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One-Pot Synthesis of (2E)- and (2E, 4E)-Unsaturated Carboxylic Acid Amides Via Organotellurium Reagents

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ABSTRACT

Di-n-butyl telluride, 2-bromoacetylisobutylamide (or 2-bromoacetylpiperidide) react directly with saturated or α,β-unsaturated aldehydes at room temperature in the presence of potassium carbonate(s) to afford (2E)-, or (2E, 4E)-unsaturated carboxylic acid amides, respectively, in excellent yields with high E stereoselectivity.

(2E)- and (2E, 4E)-Unsaturated amides constitute an important class of compounds that show both physiological activity and insecticidal properties. Such amides are unstable and difficult to access from natural products since they occur only in small amounts in plants [1]. A number of synthetic methods have appeared in the literature [2]. Nevertheless, all of them involve multiple steps to approach the title compounds. Recently, communications dealing with a facile and highly stereoselective synthesis of unsaturated amides via arsonium salts in the presence of potassium carbonate at room temperature have appeared from our laboratory [3]. In our continuing studies of the application of elementoorganic compounds of fifth and sixth group elements in organic synthesis, we wish to report an alternative method using available dibutyl telluride for the synthesis of unsaturated amides. Osuka [4] reported that moderately stabilized and stabilized telluronium ylides, generated from the corresponding telluronium salts by strong base, react with carbonyl compounds to give epoxides and alkenes, respectively. Olefination was also accomplished by treating the aromatic aldehydes with bromoacetonitrile or bromoacetophenone derivatives and dibutyl telluride with refuxing in THF [5]. We found that a variety of aldehydes react directly with dibutyl telluride and 2-bromoacetylisobutylamide (or 2-bromoacetylpiperidide) in the presence of potassium carbonate(s) at room temperature to afford (2E)- and (2E, 4E)-unsaturated amides in excellent yields with high E stereoselectivity.

$$\underset{\mathbf{1}}{Bu_2Te} + \underset{\mathbf{2}}{BrCH_2CONR^1R^2} + \underset{\mathbf{3}}{R^3CHO}$$

$R^1 = H, R^2 = i\text{-}Bu$
$R^1R^2 = (CH_2)_5\text{-}$

$$\xrightarrow[CH_3CH\ (trace\ H_2O)]{K_2CO_3(s),\ r.t.} \underset{\mathbf{4}}{R^3CH{=}CHCONR^1R^2}$$

The results are summarized in Table 1.

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TABLE 1 Unsaturated Amides 4 (A and B) from Aldehydes 3

Compounds 4	Aldehyde 3 R^3	Bromoacetylamide 2 R^1, R^2	Reaction Time (h)	Isolated Yield (%) ($E > 98\%$)
a	C_6H_5-	i-Bu	20	98
b	p-ClC_6H_4-	i-Bu	16	98
c	p-FC_6H_4-	i-Bu	24	95
d	p-$NO_2C_6H_4$-	i-Bu	5	98
e	m-$NO_2C_6H_4$-	i-Bu	5	97
f	p-$CH_3OC_6H_4$-	i-Bu	24	55
g		i-Bu	24	52
h		i-Bu	21	53
i		i-Bu	22	75
j	$C_6H_5CH{=}CH$-	i-Bu	24	91
k	$CH_3(CH_2)_8$-	i-Bu	22	63
l	p-ClC_6H_4-	$(CH_2)_5$-	7	98
m	p-FC_6H_4-	$(CH_2)_5$-	24	77
n	p-BrC_6H_4	$(CH_2)_5$-	22	76
o	p-$NO_2C_6H_4$-	$(CH_2)_5$-	5	98
p	m-$NO_2C_6H_4$-	$(CH_2)_5$-	6	98
q		$(CH_2)_5$-	24	98
r		$(CH_2)_5$-	24	78
s	$C_6H_5(CH{=}CH)$-	$(CH_2)_5$-	24	98
t	$CH_3CH{=}CH$-	$(CH_2)_5$-	22	90

This one-pot reaction is carried out at room temperature using solid potassium carbonate as a base under almost neutral conditions. The excellent yields, the high stereoselectivity, and the availability of dibutyl telluride make our method a facile one to synthesize analogues of biologically active unsaturated amides for screening.

