lizations from benzene–petroleum ether (b.p. 30–60°) the product melted at 63–64°.

Anal. Calcd. for $C_{10}H_{19}NO$: C, 70.96; H, 11.31; N, 8.28. Found: C, 70.73; H, 11.12; N, 8.39.

The *picrate*, recrystallized from acetone–ether, melted at 125–126°.

Anal. Calcd. for $C_{10}H_{19}NO \cdot C_6H_3N_3O_7$: C, 48.24; H, 5.57; N, 14.07. Found: C, 48.16; H, 5.82; N, 13.79.

3α-(2-Chloroethyl)tropane (Ve). A solution of 39.0 g. (0.23 mole) of 2-(3α-tropanyl)ethanol in 150 ml. of chloroform was saturated with hydrogen chloride. To the mixture with stirring was gradually added 33 ml. of thionyl chloride. The resulting clear solution was heated at reflux for 1 hr. and then evaporated to dryness *in vacuo*. The residual tan solid was dissolved in a minimum volume of water, the solution was made strongly basic with sodium hydroxide, and the alkaline mixture was extracted with four portions of ether. From the ether extracts, dried over potassium carbonate, was obtained 39.5 g. (92%) of chloro amine as a colorless liquid, b.p. 75–80° (0.6–0.8 mm.).

The *hydrochloride*, recrystallized from ethanol–ether, melted at 167–168°.

Anal. Calcd. for $C_{10}H_{19}NCl$: C, 53.57; H, 8.54; N, 6.25. Found: C, 53.73; H, 8.40; N, 6.18.

The *picrate*, recrystallized from water, melted at 159–160°.

Anal. Calcd. for $C_{10}H_{18}NCl \cdot C_6H_3N_3O_7$: C, 46.10; H, 5.08. Found: C, 46.25; H, 4.93.

3-(3α-Tropanyl)propionitrile (Vf). A solution of 47.0 g. (0.25 mole) of 3α-(2-chloroethyl)tropane, 48.5 g. (0.75 mole) of potassium cyanide, and 0.1 g. of sodium iodide in 250 ml. of alcohol–water (3:1) was heated at reflux for 17 hr. The reaction mixture was evaporated *in vacuo*, the residue was dissolved in water, the solution was made strongly alkaline with sodium hydroxide, and the mixture was extracted with several portions of ether. From the dried ether extracts was obtained 38.8 g. (87%) of almost colorless oil, b.p. 114–116° (0.3 mm.), $n^{24}D$ 1.4960.

The *picrate*, recrystallized from acetone–ether, melted at 150–151°.

Anal. Calcd. for $C_{11}H_{18}N_2 \cdot C_6H_3N_3O_7$: C, 50.12; H, 5.20; N, 17.19. Found: C, 49.99; H, 4.96; N, 17.15.

Ethyl 3-(3α-tropanyl)propionate (Vh). A solution of 25 g. (0.14 mole) of nitrile Vf in 100 ml. of concd. hydrochloric acid was heated at reflux for 7 hr. The cooled mixture was filtered to remove ammonium chloride (3.1 g.), and the filtrate was evaporated to dryness. The residue, from which traces of water were removed by azeotropic distillation with benzene, was dissolved in 300 ml. of dry ethanol, 5 ml. of concd. sulfuric acid was added, and the solution was heated at reflux for 6 hr. Upon working up the mixture in the usual way, 25.0 g. (80%) of ester was obtained as a colorless oil, b.p. 97–100° (0.4 mm.), $n^{24}D$ 1.4771.

Anal. Calcd. for $C_{13}H_{23}NO_2$: C, 69.29; H, 10.27; N, 6.22. Found: C, 68.76; H, 10.09; N, 6.13.

The *picrate*, recrystallized from 2-propanol, melted at 108–109°.

Anal. Calcd. for $C_{13}H_{23}NO_2 \cdot C_6H_3N_3O_7$: C, 50.21; H, 5.77. Found: C, 50.21; H, 5.48.

