Sodium Telluride in *N*-Methyl-2-pyrrolidone. Reduction of Aromatic Carbonyl Compounds to Alcohols and Formation of Pyrrolo[2,3-*d*]pyrimidines (7-Deaza-9*H*-purines) from Aromatic Nitriles

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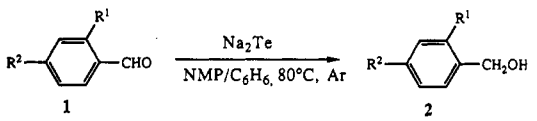
Tellurium reacts with sodium hydride (NaH) in hot *N*-methyl-2-pyrrolidone (NMP) to afford sodium telluride (Na_2Te) as a purple homogeneous solution, in which aromatic iodides are smoothly tellurated to form arenetellurolates.[1] We now wish to report that aromatic carbonyl compounds are reduced to alcohols when heated gently in this solution, while aromatic nitriles are partly converted into pyrrolo[2,3-*d*]pyrimidines (7-deaza-9*H*-purines). Although Na_2Te is known as an efficient and selective reagent for a variety of functional transformations,[2] it is inert toward carbonyl and nitrile functions in the solvent systems commonly employed, i.e., in protic solvents such as alcohol and/or water or in aprotic solvents such as *N*,*N*-dimethylformamide (DMF) and hexamethylphosphoric triamide (HMPA). The remarkable enhancement of the reducing ability of the telluride anion in NMP which had not been previously noticed was confirmed by comparing the cyclic voltammetric behavior of Na_2Te in NMP and DMF.

When aromatic aldehydes 1 are gently heated with Na_2Te in a molar ratio 1.6–2.0:1.0 in NMP/benzene (1:4) under reflux, the mixture gradually turned black under the liberation of free tellurium and after usual workup the corresponding benzyl alcohols 2 were obtained in 42–59% isolated yields (Table I). With halobenzaldehydes the reduction of the carbonyl group and displacement of the halogen atom by tellurium anion occurred competitively to give a mixture of the corresponding benzyl alcohol and diaryl ditelluride in varying ratios. A possible intervention of the Cannizzaro-type disproportionation of aldehyde[3] was ruled out from the present reaction, because carboxylic acid was not obtained in any appreciable amount.



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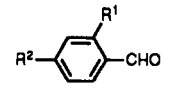
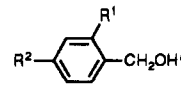
In order to clarify the origin of the hydrogen atom used in the conversion of aldehyde into alcohol, the reaction of benzaldehyde **1a** with Na_2Te was carried out in NMP-*3-d*. The benzyl alcohol **2a** obtained was found to contain deuterium in about 40% of the newly introduced hydrogen. Thus, the hydrogen source was confirmed to be the methylene group adjacent to the amide carbonyl group of NMP.

When benzophenone **3** was heated with Na_2Te in NMP/toluene (1:1) under gentle reflux for 12 h, there resulted in deep blue solution, usual workup of which gave benzhydrol in 89% isolated yield. This blue solution exhibited a weak broad ESR signal which might be attributable to a ketyl radical. However, benzopinacol could not be obtained. Attempted reduction of **3** using Na_2Te/DMF under similar conditions only led to the recovery of ketone.

Table I. Reduction of Benzaldehydes with Na_2Te



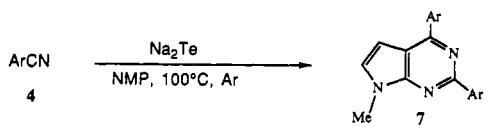


1	R^1	R^2	condns temp, °C	condns time, h	2 mp, °C, or bp, °C/mmHg	2 yield,[b] %
a	H	H	80	0.8	195–200/760	59
b	CH_3	H	80	0.6	30–31 (lit.[13] mp 33–36)	42
c	H	CH_3	80	1.0	53–56 (lit.[14] mp 57–58)	50
d	H	$(CH_3)_2CH$	90	0.5	140/21 (lit.[14] bp 135–136/26)	34

[a] Identified by direct comparison with the authentic samples. [b] Isolated yield. Not optimized.

