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10. Studies on Solubilization (Part I)
Note on the Synthesis of Some Quaternary N-(ω-Aryloxyalkyl) Piperidinium, Pyridinium, Benzyl-dimethyl-ammonium, and Trimethyl-ammonium Bromides

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(15. VIII. 70)

Summary. A series of new N-(ω-aryloxyalkyl) piperidinium, pyridinium, benzyl dimethyl ammonium, and trimethyl ammonium bromides is described.

For the study of the mechanisms of solubilization of organic substances in water, we have synthesized a series of compounds with the general formula Ar–O–$(CH_2)_n$–$\overset{\oplus}{N}$ $Br^{\ominus}$ (see tables II and III).

Table I. *Physical Properties of new α-bromo-ω-aryloxy-alkanes*
Ar—O—$(CH_2)_n$—Br

Ar	n	Yield, % (not optimized)	B.p. °C/Torr	M.p. °C	Calcd., %			Found, %		
					C	H	Br	C	H	Br
4-$(CH_3)_3C \cdot C_6H_4$	4	75.8	138–140 (0.05)		58.95	7.42	28.02	58.72	7.55	27.90
4-$[(CH_3)_3C \cdot CH_2 \cdot (CH_3)_2C] \cdot C_6H_4$	4	53.7	168–170 (0.35)		63.34	8.56	23.41	63.39	8.82	23.28
4-$C_9H_{19} \cdot C_6H_4$	4	27.5	170–175 (0.5)		64.22	8.79	22.49	63.57	8.90	22.14
3-$(CH_3O) \cdot C_6H_4$	4	66.9	148–149 (0.05)		50.98	5.83	30.84	51.20	6.10	31.40
4-$C_6H_5 \cdot C_6H_4$	4	77.5	184–185 (0.5)	78–79 (petr. ether)	62.96	5.61	26.18	63.12	5.63	26.20
1-$C_{10}H_7$	4	53.7	170–175 (0.05)	38 (ethanol)	60.23	5.42	28.62	59.79	5.58	28.25

These quaternary ammonium salts were prepared by treating eleven 1-alkyl-piperidines, 1-cyclohexylpiperidine, pyridine, benzyldimethylamine, and trimethyl-amine with seventeen α-bromo-ω-aryloxy-alkanes. The α-bromo-ω-aryloxy-alkanes were prepared by condensing [1] a phenol with an α,ω-dibromoalkane. Purity of the compounds from table III was further confirmed by paper chromatography [2].

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Table II. *ω-Aryloxyalkyl Ammonium Compounds*[a])

