotellurazole (**6**)	$C_{14}H_{11}NOTe$	64
6-Methyl-2-phenylbenzotellurazole (**7**)	$C_{14}H_{11}NTe$	56
6-Methyl-2-(4-chlorophenyl)benzotellurazole (**8**)	$C_{14}H_{10}ClNTe$	74
6-Chloro-2-phenylbenzotellurazole (**9**)	$C_{13}H_8ClNTe$	69
2-Propylbenzotellurazole (**10**)	$C_{10}H_{11}NTe$	74
6-Chloro-2-methylbenzotellurazole (**11**)	C_8H_6ClNTe	81
1-Butyl-3,4-dihydro-3-oxo-2*H*-1,4-benzotellurazolium bromide (**12**)	$C_{12}H_{16}BrNOTe$	70[a]
2H-1,4-Benzotellurazin-3(4*H*)-one (**13**)	C_8H_7NOTe	61

[a]Approximately, compound 12 was not isolated in pure form.

171.5; *m/z* (30 eV EI): 206.02 (C_6H_4Te, 54%), 213.06 ($M^+ - Te$, 32%), 342.80 (M^+, 100%). *Anal*. Calcd for $C_{13}H_8ClNTe$: C, 45.75; H, 2.36; N, 4.10. Found: C, 45.84; H, 2.66; N, 4.18.

2-(4-Methoxyphenyl)benzotellurazole (6, from 1 and 4-methoxybenzoyl chloride). Yield 305 mg, 64%, mp 112–113°C; ^1H nmr ($CDCl_3$): δ 3.87 (CH_3O), 6.93–8.16 (ArH); ^{13}C nmr ($CDCl_3$): δ 55.6, 114.4, 124.8, 126.1, 127.1, 130.2, 131.6, 133.9, 134.2, 162.1, 162.2, 172.5; *m/z* (30 eV EI): 206.1 (C_6H_4Te, 36%), 281.0 ($M^+ - Te$, 49%), 339.0 (M^+, 100%). *Anal*. Calcd for $C_{14}H_{11}NOTe$: C, 49.92; H, 3.29; N, 4.16. Found: C, 49.98; H, 3.61; N, 4.30.

6-Methyl-2-phenylbenzotellurazole (7, from 2 and benzoyl chloride). Yield 251 mg, 56%, mp 84–85°C; ^1H nmr ($CDCl_3$): δ 2.46 (CH_3), 7.25–8.08 (ArH); ^{13}C nmr ($CDCl_3$): δ 21.3, 126.0, 128.5, 128.6, 129.2, 130.9, 131.7, 134.4, 135.3, 141.2, 160.2, 171.8; *m/z* (30 eV EI): 293.2 ($M^+ - Te$, 34%), 220.1 (C_7H_6Te, 22%), 322.9 (M^+, 100%). *Anal*. Calcd for $C_{14}H_{11}NTe$: C, 52.41; H, 3.46; N, 4.37. Found: C, 52.39; H, 3.39; N, 4.42.

6-Methyl-2-(4-chlorophenyl)benzotellurazole (8, from 2 and 4-chlorobenzoyl chloride). Yield 368 mg, 74%, mp 128–129°C; ^1H nmr ($CDCl_3$): δ 2.47 (CH_3), 7.25–8.07 (ArH); ^{13}C nmr ($CDCl_3$): δ 21.4, 126.1, 128.7, 129.3, 129.5, 131.7, 134.7, 135.6, 136.8, 139.7, 160.1, 169.9; *m/z* (30 eV EI): 89.07 (84%), 227.09 ($M^+ - Te$, 34%), 356.84 (M^+, 100%). *Anal*. Calcd for $C_{14}H_{10}ClNTe$: C, 47.33; H, 2.84; N, 3.94. Found: C 47.09; H, 2.59; N, 3.93.

6-Chloro-2-phenylbenzotellurazole (9, from 3 and 4-benzoyl chloride). Yield 329 mg, 69%, mp 130–131°C; ^1H nmr ($CDCl_3$): δ 7.42–8.08 (ArH); ^{13}C nmr ($CDCl_3$): δ 126.9, 127.8, 128.6, 129.3, 130.8, 131.0, 131.4, 135.5, 140.7, 160.7, 173.7; m/z (30 eV EI): 213.2 ($M^+ - Te$, 28%), 240.1 (C_6H_3ClTe, 34%), 342.9 (M^+, 100%). *Anal*. Calcd for $C_{13}H_8ClNTe$: C, 45.75; H, 2.36; N,4.10. Found: C, 46.12; H, 2.59; N, 4.14.

General procedure for preparation of 2-alkylbenzotellurazoles. A 25-mL round-bottom flask equipped with magnetic stirring and a reflux condenser was charged with the corresponding ditelluride (**1** or **3**, 0.7 mmol) and 5 mL of acetonitrile. The mixture was stirred and briefly heated until all ditelluride had gone into solution. Stirring was continued while the temperature was allowed to return to below 50°C, followed by addition of 1.5 mmol of the corresponding acid anhydride. The mixture was subsequently heated to reflux for approximately 2 min, followed by addition of 330 mg (2.5 mmol) of 50% aq hypophosphorous acid. Heating at reflux was resumed for 15 min, during which time a color change from orange to pale gray-green was noticed. The mixture was subsequently allowed to cool to RT and poured into 10 mL of a 5% aq ammonia solution. The products separated as off-white crystals. They were collected and dissolved in petroleum ether : methyl-*tert*-butyl ether (3:1), and traces of polar impurities were removed with a 10 mm × 1.5 cm alumina-packed flash column. The tellurazoles were subsequently recrystallized from 95% ethanol.

