Heteroatom Chemistry
Volume 12, Number 4, 2001

Acyl Carbamoyl Selenides and Related Sulfur Isologues: Synthesis and X-Ray Structural Analyses

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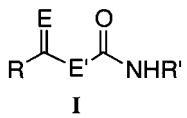
Received 12 December 2000; revised 18 January 2001

ABSTRACT: *Selenocarboxylic acids [RC(=O)SeH] were found to readily react with aryl, acyl, and arenesulfonyl isocyanates to give the corresponding acyl carbamoyl selenides* **3** *[RC(=O)SeC(=O)NHR′, R′ = aryl, C_6H_5CO, and 4-$MeC_6H_4SO_2$] in good yields. Their tautomers [RC(=O)SeC(=NR′)OH] were also detected by ^1H, ^{13}C, and ^{77}Se NMR spectroscopies. The structure of* **3** *[R = 2,6-$(MeO)_2C_6H_3$, R′ = 4-MeC_6H_4] was characterized by X-ray crystallography, which showed that this molecule is stabilized by an intramolecular hydrogen bond between the carbonyl oxygen and the NH hydrogen to form a planar six-membered ring and by nonbonded interaction of the ortho methoxy oxygen with the carbonyl oxygen or the selenium atoms. 4-Methoxybenzoyl and 4-methoxythiobenzoyl N-(4-methylphenyl)carbamoyl sulfides (***4*** *and* ***5****) were shown by X-ray crystallography to similarly have a planar intramolecular six-membered ring formed by a hydrogen bond between the carbonyl oxygen or thiocarbonyl sulfur and NH hydrogen atoms. The tautomers [RC(=E)SC(=NR′)OH; E = O or S] of* **4** *and* **5** *also were detected spectroscopically. The reactions of* **3h** *(R = 4-$MeOC_6H_4$, R′ = Ph) with sodium methoxide and p-toluidine gave sodium selenocarboxylate and the corresponding amides and urea as main products, respectively.* © 2001 John Wiley & Sons, Inc. Heteroatom Chem 12:250–258, 2001

INTRODUCTION

In general, the isolation of carbamic carboxylic mixed acid anhydrides **I** (E = E′ = O) is difficult due to the easy equilibrium between the starting compounds (RCOOH and R′NCO) and product [RC(=O)OC(=O)NHR′] [1,2] (Figure 1).

Previously, we reported the isolation of a series of dithiocarboxylic carbamic mixed acid anhydrides **I** (E = E′ = S) as crystals by reacting dithiocarboxylic acids with aryl isocyanates [3]. Since then, several carbamic thiocarboxylic mixed acid anhydrides **I** (E = O, E′ = S) have been reported by Motoki et al. [4]. However, the synthesis of other carbamic chalcogenocarboxylic mixed acid anhydrides have not been reported in the literature. This is most likely



I

E = E' = O
E = O, E' = S
E = E' = S

FIGURE 1

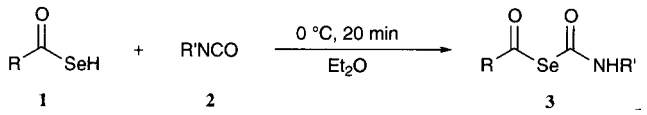
Correspondence to: Shinzi Kato.
Contract Grant Sponsor: Ministry of Education, Science, Sports and Culture of Japan.
Contract Grant Number: Grant-in-Aid for Scientific Research on Priority Areas No. 10133221.
Contract Grant Sponsor: Ministry of Education, Science, Sports and Culture of Japan.
Contract Grant Number: Grants-in-Aid for Scientific Research No. 09355032.

due to the difficulty of synthesizing chalcogenocarboxylic acids themselves [5]. Our successful isolation of selenocarboxylic acids [6] prompted us to synthesize carbamic selenocarboxylic mixed acid anhydrides. We report here the first isolation of carbamic selenocarboxylic mixed acid anhydrides and their structures which were determined by X-ray structural analyses.

RESULTS AND DISCUSSION

When a diethyl ether solution of phenyl isocyanate was added to an equimolar amount of 4-methoxybenzenecarboselenoic acid in the same solvent at room temperature, the orange solution of the selenocarboxylic acid quickly changed to a colorless suspension. Removal of the solvent and recrystallization of the resulting residue from dichloromethane/hexane gave the expected 4-methoxybenzoyl *N*-phenylcarbamoyl selenide **3h** in 95% yield as colorless crystals. Similarly, the reactions of other selenocarboxylic acids with aryl isocyanates gave the corresponding acyl carbamoyl selenides (**3a–g, k–n**) in isolated yields of 35–99% (Scheme 1). In addition, the reactions with benzoyl and *p*-tosyl isocyanates proceeded more quickly to give the corresponding *N*-benzoyl-**3i** and *N*-(*p*-tosyl)carbamoyl selenides **3j**. The structures of the products reported herein were established by IR and ^{1}H and ^{13}C NMR spectra and by elemental and X-ray structural analyses.

The resulting mixed acid anhydrides **3** are colorless crystals or colorless oils and are relatively stable thermally and insensitive toward oxygen. The aliphatic derivatives (**3a–c**) appear to be more labile than the aromatic derivatives (**3d–n**). Acetyl *N*-phenylcarbamoyl selenide **3a** decomposed at room temperature even under an argon atmosphere. Therefore, these aliphatic derivatives were not subjected to elemental analysis.



SCHEME 1

No.	RC(O)SeC(O)NHR′ R	R′	Yield [%]
3a	CH_3	C_6H_5	35
3b	i-C_4H_9	C_6H_5	95
3c	1-Adamantyl	C_6H_5	78
3d	C_6H_5	C_6H_5	99
3e	2-$CH_3C_6H_4$	C_6H_5	93
3f	4-$CH_3C_6H_4$	C_6H_5	93
3g	2-$CH_3OC_6H_4$	C_6H_5	92
3h	4-$CH_3OC_6H_4$	C_6H_5	95
3i		C_6H_5CO	61
3j		4-$CH_3C_6H_4SO_2$	97
3k	2,6-$(CH_3O)_2C_6H_3$	4-$CH_3C_6H_4$	100
3l	3-Cl-2,6-$(CH_3O)_2C_6H_2$	C_6H_5	97
3m	4-$C_6H_5C_6H_4$	C_6H_5	92
3n	1-$C_{10}H_7$	C_6H_5	93

Spectra

Previously, we reported that the NH proton chemical shifts in thioacyl carbamoyl sulfides (**I**, E = E′ = S) appeared at unusually low fields, below δ 12, indicating the presence of an intramolecular C=S···HN hydrogen bond [3]. As shown in Table 1, the chemical shifts of the NH proton appear in the range of δ 9–12, indicative of an intramolecular hydrogen bond between the carbonyl oxygen and the NH hydrogen. The carbonyl and carbamoyl carbon chemical shifts are observed in the ranges of δ 194–216 and δ 156–160, respectively. The ^{77}Se NMR signals appear at δ 630–730. Thus, these spectral data indicate that **3** exists as structure **IIa** (Figure 2).

