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Received June 13, 1988

2-*N*-Nitrosomethylaminopyridine but not 2-methylaminopyridine, 3-*N*-nitrosomethylamino- or 3-methylaminopyridine and 4-*N*-nitrosomethylamino- or 4-methylaminopyridine react with malonate esters to form the corresponding derivatives of carbamates, amides and dihydropyridines respectively. In the reaction of the 4-isomers with diisopropyl malonate small amounts of the corresponding carbamates was also formed.

*J. Heterocyclic Chem.*, **26**, 155 (1989).

Nitroso compounds, because of the polar character of the  $-N=O$  group, react with methyl or methylene groups (Erlich-Sachs reaction) in a manner similar to carbonyl groups to give imines or nitrones [1]. Depending on the reaction conditions several families of compounds were obtained as primary or secondary products in reactions which involved compounds with active methylene groups and aromatic nitroso compounds [2,3].

Continuing our previous work on nitrosation of heterocyclic amines [4,5,6,7] and in view of the interest in the carcinogenic properties [8] of the secondary nitrosamines a study of reactions which involve secondary heterocyclic aromatic nitrosamines with esters containing active methylene groups was undertaken.

### Results and Discussion.

2-*N*-nitrosomethylaminopyridine (**1a**) reacted (Table 1) with esters of type **2** to give carbamates **3** (Tables 2 and 3) presumably by the attack of **1a** on the carbonyl group and subsequent breaking of the C-C bond of **2** (Scheme 1). Reaction took place with dimethyl (**2a**), diethyl (**2b**) and diisopropyl malonate (**2e**) but not with ethyl acetoacetate (**2f**). Reaction also did not take place when the two active methylene hydrogens were replaced by methyl groups, as in the case of diethyl 2,2-dimethylmalonate (**2d**) probably because of steric hindrance.

When 2-methylaminopyridine (**1b**) was used instead of **1a** no reaction was observed even after prolong heating

Table 1  
Results of Reactions Between **2** and **1**, **4** or **7**

	<b>2</b>					<b>3</b>			<b>5</b>			<b>8</b>		
	X	Y	R <sub>1</sub>	R <sub>2</sub>	Z	Time (hours)	Yield (%)	Compound	Time (hours)	Yield (%)	Compound	Time [a] (hours)	Yield (%)	Compound
<b>a</b>	OMe	OMe	H	H	NO	2	15	<b>3a</b>	5	—	—	2 (1)	15	<b>8a</b>
<b>b</b>	OEt	OEt	H	H	NO	2	15	<b>3b</b>	1	20	<b>5a</b>	3 (1)	15	<b>8b</b>
<b>c</b>	OEt	OEt	Me	H	NO	—	—	—	2	20	<b>5b</b>	5 (5)	—	—
<b>d</b>	OEt	OEt	Me	Me	NO	2	—	—	5	—	—	—	—	—
<b>e</b>	OiP	OiP	H	H	NO	¾	15	<b>3c</b>	5	20	<b>5c</b>	3 (1)	15	<b>8c</b>
<b>f</b>	Me	OMe	H	H	NO	2	—	—	5	—	—	—	—	—
<b>a</b>	OMe	OMe	H	H	H	6	—	—	5	—	—	2 (1)	15	<b>8a</b>
<b>b</b>	OEt	OEt	H	H	H	6	—	—	1	20	<b>5a</b>	3 (1)	15	<b>8b</b>
<b>c</b>	OEt	OEt	Me	H	H	—	—	—	2	20	<b>5b</b>	5 (1)	—	—
<b>d</b>	OEt	OEt	Me	Me	H	6	—	—	5	—	—	—	—	—
<b>e</b>	OiP	OiP	H	H	H	6	—	—	5	20	<b>5c</b>	3 (1)	15	<b>8c</b>
<b>f</b>	Me	OMe	H	H	H	6	—	—	5	—	—	—	—	—

[a] In parentheses is the time of completion of the reaction in the presence of sodium methoxide.

