ysis of VII—The hydrochloride salt (VII) (100 mg., 0.00034 mole) was mixed with 2-methoxyethanol (50 ml.), and concentrated hydrochloric acid (10 ml.) was added. The mixture was refluxed for 36 hr., during which time additional quantities (5 ml.) of concentrated hydrochloric acid were added, after 8 hr. and again after 14 hr. Initially the color of the solution was light brown; on heating for 7–8 hr., some solid separated and the color of the solution turned yellow. On continued heating, the solid dissolved and the solution turned deep brown in color. After evaporation to dryness, the residue was boiled with water for 20 min. and filtered. The residue (35 mg., 40%) was thoroughly washed with acetone and crystallized from ethanol, m.p. 295–296°. $\lambda_{max.}^{EtOH}$ 390 mμ (ε 8,772); 334 mμ (ε 8,772); 250 mμ (ε 36,600).

Anal.—Calcd. for $C_{13}H_{12}N_4O_2$: C, 60.93; H, 4.68; N, 21.87. Found: C, 61.39; H, 5.04; N, 21.97.

Alkaline Hydrolysis of VII—This was conducted in the same manner as described in the preparation of IV from III. $\lambda_{max.}^{EtOH}$ identical with acid hydrolysis product.

Alkaline Hydrolysis of VI—The quaternary methyl iodide (VI) was hydrolyzed, in the same manner, and the product obtained showed the same spectrum as the acid and base hydrolysis products from VII above.

Thin-Layer Chromatography—The above compounds obtained from VII by acid and alkaline hydrolysis, respectively, were compared with IV (obtained by alkaline hydrolysis of III) by simultaneously conducted thin-layer chromatography, using alumina as the adsorbent and dimethylformamide–water (9:1) mixture as the solvent. Each compound gave a single fluorescent spot under ultraviolet light, corresponding to an R_f value of 0.55

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Keyphrases

Deoxyalloxazines (benzopteridines)
2,4-Diamino-6,7-dimethylbenzo(*g*)-pteridine—methylation
UV spectrophotometry—structure
IR spectrophotometry—structure
NMR spectrometry
TLC—UV light fluorescence

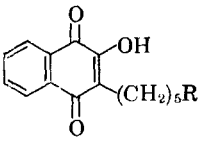
Lawsone Derivatives IV. 3-ω-Substituted Alkyllawsones and Related Compounds

By H. MACHATZKE, W. R. VAUGHAN*, C. L. WARREN, and G. R. WHITE

This paper reports the preparation of two 3-alkyllawsones in which there is a nitrogen mustard function in the ω-position. In addition, a potentially general route to the preparation of 3-ω-aminoalkyllawsones is described. The method involves introduction of an ω-amino group *via* an ω-azido group.

THE OBJECTIVES of this continuing research have been discussed in a previous paper (1), and the purpose of the present paper is to report preparation of lawsones (2-hydroxy-1,4-naphthoquinone) with 3-alkyl substituents terminating in an alkylating function. In addition, the development of a potentially general route to preparation of 3-ω-aminoalkyllawsones has been studied, and various ancillary problems have been delineated.

Two related series of lawsone derivatives have been investigated: one in which the 3-position carries a normal pentyl chain terminating in a functional group (general structural formula I, in which lower case letters refer to known starting materials involved in synthesis); and one in which the 3-position carries a normal decyl chain terminating in a functional group (general structural formula II, with notation as for I).



I

R

Ia —$CO_2C_2H_5$	*IA* —CO_2CH_3 methyl ether
Ib —COCl	*IB* —CH_2OH
Ic macrocyclic lactide derived from *Ib*	*IC* —$CON(CH_2CH_2OH)_2$
	ID —$CON(CH_2CH_2Cl)_2$

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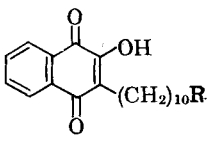