However, in the ^{1}H NMR spectrum of **3a**, two broad signals at δ 9.15 and δ 9.91 (proton ratio = 1:10) was observed, which are attributable to OH and NH protons, respectively. No appreciable change in the proton ratio was observed in the range 20°C to −60°C. The ^{13}C NMR spectra, except for the signals at δ 156.2 (CONH) and δ 202.7 (COSe) in **IIaa** (Table 2), also show small signals at δ 160.9 and δ 194.6, which are attributable to the C=N and COSe groups, respectively. In addition, in ^{77}Se NMR spectroscopy, a small sharp signal is observed at δ 835. These results apparently indicate the existence of a tautomer (**IIab**) of **IIaa**. In the ^{1}H NMR spectra of other selenides (**3b, d, h–l**), small or negligible signals of the corresponding tautomers **IIb** are observed. Table 2 shows the proton ratios of NH in **IIa** and OH in **IIb**. We also obtained ^{1}H and ^{13}C NMR spectra, which indicate the existence of the tautomers **IIIb** and **IVb** for the previously reported sulfur isologues (**I**, E = O, E′ = S [4] and E, E′ = S [3]): 4-methoxybenzoyl **4** and 4-methoxythiobenzoyl *N*-(4-methylphenyl)carbamoyl sulfides **5** (Figure 3). The proton ratios of **IIIa** and **IIIb** in **4** and **IVa** and **IVb** in **5** were 4:1 and 20:1, respectively.

X-Ray Structure

To confirm the intramolecular hydrogen bond, the X-ray structural analysis of **3** was carried out. To our knowledge, no structural analysis of acyl or thioacyl carbamoyl chalcogenides has been described in the literature. After several attempts to obtain acyl carbamoyl selenides **3** as single crystals, 2,6-dimethox-

TABLE 1 Spectral Data of Acyl Carbamoyl Selenides **3**

	RC(O)SeC(O)NHR′		*NMR ($CDCl_3$) [δ]*			
No.	*R*	*R′*	*^{1}H (NH)*	*^{13}C(O)N*	*^{13}C(O)Se*	*^{77}Se*
3a	CH_3	C_6H_5	9.91	156.2	202.7	687.5
3b	*t*-C_4H_9	C_6H_5	10.05	156.4	215.6	639.1
3c	1-Adamantyl	C_6H_5	10.10	156.8	215.3	—
3d	C_6H_5	C_6H_5	10.41	156.0	199.7	648.4
3e	2-$CH_3C_6H_4$	C_6H_5	10.27	157.0	201.8	678.4
3f	4-$CH_3C_6H_4$	C_6H_5	10.45	156.3	198.9	642.6
3g	2-$CH_3OC_6H_4$	C_6H_5	10.61	159.3	197.4	708.1
3h	4-$CH_3OC_6H_4$	C_6H_5	10.51	156.4	197.1	632.7
3i		C_6H_5CO	10.51	157.4	197.6	—
3j		4-$CH_3C_6H_4SO_2$	11.54	157.3	194.8	—
3k	2,6-$(CH_3O)_2C_6H_3$	4-$CH_3C_6H_4$	10.07	156.5	199.9	—
3l	3-Cl-2,6-$(CH_3O)_2C_6H_2$	C_6H_5	10.05	157.6	199.3	726.1
3m	4-$C_6H_5C_6H_4$	C_6H_5	10.46	156.1	198.9	—
3n	1-$C_{10}H_7$	C_6H_5	10.36	157.1	201.9	—

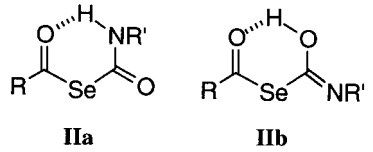


FIGURE 2

TABLE 2 Ratio of Tautomers **IIa** and **IIb**

No.	*R*	*R′*	**IIa**	**IIb**
3a	CH_3	C_6H_5	8 (**IIaa**)	1 (**IIab**)
3b	*t*-C_4H_9	C_6H_5	12 (**IIba**)	1 (**IIbb**)
3d	C_6H_5	C_6H_5	20 (**IIda**)	1 (**IIdb**)
3e	2-$CH_3C_6H_4$	C_6H_5	9 (**IIea**)	1 (**IIeb**)
3f	4-$CH_3C_6H_4$	C_6H_5	30 (**IIfa**)	1 (**IIfb**)
3g	2-$CH_3OC_6H_4$	C_6H_5	50 (**IIga**)	1 (**IIgb**)
3h	4-$CH_3OC_6H_4$	C_6H_5	30 (**IIha**)	1 (**IIhb**)
3i	3-Cl-2,6-$(CH_3O)_2C_6H_2$	C_6H_5	12 (**IIIa**)	1 (**IIIb**)

