

CHEMICAL STUDIES ON FUNGICIDES - PART III

Synthesis of α -Bromoamides

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ABSTRACT

Good methods for acylating in ethylene dichloride or in a two-phase system are presented. Forty-six compounds in two series, the α -bromoacetamides and the α -bromopropionamides, have been synthesized and characterized preliminary to fungicidal evaluation. The importance of preparing series of compounds for fungicidal study is pointed out.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

NRL Problem No. C03-06R (ONR request EN1-22/95027 4-8-46).

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CHEMICAL STUDIES ON FUNGICIDES - PART III

SYNTHESIS OF α -BROMOAMIDES

BACKGROUND

The deterioration of materials in tropical environments has been discussed many times. For the purposes of this report, it is necessary to mention only that the chief agent of destruction, the fungus, has not been conquered, and material sent to the tropics is unsuitably protected from fungal ravages.

A principal reason for this situation is lack of knowledge about fungicides. Our knowledge in this field is much poorer than that concerning insecticides and bactericides. A great part of this lack of knowledge is due to a lack of appreciation of the economic damage caused by fungi. Consequently, the work on fungicidal agents has lagged far behind that on insecticides and bactericides.

In recent years, the potentialities of organic compounds have stimulated an interest in fungus control. Unfortunately, most investigators were interested in specific applications only and at first consequently limited their tests, thereby limiting the usefulness of their results to others in this field. This has resulted in duplication of effort for other experiments, and the integration of final results has been inconclusive.

The survey initiated in this Laboratory is an attempt to place all the results in one relative order. For this purpose, a procedure giving uniform results has been adopted. In this method of assay, all factors such as solubility, diffusibility, and volatility have been eliminated as far as possible. A comprehensive survey of commercially available compounds must necessarily be supplemented by compounds available only from synthesis. This synthetic work is extremely important since all types of compounds must be covered in any survey worthy of the effort. Promising leads uncovered from commercially available organics must be carried to their logical conclusions.

Extensive correlation¹ of chemical structure with fungicidal activity has been reported from this Laboratory. More recent work, covered in another report,² has more clearly defined our paths of investigation. The purpose of this report is to record the synthesis of a number of N-alkyl- α -bromoamides, prepared because certain of their aromatic analogs studied here, the α -bromoacetanilides, were extremely toxic to fungi. It has also been noted that

¹ W. E. Weaver and W. M. Whaley, "Chemical studies on fungicides, part I, correlation of structure with fungicidity," NRL Report No. P-2877, July 1946.

² J. M. Leonard and W. E. Weaver, "Fungus inhibitive properties of organic compounds, part I, hydrocarbons and their halides," NRL Report No. C-3289, May 27, 1948.

haloacylamides are claimed as seed disinfectants.³ Since the unhalogenated amides and mono-halogenated paraffins investigated were non-toxic, the fungicidal activity of α -bromoamides may be attributed to the halogen, which is particularly reactive. This is especially true of the bromoacetamides, as contrasted with the bromopropionamides. For purposes of clarity, the properties of the α -bromoacetamides will be considered separately from those of the α -bromopropionamides.

α -BROMOACETAMIDES

There is a surprising lack of information in the literature concerning these compounds. N-Ethyl- α -bromoacetamide has been reported as a product of hydrolysis from the corresponding imide chloride.⁴ The N,N-diethyl compound was obtained by Miller and Johnson⁵ in 20% yield by treating diethylamine in aqueous alkaline solution with bromoacetyl bromide. The N-alkyl derivative was synthesized by Bergmann and co-workers⁶ in 85% yield using the method Harries⁷ gave for the chloro derivative. The rest of the α -bromoacetamides reported here are new.

