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Syntheses of β-Amidovinyltellurides and Oxazoles by Addition Reactions of Alkynes with Benzenetellurinyl Trifluoromethanesulfonate in Acetonitrile

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ABSTRACT

Benzenetellurinyl trifluoromethanesulfonate in conjunction with acetonitrile readily effected amidotellurinylation reactions with alkynes. Most of the addition reactions proceeded in a trans fashion to form the (E)-β-acetamidovinyl phenyl telluroxides. Subsequently, it was found that the prevailing Markovnikov adducts from terminal alkynes immediately isomerized to (Z)-isomers which were isolated as the corresponding vinyltellurides. On the other hand, the adducts derived from internal alkynes thermally underwent a spontaneous intramolecular cyclization to be transformed eventually into oxazoles.

INTRODUCTION

Benzenetellurinic mixed anhydrides, such as benzenetellurinyl acetate, trifluoroacetate, and trifluoromethanesulfonate, serve as superior nucleophiles toward alkenes to undergo versatile addition reactions, such as amidotellurinylation [1], aminotellurinylation [2], and oxytellurinylation [3]. These reactions serve as an entry for elegant organic syntheses based on tellurium-promoted methodology. For example, amidotellurinylation and aminotellurinylation reactions with alkenes carried out at a high temperature are accompanied by ready intramolecular cyclization of the adducts, with subsequent displacement of the introduced tellurinyl group, to give 4,5-dihydrooxazoles and oxazolidin-2-ones, respectively [1,2]. These reaction sequences constitute a facile one-pot method of synthesis of five-membered heterocycles from alkenes. If a similar reaction sequence were to occur with alkynes, one might expect, not only the initial formation of vinyltelluroxides, but also the subsequent conversion of the initial adducts into five-membered aromatic heterocycles. This has led us to examine additional reactions of alkynes with benzenetellurinyl trifluoromethanesulfonate (**1**) in acetonitrile. We wish to report here on the stereo- and regio-selective synthesis of β-actamidovinyl phenyl tellurides and the one-pot synthesis of oxazoles from alkynes [4].

RESULTS AND DISCUSSION

Treatment of phenylacetylene (**2**, R = Ph, R′ = H) with benzenetellurinyl trifluoromethanesulfonate (**1**) in acetonitrile at room temperature for 12 hours gave an amidotellurinylation product (**3**, R = Ph, R′ = H) which was isolated as β-acetamidostyryl phenyl telluride (**4**) in only 6% yield after reduction with hydrazine hydrate in methanol (Scheme 1). The reaction at reflux temperature enhanced the yield only to 16%. Although an addition of a Lewis acid, $BF_3 \cdot OEt_2$, drastically enhanced amidotellu-

Dedicated to Prof. Antonino Fava on the occasion of his seventieth birthday.

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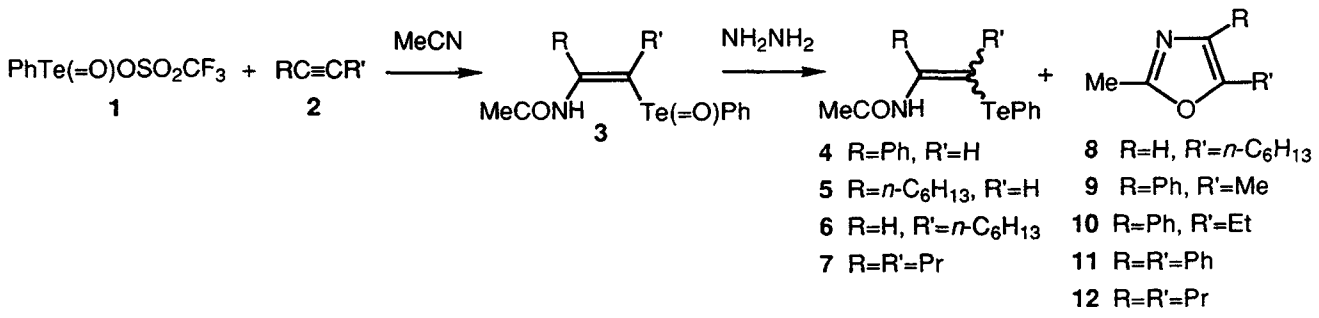