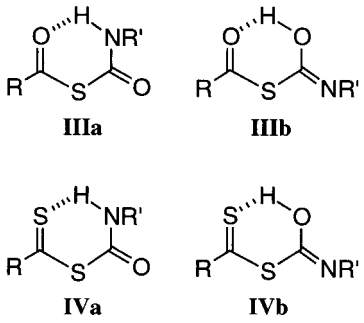


FIGURE 3

ybenzoyl *N*-(4-methylphenyl)carbamoyl selenide **3k** afforded suitable crystals for X-ray analysis. The molecular structure is shown in Figure 4. The final atomic positional parameters are listed in Table 3. Selected bond distances and angles are shown in Table 4. The C1–O1 [1.204(10) Å] and C11–O2 [1.20(1) Å] distances indicate double bonds. The C11–N1 [1.34(1) Å], C12–N1 [1.436(9) Å], C1–Se1 [1.935(10) Å], and C11–Se1 distances [1.965(9) Å] are normal, indicating single bonds. The O1–N1, N1–H1, and O1–H1 distances are 2.76(2) Å, 0.88 Å, and 1.89 Å, respectively, and the O1–H1–N1 bond angle is 173.0°, indicating the presence of a hydrogen bond between the carbonyl oxygen (O1) and the NH hydrogen (H1). Torsion angles [3.2(9)° for N1–C11–Se1–C1, 0.5(9)° for O1–C1–Se1–C11 and 8° for Se1–C11–N1–H1] indicate that the selenocarboxyl group and carbamoyl group are in approximately the same plane to give a planar intramolecular six-membered ring. In addition, the O3–O1 [2.722(7) Å] and O4–Se1 [2.959(7) Å] distances are remarkably short compared with the sum [3.04 Å for O–O; 3.42 Å for O–Se [7]) of the van der Waals radii of both the atoms, respectively, suggesting the presence of nonbonded repulsion and nonbonded attraction.

For comparison with the structures of previously isolated sulfur isologues [RC(=O/S)SC(=O)R′], the X-ray structural analyses of 4-methoxybenzoyl **4** and 4-methoxythiobenzoyl *N*-(4-methylphenyl)carbamoyl sulfides **5** were carried out. Their molecular structures are shown in Figure 4 (**b** and **c**). Selected bond lengths and angles are shown in Table 3. As expected, they have an intramolecular six-mem-

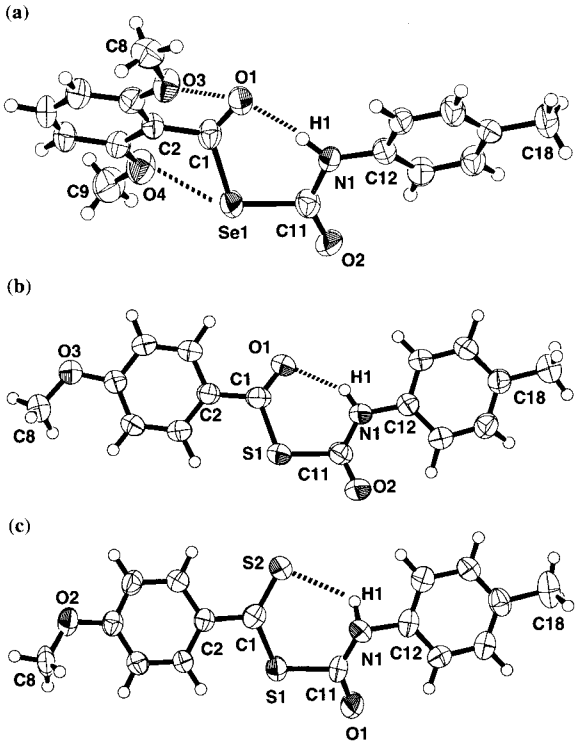


FIGURE 4 The structures of 2,6-dimethoxybenzoyl *N*-(4-methylphenyl)carbamoyl selenide **3k**, 4-methoxybenzoyl *N*-(4-methylphenyl)carbamoyl sulfide **4**, and 4-methoxythiobenzoyl *N*-(4-methylphenyl)carbamoyl sulfide **5**. The atoms are drawn with 50% probability thermal ellipsoids.

bered ring structure formed by a hydrogen bond between the carbonyl oxygen or thiocarbonyl sulfur and the NH hydrogen atoms, respectively. Presumably, this might contribute to the overall stability of the molecules. Attempts to obtain single crystals of the tautomers, **IIb, IIIb**, and **IVb** failed.

Reactions

The reaction of **3h** with sodium methoxide in diethyl ether readily proceeded at room temperature to give sodium 4-methoxybenzenecarboselenoate **6** and methyl *N*-phenylcarbamate **7** in good yields (Scheme 2).

The reaction with two equimolar amounts of *p*-toluidine under similar conditions gave *N*-4-methylphenyl 4-methoxybenzamide **8** and *N*-4-methylphenyl *N′*-phenyl urea **9** in moderate yields (Scheme 2). Compound **8** may be formed by decomposition of 4-methoxyselenocarboxylic acid 4-methylphenylammonium salt [4-$MeOC_6H_4C(=O)Se^-$ $^+NH_2C_6H_4Me$-4]. These results indicate that nucleophiles, such as alkoxides and amines, preferentially attack the carbamoyl carbon rather than the carbonyl carbon in **3**.

EXPERIMENTAL

The melting points were determined by a Yanagimoto micromelting point apparatus and are uncorrected. The IR spectra were measured on a PERKIN ELMER FT-IR 1640 instrument. The ^{1}H NMR spectra were recorded on JEOL JNM-GX-270 (270 MHz) and JEOL α-400 (399.7 MHz) instruments with Me_4Si as an internal standard. The ^{13}C NMR spectra were obtained by use of JEOL JNM-GX-270 (68 MHz) and JEOL α-400 (100.4 MHz) instruments with $CDCl_3$ as an internal standard. The ^{77}Se NMR spectra were obtained by use of a JEOL α-400 (76.2 MHz) instrument with Me_2Se as an external standard. Elemental analyses were performed by the Elemental Center of Kyoto University.

Materials

The following reagents were of commercial grade and used without further purification: phenyl, 4-methylphenyl, and *p*-toluenesulfonyl isocyanates, and *p*-toluidine (from Tokyo Kasei) and hydrogen chloride (1.0 M solution in diethyl ether) (from Aldrich). Benzoyl isocyanate [8], selenocarboxylic acids [5,6], 4-methoxybenzenecarbothioic acid [9], and 4-methoxybenzenecarbodithioic acid [10] were prepared according to the literature. Dichloromethane was distilled from diphosphorus pentaoxide and degassed. Diethyl ether was distilled from sodium diphenylketyl and degassed. Hexane was distilled from sodium metal prior to use and degassed. All of the manipulations were carried out under argon.

*3-Chloro-2,6-dimethoxybenzenecarboselenoic Acid (***1l***)*

Yellow oil; ^{1}H NMR ($CDCl_3$): δ 3.29 (br, 1H, SeH), 3.85 (s, 3H, CH_3O), 3.91 (s, 3H, CH_3O), 6.67 (d, J = 8.1 Hz, 1H), 7.35 (d, J = 8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 56.3 (CH_3O), 62.5 (CH_3O), 108.1, 119.8, 128.4, 132.2, 152.0, 154.4, 189.7 (CO).