3-(3α-Tropanyl)propionic acid hydrochloride (Vg). A solution of 4.7 g. of ester Vh in 25 ml. of concd. hydrochloric acid was heated at reflux for 3 hr. Upon evaporation of the solution *in vacuo* and recrystallization of the residue from methanol–ether, 3.1 g. of amino acid hydrochloride was obtained as colorless crystals, m.p. 194–195°.

Anal. Calcd. for $C_{11}H_{19}NO_2 \cdot HCl$: C, 56.52; H, 8.63; N, 5.99. Found: C, 56.36; H, 8.25; N, 5.94.

PHILADELPHIA, PA.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, DEFENSE ACADEMY]

Solvent-Catalyzed Michael Reaction of Derivatives of Malonic and Cyanoacetic Acids with Acrylic Acid Derivatives in Liquid Ammonia

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Acrylonitrile or ethyl acrylate can react with ethyl malonate and cyanoacetamide and their monoalkyl derivatives in liquid ammonia to yield the corresponding Michael condensation products without the use of catalysts. This Michael reaction is characterized by a catalytic action of the solvent, presumably *via* formation of the carbanion of the active methylene compound. An attempted reaction with acrylamide was unsuccessful under the same conditions. Nineteen new compounds have been prepared in the course of the present work.

In our previous paper,[1] a new modification of the Michael reaction was reported, wherein derivatives of acetamidomalonic or acetamidocyanoacetic acid were condensed with acrylonitrile, ethyl acrylate, and acrylamide in liquid ammonia without additional catalysts. The reaction was probably due to the basic character of liquid ammonia in contrast with the common organic solvents usually employed.

The present study was concerned with an extension of the new modification to the esters and amides of malonic and cyanoacetic acids and to their monoalkyl derivatives.[2] We have found that acrylonitrile can condense with ethyl malonate, cyanoacetamide, and their monoalkyl derivatives in liquid ammonia to form mono- and dicyanoethylation products in which the hydrogens of the methinyl or methylene groups have reacted. Even with limited amounts of acrylonitrile both ethyl malonate and cyanoacetamide gave exclusively dicyanoethylation products. With the less reactive ethyl acrylate as the acceptor, ethyl malonate formed only a low yield of monocarbethoxyethyla-

(1) K. Shimo and S. Wakamatsu, *J. Org. Chem.*, **26**, 3788 (1961).

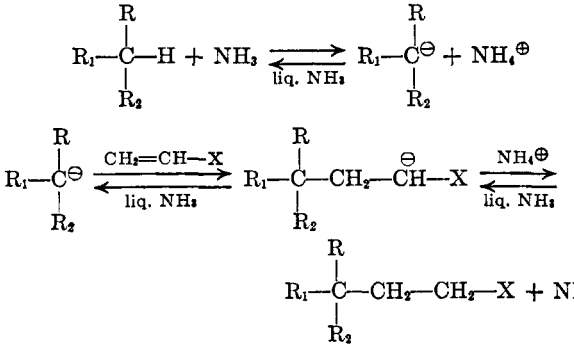
(2) Among these, the ester of cyanoacetic acid and its monoalkyl derivatives were quite unstable in liquid ammonia and were spontaneously converted to the corresponding amides, so they have not been used as starting materials in the present work.

tion product regardless of the relative amounts of the reactants. Monoalkyl malonic esters did not react appreciably with ethyl acrylate under similar conditions. On the other hand, cyanoacetamide and ethyl acrylate even in equimolar proportions gave an excellent yield of dicarbethoxyethylation product without the formation of any mono-derivative. In a similar manner, ethyl acrylate reacted with C-alkyl cyanoacetamides to form the corresponding condensation products.[3] At the end of this reaction 2 - alkyl - 2 - (2 - carbamoylethyl)cyanoacetamide was usually obtained upon treatment of the reaction mixture with ammonium chloride in liquid ammonia.

