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Monomeric N-Hydroxyethylated Amides

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Oxyethylated amides that are good wetting agents have been prepared as mixtures by allowing amides to react with ethylene oxide under base catalysis. Individual components of such mixtures can be synthesized by allowing 1-alkylaziridines to react with p-toluenesulfonic acid in monomethylethers of di-, tri- and tetraethylene glycol used as solvents. The aminoethers are then acylated, and the methylether group is removed with trimethylsilyliodide from the resulting amide ethers. The sequence should allow the synthesis of specific wetting agents of quite varied structure (alkyl, aryl groups) with an option for isotopic labeling for more detailed analysis of the wetting properties.

An authoritative review of the status of animal fat feedstocks for oleochemicals (1) served to underline the continuing interest in finding additional uses for these fats and related chemicals. One area of potential use of fatty acid derivatives, for example, is in providing chemicals that have the potential to modify the properties of soil. Soil modification by materials that act to increase watershed, i.e. that impart hydrophobic character to the soil, has been reviewed recently (2). An alternative approach to creating a new market for oleochemicals has involved the preparation and study of certain classes of nonionic wetting agents intended to increase water retentiveness of soil. In particular, Micich and Linfield have described the properties of oxyethylated benzenesulfonamides (3) and secondary amides (4) that are derived from n-alkylamines and fatty acids (Fig. 1). A subsequent study by these authors showed that polydisperse E.O. adducts of hydroxyethyl tertiary amides are outstanding wetting agents for cotton skeins and soil (5).

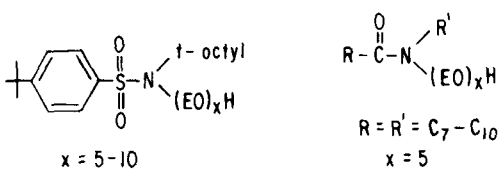

FIG. 1. Structure of fatty amides that have good wetting properties (3-5).

The conversion of a sulfonamide (or carboxamide) to a material with wetting properties has been accomplished by means of a base-catalyzed reaction with ethylene oxide that permitted rapid screening for activity-structure relationships using a fairly broad spectrum of substituents. Optimum wetting properties for benzenesulfonamides were associated with 5–10 ethylenoxy (EO) units, and for secondary amides with about 5 EO units with aliphatic alkyl groups of intermediate (ca. 8) chain length. Apparently one may expect that hydrophilic character will be bestowed on an otherwise hydrocarbon-like material if a suitable number of EO units can be fixed to the central portion of that organic compound. The ability to generate such classes of compounds, which might also effectively tap the reservoir of oleochemicals, seems firmly established. However, the optimum EO count for a particular parent molecule, as well as the details of binding to the soil and to water, remain a matter of conjecture.

A synthetic procedure that allows one to generate selected hydrophilic materials as homogeneous compounds seemed highly desirable at this juncture. In a thorough study of surface and interfacial tension of normal distribution versus homogeneous p-t-octylphenoxy-ethanols (6), similar values of measured physical properties were obtained. This implied that screening the more easily synthesized polymeric mixtures in that series could serve as a useful guide in the search for materials with desirable surfactant properties. The physical properties of the polymeric material and the homogeneous (average) counterpart were not identical, and the disparity between these became greater with increasing average EO chain length. In addition, the degree of divergence may be expected to bear a relationship to the nature of the parent molecule to which the chain is affixed. Thus, examination of a sequence of homogeneous hydrophilic candidates would provide a firmer basis for structure versus activity assessments where a finer tuning was deemed important. Secondly, the studies of oxyethylated amides were troubled in some degree by the presence of byproducts, namely aminoesters derived from N-to-O acyl migra-

tion and unalkylated amide, that added some ambiguity to the measurements of structure versus activity. Finally, the availability of a general route to pure compounds would stimulate more detailed studies of the dynamics of binding to the soil and water by these compounds. Homogeneity should simplify the interpretations of results from such studies, and a specific synthetic route allows selective introduction of isotopes for further studies.

EXPERIMENTAL PROCEDURES

Materials and apparatus. All solvents were reagent grade or better; monomethyl ethers of ethylene, diethylene and triethylene glycol were obtained from Aldrich Chemical Co., Union Carbide Co. and Fluka AG, respectively, and were dried over 4A molecular sieves. Melting points were obtained on a Fisher-Johns hot stage apparatus. Infrared (IR) data were obtained with a Perkin Elmer 13 spectrophotometer with 3% solutions in CCl_4. Thin layer chromatography was performed with Analtech silica gel G plates (25μ) that were not activated before used and with the developing solvents indicated. The Rf values are tabulated in Table 1.