$$Ar{-}O{-}(CH_2)_n{-}\overset{\oplus}{N}R_2 \cdot R'\ \ Br^{\ominus}$$

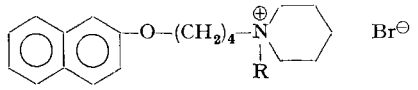
No.	Ar	n	NR_2	R′	Formula	Reaction period (h)	Cryst.	Recryst.	Yield, %	M.p., °C	Calcd., % C	H	N	Found, % C	H	N
1	C_6H_5	2	Piperidino	C_2H_5	$C_{15}H_{24}BrNO$	12	Cr–2	+	76.40	114–115 [5]	57.33	7.70	4.46	57.09	7.72	4.43
2	C_6H_5	3	Piperidino	C_2H_5	$C_{16}H_{26}BrNO$	18	Cr–2	+	76.60	119–120	58.54	7.98	4.27	57.97	8.16	4.23
3	C_6H_5	4	Piperidino	C_2H_5	$C_{17}H_{28}BrNO$	8	Cr–1	+	82.10	166–167	59.65	8.24	4.09	60.35	8.25	4.04
4	C_6H_5	5	Piperidino	C_2H_5	$C_{18}H_{30}BrNO$	18	Cr–2	+	75.80	137–138	60.67	8.49	3.93	60.92	8.37	3.94
5	4-$(CH_3)_3C\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{21}H_{36}BrNO$	25	Cr–3	+	78.56	148–151[b])	63.30	9.11	3.52	63.01	9.10	3.67
6	4-[$(CH_3)_3C\cdot CH_2\cdot (CH_3)_2C$]$\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{25}H_{44}BrNO$	48	Cr–2	+	86.34	130–134[b])	66.07	9.76	3.08	65.94	9.94	3.15
7	4-$C_9H_{19}\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{26}H_{46}BrNO$	72	Cr–1	+	58.48	118[b])[c])	66.65	9.89	2.99	66.11	10.10	3.10
8	2-$(CH_3O)\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{18}H_{30}BrNO_2$	48	Cr–1	+	82.20	146–147.5	58.06	8.12	3.76	57.72	8.18	3.88
9	3-$(CH_3O)\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{18}H_{30}BrNO_2$	48	Cr–2	+	75.27	100–102.5	58.06	8.12	3.76	57.56	8.10	3.72
10	2-$C_6H_5\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{23}H_{32}BrNO$	48	Cr–3	+	67.70	98–101	66.02	7.71	3.35	66.01	7.70	3.50
11	4-$C_6H_5\cdot C_6H_4$	4	Piperidino	C_2H_5	$C_{23}H_{32}BrNO$	18	Cr–1	+	74.33	177.5–179	66.02	7.71	3.35	65.59	7.89	3.37
12	1-$C_{10}H_7$	4	Piperidino	C_2H_5	$C_{21}H_{30}BrNO$	24	Cr–1	+	80.28	172.5–174	64.28	7.71	3.57	64.00	7.91	3.28
13	2-$C_{10}H_7$	3	Piperidino	C_2H_5	$C_{20}H_{28}BrNO$	48	Cr–2	+	62.20	148–149.5	63.49	7.46	3.70	63.13	7.52	3.90
14	2-$C_{10}H_7$	4	Piperidino	C_2H_5	$C_{21}H_{30}BrNO$	48	Cr–2	+	69.60	123–125	64.28	7.71	3.57	64.08	7.68	3.89
15	2-$C_{10}H_7$	5	Piperidino	C_2H_5	$C_{22}H_{32}BrNO$	48	Cr–2	+	68.96	139–141	65.02	7.94	3.47	64.87	8.02	3.34
16	2-$C_{10}H_7$	6	Piperidino	C_2H_5	$C_{23}H_{34}BrNO$	48	Cr–2	+	70.17	90–94	65.70	8.15	3.33	66.29	8.35	3.37
17	C_6H_5	2		Pyridino	$C_{13}H_{14}BrNO$	12	[d])	−	15.50	78[b])[c]) [6]	55.73	5.04	4.99	55.60	5.78	5.10
18	C_6H_5	3		Pyridino	$C_{14}H_{16}BrNO$	12	Cr–2	+	69.73	108–109	57.16	5.48	4.76	56.81	5.74	4.81
19	C_6H_5	4		Pyridino	$C_{15}H_{18}BrNO$	18	Cr–2	+	68.83	87–90.5	58.45	5.89	4.54	58.18	6.03	4.50
20	C_6H_5	5		Pyridino	$C_{16}H_{20}BrNO$	16	Cr–2	+	84.78	144–145.5	59.63	6.26	4.35	59.67	6.51	4.35
21	4-$(CH_3)_3C\cdot C_6H_4$	4		Pyridino	$C_{19}H_{26}BrNO$	18	−[e])	−	48.87[f])							
22	4-[$(CH_3)_3C\cdot CH_2\cdot (CH_3)_2C$]$\cdot C_6H_4$	4		Pyridino	$C_{23}H_{34}BrNO$	24	−[e])	−	58.49[f])							
23	4-$C_9H_{19}\cdot C_6H_4$	4		Pyridino	$C_{24}H_{36}BrNO$	48	−[e])	−	63.00[f])							
24	2-$(CH_3O)\cdot C_6H_4$	4		Pyridino	$C_{16}H_{20}BrNO_2$	48	−[e])	−	78.19[f])							
25	3-$(CH_3O)\cdot C_6H_4$	4		Pyridino	$C_{16}H_{20}BrNO_2$	48	−[e])	−	84.55[f])							
26	2-$C_6H_5\cdot C_6H_4$	4		Pyridino	$C_{21}H_{22}BrNO$	48	Cr–2	+	91.61	63–67	65.63	5.77	3.64	65.29	6.07	3.60
27	4-$C_6H_5\cdot C_6H_4$	4		Pyridino	$C_{21}H_{22}BrNO$	18	Cr–2	+	79.36	159–160.5	65.63	5.77	3.64	65.74	5.84	3.70
28	1-$C_{10}H_7$	4		Pyridino	$C_{19}H_{20}BrNO$	18	Cr–2	+	78.98	138–141	63.69	5.63	3.90	63.94	6.00	3.76
29	2-$C_{10}H_7$	4		Pyridino	$C_{19}H_{20}BrNO$	30	Cr–2	+	71.45	130–132.5	63.69	5.63	3.90	63.08	5.36	4.00
30	2-$C_{10}H_7$	4	2-Methylpyridino		$C_{20}H_{22}BrNO$	48	Cr–1	+	83.26	124–125.5	64.52	5.96	3.76	64.29	6.21	3.74

Table II. (cont.)