SCHEME 1

TABLE 1 Additional Reactions of Phenylacetylene (**2**, R = Ph, R′ = H) with Benzenetellurinyl Trifluoromethanesulfonate (**1**) in Acetonitrile

Run	*Additive*[a]	*Temperature*	*Time/h*	*Yield of 4/%*
1	none	rt	12	6
2	none	reflux	12	16
3	$BF_3 \cdot OEt_2$	rt	12	13
4	$BF_3 \cdot OEt_2$	reflux	2	18
5	H_2SO_4	rt	12	38
6	CF_3SO_3H	rt	12	11
7	CF_3SO_3H	reflux	2	43

[a]1.5 equiv of additive.

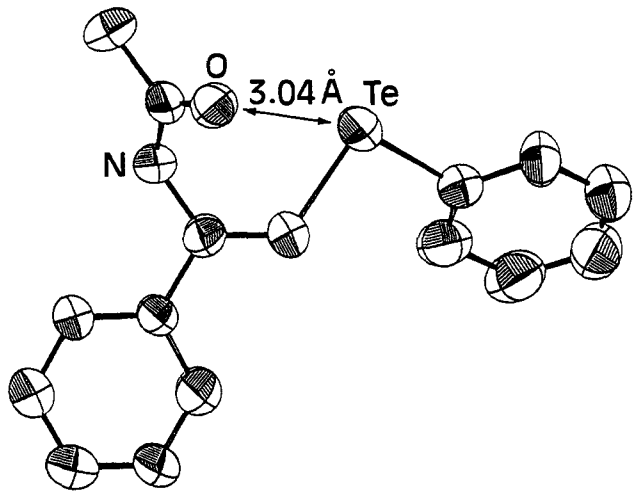


FIGURE 1 ORTEP drawing of (*Z*)-β-acetamidostyryl phenyl telluride (**4**). Hydrogen atoms are omitted.

rinylation of alkenes [1], it was less effective in the present case (Table 1). On the other hand, the addition of a 1.5 equiv amount of a Brønsted acid was fairly effective; the reactions in the presence of sulfuric acid at room temperature and trifluoromethanesulfonic acid under reflux enhanced the yield to 38 and 43%, respectively. NMR analyses showed that the structure of adduct **4** possesses a phenyltelluro group at the terminal position and an acetamido group at the internal position but did not give any information on the stereochemistry of the double bond. The structure was finally confirmed by an X-ray crystallographic analysis to have the *Z* configuration (Figure 1). Neither a regioisomer nor the *E* stereoisomer was detected. This result suggests that the amidotellurinylation of alkynes proceeds in a *cis* fashion with prevailing Markovnikov regioselectivity.

A similar reaction of 1-octyne, unlike the result with phenylacetylene, gave a mixture of products, which consisted mainly of 2-acetamido-1-octenyl phenyl telluride (**5**) and 1-(acetamidomethylene)heptyl phenyl telluride (**6**) in 28–30% yield (Table 2). The regiochemistry of these isomers was also determined by NMR analyses. The isomeric ratio of **5** with its terminal telluro group to **6** with its internal telluro group was 2:3; Markovnikov regioselectivity is absent, probably due to the steric influence of the hexyl group. With regard to stereochemistry, **5** comprised only the *Z*-form, whereas **6** remained in the *E*-form but contained a trace amount of the *Z*-form. Both *Z* configurations were confirmed by NOE difference spectra which showed an enhancement of the allylic methylene signal on irradiation at the alkenic methine signal (Figure 2). In addition to these additional products, 5-hexyl-2-methyloxazole (**8**) was formed in a trace amount.