A Hewlett-Packard (Palo Alto, California) HP-5995 GC/MS system was used to obtain electron impact (EI) spectra. The system included HP-9825, a desk-top computer, with two HP-9885 flexible disc drives controlled by HP-5995 flexible software. The unit was operated with autotune parameters obtained using perfluoro-tri-n-butylamine as a reference and the default values for the mass spectrometer temperature zones. Samples were introduced via the direct insertion probe (DIP), which was temperature programmed from 90 C to 290 C at 16 C/min. ^{13}C NMR data were obtained with Jeol GX-400 spectrometer using $CDCl_3$ as a solvent and are reported in Table 2.

Synthesis of 1-octylaziridine (1). N-octylethanolamine (17.3 g, 0.10 mol) (4) (Fig. 3) was converted to 1 by the general method of Wenker (11), whereby the sulfuric acid salt of the aminoalcohol was heated to produce the amine-sulfate ester. This was then decomposed with alkali to the aziridine 1, 10.4 g (67%): bp 88-92 C (30 mm); IR (CCl_4) 3050 cm^{-1}; EIMS (m/e) 310 (the dimeric piperazine)$^+$, 142 ($CH_2 = NHC_8H_{17}$)$^+$.

Synthesis of the 1-octylaminoethers (2). The following is representative: p-toluenesulfonic acid hydrate (4.8 g, 25 mmol) was heated in benzene for 4 hr to remove water (Deane-Starke trap). The solvent was removed with a rotary evaporator, and 2-methoxyethanol (ca. 25 ml) was added to dissolve the acid. The solution was protected from atmospheric moisture and warmed to 60-70 C with stirring. The aziridine 1 (3.1 g, 20 mmol) was added drop by drop (0.25 hr). The resulting mixture was stirred at this temperature overnight. The product was obtained by dilution of the mixture with aqueous NaOH and extraction of the amino ether 2 with diethylether. The organic phase was washed with water, dried ($MgSO_4$) and concentrated to remove the solvent. Distillation afforded 3.01 g (65%) of 2 $(C_8H_{17})NH(CH_2CH_2O)_2CH_3$: bp 94-97 C (0.3 mm); IR (CCl_4) absence of OH, broad 1110 cm^{-1}; EIMS (m/e) 231 (M)$^+$, 200 (M-31)$^+$ (loss of OCH_3), 142 ($CH_2 = NHC_8H_{17}$)$^+$. Similarly were obtained 2 (C_8H_{17}) NH $(CH_2CH_2O)_3CH_3$: bp 130-133 C (0.4 mm); C_8H_{17}) NH $(CH_2CH_2)_4CH_3$: bp 152-160 C (0.4 mm); $(C_8H_{17})NH(CH_2CH_2O)_5CH_3$: bp 184-194 C (0.6 mm). The IR spectra were essentially the same, and the diagnostic EIMS fragments were analogous.

Synthesis of the 1-octyl-1-octanoylaminoethers (3). The following is representative: aminoether 2, $(C_8H_{17})NH(CH_2CH_2O)_2CH_3$, (2.75 g, 11.9 mmol) was dissolved in CH_2Cl_2 (40 ml) containing Et_3N (2.0 ml, 14.3 mmol). The solution was cooled (0-5 C) and stirred while a solution of octanoyl chloride (2.3 ml, 13.1 mmol) in CH_2Cl_2 (10 ml) was added drop by drop. The resulting mixture was stirred at room temperature for 2 hr and was then worked up by extraction into ether, and worked up thereafter in the usual manner of a typical organic chemical reaction. The crude product was chromatographed on silica gel (100 g) with $CHCl_3$. The product 3 bearing 2 EO units eluted in a volume of 200-600 ml as judged by

TABLE 1

Thin Layer Chromatograph R_f Values

(EO)$_x$	2 (Aminoethers)[a]	3 (Amidoethers)[b]	4 (Amidoalcohols)[c]
1	—	—	0.72
2	0.46	0.38	0.68
3	0.37	0.26	0.64
4	0.32	0.18	0.59
5	0.23	0.10	0.52

[a]10:1 $CHCl_3$-methanol + 0.5% NH_4OH.
[b]7:3 Ethyl acetate - hexane.
[c]Wet methyl ethyl ketone.