EXPERIMENTAL

Proton nuclear magnetic resonace (^{1}H NMR) spectra were determined with a Varian EM-360L (60 MHz) or an XL-200 (200 MHz) spectrometer using tetramethylsilane as an internal standard. Infrared (IR) spectra were recorded on an IR-440 instrument. Mass spectral data were obtained with electron ionization (EI) on a Finnigan 4021 spectrometer.

All reactions were carried out under nitrogen, and melting points are uncorrected. Di-*n*-butyl telluride [6], 2-bromoacetylisobutylamide [7], and 2-bromoacetylpiperidide [8], were prepared according to literature methods.

General Procedure

Dibutyl telluride (3 mmol), 2-bromoacetylisobutylamide or 2-bromoacetylpiperidide (3 mmol), the aldehyde (2 mmol), K_2CO_3 (3 mmol), and CH_3CN/H_2O (14 ml/90 μL) were mixed under N_2 and stirred at room temperature for several hours. After the reaction had been completed (monitored by thin-layer chromatography [TLC]), the reaction mixture was passed through a short column of silica gel to remove most of dibutyltelluroxide and inorganic salts. The desired product was obtained by flash column chromatography.

N-*Isobutyl 3-phenyl-(2E)-propenoic acid amide* (**4a**)

Using the general procedure, **4a** was obtained from benzaldehyde and 2-bromoacetylisobutylamide, m.p. 110–112°C (Lit. [4] m.p. 107–108°C); results by IR (KCl), 3250, 1655, 1615, 990 cm^{-1}; by MS, *m/z* (relative intensity), 203 (M^+, 19), 146(21), 131(100), 103(26), 77(13); by ^{1}H NMR (200 MHz, $CDCl_3$-TMS,

δ) 7.59 (*d*, *J* = 16 Hz, 1H), 7.34 (*m*, 5H), 6.36 (*d*, *J* = 16 Hz, 1H), 5.81 (*br*, 1H), 3.17 (t, 2H), 1.76 (*m*, 1H), 0.93 (*d*, *J* = 6 Hz, 6H).

N-*Isobutyl 3-*(p-*chlorophenyl*)-(*2E*)-*propenoic acid amide* (**4b**)

4b was prepared from *p*-chlorobenzaldehyde and 2-bromoacetylisobutylamide, m.p. 133–134°C (Lit. [4] m.p. 123°C); results by IR (KCl), 3290, 1650, 1615, 980 cm^{-1}; by MS, *m/z* (relative intensity), 238 (M^+ + 1, 20), 180(24), 165(100), 137(16), 102(15); by ^1H NMR (200 MHz, $CDCl_3$-TMS, δ), 7.53 (*d*, *J* = 16 Hz, 1H), 7.31 (*m*, 4H), 6.30 (*d*, *J* = 16 Hz, 1H), 5.69 (*br*, 1H), 3.17 (t, 2H), 1.82 (*m*, 1H), 0.92 (*d*, *J* = 6 Hz, 6H).

N-*Isobutyl 3-*(p-*fluorophenyl*)-(*2E*)-*propenoic acid amide* (**4c**)

4c was prepared from *p*-fluorobenzaldehyde and 2-bromoacetylisobutylamide, m.p. 102–103°C; results by IR (KCl), 3230, 1655, 1615, 985 cm^{-1}; by MS, *m/z* (relative intensity), 221 (M^+, 32), 164(30), 149(100), 121(27), 101(26); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.58 (*d*, *J* = 16 Hz, 1H), 7.31 (*m*, 4H), 6.31 (*d*, *J* = 16 Hz, 1H), 5.75 (*br*, 1H), 3.21 (t, 2H), 1.88 (*m*, 1H), 0.92 (*d*, *J* = 16 Hz, 6H). The analysis calculated for $C_{13}H_{16}FNO$ (221.03), C, 70.56; H, 7.29; N, 6.73; found, C, 70.37; H, 7.09; N, 6.20.