No.	Ar	n	NR_2	R′	Formula	Reaction period (h)	Cryst.	Recryst.	Yield, %	M.p., °C	Calcd., % C	Calcd., % H	Calcd., % N	Found, % C	Found, % H	Found, % N
31	C_6H_5	2	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{17}H_{22}BrNO$	24	Cr–2	+	56.54	122–124 [7]	60.72	6.59	4.16	60.25	6.67	4.33
32	C_6H_5	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{19}H_{26}BrNO$	24	Cr–2	+	75.54	128–130	62.64	7.19	3.84	62.22	7.37	3.81
33	C_6H_5	5	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{20}H_{28}BrNO$	24	Cr–2	+	52.90	139–140.5	63.49	7.46	3.70	62.87	7.61	3.70
34	4-$(CH_3)_3C \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{23}H_{34}BrNO$	24	–[e]	–	64.60[f]							
35	4-[$(CH_3)_3C \cdot CH_2 \cdot (CH_3)_2C$]$\cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{27}H_{42}BrNO$	48	Cr–2	+	57.68	84–88	68.05	8.88	2.94	67.65	8.64	3.13
36	4-$C_9H_{19} \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{28}H_{44}BrNO$	48	Cr–5	+	61.40	59–61	68.55	9.04	2.85	67.46	9.07	2.90
37	2-$(CH_3O) \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{20}H_{28}BrNO_2H_2O$	48	from water	–	62.70	81–86	58.26	7.28	3.39	58.68	7.36	3.40
38	3-$(CH_3O) \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{20}H_{28}BrNO_2$	98	–[e]	–	68.80[f]							
39	2-$C_6H_5 \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{25}H_{30}BrNO$	42	freeze dried	–	59.00	38–40	68.18	6.87	3.18	67.53	7.20	3.23
40	4-$C_6H_5 \cdot C_6H_4$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{25}H_{30}BrNO$	48	Cr–1	+	54.94	179–184	68.18	6.87	3.18	67.29	6.90	3.38
41	1-$C_{10}H_7$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{23}H_{28}BrNO$	24	Cr–2	+	54.10	139–141	66.66	6.81	3.38	66.12	6.92	3.38
42	2-$C_{10}H_7$	4	$N(CH_3)_2$	$CH_2 \cdot C_6H_5$	$C_{23}H_{28}BrNO$	48	Cr–1	+	71.98	159–164	66.66	6.81	3.38	66.01	7.20	3.43
43	C_6H_5	2	$N(CH_3)_2$	CH_3	$C_{11}H_{18}BrNO$	4w[g]	CH_3OH	+	84.90	165–167 [8, 9]	50.78	6.97	5.38	50.86	6.82	5.40
44	C_6H_5	3	$N(CH_3)_2$	CH_3	$C_{12}H_{20}BrNO$	30 min	CH_3OH	+	58.97	152–153 [8]	52.56	7.35	5.11	52.90	7.11	4.91
45	C_6H_5	4	$N(CH_3)_2$	CH_3	$C_{13}H_{22}BrNO$	4 w[g]	CH_3OH	+	75.70	172.5–174	54.17	7.69	4.86	54.45	7.85	5.00
46	C_6H_5	5	$N(CH_3)_2$	CH_3	$C_{14}H_{24}BrNO$	30 min	CH_3OH	+	75.16	175–179.5	55.63	8.00	4.63	56.10	8.34	4.67
47	4-$(CH_3)_3C \cdot C_6H_4$	4	$N(CH_3)_2$	CH_3	$C_{17}H_{30}BrNO$	4 w[g]	CH_3OH	+	58.80	135–139	59.30	8.78	4.07	59.26	8.94	3.97
48	3-$(CH_3O) \cdot C_6H_4$	4	$N(CH_3)_2$	CH_3	$C_{14}H_{24}BrNO_2$	15 min	Cr–2	+	89.80	89–91.5	52.83	7.60	4.40	52.12	7.63	4.28
49	2-$C_6H_5 \cdot C_6H_4$	4	$N(CH_3)_2$	CH_3	$C_{19}H_{26}BrNO$	1	CH_3OH	+	69.50	132–134	62.64	7.19	3.84	62.26	7.51	4.04
50	2-$C_{10}H_7$	4	$N(CH_3)_2$	CH_3	$C_{17}H_{24}BrNO$	4 w[g]	Cr–1	+	87.80	180–182	60.36	7.15	4.14	59.97	7.40	4.20

a) Compounds 1–42 prepared by general procedure 1; compounds 44, 46, 48 and 49 by general procedure 2, and compounds 43, 45, 47 and 50 by general procedure 3.
b) Very hygroscopic.
c) Moistening starts at considerably lower temperature.
d) Crystallized directly from the reaction concentrate.
e) Not obtained in crystalline form; not analyzed.
f) Yield calcd. on basis of Br^- content of final solution.
g) w = week.

