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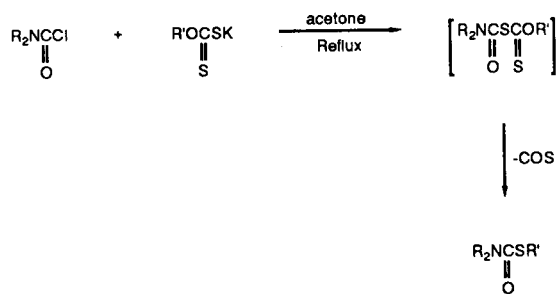
Monsanto Agricultural Company, A Unit of Monsanto Company,  
 800 North Lindbergh Boulevard, St. Louis, MO 63167

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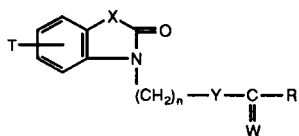
The reaction of 3-(2-hydroxyethyl)-2-benzothiazolinone with methyl, phenyl isocyanate, or dimethylcarbamoyl chloride afforded the carbamates **1-4**. The carbanilate **5** was prepared by the reaction of 2-benzothiazolinone with 3-chloropropyl-*N*-methylcarbanilate under basic conditions. The reaction of the appropriate 2-benzothiazolinone with the appropriate 2-chloro or 3-chloropropyl disubstituted thiocarbamate under basic conditions furnished the thiocarbamates **6-14**. The thiocarbamate **15** was prepared by the reaction of sodium diisopropylthiocarbamate with 3-(chloromethyl)-2-benzothiazolinone. The reaction of 3-(chloromethyl)-2-benzothiazolinone and related compound with the appropriate sodium or triethylamine salt of disubstituted dithiocarbamate afforded the dithiocarbamate **16-22**. The reaction of the appropriate xanthate with the potassium salt of bromoacetic acid and the appropriate secondary amine afforded the thionocarbamates **23-29**. The thionocarbamate **30** was synthesized by the reaction of 5-chloro-2-benzothiazolinone with 3-chloropropyl diethylthionocarbamate.

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During the past thirty-five years, the following nine commercial herbicides: 2-chloroallyl diethylthiocarbamate [1a,b], *cis*- and *trans*-2,3-dichloroallyl diisopropylthiocarbamate [2a,b,c], 2,3,3-trichloroallyl diisopropylthiocarbamate [2a,b,c], *p*-chlorobenzyl diethylthiocarbamate [3], ethyl dipropylthiocarbamate [7a,b,c], ethyl disecbutylthiocarbamate [5a,b,c], propyl *N*-butyl *N*-ethylthiocarbamate [4a,b,c], ethyl hexamethyleniminecarbothioate [4a,b,c], and 4-chloro-2-butyne *m*-chlorocarbanilate [5] were discovered. Moreover, in 1980 we reported [6] a new route for the synthesis of disubstituted-thiocarbamates *via* a sulfur transfer agent as illustrated by the following reaction:



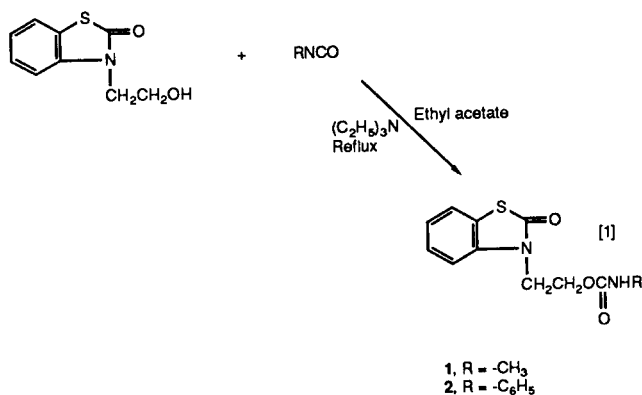
Because of their outstanding biological activity, it appeared expedient to continue our research effort in this area of chemistry. Accordingly, the purpose of this investigation was to synthesize the following new carbamates, thiocarbamates, dithiocarbamates and thionocarbamates:



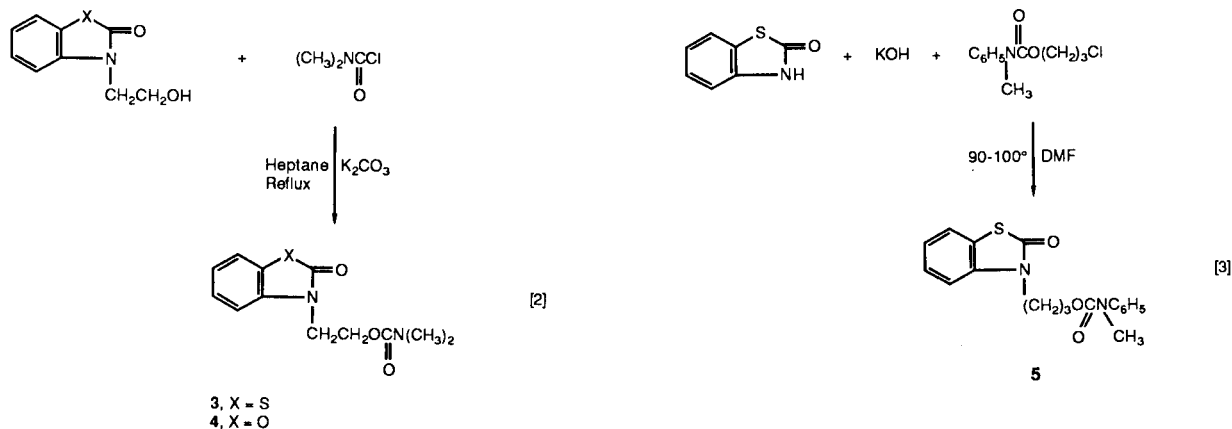
T = H, 5-Cl, 6-Br, or 6-OC<sub>2</sub>H<sub>5</sub>. X = S or O.  
 n = 1,2, or 3. Y = W = O or S.

Y = S, W = O. Y = O, W = S.  
 R = -NHalkyl, -(alkyl)<sub>2</sub>, -N(CH<sub>2</sub>)<sub>2</sub> or -N(CH<sub>2</sub>)<sub>6</sub>O.

The reaction of methyl isocyanate or phenyl isocyanate with 3-(2-hydroxyethyl)-2-benzothiazolinone [7] afforded 2-(2-oxobenzothiazolin-3-yl)ethyl methylcarbamate (**1**) and 2-(2-oxobenzothiazolin-3-yl)ethyl carbanilate (**2**), respectively (Reaction 1).



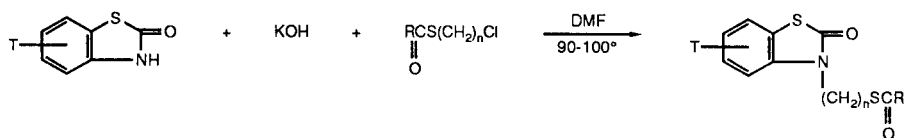
2-(2-Oxobenzothiazolin-3-yl)ethyl dimethylcarbamate (**3**) and 2-(2-oxobenzoxazolin-3-yl)ethyl dimethylcarbamate (**4**) were prepared by the reaction of 3-(2-hydroxyethyl)-2-benzothiazolinone [7] or 3-(2-hydroxyethyl)-2-benzoxazolinone [7] with dimethylcarbamoyl chloride under basic conditions (Reaction 2).



The treatment of 2-benzothiazolinone [8] with 3-chloropropyl-*N*-methylcarbanilate under basic conditions afforded 3-(2-oxobenzothiazolin-3-yl)propyl *N*-methylcarbanilate (5) (Reaction 3).