Seeing the enhanced reducing ability of Na_2Te in NMP, we next turned our attention toward aromatic nitriles which had been known to be inert to Na_2Te. When benzonitrile **4a** was heated with Na_2Te/NMP at 110 °C for 7 h under argon, we were surprised to obtain an appreciable amount of a crystalline nitrogen-containing solid besides unchanged nitrile and polymeric material. This product was assigned the structure of 9-methyl-2,6-diphenyl-7-deaza-9*H*-purine, **7a**, based on its spectral data and elemental analysis and confirmed by an independent synthesis as shown in Scheme I (route b). Neither benzylamine nor benzylideneimine was detected in the product mixture. Methylbenzonitriles **4b,c** also gave 7-deaza-9*H*-purines **7b,c** in comparable yields (Table II), but aliphatic nitriles such as propionitrile and isobutyronitrile remained intact. Simple heating of aromatic nitriles and NaH in NMP did not produce any 7-deaza-9*H*-purines **7**.

In view of the pharmacological importance of deazapurine derivatives[4] as well as of the known tedious procedures involved in constructing the 7-deazapurine framework,[5,6] the present one-pot formation of **7a–c** appeared at first to be of considerable synthetic value. However, the attempts to improve the yields of **7** were fruitless; reactions under forced conditions (prolonged reaction time or higher temperatures) only resulted in the increase of polymeric products.



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(2) For recent reviews, see: Irgolic, K. J. *Houben-Weyl Methoden der Organischen Chemie, 4th ed.*; Georg Thieme Verlag: Stuttgart, 1990; Vol. E12b. Petragnani, N.; Comasseto, J. V. *Synthesis* **1991**, 793.
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Scheme I

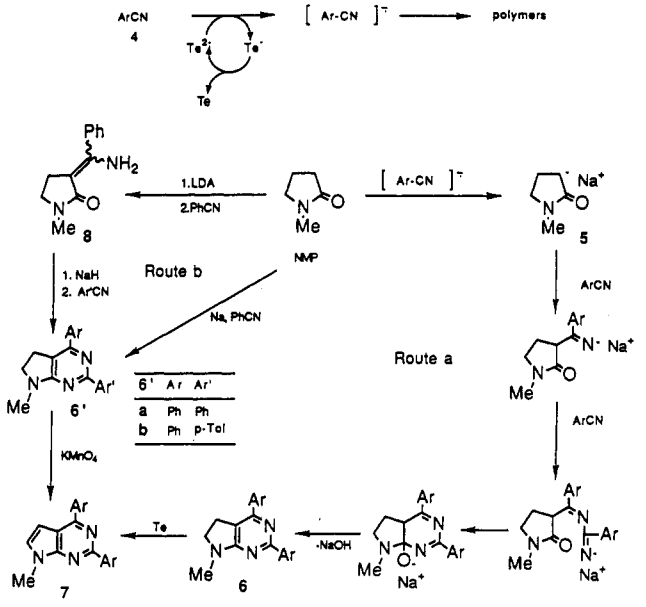

Table II. 7-Deazapurines Obtained from Benzonitriles

ArCN		condns		7-deazapurine 7	
4	Ar	temp, °C	time, h	mp, °C	yield,[a] %
a	C_6H_5	110	7	159–160	15
b	4-$H_3CC_6H_4$	100	13	150–152	16
c	3-$H_3CC_6H_4$	100	8	125–127	28

[a] Isolated yield. Not optimized.

7-Deaza-9*H*-purines **7** are most likely formed by the cyclocondensation between two molecules of aromatic nitrile and one molecule of NMP via route a shown in Scheme I. The anion radical generated from nitrile via the electron transfer from Te^{2-} ion abstracts the α-hydrogen from NMP to yield the lactam enolate anion **5**, which adds successively to two molecules of aromatic nitrile to form 7,8-dihydro-7-deaza-9*H*-purine **6**. Compound **6** is dehydrogenated by the nascent tellurium to yield 7-deazapurine **7**. 1,1-Diphenylethylene as a radical trap did not influence the formation of **7**.

