Table I. Characterization Data of 1-Amino-4-anilinoanthraquinone 2-Ethers (Dyes III)

compd	R^1	R^2	synthesis method and recryst solv[a]	crude prod yield, %	λ_{max} (log ϵ) in C_6H_5Cl[b]	mp, °C
IIIa	H	H			454 s (3.93), 573 (4.11), 610 (4.12)	
IIIb	Br	H			584 (4.12), 621 (4.11)	
IIIc	OC_6H_5	H	1A	75.7	528 s (3.96), 536 (4.18), 600 (4.19)	189–190
IIId	$OC_6H_4CH_3$-*p*	H	1A	73.8	530 s (3.93), 562 (4.15), 600 (4.16)	168
IIIe	$OC_6H_4OCH_3$-*p*	H	1A	94.5	534 s (4.02), 562 (4.20), 600 (4.21)	184
IIIf	$OOCH_3$-*m*	H	2A	69.0	534 s (3.92), 564 (4.12), 600 (4.13)	120
IIIg	OC_6H_4OH-*p*	H	1A	80.3	534 s (3.94), 562 (4.14), 600 (4.14)	220
IIIh	$OC_6H_4NH_2$-*p*	H	1A	128.0	534 s (3.94), 561 (4.15), 600 (4.16)	202
IIIi	$OC_6H_4C(CH_3)_2$-*p*-C_6H_4OH-*p*	H	1A	54.7	536 s (3.90), 564 (4.15), 600 (4.16)	110
IIIj	OCH_3	H	3B	126.0	530 s (4.00), 552 (4.18), 582 (4.20)	165
IIIk	OCH_3	CH_3	3C	111.0	530 s (4.00), 554 (4.19), 5.93 (4.21)	118
IIIl	OC_6H_5	CH_3	1B	87.4	568 (3.15), 604 (4.15)	148
IIIm	$OC_6H_4CH_3$-*p*	CH_3	1C	85.7	566 (4.16), 602 (4.18)	194
IIIn	$OC_6H_4OCH_3$-*p*	CH_3	1C	81.8	534 s (3.91), 565 (4.16), 602 (4.18)	200
IIIo	OCH_3	OCH_3	1A	103.0	530 s (3.90), 556 (4.09), 592 (4.11)	118
IIIp	OC_6H_5	OCH_3	1A	82.5	536 s (3.98), 568 (4.18), 600 (4.18)	128
IIIq	$OC_6H_4CH_3$-*p*	OCH_3	1A	94.3	536 s (3.98), 602 (4.18), 602 (4.18)	182
IIIr	$OC_6H_4OCH_3$-*p*	OCH_3	1C	86.4	536 s (4.00), 568 (4.18), 600 (4.18)	204

[a] A = acetone. B = ethanol. C = toluene. [b] S = shoulder.

g), mp 165–166 °C. The compound was confirmed by mass spectrometry and elemental analysis.

The other bands were also isolated and their structures confirmed by mass spectrometry. Similarly prepared from appropriate bromoaryl compounds were other dyes (III) listed in Table I.

Registry No. I (X = Br), 81-49-2; II (R = H, X = CH_3O), 2872-48-2; IIIa, 4395-65-7; IIIb, 1564-71-2; IIIc, 86397-46-8; IIId, 90791-31-4; IIIe, 90791-32-5; IIIf, 90791-33-6; IIIg, 90791-34-7; IIIh, 90791-35-8; IIIi, 90791-36-9; IIIj, 90791-37-0; IIIk, 90791-38-1; IIIl, 90822-50-7; IIIm, 90791-39-2; IIIn, 90791-40-5; IIIo, 90791-41-6; IIIp, 90791-42-7; IIIq, 90791-43-8; IIIr, 90791-44-9; $C_6H_5NH_2$, 62-53-3; C_6H_5OH, 108-95-2; *m*-$CH_3OC_6H_4OH$, 150-19-6; C_6H_5Br, 108-86-1.

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Synthesis of Some Bis(ammonium) Salts of 2,4-Dinitro-3-(2-furyl)glutarodinitrile

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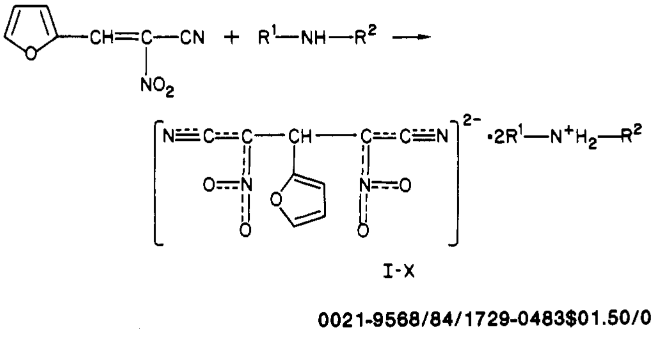
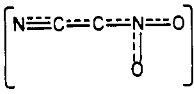
A number of bis(ammonium) salts of 2,4-dinitro-3-(2-furyl)glutarodinitrile are synthesized from 1-nitro-1-cyano-2-(2-furyl)ethene via reaction with aliphatic and alicyclic amines. The structure of the products is supported by IR and NMR spectra.