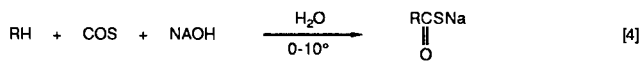
The intermediates, 2-chloroethyl and 3-chloropropyl di-substituted-thiolcarbamates [9a,b] were prepared as illustrated by the following reactions 4 and 5:

Table 1

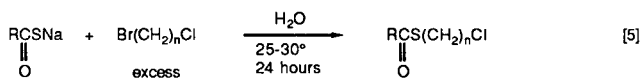


No.	T	n	R	Mp°	Crude % Yield	Empirical formula	C	Analysis %		
								Calcd./Found	H	N
6	5-Cl	3	-N(C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	[a]	90	C <sub>12</sub> H <sub>13</sub> ClN <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	52.76 52.86	5.99 5.80	7.24 7.52	16.57 16.77
7	6-OC <sub>2</sub> H <sub>5</sub>	3	-N(C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	[a]	80	C <sub>14</sub> H <sub>28</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	57.24 57.14	7.12 7.22	7.06 7.34	16.17 15.97
8	H	3	-N(C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	[a]	90	C <sub>17</sub> H <sub>24</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	57.92 57.80	6.86 6.56	7.95 8.06	18.19 17.98
9	H	3	-N(CH <sub>3</sub> ) <sub>2</sub>	[a]	74	C <sub>13</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	52.67 52.50	5.44 5.24	9.45 9.50	21.64 21.49
10	H	3	-N(CH(CH <sub>3</sub> ) <sub>2</sub> ) <sub>2</sub>	[a]	88	C <sub>17</sub> H <sub>24</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	57.92 57.85	6.86 6.66	7.95 8.18	18.19 17.90
11	5-Cl	3	-N(CH <sub>3</sub> ) <sub>2</sub>	[a]	83	C <sub>13</sub> H <sub>15</sub> ClN <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	47.19 47.01	4.57 4.39	8.47 8.29	19.38 19.18
12	5-Cl	2	-N(C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	[a]	81	C <sub>16</sub> H <sub>21</sub> ClN <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	51.53 51.26	5.68 5.58	7.51 7.64	17.20 17.10
13	5-Cl	3	-N(CH(CH <sub>3</sub> ) <sub>2</sub> ) <sub>2</sub>	109-110	44	C <sub>17</sub> H <sub>23</sub> ClN <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	52.76 52.68	5.99 5.84	7.24 7.11	16.57 16.87
14	H	2	-N(CH <sub>3</sub> ) <sub>2</sub>	82-84	50	C <sub>12</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	51.04 51.21	5.00 4.90	9.92 9.70	22.71 22.98

[a] Amber viscous liquid.

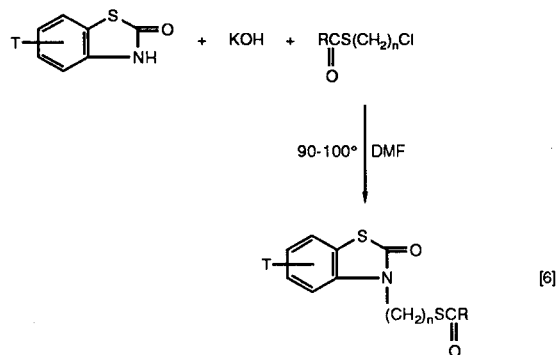


35% excess



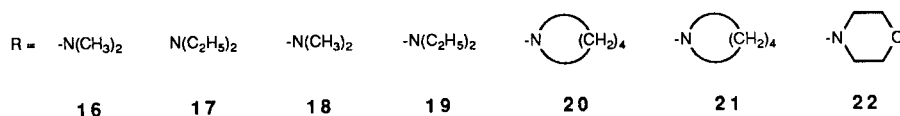
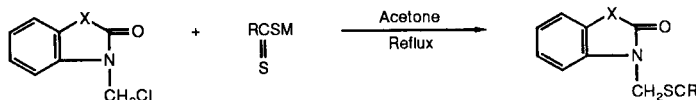
R = -N(CH<sub>3</sub>)<sub>2</sub>, -N(C<sub>3</sub>H<sub>7</sub>)<sub>2</sub>, -N(CH(CH<sub>3</sub>)<sub>2</sub>)<sub>2</sub>  
 n = 2 or 3.