In order to substantiate the above mechanism, the enamine **8** was prepared from benzonitrile and NMP via an independent route (route b in Scheme I) and treated with NaH in the presence of an equimolar amount of 4-methylbenzonitrile. As expected, 7,8-dihydro-7-deaza-9*H*-purine **6'b** with two different aryl substituents was obtained in 8–10% isolated yield. Interestingly, when benzonitrile was heated with metallic sodium in NMP at 80 °C, 7,8-dihydro-7-deaza-9*H*-purine **6'a** was obtained in a low yield and subsequent treatment of **6'a** with $KMnO_4$ in acetone gave **7a**. These findings suggested a possible role of the nascent tellurium in the dehydrogenation of **6** to **7**. Although sulfur and selenium are long-known dehydrogenating agents for hydroaromatic compounds,[7] such a transformation with tellurium is, as far as we know, unprecedented. We have examined the dehydrogenation of several hydroarenes and indolines with finely powdered tellurium in NMP at 100–120 °C. Hydroarenes such as tetralin and decalin were inert, but indolines were dehydrogenated to give the expected indoles. Thus, free tellurium was confirmed to dehydrogenate some hydroazaarenes, just as the lower chalcogen elements do for hydroarenes.

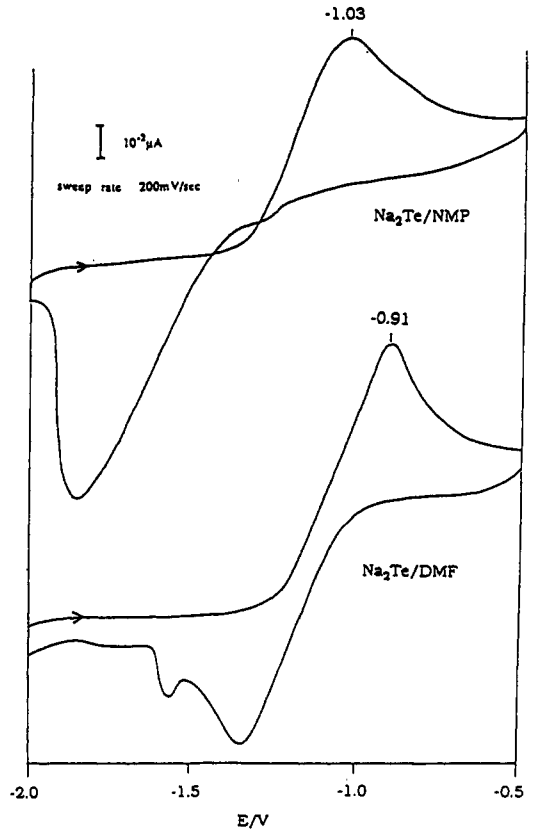

Figure 1. Cyclic voltammograms of Na_2Te in NMP (top) and DMF (bottom) at 23 °C.

In order to compare the reducintg power of Na_2Te/NMP and Na_2Te/DMF, the electromeasurements were performed for each of the solvent systems using the ferrocenium/ferrocene redox couple as the standard. The peak potentials of the oxidation route of Te^{2-} ion in NMP and DMF were −1.03 and −0.91 V, respectively, indicating the enhancement of the reducing ability of Te^{2-} in NMP as compared with that in DMF (Figure 1).[8,9] A standard oxidation–reduction potential E° of benzophenone was also measured in these solvents, and the same value of −2.26 V was obtained. This result may indicate a transition state in which the electron transfer to the ketone to form a ketyl is not influenced by the change of solvent, thus providing an indirect support for the enhanced reducing ability of Te^{2-} in NMP.

In summary, we have shown that the reducing power of Na_2Te is enhanced in hot NMP to such a level as to reduce aromatic carbonyl compounds to the corresponding alcohols. In the presence of Na_2Te, aromatic nitriles cyclocondense with NMP to form 2,6-diaryl-9-methyl-7-deaza-9*H*-purines in low to moderate yields.

