Heteroatom Chemistry
Volume 8, Number 5, 1997

Studies on the Formation of Alkyl Radicals from Alkyl Aryl Tellurides and Their Application to the Synthesis of Carbocyclic *C*-Nucleoside Analogs

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Received 22 November, 1996; Revised 3 February 1997

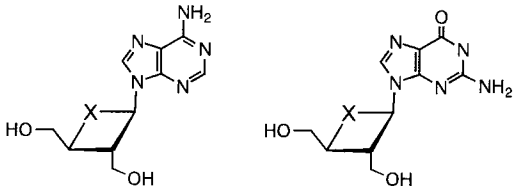
ABSTRACT

The photochemically initiated reactions and the atmospherically initiated reactions of alkyl aryl tellurides with electron-deficient heteroaromatic bases in the presence of N-acetoxy-2-thiopyridone and triethylborane, respectively, have been investigated. These reactions were applied to the preparation of carbocyclic four-membered C-nucleoside analogs. © 1997 John Wiley & Sons, Inc. Heteroatom Chem **8***: 411–419, 1997*

INTRODUCTION

Nucleoside chemistry has been widely studied as a significant factor in the functioning of antibacterial, antiviral, and antitumor agents [1]. Especially, in order to improve the antiviral activities of nucleosides, extensive modifications have been made on both the sugar and the base moieties. The replacement of the furanose ring by a carbocyclic ring leads to the synthesis of carbocyclic analogs of nucleosides. Carbocyclic nucleoside analogs have also been of interest for their high stabilities in addition to their high biological activities [2]. Recently, the synthesis of four-membered carbocyclic nucleoside analogs, which possess potential antiviral activities, has gained increasing interest due to their biological properties [3]. For example, Oxetanocin A (**IA**) (Scheme 1) is a naturally occurring nucleoside antibiotic that exhibits both antiviral and antitumour activities [4]. Oxetanocin G (**IIA**) has been found to exhibit antiviral activity [5]. The observed biological activities have encouraged organic chemists to undertake the synthesis of (**IB**) and (**IIB**), respectively [6], and, in particular, the enantiomerically pure guanine derivatives (**IIB**) were found to have very high activity against herpes [7].

As a part of our studies concerning the synthesis of carbocyclic nucleosides [8], we now report the syntheses of cyclobutyl nucleosides by the uses of a



I A X = O
I B X = CH_2
II A X = O
II B X = CH_2

SCHEME 1

Dedicated to Prof. William E. McEwen on the occasion of his seventy-fifth birthday.

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 CCC 1042-7163/97/050411-09

photochemically initiated radical reaction and an atmospherically initiated radical reaction, involving alkyl aryl tellurides. As compared with an ionic reaction, a radical reaction has the following advantages: (1) mild reaction conditions, (2) a one-pot procedure, and (3) neutral reaction conditions. Thus, with the availability of the radical reactions, the synthesis of four-membered carbocyclic nucleosides has attracted our interest. However, at first, as a model study, the reactions of cycloalkyl aryl chalcogenides with lepidine in the presence of either *N*-acetoxy-2-thiopyridone or triethylborane to give 2-cycloalkyl-4-methylquinolines were studied.

RESULTS AND DISCUSSION

Reactivity of Chalcogenides

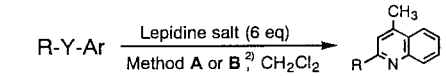
In order to study the effect of chalcogen atoms in the mechanistically complex radical reactions under consideration, the reactions of alkyl aryl sulfides, selenides, and tellurides with lepidinium salts have been investigated. As shown in Table 1, adamantyl 4-methoxyphenyl sulfide, adamantyl 4-methoxyphenyl selenide, and adamantyl 4-methoxyphenyl telluride were prepared and irradiated with a tungsten lamp (500 W) in the presence of *N*-acetoxy-2-thiopyridone and a lepidinium salt (method A). Adamantyl 4-methoxyphenyl telluride showed the highest reactivity (entry 3), while the corresponding selenide and sulfide did not undergo the desired reactions at all. This difference might come from the much higher electron density at the tellurium atom than that at the selenium or sulfur atom. Thus, the telluride was easily attacked by the methyl radical presumed to be formed from the *N*-acetoxy-2-thiopyridone, since the methyl radical is thought to behave as an electrophilic radical [9]. Then, other alkyl aryl tellurides, such as cyclohexyl and *n*-pentyl 4-methoxyphenyl tellurides, were prepared and treated with lepidine under the same conditions. The same results were obtained by the atmospherically initiated radical reactions with triethylborane (method B). The results were favorable, and the high reactivity of the tellurides shown in the above reactions prompted our interest to apply the method to the preparation of four-membered carbocyclic *C*-nucleosides.

p-Substituent Effect of Alkyl Aryl Tellurides

Our next objective was to carry out a model study on the *p*-substituent effect of alkyl aryl tellurides. Cyclohexyl 4-methoxyphenyl telluride, cyclohexyl 4-methylphenyl telluride, cyclohexyl phenyl telluride, and cyclohexyl 4-bromophenyl telluride were prepared and irradiated with a tungsten lamp in the presence of *N*-acetoxy-2-thiopyridone and a lepidinium salt to afford the corresponding 2-cyclohexyl-4-methylquinoline (**4**) (method A). ^{1}H-, ^{13}C-, and ^{125}Te-NMR chemical shift values of compounds **3** and the yields of products are depicted in Table 2.

Table 2 shows that the compound **4** could be obtained in good yield either with an electron-donating substituent or an electron-withdrawing one (entries 1 and 4) present on the aryl group of the alkyl aryl telluride. The reason might be as follows: in entry 1, the methyl radical behaves as an electrophilic radical, while in entry 4, the methyl radical appears to react more like a nucleophilic radical. To buttress this speculation, *N*-propionoxy-2-thiopyridone, the precursor of the more nucleophilic ethyl radical, was prepared and treated with a cyclohexyl aryl telluride and the lepidinium salt under the same conditions. If our speculations were correct, the yields should gradually increase from entries 1 to 4 (R = C_2H_5).