Scheme 1

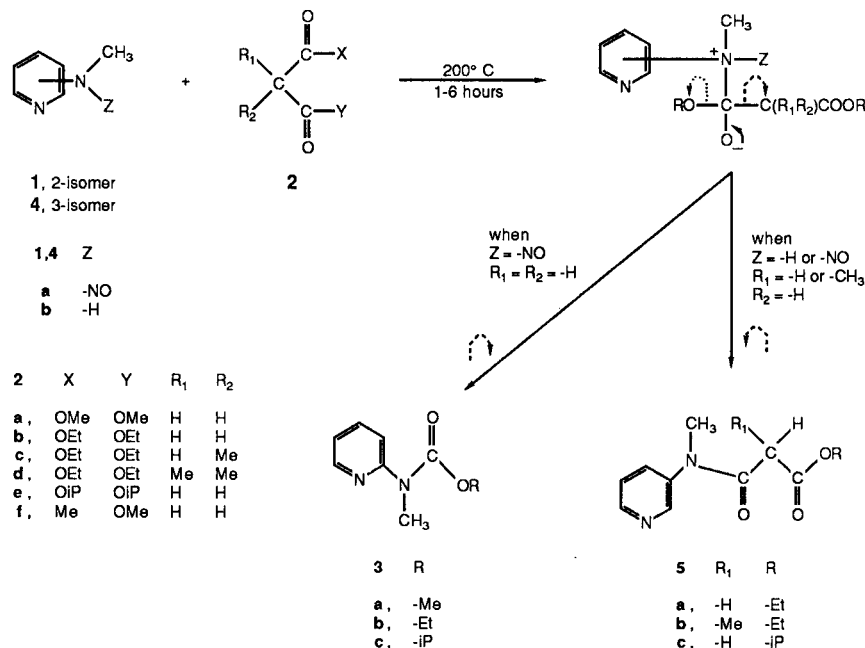


Table 2

Analytical Data of Products 3, 5, 6, and 8

Product	Molecular Formula	Calcd. (%)			Found (%)		
		C	H	N	C	H	N
3a	C <sub>9</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub>	57.83	6.02	16.87	57.74	6.10	16.62
3b	C <sub>9</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>	60.00	6.67	15.55	59.78	6.72	15.68
3c	C <sub>10</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub>	61.86	7.22	14.43	61.54	7.40	14.16
5a	C <sub>11</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub>	59.46	6.31	12.61	58.91	6.14	12.84
5b	C <sub>12</sub> H <sub>16</sub> N <sub>2</sub> O <sub>3</sub>	61.02	6.78	11.86	60.44	7.22	11.57
5c	C <sub>12</sub> H <sub>16</sub> N <sub>2</sub> O <sub>3</sub>	61.02	6.78	11.86	60.78	7.14	11.35
6	C <sub>12</sub> H <sub>15</sub> NO <sub>3</sub>	65.16	6.79	6.33	64.94	7.02	6.52
8a	C <sub>11</sub> H <sub>13</sub> NO <sub>4</sub>	59.19	5.82	6.27	59.47	6.31	5.88
8b	C <sub>14</sub> H <sub>19</sub> NO <sub>4</sub>	63.39	7.16	5.28	63.43	6.96	5.44
8c	C <sub>17</sub> H <sub>23</sub> NO <sub>4</sub>	66.44	8.14	4.56	66.75	7.59	4.88

(Table 1). This observation suggests that the nitroso moiety may perhaps be a prerequisite for these reactions to take place.

The products **3** were hydrolysed with sodium hydroxide solution or hydrochloric acid to give **1b** (Tables 2 and 3). This hydrolysis pattern is consistent with that of carbamic esters [9]. The ir spectra of **3** showed an absorption at 1710 cm<sup>-1</sup> which is in agreement with the absorption of the carbonyl group of carbamates [10a] (Table 3). The <sup>13</sup>C chemical shift of the carbonyl carbon of the **3** is a singlet

which remains unaffected even under off-resonance decoupling. Depending on the group R in **3** a slight shift in the absorption of the carbonyl carbon is observed from 155.9 ppm for R = CH<sub>3</sub> to 155.1 ppm for R = CH(CH<sub>3</sub>)<sub>2</sub> presumably because of an increase in the +I effect of R group. These values agree with those in the literature [11]. The carbonyl shifts may be differentiated from those of carbon-2 of the pyridine nucleus (Table 3) since the latter show much smaller change (154.9-155.1 ppm).