Internal alkynes behaved differently from terminal alkynes. The reaction of 1-phenyl-1-propyne with **1** in acetonitrile at room temperature for 12 hours or under reflux for 2 hours gave 2,5-dimethyl-4-phenyloxazole (**9**) in 44–46% yield. Neither possible addition product nor the regioisomeric oxazole derivative was formed. The reactions of 1-phenyl-1-butyne and of diphenylacetylene also gave only the corresponding oxazoles **10** and **11**, respectively, in good yields. On the other hand, the reaction of 4-octyne at room temperature for 12 hours gave a mixture of (*Z*)-2-acetamido-1-propyl-1-pentenyl phenyl telluride (**7**) in 36% yield and 4,5-dibutyl-2-methyloxazole (**12**) in 54% yield. This re-

TABLE 2 Additional Reactions of Alkynes (**2**) with Benzenetellurinyl Trifluoromethanesulfonate (**1**) in Acetonitrile

Run	*Alkyne*	*Temp*	*Time/h*	*Product*	*Yield/%*[a]
1	R=Ph, R′=H	reflux	2	(*Z*) − **4**	43
2	R=*n*-C_6H_{13}, R′=H	rt	12	(*Z*) − **5** + (*E*) − **6**[b]	30
3	R=*n*-C_6H_{13}, R′=H	50°C	2	(*Z*) − **5** + (*E*) − **6**[b]	28
4	R=Ph, R′=Me	rt	12	**9**	46
5	R=Ph, R′=Me	reflux	2	**9**	44
6	R=Ph, R′=Et	reflux	2	**10**	57
7	R=R′=Ph	reflux	2	**11**	75
8	R=R′=Pr	rt	12	(*Z*) − **7**	36
				12	54
9	R=R′=Pr	50°C	2	(*Z*) − **7**	19
				12	57
10	R=R′=Pr	−10°C	24	(*E*) − **7**	63
				12	13
11	R=R′=Pr	−10°C then rt	24	(*Z*) − **7**	10
				12	72

[a]*Yields of products after chromatographic separation.*
[b]Isomeric ratio **5**:**6** = 2:3. Besides (*Z*) − **5** and (*E*) − **6**, trace amounts of (*Z*) − **6** and **8** were formed.

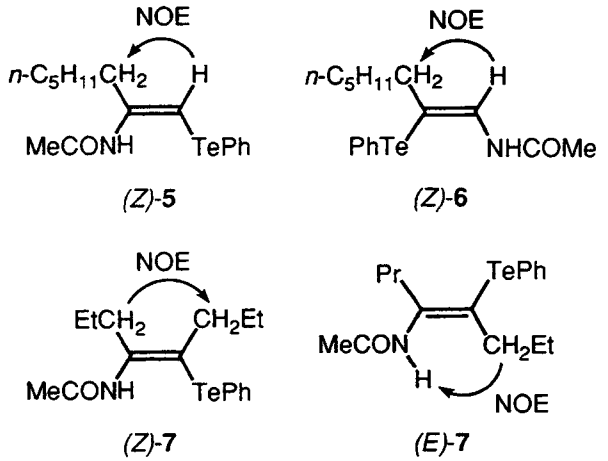


FIGURE 2 Nuclear Overhauser effects observed in the NOE difference spectra.

