

Anal. Calcd. for $C_8H_6O_3$: C, 52.63; H, 5.30. Found: C, 52.25, 52.18; H, 5.31, 5.39.

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Some Substituted Benzalmalononitriles¹

The substituted benzalmalononitriles listed in Table I were prepared essentially by the procedure of Corson and Stoughton.²

Acetylalanine N-*n*-butylamide was prepared from the ethyl ester and *n*-butylamine, m. p. 114–115.2° from nitromethane.

Anal. Calcd. for $C_9H_{13}O_2N_2$: C, 58.04; H, 9.74; N, 15.05. Found: C, 58.10; H, 9.26; N, 14.88.

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N,N-Dicyclohexylformamide

In the course of the preparation of tertiary amines

TABLE I
SUBSTITUTED BENZALMALONONITRILES

Substituents	Reaction solvent ^a	Cryst. solvent ^a	Yield, %	M. p., °C.	Formula	Analyses, ^b %					
						Carbon		Hydrogen			
						Calcd.	Found	Calcd.	Found		
3-Chloro	<i>t</i> -Amyl	<i>n</i> -Butyl	85	116–117	$C_{10}H_5ClN_2$	63.68	63.86	63.79	2.67	2.75	2.73
4-Chloro	<i>i</i> -Propyl	Ethyl	80	162–163	$C_{10}H_5ClN_2$	63.68	63.79	63.69	2.67	2.84	2.76
2-Bromo	<i>n</i> -Butyl	<i>n</i> -Butyl	95	90–90.5	$C_{10}H_5BrN_2$	51.53	51.63	51.62	2.16	2.32	2.15
3-Bromo	<i>t</i> -Amyl	<i>n</i> -Butyl	81	109.5–110	$C_{10}H_5BrN_2$	51.53	51.64	51.62	2.16	2.24	2.23
3-Iodo	<i>t</i> -Amyl	<i>n</i> -Butyl	85	107–108	$C_{10}H_5IN_2$	42.88	43.01	42.93	1.80	1.90	1.91
4-Nitro	Ethyl	Ethyl	41	159–160	$C_{10}H_5N_3O_2$	60.30	60.42	60.30	2.53	2.66	2.68
3-Hydroxy	Ethyl	<i>t</i> -Amyl	59	151.5–153	$C_{10}H_5N_2O$	70.58	70.37	70.50	3.55	3.65	3.64
2-Methyl	<i>i</i> -Propyl	<i>n</i> -Butyl	51	104–106	$C_{11}H_5N_2$	78.55	78.55	78.57	4.79	4.79	4.93
3-Methyl	<i>i</i> -Propyl	<i>n</i> -Butyl	38	133–134	$C_{11}H_5N_2$	78.55	78.52	78.66	4.79	4.87	4.94
2,6-Dichloro	Ethyl	Hexane	56	89–90	$C_{10}H_4Cl_2N_2$	53.84	53.91	53.93	1.81	2.01	1.88
2-Chloro-5-nitro	<i>i</i> -Propyl	Ethyl	78	119–120	$C_{10}H_4ClN_3O_2$	51.41	51.51	51.60	1.73	1.87	1.86
2,4,6-Trichloro-3-hydroxy	<i>i</i> -Propyl	Benzene	70	135–136	$C_{10}H_3Cl_3N_2O$	43.90	44.12	44.05	1.10	1.26	1.26
4-(2-Chloroethyl-mercapto)	Ethyl	<i>t</i> -Amyl	..	87–88	$C_{12}H_9ClN_2S$	57.94	57.84	57.70	3.67	3.73	3.63
4-Methoxymethyl	Ethyl	Ethyl	61	72–73	$C_{12}H_{10}N_2O$	72.71	72.79	72.78	5.10	5.13	5.15
2-Thiophenyl-malononitrile	Ethyl	Ethyl	50	95–96	$C_8H_5SN_2$	59.98	60.06	60.00	2.52	2.61	2.62
1-Naphthylmalono-nitrile	Ethyl	Ethyl	70	170–171.5	$C_{14}H_9N_2$	82.33	82.54	82.66	3.95	4.09	4.15

^a Alcohols except as noted. ^b Microanalyses by Huffman Microanalytical Laboratories, Denver, Colo.

(1) These compounds were prepared for the Office of Scientific Research and Development under Contract OEMsr-136 with Stanford University.

(2) Corson and Stoughton, *THIS JOURNAL*, **50**, 2825 (1928).

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Some Acylamino Acid Esters and Amides

The following previously undescribed esters and amides of butyrylglycine and acetylalanine have been prepared and characterized.

from N,N-dialkyl amides, there was occasion to prepare N,N-dicyclohexylformamide, a white, wax-like compound. To the best of our knowledge, the preparation of this substance has not been reported previously.

Fifty ml. of water and 108.4 g. of 85% formic acid were added to a 500-ml., 3-neck, round-bottom flask equipped with a dropping funnel in one side neck, a water-cooled reflux condenser in the other side neck and a mercury-sealed mechanical agitator in the center neck. The solution was warmed to 50°, when the dropwise addition of 90.5 g. of dicyclohexylamine was begun. After the amine was completely added, the solution was allowed to agitate for fifteen minutes, when it was transferred to a Claisen flask. The water and some of the excess formic acid were removed by distillation at atmospheric pressure, followed

TABLE I
BUTYRYLGLYCINE DERIVATIVES

Compound		Ethyl ester		N- <i>n</i> -Butylamide		Anilide	
Prepn. method		Esterify acid		Ester and amine		Na salt and aniline	
M. or b. p., °C.		B. 136° (5 mm.)		M. 147.5–148.5		M. 158.5–159.5	
Analyses, %	Carbon	Calcd.	55.5		60.0		65.43
		Found	55.23		60.09		65.5
	Hydrogen	Calcd.	8.73		10.06		7.32
		Found	8.58		9.75		7.22
	Nitrogen	Calcd.	8.08		14.0		12.72
		Found	7.95		14.16		12.96