It was found that isopropyl and *sec*-butyl cyanoacetamide, however, did not undergo carbethoxyethylation under these conditions, probably owing to steric hindrance of the secondary alkyl group attached to the 2-carbon. Malonamide and C-alkyl malonamides failed to condense with either acrylonitrile or ethyl acrylate. Acrylamide did not react with derivatives of malonic and cyanoacetic acids in liquid ammonia, and unexpectedly acrylonitrile did not react with derivatives of nitromalonic acid. We believe the explanation that the methinyl group between the nitro and carbonyl groups forms a stable unreactive ammonium salt.

The results are summarized in Table I.

A suggested mechanism is shown below.



Where R = H, alkyl, or phenyl
R_1, R_2 = CN, $CO_2C_2H_5$, or $CONH_2$
X = CN or $CO_2C_2H_5$

It must be noted that not only the active methylene compound, but also ammonia itself can react with acrylonitrile as reported.[4] There are thus two competitive reactions at the same time. Actually when the cyanoethylation took place very slowly, the major product was bis(2-cyanoethyl)amine.

EXPERIMENTAL[7]

Derivatives of malonic and cyanoacetic acids used as starting materials in this work were prepared by known methods.[8] Acrylonitrile, ethyl acrylate and acrylamide were commercially available. The Michael reaction in liquid ammonia was generally run by two different methods. Method A consisted of treating the reactants with liquid ammonia at room temperature under pressure in a glass pressure vessel.[9] It was advisable to provide a cooling bath of ice water because of an exothermic character of the reaction. Method B consisted of treating the reactants at atmospheric pressure below the boiling point of liquid ammonia (−50°) wherein the acrylonitrile was added dropwise to the stirred solution of the other component in liquid ammonia. These are illustrated in typical examples as follows.

4,4-Dicarbethoxypimelonitrile (*Method B*). To a stirred solution of 16.0 g. (0.1 mole) of ethyl malonate in 150 cc. of liquid ammonia, there was added 10.6 g. (0.1 mole) of acrylonitrile dropwise during the course of 40 min. while cooling to −50°. The mixture was stirred for 2 hr. after all the acrylonitrile was added, and then the ammonia was evaporated. The residual crystalline product was dried in vacuum to give 25.8 g. (97%) of 4,4-dicarbethoxypimelonitrile which melted at 55–62°. Upon crystallization from ethanol, the melting point was raised to 61–63.5°. A quantitative yield (based on acrylonitrile) of the same product was obtained from equimolar quantities of acrylonitrile and ethyl malonate under similar conditions, whereas no monocyanoethylation product was produced.

Ethyl 2-isopropyl-2-(2-cyanoethyl)malonamate (*Method A*). To a mixture of 10.11 g. (0.05 mole) of ethyl 2-isopropylmalonate and 2.65 g. (0.05 mole) of acrylonitrile in a glass pressure vessel was added 50 cc. of liquid ammonia. The clear solution was held at room temperature for 2 hr., then the ammonia was evaporated. The residual oil was fractionated in vacuum to give 4.3 g. (38%) of the product boiling at 174° (2 mm.) which solidified on cooling. After recrystallization from ligroin (b.p. 78–108°)-benzene, the solid melted at 71–73.5°. Unchanged ethyl 2-isopropylmalonate, 3.3 g. (33%) was recovered.

Ethyl 4-cyano-4-carbamoylpimelate (*Method B*). Ethyl acrylate (10.0 g.) (0.1 mole) was added dropwise to a stirred solution of 8.4 g. (0.1 mole) of cyanoacetamide and 150 cc. of liquid ammonia during 20 min. while maintaining the reaction temperature at about −50°. The mixture was stirred for 1 hr. longer to complete the reaction, then the ammonia was evaporated. The remaining solids were washed with water to yield 13.8 g. (97% based on ethyl acrylate) of the product which after recrystallization from dilute ethanol melted at 111–112°.