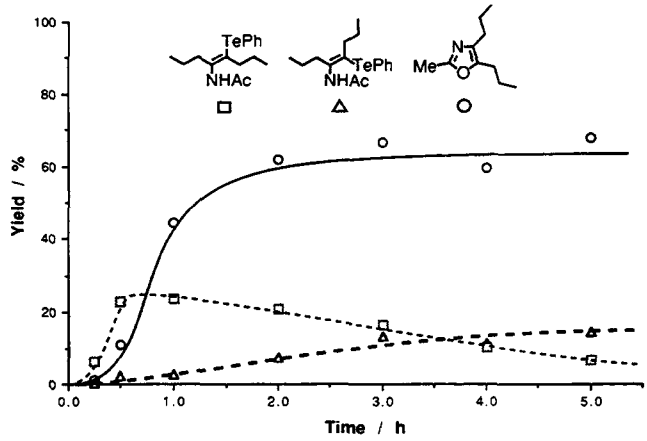
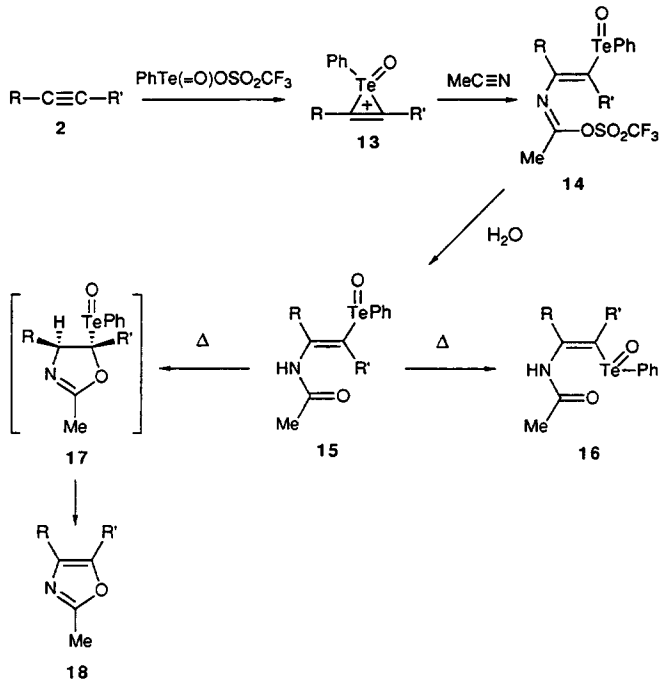


FIGURE 3 Time-dependent formation of (*E*)-adduct **7**, (*Z*)-adduct **7**, and oxazole **12** in the reaction of 4-octyne with benzenetellurinyl trifluoromethanesulfonate (**1**) in acetonitrile at room temperature.

action was greatly temperature-dependent; the reaction at 50°C for 2 hours gave (*Z*)-**7** in 19% yield and **12** in 57% yield, but that at −10°C for 24 hours gave, besides oxazole **12** (13% yield), another addition product (*E*)-**7** (63% yield). In addition, the treatment of 4-octyne at −10°C for 24 hours and then at room temperature for 24 hours gave a mixture of (*Z*)-**7** (10% yield) and **12** (72% yield). The stereochemistry of **7** was also confirmed by an NOE experiment (Figure 2). These results indicate that (*E*)-**7** is initially formed, and this is thermally converted into either (*Z*)-**7** or **12**. This hypothesis was further confirmed by the time-dependent formation of these products, followed in the reaction at room temperature, as shown in Figure 3.

Contrary to the early consideration based on the results of the reaction with phenylacetylene, the results of the 4-octyne reaction clearly indicate that the amidotellurinylation of alkynes **2** proceeds in a *trans* fashion, as shown in Scheme 2. The initially formed *E*-addition product **15** subsequently undergoes either ready thermal isomerization to the *Z*-isomer **16** or to an intramolecular cyclization product to give eventually the oxazole **18**. The former configurational change of the double bond is facilitated by conjugation with both amido and tellurinyl groups. The thermodynamical stability of the *Z*-telluroxides of **4**, **5**, and **7**, as compared to the *E*-isomers, is presumably ascribable to the following two factors. The first is an intramolecular nonbonded Te-O interaction between the amido and tellurinyl groups of the *Z*-form, this assumption being supported by the structure of adduct **4** in



SCHEME 2

which the distance (3.04 Å) between Te and O is very short as compared with the van der Waals contact distance (3.6 Å). The second is the steric influence of the functional groups attached at the double bond; the steric hindrance between R and the phenyltellurinyl groups of the *E*-form seems to be especially crucial, because the telluroxide of **6** free from such steric hindrance remains in the *E*-form. On the other hand, the latter intramolecular cyclization presumably involves a telluroxide elimination reaction of an intermediate **17**. Since a telluroxide elimination occurs more readily with highly branched alkyl telluroxides [5], this transformation is favorable for telluroxides **15** formed from internal alkynes rather than from terminal alkynes.