2-Propylbenzotellurazole (10, from 1 and butanoic anhydride). Colorless plates, yield 276 mg, 74%, mp 71–72°C; ^1H nmr ($CDCl_3$): δ 1.04 (CH_3), 1.84 (–CH_2–), 3.02 (–CH_2–), 7.11–8.10 (ArH); ^{13}C nmr ($CDCl_3$): δ 13.7, 24.7, 45.4, 124.8, 125.7, 126.8, 131.8, 134.2, 160.6, 178.80; *m/z* (30 eV EI): 275.0 (M^+, 100%). *Anal*. Calcd for $C_{10}H_{11}NTe$: C, 44.03; H, 4.06; N, 5.13. Found: C, 43.86; H, 4.44; N, 5.15.

6-Chloro-2-methylbenzotellurazole (11, from 3 and acetic anhydride). Colorless needles, yield 269 mg, 81%, mp 126–127°C; ^1H nmr ($CDCl_3$): δ 2.84 (CH_3), 7.37–7.94 (ArH); ^{13}C nmr ($CDCl_3$): δ 30.8, 125.9, 127.4, 130.4, 131.1, 136.6, 159.2, 170.9; m/z (30 eV EI): 151.2 ($M^+ - Te$, 26%), 240.1 (C_6H_4ClTe, 32%), 280.9 (M^+, 100%). *Anal*. Calcd for C_8H_6ClNTe: C, 34.42; H, 2.17; N,5.02. Found: C, 34.40; H, 2.39; N, 5.00.

Reaction of bis(2-aminophenyl) ditelluride (1) with chloroacetyl chloride. A 25-mL round-bottom flask equipped with magnetic stirring and a reflux condenser was charged with 300 mg (0.68 mmol) of **1** and 5 mL of acetonitrile. The mixture was stirred and briefly heated until all ditelluride had gone into solution. Pyridine (119 mg, 1.50 mmol) was added, followed by dropwise addition of 154 mg (1.36 mmol) of chloroacetyl chloride. The flask was capped and set aside for 5 days. After this time, approximately 90 mg of solids had precipitated, which consisted of a white crystalline solid mixed with elemental tellurium and contained several well-formed crystals suitable for X-ray crystallography. The white compound was identified as 1, 1-dichloro-2*H*-1,4-benzotellurazin-3(4*H*)-one, an ORTEP plot of which is shown in Figure 1. In light of the low yield of this procedure, no attempt was made to purify the product.

1-Butyl-3,4-dihydro-3-oxo-2H-1,4-benzotellurazolium bromide (12) and 2H-1,4-benzotellurazin-3(4H)-one (13). A 50-mL round-bottom flask equipped with magnetic stirring, reflux condenser, and a nitrogen purge line was charged with 500 mg (1.14 mmol) of **1** and 8 mL of methanol. The mixture was heated to reflux, purged with nitrogen, and sodium borohydride added until the color of the ditelluride had faded (approximately 110 mg consumed). A solution of 327 mg (2.39 mmol, 5% excess) of 1-bromobutane in 1-mL methanol was added, and refluxing resumed for an additional 5 min. Subsequently, the flask was allowed to cool to RT and 379 mg (2.73 mmol, 20% excess) of bromoacetic acid added before capping and setting aside for 3 days. After this time, the crude telluronium salt **12** had precipitated as colorless crystals. The flask was chilled in a freezer for 1 h and the product collected by filtration, yield, 630 mg, 70%. It was dried and used without purification.

The crude telluronium salt (**12**, 300 mg, 0.75 mmol) was placed in an open-ended 15 mm × 15 cm reaction tube equipped with magnetic stirring, and 2 mL of DMF was added. The mixture was heated to gentle boiling for 15 min, allowing 1-bromopropane to escape. The tube was then allowed to cool to RT and 10 mL of water added. The product precipitated as crystalline solid and was collected by filtration. It was dissolved in THF, traces of polar impurities were removed with a 10 mm × 1.5 cm alumina-packed flash column, and the product was crystallized from 95% ethanol. Yellow needles, yield 120 mg, 61 %, mp 206–208°C; ir (potassium bromide): 3165, 3263 (NH), 1643 (CO); ^1H nmr ($CDCl_3$): δ 3.38 (2H), 6.89–7.54 (ArH), 8.52 (NH); ^{13}C nmr ([$^1H_6$]-DMSO): δ 1.5, 103.7, 119.1, 123.6, 128.4, 136.5, 142.8, 169.1; *m/z* (30 eV EI): 132.07 (C_8H_6NO, 100%), 220.9 (C_6H_5NTe, 14%), 262.85 (M^+, 54%). *Anal*. Calcd for C_8H_7NOTe: C,36.85; H, 2.72; N,5.37. Found: C, 37.04; H, 3.11; N, 5.34.

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