The reaction of 2-benzothiazolinone [8], 5-chloro-2-benzothiazolinone [10] or 6-ethoxy-2-benzothiazolinone [11] with the appropriate 2-chloro or 3-chloropropyl disubstituted-thiolcarbamate [9a,b] under basic conditions afforded 3-(2-oxo-benzothiazolin-3-yl)propyl disubstituted-thiolcarbamates and related compounds **6-14** (Reaction 6).



T, n, R are shown in Table 1.

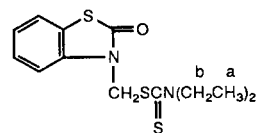
Table 2



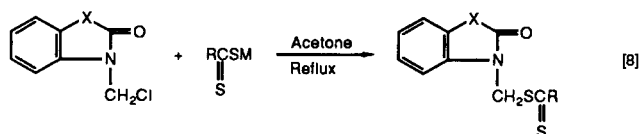
No.	X	M	Mp°	Crude % Yield	Empirical formula	C	Analysis %		
							Calcd./Found	H	N
16 [c]	S	Na · 2H <sub>2</sub> O	135-136 [a]	95	C <sub>11</sub> H <sub>12</sub> N <sub>2</sub> OS <sub>3</sub>	46.45	4.25	9.85	33.82
						46.46	4.28	9.90	33.75
17 [d]	S	H · N(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub>	112-113 [b]	99	C <sub>13</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	49.97	5.16	8.97	30.78
						49.85	5.17	8.95	30.73
18	O	Na · 2H <sub>2</sub> O	163-164 [a]	95	C <sub>11</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	49.23	4.51	10.44	23.90
						49.35	4.66	10.39	23.76
19	O	Na · 3H <sub>2</sub> O	88-89 [b]	99	C <sub>13</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	52.68	5.44	9.45	21.63
						52.68	5.45	9.43	21.61
20	S	H · N(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub>	165-167 [a]	97	C <sub>13</sub> H <sub>14</sub> N <sub>2</sub> OS <sub>3</sub>	50.29	4.55	9.02	30.98
						50.22	4.58	9.04	31.00
21	O	H · N(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub>	148-149 [a]	95	C <sub>13</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	53.04	4.79	9.52	21.78
						53.12	4.81	9.53	21.75
22	O	H · N(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub>	140-141 [a]	97	C <sub>13</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	50.30	4.55	9.03	20.66
						50.50	4.67	8.97	20.56

[a] Recrystallization from ethyl acetate. [b] Recrystallization from 2-propanol.

[c] <sup>1</sup>H nmr (deuteriochloroform): δ 3.30 and 3.5 (2s, 6, N(CH<sub>3</sub>)<sub>2</sub>, restricted rotation, 5.90 (s, 2, NCH<sub>2</sub>), 6.85-7.60 (m, 4, ArH). [d] <sup>1</sup>H nmr (deuteriochloroform): δ 1.20 (t, 6, a), 3.60 and 3.95 (2q, 4, b), restricted rotation, 5.90 (s, 2, NCH<sub>2</sub>), 6.80-7.70 (m, 4, ArH).

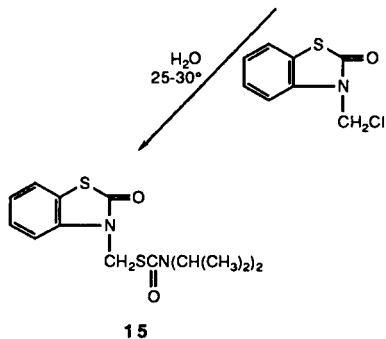
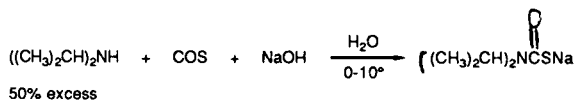


(2-Oxobenzothiazolin-3-yl)methyl diisopropylthiolcarbamate (**15**) was prepared by the reaction of sodium diisopropylthiolcarbamate with 3-(chloromethyl)-2-benzothiazolinone [12] (Reaction 7).

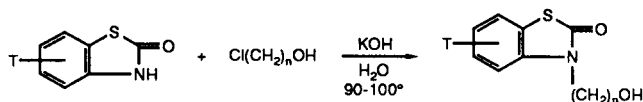


X, R and M are shown in Table 2.