In continuation of our investigations on the reaction of cyanonitroalkenes with amines (*1–4*), we carried out the reaction of 1-nitro-1-cyano-2-(2-furyl)ethene (*5*) with aliphatic and alicyclic amines and obtained the corresponding bis(ammonium) salts of 2,4-dinitro-3-(2-furyl)glutarodinitrile (I–X).



I-X

The structures of the compounds follow from elemental analyses, spectroscopic data (Table I), and some chemical reactions. There are several absorption bands in the IR spectra in the region 1470–1080 cm^{-1} and a band at 2210–2195 cm^{-1} which correspond to the vibrations of the conjugated anionic system (*1–4*, *6*, *7*)



This anion is also confirmed by the presence of the three furyl protons at δ 7.40, 6.24, and 5.96 and one proton at δ 5.40 in NMR spectra. The presence of ammonium counterion is proved by the broad absorption band at 3300–2600 cm^{-1} in IR spectra (*6*, *8*), by a signal at δ 7.76 or 8.40 (D_2O exchanged) in NMR spectra, and also by acidification of the salts with excess hydrobromic acid, when the respective ammonium bromides were obtained quantitatively.

All salts are crystalline solids, easily soluble in water and polar solvents, insoluble in ether, benzene, chloroform, tetrachloromethane, and petroleum ether. They must be kept in dark

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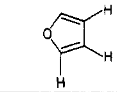
Table I. Bis(ammonium) Salts of 2,4-Dinitro-3-(2-furyl)glutarodinitrile[a]

					IR (in KBr) ν, cm^{-1}		
no.	R^1	R^2	yield, %	mp, °C	$N{\equiv}C$	^+NH_n	$[NC{=}C{=}NOO]^-$
I	H	n-C_3H_7	98.0	117–118	2205	3300–2700	1460, 1310, 1230, 1080
II	H	n-C_4H_9	95.9	108–109	2195	3200–2700	1470, 1310, 1230, 1080
III	H	$C(CH_3)_3$	60.0	155–167	2205	3300–2700	1440, 1330, 1245, 1090
IV	H	C_6H_{11}	80.3	138–139	2200	3200–2800	1460, 1320, 1240, 1100
V	H	C_8H_{17}	70.9	119–120	2210	3300–2800	1480, 1320, 1230, 1080
VI	H	$C_{12}H_{25}$	71.0	118–119	2210	3300–2700	1470, 1320, 1230, 1080
VII	H	$C_{16}H_{33}$	71.0	121–122	2210	3300–2800	1470, 1320, 1240, 1080
VIII	H	$C_{17}H_{35}$	73.3	108–110	2210	3300–2800	1470, 1320, 1240, 1080
IX	C_2H_5	C_2H_5	75.3	129–130	2195	3200–2700	1465, 1320, 1225, 1090
X	$CH_2{-}CH_2{-}CH_2$ $CH_2{-}\quad{-}CH_2$		82.9	129–130	2195	3100–2600	1460, 1310, 1210, 1100

			NMR δ				
no.	R^1	R^2	CH_3	CH_2	CH		^+NH_n
III	H	$C(CH_3)_3$	1.22 (s, 18 H)		5.40 (s, 1 H)	5.96 (d, 1 H) 6.24 (t, 1 H) 7.40 (d, 1 H)	7.76 (s, 6 H)
IX	C_2H_5	C_2H_5	1.16 (t, 12 H)	2.88 (q, 8 H)	5.40 (s, 1 H)	5.96 (d, 1 H) 6.24 (t, 1 H) 7.40 (d, 1 H)	8.40 (s, 4 H)

[a] Elemental analyses (C, H, N) in agreement with theoretical values have been obtained and were submitted for review.

containers at 0–5 °C, because of their decomposition at room temperature in daylight.

Experimental Section

A solution of 1-nitro-1-cyano-2-(2-furyl)ethene (0.01 mol) in dioxane is added in drops to the solution of freshly distilled amine (0.01 mol) in 60 cm^3 of diethyl ether and 10 cm^3 of methanol at room temperature with vigorous stirring. About 10 min after, the precipitate separated is filtered and washed with diethyl ether. The obtained bis(ammonium) salts of 2,4-dinitro-3-(2-furyl)glutarodinitrile are purified through reprecipitation with ether from methanolic solution.

Registry No. I, 81548-95-0; II, 81548-96-1; III, 81548-97-2; IV, 81548-98-3; V, 91328-21-1; VI, 91328-22-2; VII, 91328-23-3; VIII, 91328-24-4; IX, 81548-99-4; X, 81549-00-0; 1-nitro-1-cyano-2-(2-furyl)ethene, 67075-62-1; 1-propanamine, 107-10-8; 1-butanamine, 109-73-9; 2-methyl-2-propanamine, 75-64-9; cyclohexylamine, 108-91-8; 1-octanamine, 111-86-4; 1-dodecanamine, 124-22-1; 1-hexadecanamine, 143-27-1; 1-heptadecanamine, 4200-95-7; *N*-ethylethanamine, 109-89-7; piperidine, 110-89-4.

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Correction

Densities of the Molten Salt System Cesium Bromide–Cuprous Bromide. Gert J. Lamprecht* and G. D. Peckham, *J. Chem. Eng. Data* **1977**, *22*, 329.

This paper was originally published with a single author. G. D. Peckham should have been cited as a coauthor.