*4-Biphenylcarboselenoic Acid (***1m***)*

Red solid; IR (KBr): 2290 (SeH) cm^{-1} [15], dec.: 54–56°C; ^{1}H NMR ($CDCl_3$): δ 4.85 (br, 1H, SeH), 7.38–8.24 (m, 9H); ^{13}C NMR ($CDCl_3$): δ 127.8, 127.9, 128.0, 128.9, 129.1, 129.7, 129.8, 131.1, 203.2 (CO).

X-Ray Structure Analysis

All measurement were carried out on a Rigaku AFC7R diffractometer with graphite monochromated MoKα radiation (λ = 0.71069 Å). All of the structures were solved and refined using the teXsan

TABLE 3 Crystal Data and Experimental Crystallographic Details for Compounds **3k, 4**, and **5**

Compound	**3k**	**4**	**5**
Empirical formula	$C_{17}H_{17}NO_4Se$	$C_{16}H_{15}NO_3S$	$C_{16}H_{15}NO_2S_2$
M	378.29	301.36	317.42
Crystal size	0.34 × 0.13 × 0.13	0.20 × 0.20 × 0.30	0.34 × 0.13 × 0.13
Color/shape	colorless/needle	colorless/needle	orange/needle
Crystal system	orthorhombic	triclinic	monoclinic
Space group	$Pna2_1$	$P\bar{1}$	$P2_1/n$
a/Å	13.976(1)	12.389(3)	4.101(4)
b/Å	8.520(1)	15.489(3)	21.076(3)
c/Å	13.753(2)	4.132(2)	17.731(3)
α/°		91.19(3)	
β/°		90.80(3)	94.65(4)
γ/°		112.89(1)	
V/Å^3	1637.6(3)	730.1(4)	1527(1)
Z	4	2	4
F(000)	768.00	316.00	664.00
D_0/g cm^{-3}	1.534	1.371	1.380
μ(MoKα)/cm^{-1}	23.12	2.31	3.51
Temp (°C)	23 ± 1	23 ± 1	23 ± 1
2θ max (°)	55.0	55.0	55.0
Scan Rate (° min^{-1})	16.0	16.0	16.0
Data collected	$+h, +k, +l$	$+h, \pm k, \pm l$	$+h, +k, \pm l$
Total data collected, unique, observed	2170, 1954, 1170 (I > 2σ(I))	3502, 3344, 1681 (I > 2σ(I))	4112, 3624, 1158 (I > 2σ(I))
No. of variable	209	190	190
Residuals: *R*1: *wR*[a]	0.086, 0.125	0.060, 0.066	0.092, 0.220
Goodness of fit	1.02	1.28	1.09
Final diff. map max, min (e Å^{-3})	−0.60, 0.55	−0.24, 0.23	−0.58, 0.58

[a]$R1 = \Sigma||F_0| - |F_0||/\Sigma|F_0|$ (for $I > 2.0\sigma(I)$ data), $wR = [(\Sigma w(F_0^2 - F_c^2)^2/\Sigma w(F_0^2)^2)]^{1/2}$, where $w = 1/\sigma^2(F_0^2)$.

crystallographic software package. The cell dimensions were determined by a least-squares refinement of the diffractometer angles for 25 automatically centered reflections. Three standard reflections were measured every 150 reflections, and no decay was detected. An empirical absorption correction (Ψ Scan) was applied. The structures were solved by direct methods (SHELXS86) [11] and expanded using DIRDIF94 [12]. Scattering factors for neutral atoms were from Cromer and Waber [13], and anomalous dispersion [14] was used. A full-matrix least-squares refinement was executed with nonhydrogen atoms being anisotropic. The final least-squares cycle included fixed hydrogen atoms at calculated positions of which each isotropic thermal parameter was set to 1.2 times that of the connecting atom. Crystal data and measurement description are summarized in Table 3.

Preparation of Single Crystals

2,6-Dimethoxylbenzoyl *N*-(4-methylphenyl)carbamoyl selenide **3k** was crystallized from diethyl ether/hexane (1:1) at 18°C during a period of four days. 4-Methoxybenzoyl *N*-(4-methylphenyl)carbamoyl sulfide **4** was crystallized from CH_2Cl_2/hexane (1:5) at 23°C for three days. 4-Methoxythiobenzoyl *N*-(4-methylphenyl)carbamoyl sulfide **5** was crystallized from $CHCl_3$/hexane (1:2) at 23°C for two days. These crystal samples were cut from grown crystals, coated with an epoxy resin, and mounted on a glass fiber.

*Synthesis of Acyl Carbamoyl Selenides (**3**)*

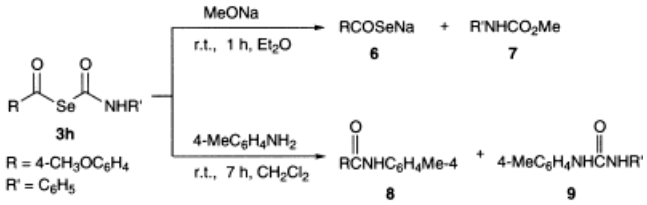
The synthesis of 4-methoxybonzoyl *N*-phenylcarbamoyl selenide **3h** is described in detail as a typical procedure.