The intermediates, S-potassium O-[2-(2-oxobenzothiazolin-3-yl)ethyl]xanthic acid and related xanthates [7], were prepared as illustrated by the following reaction 9.

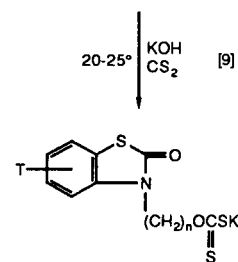


[7]



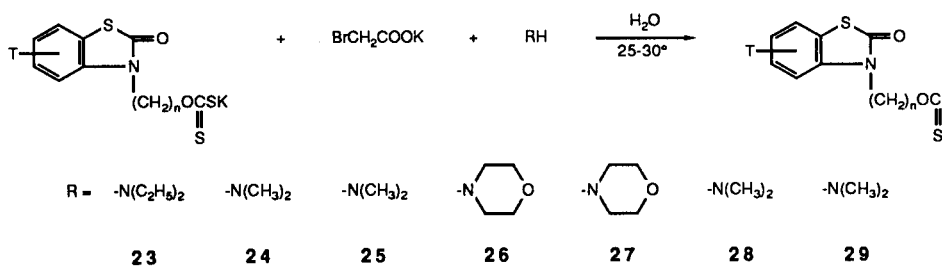
T = H, 6-Br or 6-OC<sub>2</sub>H<sub>5</sub>,  
n = 2 or 3

[9]



The reaction of 3-(chloromethyl)-2-benzothiazolinone [12] or 3-(chloromethyl)-2-benzoxazolinone [12] with the appropriate sodium or triethylamine salt of disubstituted-dithiocarbamate (Table 2) afforded (2-oxobenzothiazolin-3-yl or 2-oxobenzoxazolin-3-yl)methyl disubstituted-dithiocarbamates **16-22** (Reaction 8).

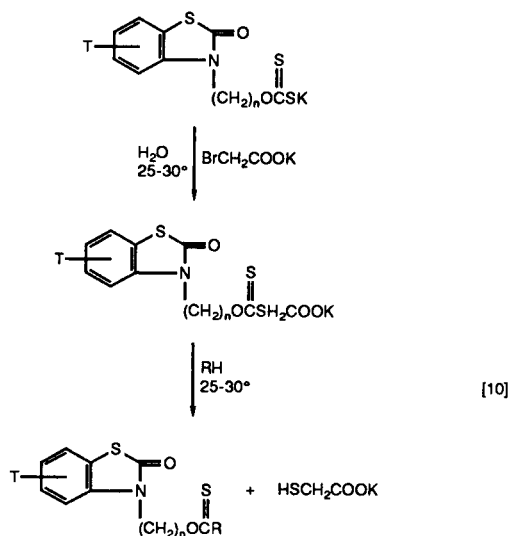
Table 3



No.	T	n	Mp°	Crude % Yield	Empirical formula	Analysis %			
						C	H	N	S
<b>23</b>	H	2	89-90 [a]	47	C <sub>14</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	54.16	5.84	9.02	20.65
						54.01	5.65	8.96	20.64
<b>24</b>	H	2	126-127 [a]	52	C <sub>12</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	51.04	5.00	9.92	22.71
						51.21	4.90	9.72	22.59
<b>25</b>	H	3	114-115 [a]	98	C <sub>13</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	52.67	5.44	9.45	21.63
						52.87	5.51	9.54	21.82
<b>26</b>	H	2	147-148 [b]	70	C <sub>14</sub> H <sub>16</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	51.83	4.97	8.64	19.77
						51.65	4.83	8.48	19.96
<b>27</b>	H	3	122-123 [b]	70	C <sub>15</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	53.23	5.36	8.28	18.95
						53.10	5.20	8.08	19.15
<b>28</b>	6-Br	2	131-132 [b]	76	C <sub>12</sub> H <sub>13</sub> BrN <sub>2</sub> O <sub>2</sub> S <sub>2</sub>	39.88	3.63	7.75	17.75
						39.79	3.66	7.68	17.66
<b>29</b>	6-OC <sub>2</sub> H <sub>5</sub>	2	121-122 [a]	49	C <sub>14</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	51.51	5.56	8.58	19.64
						51.32	5.57	8.50	19.83