Analytical results and physical properties of all the compounds prepared are given in Table I. All the monosubstituted amides were low-melting solids, while the disubstituted compounds were liquids as anticipated from well-known theories of association.⁸ The properties of the bromoacetamides do not render them unsuitable for many fungicidal purposes. Extensive physiological tests would necessarily have to be carried out to determine their limit of usefulness. The lower liquid amides were somewhat lachrymatory and sternutatory, though not as markedly as the corresponding bromo esters. Some of the higher members of this series exhibited these properties on heating, but it was not necessary to work in hoods and the compounds were only mildly irritating to the skin. The di-isopropyl and the di-n-butyl compounds were yellow, indicating traces of impurity, since all the other liquid compounds were water-white and remained so for several months. The analytical results indicate that several products were difficult to purify despite repeated distillations and alternate methods of preparation. There seems to be no explanation for this difficulty, except that some secondary amide formation might have occurred. An attempt was made to overcome this deficiency by using a different method of synthesis. The low bromine analyses for these compounds, however, seem to rule out secondary amide formation. Except for the di-n-butyl and the n-heptyl derivatives whose nitrogen analyses were satisfactory, the molar refractivity measurements indicated a good degree of purity.

The standard practice of conducting acylations with acid halides in ether⁹ was discarded in favor of the much superior ethylene dichloride. As pointed out by Wacker¹⁰ and by

³ Maier-Bode, German Patent 695,907.

⁴ J. v. Braun, F. Jostes and W. Münch, Ann., **453**, 113 (1927).

⁵ L. L. Miller and J. R. Johnson, J. Org. Chem., **1**, 135-140 (1936).

⁶ M. Bergmann, F. Dreyer and F. Radt, Ber., **54B**, 2139 (1921).

⁷ C. Harries and I. Petersen, ibid., **43**, 634-639 (1910).

⁸ A. Hantzsch, ibid., **64**, 661-667 (1931); A. M. Buswell, W. H. Rodebush and M. F. Roy, J. Am. Chem. Soc., **60**, 2239-2244 (1938).

⁹ W. A. Jacobs, M. Heidelberger and I. P. Rolf, ibid., **41**, 458-74 (1919); W. M. Dehn, ibid., **34**, 1399-1409 (1912).

¹⁰ Wacker, Chem. Ztg., **45**, 266 (1920).

TABLE I
 α -BROMOACETAMIDES, $\text{BrCH}_2\text{C}(\text{N}^{\text{O}}\text{R})\text{N}^{\text{R}'}\text{R}^{\text{R}}$