To a solution of sodium 4-methoxybenzenecarboselenoate (0.89 g, 3.0 mmol) in diethyl ether (10 mL), contained in a 20 mL two-necked round-bottom flask, 1.0 M hydrogen chloride in diethyl ether (2.6 mL) was added. The mixture was stirred at 0°C for 10 minutes. Filtration of the precipitates (NaCl and excess of sodium 4-methoxybenzenecarboselenoate) and removal of the solvent under reduced pressure (22°C/53.3Pa) gave 0.56 g (2.6 mmol) of 4-methoxybenzene-carboselenoic acid as yellow solid) [5]. The solid was dissolved in diethyl ether (10 mL). A solution of phenylisothiocyanate (0.30 g, 2.6 mmol) in diethyl ether (5.0 mL) was added and

TABLE 4 Selected Bond Lengths (Å) and Bond Angles (°) of **3k, 4**, and **5**

3k			
Bond Lengths			
Se1–C1	1.935(10)	N1–H1	0.88
Se1–C11	1.965(9)	N1···O1	2.76(2)
O1–C1	1.204(10)	O1···H1	1.89
O2–C11	1.20(1)	Se1···O4	2.959(7)
N1–C11	1.34(1)	O1···O3	2.722(7)
N1–C12	1.436(9)		
Angles			
Se1–C1–O1	121.9(6)	O2–C11–N1	127.8(8)
C1–Se1–C11	106.0(4)	C11–N1–C12	124.7(8)
Se1–C11–N1	116.2(6)	N1–H1···O1	173.0
Torsion Angles			
Se1–C1–C2–C7	48(1)	O1–C1–C2–C3	42(1)
N1–C11–Se1–C1	3.2(8)	O2–C11–N1–C12	0(1)
O1–C1–Se1–C11	0.5(9)	Se1–C11–N1–H1	8

4				*5*			
Bond Lengths				Bond Lengths			
S1–C1	1.791(3)	N1–C11	1.334(4)	S1–C1	1.744(6)	N1–C11	1.322(8)
S1–C11	1.817(4)	N1–C12	1.422(4)	S1–C11	1.844(6)	N1–C12	1.448(8)
O1–C1	1.218(4)	N1–H1	0.85	S2–C1	1.632(7)	N1–H1	0.95
O2–C11	1.202(4)	N1···O1	2.705(3)	O1–C11	1.197(7)	N1···S2	3.062(5)
Angles				Angles			
S1–C1–O1	122.8(3)	O2–C11–N1	128.4(3)	S1–C1–S2	128.0(4)	O1–C11–N1	130.3(6)
C1–S1–C11	109.3(2)	C11–N1–C12	127.5(3)	C1–S1–C11	114.5(3)	C11–N1–C12	126.6(6)
S1–C11–N1	117.2(3)	N1–H1···O1	146.0	S1–C11–N1	117.3(5)	N1–H1···S2	111.8
Torsion Angles				Torsion Angles			
S1–C1–C2–C7	5.0(5)	O1–C1–C2–C3	4.8(5)	S1–C1–C2–C7	20.6(8)	S2–C1–C2–C3	19.6(9)
N1–C11–S1–C1	3.9(3)	O2–C11–N1–C12	2.2(6)	N1–C11–S1–C1	2.4(7)	O1–C11–N1–C12	5(1)
O1–C1–S1–C11	2.0(4)	S1–C11–N1–H1	10	S2–C1–S1–C11	6.3(6)	S1–C11–N1–H1	54



SCHEME 2

stirred at 20°C for 10 minutes (The color of the solution changed from red to colorless). Removal of the solvent under reduced pressure (22°C/53.3Pa) gave 0.81 g (95%) of crude 4-methoxybenzoyl *N*-phenylcarbamoyl selenide **3h** as a colorless solid. Recrystallization of the solid from a mixed solvent of dichloromethane (3 mL) and hexane (1 mL) at −20°C during 1 hour yielded 0.46 g (59%) of **3h** as colorless needles.

Acetyl N-Phenylcarbamoyl Selenide (3a). Colorless needles (35% yield); dec.: 81°C; IR (KBr): 3252 (NH), 1715 (*CO*Se), 1682 (*CO*NH), 1557 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): **IIaa,** δ 2.50 (s, 3H, CH_3), 7.15 (t, J = 7.6 Hz, 1H, NHPh), 7.35 (t, J = 7.6 Hz, 2H, NHPh), 7.53 (d, J = 7.6 Hz, 2H, NHPh), 9.91 (br, 1H, NH); **IIab,** δ 2.54 (s, 3H, CH_3), 9.15 (br, 1H, OH); ^{13}C NMR ($CDCl_3$): **IIaa,** δ 35.0 (CH_3), 119.9, 125.0, 129.2, 137.1, 156.2 (CONH), 203.7 (COSe); **IIab,** δ 35.8 (CH_3), 119.9, 124.8, 129.6, 137.4, 160.9 (C=N), 194.6 (COSe); ^{77}Se NMR ($CDCl_3$): **IIaa,** δ 687.5; **IIab,** δ 835.9. This compound is too unstable to subject in elemental analysis.

*1,1-Dimethylethanecarbonyl N-Phenylcarbamoyl Selenide (***3b***).* Colorless oil (95% yield); IR (Neat): 3246 (NH), 1718 (*CO*Se), 1670 (*CO*NH), 1549 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): **IIba,** δ 1.30 (s, 9H, CH_3), 7.14 (t, J = 7.9 Hz, 1H, NH*Ph*), 7.34 (t, J = 7.9 Hz, 2H, NH*Ph*), 7.53 (d, J = 7.9 Hz, 2H, NH*Ph*), 10.05 (br, 1H, NH); **IIbb,** δ 1.32 (s, 9H, CH_3), 7.07 (t, J = 8.3 Hz, 2H), 7.27 (t, J = 8.3 Hz, 1H), 7.53 (d, J = 8.3

Hz, 2H); ^{13}C NMR ($CDCl_3$): **IIba,** δ 26.0 (CH_3C), 50.8 (CH_3C), 119.8, 124.9, 129.1, 137.1, 156.4 (CONH), 215.6 (COSe); ^{77}Se NMR ($CDCl_3$): **IIba:** δ 639.1. This compound is too unstable to be subject to elemental analysis.

*1-Adamantanecarbonyl N-Phenylcarbamoyl Selenide (***3c***).* Colorless crystals (78% yield); dec.: 103–105°C; IR (KBr): 3229 (NH), 1715 (*CO*Se), 1664 (*CO*NH), 1555 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.75 (br, 6H, Ad), 1.97 (br, 6H, Ad), 2.11 (br, 3H, Ad), 7.14 (t, J = 7.7 Hz, 1H, NH*Ph*), 7.34 (t, J = 7.7 Hz, 2H, NH*Ph*), 7.55 (d, J = 7.7 Hz, 2H, NH*Ph*), 10.10 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 28.0 (Ad), 36.2 (Ad), 38.9 (Ad), 53.1 (Ad), 119.9, 124.9, 129.2, 137.2, 156.8 (CONH), 215.3 (COSe). This compound is too unstable to be subject to elemental analysis.