Table III. *ω-Aryloxyalkyl Ammonium Compounds* (prepared by general procedure 1)



No.	R	Formula	Reaktion period. (hours)	Cryst.	Recryst.	Yield, %	M.p., °C	Calcd., %			Found, %		
								C	H	N	C	H	N
51	n-C_3H_7	$C_{22}H_{32}BrNO$	48	Cr–2	+	79.4	149–151	65.02	7.94	3.44	65.04	7.74	3.36
52	n-C_4H_9	$C_{23}H_{34}BrNO$	48	Cr–2	+	54.2	119–124	65.70	8.15	3.33	66.02	8.42	3.47
53	n-C_5H_{11}	$C_{24}H_{36}BrNO$	40	Cr–2	+	73.6	122–128	66.35	8.35	3.22	66.14	8.50	3.20
54	n-C_6H_{13}	$C_{25}H_{38}BrNO$	48	Cr–2	+	57.9	138–141	66.95	8.54	3.12	67.12	8.50	3.07
55	cy-C_6H_{11}	$C_{25}H_{36}BrNO$	48	Cr–1	+	73.3	177.5–180	67.25	8.13	3.14	66.60	8.22	3.44
56	n-C_7H_{15}	$C_{26}H_{40}BrNO$	48	Cr–2	+	73.5	136.5–138	67.52	8.72	3.03	67.87	8.79	3.30
57	n-C_8H_{17}	$C_{27}H_{42}BrNO$	40	Cr–1	+	78.3	133–136	68.05	8.88	2.94	67.73	8.82	2.94
58	n-C_9H_{19}	$C_{28}H_{44}BrNO$	48	Cr–1	+	79.5	110–115	68.55	9.04	2.85	68.85	9.22	3.00
59	n-$C_{10}H_{21}$	$C_{29}H_{46}BrNO$	48	Cr–1	+	80.3	118–122.5	69.03	9.19	2.78	69.99	9.36	3.06
60	n-$C_{11}H_{23}$	$C_{30}H_{48}BrNO$	48	Cr–1	+	79.4	123.5–127	69.48	9.33	2.70	69.24	9.36	2.90
61	n-$C_{12}H_{25}$	$C_{31}H_{50}BrNO$	48	Cr–1	+	90.2	115–118	69.90	9.46	2.63	69.69	9.57	2.88
62	n-$C_{14}H_{29}$	$C_{33}H_{54}BrNO$	48	Cr–1	+	77.5	98–101	70.69	9.71	2.49	70.35	9.32	2.65

Experimental[2]). – *1-Alkylpiperidines.* Eleven 1-alkylpiperidines (alkyl = n-C_nH_{2n+1} with n = 3 to 12, and 14) were prepared according to *Maklajev & Petrova* [3]; new compounds (alkyl, yield, b.p./Torr): n-nonyl, 71%, 158–160°/25; n-undecyl, 67.7%, 156–159°/6; n-tetradecyl, 75.5%, 170–171°/3. 1-Cyclohexylpiperidine was made according to *Smith & Macdonald* [4].

α-Bromo-ω-aryloxy-alkanes (new compounds, see Table I). Procedure see [1]. Phenols used: phenol, 2-methoxyphenol, 2-phenylphenol, β-naphthol, besides the phenols ArOH with Ar mentioned in table I. 4-Nonylphenol used: mixture of 2,4-dimethyl-4-(*p*-hydroxyphenyl)-heptane and 2,4-dimethyl-2-(*p*-hydroxyphenyl)-heptane.

ω-Aryloxyalkyl ammonium bromides (Tables II & III). – *General Procedure 1.* A mixture of the appropriate tertiary amine (0.11 mole) and the α-bromo-ω-aryloxy-alkane (0.1 mole) was refluxed in absolute ethanol (10 ml) for 8–48 h at 100–120°. Towards the end of reaction, the mixture became very viscous and slightly coloured. The solvent was removed under anhydrous conditions. The product was isolated by one of the methods for crystallization described below.

