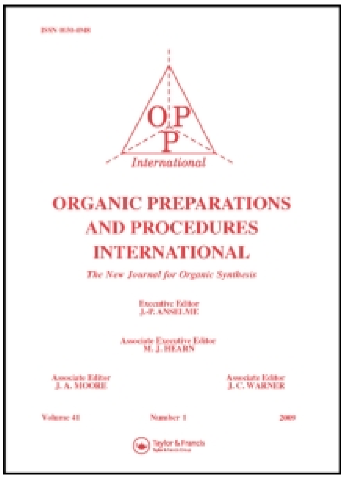

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# 3- AND 4-(7-BENZ[a]ANTHRACENYL)-N-(2-CHLOROETHYL)BENZAMIDES AND 3- AND 4-(9-ANTHRACENYL)-N-(2-CHLOROETHYL)BENZAMIDES

F. A. Vingiello[a][b]; M. P. Rorer[a]; M. A. Ogliaruso[a]
[a] Department of Chemistry, Virginia Polytechnic Institute and State University, Blacksburg, Virginia [b] Department of Chemistry, Northeast Louisiana State College, Monroe, Louisiana

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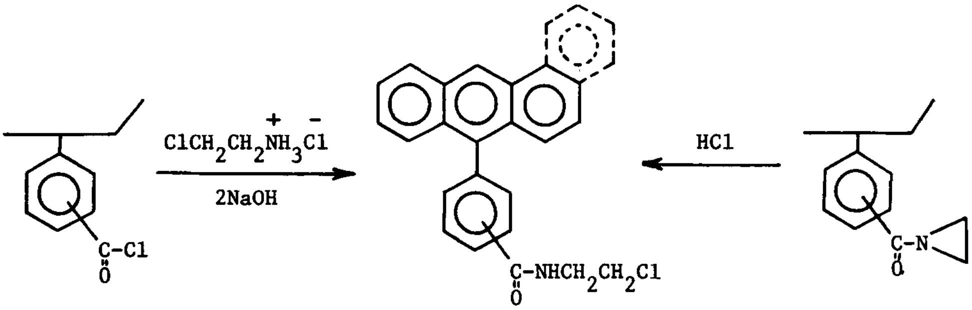
# 3- AND 4-(7-BENZ[a]ANTHRACENYL)-N-(2-CHLOROETHYL)BENZAMIDES
# AND
# 3- AND 4-(9-ANTHRACENYL)-N-(2-CHLOROETHYL)BENZAMIDES

F. A. Vingiello,[1a] M. P. Rorer[1b] and M. A. Ogliaruso[1c]

Department of Chemistry
Virginia Polytechnic Institute and State University
Blacksburg, Virginia 24061

Two methods have been used to prepare N-(2-chloroethyl)amides; reaction of hydrochloric acid with 1-aroylaziridines[2] and by reaction of 2-chloroethylamine hydrochlorides with acid chlorides.[3] However, very little has been done in the way of extending these preparative reactions for N-(2-chloroethyl)amides to polycyclic compounds. We now report the preparation of four polycyclic N-(2-chloroethyl)amides, 3- and 4-(7-benz-[a]anthracenyl)-N-(2-chloroethyl)benzamides (9 and 10) and 3- and 4-(9-anthracenyl)-N-(2-chloroethyl)benzamides (11 and 12), using both of the methods described above.



1, 2, 3, 4 9, 10, 11, 12 5, 6, 7, 8

## EXPERIMENTAL[4]

N-(2-Chloroethyl)amides. General Procedures.

The four polycyclic N-(2-chloroethyl)amides 9, 10, 11 and 12 were each prepared from the acid chlorides (Method I) and from polycyclic 1-aroylaziridines (Method II). Products obtained from both methods were identical.