3-N-Nitrosomethylaminopyridine (**4a**) and 3-methylaminopyridine (**4b**) reacted with **2** (Table 1) to give amide derivatives **5** (Tables 2 and 3) presumably by an attack on the carbonyl group and subsequent removal of an alkoxy group of **2** (Scheme 1). The formation of amides is to be expected since position-3 of the pyridine nucleus of **4** is very little affected by the ring nitrogen thus making these pyridine derivatives to behave like the respective aromatic amines [13]. This is shown by the fact that *N*-nitroso-*N*-methylaniline reacts with **2b** to give *N*-methyl-*N*-phenyl- $\alpha$ -carboethoxyacetamide (**6**) (Tables 2 and 3). However, no reaction was observed with **2a** or **2f** since in the former case the methoxy is not as good a leaving group as the ethoxy or isopropoxy. Moreover, as in the case of **1a**, no reaction was observed between **4a** and **2d** presumably because of steric hindrance. However reaction was observed with **2c**. Also **4b** reacted in a similar manner as **4a** giving lower yields of **5** thus indicating that the nitroso moiety is not a prerequisite for these reactions to occur (Tables

Table 3

<sup>1</sup>H, <sup>13</sup>C NMR, IR and Mass Spectra of **3**, **5**, **6**, **8**, and **9**

Product	<sup>1</sup> H NMR δ ppm [a]	<sup>13</sup> C NMR δ ppm [a]	IR cm <sup>-1</sup>	MS (RI) m/e (%)
<b>3a</b>	3.44 (s, 3H)	34.2 (q)	2945 (w), 1710 (s)	166 (9)
	3.79 (s, 3H)	53.0 (q)	1590 (m), 1570 (m)	108 (10)
	7.0-8.3 (m, 4H)	118.9 (d)	1475 (m), 1435 (s)	107 (100)
		119.5 (d)	1360 (s), 1310 (m)	80 (11)
		137.2 (d)	1280 (m), 1180 (m)	79 (24)
		147.5 (d)	1140 (s), 1110 (m)	78 (56)
		154.9 (s)	780 (m), 760 (m)	
		155.9 (s)	740 (w)	
<b>3b</b>	1.31 (t, 3H)	14.5 (q)	2965 (w), 1710 (s)	180 (9)
	3.44 (s, 3H)	34.1 (q)	1590 (m), 1570 (m)	135 (6)
	4.24 (q, 3H)	62.0 (t)	1475 (m), 1425 (s)	108 (50)
	7.0-8.3 (m, 4H)	119.0 (d)	1380 (s), 1345 (s)	107 (100)
		119.4 (d)	1310 (m), 1275 (m)	80 (29)
		137.1 (d)	1140 (s), 1110 (m)	79 (61)
		147.5 (d)	780 (m), 765 (w)	78 (69)
		155.0 (s)	740 (w)	
		155.4 (s)		
<b>3c</b>	1.30 (d, 6H)	22.1 (q)	2965 (w), 1710 (s)	194 (7)
	3.43 (s, 3H)	34.0 (q)	1590 (m), 1570 (m)	135 (21)
	5.03 (m, 1H)	69.6 (d)	1475 (m), 1425 (s)	108 (100)
	7.0-8.3 (m, 4H)	119.0 (d)	1370 (s), 1275 (m)	107 (74)
		119.3 (d)	1150 (s), 1095 (m)	80 (71)
		136.9 (d)	775 (m), 765 (w)	79 (89)
		147.5 (d)	735 (w)	78 (86)
155.1 (s)				
<b>5a</b>	1.23 (t, 3H)	14.0 (q)	2970 (w), 1735 (s)	222(9)
	3.22 (s, 2H)	37.1 (q)	1665 (s), 1585 (m)	177 (7)
	3.34 (s, 3H)	41.8 (t)	1480 (m), 1420 (s)	149 (9)
	4.13 (q, 2H)	61.1 (t)	1380 (s), 1325 (m)	135 (16)
	7.3-8.5 (m, 4H)	123.9 (d)	1250 (m), 1150 (m)	108 (100)
		134.6 (d)	1110 (m), 1015 (m)	107 (93)
		140.4 (s)	815 (w), 710 (m)	80 (13)
		148.8 (d)		79 (13)
		148.9 (d)		78 (28)
165.5 (s)				
167.3 (3)				
<b>5b</b>	1.23 (t, 3H)	14.1 (q)	2965 (w), 1740 (s)	236 (11)
	1.34 (d, 3H)	38.0 (q)	1665 (s), 1585 (m)	191 (8)
	3.33 (s, 3H)	43.8 (d)	1480 (m), 1420 (s)	135 (13)
	3.72 (q, 1H)	61.2 (t)	1385 (s), 1320 (m)	108 (100)
	4.10 (q, 2H)	124.3 (d)	1260 (m), 1180 (m)	107 (36)
	7.3-8.5 (m, 4H)	135.0 (d)	1110 (m), 1080 (m)	
		140.2 (s)	1015 (m), 815 (w)	
		149.1 (d)	710 (m)	
		149.3 (d)		
		170.0 (s)		
170.3 (s)				