General Procedure 2. The α-bromo-ω-aryloxy-alkane (0.1 mole) was dissolved in a solution of trimethylamine (0.15 mole) in 100 ml absolute ethanol. The mixture was heated in an autoclave at 110–120° for 15 to 30 min, then the solvent and excess amine were evaporated. The solid residue was refluxed with activated charcoal in 100 ml methanol for 1 h, filtered and the filtrate concentrated to 50 ml. The quaternary bromide crystallized on cooling to 0°.

General Procedure 3. The α-bromo-ω-aryloxy-alkane (0.1 mole) was reacted at room temperature for 4 to 5 weeks with trimethylamine (0.15 mole) in 100 ml absolute ethanol, in a sealed bottle. The quaternary salt was recovered as described in general procedure 2.

Procedures for Crystallization (Cr-1 to Cr-5). – Cr-1. The crude reacted mixture was kept overnight at 0°. The crystals formed were filtered off, washed with dry acetone and dried over sodium hydroxide pellets.

Cr-2. When Cr-1 failed, the solvent and excess amine were evaporated under reduced pressure in anhydrous conditions. The residue was dissolved in a very small quantity of dry acetone and kept at room temperature.

Cr-3. When Cr-2 failed, more acetone was added to the solution, followed by slow addition of dry ether until the solution became slightly turbid. By adding a few drops of acetone a clear solution in acetone-ether mixture was obtained which was kept for a week at – 15 to – 25° C, for crystallization.

Cr-4. When Cr-3 failed, the solvents were removed under reduced pressure and a fresh quantity of dry acetone was added to the residue. The solution was cooled to – 50°C, then dry ether was slowly added. The precipitated product was filtered and dried.

Cr-5. When Cr-1 to Cr-4 failed, the following solvents were tried: ethyl acetate, methanol-ethyl acetate, isopropyl ether, 2-methoxy-ethanol, and dioxane, as described for Cr-2.

When crystallization failed, the solvents from Cr-5 were removed and the residue was dissolved in 200 ml of water. The solution was extracted three times with ether (100 ml each time). The aqueous layer was concentrated to 100 ml, decolourized with activated charcoal, and freeze dried. If no solid product could be obtained, the viscous mass was dissolved in a small quantity of water and this syrup was stored; the concentration of the quaternary ammonium salt was determined by assay of Br^-.

Recrystallization was from mixtures of methanol and acetone or ethyl acetate using activated charcoal where necessary.

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11. Über siliciumaromatische Verbindungen
III. Trimethylsilyl- und Triphenylsilyl-substituierte Farbstoffe der Anthrachinonreihe

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Summary. The influence of the presence of trimethylsilyl and triphenylsilyl groups in substituents of anthraquinone on the properties of anthraquinone vat, acid, and dispersion dyes was investigated.

In früheren Mitteilungen [1] [2] haben wir über Trimethylsilylphtalsäuren und davon abgeleitete Farbstoffe aus der Reihe der Phtaleine, Phtalocyanine und Chinophtalone berichtet. Die vorliegende Arbeit dehnt diese Untersuchung auf Anthrachinonfarbstoffe aus (s. auch [3] [4] [5]), und zwar sowohl auf Küpen- wie auf Säure- und Dispersionsfarbstoffe.

1. Küpenfarbstoffe. – Als einfachster Typ der Küpenfarbstoffe wurden die Acylamino-anthrachinone aus verschiedenen Amino-anthrachinonen und trimethylsilylsubstituierten Mono- und Dicarbonsäuren gewählt. Tabelle 1 zeigt die von uns benützten, teilweise neuen Carbonsäuren, Tabelle 2 die verwendeten Amino-anthrachinone.

Die Farbstoffe wurden in bekannter Weise durch Umsetzung der Carbonsäurechloride mit den Amino-anthrachinonen in siedendem *o*-Dichlorbenzol hergestellt.

Die zum Vergleich hergestellten Farbstoffe mit Acyl = *t*-Butylbenzoyl zeigten sämtlich eine wesentlich geringere Affinität zu Baumwolle sowie geringere Lichtechtheit. Verglichen mit den Farbstoffen ohne Silylreste lassen die Silylgruppen enthaltenden Farbstoffe in den UV.-Absorptionsspektren fast keine Verschiebung des Absorptionsmaximums erkennen (s. Figur).

Die Maxima liegen bei beiden Verbindungsarten, trimethylsilyl- und *t*-butylsubstituierten, praktisch an gleicher Stelle, 301–304 nm. Nur bei höheren Wellenlängen tritt eine leichte bathochrome Verschiebung von 5 nm bei den *t*-butyl-substituierten Verbindungen ein. Daraus ergibt sich, dass die Trimethylsilylgruppe und die *t*-Butylgruppe praktisch dieselbe Elektronendonatorwirkung auf den Kern bewirken.