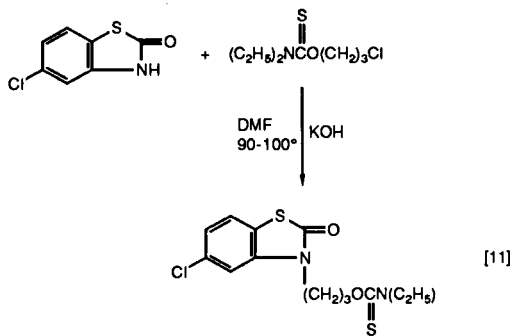
[a] Recrystallization from 2-propanol. [b] Recrystallization from ethyl acetate.

The reaction of the above xanthates with the potassium salt of bromoacetic acid and the appropriate secondary amine afforded 2-(2-oxobenzothiazolin-3-yl)ethyl disubstituted-thionocarbamates **23-29** (Reaction 10).

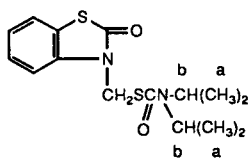


T, n and R are shown in Table 3.

3-(5-Chloro-2-oxobenzothiazolin-3-yl)propyl diethylthionocarbamate (**30**) was prepared by the reaction of 5-chloro-2-benzothiazolinone [10] with 3-chloropropyl diethylthionocarbamate [13] under basic conditions (Reaction 11).

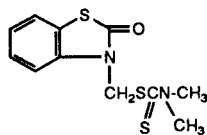


It is noteworthy to contrast the chemical shifts for the thiolcarbamate **15** and the dithiocarbamate **16**.



**15**

1.32 (d, 12, a)  
3.79 (heptet, 2, b)  
5.58 (s, 2, NCH<sub>2</sub>)  
7.05-7.62 (m, 4, ArH)



**16**

3.30 (s, 3, NCH<sub>2</sub>) Restricted  
3.55 (s, 3, NCH<sub>2</sub>) rotation  
5.90 (s, 2, NCH<sub>2</sub>)  
6.85-7.60 (m, 4, ArH)

In **16**, the N(CH<sub>3</sub>) amino protons were nonequivalent whereas in **15** the amino protons N(CH(CH<sub>3</sub>)<sub>2</sub>)<sub>2</sub> were equivalent.

## EXPERIMENTAL

The <sup>1</sup>H nmr spectra were obtained with a Varian T-60 NMR spectrometer. The chemical shifts are reported in δ, using tetramethylsilane as reference. All melting points were taken upon a Fisher-Johns block and are uncorrected.

2-(2-Oxobenzothiazolin-3-yl)ethyl Methylcarbamate (**1**) and 2-(2-Oxobenzothiazolin-3-yl)ethyl Carbanilate (**2**).

To a stirred charge containing 19.5 g (0.1 mole) of 3-(2-hydroxyethyl)-2-benzothiazolinone [7], 100 ml of ethyl acetate and 1 ml of triethylamine, 0.1 mole of methyl or phenyl isocyanate was added in one portion. The stirred reaction mixture was heated at reflux for 6 hours and at 25-30° for 18 hours. After cooling to 5°, the reaction mixture was stirred at 0-10° for 1 hour. The solids, **1** and **2**, were collected by filtration and air-dried at 25-30°; **1** had mp 107-108°, after recrystallization from ethanol, and **2** had mp 122-123°, after recrystallization from ethanol and were obtained in 95 and 86% yield, respectively.

*Anal.* (**1**) Calcd. for C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>S: C, 52.37; H, 4.79; N, 11.10; S, 12.71. Found: C, 52.47; H, 4.83; N, 11.05; S, 12.90.

*Anal.* (**2**) Calcd. for C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S: C, 61.13; H, 4.49; N, 8.91; S, 10.20. Found: C, 61.03; H, 4.40; N, 8.95; S, 10.49.