Table 3 (continued)

Product	<sup>1</sup> H NMR δ ppm [a]	<sup>13</sup> C NMR δ ppm [a]	IR cm <sup>-1</sup>	MS (RI) m/e (%)
<b>5c</b>	1.21 (d, 6H)	21.7 (q)	2970 (w), 1730 (s)	236 (71)
	3.21 (s, 2H)	37.7 (q)	1670 (s), 1580 (m)	193 (14)
	3.34 (s, 3H)	41.9 (t)	1480 (m), 1420 (s)	177 (33)
	4.98 (m, 1H)	69.1 (d)	1380 (s), 1320 (m)	149 (14)
	7.3-8.5 (m, 4H)	124.4 (d)	1290 (m), 1090 (s)	135 (57)
		134.8 (d)	965 (m), 815 (w)	108 (100)
		140.3 (s)	710 (m)	107 (99)
		148.9 (d)		80 (16)
		149.1 (d)		79 (24)
		166.0 (s)		78 (55)
		166.8 (s)		
<b>6</b>	1.22 (t, 3H)	14.1 (q)		
	3.20 (s, 2H)	37.4 (q)		
	3.30 (s, 3H)	41.6 (t)		
	4.11 (q, 2H)	61.2 (t)		
	7.0-7.5 (m, 5H)	127.3 (d)		
		128.3 (d)		
		129.9 (d)		
		143.6 (s)		
166.1 (s)				
167.6 (s)				
<b>8a</b>	3.56 (s, 3H)	43.2 (q)	3050 (w), 2970 (w)	223 (52)
	3.72 (s, 6H)	50.9 (q)	2930 (w), 2830 (w)	192 (100)
	6.96 (d, 2H)	93.0 (s)	1670 (s), 1630 (s)	165 (23)
	7.58 (d, 2H)	113.9 (d)	1505 (s), 1440 (s)	162 (29)
		136.7 (d)	1365 (s), 1310 (w)	134 (55)
		150.0 (s)	1420 (s), 1200 (s)	122 (23)
		169.3 (s)	1070 (s), 1040 (s)	107 (35)
			1015 (s), 945 (m)	104 (13)
			895 (s), 840 (s)	93 (28)
			790 (m), 740 (s)	78 (10)
			77 (10)	
<b>8b</b>	1.28 (t, 6H)	14.5 (q)	3060 (w), 2960 (m)	265 (70), 236 (3)
	1.37 (t, 3H)	16.0 (q)	2920 (w), 2890 (w)	220 (100), 193 (50)
	3.75 (q, 2H)	51.4 (t)	1690 (m), 1645 (s)	192 (60), 176 (70)
	4.20 (q, 4H)	59.3 (t)	1515 (s), 1445 (w)	165 (26), 150 (50)
	7.00 (d, 2H)	94.0 (s)	1405 (m), 1385 (m)	148 (86), 136 (13)
	7.49 (d, 2H)	113.7 (d)	1350 (w), 1315 (w)	121 (60), 107 (40)
		135.4 (d)	1230 (s), 1180 (m)	104 (37), 92 (36)
		149.8 (s)	1150 (w), 1070 (m)	91 (34), 80 (26)
		169.0 (s)	1040 (s), 1020 (m)	79 (20), 78 (22)
			950 (m), 840 (m)	77 (17), 65 (37)

Table 3 (continued)