II

R

IIa —OH IIA —OSO_2SCH_3 methyl ether

IIb —Br IIB —$OCON(CH_2CH_2Cl)_2$

IIC —N_3

IID —$NH_2 \cdot HO_2CCH_3$

IIE —$NHCOCH_3$

DISCUSSION

Series I—In this series the preparation of methyl 6-(2-methoxy-1,4-naphthoquinon-3-yl)hexanoate (I*A*) and of 6-(2-hydroxy-1,4-naphthoquinon-3-yl)hexanol are trivial, the former involving boron trifluoride-etherate catalyzed trans-methylation of I*a* (1) followed by treatment with dimethyl sulfate of the sodium salt; and the latter involving lithium aluminohydride reduction of I*a* followed by silver oxide oxidation.

However, the preparation of *N,N*-bis(2-chloroethyl)-6-(2-hydroxy-1,4-naphthoquinon-3-yl)hexanamide (I*D*) from *N,N*-bis(2-hydroxyethyl)-6-(2-hydroxy-1,4-naphthoquinon-3-yl)hexanamide (I*C*) proved singularly difficult. In the first place treatment of I*a*, I*b*, and I*c* with varying amounts of diethanolamine afforded in each case a product resembling that expected, but in no instance could an analytically satisfactory sample be obtained owing to the high polarity and hydrophilic character of the material. The general appearance and physical characteristics of the products obtained from I*a*, I*b*, and I*c* were invariably the same, the reported procedures being typical of many runs of each. And while no absolute proof of structure is available, the fact that the infrared spectra of all products correspond in the significant bands, which in turn are favorably comparable to those in the infrared spectra of 6-(2-hydroxy-1,4-naphthoquinon-3-yl)-hexanamide and I*D*, strongly suggests that one obtained I*C* from any of the reported procedures in comparable yield and purity. Unquestionably, the difficulty in converting I*C* to I*D* was a consequence of the difficulty of obtaining analytically pure I*C*. However, in one instance one of the authors (H. M.) was able to effect the conversion by treating a sample of I*C* with thionyl chloride.

Series II—In this series the preparation of the first two members was a relatively simple matter. But is is interesting to note that intended methanesulfonation of the decanol, II*a* (1) surprisingly resulted in simultaneous *methylation* of the 2-hydroxyl group when the reaction was carried out under very mild conditions (methanesulfonyl chloride and excess pyridine in dry benzene at room temperature), giving 10-(2-methoxy-1,4-naphthoquinon-3-yl)decyl methanesulfonate (II*A*). There can be no question of the structure, since the infrared spectrum lacks hydroxyl absorption, the NMR spectrum shows *O*-methyl as well as *S*-methyl, and II*A* is insoluble in alkali, retaining its color rather than turning red, as do free lawsones.

No problems were encountered in the reaction of the decanol (II*a*) with *N,N*-bis(2-chloroethyl)-carbamoyl chloride (2) in pyridine to give *N,N*-bis(2-chloroethyl)-10-(2-hydroxy-1,4-naphthoquinon-3-yl)decyl carbamate (II*B*).

The remaining compounds in this series constitute the culmination of an exhaustive series of attempts to introduce an amine function bonded to carbon at the terminus of a 3-alkyl chain in lawsone. It was originally intended to use the decyl bromide (II*b*) (3) as a substrate for reaction with diethanolamine or bis(2-chloroethyl)amine; and when these two amines failed to give characterizable products, ammonia was also tried, with equally unsatisfactory results. Certainly these failures are attributable in part to complex additions to the lawsone system, such as are described in the preceding paper in this series.

Confronted with this discouraging experience, the authors turned their attention in the final phases of the study to preparation of 10-(2-hydroxy-1,4-naphthoquinon-3-yl)decylamine, which was actually isolated as its acetate salt (II*D*) and was characterized as such and as its acetyl derivative (II*E*). The availability of II*D* should permit future study of methods for *N*-hydroxyethylation and *N*-chloroethylation of ω-(2-hydroxy-1,4-naphthoquinon-3-yl)alkylamines, especially since there is no *a priori* reason why the authors' method of synthesis for II*D* should not be general.