2-(2-Oxobenzothiazolin-3-yl)ethyl Dimethylcarbamate (**3**) and 2-(2-Oxobenzoxazolin-3-yl)ethyl Dimethylcarbamate (**4**).

To a stirred slurry containing 0.2 mole of 3-(2-hydroxyethyl)-2-benzothiazolinone [7] or 3-(2-hydroxyethyl)-2-benzoxazolinone [7], 28 g (0.2 mole) of potassium carbonate and 200 ml of heptane, 26.6 g (0.25 mole) of dimethylcarbonyl chloride was added in one portion. The stirred reaction mixture was heated at reflux for 7 hours and at 25-30° for 18 hours. After the addition of 800 ml of water, stirring was continued at 25-30° for 1 hour. The solids, **3** and **4**, were collected by filtration, washed with water until neutral and air-dried at 25-30°; **3** had mp 74-75°, after recrystallization from heptane-2-propanol and **4**, mp 82-83°, after recrystallization from 2-propanol, were obtained in 77 and 72% yield, respectively.

*Anal.* (**3**) Calcd. for C<sub>12</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S: C, 54.12; H, 5.30; N, 10.52; S, 12.10. Found: C, 54.32; H, 5.25; N, 10.43; S, 12.10.

*Anal.* (**4**) Calcd. for C<sub>12</sub>H<sub>14</sub>N<sub>2</sub>O<sub>4</sub>: C, 57.59; H, 5.64; N, 11.20. Found: C, 57.62; H, 5.65; N, 10.92.

3-(2-Oxobenzothiazolin-3-yl)propyl *N*-Methylcarbanilate (**5**).

To a stirred charge containing 15.1 g (0.1 mole) of 2-benzothiazolinone [8], 6.6 g (0.1 mole) of 85% potassium hydroxide and 150 ml of dimethylformamide, 22.8 g (0.1 mole) of 3-chloropropyl *N*-methylcarbanilate was added in one portion. The stirred reaction mixture was heated at 90-100° for 24 hours. After cooling to 25°, 500 ml of water and 600 ml of ethyl ether were added and stirring continued at 25-30° for 15 minutes. The separated ethyl layer was washed with water until neutral and dried over sodium sulfate. The ether was removed *in vacuo* at maximum temperature of 80-90° at 1-2 mm Hg; **5**, a dark amber viscous liquid, was obtained in 82% yield.

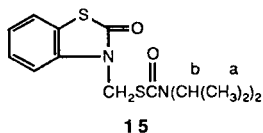
*Anal.* Calcd. for C<sub>18</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>S: C, 63.13; H, 5.30; N, 8.18; S, 9.36. Found: C, 63.03; H, 5.20; N, 8.38; S, 9.42.

### 3-(2-Oxobenzothiazolin-3-yl)propyl Disubstituted-thiolcarbamate and Related Compounds 6-14.

To a stirred charge containing 0.11 mole of 2-benzothiazolinone [8], 5-chloro-2-benzothiazolinone [10] or 6-ethoxy-2-benzothiazolinone [11], 7.3 g (0.11 mole) of 85% potassium hydroxide and 200 ml of dimethylformamide, 0.1 mole of 2-chloroethyl or 3-chloropropyl disubstituted-thiolcarbamate [9a,b] was added and then heated at 90-100° for 24 hours. For **13** and **14**, after cooling to 5° and the addition of 600 g of ice water, stirring was continued at 0-10° for 2 hours. The solid was collected by filtration, washed with water until neutral and air-dried at 25-30°. For **6-12**, after cooling to 25°, 500 ml of water and 600 ml of ethyl ether were added, and stirring continued at 25-30° for 15 minutes. The separated ether was washed with water until neutral and dried over sodium sulfate. The ether was removed *in vacuo* at a maximum temperature of 80-90° at 1-2 mm Hg. The data are summarized in Table 1.