R	R'	Method	% Yield	B.p. ^a		M.p. ^a °C.	^{20}b n _D	^{20}b d ₄	MR		Formula	Nitrogen, %	
				°C.	mm.Hg				Calcd.	Obs.		Calcd.	Found
CH ₃ -	H-	Ia	61	0.6	44-45						C ₃ H ₆ BrNO	9.22	9.07
		IIb	122	20									
CH ₃ -	CH ₃ -	IIa	23	63-65	liq.	1.5097	1.5412	32.20	32.20		C ₄ H ₈ BrNO	8.44	8.35
		IIIb	32	115-116									
C ₂ H ₅ -	H-	IIa	82	72-77	47 ^c						C ₄ H ₈ BrNO	8.44	8.35
		III	33	119-121									
C ₂ H ₅ -	C ₂ H ₅ -	IIb	67	82	liq. ^d	1.4963	1.3745	41.44	41.25		C ₆ H ₁₂ BrNO	7.22	6.98
n-C ₃ H ₇ -	H-	Ia	75	70	ca.20	1.4978	1.4455	36.82	36.49		C ₅ H ₁₀ BrNO	7.78	7.09
		Ie	45	73-75									7.40
n-C ₃ H ₇ -	n-C ₃ H ₇ -	Ia	90	98	liq.	1.4880	1.2686	50.68	50.42		C ₆ H ₁₂ BrNO	6.31	6.26
i-C ₃ H ₇ -	H-	Ib	86	70-72	66-67						C ₅ H ₁₀ BrNO	7.78	7.72
i-C ₃ H ₇ -	i-C ₃ H ₇ -	Ib	59	72-73	liq.	1.4855	1.2651	50.68	50.34		C ₆ H ₁₂ BrNO	6.31	6.44
CH ₂ =CH-CH ₂ -	H-	Ib	76	90	ca.25 ^e						C ₅ H ₈ BrNO	7.87	7.35
		Ie	41	87									8.28
n-C ₄ H ₉ -	H-	Ia	77	76	30-33						C ₆ H ₁₂ BrNO	7.22	6.97
		Ic	28	95									
n-C ₄ H ₉ -	n-C ₄ H ₉ -	Ia	90	101-102	liq.	1.4840	1.2084	59.92	59.19		C ₁₀ H ₂₀ BrNO	5.60	5.59
		Id	62	102-105									
i-C ₄ H ₉ -	H-	Ia	83	99	37-40						C ₆ H ₁₂ BrNO	7.22	7.03
i-C ₄ H ₉ -	i-C ₄ H ₉ -	Ia	84	100	liq.	1.4835	1.2016	59.92	59.47		C ₁₀ H ₂₀ BrNO	5.60	5.64
CH ₃ CH ₂ CH(CH ₃)-	H-	Ia	85	83	53-54						C ₆ H ₁₂ BrNO	7.22	6.74
		Ie	38	75-76									6.82
CH ₃ CH ₂ CH(CH ₃)-	CH ₃ CH ₂ CH(CH ₃)-	Ia	62	95	liq.	1.4873	1.2073	59.92	59.63		C ₁₀ H ₂₀ BrNO	5.60	5.60
n-C ₅ H ₁₁ -	H-	Ia	82	107	ca.25	1.4900	1.3108	46.06	46.01		C ₇ H ₁₄ BrNO	6.73	6.47
n-C ₅ H ₁₁ -	n-C ₅ H ₁₁ -	Ia	83	113	liq.	1.4783	1.1091	69.16	71.05		C ₁₂ H ₂₄ BrNO	5.04	4.76
CH ₃ CH ₂ CH ₂ CH(CH ₃)-	H-	Ia	87	100	ca.30	1.4878	1.3019	46.06	46.04		C ₇ H ₁₄ BrNO	6.73	6.43
n-C ₆ H ₁₃ -	H-	Ia	83	99	ca.25	1.4875	1.2617	50.48	50.63		C ₈ H ₁₆ BrNO	6.31	6.46
CH ₃ CH ₂ CH(CH ₂ H ₅)CH ₂ -	H-	Ia	83	95	liq.	1.4930	1.2801	50.48	50.43		C ₈ H ₁₆ BrNO	6.31	5.99
n-C ₇ H ₁₅ -	H-	Ia	81	110	ca.20	1.4870	1.2357	55.30	54.93		C ₉ H ₁₈ BrNO	5.93	5.71
n-C ₈ H ₁₇ -	H-	Ia	80	119-120	ca.26						C ₁₀ H ₂₀ BrNO	5.60	5.52
n-C ₁₀ H ₂₁ -	H-	Ia	79	130	38-40						C ₁₂ H ₂₄ BrNO	5.04	5.17

^aAll temperatures are uncorrected. ^bSome of these values were obtained on supercooled liquids. Cyon Braun, Jostes and Munch⁴ reported n.p. 49°. dMiller and Johnson⁵ reported b.p. 114-17°/9 mm. eBergmann, Dreyer and Radt⁶ reported b.p. 85°/0.3-0.4 mm. and noted compound was solid at room temperature but melted on warming in the hand. fCalcd.: Br, 44.39. Found: Br, 43.71, 44.10. gCalcd.: Br, 44.69. Found: 44.10, 43.85. hCalcd.: Br, 41.18. Found: 41.32, 41.33. iMicro-analyses by Arlington Laboratories, Fairfax, Virginia.

Seymour¹¹, the latter product can be used without drying, is easily removed at reduced pressures without foaming, can be recovered for re-use, is inexpensive, apparently affords excellent yields, is relatively non-flammable, and does not form explosive peroxides.