By contrast with wholly unsuccessful attempts to obtain ω-(2-hydroxy-1,4-naphthoquinon-3-yl)alkylamines by various reduction processes applied to amides, notably by means of complex metal hydrides, the route involving reaction of the decyl bromide II*b* with azide ion, followed by hydrogenation was relatively straightforward. The only problem was in preparation of 10-(2-hydroxy-1,4-naphthoquinon-3-yl)decyl azide (II*C*) which had to be carried out in *N,N*-dimethylformamide using lithium azide instead of the more conventional sodium azide-methanol reaction, which led to recovery of unchanged II*b*. Reduction of II*C* to II*D* was achieved by hydrogenation at 3 Atm. over Adams' catalyst, followed by air oxidation, and the product proved to be an extremely stable acetate salt, which could readily be converted to the acetamide (II*E*).

EXPERIMENTAL[1]

Methyl 6-(2-hydroxy-1,4-naphthoquinon-3-yl)-hexanoate—In an attempt to methylate the free hydroxyl group of I*a*, 5.1 Gm. (16.1 mmoles) of this ethyl ester was dissolved in 100 ml. of methanol, and 12 ml. of boron trifluoride-etherate was added. The mixture was held at 70°, with stirring, overnight and then was held at 75–80° for 24 hr. Evaporation to dryness under reduced pressure left a yellow solid, m.p. 79–83°, and recrystallization from petroleum ether (b.p. 60–75°) raised this to 88–90°. Some 85% of this transesterified material was recovered in all.

Methyl 6-(2-methoxy-1,4-naphthoquinon-3-yl)-hexanoate IA—A sodium salt was prepared by adding 297 mg. of sodium (12.9 mmoles) to a solution of the above compound (3.9 Gm., 12.9 mmoles)

[1] Melting points uncorrected. Kofler block method indicated where used. Microanalyses by Spang Microanalytical Laboratory, Ann Arbor, Mich. Infrared spectra of Nujol mulls obtained with Perkin-Elmer model 21 double beam spectrometer. NMR spectra obtained with Varian A-60 spectrometer, 60 Mc., internal TMS reference. We are indebted to Mr. B. E. Wenzel, Mr. Günter Schütze, and Mr. Frank Parker for preparation of most of these spectra.

in 40 ml. of methanol, and the resulting red solution was evaporated to dryness under reduced pressure.

The red sodium salt was heated to 120–130° and treated, with stirring, with 1.64 Gm. (13 mmoles) of dimethyl sulfate. Some yellowing was noted, and more dimethyl sulfate (1.64 Gm., 13 mmoles) was added. After about 1 hr. all red color had disappeared, leaving a brown-yellow reaction mixture. Dilution with water (10 ml.) and addition of 10 ml. of saturated sodium carbonate solution resulted in production of the previous dark red color. Next 60 ml. of 5% hydrochloric acid and 6 ml. of concentrated hydrochloric acid was added and the mixture was extracted with chloroform. After drying (magnesium sulfate), filtration, and evaporation, there was left an oil which crystallized on being triturated with petroleum ether (b.p. 40–60°). Recrystallization from petroleum ether (b.p. 60–75°) afforded 2.339 Gm. (60%) of starting material (infrared spectrum and m.p.).

The mother liquor was concentrated and cooled to −5°, whereupon yellow needles precipitated, m.p. 43°. Recrystallization from the same solvent raised the m.p. to 47° and finally to a constant 57° (805 mg., with an additional 815 mg., m.p. 55° obtained as a second crop: 40% total). The infrared spectrum showed no hydroxyl absorption.

Anal.—Calcd. for $C_{18}H_{20}O_5$: C, 68.34; H, 6.37. Found: C, 68.46; H, 6.52.