*Benzoyl N-Phenylcarbamoyl Selenide (***3d***).* Colorless crystals (99% yield); dec.: 97–99°C; IR (KBr): 3222 (NH), 1728 (*CO*Se), 1645 (*CO*NH), 1554 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): **IIda,** δ 7.18 (t, J = 7.6 Hz, 1H, NH*Ph*), 7.38 (t, J = 7.6 Hz, 2H, NH*Ph*), 7.53 (t, J = 7.9 Hz, 2H, *Ph*CO), 7.61 (d, J = 7.6 Hz, 2H, NH*Ph*), 7.69 (t, J = 7.9 Hz, 1H, *Ph*CO), 7.94 (d, J = 7.9 Hz, 2H, *Ph*CO), 10.41 (br, 1H, NH); **IIdb,** δ 7.31 (t, J = 7.3 Hz); ^{13}C NMR ($CDCl_3$): **IIda,** δ 120.0, 125.1, 127.6, 129.2, 129.3, 135.2, 137.2, 138.0, 156.0 (CONH), 199.7 (COSe); ^{77}Se NMR ($CDCl_3$): **IIda:** δ 648.4. Anal. calcd for $C_{14}H_{11}NO_2Se$: C, 55.28; H, 3.64. Found: C, 55.20; H, 3.57.

*2-Methylbenzoyl N-Phenylcarbamoyl Selenide (***3e***).* Colorless crystals (93% yield); dec.: 65–66°C; IR (KBr): 3225 (NH), 1718 (*CO*Se), 1662 (*CO*NH), 1549 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): **IIea:** δ 2.55 (s, 3H, CH_3), 7.10–7.80 (m, 9H), 10.27 (br, 1H, NH); **IIeb:** δ 2.51 (s, 3H, CH_3), 9.04 (br, 1H, OH); ^{13}C NMR ($CDCl_3$): **IIea:** δ 20.9 (CH_3), 120.0, 124.8, 125.1, 126.4, 129.2, 129.6, 132.3, 133.4, 137.0, 137.1, 157.0 (CONH), 201.8 (COSe); ^{77}Se NMR ($CDCl_3$): **IIea:** δ 678.7. Anal. calcd for $C_{15}H_{13}NO_2Se$: C, 56.61; H, 4.12. Found: C, 56.36; H, 4.21.

*4-Methylbenzoyl N-Phenylcarbamoyl Selenide (***3f***).* Colorless crystals (93% yield); dec.: 97–99°C; IR (KBr): 3219 (NH), 1693 (*CO*Se), 1645 (*CO*NH), 1547 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): **IIfa,** δ 2.43 (s, 3H, CH_3), 7.17 (t, J = 7.9 Hz, 1H, NH*Ph*), 7.31 (d, J = 8.3 Hz, 2H, C_6H_4CO), 7.37 (t, J = 7.9 Hz, 2H, NH*Ph*), 7.61 (d, J = 7.9 Hz, 2H, NH*Ph*), 7.83 (t, J = 8.3 Hz, 2H, C_6H_4CO), 10.45 (br, 1H, NH); **IIfb,** δ 2.42 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$): **IIfa,** δ 21.9 (CH_3), 120.0, 125.0, 127.7, 129.2, 130.0, 135.5, 137.2, 146.7, 156.3 (CONH), 198.9 (COSe); ^{77}Se NMR ($CDCl_3$): **IIfa,** δ 642.6. Anal. calcd for $C_{15}H_{13}NO_2Se$: C, 56.61; H, 4.12. Found: C, 56.60; H, 3.90.

*2-Methoxybenzoyl N-Phenylcarbamoyl Selenide (***3g***).* Colorless crystals (92% yield); dec.: 95–97°C; IR (KBr): 3257 (NH), 1707 (*CO*Se), 1614 (*CO*NH), 1556 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): **IIga,** δ 4.00 (s, 3H, CH_3O), 7.04 (d, J = 7.7 Hz, 1H, C_6H_4CO), 7.08 (t, J = 7.7 Hz, 1H, C_6H_4CO), 7.15 (t, J = 7.5 Hz, 1H, NH*Ph*), 7.36 (t, J = 7.5 Hz, 2H, NH*Ph*), 7.59 (t, J = 7.7 Hz, 1H, C_6H_4CO), 7.63 (d, J = 7.5 Hz, 2H, NH*Ph*), 7.87 (d, J = 7.7 Hz, 1H, C_6H_4CO), 10.61 (br, 1H, NH): **IIgb,** δ 3.93 (s, 3H, CH_3O); ^{13}C NMR ($CDCl_3$): **IIga,** δ 55.7 (CH_3O), 112.4, 120.1, 121.1, 124.8, 126.7, 129.2, 129.4, 135.9, 137.4, 159.3 (CONH), 160.2, 197.4 (COSe); ^{77}Se NMR ($CDCl_3$): **IIga,** δ 708.1. Anal. calcd for $C_{15}H_{13}NO_3Se$: C, 53.90; H, 3.92. Found: C, 53.98; H, 4.08.

*4-Methoxybenzoyl N-Phenylcarbamoyl Selenide (***3h***).* Colorless crystals (95% yield); dec.: 106–108°C; IR (KBr): 3224 (NH), 1728 (*CO*Se), 1645 (*CO*NH), 1572 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): **IIha,** δ 3.90 (s, 3H, CH_3O), 6.98 (d, J = 9.0 Hz, 2H, C_6H_4CO), 7.16 (t, J = 7.8 Hz, 1H, NH*Ph*), 7.37 (t, J = 7.8 Hz, 2H, NH*Ph*), 7.60 (d, J = 7.8 Hz, 2H, NH*Ph*), 7.91 (d, J = 9.0 Hz, 2H, C_6H_4CO), 10.51 (br, 1H, NH); **IIhb,** δ 3.86 (s, 3H, CH_3O); ^{13}C NMR ($CDCl_3$): **IIha,** δ 55.8 (CH_3O), 114.5, 119.9, 124.9, 129.2, 130.2, 130.6, 137.3, 156.4 (CONH), 165.3, 197.1 (COSe); ^{77}Se NMR ($CDCl_3$): **IIha,** δ 632.7. Anal. calcd for $C_{15}H_{13}NO_3Se$: C, 53.90; H, 3.92. Found: C, 53.65; H, 4.02.