N-*Isobutyl 3-*(p-*nitrophenyl*)-(*2E*)-*propenoic acid amide* (**4d**)

4d was prepared from *p*-nitrobenzaldehyde and 2-bromoacetylisobutylamide, m.p. 150–151°C (Lit. [4] 145–146°C); results by IR. (KCl), 3300, 1660, 1620, 975 cm^{-1}; by MS, *m/z* (relative intensity), 248 (M^+, 38), 233(21), 205(13), 176(100), 127(45); by ^1H NMR (200 MHz, $CDCl_3$-TMS, δ) 8.20 (d, J = 9 Hz, 2H), 7.65 (*d*, *J* = 16 Hz, 1H), 7.61 (*d*, *J* = 9 Hz, 2H), 6.58 (*d*, *J* = 16 Hz, 1H), 6.06 (*m*, 1H), 3.22 (t, 2H), 1.85 (*m*, 1H) 0.95 (*d*, *J* = 6 Hz, 6H).

N-*Isobutyl 3-*(m-*nitrophenyl*)-(*2E*)-*propenoic acid amide* (**4e**)

4e was prepared from *m*-nitrobenzaldehyde and 2-bromoacetylisobutylamide, m.p. 114–115°C; results by IR (KCl), 3300, 1660, 1620, 980 cm^{-1}; by MS, *m/z* (relative intensity), 248 (M^+, 32), 233(12), 205(7), 175(100), 102(18); by ^1H NMR (60 MHz, $CDCl_3$–TMS, δ), 7.75 (*m*, 5H), 6.58 (*d*, *J* = 16 Hz, 1H), 6.10 (*m*, 1H), 3.25 (t, 2H), 1.85 (*m*, 1H), 0.95 (*d*. *J* = 6 Hz, 6H). The analysis calculated for $C_{13}H_{16}N_2O_3$ (248.28), C, 62.88; H, 6.50; N, 11.29; found, C, 62.42; H, 6.32; N, 11.08.

N-*Isobutyl 3-*(p-*methoxyphenyl*)-(*2E*)-*propenoic acid amide* (**4f**)

4f was prepared from *p*-methoxybenzaldehyde and 2-bromoacetylisobutylamide. m.p. 109–110°C; result by IR. (KCl), 3300, 1650, 1600, 830 cm^{-1}; by MS, *m/z* (relative intensity), 233 (M^+, 42), 176(65), 161(100), 133(7), 121(10); by ^1H NMR (200 MHz, $CDCl_3$-TMS, δ) 7.61 (*d*, *J* = 16 Hz, 1H), 7.41 (*d*, *J* = 9 Hz, 2H), 6.81 (*d*, *J* = 9 Hz, 2H), 6.40 (*m*, 1H), 6.38 (*d*, *J* = 16 Hz, 1H), 3.79 (*s*, 3H), 3.19 (t, 2H), 1.85 (*m*, 1H), 0.95 (*d*, *J* = 7 Hz, 6H). The analysis calculated for $C_{14}H_{19}NO_2$ (233.30), C, 72.07; H, 8.21; N, 6.01; found, C, 72.17; H, 8.64; N, 6.24.

N-*Isobutyl 3-*(*3,4-methylenedioxyphenyl*)-(*2E*)-*propenoic acid amide* (**4g**)

4g was prepared from 3,4-methylenedioxybenzaldehyde and 2-bromoacetylisobutylamide. m.p. 102–103°C (Lit. [4] 106–107°C); result by IR. (KCl), 3250, 1650, 1600, 980 cm^{-1}; by MS, *m/z* (relative intensity), 247 (M^+, 50), 190(57), 174(100), 144(32), 89(18); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ) 7.43 (*d*, *J* = 16 Hz, 1H), 6.85 (*m*, 4H), 5.86 (*s*, 2H), 5.70 (*m*, 1H), 3.15 (t, 2H), 1.82 (*m*, 1H), 0.95 (*d*, *J* = 6 Hz, 6H).