In conclusion, the present amidotellurinylation with terminal alkynes provides a stereo- and regio-selective synthetic method of (*Z*)- or (*E*)-β-amidovinyl phenyl tellurides, which might be useful intermediates in modern organic syntheses [6]. In addition, the results with internal alkynes provide a novel synthetic method for oxazoles. The numerous synthetic methods for formation of oxazole derivatives have been hitherto developed in connection with their pharmaceutical properties. The present method is advantageous for its one-pot access from alkynes [7].

EXPERIMENTAL SECTION

General

All chemicals and solvents were of reagent grade. Melting points were uncorrected. Boiling points were measured by use of a Kugelrohr apparatus. All the reactions were carried out under a nitrogen atmosphere. NMR spectra were recorded on a JEOL EX-270 spectrometer (^{1}H NMR at 270 MHz and ^{13}C NMR at 67.8 MHz) in deuteriochloroform using tetramethylsilane as an internal standard. IR spectra were taken on a Hitachi 260-30 spectrometer with a KBr disk or by a neat method. MS spectra were measured at 70 eV on a Shimadzu QP-1000A spectrometer or on a Hitachi M-80B spectrometer using a direct insertion technique. A general procedure for amidotellurinylation of alkynes is described in the following conversion of phenylacetylene to (*Z*)-β-acetamidostyryl phenyl telluride (**4**). Separation of the product mixtures in the reactions of 1-octyne and 4-octyne was carried out by column chromatography on silica gel using 1:9 ethyl acetate-chloroform as eluent, in which the oxazole, the *Z*-adduct, and the *E*-adduct were very closely eluted in that order.

(Z)-β-Acetamidostyryl Phenyl Telluride (**4**)

Benzenetellurinyl trifluoromethanesulfonate (1.2 mmol) was generated in situ by treatment of benzenetellurinic anhydride (274 mg, 0.6 mmol) with trifluoromethanesulfonic acid (270 mg, 1.8 mmol) in acetonitrile (5 mL) for a few minutes at room temperature. To this solution was added phenylacetylene (105 mg, 1.0 mmol), and the mixture was refluxed for 2 hours and then reduced with hydrazine hydrate (5 mL) in methanol (20 mL) at 60°C for 10 minutes. After evaporation of the solvent under reduced pressure, the residue was taken up with chloroform (50 mL), washed with brine (30 mL × 3), and dried over anhydrous magnesium sulfate. The chloroform solution was chromatographed on silica gel using 1:9 ethyl acetate-chloroform as eluent to give **4** (159 mg, 43% yield). Yellow columns from benzene; dp 148–149°C. IR (KBr) 3200–3300 (NH), 1640 (CONH) cm^{-1}. ^{1}H NMR δ 2.16 (s, 3H, CH_3), 6.80 (br s, 1H, NH), 7.09 (s, 1H, olefinic CH), 7.2–7.4 (m, 8H, ArH), 7.76 (d, J = 7.3 Hz, 2H, ArH). ^{13}C NMR δ 23.6 (CH_3), 99.2 (olefinic CTe), 116.0 (aromatic CTe), 126–138 (aromatic and olefinic CH), 141.5 (aromatic C), 168.5 (C=O). MS m/z 367 (M^+, ^{130}Te), 207, 160, 77. Anal. calcd for $C_{16}H_{15}NOTe$: C, 52.67; H, 4.14; N, 3.84; found: C, 52.55; H, 4.10; N, 3.81%.