TABLE 2

^{13}C NMR Data

Compound[a]	a	b	c	d	e	f	g	h	i	OCH_3	Other
2, x = 1	14.09	22.68	31.88	(27.43	29.30	29.58	30.24)	(49.41	50.09)	59.02	CH_2O's 70.72
2, x = 2	14.10	22.68	31.86	(27.43	29.30	29.58	30.24)	(49.41	50.08)	59.03	CH_2O's 70.72
2, x = 3	14.09	22.66	31.86	(27.43	29.28	29.57	30.16)	(49.35	50.05)	59.00	CH_2O's 70.72
2, x = 4	14.04	22.62	31.82	(27.43	29.24	29.52	30.16)	(49.35	50.01)	58.94	CH_2O's 70.72
$(C_8H_{17})NCH_2CH_2OH$	14.10	22.68	31.88	(27.35	29.31	29.55	30.22)	(49.64	51.21)	CH_2OH 60.90	
$(C_8H_{17})N$	14.12	22.72	31.92	(27.54	29.34	29.69	29.92)	62.22	27.20		

[a]^{13}C NMR spectra were essentially consistent with assigned structures. Compounds 3 and 4 (Fig. 3) exhibited a signal at ca. 173 as expected. The carbonyl carbon signal of 4- (single EO unit) was at 175.6 ppm, consistent with its intramolecularly H-bonded structure.

monitoring 100 ml fractions by TLC. The yield of 3 so obtained was 80%. A sample was distilled bulb-to-bulb: bp 175–180 C (0.2 mm) ; IR (CCl_4) absence of OH, 1645, 1110 (broad) cm^{-1}; EIMS (m/e) 357.6 $(M)^+$, 298 $(M\text{-}CH_2\text{-}CH_2OCH_3)^+$, 282 $(M\text{-}OCH_2CH_2OCH_3)^+$, 142 $(CH_2 = NHC_8H_{17})^+$. Similarly were obtained 3 bearing 3 EO units: bp 200–210 C (0.1 mm); 4 EO units: bp 215–225 C (0.1 mm); 5 EO units: bp 230–235 C (0.1 mm). The IR spectra were essentially the same, and the diagnostic EIMS fragments were M^+, $[M\text{-}O(CH_2CH_2O)_{m-1}CH_3]^+$ and 142 $(CH_2 = NH_2C_8H_{17})^+$.

Synthesis of 1-octyl-1-octanoylaminoalcohols (4). A solution of trimethylsilyl iodide was prepared by adding trimethylsilyl chloride (1.4 ml, 10.9 mmol) to a stirred and cooled (0–5 C) solution of NaI (1.65 g, 10.9 mmol) in dry acetonitrile under nitrogen. The mixture was stirred for 0.25 hr, and then the 1-octyl-1-octanoylaminoether, 3 (2 EO units) was added. The resulting mixture was allowed to stir overnight at ambient temperature. The product was obtained by diluting the mixture with water (bisulfite used to decolorize the mixture) and extracting with ether. The organic phase was washed with water, dried (Na_2SO_4) and concentrated to remove solvent. Column chromatography using silica gel (10 g) was employed using ethyl acetate-hexane mixtures and 25 ml fractions to purify the product. The starting material 3 was recovered in fractions 2–4 (10–35% ethyl acetate); the product 4 was obtained in fractions 6–8 (60–80% ethyl acetate). The chromatography was monitored by TLC. The yields of 4 varied considerably (10–60%) and no effort was made to optimize conditions because these could vary with the number of EO units; i.e., might have to be established for each product. A sample was distilled bulb-to-bulb to give 4 bearing 2 EO units: bp 180 C/0.1 mm; IR (CCl_4) 3440 (broad), 1640 1120, 1060 cm^{-1}; EIMS (m/e) 343.5 $(M)^+$, 298 $(M\text{-}CH_2\text{-}CH_2OH)^+$, 244 $(M\text{-}C_7H_{15})^+$, 142 $(CH_2 = NH_2C_8H_{17})^+$. In similar manner were obtained 4 bearing 3 EO units: bp 210–215 C (0.1 mm); 4 EO units: bp 215-220 C (0.1 mm); 5 EO units: bp 235–245 C (0.1 mm). The IR spectra were essentially the same, and diagnostic EIMS fragments were M^+, $[M\text{-}(CH_2CH_2O)_{n-1}H]^+$, 142 $(CH_2 = NHC_8H_{17})$.