Experimental Section

Commercial aromatic aldehydes and nitriles and benzophenone were purified by recrystallization or distillation. Sodium hydride

(7) Plattner, A.; Armstrong, E. C. Dehydrogenation with Sulfur, Selenium and Platinum Metals. *Newer Methods of Preparative Organic Chemistry*; Interscience: London, 1948; p 21.

(8) Standard oxidation–reduction potential of the tellurium anion in aqueous solution was reported: de Bethume, A. J.; Loud, N. A. S. *Standard Aqueous Electrode Potentials and Temperature Coefficients at 25 °C*; Hampel, C. A., Ed.; Skokie: Chicago, 1964; Vol. III.

(9) A unique pattern of the cyclic voltammogram of Na_2Te/NMP is suggestive of some unknown electrochemical process occurring on the reduction route, which alters the surface conditions of the electrode (Prof. S. Okazaki).

(NaH; 60% dispersion in mineral oil) was washed with dry hexane prior to use. Tellurium (99.999%) was used as commercially obtained. *N*-Methyl-2-pyrrolidone (NMP) and *N,N*-dimethylformamide (DMF) were distilled from calcium hydride and stored over 4-Å molecular sieves. NMP-*3-d* was prepared by quenching the lithiated NMP with deuterium oxide. Melting points were determined on a Yanagimoto hot-plate apparatus and are uncorrected. ^{1}H-NMR spectra were recorded in $CDCl_3$ on a Varian Gemini 200-MHz NMR spectrometer. *J* values are given in Hz. IR spectra were measured as KBr pellets with a Shimadzu FTIR-8100S infrared spectrophotometer. Mass spectra (EI) were determined at 70 eV on a Shimadzu GCMS-QP2000A mass spectrometer. Electromeasurements were made on a PAR Model 174 polarographic analyzer.

Preparation of Sodium Telluride. Sodium telluride was obtained either as a deep purple homogeneous solution by heating tellurium and NaH in a molar ratio of 1:2 in NMP at 100–110 °C under argon or as a pale yellow chalky suspension by heating both reagents similarly in DMF at 140 °C.

Reduction of Aromatic Aldehydes. General Procedure. To a solution of Na_2Te (prepared from Te (170 mg, 1.3 mmol), NaH (70 mg, 2.9 mmol)) in NMP (2.5 mL) was added acetic acid (1.2 mg, 0.02 mmol)[10] followed by aldehyde **1** (2.1 mmol) in dry benzene (10 mL) at 80 °C under argon, and the resulting mixture was kept at this temperature for 0.5–1.0 h. During the course of this period, the color of the solution gradually changed from deep purple to blue and finally to black. The progress of the reaction was monitored by TLC. After disappearance of the starting material (0.5–1.0 h), the reaction was quenched by the addition of 10% hydrochloric acid (0.1 mL), and the mixture was briefly aerated with vigorous stirring. Deposited tellurium was filtered off, and the filtrate was partitioned twice between ethyl acetate (20 mL) and saturated brine (10 mL). The organic phase was separated and dried over Na_2SO_4. The solvent was removed under reduced pressure, and the residue was chromatographed over silica gel using hexane/ethyl acetate (6:1) as the eluent to give crude product, benzyl alcohol **2**, which was purified either by Kugelrohr distillation (liquid) or by recrystallization (solid).

Reduction of Benzophenone (3). A solution of Na_2Te (prepared from Te (180 mg, 1.4 mmol) and NaH (100 mg, 4.2 mmol)) in NMP (5 mL) was diluted with dry toluene (5 mL), and a solution of benzophenone (270 mg, 1.5 mmol) in dry toluene (1 mL) was added in one portion. The mixture was heated at 110 °C under argon. After 12 h 10% hydrochloric acid (0.6 mL) was added to the resulting blue reaction mixture. Elemental tellurium was filtered off, and the filtrate was evaporated almost to dryness under reduced pressure. The residue was chromatographed over silica gel using hexane/ethyl acetate (6:1) as the eluent to afford benzhydrol (240 mg, 89%), mp 62–64 °C (lit.[11] mp 64–66 °C), identical with the authentic specimen.