(Z)-2-Acetamido-1-octenyl Phenyl Telluride (**5**)

Yellow oil. ^{1}H NMR δ 0.88 (t, J = 6.4 Hz, 3H, CH_3), 1.2–1.35 (m, 6H, CH_2), 1.46 (m, 2H, CH_2), 2.01 (s, 3H, CH_3), 2.65 (t, J = 7.4 Hz, CH_2), 6.16 (s, 1H, olefinic CH), 7.1–7.3 (m, 4H, ArH and NH), 7.60 (dd, J = 5.8, 1.8 Hz, ArH). ^{13}C NMR δ 14.1 (CH_3), 22.6 (CH_2), 24.0 (CH_3), 28.2, 28.7, 31.6, 36.2 (CH_2), 85.8 (olefinic CTe), 115.4 (aromatic CTe), 127.5, 129.5, 136.1 (aromatic CH), 147.4 (olefinic CNH), 168.0

(C=O). MS *m/z* 375 (M^+, ^{130}Te), 207, 168, 126, 77. HRMS calcd for $C_{16}H_{23}NOTe$: *m/z* 373.0826 (Te = 127.9047); found: *m/z* 373.0777.

(E)-1-(Acetamidomethylene)heptyl Phenyl Telluride (**6**)

Yellow oil. IR (KBr) 3100–3500 (NH), 1670 (CONH) cm^{-1}. ^1H NMR δ 0.85 (t, *J* = 6.9 Hz, 3H, CH_3), 1.15–1.3 (m, 6H, CH_2), 1.45 (m, 2H, CH_2), 2.08 (s, 3H, CH_3), 2.32 (t, *J* = 7.2 Hz, CH_2), 7–8 (br, 1H, NH), 7.15–7.3 (m, 3H, ArH), 7.47 (s, 1H, olefinic CH), 7.65 (d, *J* = 6.3 Hz, 2H, ArH). ^{13}C NMR δ 14.1, 23.4 (CH_3), 22.6, 28.7, 29.1, 31.7, 34.8 (CH_2), 99.5 (olefinic CTe), 113.9 (aromatic CTe), 127.5, 129.2, 137.2 (aromatic CH), 133.4 (olefinic CNH), 166.9 (C=O). MS *m/z* 375 (M^+, ^{130}Te), 207, 168, 126, 77. HRMS calcd for $C_{16}H_{23}NOTe$: *m/z* 373.0826 (Te = 127.9047); found: *m/z* 373.0865. Compound **6** contained a trace of (*Z*)-isomer, which was detected by the following spectral peaks: ^1H NMR δ 0.85 (t, *J* = 6.6 Hz, 3H, CH_3), 1.15–1.3 (m, 6H, CH_2), 1.49 (m, 2H, CH_2), 1.98 (s, 3H, CH_3), 2.44 (t, *J* = 7.4 Hz, 2H, CH_2), 6.95 (d, *J* = 11.2 Hz, 1H, CH), 7.15–7.3 (m, 3H, ArH), 7.54 (d, *J* = 6.3 Hz, 2H, ArH), 7.62 (br d, *J* = 11.2 Hz, 1H, NH). ^{13}C NMR δ 14.0, 23.3 (CH_3), 22.5, 28.3, 30.2, 31.6, 40.3 (CH_2), 102.0 (olefinic CTe), 112.8 (aromatic CTe), 127.6, 129.7, 135.9, 136.0 (aromatic and olefinic CH), 167.2 (C=O).