Synthesis of 1-octyl-1-octanoylaminoethanol (4) (1 EO) unit . A solution of lithium diisopropylamide was prepared from diisopropylamine (4.2 ml, 30 mmol) and n-butyllithium (11.5 ml of 2.6 M) in dry tetrahydrofuran (50 ml) under nitrogen. The N-octyloctanamide (4) (5.1 g, 20 mmol) was added directly, and the resulting mixture was stirred for 0.25 hr at room temperature. The solution of lithiated amide was cooled (−78 C) and hexamethylphosphoric (10.4 ml, 60 mmol) and ethylene oxide (1.5 ml, 30 mmol) were injected sequentially, the latter from a precooled syringe.

The mixture was allowed to stir coming to room temperature overnight. The crude product was obtained by diluting the mixture with water and extracting with ether. The organic phase was washed with dil HCl and then water. The solution was dried (Na_2SO_4), and the solvent was removed by rotary evaporation to produce 4 (1 EO unit) quantitatively and free of amide and rearranged aminoesters as judged by TLC: IR (CCl_4) 3630 (broad), 1625 cm^{-1}; EIMS (m/e) 299 $(M)^+$, 268 $(M\text{-}CH_2OH)^+$, 200 $(M\text{-}C_7H_{15})^+$, 142 $(CH_2 = NHC_8H_{17})^+$.

RESULTS AND DISCUSSION

Because more obvious routes to the desired structural types failed, it would be useful to briefly mention them and the apparent basis for failure. Potentially useful synthetic intermediates preparable from homogeneous polyethylene glycols are the protected halohydrins (Fig. 2). In fact, the monotetrahydropyranyl ether (THP) of triethylene glycol has been reported and was employed as its alkoxide anion for a displacement reaction in the preparation of phase-transfer catalysts (7). We prepared several chloro and iodopolyethyleneoxy THP ethers by standard procedures; the compounds with EO > 1 do not appear to have been reported. The key step in the sequence was to be a displacement of the halide ion using the N-anion of the amide obtained by deprotonation of the amide with, e.g., lithium diisopropylamide. It is well established that β-ether oxygen retards SN2 displacement reactions (8), and this sluggishness of reaction coupled with the mild temperatures required with amide ions (room temperature or lower) combined to thwart alkylation.

Hydroxyethylated amides, i.e. amides bearing one EO unit, are easily obtained by reaction of amide anions with ethylene oxide (9,10) (Fig. 2). Ethylene oxide reacts much more rapidly with amide anions than with the

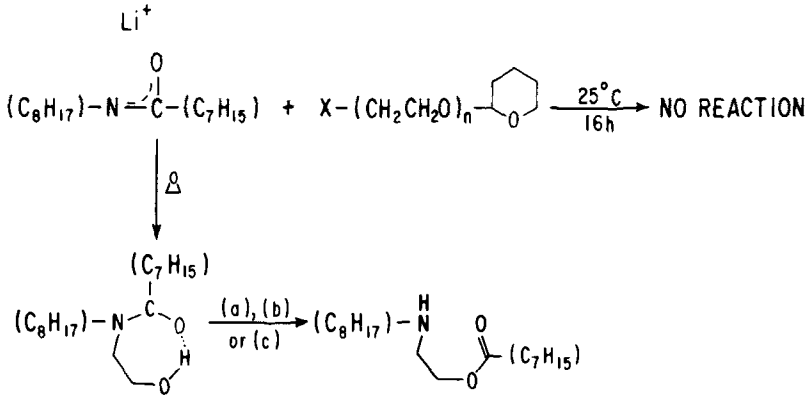

FIG. 2. Nominal approaches to N-(EO)-substituted secondary amides that were unsuccessful; (a) pyridine, methanesulfonyl chloride; (b) butyllithium, tosyl chloride, lithium bromide; (c) triphenlphosphine dibromide.

oxyanions that are formed as products, so there is essentially no polymerization as long as only one equivalent of ethylene oxide is used. If the hydroxyl group of such an adduct could be transformed to a displaceable unit (halide, mesylate), the alkoxide ion of a mono-THP- protected polyethylene glycol could perform the required displacement reaction. This reversal of roles, however, failed because the resulting hydroxyethylated amide underwent rearrangement to the corresponding aminoester (Fig. 2). In fact, the amide carbonyl of this short (one) EO substituted compound absorbs at 1625 cm^{-1}, fully 20 cm^{-1} lower than other amides reported here, presumably by hydrogen bonding, indicating a favorable location for potential bond formation to the carbonyl by the hydroxyl group.