α -BROMOPROPIONAMIDES

Like the α -bromoacetamides, there is little information in the literature concerning the α -bromopropionamides. N-Ethyl- α -bromopropionamide¹², N,N-diethyl- α -bromopropionamide¹³ and N,N-dimethyl- α -bromopropionamide¹⁴ were prepared by treating the appropriate amine dissolved in ether with the bromo acid bromide or chloride. As may be seen from Table II, the constants given by Freudenberg and Markert for the dimethyl derivative differ markedly from those found in the present work. But identity of their product was not supported by analytical data, whereas the dimethyl compound prepared in this Laboratory checked satisfactorily both nitrogen analysis and molecular refractivity.

Physical properties and analyses of all the propionamides prepared are given in Table II. The monosubstituted amides of lowest and highest molecular weight were solids, while the intermediate compounds melted below room temperature. All the disubstituted amides were liquid. The first members of the series were only slightly lachrymatory, less so than the corresponding bromoacetamides. No skin irritation was observed with any of the bromopropionamides. All were perfectly colorless when pure, and less difficulty was experienced in purifying these derivatives than was encountered in the bromoacetamide series.

Some difficulty was experienced in distilling certain primary amine derivatives possessing high melting points. For example, with N-isopropyl- α -bromoacetamide, the solvent was removed and the residue recrystallized from water and ethanol. As a rule, the melting points for the propionamides were higher than for the corresponding acetamides.

It is noteworthy that in the propionamides series the yields obtained from amine hydrochlorides were comparable to those obtained from the free amine in anhydrous medium. This was not evident in the work on α -bromoacetamides, though otherwise the results were very similar.

EXPERIMENTAL

The bromoacetyl bromide, α -bromopropionyl bromide, diethylamine, di-n-butylamine, allylamine, and the amine hydrochlorides were obtained from Eastman Kodak Co.; the isopropylamine from Commercial Solvents Corporation, and the ethylene dichloride from Carbide and Carbon Chemicals Corporation. The other amines used were supplied by Sharples Chemicals, Inc. None of the reagents was purified before use.

¹¹ K. M. Seymour, J. Chem. Ed., **16**, 285-287 (1939).

¹² J. v. Braun, F. Jostes and A. Heymons, Ber., **60B**, 92-102 (1927).

¹³ K. Freudenberg and L. Markert, ibid., **60B**, 2247-58 (1927).

¹⁴ Phou-TiSouo and Lin Tchou, Bull. faculté sci. univ. franco-chinoise Peiping, **No. 5**, 13-22 (1935); C. A.; **30**, 4465 (1936).

Method Ia. In a 500-cc, three-necked round-bottomed flask equipped with a pentane thermometer, a dropping funnel, and a mechanical stirrer, were placed 150 cc of ethylene dichloride and 0.2 mole of the amine. This solution was cooled to and maintained at -10°C by a dry ice-ethanol bath while 0.1 mole of bromoacetyl bromide in 25 cc of ethylene dichloride was added during a period of 5 to 10 minutes. After stirring for five minutes longer, the mixture was filtered with suction to remove the precipitated amine hydrobromide. The filtrate was washed first with several 25-cc portions of very dilute hydrochloric acid, finally with water, and then dried over anhydrous magnesium sulfate. After removal of the solvent by vacuum distillation (water-pump), the product was distilled at pressures obtainable with a mechanical pump.

Method Ib. This method was the same as Ia except that the filtrate was not washed because the lower members of the series were water soluble. Fortunately, the amine hydrobromides of the lower molecular weight amines are not very soluble in ethylene dichloride, making this washing unnecessary.

Method Ic. This method was identical with Ia, except that absolute ether was substituted for ethylene dichloride. Commercial ether, when used as the solvent, contained sufficient ethanol to yield appreciable quantities of ethyl α -bromoacetate.

Method Id. A solution of 0.2 mole of amine in 120 cc of benzene was shaken in an ice bath while 0.1 mole of bromoacetyl bromide in 25 cc of benzene was gradually added. The next day the crystals of amine hydrobromide were filtered off, the filtrate dried and distilled. Methods Ia and Ib are definitely superior to Ic and Id, the latter two methods giving much lower yields. Moreover, ether and benzene are inflammable solvents which necessarily must be handled with care.