6 - (2 - Hydroxy - 1,4 - naphthoquinon - 3 - yl)-hexanol (IB)—To a vigorously stirred suspension of lithium aluminohydride (LAH) (0.57 Gm., 15 mmoles) in 25 ml. of tetrahydrofuran (THF) (distilled from LAH) was added a solution of 3.16 Gm. (10.0 mmoles) of I*a* (1) in 25 ml. of similarly dried THF. Residue of *Ia* was washed into the reaction vessel with an additional 10 ml. of dry THF, and the brownish-red mixture was refluxed for 4 hr. and then cooled to 0°. Next, 6 ml. of ice water was added followed by 26 ml. of 6 *M* hydrochloric acid. Extraction with three 25-ml. portions of ether followed, and the combined ethereal extracts were vigorously stirred with 3.47 Gm. (15 mmoles) of freshly precipitated silver oxide. The mixture was then dried over anhydrous magnesium sulfate and filtered. Removal of the ether under reduced pressure left 2.73 Gm. of a dark brown solid, m.p. 86–87°, which was purified by digestion with three 200-ml. portions of petroleum ether (b.p. 90–100°) on the steam bath. The extracts were combined and evaporated under reduced pressure leaving a solid which was recrystallized from benzene to give 1.24 Gm. (45%) of *IB*, m.p. 103.5–104.0°. The infrared spectrum is appropriate for *IB:*

Anal.—Calcd. for $C_{16}H_{18}O_4$: C, 70.05; H, 6.61; mol. wt., 274. Found: C, 69.99; H, 674; mol. wt., 265.

An additional 0.30 Gm. (11%) of less pure *IB*, m.p. 98°, was slowly deposited from the mother liquor.

N,N - Bis(2 - hydroxyethyl) - 6 - (2 - hydroxy-1,4-naphthoquinon-3-yl)hexanamide (IC)—(*a*) From ethyl 6-(2-hydroxy-1,4-naphthoquino-3-yl)hexanoate (I*a*) (1). A mixture of 0.64 Gm. of I*a* (2 mmoles) and 0.22 Gm. (2 mmoles) of diethanolamine in 10 ml. of ethanol, with which 6 mg. of sodium had been allowed to react, was heated in a sealed tube for 3 hr. at 160°. The product, a dark red tar, was taken up in 30 ml. of chloroform at 65°, concentrated and then chromatographed on Florosil (10 × 2-in. column). After elution of traces of a pale yellow band and a broad pink band (with chloroform), further eluates obtained with chloroform and then methanol possessed infrared spectra comparable in most respects to that of *IC* obtained from I*c* (below). (*b*) From 6-(2-hydroxy-1,4-naphthoquinon-3-yl)hexanoyl chloride (I*b*). A solution of I*b* (1.06 Gm., 0.35 mmoles) and diethanolamine (4.0 Gm., 38 mmoles) in 10 ml. of toluene was stirred with refluxing for 15 hr. Next 45 ml. of concentrated hydrochloric acid was added, and the lower layer was continuously extracted with ether for 24 hr., the original toluene being part of the extraction medium. Evaporation of the filtered red organic extracts left a deep red solid possessing an infrared spectrum comparable in most respects to that of *IC* obtained from I*c* (below). (*c*) From the macrolactide (I*c*). A mixture of 1.0 Gm. of I*c* and 5 ml. of diethanolamine was heated at 100–150° for 2 hr. and then was allowed to stand overnight at room temperature. Next the solution was filtered, acidified with excess hydrochloric acid, and continuously extracted with ether. The ethereal extract was then diluted with 20 ml. of methanol and dried over anhydrous magnesium sulfate. Filtration and removal of the solvent left a red oil whose infrared spectrum possessed the expected characteristic bands for *IC:* (Nujol) 3400–3160; 1740 (shoulder); 1732–1720; 1672; 1650; 1640; 1600; 1470; 1440; 1372; 1280; 1220; 730 cm.$^{-1}$ In no case could *IC* be obtained sufficiently pure for satisfactory analysis.

N,N - Bis - (2 - chloroethyl) - 6 - (2 - hydroxy - 1,4-naphthoquinon-3-yl)hexanamide (ID)—The crude product from the previous experiment was dissolved in 10 ml. of absolute chloroform and treated with 2 ml. of thionyl chloride at ice-bath temperature with subsequent stirring for 2 hr. at room temperature. Next the excess thionyl chloride was removed by evaporation *in vacuo* followed by two additions and vacuum distillations of benzene. The residual red gum was taken up in chloroform and diluted with dry ether, whereupon yellow crystals separated (0.30 Gm.), m.p. 111–112° (Kofler). The infrared spectrum is consistent for *ID*.