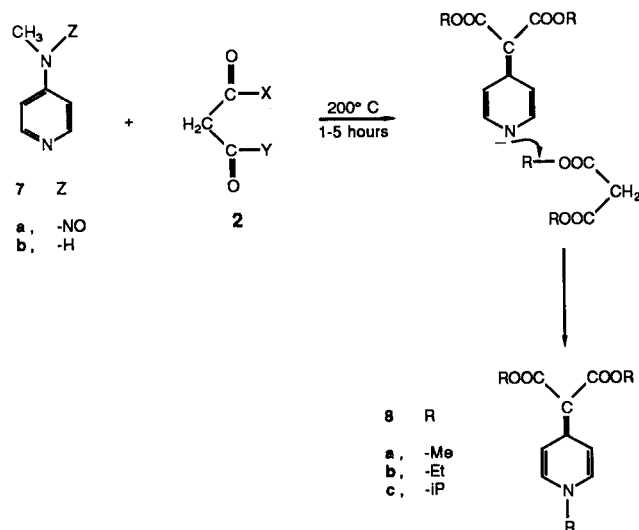
Product	<sup>1</sup> H NMR δ ppm [a]	<sup>13</sup> C NMR δ ppm [a]	IR cm <sup>-1</sup>	MS (RI) m/e (%)
<b>8c</b>	1.26 (d, 12H)	22.1 (q)	2965 (s), 2930 (m)	307 (70)
	1.40 (d, 6H)	22.6 (q)	1660 (s), 1625 (s)	248 (72)
	3.80 (m, 1H)	57.7 (d)	1515 (s), 1465 (w)	221 (62)
	5.10 (m, 2H)	66.2 (d)	1400 (s), 1380 (s)	206 (48)
	7.00 (d, 2H)	95.0 (s)	1320 (w), 1300 (w)	179 (50)
	7.48 (d, 2H)	113.6 (d)	1240 (s), 1180 (s)	164 (40)
		133.4 (d)	1160 (s), 1100 (s)	137 (38)
		149.8 (s)	1060 (m), 1040 (s)	135 (100)
		168.6 (s)	980 (s), 835 (m)	120 (50)
				93 (14)
			92 (20)	
			91 (16)	
<b>9</b>	1.30 (d, 6H)	22.1 (q)	2790 (w), 2930 (w)	
	3.33 (s, 3H)	35.7 (q)	1710 (s), 1590 (s)	
	5.03 (m, 1H)	70.4 (d)	1505 (m), 1375 (s)	
	7.30 (d, 2H)	117.5 (d)	1330 (m), 1295 (m)	
	8.50 (d, 2H)	150.2 (d)	1275 (m), 1240 (m)	
		154.5 (s)	1150 (s), 1100 (s)	
			985 (w), 895 (w)	
			820 (m), 790 (w)	
		765 (w)		

[a] The <sup>1</sup>H and <sup>13</sup>C nmr spectra were obtained in deuteriochloroform.

1 and 2). The alkaline or acid hydrolysis of **5** gave **4b** (Tables 2 and 3). The ir spectra of **5** showed two peaks one at 1735 cm<sup>-1</sup>, which was assigned to the carbonyl of the ester group, and one at 1665 cm<sup>-1</sup> which was assigned to the carbonyl of the amide group [10b]. In the <sup>13</sup>C nmr spectra there are 3 peaks which remain as singlets even under off-resonance decoupling. Two of these singlets between 165-170 ppm are attributed to the two carbonyl groups, whilst the third singlet at about 140 ppm to C-3 of the pyridine nucleus of **5**. Of the two carbonyl peaks, one at the higher field is attributed to the carbonyl carbon of the amide group (Table 3).

The reaction of 4-*N*-nitrosomethylamino- **7a** and 4-methylaminopyridine (**7b**) with **2** (Table 1) lead to the formation of substituted dihydropyridines **8** (Tables 2 and 3) presumably by the attack of **2** on carbon-4 of **7a** and **7b** and the addition of an alkyl group on the ring nitrogen (Scheme 2). Moreover, these reactions, in contrast to those of **2** and **3** substituted pyridines, are accelerated by sodium methoxide. However, although reaction occurred with **2a**, **2b** and **2e** no reaction was observed with **2c**, *i.e.* when a methyl is present on the methylene carbon of **2**. In the reaction **7a** with **2e** a small amount (7%) of isopropyl *N*-methyl-*N*-(4-pyridyl)carbamate (**9**) was obtained. This behaviour is similar to that of **1a**.

Scheme 2

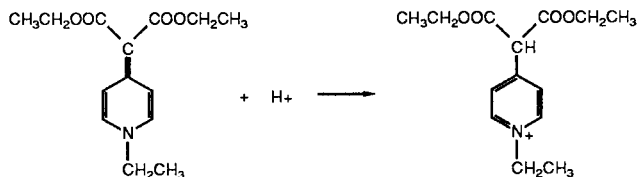


The ir spectra of **8** reveal a strong absorption at 1660-1690 cm<sup>-1</sup> which is attributed to the carbonyl groups and another strong absorption at 1625-1645 cm<sup>-1</sup> which is characteristic of the dihydropyridines [12].