N-*Isobutyl 3-cyclohexyl-*(*2E*)-*propenoic acid amide* (**4h**)

4h was prepared from cyclohexanecarboxaldehyde and 2-bromoacetylisobutylamide. m.p. 109–110°C (Lit. [4] m.p. 107–108°C); result by IR. (KCl), 3300, 1640, 1580, 990 cm^{-1}; by MS, *m/z* (relative intensity) 209 (M^+, 50), 154(18), 137(100), 126(29), 55(49); by ^1H NMR (60 MHz, $CDCL_3$-TMS, δ) 6.78 (*m*, 1H), 5.67 (*d*, *J* = 16 Hz, 1H), 5.50 (*m*, 1H), 3.12 (t, 2H), 1.47 (*m*, 11H), 0.90 (*d*, *J* = 6 Hz, 6H).

N-*Isobutyl 3-*(*2-naphthyl*)-(*2E*)-*propenoic acid amide* (**4i**)

4i was prepared from 2-naphthaldehyde and 2-bromoacetylisobutylamide. m.p. 143–144°C; result by IR. (KCl), 3200, 1650, 1610, 990 cm^{-1}; by MS, *m/z* (relative intensity), 253 (M^+, 46), 196(65), 181(100), 152(44), 127(8); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.65 (*m*, 8H), 6.48 (*d*, *J* = 16 Hz, 1H), 5.75 (*m*, 1H), 3.22 (t, 2H), 1.78 (*m*, 1H) 0.92 (*d*, *J* = 6 Hz, 6H). The analysis calculated for $C_{17}H_{19}NO$ (253.33), C, 80.59; H, 7.56; N, 5.52; found, C, 80.35; H, 7.59; N, 5.38.

N-*Isobutyl 5-phenyl-*(*2E, 4E*)-*pentadienoic acid amide* (**4j**)

4j was prepared from cinnamaldehyde and 2-bromoacetylisobutylamide. m.p. 154–155°C (Lit.

[4] 155–156°C); result by IR. (KCl), 3300, 1640, 1610, 990 cm^{-1}; by MS, *m/z* (relative intensity), 229 (M^+, 50), 172(16), 157(100), 128(67), 96(51); by ^1H NMR (200 HMz, C_6D_6, δ) 7.72 (*dd*, *J*2, 3 = 14.9 Hz, *J*3, 4 = 10.8 Hz, 1H), 7.15 (*m*, 5H), 6.78 (*dd*, *J*4, 5 = 15.4 Hz, *J*3, 4 = 10.8 Hz, 1H), 6.55 (*d*, *J*4,5 = 15.4 Hz, 1H), 5.69 (*d*, *J*2, 3 = 14.9 Hz, 1H), 5.22 (*br*, 1H), 3.14 (*m*, 2H), 1.70 (*m*, 1H), 0.82 (*d*, *J* = 6.7 Hz, 6H).

N-*Isobutyl-2E-dodecenoic acid amide* (**4k**)

4k was prepared from decylaldehyde and 2-bromoacetylisobutylamide. m.p. 58–60°C; result by IR (KCl), 3300, 1660, 1620, 980 cm^{-1}; by MS, *m/z* (relative intensity), 254 (M^+ + 1, 100), 253 (M^+, 8), 238(16), 181(30), 126(12); by ^1H NMR (200 MHz, $CDCl_3$-TMS, δ) 6.85 (*dt*, *J* = 15.5, 7.5 Hz, 1H), 5.78 (*d*, *J* = 15.5 Hz, 1H), 5.56 (*br*, 1H, NH), 3.16 (t, 2H), 2.19 (*m*, 2H), 1.76 (*m*, 2H), 1.82 (*m*, 1H), 1.26 (*m*, 12H), 0.92 (*d*, *J* = 6.7 Hz, 6H), 0.88 (t, 3H). The analysis calculated for $C_{16}H_{31}NO$ (253.42), C, 75.83; H, 12.33; N, 5.53; found, C, 75.71; H, 12.56; N, 5.42.