### (2-Oxobenzothiazolin-3-yl)methyl Diisopropylthiolcarbamate (15).

To a stirred charge containing 15 g (0.15 mole) of diisopropylamine, 8 g (0.1 mole) of 50% sodium hydroxide and 50 ml of water, 7.8 g (0.15 mole) of 85% carbonyl sulfide was added at 0-10° over a 15 minute period. To this stirred mixture, 19.6 g (0.1 mole) of 3-(chloromethyl)-2-benzothiazolinone [12] and 100 ml of tetrahydrofuran were added in one portion. The reaction mixture was stirred at 0-20° for the first 4 hours and at 25-30° for 2 days. After the addition of 800 g of ice water, stirring was continued at 0-10° for 30 minutes. The solid was collected by filtration, washed with water until neutral and air-dried at 25-30°. Crude **15** was obtained in 86% yield. After recrystallization from heptane-2-propanol, **15** melted at 118-120°; <sup>1</sup>H nmr (deuteriochloroform): δ 1.32 (d, 12, a), 3.79 (heptet, 2, b), 5.58 (s, 2, NCH<sub>2</sub>), 7.05-7.62 (m, 4, ArH).



*Anal.* Calcd. for C<sub>15</sub>H<sub>20</sub>N<sub>2</sub>O<sub>2</sub>S<sub>2</sub>: C, 55.53; H, 6.21; N, 8.63; S, 19.76. Found: C, 55.48; H, 6.24; N, 8.62; S, 19.83.

### (2-Oxobenzothiazolin-3-yl or 2-oxobenzoxazolin-3-yl)methyl Disubstituted-dithiocarbamates 16-22.

To a stirred charge containing 0.11 mole of the appropriate sodium or triethylamine salt of disubstituted-dithiocarbamic acid in 200 ml of acetone, 0.1 mole of 3-(chloromethyl)-2-benzothiazolinone [12] or 3-(chloromethyl)-2-benzoxazolinone [12] was added in one portion. The stirred reaction mixture was heated at reflux for 24 hours. After cooling to 5°, 800 g of ice water was added and stirring continued at 0-10° for 30 minutes. The solids were collected by filtration, washed with water until neutral and air-dried at 25-30°. The data are summarized in Table 2.

### 2-(2-Oxobenzothiazolin-3-yl)ethyl Disubstituted-thionocarbamate and Related Compounds 23-29.

To a stirred solution containing 20.9 g (0.15 mole) of bromoacetic acid in 250 ml of water, 12 g (0.08 mole) of potassium carbonate was added in small portions (foaming) until pH 8 was obtained. To this stirred solution 0.15 mole of the appropriate xan-

thate [7] was added in one portion and stirring continued at 25-30° for 1.5 hours. This was followed by the addition of 0.3 mole of dimethylamine, diethylamine or morpholine, and stirring was continued at 25-30° for 25 hours. After the addition of 200 g of ice water, the stirred reaction was held at 0-10° for 30 minutes. The solids were collected by filtration, washed with water until neutral and air-dried at 25-30°. The data are summarized in Table 3.

### 3-(5-Chloro-2-oxobenzothiazolin-3-yl)propyl Diethylthionocarbamate (30).

To a stirred charge containing 20.4 g (0.11 mole) of 5-chloro-2-benzothiazolinone [10], 7.3 g (0.11 mole) of 85% potassium hydroxide and 200 ml of dimethylformamide, 21 g (0.1 mole) of 3-chloropropyl diethylthionocarbamate [13] was added in one portion. The stirred reaction mixture was heated at 90-100° for 24 hours. After cooling to 25°, 500 ml of water and 600 ml of ethyl ether were added and stirring continued at 25-30° for 30 minutes. The separated ether layer was washed with water until neutral and dried over sodium sulfate. The ether was removed *in vacuo* at maximum temperature of 80-90° at 1-2 mm Hg; **30**, an amber viscous liquid, was obtained in 81% yield.

*Anal.* Calcd. for C<sub>15</sub>H<sub>15</sub>ClN<sub>2</sub>O<sub>2</sub>S<sub>2</sub>: C, 50.19; H, 5.34; N, 7.81; S, 17.87. Found: C, 50.30; H, 5.40; N, 8.00; S, 17.80.

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