Method Ie. This method differed from Method Ia only in that three equivalents of amine were used instead of two. This procedure was used for three of the compounds that gave low nitrogen analyses. The use of three equivalents of amine was attempted in an effort to reduce secondary amide formation, which might have accounted for the low nitrogen values.

Method IIa. For the preparation of amides from amine hydrochlorides, an adaptation of the method of McKie¹⁵ was used. In the reaction flask were placed 150 cc of ethylene dichloride, 50 cc of 40% (W/V) sodium hydroxide solution, and 0.2 mole of amine hydrochloride. After cooling to -10°C , 0.2 mole of bromoacetyl bromide in 25 cc of ethylene dichloride was added dropwise. The mixture was filtered, the layers separated, and the ethylene dichloride layer dried. The product was then distilled after removal of the ethylene dichloride. This two-phase acylation is extremely useful but has not been widely exploited.

Method IIb. This method differed from IIa only in that 20% (W/V) sodium hydroxide solution was used. Yields by this procedure were definitely inferior to those obtained by IIa.

Method III. The 500-cc flask containing 150 cc of chloroform, 0.4 mole of pyridine, and 0.2 mole of amine hydrochloride was cooled to -5°C , and 0.2 mole of bromoacetyl bromide was added during an hour while the temperature was kept below 0°C . The mixture was stirred for half an hour longer. The light-yellow reaction mixture contained no separated hydrobromides. After washing consecutively with approximately 10% hydrochloric acid and water, the solution was dried and distilled. Compared to Method IIa, the yield from this procedure was so poor that it was not used except for one derivative.

¹⁵ P. V. McKie, *J. Chem. Soc.*, 123, 2213-7 (1923).

TABLE II
 α -BROMOPROPIONAMIDES, $\text{CH}_3\text{CHBrC-N}^{\text{O,R}}\text{-R}'$