Anal.—Calcd. for $C_{20}H_{23}Cl_2NO_4$: C, 58.20; H, 5.63; Cl, 17.20; N, 3.39. Found: C, 58.33; H, 5.80; Cl, 17.06; N, 3.50.

Numerous attempts to repeat this procedure using I*c* from *a*, *b*, and various modifications of *c* were unsuccessful.

10 - (2 - Methoxy - 1,4 - naphthoquinon - 3 - yl)-decyl methanesulfonate (IIA)—A solution of 2.2 Gm. of II*a* [10-(2-hydroxy-1,4-naphthoquinon-3-yl)-decanol (2)] (6.7 mmoles) and 2.3 Gm. (20 mmoles) of methanesulfonyl chloride in 10 ml. of dry benzene containing 5 ml. of dry pyridine was stirred for 8 hr. at room temperature. The solvent was distilled off and the residue taken up in ether and filtered. The ether solution was washed with dilute hydrochloric acid and water and dried over anhydrous magnesium sulfate. Removal of the ether left a yellow oil which was dried *in vacuo* over phosphorus pentoxide. After 4 days, the oil crystallized and then was recrystallized from acetone–water: (2.0 Gm.), m.p. 57–58° Kofler.

Anal.—Calcd. for $C_{22}H_{30}O_6S$: C, 62.53; H, 7.16; S, 7.59. Found: C, 62.12; H, 7.14; S, 7.51.

The infrared spectrum shows no hydroxyl absorption. Treatment with 5% sodium hydroxide at room temperature resulted in no observable color change, although on prolonged standing (over 30 min.) the compound slowly became dark red. The NMR spectrum (in trifluoroacetic acid, 60 Mc.) shows a 4-proton complex multiplet centered at $\sim\tau = 2$, a multiplet integrating for 8 protons at $\tau = 5.85$–6.30 which may be assigned to CH_3O—, CH_3SO_3—, and —CH_2O—. The next band is a broad unresolved multiplet centered at $\tau = 7.20$ (two protons, —CH_2– adjacent to the quinone nucleus), and finally there is a 16-proton multiplet centered at $\tau = 8.60$ (remaining methylenes).

N,N - Bis(2 - chloroethyl) - 10 - (2 - hydroxo - 1,4-naphthoquinon-3-yl)decyl carbamate (IIB)—A solution of 1.7 Gm. (5 mmoles) of II*a* and 0.95 Gm. (5 mmoles) of *N,N*-bis(2-chloroethyl)carbamyl chloride (2) in 5 ml. of pyridine was stirred at room temperature for 8 hr. during which time pyridine hydrochloride precipitated. Water was then added and the mixture was acidified with hydrochloric acid and extracted with ether, the ethereal extracts being washed with water and dried over anhydrous magnesium sulfate. Evaporation of the ether left a yellow oil which was taken up in methanol–water and deposited yellow crystals on being cooled. These were dried over phosphorus pentoxide: 2.2 Gm., m.p. 57–58° Kofler; recrystallized from methanol, m.p. 60–61° Kofler.

Anal.—Calcd. for $C_{25}H_{33}Cl_2NO_5$: C, 60.24; H, 6.67; N, 2.81. Found: C, 60.59; H, 6.79; N, 2.48. The infrared spectrum shows a broad absorption for hydroxyl.