The successful route, outlined in Figure 3, takes advantage of the reactivity of aziridines with alcohols under acidic conditions to form aminoethers. Based on the work of Micich and Linfield (4,5), the n-octyl and n-octanoyl groups were chosen as substituents for the desired final products. Octylamine was converted to N-2-hydroxyethyl-N-octylamine (5) and then transformed to an aziridine via its sulfate ester by the method of Wenker (11) (Fig. 3). Conversion of the aziridine to a β-aminoether required the chosen (homogeneous) polyethylene glycol to be mono-protected by a blocking group that is relatively insensitive to acid and that could be cleaved without disrupting the EO chain or the amide group. Several blocks were possible, and we chose to use a methyl ether because several monomethoxy polyethylene glycols are commercially available, and methyl ethers provide simpler ^{13}C and ^{1}H NMR spectra.

If the aziridine was allowed to react with the monomethoxy polyethylene glycol using only a catalytic quantity of acid (p-toluenesulfonic acid), the product was largely polymeric even though the alcohol was used as a solvent. Even with equimolar amounts of the anhydrous acid, close to 50% of the product was still a mobile undistillable liquid. Evidently the aziridinium ion that is formed has ample time to react with unprotonated aziridine and thereby established low molecular weight polymers. The best procedure involved adding the aziridine to ca. 2.5 equivalents of anhydrous p-toluenesulfonic acid in dried alcohol at 50-60 C. Yields ranged from 56-65% for the aminoethers. The aminoethers were N-acylated with octanoyl chloride/triethylamine in methylene chloride in the usual manner with yields of 70-80% after column chromatography.

Removal of the methyl ether block was accomplished with trimethyl silyliodide (TMSI) in acetonitrile (12). It has been reported that reaction of this reagent (> 2 equivalents) with methyl cyclohexyl ether produces the two corresponding iodides, although if exposure of the ether is limited to only slightly more than one equivalent of TMSI the predominant iodide product is methyl iodide (95:5) (13). The reaction is envisioned as occurring via SN2 displacement of a methyl (or alkyl) group from a trimethylsilyloxonium ion (Fig. 3). Steric hindrance to the reaction on the cyclohexyl side is the reason for differential reactivities of the alkyl residues. Because of the aforementioned retardation to such displacements in polyoxyethyl halides engendered by β-ether oxygen, we imagined that a favorable split would occur in the amide-polyethyleneoxy methyl ethers, and this apparently was the case. Conditions for this last reaction were not optimized, although the following observations were made: the cleavage with TMSI is quite slow, and reaction occurs almost exclusively at the desired methyl ether goup. Alternative ether cleavages would result in smaller EO-bearing units that would be detected easily by TLC and during distillation of the final amide-alcohol. No evidence was obtained for an iodo-polyethyleneoxyamide (TLC, column chromatographic collections subjected to flame-Beilstein test), and only the starting material (amide ether) was recovered. A persistent byproduct, however, gave a slow TLC spot that exhibited ester carbonyl at 1740 cm^{-1}. This material probably was a rearranged aminoester, though this point was not examined in detail (Fig. 4). A large excess of TMSI did use up the starting material, generated greater quantities of the ester product, and gave evidence of alternative ether cleavages. Evidently the best procedure entails the use of 1.5 equivalents of TMSI and a 24-hr reaction period providing about 30-40% yields each of the amide alcohol and amide ether recovered after column chromatography.