TABLE 1 Reactivity of Chalcogenides



Entry	1 R-Y-Ar[1)]	Yields / % of 2 Method A	Method B
1	Ad-S-An	0	0
2	Ad-Se-An	0	<10
3	Ad-Te-An	81	95
4	Cy-Te-An	65	66
5	*n*-Pen-Te-An	0	0

1) Ad = 1-Adamantyl; An = *p*-Anisyl; Cy = Cyclohexyl; *n*-Pen = *n*-Pentyl
2) See experimental section

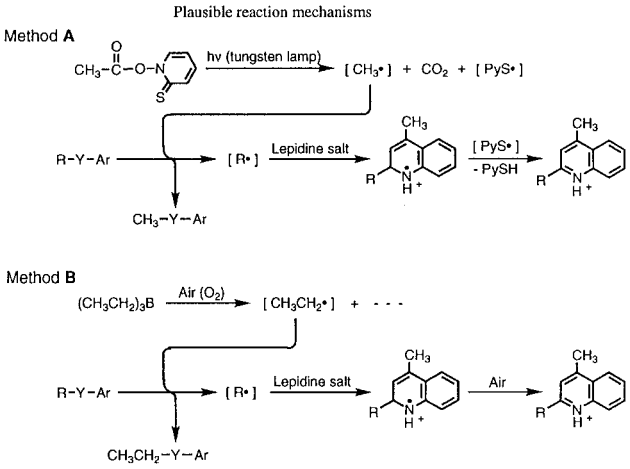
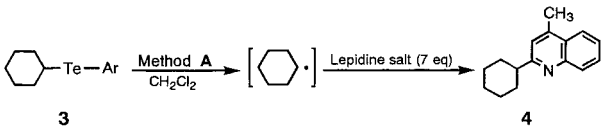


TABLE 2 Substituent Effect of Alkyl Aryl Tellurides



3 4

Entry	Ar–	δ (ppm, $CDCl_3$) methine position ^{125}Te	^{13}C	^{1}H	Yield / % of **4** R = CH_3	R = CH_3CH_2
1	CH_3O–	635	27.6	3.38	65	20
2	CH_3–	644	27.7	3.43	32	22
3	H–	655	27.9	3.49	31	25
4	Br–	666	28.5	3.47	61	34

Method **A** : R–C(=O)–O–N(S) (R = CH_3 or CH_3CH_2)

Actually, the yields described in Table 2 increased from 21% to 34% gradually and can be argued to support our speculation. Unfortunately, *N*-chloroacetoxy-2-thiopyridone, the putative precursor of the electrophilic chloromethyl radical, could not be prepared because of rapid hydrolysis and decomposition to chloromethyl pyridyl sulfide. Practically, as compared with cyclohexyl *p*-bromophenyl telluride, cyclohexyl *p*-methoxyphenyl telluride could be prepared more easily. So the *p*-methoxyphenyl group was chosen as the preferred aromatic group in the following reactions.

Reactivity of Cyclobutyl Radical

Finally, again as a model study, the reactivity of the cyclobutyl radical toward the electron-deficient heteroaromatic bases, such as lepidine, as compared with the reactivities of cyclopropyl, cyclopentyl, and cyclohexyl radicals, was studied. The radicals were formed from the corresponding cycloalkyl *p*-methoxyphenyl tellurides (**5**) using methods A and B, and then their reactions with protonated lepidine were carried out under the same reaction conditions to give compounds **6.**

Here, the reactions utilizing *N*-acetoxy-2-thiopyridone and triethylborane were effective. However, *N*-propionoxy-2-thiopyridone was not effective because of the formation of ethyl pyridyl sulfide and 2-ethyl-4-methylquinoline. The results are shown in Figure 1 and suggest that cyclohexyl, cyclopentyl, and cyclobutyl radicals, possessing similar reactivities, are more reactive than the cyclopropyl radical. This could be explained by the increasing s-character of the cyclopropyl radical that reduces its nucleophilicity. The reactivity of these cycloalkyl radicals corresponds fairly well to the calculated ionization potentials of those radicals based on **AM1** calculations.

Thus, the present method makes it possible to form the cyclobutyl radical from cyclobutyl *p*-methoxyphenyl telluride by methods A and B and to prepare the cyclobutylated heteroaromatic bases.

Preparation of Cyclobutyl C-Nucleoside Analogs

The compound **8** was prepared from allyl bromide in four steps, as outlined in Scheme 2, based on the literature method [10]. Compound **8** was treated with sodium borohydride in ethanol solution at 0°C to give 3-(benzyloxymethyl)cyclobutanol **9** as a mixture of stereoisomers (*cis/trans:* 4/1). The alcohol **9** was converted into the mesylate **10** that was subsequently treated with the 4-methoxyphenyl telluride anion to produce the corresponding cyclobutyl *p*-methoxyphenyl telluride **11** (*cis/trans:* 1/4). This telluride had sufficient stability to enable us to carry out the following reactions. The telluride **11** was irradiated (500 W tungsten lamp) in the presence of *N*-acetoxy-2-thiopyridone and various heteroaromatic bases under an argon atmosphere (method A) or treated with triethylborane in the presence of heteroaromatic bases under aerobic conditions (method B) to give the corresponding products **12** in moderate yields as shown in Table 3. Compounds **12** could be easily deprotected with boron trichloride to give compounds **13** in high yields (Scheme 3).

The key step for the synthesis of cyclobutyl *C*-nucleosides is the radical substitution reaction of the cyclobutyl radical with the appropriate heteroaromatic bases. The present method has advantages such as the short synthetic route to *C*-nucleosides containing a cyclobutyl ring in place of a sugar moiety, facile deprotection, and its application to various heteroaromatic bases. Further work along these lines is underway.

