Anal. Calcd. for  $C_6H_6O_3$ : C, 52.63; H, 5.30. Found: C, 52.25, 52.18; H, 5.31, 5.39.

DEPARTMENT OF CHEMISTRY STANFORD UNIVERSITY

STANFORD, CALIF.

C. D. HEATON C. R. Noller

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## Some Substituted Benzalmalononitriles1

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

Acetylalanine N-n-butylamide was prepared from the ethyl ester and n-butylamine, m. p.  $114-115.2^{\circ}$  from nitromethane.

Anal. Calcd. for  $C_9H_{18}O_2N_2$ : C, 58.04; H, 9.74; N, 15.05. Found: C, 58.10; H, 9.26; N, 14.88.

VENABLE CHEMICAL LABORATORY

University of North Carolina Richard H. Wiley Chapel Hill, North Carolina Leonard L. Bennett, Jr.

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## N,N-Dicyclohexylformamide

In the course of the preparation of tertiary amines

TABLE I

SUBSTITUTED BENZALMALONONITRILES											
	Reaction	Cryst.	Yield,	М. р.,		Carbon Analyses,			b %		
Substituents	solvent <sup>a</sup>	solventa	%	M. p., °C.	Formula	Calcd.		und	Calcd.		und
3-Chloro	t-Amyl	n-Butyl	85	116-117	$C_{10}H_5C1N_2$	63.68	63.86	63.79	2.67	2.75	2.73
4-Chloro	i-Propyl	Ethyl	80	162-163	$C_{10}H_5C1N_2$	63.68	63.79	63.69	2.67	2.84	2.76
2-Bromo	n-Butyl	n-Butyl	95	90-90.5	$C_{10}H_5BrN_2$	51.53	51.63	51.62	2.16	2.32	2.15
3-Bromo	t-Amyl	n-Butyl	81	109.5-110	$C_{10}H_5BrN_2$	51.53	51.64	51.62	2.16	2.24	2.23
3-Iodo	t-Amyl	n-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	t-Amyl	59	151.5-153	$C_{10}H_6N_2O$	70.58	70.37	<b>7</b> 0. <b>5</b> 0	3.55	3.65	3.64
2-Methyl	i-Propyl	n-Butyl	51	104-106	$C_{11}H_8N_2$	78.55	78.55	78.57	4.79	4.79	4.93
3-Methyl	i-Propyl	n-Butyl	38	133-134	$C_{11}H_8N_2$	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-Propyl	Ethyl	78	119-120	$C_{10}H_4C1N_3O_2$	51.41	51.51	51.60	1.73	1.87	1.86
2,4,6-Trichloro-3- hydroxy	i-Propyl	Benzene	<b>7</b> 0	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	t-Amyl		87–88	$C_{12}H_9C1N_2S$	57.94	57.84	<b>57</b> . <b>7</b> 0	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-Thiophenal- malononitrile	Ethyl	Ethyl	<b>5</b> 0	95–96	$C_8H_4SN_2$	59.98	60.06	60.00	2.52	2.61	2.62
1-Naphthalmalono- nitrile	Ethyl	Ethyl	<b>7</b> 0	170–171.5	$C_{14}H_8N_2$	82.33	82.54	82.66	3.95	4.09	4.15

<sup>&</sup>lt;sup>a</sup> Alcohols except as noted. <sup>b</sup> Microanalyses by Huffman Microanalytical Laboratories, Denver, Colo.

DEPARTMENT OF CHEMISTRY STANFORD UNIVERSITY STANFORD, CALIFORNIA

H. G. Sturz C. R. Noller

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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.

stance 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

Compound			Ethyl ester	N-n-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			
Analy- ses, { %	Carbon {	Caled.	55.5	<b>60</b> .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		
	$\left\{ egin{array}{l}  ext{Nitrogen}  ight.  ight.  ight.$	Calcd.	<b>8</b> .08	14.0	12.72		
		Found	7.95	14.16	12.96		

BUTYRYLGLYCINE DERIVATIVES

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

<sup>(2)</sup> Corson and Stoughton, This Journal, 50, 2825 (1928).