4-Cyano-4-carbamoylpimelamide (*Method A*). A mixture of 2.1 g. (0.025 mole) of cyanoacetamide, 5.0 g. (0.05 mole) of ethyl acrylate, and 25 cc. of liquid ammonia in a glass pressure vessel was held at room temperature for 80 min. Then 0.1 g. of ammonium chloride was added in order to cause ammonolysis, and after standing for about 24 hr. the ammonia was evaporated. The remaining solids were washed with water to give 3.4 g. (60%) of 4-cyano-4-carbamoyl-

(3) The carbethoxyl group included in each of these substances was relatively readily subjected to ammonolysis in the course of the reaction in liquid ammonia, and so it was sometimes difficult to isolate the products in pure forms. It was therefore advisable to treat the reaction mixtures with ammonium chloride in order to cause ammonolysis and to get the products in the form of the corresponding amide.

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TABLE I

MICHAEL REACTION OF DERIVATIVES OF MALONIC AND CYANOACETIC ACIDS WITH ACRYLIC ACID DERIVATIVES IN LIQUID AMMONIA

Compound	$CH_2=CH-X$[a] X	Method[b]	Product	Yield, %[c]	M.P. or B.P. (Mm.)	Recryst. Solvent		Calcd.	Found
$RCH(CO_2C_2H_5)_2$									
R=H	CN	B	$(C_2H_5O_2C)_2C(CH_2CH_2CN)_2$	99[d]	61–63.5[e]	Ethanol	N	10.52	10.58
H	$CO_2C_2H_5$	B	$(C_2H_5O_2C)_2CHCH_2CH_2CO_2C_2H_5$	29[f]	130(3)[g]		C	55.38	55.15
							H	7.75	7.62
C_2H_5	CN	B	$(C_2H_5O_2C)_2C(C_2H_5)CH_2CH_2CN$	61[h]	47–48[i]	Ethanol	C	59.73	59.74
							H	7.94	7.86
							N	5.81	5.95
i-C_3H_7	CN	A	$C_2H_5O_2C(H_2NOC)C(i\text{-}C_3H_7)CH_2CH_2CN$	38[j]	71–73.5[k]	Ligroin (b.p. 78–108°)–benzene	C	58.38	58.29
							H	8.02	7.80
							N	12.38	12.45
$C_6H_5CH_2$	CN	A	$(C_2H_5O_2C)_2C(CH_2C_6H_5)CH_2CH_2CN$	45[l]	176–178(1)[m]		C	67.31	67.17
							H	6.98	6.93
							N	4.62	4.59
$RCH(CN)CONH_2$									
R=H	CN	B	$H_2NOCC(CN)(CH_2CH_2CN)_2$	94[d,n]	118–119[o]	Water	N	29.46	29.31
H	$CO_2C_2H_5$	B	$H_2NOCC(CN)(CH_2CH_2CO_2C_2H_5)_2$	97[d]	111–112[k]	Dil. ethanol	N	9.86	9.78
H	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(CH_2CH_2CONH_2)_2$	60[q]	209.5[k]	Water	N	24.77	25.25
C_2H_5	CN	A	$H_2NOCC(CN)(C_2H_5)CH_2CH_2CN$	93	111–112.5[k]	Dil. ethanol	N	25.45	25.35
C_2H_5	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(C_2H_5)CH_2CH_2CONH_2$	66	189–191[k]	Water	N	21.65	21.78
n-C_3H_7	CN	A	$H_2NOCC(CN)(n\text{-}C_3H_7)CH_2CH_2CN$	88	86–88[k]	Dil. ethanol	N	23.45	23.50
n-C_3H_7	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(n\text{-}C_3H_7)CH_2CH_2CONH_2$	88	173–174.5[k]	Dil. ethanol	N	21.30	21.17
i-C_3H_7	CN	A	$H_2NOCC(CN)(i\text{-}C_3H_7)CH_2CH_2CN$	82[r]	129–131[k]	Dil. ethanol	N	23.45	23.50
n-C_4H_9	CN	A	$H_2NOCC(CN)(n\text{-}C_4H_9)CH_2CH_2CN$	92	106–107.5[k]	Dil. ethanol	N	21.76	21.76
n-C_4H_9	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(n\text{-}C_4H_9)CH_2CH_2CONH_2$	97	159–160.5[k]	Ethanol	N	19.89	19.97
i-C_4H_9	CN	A	$H_2NOCC(CN)(i\text{-}C_4H_9)CH_2CH_2CN$	85	144.5[k]	Dil. ethanol	N	21.76	21.35
i-C_4H_9	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(i\text{-}C_4H_9)CH_2CH_2CONH_2$	87	151–152[k]	Dil. ethanol	N	19.89	19.76
s-C_4H_9	CN	A	$H_2NOCC(CN)(s\text{-}C_4H_9)CH_2CH_2CN$	62[s]	110–115[k]	Benzene	N	21.76	21.86
C_6H_5	CN	A	$H_2NOCC(CN)(C_6H_5)CH_2CH_2CN$	86	84.5–87[k]	Benzene	N	19.71	19.83
C_6H_5	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(C_6H_5)CH_2CH_2CONH_2$	69	159–160[k]	Ethanol	N	18.16	18.32
$C_6H_5CH_2$	CN	A	$H_2NOCC(CN)(CH_2C_6H_5)CH_2CH_2CN$	Quantitative	133–135[k]	Ethanol	N	18.50	18.12
$C_6H_5CH_2$	$CO_2C_2H_5$	A[p]	$H_2NOCC(CN)(CH_2C_6H_5)CH_2CH_2CONH_2$	74	151.5–153.5[k]	Water	N	17.13	17.04