EXPERIMENTAL

General

Melting points were determined on a Yamato Model MP-21 instrument. IR spectra were recorded on a Hitachi 215 spectrometer. ^{1}H- and ^{13}C-NMR spectra were taken [deuteriochloroform with tetramethylsilane (TMS) as the internal reference] with JNM-FX-270, JNM-GSX-400, and JNM-GSX-500 spectrometers. Chemical shifts (δ) are expressed as δ values, relative to TMS, and *J* values are given in hertz. Carbon signals were assigned by DEPT and INEPT pro-

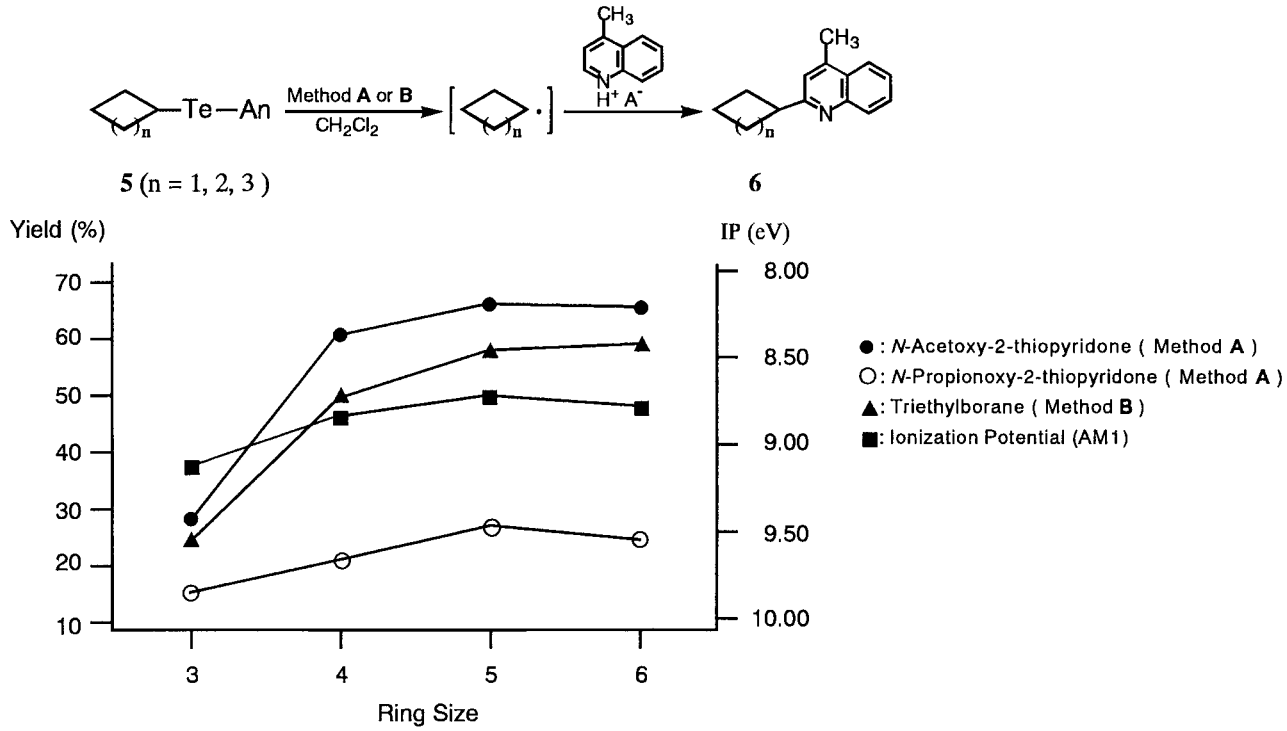
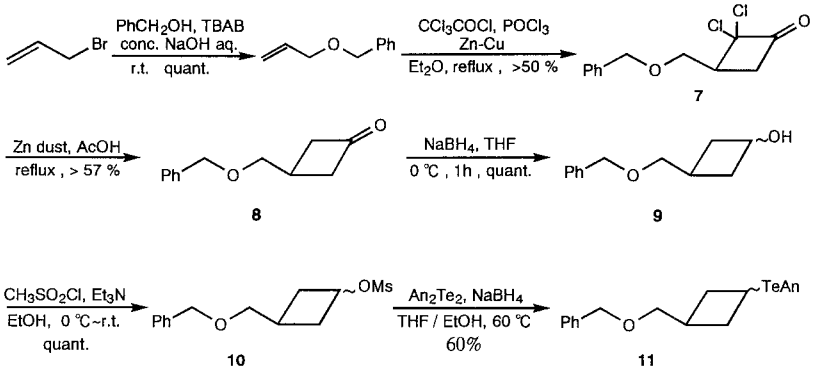


FIGURE 1 Reactivities of cycloalkyl radicals.



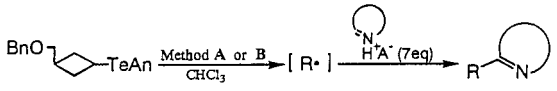
SCHEME 2 Synthesis of cyclobutyl telluride derivative.

grams. 2D-NMR (COSY and NOESY) data were recorded on JEOL JNM-GSX-400 and JNM-GSX-500 spectrometers. Mass spectra were obtained on Hitachi M-60 and JEOL HX-110 mass spectrometers. Elemental analyses were performed on a Perkin-Elmer 240 elemental analyzer at the Chemical Analysis Center of Chiba University. TLC analyses were performed on thin-layer analytical plates of Kieselegel $60F_{254}$ (E. Merck, Darmstadt) and Wakogel B-5F. Silica gel column chromatography was carried out on Wakogel C-200 or C-300. Reactions were carried out under a dry argon atmosphere unless otherwise stated.