3-(p-*Chlorophenyl*)-(2*E*)-*propenoic acid piperidide* (**4l**)

4l was obtained from *p*-chlorobenzaldehyde and 2-bromoacetylpiperidide using the general procedure. It has m.p. 135–136°C; result by IR (KCl), 2920, 1640, 1590, 990 cm^{-1}; by MS, *m/z* (relative intensity), 250 (M^+ + 1, 43), 165(59), 138(50), 102(55), 84(100); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.45 (*m*, 5H), 6.83 (*d*, *J* = 16 Hz, 1H), 3.55 (*br*, 4H), 1.60 (*m*, 6H). The analysis calculated for $C_{14}H_{16}ClNO$ (249.73), C, 67.33; H, 6.46; Cl, 14.20; N, 5.61; found, C, 66.96; H, 6.29; Cl, 13.85; N, 5.45.

3-(p-*Fluorophenyl*)-(2*E*)-*propenoic acid piperidide* (**4m**)

4m was prepared from *p*-fluorobenzaldehyde and 2-bromoacetylpiperidide, m.p. 138–139°C; result by IR (KC1), 2900, 1640, 1580, 1500, 985 cm^{-1}; by MS, *m/z* (relative intensity), 234 (M^+ + 1, 66), 149(100), 138(45), 101(70), 84(95); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.57 (*m*, 5H), 6.80 (*d*, *J* = 16 Hz, 1H), 3.58 (*br*, 4H), 1.62 (*m*, 6H). The analysis calculated for $C_{14}H_{16}FNO$ (233.27), C, 72.08; H, 6.91; N, 6.00; found, C, 71.98; H, 6.82; N, 5.95.

3-(p-*Bromophenyl*)-(2*E*)-*propenoic acid piperidide* (**4n**)

4n was prepared from *p*-bromobenzaldehyde and 2-bromoacetylpiperidide, m.p. 134–135°C; result by IR (KCl), 2900, 1640, 1600, 980 cm^{-1}; by MS, *m/z* (relative intensity), 295 (M^+ + 1, 44), 209(51), 138(54), 102(59), 84(100); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.51 (*m*, 5H), 6.80 (*d*, *J* = 16 Hz, 1H), 3.55 (*br*, 4H), 1.62 (*m*, 6H). The analysis calculated for $C_{14}H_{16}BrNO$ (294.18), C, 57.16; H, 5.48; N, 4.76; Br, 27.16; found, C, 57.46; H, 5.52; N, 4.62; Br, 27.29.

3-(p-*Nitrophenyl*)-(2*E*)-*propenoic acid piperidide* (**4o**)

4o was prepared from *p*-nitrobenzaldehyde and 2-bromoacetylpiperidide, m.p. 174–175°C; result by IR (KCl), 2950, 1650, 1600, 1500, 980 cm^{-1}; by MS, *m/z* (relative intensity), 260 (M^+, 36), 176(37), 138(60), 102(59), 84(100); by ^1H NMR (60 MHz, $CDCl_3$-TMS δ), 8.25 (*d*, 2H), 7.80 (*d*, 2H), 7.72 (*d*, *J* = 15.5 Hz, 1H), 7.04 (*d*, *J* = 15.5 Hz, 1H), 3.60 (*br*, 4H), 1.80 (*m*, 6H). The analysis calculated for $C_{14}H_{16}N_2O_3$ (260.28), C, 64.60; H, 6.20; N, 10.76; found, C, 64.72; H, 6.27; N, 10.53.