(E)-2-Acetamido-1-propyl-1-pentenyl Phenyl Telluride (**7**)

Yellow solid; mp 72–74°C. IR (KBr) 3200 (NH), 1650, 1620 (CONH) cm^{-1}. ^1H NMR δ 0.88 (t, *J* = 7.3 Hz, 3H, CH_3), 0.76 (t, *J* = 7.3 Hz, 3H, CH_3), 1.44 (sex, *J* = 7.4 Hz, 4H, CH_2), 2.03 (s, 3H, CH_3), 2.26 (t, *J* = 7.4 Hz, 2H, CH_2), 2.68 (t, *J* = 7.6 Hz, 2H, CH_2), 7–8 (br, 1H, NH), 7.1–7.3 (m, 3H, ArH), 7.69 (d, *J* = 6.9 Hz, 2H, ArH). ^{13}C NMR δ 13.5, 13.8, 23.6 (CH_3), 21.5, 22.8, 38.1, 39.9 (CH_2), 114.0, 116.1 (olefinic and aromatic CTe), 127.7, 129.3, 138.1 (aromatic CH), 139.9 (olefinic CNH), 168.2 (C=O). MS *m/z* 374 (M^+ − 1), 207, 169, 126. Anal. calcd for $C_{16}H_{23}NOTe$: C, 51.53; H, 6.22; N, 3.76; found: C, 51.50; H, 6.03; N, 3.70%.

(Z)-2-Acetamido-1-propyl-1-pentenyl Phenyl Telluride (**7**)

Yellow solid; mp 67–68°C. IR (neat) 3300 (NH), 1650 (CONH) cm^{-1}; ^1H NMR δ 0.83 (t, *J* = 6.9 Hz, 3H, CH_3), 0.92 (t, *J* = 7.3 Hz, 3H, CH_3), 1.48 (sex, *J* = 6.9 Hz, 4H, CH_2), 1.89 (s, 3H, CH_3), 2.32 (t, *J* = 6.6 Hz, 2H, CH_2), 2.66 (t, *J* = 6.9 Hz, CH_2), 7–8 (br, 1H, NH), 7.15–7.3 (m, 3H, ArH), 7.63 (d, *J* = 6.9 Hz, 2H, CH_2), ^{13}C NMR δ 13.3, 13.6, 23.4 (CH_3), 21.5, 23.3, 31.1, 37.6 (CH_2), 112.7, 113.9 (olefinic and aromatic CTe), 127.5, 129.1, 137.4 (aromatic CH), 140.7 (olefinic CNH), 168.4 (C=O). MS *m/z* 374 (M^+ − 1), 207, 169, 126. Anal. calcd for $C_{16}H_{23}NOTe$: C, 51.53; H, 6.22; 3.76; found: C, 51.51; H, 6.21; N, 3.76%.

5-Hexyl-2-methyloxazole (**8**)

Colorless oil. ^1H NMR δ 0.89 (t, *J* = 7.0 Hz, 3H, CH_3), 1.24–1.37 (m, 6H, CH_2), 1.60 (m, quin, *J* = 7.0 Hz, CH_2), 2.42 (s, 3H, CH_3), 2.47 (t, *J* = 7.0 Hz, CH_2), 7.22 (s, 1H, ArH). HRMS calcd for $C_{10}H_{17}NO$: *m/z* 167.1310; found *m/z* 167.1329.

2,5-Dimethyl-4-phenyloxazole (**9**)

Colorless oil; bp 120°C/50 Torr (lit., bp 134°C/11 Torr [8]). ^1H NMR δ 2.44 (s, 3H, CH_3), 2.47 (s, 3H, CH_3), 7.27 (t, *J* = 7.3 Hz, 1H, ArH), 7.39 (t, *J* = 7.9 Hz, 2H, ArH), 7.60 (d, *J* = 7.3 Hz, 2H, ArH). ^{13}C NMR δ 11.7, 13.8 (CH_3), 126.6, 127.0, 128.5, 143.3 (phenylic C), 132.5, 134.3, 159.1 (oxazolic C). MS *m/z* 173 (M^+), 158, 130, 104. Anal. calcd for 1:1 picrate (dp 121–122°C): C, 50.76; H, 3.51; N, 13.93; found: C, 50.62; H, 3.47; N, 13.87%.