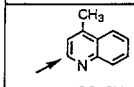
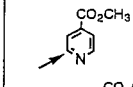
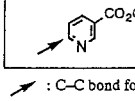
General Procedure for the Alkylation of Heteroaromatic Bases

Method A. A mixture of the chalcogenide (1 mmol), 7 equiv. of 4-methylquinolinium trifluoroacetate, and 2 or 4 equiv. of *N*-acetoxy-2-thiopyridone was dissolved in dry dichloromethane (4 mL) under an argon atmosphere. The solution was irradiated with a 500 W tungsten lamp for 1 hour at 30–40 °C. The resulting solution was hydrolyzed with sat. aq. sodium hydrogen carbonate. The organic layer was extracted with $CHCl_3$ twice and dried over Na_2SO_4. After removal of the solvent under reduced pressure,

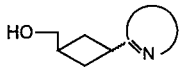
TABLE 3 Preparation of Cyclobutyl *C*-Nucleosides



11 (*cis* / *trans* : *1* / *4*) 12

Base	Yields / % (*cis* / *trans*) Method A	Method B
 (a)	68 (*cis* / *trans* = 1 / 1)	63
 (b)	40 (*cis* only)	32
 (c)	38 (*cis* / *trans* = 1 / 1)	30

: C–C bond forming position.



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SCHEME 3

the residual oil was purified by pTLC on silica gel ($CHCl_3$, or Hexane/EtOAc = 2/1–3/1).

Method B. A mixture of a chalcogenide (1 mmol) and 7 equiv. of 4-methylquinolinium trifluoroacetate was dissolved in dry dichloromethane (4 mL). To the solution was added triethylborane (4 equiv.) as a THF solution (1 M). Triethylborane was again added after 2 and 4 hours, respectively. Then the mixture was stirred for 7–8 hours at room temperature under aerobic conditions. The resulting solution was hydrolyzed with sat. aq. $NaHCO_3$. The organic layer was extracted with CH_2Cl_2 twice and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the residual oil was purified by pTLC on silica gel ($CHCl_3$, or Hexane/EtOAc = 2/1–3/1).

Preparation of Adamantyl 4-Methoxyphenyl Sulfide and Adamantyl 4-Methoxyphenyl Selenide [11]

The reaction was carried out with a molar ratio of disulfide/the [bis (1-adamantanecarboxy)iodo]arene of 0.7/1.0. A solution of the disulfide and the [bis (1-adamantanecarboxy)iodo]arene in CH_2Cl_2 (5 mL) was irradiated with a high-pressure mercury lamp (400 W) for 3 hours at 30°C. The resulting solution was hydrolyzed with sat. aq. sodium hydrogen carbonate (20 mL). The organic layer was extracted with CH_2Cl_2 (3 × 20 mL) and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the residual oil was purified by pTLC on silica gel (Hexane/EtOAc = 4/1–2/1).

1-Adamantyl 4-Methoxyphenyl Sulfide. Mp 65.0–66.0°C; IR (KBr) 2860, 1580, 1480, 1340, 1240, 835, and 800 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.64–2.14 (15H, m, Ad), 3.82 (3H, s, OCH_3), 6.84 (2H, d, J = 8.9 Hz, Ar), 7.40 (2H, d, J = 8.9 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ= 29.94 (Ct, Ad), 36.20 (Cs, Ad), 43.44 (Cs, Ad), 47.42 (Cq, Ad), 55.27 (Cp, CH_3), 113.80 (Ct, Ar), 121.40 (Cq, Ar), 139.01 (Ct, Ar), 160.14 (Cq, Ar). Found: C, 73.85; H, 8.30%. Calcd for $C_{17}H_{22}OS$: C, 74.40; H, 8.08%; HRMS (FAB) found: 274.1389. Calcd for $C_{17}H_{22}OS$: 274.1391.

1-Adamantyl 4-Methoxyphenyl Selenide. IR (KBr) 2870, 1580, 1490, 1350, 1250, and 830 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.55–1.94 (15H, m, Ad), 3.82 (3H, s, CH_3), 6.82 (2H, d, J = 8.8 Hz, Ar), 7.51 (2H, d, J = 8.8 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 30.67 (Ct, Ad), 36.20 (Cs, Ad), 44.50 (Cs, Ad), 46.43 (Cq, Ad), 55.21 (Cp, CH_3), 114.12 (Ct, Ar), 116.97 (Cq, Ar); 139.69 (Ct, Ar), 159.96 (Cq, Ar); HRMS (FAB) found: 322.0845. Calcd for $C_{17}H_{22}O^{80}Se$: 322.0837.

Typical Procedure for the Preparation of Diaryl Ditellurides [12]

Method 1. A mixture of tellurium tetrachloride (9 mmol) and an arene (27 mmol) was refluxed in CCl_4 (64 mL) for 6–24 hours. The reaction mixture was cooled in an ice bath and filtered to give a gray solid. This compound was dissolved and stirred in a two-phase system with CH_2Cl_2 (38 mL) and distilled water (38 mL). $Na_2S_2O_5$ (0.8 equiv.) was added in small portions over a period of 5 minutes, and the reaction mixture was stirred for a further 1.5 hours at room temperature. The resulting solution was extracted with $CHCl_3$ twice, and the extract was concentrated and dried in vacuo. A reddish-brown solid was obtained in 90–96% yield.

Method II. *t*-Butyllithium (16 M, 24 mmol) was added dropwise to a solution of an aryl bromide (12 mmol) in THF (60 mL) at −78°C under an argon atmosphere. After 1 hour, the cooling bath was removed and the mixture was allowed to warm at room temperature for 30 minutes. Tellurium powder (12 mmol) was then added rapidly. After 2 hours, the mixture was poured into a separatory funnel containing $K_3Fe(CN)_6$ (12 mmol) in water (220 mL). The

product was extracted with $CHCl_3$ (60 mL). After drying of the extract with Na_2SO_4, it was filtered through a Celite pad and purified by flash chromatography (hexane) to give 50–65% of the ditelluride.

Bis(4-methoxyphenyl) Ditelluride. Mp 58.5–60.5°C (Ref. [12] mp 58–59°C); IR (KBr) 1490, 1290, 1250, 1070, 1030, 810 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 3.80 (6H, s, OCH_3), 6.75 (4H, d, J = 8.8 Hz, Ar), 7.70 (4H, d, J = 8.8 Hz, Ar).