Attempted Reduction of Aromatic Nitriles. General Procedure. Aromatic nitrile **4** (1.4 mmol) was added in one portion to a solution of Na_2Te (prepared from Te (360 mg, 2.8 mmol) and NaH (150 mg, 6.3 mmol)) in NMP (7 mL), and the mixture was heated at 100–110 °C for 7–13 h. During the course of this period, the color of the solution changed from deep purple to black. After disappearance of the starting material, the reaction mixture was cooled and aerated with stirring at room temperature. Benzene (5 mL) was added, and free tellurium was filtered off. The filtrate was extracted three times with a mixture of ethyl acetate (7 mL) and water (10 mL). The combined extracts were dried over Na_2SO_4, and the solvent was removed under reduced pressure. The residue was chromatographed over silica gel using hexane/ethyl acetate (5:1) as the eluent to furnish 7-deaza-9*H*-purine **7** as a crystalline solid.

7-Methyl-2,4-diphenylpyrrolo[2,3-*d*]pyrimidine (7a): yield 15%; colorless crystals; mp 159–160 °C; ^{1}H NMR ($CDCl_3$) δ 8.6–8.7 (2 H, m), 8.2–8.3 (2 H, m), 7.4–7.7 (6 H, m), 7.20 (1 H, d, *J* = 3.6), 6.81 (1 H, d, *J* = 3.6), 3.95 (3 H, s); ^{13}C NMR δ 29.77, 100.27, 113.85, 128.01, 128.25 (2 C), 128.56 (2 C), 129.00 (2 C), 129.42 (2 C), 129.76 (2 C), 138.71, 139.00, 152.96, 156.79, 157.48; IR (KBr), 1570, 1540, 1390, 1290, 940, 770, 720, 690 cm^{-1}; MS (EI) *m/z* 285 (M$^+$, 100), 181 (14), 140 (11), 77 (12). Anal. Calcd for $C_{19}H_{15}N_3$: C, 79.98; H, 5.30; N, 14.73. Found: C, 79.94; H, 5.20; N, 14.67.

7-Methyl-2,4-bis(4-methylphenyl)pyrrolo[2,3-*d*]pyrimidine (7b): yield 16%; colorless crystals; mp 150–152 °C; ^{1}H NMR ($CDCl_3$) δ 8.56 (2 H, d, *J* = 8.3), 8.18 (2 H, d, *J* = 8.2), 7.37 (2 H, d, *J* = 8.4), 7.30 (2 H, d, *J* = 8.1), 7.18 (1 H, d, *J* = 3.5), 6.80 (1 H, d, *J* = 3.5), 3.96 (3 H, s), 2.46 (3 H, s), 2.43 (3 H, s); ^{13}C NMR δ 21.45 (2 C), 30.98, 100.26, 113.53, 127.97, 128.91 (2 C), 129.05 (2 C), 129.34 (2 C), 129.49 (2 C), 136.13, 136.49, 139.40, 139.94, 153.07, 156.87, 157.71; IR (KBr) 1580, 1520, 1380, 1170, 935, 800, 715 cm^{-1}; MS (EI) *m/z* 313 (M$^+$, 100), 195 (7), 157 (9), 91 (5). Anal. Calcd for $C_{21}H_{19}N_3$: C, 80.48; H, 6.11; N, 13.41. Found: C, 80.57; H, 6.11; N, 13.58.

7-Methyl-2,4-bis(3-methylphenyl)pyrrolo[2,3-*d*]pyrimidine (7c): yield 28%; colorless crystals; mp 125–127 °C; ^{1}H NMR ($CDCl_3$) δ 8.4–8.5 (2 H, m), 8.0–8.1 (2 H, m), 7.2–7.5 (4 H, m), 7.18 (1 H, d, *J* = 3.6), 6.79 (1 H, d, *J* = 3.5), 3.95 (3 H, s), 2.50 (3 H, s), 2.49 (3 H, s); IR (KBr) 1570, 1540, 1520, 1390, 1370, 1270, 1090, 790, 710 cm^{-1}; MS (EI) *m/z* 313 (M$^+$, 100), 195 (3), 157 (6), 91 (5). Anal. Calcd for $C_{21}H_{19}N_3$: C, 80.48; H, 6.11; N, 13.41. Found: C, 80.84; H, 6.26; N, 13.03.