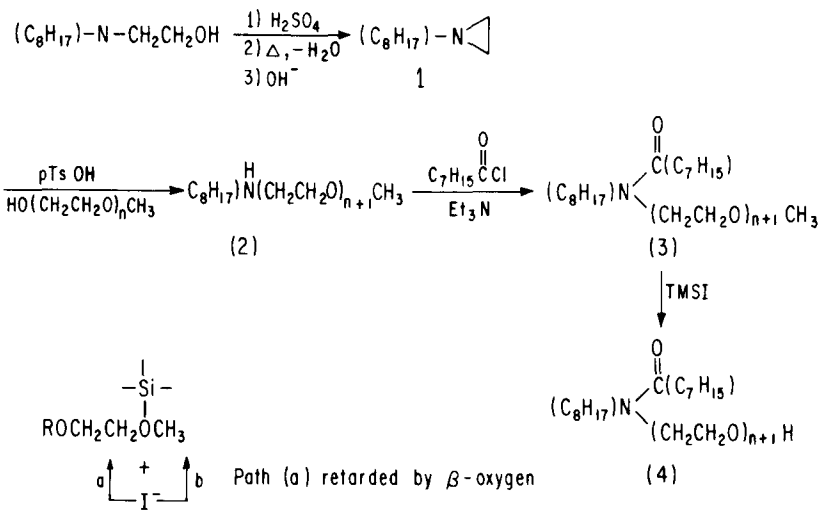

FIG. 3. Synthesis of N-(EO)-substituted secondary amides.

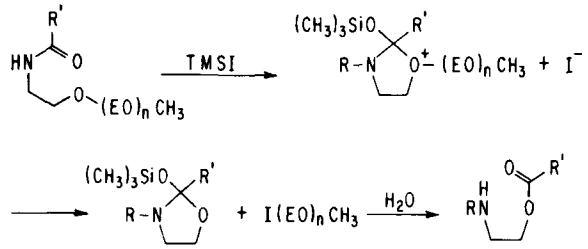

FIG. 4. Possible source of ester by-product from ether cleavage.

More detailed investigation of surfactant properties of monomeric N-hydroxyethylated amides will be performed in conjunction with studies of interactions of such compounds with soil.

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Synthesis and Properties of New Cationic Surfactants

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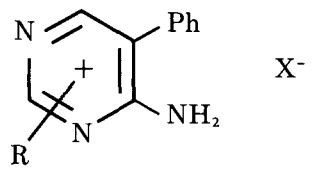
New pyrimidinium cationic surfactants containing a positively charged nitrogen atom of the pyrimidine ring and a long-chain alkyl group of 6,8,10,12,14 carbon atoms were prepared. The structural assignment of these compounds was made on the basis of the elemental analysis and spectroscopic data, infrared, nuclear magnetic resonance and mass spectroscopy. Surface tension and after-treatment of direct dyed cotton fabrics were determined for these surfactants. Antibacterial activities also were examined.

Cationic surfactants with a positively charged nitrogen atom and one or more hydrophobic long-chain alkyl substituents display antimicrobial activities (1). They are also used as auxiliaries in dyeing fabric (1,2). The little known chemistry and properties of pyrimidinium cationic surfactants (3) encouraged us to prepare and study the properties of such surfactants having the general formula:



$R=C_nH_{2n+1}$, n=6,8,10,12,14
X=I, Br

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The above compounds were obtained in good yields (60-80%) by the reaction of 4-amino-5-phenyl-pyrimidine 1 with excess of alkylhalides 2 under reflux in methanol for 24 hr (Scheme 1).

The structural assignment of the prepared compounds 3 was made on the basis of elemental analysis and spectroscopic data (IR, NMR, MS) summarized in Table 1.

EXPERIMENTAL PROCEDURES

All melting points are uncorrected and were determined with a Koffler hot-stage apparatus. IR spectra were obtained with a Perkin-Elmer 281 B spectrophotometer, NMR spectra reported in δ units were recorded with a Varian A-60A spectrometer using D_2O as solvent and tetramethylsilane as an external standard. The mass spectra were measured with a Hittachi-Perkin-Elmer Model RMU-6L spectrometer with an ionization energy of 70 eV. Elemental analyses were performed with a Perkin-Elmer analyzer Model 240 B.

Preparation of starting materials. 4-Amino-5-phenyl-pyrimidin 1 was prepared according to the procedure described in the literature (4).

General procedure for the preparation of halide salts of 1(3)-alkyl-4-amino-5-phenyl-pyrimidines 3. A mixture of 0.01 mol (1.71 g) of 1 and 0.03 mol of 2 was carried out in absolute methanol and refluxed with stirring for 24 hr. The residue was evaporated and dissolved in hot water. The compounds 3 were precipitated after cooling and recrystallized from water.

Study of surface activity: Surface tension. Measure-