Diphenyl Ditelluride. Mp 64.5–66.3°C (Ref. [12] mp 64–65°C); IR (KBr) 1470, 1430, 1060, 890, 740 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 6.97–7.84 (10H, m, Ar).

Bis(4-methylphenyl) Ditelluride. Mp 50.5–51.2°C (Ref. [12] mp 51–52°C); IR (KBr) 1540, 1480, 1010, 790 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 2.37 (6H, s, CH_3), 7.00 (4H, d, J = 8.1 Hz, Ar), 7.67 (4H, d, J = 8.1 Hz, Ar).

Bis(4-bromophenyl) Ditelluride. Mp 151.5–152.3°C (Ref. [12] mp 151–152°C); IR (KBr) 1480, 1440, 1370, 1070, 1050, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 7.30 (4H, d, J = 8.2 Hz, Ar), 7.61 (4H, d, J = 8.2 Hz, Ar).

Preparation of Adamantyl 4-Methoxyphenyl Telluride [13]. Bis(4-methoxyphenyl) ditelluride (1.5 mmol) and *N*-adamantanecarboxy-2-thiopyridone (0.75 mmol) were dissolved in dichloromethane and stirred at room temperature. After 2 hours, *N*-adamantanecarboxy-2-thiopyridone (0.375 mmol) was added again and stirred for another 2 hours under room light. The reaction mixture was concentrated and purified by column chromatography (hexane / $CHCl_3$ = 6 / 5) to give an orange solid in about 80% yield.

Adamantyl 4-Methoxyphenyl Telluride. IR (KBr) 2860, 1570, 1480, 1280, 1250, 1180, 1030, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.72 (6H, bs, Ad), 1.90 (3H, bs, Ad), 2.15–2.20 (6H, m, Ad), 3.82 (3H, s, OCH_3), 6.78 (2H, dd, J = 8.9 and 2.3 Hz, Ar), 7.74 (2H, dd, J = 8.9 and 2.3 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 31.28 (Ct, Ad), 34.94 (Cq, Ad), 36.31 (Cs, Ad), 47.20 (Cs, Ad), 55.10 (Cp, OCH_3), 101.20 (Cq, Ar), 114.82 (Ct, Ar), 143.81 (Ct, Ar), 159.96 (Cq, Ar); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te) δ = 837.81; HRMS (FAB) found: 372.0739. Calcd. for $C_{17}H_{22}{}^{130}Te$: 372.0733.

Typical Procedure for Preparation of Alkyl Aryl Tellurides. Bis(4-methoxyphenyl) ditelluride (0.5 mmol) was dissolved in a mixture of THF (10 mL) and EtOH (5 mL). $NaBH_4$ (4 equiv.) was added to the solution. After 30 minutes, an alkyl bromide (3 equiv.) was added to this solution, then the mixture was heated at 50°C under an Ar atmosphere for 5 hours. The resulting solution was extracted with $CHCl_3$ twice and the extract dried over Na_2SO_4. This extract was concentrated and purified by column chromatography (hexane / $CHCl_3$ = 6 / 5). An orange oil was obtained in 80% yield.

n-Pentyl 4-Methoxyphenyl Telluride. IR (NaCl) 2880, 2840, 1590, 1490, 1280, 1250, 1180, 1030, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 0.88 (3H, t, J = 7.6 Hz, CH_3), 1.22–1.38 (4H, m, CH_2), 1.75 (2H, quintet, J = 8.0 Hz, CH_2), 2.82 (2H, t, J = 8.0 Hz, CH_2Te), 3.80 (3H, s, OCH_3), 6.76 (2H, dd, J = 8.9 and 2.2 Hz, Ar), 7.72 (2H, dd, J = 8.9 and 2.2 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 9.05 (Cs, CH_2), 13.98 (Cp, CH_3), 22.00 (Cs, CH_2), 31.42 (Cs, CH_2), 34.07 (Cs, CH_2), 55.13 (Cp, OCH_3), 100.62 (Cq, Ar), 115.07 (Ct, Ar), 140.89 (Ct, Ar), 159.63 (Cq, Ar); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te) δ = 455.202; HRMS (FAB) found 308.0428. Calcd. for $C_{12}H_{18}O^{130}Te$: 308.0420.

Cyclohexyl 4-Methoxyphenyl Telluride. The yield was 93%. IR (NaCl) 2880, 2840, 1570, 1480, 1280, 1240, 1170, 1030, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.20–1.40 (2H, m, CH_2), 1.50–1.70 (6H, m, CH_2), 2.00–2.12 (2H, m, CH_2), 3.38 (1H, tt, J = 11.6 and 3.9 Hz, CH), 3.80 (3H, s, OCH_3), 6.76 (2H, dd, J = 8.9 and 2.0 Hz, Ar), 7.72 (2H, dd, J = 8.9 and 2.0 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 25.82 (Cs, CH_2), 27.57 (Ct, CHTe), 28.21 (Cs, CH_2), 36.24 (Cs, CH_2), 55.10 (Cp, OCH_3), 100.68 (Cq, Ar), 114.95 (Ct, Ar), 142.36 (Ct, Ar), 159.77 (Cq, Ar); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te) δ = 635.02; HRMS (FAB) found 320.0420. Calcd. for $C_{13}H_{18}{}^{130}Te$: 320.0420.

Cyclohexyl 4-Methylphenyl Telluride. The yield was 50%. IR (NaCl) 2880, 2840, 1540, 1480, 1020, 790 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.22–1.40 (2H, m, CH_2), 1.52–1.72 (6H, m, CH_2), 2.04–2.12 (2H, m, CH_2), 2.35 (3H, s, CH_3), 3.43 (1H, tt, J = 10.0 and 3.6 Hz, CHTe), 7.03 (2H, d, J = 7.8 Hz, Ar), 7.69 (2H, dd, J = 7.8 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 21.27 (Cp, CH_3), 25.82 (Cs, CH_2), 27.72 (Ct, CHTe), 28.20 (Cs, CH_2), 36.31 (Cs, CH_2), 107.42 (Cq, Ar), 129.96 (Ct, Ar), 137.81 (Cq, Ar), 140.41 (Ct, Ar); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te) δ = 644.04; HRMS (FAB) found 304.0478. Calcd. for $C_{13}H_{18}{}^{130}Te$: 304.0471.