7-Methyl-2,4-diphenyl-5,6-dihydropyrrolo[2,3-*d*]pyrimidine (6′a). To a mixture of sodium metal (200 mg, 8.7 mmol), NMP (8 mL), and dry benzene (3 mL) was added benzonitrile (2.5 mL, 24 mmol) in one portion, and the resulting mixture was heated at 80 °C for 14 h. The reaction was quenched by the addition of 2-propanol (1 mL) followed by water (10 mL), and the product was extracted twice with a mixture of ethyl acetate (50 mL) and water (25 mL). The extracts were combined and dried, and the solvent was removed under reduced pressure. The residue was chromatographed on silica gel to give compound **6′a** as colorless crystals (440 mg, 13%): mp 181–183 °C; ^{1}H NMR ($CDCl_3$) δ 8.5–8.6 (2 H, m), 8.0–8.1 (2 H, m), 7.4–7.5 (6 H, m), 3.58 (2 H, t, *J* = 8.2), 3.27 (2 H, t, *J* = 8.3), 3.08 (3 H, s); ^{13}C NMR δ 26.01, 31.24, 51.03, 113.85, 128.00 (4 C), 128.30 (2 C), 128.99 (2 C), 129.67 (2 C), 138.27, 138.86, 154.10, 163.06, 168.70; IR (KBr) 1600, 1570, 1540, 1380, 750, 690 cm^{-1}; MS (EI) *m/z* 287 (M$^+$, 61), 286 (100), 77 (14). Anal. Calcd for $C_{19}H_{17}N_3$: C, 79.41; H, 5.96; N, 14.62. Found: C, 79.44; H, 5.94; N, 14.53.

7-Methyl-4-phenyl-2-(4-methylphenyl)-5,6-dihydropyrrolo[2,3-*d*]pyrimidine (6′b). To a solution of enamine **8** (220 mg, 1.1 mmol) in NMP (4 mL) was added NaH (40 mg, 1.7 mmol) at room temperature under argon. After 40 min *p*-tolunitrile (140 mg, 1.2 mmol) was added to the solution. The resulting mixture was gradually heated to 80 °C and kept at this temperature for 17 h, during the course of which NaH (60 mg, 2.5 mmol) was added in two portions to the reaction mixture at an interval of 4 hours. Usual workup gave compound **6′b** as a colorless solid (25 mg, 8%): mp 150–153 °C; ^{1}H NMR ($CDCl_3$) δ 8.4–8.6 (2 H, m), 8.0–8.1 (2 H, m), 7.4–7.5 (3 H, m), 7.2–7.3 (2 H, m), 3.61 (2 H, t, *J* = 7.8), 3.30 (2 H, t, *J* = 8.0), 3.11 (3 H, s), 2.41 (3 H, s); MS (EI) *m/z* 301 (M$^+$, 62), 300 (100), 91 (23), 77 (35). Anal. Calcd for $C_{20}H_{19}N_3$: C, 79.70; H, 6.35; N, 13.94. Found: C, 79.81; H, 6.49; N, 13.76.

Dehydrogenation of 1,2-Dimethylindoline with Tellurium. A mixture of finely powdered tellurium (170 mg, 1.3 mmol), 1,2-dimethylindoline (200 mg, 1.4 mmol), and NMP (3 mL) was heated at 120 °C for 48 h under an argon atmosphere. Usual workup of the reaction mixture gave 1,2-dimethylindole, 40 mg (34% based on 60% conversion) in addition to unchanged starting material: mp 50–53 °C (lit.[12] mp 54–55 °C); ^{1}H NMR ($CDCl_3$) δ 7.50 (1 H, d, *J* = 7.7), 7.0–7.3 (3 H, m), 6.22 (1 H, s), 3.61 (3 H, s), 2.39 (3 H, s); MS (EI) *m/z* 145 (M$^+$, 78), 144 (100).