10 - (3 - Hydroxy - 1,4 - naphthoquinon - 3 - yl)-decyl Azide (IIC)—To a stirred solution of II*b* [10-(2-hydroxy-1,4-naphthoquinon-3-yl)decyl bromide (3)] (1.95 Gm., 5.0 mmoles) in 60 ml. of dry anhydrous magnesium sulfate], distilled *N,N*-dimethylformamide (DMF) there was added lithium azide. This was prepared from 6.5 Gm. (0.1 mmole) of sodium azide and 7.05 Gm. (0.055 mmole) of lithium sulfate monohydrate dissolved in 35 ml. of warm water, followed by slow addition of 95% ethanol (175 ml.). Sodium sulfate was filtered off and evaporation of the filtrate under reduced pressure at 80° afforded a yellowish solid which was dried azeotropically with benzene and then used without further purification (4). The reaction mixture, a red solution, was stirred at 95–100° for 16 hr. (5) and then evaporated under reduced pressure to a dark red gum. To this was added 100 ml. of water followed by 10 ml. (excess) concentrated hydrochloric acid (pH = 2 at end of addition). The mixture was then extracted with ether (100 ml. then five 25-ml. portions). Evaporation of the dried (anhydrous magnesium sulfate), filtered ethereal extracts and recrystallization of the yellow residue from petroleum ether (b.p. 60–75°) afforded 1.57 Gm. (94%) of II*C*, m.p. 80.5°.

Anal.—Calcd. for $C_{20}H_{25}N_3O_3$: C, 67.58; H, 7.09; N, 11.82. Found: C, 67.48; H, 7.11; N, 11.96.

10 - (2 - Hydroxy - 1,4 - naphthoquinon - 3 - yl)-decylamine Acetate Salt (IID)—A 2.02-Gm. (5.70 mmoles) sample of II*C* suspended in 200 ml. of absolute ethanol containing 0.25 Gm. of Adams' catalyst was shaken for 19 hr. under 50 p.s.i. of hydrogen. No pressure drop was observed, but a red solid precipitated. This was filtered off and washed with glacial acetic acid until the washings were colorless. The experiment was repeated, and the combined ethanolic acetic acid filtrates were oxidized by bubbling an air stream through the solution for 3 days, by which time the solvent had evaporated leaving an intense red solid, m.p. 190–195°. This was dried over solid sodium hydroxide: 4.093 Gm. (97.5% $B \cdot HO_2CCH_3$).

The product was dissolved in glacial acetic acid, precipitated by addition of water, and collected by centrifugation to give an intensely crimson product, m.p. 196°, with darkening above 185°.

Anal.—Calcd. for $C_{22}H_{31}NO_5$: C, 71.52; H, 8.43; N, 3.79. Found: C, 71.81; H, 8.09; N, 3.87.

***N* - 10 - (2 - Hydroxy - 1,4 - naphthoquinon - 3-yl)decylacetamide (IIE)**—A 426-mg. sample of I*D* was added to 10 ml. of glacial acetic acid to give an orange solution. Next, 1.50 ml. of acetic anhydride was added and the mixture was stirred at room temperature for 2 hr. during which time a yellow precipitate appeared. The mixture was then held at −5° for 2 hr. during which time the color of the precipitate changed. Filtration afforded a pale primrose solid (*A*) (315 mg., m.p. 146°). This was washed with 10 ml. of glacial acetic acid and the filtrate and washings were diluted with water to give 185 mg. of orange solid (*B*) (m.p. 144°).

Both of the products were recrystallized from ethanol-water, the former affording a pale primrose substance softening >140°, m.p. 150° (*A*) and the latter a yellow solid softening >137°, m.p. 146–147° (*B*), and both analyzed correctly for II*E*.

Anal.—Calcd. for $C_{22}H_{29}NO_4$: C, 71.13; H, 7.87; N, 3.77; mol. wt., 371. Found (*A*): C, 70.51, 70.78; H, 7.84, 7.95; N, 4.00; mol. wt., 367, 371. Found (*B*): C, 70.50, 70.76; H, 7.84, 7.94; N, 3.82; mol. wt., 380, 378.

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Keyphrases

Lawsone derivatives
3-Alkyl 2-hydroxy-1,4-naphthoquinones—synthesis
3-ω-Aminoalkyl 2-hydroxy-1,4-naphthoquinones—synthesis
Column chromatography—separation
IR spectrophotometry—structure
NMR spectrometry