Cyclohexyl Phenyl Telluride. The yield was 80%.

IR (NaCl) 1470, 1430, 1060, 990, 740 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.20–1.40 (2H, m, CH_2), 1.52–1.70 (6H, m, CH_2), 2.05–2.18 (2H, m, CH_2), 3.49 (1H, tt, J = 10.4 and 3.7 Hz, CHTe), 7.17–7.33 (3H, m, Ph), 7.79 (2H, dd, J = 8.3 and 1.3 Hz, Ph); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 25.82 (Cs, CH_2), 27.98 (Ct, CHTe), 28.17 (Cs, CH_2), 36.33 (Cs, CH_2), 111.61 (Cq, Ph), 127.72 (Ct, Ph), 128.99 (Ct, Ph), 140.01 (Ct, Ph); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te), δ = 655.24; HRMS (FAB) found 290.0321. Calcd. for $C_{12}H_{16}{}^{130}Te$: 290.0393.

Cyclohexyl 4-Bromophenyl Telluride. The yield was 30%. IR (NaCl) 2890, 2820, 1460, 1440, 1370, 1070, 1010, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$), δ = 1.20–1.45 (2H, m, CH_2), 1.50–1.78 (6H, m, CH_2), 2.00–2.15 (2H, m, CH_2), 3.47 (1H, tt, J = 10.3 and 3.7 Hz, CHTe), 7.32 (2H, dt, J = 8.2 and 2.0 Hz, Ar), 7.63 (2H, dt, J = 8.2 and 2.0 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 25.76 (Cs, CH_2), 28.17 (Cs, CH_2), 28.49 (Ct, CHTe), 36.27 (Cs, CH_2), 109.76 (Cq, Ar), 122.70 (Cq, Ar), 132.22 (Ct, Ar), 141.73 (Ct, Ar); ^{125}Te NMR (82.5 MHz, $CDCl_3$, Me_2Te) δ = 666.68; HRMS (FAB) found 367.9417. Calcd. for $C_{12}H_{15}{}^{79}Br{}^{130}Te$: 367.9419.

2-(1-Admantyl)-4-methylquinoline. Mp 120.3–122.0°C; IR (KBr) 3040, 2880, 2840, 1590, 1440, 760 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.80 (6H, bs, Ad), 2.07 (9H, bs, Ad), 2.60 (3H, s, CH_3), 7.15 (1H, s, Ar 3-H), 7.30–7.60 (2H, m, Ar 6- and 7-H), 7.80 (1H, d, J = 9.0 Hz, Ar 5-H), 7.90 (1H, d, J = 9.0 Hz, Ar 8-H); MS (FAB) M^+ = 227, $C_{20}H_{23}N$; found: C, 86.42; H, 8.27; N, 5.23%. $C_{20}H_{23}N$ requires C, 86.59; H, 8.36; N, 5.05%.

2-Cyclohexyl-4-methylquinoline. Oil; IR (NaCl) 2910, 1600, 1450, 770 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.25–2.05 (10H, m, Cy), 2.68 (3H, s, CH_3), 2.88 (1H, m, Cy), 7.15 (1H, s, Ar 3-H), 7.34–7.70 (2H, m, Ar 6- and 7-H), 7.95 (1H, d, J = 9.0 Hz, Ar 5-H), 8.20 (1H, d, J = 9.0 Hz, Ar 8-H); MS (FAB) M^+ = 225, $C_{16}H_{19}N$. Found: C, 85.31; H, 8.59; N, 6.49%. $C_{16}H_{19}N$ requires C, 85.28; H, 8.50; N, 6.22%.

Cyclopropyl 4-Methoxyphenyl Telluride. Oil; IR (NaCl) 2975, 1700, 1425, 1360, 1225 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$), δ = 0.73–1.15 (4H, m, cyclopropyl), 2.15–2.18 (1H, m, cyclopropyl), 3.81 (3H, s, OCH_3), 6.80 (2H, dd, J = 8.8 and 1.8 Hz, Ar), 7.67 (2H, dd, J = 8.8 and 1.8 Hz, Ar); HRMS (FAB) found 277.9951. Calcd. for $C_{10}H_{12}O{}^{130}Te$: 277.9950.

Cyclobutyl 4-Methoxyphenyl Telluride. Oil; IR (NaCl) 2940, 1580, 1480, 1280, 1240, 1180, 1030, 820 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$), δ = 1.90–2.04 (2H, m, cyclobutyl), 2.17–2.28 (2H, m, cyclobutyl), 2.38–2.47 (2H, m, cyclobutyl), 3.81 (3H, s, OCH_3), 4.02 (1H, quintet, J = 8.4 Hz, CH), 6.78 (2H, dd, J = 8.8 and 2.2 Hz, Ar), 7.69 (2H, dd, J = 8.8 and 2.2 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 16.62 (Ct, CHTe), 23.09 (Cs, CH_2), 32.67 (Cs, CH_2), 55.12 (Cp, OCH_3), 101.08 (Cq, Ar), 115.01 (Ct, Ar), 141.95 (Ct, Ar), 159.78 (Cq, Ar); HRMS (FAB) found 292.0107. Calcd. for $C_{11}H_{14}O{}^{130}Te$: 292.0107.