When dihydrodeazapurine **6′a** was similarly treated with tellurium powder at 100 °C for 28 h, deazapurine **7a** was obtained in 48% yield at 55% conversion.

1-Methyl-3-(aminophenylmethylene)-2-pyrrolidone (8). NMP (2.9 mL, 30 mmol) was added to a solution of lithium

(10) Since an excess of NaH appeared to work adversely toward the reduction of aldehydes, it was carefully removed by the addition of acetic acid.

(11) Truett, W. L.; Moulton, W. N. *J. Am. Chem. Soc.* **1951**, *73*, 5913.

(12) McDonald, B. G.; Proctor, G. R. *J. Chem. Soc., Perkin Trans. 1* **1975**, 1446.

(13) Bransen, W. R.; Hauser, C. R. *Organic Syntheses*; Wiley: New York, 1963; Collect. Vol. IV, p 582.

(14) Mozingo, R.; Folkers, K. *J. Am. Chem. Soc.* **1948**, *70*, 229.

diisopropylamide (32 mmol) in THF (30 mL) at 0 °C under argon, and the mixture was stirred at room temperature for 1 h. Then benzonitrile (3.2 g, 31 mmol) was added slowly at 0 °C, and the resulting mixture was allowed to stir for 4 h at room temperature. The color of the solution gradually changed from pale yellow to red. The reaction was quenched by the addition of water (5 mL), and the product was extracted twice with a mixture of ethyl acetate (50 mL) and a saturated brine (25 mL). The extracts were combined and dried over Na_2SO_4, and the solvent was removed under reduced pressure. The syrupy residue was distilled by Kugelrohr (250 °C/2.5 mmHg) to obtain a yellow oil, which solidified upon standing in a refrigerator and was recrystallized from hexane–chloroform (2:1) to give 8 as yellow crystals (4.7 g, 78%): mp 101–103 °C; ^{1}H NMR ($CDCl_3$) δ 7.3–7.5 (5 H, m), 5.91 (2 H, br s), 3.26 (2 H, t, J = 7.3), 2.85 (3 H, s), 2.62 (2 H, t, J = 7.2); IR (KBr) 1650, 1560, 1490, 1430, 1400, 1290 cm^{-1}; MS (EI) m/z 202 (M^+, 100), 144 (31), 130 (78), 105 (13), 98 (25), 77 (30). Anal. Calcd for $C_{12}H_{14}N_2O$: C, 71.26; H, 6.98; N, 13.85; O, 7.91. Found: C, 71.25; H, 7.03; N, 14.14; O, 8.01.

Measurement of Cyclic Voltammetry. The working electrode was a 0.5-mm diameter Pt disk, and a double-junctioned assembly of a Ag/Ag$^+$ reference electrode was employed to record potentials. The counter electrode was a platinum wire (0.5 mm in diameter). The temperature of the electrochemical cell was 23 °C. Cetyltrimethylammonium bromide was employed as a supporting electrolyte. The concentration of Na_2Te was 54 mM. An NMP solution of Na_2Te was prepared by heating a mixture of Te (55.3 mg, 0.433 mmol), NaH (31.2 mg, 1.30 mmol), and NMP (2 mL) and was diluted by the addition of the same solvent (5 mL). In the case of a DMF solution, the initially formed chalky suspension was diluted with DMF (5 mL) and then stirred at 140 °C for 30 min to obtain a deep purple homogeneous solution. Cetyltrimethylammonium bromide (80 mg) dissolved in the respective solvents (1 mL) was added to each of the Na_2Te solutions, and the solutions thus obtained were subjected to the cyclic voltammetry measurement under nitrogen. Measurements of the standard oxidation–reduction potential of benzophenone in NMP and DMF were made for 40 mM solutions using tetraethylammonium perchlorate as a supporting electrolyte.

Acknowledgment. We thank Prof. S. Okazaki of Ehime University, Dr. M. Oyama and Mr. M. Satake of our department for valuable discussions and also for measurement of cyclic voltammetry.