As expected [12] dihydropyridine derivative **8b** was converted to the corresponding pyridine derivative under

acidic conditions by protonating the exocyclic carbon. The  $^1\text{H}$  nmr spectra show a shift of the dihydropyridine hydrogens to the aromatic region and the appearance of a singlet in the aliphatic region which corresponds to the added proton (Scheme 3).

Scheme 3



The results are confirmed by decoupled and off-resonance decoupled  $^{13}\text{C}$  nmr spectroscopy. There is also agreement of the ir spectra of **9** of the carbamates **5c** obtained in the reaction between **1a** and **2e** with the characteristic carbonyl absorption at  $1710\text{ cm}^{-1}$  (Table 3).

The difference in the behaviour between the 2-isomers **1**, which form carbamic esters, and the 4-isomers **7**, which form mainly 4-dihydropyridines, may be rationalized on the grounds that, apart from steric factors which would favour the formation of 4-dihydropyridines **8**, carbon-4 in the 4-isomers seems to have a greater electrophilic character than carbon-2 in the 2-isomers presumably because of its greater separation of charges in the 4-isomers.

It is also noteworthy that no reaction was observed between the aminopyridine derivatives, **1**, **4** and **7**, and ethyl acetoacetate (**2f**) presumably because the methylene group of **2f** is not activated enough to participate in these reactions.

#### EXPERIMENTAL

The nmr, ir and ms spectra were obtained with a Varian FT-80A spectrometer, an SP-1100 Pye Unicam spectrophotometer and Hitachi Perkin Elmer RMU-6L spectrometer respectively. Gas chromatography was carried out on Pye Unicam GCV chromatograph. Elemental analyses were carried out by Dr. Ch. Mantzos (National Hellenic Research Foundation).

#### Materials.

Dimethyl, diethyl malonate and ethyl acetoacetate were purified by distillation under vacuum. 2-Methylamino- [13], 3-methylamino- [14] and 4-methylaminopyridine [15], 2-methylamino-*N*-nitroso- [4], 3-methylamino-*N*-nitroso- [6] and 4-methylamino-*N*-nitrosopyridine [4], and *N*-methyl-*N*-nitrosoaniline [16] were prepared by known methods.

#### Diethyl 2-Methylmalonate [17].

A mixture of 70 g of dimethyl sulfoxide (DMSO) and 5 g of sodium

hydride 55-60% ( $\approx 0.1$  mole) was stirred in a three neck flask equipped with a calcium chloride tube, and 16 g of diethyl malonate added slowly under cooling. After 10 minutes, 16 g of methyl iodide ( $\approx 0.12$  mole) was added dropwise. The slurry mixture was stirred overnight and next day the reaction products were clarified with filtration and the precipitate washed with diethyl ether. The filtrates were washed with water many times to remove DMSO and the organic phase was dried over calcium chloride. Finally under vacuum distillation 16.1 g was obtained consisting (glc) of a mixture of: 76% diethyl 2-methylmalonate (70%), 21% diethyl 2,2-dimethyl malonate, and 3.5% diethyl malonate.

This mixture was used as such because no interference was expected from diethyl 2,2-dimethylmalonate in the relevant reactions whilst diethyl malonate was present in insignificant amounts (Table 1).

#### Diethyl 2,2-Dimethylmalonate [17].

The reaction was carried out as for diethyl 2-methylmalonate with the exception that a second lot of sodium hydride followed with a second lot of methyl iodide were added. Finally under vacuum distillation (1 mm Hg,  $52^\circ$ ) 15.7 g (84%) of diethyl 2,2-dimethylmalonate was obtained which shows by glc only trace amounts of diethyl 2-methylmalonate and unreacted diethyl malonate;  $^1\text{H}$  nmr (deuteriochloroform):  $\delta$  (ppm) 1.02 (t, 6H), 1.16 (s, 6H), 3.95 (q, 4H);  $^{13}\text{C}$  nmr (deuteriochloroform):  $\delta$  (ppm) 14.1 (q), 22.8 (q), 49.9 (s), 172.8 (s).

#### Reaction Procedure.

Reactions were carried out in a screw capped tube at  $200^\circ$  by mixing 10 mmoles of the amine or nitrosamine with 50 mmoles of malonic ester. The reaction was followed at regular intervals by tlc using the appropriate solvent systems (Table 1). The reaction was stopped after the disappearance of the amine or nitrosamine. The reaction mixture was passed through column of silica gel and the fractions collected. The products were obtained in pure form by preparative tlc (Silica gel G) followed by preparative glc on a SE-30 column.

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