Method I. A solution of 0.042 mole of acid chloride (1, 2, 3 or 4)[5] in 300 ml of benzene was added over a 15 min. period to a mixture of 6.1 g (0.053 mole) of 2-chloroethylamine hydrochloride and 5.2 g (0.13 mole) of sodium hydroxide in 100 ml of benzene and 200 ml of water contained in a flask immersed in an ice bath at 0°. The mixture was magnetically stirred at 0° for 3 hrs., the precipitated product removed by filtration and dried at 50° (10 mm) for 5 hrs.

Method II. Anhydrous hydrogen chloride was bubbled vigorously through a solution of 0.0083 mole of polycyclic 1-aroylaziridine (5, 6, 7 or 8)[5] in 100 ml of chloroform at room temperature for 15 min. The solution was then transferred to a separatory funnel, washed successively with 100 ml of water, 100 ml of 10% sodium bicarbonate solution, two 100 ml portions of water, and the chloroform layer then separated and dried over sodium sulfate. The dried solution was filtered, concentrated, and the solid product isolated.

### TABLE I

Physical Data for Polycyclic N-(2-Chloroethyl)benzamides

| | Yield (%) | | Spectral Data | |
|---|---|---|---|---|
| Compound | Method I (mp) | Method II (mp) | Infrared[a] (cm$^{-1}$) | nmr (δ) |
| 9 white nuggets | 86 (136-138)[b] | 71 (135-138)[c] | 3050, 2950, 1633, 900-700 | 5.4-4.0 (m, 15, ArH), 1.99 (s, 4, CH$_2$-CH$_2$)[d] |
| 10 white powder | 83 (204-208)[e] | 90 (203-208)[f] | 3050, 2950, 1635, 900-700 | 5.1-4.1 (m, 15, ArH), 2.0 (s, 4, CH$_2$-CH$_2$)[g] |
| 11 yellow needles | 84 (168-169)[b] | 78 (167-169)[c] | 3049, 2950, 1633, 900-700 | 4.7-3.9 (m, 13, ArH), 2.0 (s, 4, CH$_2$-CH$_2$)[d] |
| 12 pale yellow scales | 85 (204-208)[e] | 89 (203-208)[f] | 3050, 2948, 1635, 900-700 | 4.7-3.9 (m, 13, ArH), 2.0 (s, 4, CH$_2$-CH$_2$)[g] |

[a] As KBr pellets. [b] One crystallization from EtOAc. [c] Five crystallizations from EtOAc. [d] In CDCl$_3$. [e] One crystallization from benzene. [f] Four crystallizations from benzene. [g] In DMSO.

### TABLE II

Elemental Analysis for Polycyclic N-(2-Chloroethyl)benzamides

| | | % Calcd. | | | | % Found | | | |
|---|---|---|---|---|---|---|---|---|---|
| C'mpd. | Formula | C | H | N | Cl | C | H | N | Cl |
| 9 | $C_{27}H_{20}ClNO$ | 79.10 | 4.93 | 3.42 | 8.65 | 79.22 | 4.90 | 3.31 | 8.56 |
| 10 | $C_{27}H_{20}ClNO$ | 79.10 | 4.93 | 3.42 | 8.65 | 79.12 | 4.90 | 3.24 | 8.55 |
| 11 | $C_{23}H_{18}ClNO$ | 76.76 | 5.05 | 3.89 | 9.85 | 76.82 | 5.08 | 3.84 | 9.71 |
| 12 | $C_{23}H_{18}ClNO$ | 76.76 | 5.05 | 3.89 | 9.85 | 76.84 | 5.17 | 3.73 | 9.81 |

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1. a) Present address: Northeast Louisiana State College, Department of Chemistry, Monroe, Louisiana, 71201; b) Abstracted from the Ph.D. Thesis of M.P.R., Nov. 1969; c) To whom inquiries should be sent.

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4. The infrared spectra were recorded on a Perkin-Elmer Model 621 spectrophotometer using potassium bromide disks. The nmr data were obtained from a Varian A-60 spectrometer. Line positions are recorded as ppm from internal tetramethylsilane ($\delta$ scale). All melting points are corrected.

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