*4-Methoxybenzoyl N-(Benzoyl)carbamoyl Selenide (***3i***).* Colorless crystals (61% yield); dec.: 81–83°C; IR (KBr): 3373 (NH), 1769 (*CO*Se), 1642 (*CO*NH), 1574 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.91 (s, 3H, CH_3O), 7.00 (d, J = 8.0 Hz, 2H, C_6H_4CO), 7.45 (t, J = 7.8 Hz, 1H, NH*Ph*), 7.58 (t, J = 7.8 Hz, 2H, NH*Ph*), 7.93 (d, J = 7.8 Hz, 2H, NH*Ph*), 8.15 (d, J = 8.0 Hz, 2H, C_6H_4CO), 10.51 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 55.8 (CH_3O), 114.7, 128.3, 128.6, 130.0, 130.5, 131.1, 133.5, 157.4 (SeCONH), 162.7 (PhCONH), 165.8, 197.6 (COSe).

*4-Methoxybenzoyl N-(Tosyl)carbamoyl Selenide (***3j***).* Colorless crystals (97% yield); dec.: 99–100°C; IR (KBr): 3365 (NH), 1719 (*CO*Se), 1646 (*CO*NH), 1509 (NH) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.44 (s, 3H, CH_3), 3.90 (s, 3H, CH_3O), 6.98 (d, J = 8.5 Hz, 2H, C_6H_4CO), 7.35 (d, J = 8.0 Hz, 2H, $C_6H_4SO_2$), 7.84 (d, J = 8.0 Hz, 2H, $C_6H_4SO_2$), 8.00 (d, J = 8.5 Hz, 2H, C_6H_4CO), 11.54 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 21.7 (CH_3), 55.8 (CH_3O), 114.7, 128.5, 128.6, 129.7, 129.8, 135.5, 145.5, 157.3 (CONH), 165.9, 194.8 (COSe). Anal. calcd for $C_{16}H_{15}NO_5SSe$: C, 46.61; H, 3.67. Found: C, 46.74; H, 3.46.

*2,6-Dimethoxybenzoyl N-(4-Methylphenyl)carbamoyl Selenide (***3k***).* Colorless needles (100% yield); dec.: 118–122°C; IR (KBr): 3231 (NH), 1695 (*CO*Se), 1654 (*CO*NH), 1593, 1523 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.33 (s, 3H, CH_3), 3.83 (s, 6H, CH_3O), 6.58 (d, J = 7.3 Hz, 2H), 7.16 (d, J = 6.7 Hz, 2H), 7.35 (t, J = 6.7 Hz, 1H), 7.50 (d, J = 7.3 Hz, 2H), 10.08 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 20.9 (CH_3), 56.2 (CH_3O), 104.3, 120.0, 129.6, 133.1, 134.4, 134.9, 156.5, 158.3 (CONH), 199.9 (COSe). Anal. calcd for $C_{17}H_{17}NO_4Se$: C, 53.98; H, 4.53. Found: C, 53.84; H, 4.41.

*3-Chloro-2,6-dimethoxybenzoyl N-Phenylcarbamoyl Selenide (***3l***).* Colorless crystals (97% yield); dec.: 84–86°C; IR (KBr): 3240 (NH), 1714 (*CO*Se), 1655 (CONH), 1547 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): **IIIa,** δ 3.86 (s, 3H, CH_3O), 3.95 (s, 3H, CH_3O), 6.70 (d, J = 9.3 Hz, 1H, C_6H_2CO), 7.18 (t, J = 7.8 Hz, 1H, NH*Ph*), 7.38 (t, J = 7.8 Hz, 2H, NH*Ph*), 7.43 (d, J = 7.8 Hz, 1H, C_6H_2CO), 7.61 (d, J = 7.8 Hz, 2H, NH*Ph*), 10.05 (br, 1H, NH); IIIb, δ 3.87 (s, 3H, CH_3O), 3.96 (s, 3H, CH_3O); ^{13}C NMR ($CDCl_3$); IIIa, δ 56.5 (CH_3O), 62.7 (CH_3O), 108.3, 119.9, 120.0, 125.1, 125.5, 129.2, 133.4, 137.2, 152.6, 155.0, 157.6 (CONH), 199.3 (COSe); ^{77}Se NMR ($CDCl_3$): **IIIa,** δ 726.1. Anal. calcd for $C_{16}H_{14}ClNO_4Se$: C, 48.20; H, 3.54. Found: C, 48.29; H, 3.56.

*4-Biphenylcarbonyl N-Phenylcarbamoyl Selenide (***3m***).* Colorless crystals (92% yield); dec.: 106–108°C; IR (KBr): 3223 (NH), 1693 (*CO*Se), 1655 (*CO*NH), 1517 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.17–7.99 (m, 14H), 10.46 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 120.0, 125.0, 127.2, 127.3, 127.8, 128.2, 128.8, 129.0, 129.1, 137.1, 139.2, 148.0, 156.1 (CONH), 198.9 (COSe).

*1-Naphthalenecarbonyl N-Phenylcarbamoyl Selenide (***3n***).* Colorless crystals (93% yield); dec.: 87–90°C; IR (KBr): 3234 (NH), 1725 (*CO*Se), 1655 (*CO*NH), 1555 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.20–8.57 (m, 12H), 10.36 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 120.0, 124.5, 124.7, 125.1, 127.2, 128.2, 128.7, 129.0, 129.3, 129.4, 134.0, 134.9, 135.5, 137.2, 157.1 (CONH), 201.9 (COSe). Anal. calcd for $C_{18}H_{13}NO_2Se$: C, 61.03; H, 3.70. Found: C, 61.02; H, 3.92.