[a] A molar equivalent per mole of $RCH(CO_2C_2H_5)_2$ or $RCH(CN)CONH_2$ was used unless otherwise stated. [b] See Experimental. [c] Yields based on $RCH(CO_2C_2H_5)_2$ or $RCH(CN)CONH_2$. [d] Based on $CH_2=CHX$. [e] Lit.[5] reported m.p. 62°. [f] Recovered 67% of ethyl malonate. Practically the same yield of ethyl 2-carbethoxyglutarate was obtained from the reaction of two moles of ethyl acrylate and one mole of ethyl malonate. The product was readily converted to 2-carbamoylethylglutaramide[k] in the presence of ammonium chloride in liquid ammonia, m.p. 180.5–181.5°. *Anal.* Calcd. for $C_6H_{11}N_3O_3$: N, 24.27. Found: N, 24.48. [g] Lit.[6] reported b.p. 99–108° (0.5 mm.). [h] When the reaction was carried out with ethyl acrylate under method A (with footnote *p*), a 84% of ethyl ethylmalonamate together with a small amount of ethylmalonamide was recovered. [i] Ref. 5, m.p. 47°. [j] Recovered 33% of ethyl isopropylmalonate. Ethyl 2-isopropyl-2-(2-cyanoethyl)malonamate upon ammonolysis in liquid ammonia gave 2-isopropyl-2-(2-cyanoethyl)malonamide. [k] m.p. 177–178°. *Anal.* Calcd. for $C_9H_{15}N_3O_2$: N, 21.31. Found: N, 21.38. [k] New compound. [l] Recovered 40% of ethyl benzylmalonate. When the reaction was carried out with ethyl acrylate under method A (with footnote *p*), a 84% of benzylmalonamide was recovered. [m] Ref. 5, b.p. 175–180° (1 mm.). [n] With two moles of acrylonitrile in A, the yield was 87%. [o] Ref. 5, m.p. 118°. [p] After the reaction was complete, ammonium chloride was then added to the reaction mixture in order to cause ammonolysis, and the reaction product was isolated as the corresponding amide. [q] Two molar equivalents of ethyl acrylate were used per mole of cyanoacetamide. [r] Recovered 85% of isopropylcyanoacetamide when the reaction was carried out with ethyl acrylate under method A (with footnote *p*). [s] Recovered 75% of *sec*-butylcyanoacetamide when the reaction was carried out with ethyl acrylate under method A (with footnote *p*).

pimelamide which melted at 205°. Upon recrystallization from water the melting point was raised to 209.5°.