5-Ethyl-2-methyl-4-phenyloxazole (**10**)

Colorless oil; bp 115°C/6 Torr (lit., bp 134.5°C/11 Torr [8]). ^1H NMR δ 1.28 (t, *J* = 7.3 Hz, 3H, CH_3), 2.45 (s, 3H, CH_3), 2.85 (q, *J* = 7.3 Hz, 2H, CH_2), 7.2–7.5 (m, 5H, ArH). MS *m/z* 187 (M^+), 172, 144, 130, 117, 103, 89, 77. Anal. calcd for 1:1 picrate salt (dp 118–120°C): C, 51.93; H, 3.87; N, 13.46; found: C, 51.90; H, 3.85; N, 13.41%.

4,5-Diphenyl-2-methyloxazole (**11**)

Colorless oil; bp 155°C/10 Torr (lit., bp 214°C/17 Torr [9]). ^1H NMR δ 2.48 (s, 3H, CH_3), 7.2–7.3 (m, 6H, ArH), 7.55 (dd, *J* = 8.2, 1.6 Hz, 2H, ArH), 7.64 (dd, *J* = 8.2, 1.6 Hz, 2H, ArH). ^{13}C NMR δ 13.9 (CH_3), 126.4, 127.8, 127.9, 128.3, 128.5, 128.6, 129.0, 132.5, 135.1, 145.3, 160.1 (aromatic C). MS *m/z* 235 (M^+), 165, 104, 77. Anal. calcd for $C_{16}H_{13}NO$: C, 81.68; H, 5.57; N, 5.59; found: C, 81.57; H, 5.52; N, 6.00%.

4,5-Dipropyl-2-methyloxazole (**12**)

Yellow oil; bp 85°C/50 Torr (lit., bp 85–86°C/14 Torr [10]). ^1H NMR δ 0.90 (m, 3H, CH_3), 0.95 (m, 3H, CH_3), 1.55–1.7 (m, 4H, CH_2), 2.35 (t, *J* = 7.26 Hz, 2H, CH_2), 2.37 (s, 3H, CH_3), 2.51 (t, *J* = 7.4 Hz, 2H, CH_2). ^{13}C NMR δ 13.6, 13.8, 13.9 (CH_3), 21.8, 22.4, 26.5, 27.7 (CH_2), 134.2, 146.8, 159.0 (oxazolic C). MS *m/s* 167 (M^+), 152, 138, 91. Anal. calcd for 1:1 picrate (mp 84°C): C, 48.49; H, 5.09; N, 14.14; found: C, 48.46; H, 5.07; N, 14.08%.

Crystal data of **4**: $C_{16}H_{15}NOTe$, *M* = 364.90, monoclinic, space group $P2_1/c$, *a* = 16.591(1), *b* = 10.149(1), *c* = 8.726(1) Å, β = 91.42(1)°, *V* = 1468.9(3)

Å^3, $Z = 4$, $Dx = 1.651$ g cm^{-3}, graphite-monochromated Cu-K_α radiation, crystal dimensions 0.60 × 0.05 × 0.04 mm, μ(Cu-K_α) = 154.8 cm^{-1}. Rigaku AFC-6C diffractometer, 2061 unique reflections having $|F_0| \geq 1.0\sigma(F_0)$. The intensity of the three standard reflections, which were measured every 100 reflections, showed evidence of decay which finally reached 24% and was used for intensity corrections of the collected data. The structure was solved by the Monte-Carlo direct method using the MULTAN78 program system for the selection of the initial set of phase and refined by the full-matrix least-squares program without absorption correction. Anisotropic temperature factors were used for the refinement, and hydrogen atoms were not included in the refinement (i.e., number of parameters = 1729), $R = 0.068$, $R_w = 0.063$. Atomic coordinates, bond lengths and angles, and thermal parameters were deposited at the Cambridge Crystallographic Data Center.

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