R	R'	Method	Yield, %	B.p. ^a		M.p. ^a °C.	n _D ²⁰	d ₄ ²⁰	[M] ^R		Formula	Nitrogen, % ^e		
				°C.	mm. Hg				Calcd.	Obs.		Calcd.	Found	
CH ₃ -	H-	Ila	89	80-81	2	40					C ₄ H ₈ BrNO	8.44	8.51	8.21
CH ₃	CH ₃ -	Ila	85	75	3	liq. ^b	1.4979	1.4284	36.82	36.99	C ₅ H ₁₀ BrNO	7.78	8.12	7.99
		Ib	83	77	3									
C ₂ H ₅ -	H-	Ila	81	82	2	62 ^c					C ₆ H ₁₀ BrNO	7.78	7.61	7.71
		Ib	89	82	2									
C ₂ H ₅ -	C ₂ H ₅ -	Ib	79	84	1.6	liq. ^d	1.4862	1.2947	46.06	46.16	C ₇ H ₁₄ BrNO	6.73	6.94	6.80
n-C ₃ H ₇ -	H-	Ib	85	81	0.45	33					C ₈ H ₁₂ BrNO	7.22	6.87	6.98
n-C ₃ H ₇ -	n-C ₃ H ₇ -	Ib	87	86	0.31	liq.	1.4830	1.2218	55.30	55.20	C ₉ H ₁₈ BrNO	5.93	6.13	6.08
i-C ₃ H ₇ -	H-	Ib	98	Sublimes		115-117					C ₈ H ₁₂ BrNO	7.22	7.25	7.20
i-C ₃ H ₇ -	i-C ₃ H ₇ -	Ia	74	78-80	0.25	liq.	1.4820	1.2356	55.30	55.49	C ₉ H ₁₈ BrNO	5.93	5.75	5.58
CH ₂ =CH-CH ₂ -	H-	Ia	83	84-85	0.30	37-38					C ₈ H ₁₀ BrNO	7.29	7.25	7.18
n-C ₄ H ₉ -	H-	Ia	79	88	0.37	liq.	1.4850	1.2959	46.06	46.03	C ₇ H ₁₄ BrNO	6.73	6.45	6.80
n-C ₄ H ₉ -	n-C ₄ H ₉ -	Ia	80	106	0.23	liq.	1.4792	1.1605	64.54	64.58	C ₁₁ H ₂₂ BrNO	5.32	5.20	5.25
i-C ₄ H ₉ -	H-	Ia	84	88	0.35	67					C ₇ H ₁₄ BrNO	6.73	6.85	6.62
i-C ₄ H ₉ -	i-C ₄ H ₉ -	Ia	83	102	0.35	liq.	1.4790	1.1591	64.54	64.64	C ₁₁ H ₂₂ BrNO	5.32	5.36	5.59
CH ₃ CH ₂ CH(CH ₃)-	H-	Ia	72	Sublimes		83					C ₇ H ₁₄ BrNO	6.73	6.56	6.51
CH ₃ CH ₂ CH(CH ₃)-	CH ₃ CH ₂ CH(CH ₃)-	Ia	76	91	0.50	liq.	1.4841	1.1981	64.54	63.06	C ₁₁ H ₂₂ BrNO	5.32	5.24	5.21
n-C ₅ H ₁₁ -	H-	Ia	81	105	0.45	liq.	1.4840	1.2503	50.66	50.83	C ₈ H ₁₆ BrNO	6.31	6.40	6.36
n-C ₅ H ₁₁ -	n-C ₅ H ₁₁ -	Ia	61	124-125	0.25	liq.	1.4778	1.1157	73.78	74.12	C ₁₃ H ₂₆ BrNO	4.79	4.89	4.98
CH ₃ CH ₂ CH ₂ CH(CH ₃)-	H-	Ia	94	Sublimes		63					C ₈ H ₁₆ BrNO	6.31	6.15	6.35
n-C ₆ H ₁₃ -	H-	Ia	87	108-110	0.25	liq.	1.4820	1.2105	55.30	55.62	C ₉ H ₁₈ BrNO	5.93	6.01	5.99
CH ₃ CH ₂ CH(C ₂ H ₅)CH ₂ -	H-	Ia	82	101-102	0.25	liq.	1.4862	1.2285	55.30	55.21	C ₉ H ₁₈ BrNO	5.93	6.14	5.95
n-C ₇ H ₁₅ -	H-	Ia	77	114-115	0.35	ca.20	1.4807	1.1874	59.92	59.93	C ₁₀ H ₂₀ BrNO	5.60	6.01	5.84
n-C ₈ H ₁₇ -	H-	Ia	77	121-122	0.25	42-43					C ₁₁ H ₂₂ BrNO	5.32	5.53	5.35
n-C ₁₀ H ₂₁ -	H-	Ia	72	126-128	0.06	33-34					C ₁₃ H ₂₆ BrNO	4.79	4.92	5.01

^aAll temperatures are uncorrected. ^bFreudenberg and Markert¹³ reported b.p. 44°/2 mm. von Braun, Jostes and Heymons¹² reported m.p. 60° and b.p. 114-115°/16 mm. ^cFreudenberg and Markert¹³ reported b.p. 87°/2 mm., Souo and Tchou⁴ reported b.p. 119-121°/15 mm. ^eMicro-analyses by Arlington Laboratories, Fairfax, Virginia.

SUMMARY AND CONCLUSIONS

A good method of conducting acylations in ethylene dichloride and the advantages obtained by the use of this solvent have been discussed.

Similarly, a general procedure using two-phase acylation for water-soluble materials has been presented.

The synthesis and physical properties of forty-six α -bromoacetamides and α -bromopropionamides prepared in this Laboratory have been described.

While the mycological findings are being presented separately, it is definite that the bromoacetamides are much more fungicidal than the bromopropionamides. The importance of continued preparation and evaluation of series of compounds must not be underestimated. Such correlations as are to be found within these groups of compounds will contribute to our understanding of fungicidal activity. On the basis of previous work, additional series of compounds are being prepared, preliminary to their evaluation.