Cyclopentyl 4-Methoxyphenyl Telluride. Oil; IR (NaCl) 2950, 1600, 1500, 1290, 1260, 1180, 1030, 830 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ = 1.45–1.60 (2H, m, CH_2), 1.60–1.75 (4H, m, CH_2), 2.20–2.10 (2H, m, CH_2), 3.49 (1H, quintet, J = 7.3 Hz, CH), 3.79 (3H, s, OCH_3), 6.75 (2H, dd, J = 8.8 and 2.2 Hz, Ar), 7.72 (2H, dd, J = 8.8 and 2.2 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 23.49 (Ct, CHTe), 25.29 (Cs, CH_2), 35.84 (Cs, CH_2), 55.03 (Cp, OCH_3), 101.32 (Cq, Ar), 114.89 (Ct, Ar), 141.89 (Ct, Ar), 159.68 (Cq, Ar); HRMS (FAB) found 306.0264. Calcd. for $C_{12}H_{16}O{}^{130}Te$: 306.0263.

Preparation of [3-(Benzyloxymethyl)cyclobutyl] 4-Methoxyphenyl Telluride Derivative: 3-(Benzyloxymethyl)cyclobutanol was Prepared with the Literature Method [10]

3-(Benzyloxymethyl)cyclobutanol (4 mmol) and triethylamine (7.2 mmol) were dissolved in dry THF (15 mL). This solution was then cooled to 0°C and methanesulfonyl chloride (7.2 mmol) in dry THF (5 mL) was added dropwise to the solution, and the mixture was stirred for 1 hour at room temperature and then filtered through a Celite pad. The filtrate was evaporated under reduced pressure to afford a light yellow oil. The residue was treated with Na^+AnTe^- prepared by reduction of An_2Te_2 (0.75 mmol) with $NaBH_4$ (2.4 mmol) in a mixture of THF (10 mL) and EtOH (5 mL). The mixture was stirred for 6 hours at 45 °C. After the reaction, the mixtures was treated with water. The residue was extracted with $CHCl_3$ (3 × 40 mL) and the extract dried over Na_2SO_4. The solvent was evaporated to yield the crude product. It was purified by pTLC on silica gel ($CHCl_3$) to give the telluride **11** in 70% yield (*cis:trans* = 1:4). Oil; IR (NaCl) 2900, 2850, 2350, 1585, 1490, 1290, 1250, 1180, 1110, 1040, 840, 750, 710 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.32–2.37 (4H, m, cyclobutyl), 2.66–2.70 (1H, m, cyclobutyl-CH), 3.48 (2H, d, J = 7.0 Hz, OCH_2), 3.79 (3H, s, OCH_3), 4.02 (1H, quintet, J = 7.8 Hz, CHTe), 4.48 (2H, s, $PhCH_2$), 6.77 (2H, dd, J = 8.8 and 2.2 Hz, Ar), 7.25–7.35 (5H, m, Ph), 7.69 (2H, dd, J = 8.8 and 2.2 Hz, Ar); HRMS

(FAB) found 412.0868. Calcd. for $C_{19}H_{22}O_2{}^{130}Te$: 412.0683.

Typical Procedure for the Preparation of Carbocyclic C-Nucleoside Analogs 12

Method A. To a solution of [3-(benzyloxymethyl)cyclobutyl] 4-methyoxyphenyl telluride (0.5 mmol, 205 mg) in dry $CHCl_3$ (4 mL) were added the trifluoroacetate salt of heteroaromatic compounds (3.5 mmol) and *N*-acetoxy-2-thiopyridone (338 mg, 2.5 mmol) at 0°C. After having been stirred for 5 minutes, the mixture was irradiated with a 500 W tungsten lamp for 1 hour at the range of 30–35°C, then the mixture was hydrolyzed with sat. aq. $NaHCO_3$, extracted with dichloromethane, the extract was dried over Na_2SO_4, filtered, and finally concentrated. The residue was chromatographed (ethyl acetate / hexane: 1 / 2) to give the products of **12a–c** in moderate yields.

Method B. To a solution of the [3-(benzyloxymethyl)cyclobutyl] 4-methoxyphenyl telluride (0.5 mmol, 205 mg) in dry $CHCl_3$ (4 mL) were added the trifluoroacetate salt of a heteroaromatic compound (3.5 mmol) and triethylborane (4 equiv.) as a THF solution (1 M) at room temperature. Triethylborane was again added after 2 and 4 hours, respectively. Then, the mixture was stirred for 7–8 hours at room temperature under aerobic conditions. The resulting solution was hydrolyzed with sat. aq. $NaHCO_3$. The organic layer was extracted with $CHCl_3$ twice and the extract dried over Na_2SO_4. After removal of the solvent under reduced pressure, the residual oil was purified by pTLC on silica gel (hexane / EtOAc = 2 / 1).

2-[cis-3-(Benzyloxymethyl)cyclobutyl]-4-methylquinoline **12a.** Oil; IR (NaCl) 2950, 2800, 1590, 1440, 1090, 760, 740, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.27–2.33 (2H, m, cyclobutyl), 2.55–2.66 (2H, m, cyclobutyl), 2.66 (3H, s, CH_3), 2.65–2.80 (1H, m, cyclobutyl), 3.66 (2H, d, J = 7.0 Hz, OCH_2), 3.83 (1H, quintet, J = 8.2 Hz, CH), 4.58 (2H, s, OCH_2Ph), 7.19 (1H, s, Ar 3-H), 7.28–7.39 (5H, m, Ph), 7.48 (1H, t, J = 8.3 Hz, Ar 6-H), 7.65 (1H, t, J = 8.3 Hz, Ar 7-H), 7.92 (1H, d, J = 8.3 Hz, Ar 5-H), 8.05 (1H, d, J = 8.3 Hz, Ar 8-H); ^{13}C NMR (100 MHz, $CDCl_3$), δ = 18.72 (Cp, CH_3), 29.90 (Cs, CH_2), 30.91 (Ct, CH), 39.20 (Ct, CH), 73.06 (Cs, CH_2), 74.21 (Cs, CH_2), 120.28, 123.50, 125.38, 127.49, 127.63, 128.35, 128.93, 129.47 (Ct, Ar), 126.80, 138.61, 144.18, 147.52, 164.73 (Cq, Ar); NOE (Ar-3H ⟷ Ph$\underline{CH_2}$) was observed; HRMS (FAB) found 318.1862. Calcd. for $C_{22}H_{24}NO$ 318.1858. (Found: C, 82.74; H, 7.53 ; N, 4.42%. Calcd. for $C_{22}H_{23}NO$: C, 82.98; H, 7.59; N, 4.39%.)