2-Benzyl-2-(2-cyanoethyl)cyanoacetamide (Method A). A mixture of 3.48 g. (0.02 mole) of 2-benzylcyanoacetamide and 1.06 g. (0.02 mole) of acrylonitrile in a glass pressure vessel was treated with 30 cc. of liquid ammonia for 2 hr. at room temperature. Then the ammonia was evaporated, and the residual crystalline product was washed with water; yield 4.5 g. (quantitative). The melting point was 133–135° after recrystallization from ethanol.

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YOKOSUKA, JAPAN

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF SOUTHERN CALIFORNIA]

The Synthesis of Some Substituted 5-Bromopentylamine Hydrobromides

RONALD F. BROWN AND GEORGE H. SCHMID

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The preparation of the hydrobromides of 5-bromopentylamine and of 5-bromo-1,1-, 2,2-, 3,3-, and 4,4-dimethylpentylamines is described.

For a continuation of our study of the effects of *gem*-dialkyl substitution on the rates of cyclization of ω-bromoalkylamines,[2] it became necessary to prepare a series of 5-bromopentylamine hydrobromides. This paper reports the synthesis of the hydrobromides of 5-bromopentylamine, 5-bromo-1,1-dimethylpentylamine, 5-bromo-2,2-dimethylpentylamine, 5-bromo-3,3-dimethylpentylamine, and 5-bromo-4,4-dimethylpentylamine.

Of the five compounds listed only the unsubstituted 5-bromopentylamine hydrobromide has been prepared previously. Although Freundlich[3] studied the rates of cyclization of a series of bromoalkylamines, he did not describe in detail the method of preparation nor the physical properties of the 5-bromopentylamine hydrobromide used. Blank[4] seems to have been the first to report this compound in the literature but neither he nor von Braun and Steindorff[5] gave any physical constants. Keimatsu and Takamoto[6] reported the preparation of the amine by the action of phosphorus tribromide in chloroform upon 5-hydroxypentylamine. Our product was synthesized from 1,4-dibromobutane by conversion to 1-bromo-4-phenoxybutane followed by replacement of the remaining bromine by cyanide ion and reduction with lithium aluminum hydride to give 5-phenoxypentylamine. Cleavage of the ether with hydrobromic acid[7] then gave the product in excellent yield. Our salt, upon treatment with alkali, gave piperidine which was characterized in several ways. However, we are at a loss to explain the discrepancy between the properties of our product and those reported by Keimatso and Takamoto. Since they report a b.p. 78–79° (749 mm.) and we found the cyclization to proceed with rapidity at room temperature, it seems strange that the amine would exist long enough to allow distillation to occur as the unchanged amine.

The second compound in our list, 5-bromo-1,1-dimethylpentylamine hydrobromide was obtained by the action of the Grignard reagent from 1-bromo-4-ethoxybutane on acetone followed by the Ritter and Kalisch[8] conversion of the resulting tertiary alcohol to the corresponding amine, 5-ethoxy-1,1-dimethylpentylamine, which underwent ether cleavage smoothly with hydrobromic acid to give the product. On our first attempt to use the Ritter and Kalisch reaction the phenoxy group was present rather than the ethoxy and only a 10% yield was obtained. Since this reaction probably proceeds *via* the carbonium ion from the tertiary alcohol, it seemed likely that the ion was attacking other phenoxy groups in the reaction mixture as well as the hydrogen cyanide which is the normal course for this reaction.[9] This explanation was substantiated by the isolation of a high-boiling viscous oil as the major product, and by an increase in the yield of the amine to 66% when ethoxy was used in place of the phenoxy group in the tertiary alcohol. The final product, 5-bromo-1,1-dimethylpentylamine hydrobromide, gave 2,2-dimethylpiperidine upon cyclization by treatment with alkali.

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(1) This work was supported by a grant from the National Science Foundation. It is based on a dissertation submitted by G. H. Schmid to the Graduate School of the University of Southern California in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

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