*4-Methoxybenzoyl N-(4-Methylphenyl)carbamoyl Sulfide (***4***).* Colorless crystals; dec.: 136–142°C; IR (KBr): 3209 (NH), 1714 (COS), 1705 (CONH), 1542 (NH) cm^{-1}; ^1H NMR ($CDCl_3$): **IIIa,** δ 2.34 (s, 3H, CH_3), 3.90 (s, 3H, CH_3O), 6.98 (d, J = 8.8 Hz, 2H), 7.17 (d, J = 8.6 Hz, 2H), 7.48 (d, J = 8.6 Hz, 2H), 7.98 (d, J = 8.8 Hz, 2H), 10.82 (br, 1H, NH); **IIIb,** δ 2.32 (s, 3H, CH_3), 3.87 (s, 3H, CH_3O), 6.94 (d, J = 8.3 Hz, 2H), 7.10 (d, J = 8.3 Hz, 2H), 7.88 (d, J = 7.7 Hz, 2H), 8.05 (d, J = 7.7 Hz, 2H); ^{13}C NMR ($CDCl_3$): **IIIa,** δ 21.0 (CH_3), 55.7 (CH_3O), 114.4, 120.2, 124.5, 128.5, 129.7, 130.2, 134.7, 158.3 (CONH), 165.2, 191.9 (COS)

*4-Methoxythiobenzoyl N-(4-Methylphenyl)carbamoyl Sulfide (***5***).* Orange needles; dec.: 120–121°C [3]; IR (KBr): 3282 (NH), 1712 (CO), 1541 (NH), 1248 (CS) cm^{-1}; ^1H NMR ($CDCl_3$): **IVa,** δ 2.34 (s, 3H, CH_3), 3.88 (s, 3H, CH_3O), 6.91 (d, J = 9.3 Hz, 2H), 7.17 (d, J = 8.3 Hz, 2H), 7.49 (d, J = 8.3 Hz, 2H), 8.05 (d, J = 9.3 Hz, 2H), 11.51 (br, 1H, NH); **IVb,** δ 2.31 (s, 3H, CH_3), 3.86 (s, 3H, CH_3O), 6.85 (d, J = 8.8 Hz, 2H), 6.96 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.3 Hz, 2H), 8.10 (d, J = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$): **IVa,** δ 21.0 (CH_3), 55.7 (CH_3O), 114.0, 120.3, 124.4, 129.5, 129.8, 130.1, 135.1, 158.2 (CONH), 165.0, 224.8 (CSS); **IVb,** δ 20.8 (CH_3), 55.7 (CH_3O), 113.5, 120.1, 129.4, 129.5, 134.3, 138.2.

*Reactions of Acyl Carbamoyl Selenides (***3***) with Sodium Methoxide and p-Toluidine*

*Reaction of 4-Methoxybenzoyl N-Phenylcarbamoyl Selenide (***3h***) with Sodium Methoxide.* 4-Methoxybenzoyl *N*-phenylcarbamoyl selenide **3h** (0.17 g, 0.5 mmol) and sodium methoxide (0.03 g, 0.5 mmol) were stirred in diethyl ether (5 mL) at 24°C for 1 hour. The solvent was evaporated under reduced pressure to give a yellow solid containing sodium 4-methoxybenzenecarboselenoate **6**. To the solid, iodomethane (1 mL) was added, and the mixture was stirred at 24°C for 1 hour. To the reaction mixture was added diethyl ether (3 mL). The resulting precipitates (NaI containing methyl *N*-phenylcarbamate **7**) was filtered off. Evaporation of the solvent from the filtrate under reduced pressure gave 0.11 g (98%) of *Se*-methyl 4-methoxybenzenecarboselenoate, which was identified by comparison of the IR and ^1H NMR spectra with those of the authentic sample.

Methyl N-phenylcarbamate **7.** m.p.: 43–45°C; IR (KBr): 3321 (NH), 1714 (*CO*) cm^{-1}; ^1H NMR ($CDCl_3$): δ 3.72 (s, 3H, CH_3), 7.02 (t, J = 7.8 Hz, 1H), 7.02 (br, 1H, NH), 7.25 (t, J = 7.8 Hz, 2H), 7.38 (d, J = 7.8 Hz, 2H); ^{13}C NMR ($CDCl_3$): δ 52.1 (CH_3), 123.3, 129.5, 128.8, 137.9, 154.3 (CO).

Reaction of **3h** *with p-Toluidine.* To a solution of 4-methoxybenzoyl N-phenylcarbamoyl selenide 3h (0.26 g, 0.8 mmol) in dichloromethane (10 mL) p-toluidine (0.17 g, 1.6 mmol) was added and the mixture was stirred at room temperature for 7 h. Af-

ter evaporation of the solvent under reduced pressure, diethyl ether (5 mL) was added. Collection of the resulting precipitates by filtration gave 0.16 g (85% yield) *N*-4-methylphenyl *N′*-phenyl urea **9** as colorless needles. Removal of the diethyl ether from the filtrate under reduced pressure gave 0.11 g (57% yield) of N-4-methylphenyl 4-methoxybenzamide **8** as colorless crystals.

*N-4-Methylphenyl 4-Methoxybenzamide (***8***).* Colorless crystals; m.p.: 156–157°C; IR (KBr): 3340 (NH), 1651 (CO) cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.31 (s, 3H, CH_3), 3.83 (s, 3H, CH_3O), 6.90 (d, J = 8.8 Hz, 2H), 7.12 (d, J = 8.3 Hz, 2H), 7.50 (d, J = 8.3 Hz, 2H), 7.80 (d, J = 8.8 Hz, 2H), 7.95 (br, 1H, NH); ^{13}C NMR ($CDCl_3$): δ 20.9 (CH_3), 55.4 (CH_3O), 113.8, 120.4, 127.2, 128.9, 129.5, 133.9, 135.6, 162.3 (CO), 165.3.

*N-4-Methylphenyl N′-Phenyl Urea (***9***).* Colorless needles; m.p.: 204–205°C; IR (KBr): 3303 (NH), 1635 (CO) cm^{-1}; ^1H NMR ($CDCl_3$ + DMSO-d^6): δ 2.27 (s, 3H, CH_3), 6.94 (t, J = 7.7 Hz, 1H), 7.05 (d, J = 8.3 Hz, 2H), 7.24 (t, J = 7.7 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H), 7.43 (d, J = 7.7 Hz, 2H), 8.42 (br, 1H, NH), 8.49 (br, 1H, br); ^{13}C NMR ($CDCl_3$ + DMSO-d^6): δ 20.4 (CH_3), 118.1, 118.3, 121.5, 128.5, 129.0, 130.6, 137.0, 139.7, 152.6 (CO).

ACKNOWLEDGMENTS

S. K. thanks Prof. Takashi Kawamura and Dr. Masahiro Ebihara (Gifu University) for invaluable assistance with crystallography and Mr. Kohji Maeshima for assistance of the experiment.

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