2-[trans-3-(Benzyloxymethyl)cyclobutyl]-4-methylquinoline **12a.** Oil; IR (NaCl) 2950, 2800, 1590, 1440, 1090, 760, 740, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.22–2.30 (2H, m, cyclobutyl), 2.54–2.63 (2H, m, cyclobutyl), 2.66 (3H, s, CH_3), 2.63–2.80 (1H, m, cyclobutyl), 3.53 (2H, d, J = 5.9 Hz, OCH_2), 3.71 (1H, quintet, J = 8.4 Hz, CH), 4.56 (2H, s, $PhCH_2$), 7.20 (1H, s, Ar 3-H), 7.26–7.40 (5H, m, Ph), 7.50 (1H, t, J = 8.2 Hz, Ar 6-H), 7.67 (1H, t, J = 8.2 Hz, Ar 7-H), 7.94 (1H, d, J = 8.2 Hz, Ar 5-H), 8.05 (1H, d, J = 8.2 Hz, Ar 8-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 19.04 (Cp, CH_3), 31.62 (Ct, CH), 31.68 (Cs, CH_2), 39.05 (Ct, CH), 73.34 (Cs, OCH_2), 75.06 (Cs, OCH_2), 120.52, 123.84, 125.73, 127.78, 127.89, 128.64, 129.25, 129.85 (Ct, Ar), 127.18, 139.02, 144.46, 147.82, 164.75 (Cq, Ar); HRMS (FAB) found 318.1857. Calcd. for $C_{22}H_{24}NO$: 318.1858.

Methyl 2-[cis-3-(Benzyloxymethyl)cyclobutyl] isonicotinate **12b.** Oil; IR (NaCl) 2925, 2825, 1720, 1550 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.22–2.30 (2H, m, cyclobutyl), 2.45–2.55 (2H, m, cyclobutyl), 2.65–2.75 (1H, m, cyclobutyl), 3.65 (2H, d, J = 6.9 Hz, OCH_2), 3.78 (1H, J = 8.3 Hz, CHAr), 3.95 (3H, s, OCH_3), 4.58 (2H, s, $PhCH_2$), 7.22–7.40 (5H, m, Ph), 7.65 (1H, d, J = 5.1 Hz, Ar 5-H), 7.74 (1H, s, Ar 3-H), 8.71 (1H, d, J = 5.1 Hz, Ar 6-H); NOE ($PhCH_2OCH_2$ ⟷ Ar 6-H, Ph ⟷ CH_3) was observed; HRMS (FAB) found 312.1598. Calcd. for $C_{19}H_{22}NO_3$: 312.1600.

Methyl 6-[3-(Benzyloxymethyl)cyclobutyl] nicotinate **12c** *(mixture of cis / trans: 1 / 1).* Oil; IR (NaCl) 2900, 2820, 1720, 1500 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.05–2.18 (2H, m, cyclobutyl), 2.45–2.58 (2H, m, cyclobutyl), 2.62–2.72 (1H, m, cyclobutyl), 3.49 (2H, d, J = 6.2 Hz, OCH_2), 3.60 (1H, quintet, J = 7.0 Hz, CHAr), 3.94 (3H, s, CH_3), 4.53 (2H, s, $PhCH_2$), 7.20–7.40 (5H, m, Ph), 7.23 (1H, d, J = 8.1 Hz, Ar 5-H), 8.18 (1H, dd, J = 8.1 and 1.5 Hz, Ar 4-H), 9.13 (1H, d, J = 1.5 Hz, Ar 2-H); HRMS (FAB) found 312.1516. Calcd. for $C_{19}H_{22}NO_3$: 312.1600.

Typical Procedure for the Deprotection of Compounds **12.** Each compound **12** (0.17 mmol) was dissolved in $CHCl_3$ (12 mL) at −78°C. After addition of BCl_3 (1 M solution of CH_2Cl_2), the mixture was stirred for 1 hour at −78°C and then was warmed up to 0°C. Then solid $NaHCO_3$ was added and the mixture stirred for another 5 minutes. The reaction mixture was filtered, concentrated, and the residue purified by PTLC ($CHCl_3$/Methanol: 9/1) to give compound **13** (34 mg) in 90% yield.

2-[trans-3-(Hydroxymethyl)cyclobutyl]-4-methy-

lquinoline **13a.** Oil; IR (NaCl) 3300, 2900, 2860, 2800, 1580, 1540, 1430, 1020, 760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ = 2.26–2.40 (2H, m, CH_2Ar), 2.50 (1H, bs, OH), 2.55–2.71 (3H, m, cyclobutyl-CH_2), 2.68 (3H, s, CH_3), 3.69 (1H, quintet, J = 8.4 Hz, CHAr), 3.75 (2H, d, J = 4.4 Hz, OCH_2), 7.14 (1H, s, Ar 3-H), 7.52 (1H, t, J = 8.3 Hz, Ar 6-H), 7.69 (1H, t, J = 8.3 Hz, Ar 7-H), 7.95 (1H, d, J = 8.3 Hz, Ar 5-H), 8.12 (1H, d, J = 8.3 Hz, Ar 8-H); HRMS (FAB) found 228.1391. Calcd. for $C_{15}H_{18}NO$: 228.1388. (Found: C, 7.93; H, 79.01; N, 6.16%. Calcd. for $C_{15}H_{17}NO$: C, 7.95; H, 78.91; N, 6.14%.)

ACKNOWLEDGMENT

We thank Dr Soichi Sato of Tsukuba University for Tellurium NMR measurements.

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