3-(m-*Nitrophenyl*)-(2*E*)-*propenoic acid piperidide* (**4p**)

4p was prepared from *m*-nitrobenzaldehyde and 2-bromoacetylpiperidide, m.p. 125–126°C; result by IR (KCl), 2900, 1640, 1600, 1520, 985 cm^{-1}; by MS, *m/z* (relative intensity), 261 (M^+ + 1, 29), 176(11), 138(27), 102(33), 84(100); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ) 7.71 (*m*, 5H), 7.00 (*d*, *J* = 16 Hz, 1H), 3.63 (*br*, 4H) 1.68 (*m*, 6H). The analysis calculated for $C_{14}H_{16}N_2O_3$ (260.28), C, 64.60; H, 6.20, N, 10.76; found, C, 64.60; H, 6.14; N, 10.65.

3-(3,4-*Methylenedioxyphenyl*)-(2*E*)-*propenoic acid piperidide* (**4q**)

4q was prepared from 3,4-methylenedioxybenzaldehyde and 2-bromoacetylpiperidide, m.p. 90–91°C; result by IR (KCl), 2900, 1640, 1600, 1490, 980 cm^{-1}; by MS, *m/z* (relative intensity), 259 (M^+, 46), 175(86), 145(63), 138(14), 84(100); by ^1H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.52 (*d*, *J* = 16 Hz, 1H), 6.85 (*m*, 4H), 5.95 (*s*, 2H), 3.55 (*br*, 4H), 1.62 (*m*, 6H). The analysis calculated for $C_{15}H_{17}NO_3$ (259.29), C, 69.48; H, 6.61; N, 5.40; found, C, 69.30; H, 6.54; N, 5.32.

3-(2-*naphthyl*)-(2*E*)-*propenoic acid piperidide* (**4r**)

4r was prepared from 2-naphthyldehyde and 2-bromoacetylpiperidide, m.p. 139–140°C; result by IR (KCl), 2900, 1640, 1590, 980 cm^{-1}; by MS, *m/z* (relative intensity), 266 (M^+ + 1, 53), 182(100),

152(36), 138(13.2), 85(44); by ^{1}H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.58 (*m*, 8H), 7.00 (*d*, J = 16 Hz, 1H), 3.62 (*br*, 4H), 1.60 (*m*, 6H). The analysis calculated for $C_{18}H_{19}NO$ (265.34), C, 81.47; H, 7.22; N, 5.28; found, C, 81.47; H, 7.22; N, 5.05.

5-Phenyl-(2E, 4E)-pentadienoic acid piperdide (***4s***)

4s was prepared from cinnamaldehyde and 2-bromoacetylpiperide, m.p. 200–201°C (Lit. [9] 203°C); result by IR (KCl), 2900, 1640, 1600, 1000 cm^{-1}; by MS, *m/z* (relative intensity), 241 (M^+, 97), 157(97), 137(50), 128(86), 84(100); by ^{1}H NMR (60 MHz, CCl_4-TMS, δ), 7.35 (m, 8H), 6.33 (*d*, J = 14 Hz, 1H), 3.49 (*m*, 4H), 1.60 (*m*, 6H).

(2E, 4E)-Hexadienoic acid piperdide (***4t***)

4t was prepared from crotylaldehyde and 2-bromoacetylpiperide, m.p. 77–78°C (Lit. [9] 83–84°C); result by IR (KCl), 2910, 1620, 1600, 1000 cm^{-1}; by MS, *m/z* (relative intensity), 180 (M^+ + 1, 100), 179 (M^+, 35), 164(15), 138(10), 84(39); by ^{1}H NMR (60 MHz, $CDCl_3$-TMS, δ), 7.30 (*m*, 1H), 6.0–6.4 (*m*, 2H), 6.03 (*d*, J = 15 Hz, 1H), 3.55 (*m*, 4H), 1.83 (*d*, J = 8 Hz, 3H), 1.73 (*m*, 6H).

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