

TABLE I

Substituted 2-pyrrolidone R	R ¹	R ²	Yield, %	B. p., °C.	Mm.	Formula	Nitrogen, % ^a	
							Calcd.	Found
Dodecyl ^b	H	H	83	202-205	11	C ₁₆ H ₃₁ NO	5.5	5.8
Tridecyl ^c	H	H	61	210-213	9.5	C ₁₇ H ₃₃ NO	5.2	5.3
Tetradecyl ^d	H	H	72	201-201.5	2.5	C ₁₈ H ₃₅ NO	5.0	5.0
Hexadecyl ^d	H	H	72	213-215	2.5	C ₂₀ H ₃₉ NO	4.5	4.5
Octadecyl ^{b,d}	H	H	78	219-221	1.5	C ₂₂ H ₄₃ NO	4.2	4.0
Benzyl ^b	H	H	33	168-170	14	C ₁₁ H ₁₃ NO	8.0	7.9
4-Diethylamino-1-methylbutyl	H	H	77	162-166	11	C ₁₃ H ₂₅ N ₂ O	12.4	12.4
Dodecyl	H	CH ₃	56	185-187	3.5	C ₁₇ H ₃₃ NO	5.2	5.1
Tetradecyl	H	CH ₃	45	189-190	1.5	C ₁₉ H ₃₇ NO	4.7	4.8
Hexadecyl ^d	H	CH ₃	31	200-201	1	C ₂₁ H ₄₁ NO	4.3	4.2
Octadecyl ^d	H	CH ₃	33	219-222	1.2	C ₂₃ H ₄₅ NO	4.0	3.8
Dodecyl	CH ₃	C ₂ H ₅	48	162-172	4	C ₁₉ H ₃₇ NO	4.7	4.9

^a The analyses were performed by Mrs. J. D. Nevins and Mrs. R. C. Schropp of the Monsanto Analytical Laboratory. ^b Preparation mentioned, but compound not described, in ref. (1b). ^c Prepared by Mr. H. L. Morrill. ^d Waxy solid.

tested *in vitro*, were found to have no bacteriostatic activity. The 2-pyrrolidones with the dodecyl, tetradecyl, octadecyl and 4'-diethylamino-1'-methylbutyl groups in the 1-position were inactive against experimental tuberculosis in guinea pigs. These products were evaluated for chemotherapeutic and pharmacologic action in The Lilly Research Laboratories.

Experimental

Preparation of 2-Pyrrolidones.—Equimolar quantities of the amine and the lactone were heated with agitation at 110-130° for about three hours and then at 250-270° for three to six hours while distilling off water. The excess reactants were distilled off under reduced pressure and the N-substituted pyrrolidone was distilled.

Long chain amines,² benzylamine and 1-diethylamino-4-aminopentane reacted satisfactorily, giving 35-85% yields of the corresponding pyrrolidones.

γ -Butyrolactone, γ -valerolactone and γ -ethyl- γ -valerolactone³ reacted without difficulty with the amines used, although it is clearly seen that under the same conditions γ -valerolactone gives lower yields than does γ -butyrolactone.

The several pyrrolidones prepared were high-boiling liquids or waxy solids.

(2) Gift from Armour and Company. Tridecylamine was prepared in 55% yield by Dr. E. L. Hatlelid using the procedure of Ralston, Selby, Pool and Potts, *Ind. Eng. Chem.*, **32**, 1093 (1940); the dodecylamine (35%) obtained as a by-product boiled at 240-242° (8 mm.), while Hoerr, Harwood and Ralston, *J. Org. Chem.*, **9**, 201 (1944), reported m. p. 56.5°. Didodecylamine, b. p. 263-265° (27 mm.), was obtained in a preparation of dodecylamine; Wibaut, Heierman and Wagtenonk, *Rec. trav. chim.*, **57**, 456 (1938), reported 195° (0.7 mm.).

(3) Grignard, *Compt. rend.*, **135**, 629 (1902). See Cason, Adams, Bennett and Register, *This Journal*, **66**, 1764 (1944), for an improved method of making γ, γ -dialkyl-butyrolactones.

MONSANTO CHEMICAL CO.
RESEARCH LABORATORIES
ST. LOUIS 4, MISSOURI

RECEIVED OCTOBER 21, 1946

NEW COMPOUNDS

Cadalene and Eudalene Trinitrotoluates

Cadalene (50 mg.) and trinitrotoluene (58 mg.) were heated in methanol solution on the water-bath for a few minutes. On cooling, long yellow glistening needles of

cadalene trinitrotoluate crystallized out, m. p. 83°, not raised by further crystallization from methanol.

Anal. Calcd. for C₁₅H₁₈·C₇H₅N₃O₆: C, 62.12; H, 5.45. Found: C, 62.04; H, 5.56.

Eudalene trinitrotoluate, similarly prepared from eudalene (87 mg.) and trinitrotoluene (103 mg.), formed short, dull yellow needles, m. p. 62-63°, not raised by further crystallization.

Anal. Calcd. for C₁₄H₁₆·C₇H₅N₃O₆: C, 61.31; H, 5.15; N, 10.21. Found: C, 61.02; H, 5.15; N, 10.4.

We are indebted to Mr. A. R. Penfold for a specimen of eudesmol from which the eudalene was prepared by dehydrogenation, to the Chemical Society and the Australian and New Zealand Association for the Advancement of Science for grants and one of us (W. I. T.) for a Duffus Lubbecki Scholarship.

The analyses are by Drs. Weiler and Strauss.

AUCKLAND UNIVERSITY COLLEGE LINDSAY H. BRIGGS
AUCKLAND, NEW ZEALAND WILLIAM I. TAYLOR

RECEIVED NOVEMBER 5, 1946

2-Arylamino-4-chlorobenzoic Acids and 9-Chloroacridines⁴

2-Anilino-4-chlorobenzoic Acid, C₈H₅NHClO₂.—A mixture of 153 g. (0.8 mole) of 2,4-dichlorobenzoic acid, 93 g. (1 mole) of freshly distilled aniline, 111 g. (0.8 mole) of anhydrous potassium carbonate, 4 g. of copper bronze and 600 ml. of *n*-pentyl alcohol was heated under reflux with stirring for five hours. One hundred ml. of 35% potassium hydroxide solution was added, and the excess aniline and pentyl alcohol was removed by steam distillation. The residue was filtered and acidified (concentrated hydrochloric acid). The purple solid which separated was removed by filtration and washed with water. Crystallization of the moist product from alcohol gave purple needles, and a second recrystallization from benzene gave 105 g. (53% yield) of slightly grey needles melting at 201°, cor.

Anal. Calcd. for C₁₃H₁₀ClNO₂: Cl, 14.31; neut. equiv., 247.7. Found: Cl, 14.08; neut. equiv., 247.6, 248.5.

2-*o*-Toluidino-4-chlorobenzoic Acid.—Similar directions were followed. From 0.8 mole of 2,4-dichlorobenzoic acid and *o*-toluidine was obtained 75 g. (36%) of the desired acid, m. p. 208°, cor. after crystallization from alcohol.

Anal. Calcd. for C₁₄H₁₂ClNO₂: Cl, 13.54; neut. equiv., 261.7. Found: Cl, 13.40; neut. equiv., 262.1, 260.3.

(1) This report is based on work done under contracts, recommended by the National Defense Research Committee and the Committee on Medical Research, between the Office of Scientific Research and Development and Northwestern University.