Procedure. Formaldehyde was generally used in 37% aqueous solution (Formalin) while chloral was used as its hydrate. The nitroalkane, carbonyl compound, and amine were mixed and added to the dropping funnel. Although triethylamine was usually employed, a number of other amines were tested. Thus, the following amines gave the indicated optimum conversion of 1-nitropropane to 2-nitro-1-butene: *n*-butylamine, 3.1%; diethylamine, 7.0%; piperidine, 11.6%; benzyltrimethylammonium hydroxide, 13.8%; pyridine, 3.6%; triethylamine, 18.0%; (all the above using 0.45 mole per cent of amine), and triethylamine, 40.2%; trimethylamine, 38.0%; tributylamine, 3.2%; tribenzylamine, 9.2% (using 0.23 mole per cent of amine). With tertiary amines warming occurred and the mixtures became homogeneous. With other amines the mixture usually remained heterogeneous and had to be stirred continuously throughout the run. With the reactor at the proper internal temperature as measured by a thermocouple the reactants were added dropwise at a rate of about 1 mole of nitroalkane per 20 min. At the end of the run the product was removed from the receiver, mixed with 200 cc. of ether, the mixture washed successively with 10% hydrochloric acid, saturated aqueous sodium bicarbonate solution, and water, and dried over sodium sulfate and fractionally distilled, eventually under vacuum. The fraction boiling in the nitro alkane–nitro olefin range was collected and weighed and its refractive index determined. From these data were calculated the per cent conversions. In the cases of the formaldehyde condensation products, a 5.0-g. aliquot of the fraction was dissolved in a solution containing 5.4 g. of *p*-toluidine in 20 ml. of 95% ethanol cooled to 0°. The mixture became warm and turned red in color. It was heated for 10 min. on a steam bath and allowed to stand overnight. The yellow crystals which formed were dried and weighed and then recrystallized from ethanol to obtain an accurate melting point. The conversions to these derivatives of the nitro olefins varied with the percentage of the nitro olefin present in the product mixture and averaged about 70% in a typical mixture containing 85% nitro olefin.

In the cases of the chloral condensation products a 0.02 mole equivalent of the nitro olefin fraction was dissolved in 25 ml. of 95% ethanol, heated to boiling, and phenylhydrazine, 1.8 g. (0.02 mole), added dropwise. The solution turned red in a few seconds. Water was added dropwise until the solution became faintly cloudy and the mixture was allowed to cool. The yellow crystals obtained were dried, weighed, and recrystallized from ethanol to obtain an accurate melting point. The conversions to these derivatives of the trichloronitro olefins were as follows: propene, 99%; butene, 99%; pentene, 79%; hexene, 51%. All of the chloral condensation products gave a positive test[9] for the $-CCl_3$ group.

Nitro alcohols and their acetate esters. The nitro alcohols and esters prepared in the liquid phase were obtained by published procedures.[10]

Nitro olefins. (*a*) *By dehydration of nitro alcohols with phthalic anhydride.* The procedure of Buckley and Scaife[11] gave good results in all cases except with 1,1,1-trichloro-3-nitro-2-hexanol. In this case a small yield of the olefin was obtained by dehydration with phosphorous pentoxide.[6]

(*b*) *By pyrolysis of the acetate esters.* The procedure of Gold[12] gave good results except again in the case of 1,1,1-trichloro-3-nitro-2-hexyl acetate which gave no nitro olefin.

Lafayette, Ind.

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[9] L. H. W. Doughty, *J. Am. Chem. Soc.*, 41, 1129 (1919).

[10] H. B. Hass and B. M. Vanderbilt, *Ind. Eng. Chem.*, 32, 34 (1940).

[11] G. D. Buckley and C. W. Scaife, *J. Chem. Soc.*, 1471 (1947).

[12] M. H. Gold, *J. Am. Chem. Soc.*, 68, 2544 (1946).

[Contribution from the Organic Chemistry Department, Research Division, Abbott Laboratories]

Effect of Nuclear Substitution on the Reaction of Aromatic Amines with Ethylene Oxide

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A study was made of the effect of nuclear substitution in aromatic amines on their reactivity with ethylene oxide. The steric effects of some *ortho* substituents are noted. Seventeen new *N*,*N*-bis(2-hydroxyethyl) aromatic amines are reported.

The need in this laboratory for some *N*,*N*-bis-(2-hydroxyethyl)anilines gave us the opportunity to observe the effect of nuclear substitution on the reaction of aromatic amines with ethylene oxide. Our interest had been aroused by the work of Ross[1] who reported successful reactions with *o*-, *m*- and *p*-toluidine and *o*- and *p*-anisidine at 90° for sixteen hours. He found it necessary to raise the temperature to 150° to get bis substitution with *p*-chloroaniline, but reported failure with both *o*- and *m*-chloroaniline at this temperature. As a result of these reactions and those of other aromatic amines in his work, he suggested that bishydroxyethylation was dependent on the basicity of the aromatic amine.

The reported values of the base strength of the chloroanilines,[2] showing that *p*-chloroaniline is the strongest base of the group and *o*-chloroaniline the weakest base, seem to support Ross' premise.

Using base strength as a criterion of reactivity, the observed values of *o*-anisidine and *o*-phenetidine[3] would suggest little difference in reaction of these compounds with ethylene oxide. From a study of molecular models, however, it appeared that steric effects would be a factor.

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[1] W. C. J. Ross, *J. Chem. Soc.*, 183 (1949).

[2] J. M. Vandenbelt, *Anal. Chem.*, 26, 726 (1954).

[3] N. F. Hall and M. R. Sprinkle, *J. Am. Chem. Soc.*, 54, 3469 (1932).

In order to compare the effects of the various substituents, we used a modification of the work of Gabel[4] taking as a standard reaction of the aromatic amine with two equivalents of ethylene oxide for five hours at 90–100°. It was our opinion that the long heating period used by Ross would fail to show any subtle differences in the effect of the methoxy and ethoxy groups (and perhaps slightly larger ones) in the *ortho* position on reactivity.

At the same time to study the correlation between base strength and the ease of obtaining bis addition, the relative order of basicity of some of the amines was determined (see Table I.)

TABLE I

MILLIVOLTAGE HALF-NEUTRALIZATION VALUES[a]

Anilines[b]	E 1/2 (mv.)[c]
o-Ethoxy	298
p-Phenoxy	300
o-Methoxy	305
o-Allyloxy	315
o-Butoxy	325
o-Phenoxy	375
p-Chloro	378
m-Chloro	404
o-Methylmercapto	405
o-Fluoro	435
m-Trifluoromethyl	440
p-Trifluoromethyl	456
o-Chloro	464

[a] Essentially, the method of H. K. Hall, Jr., *J. Phys. Chem.*, **60**, 63 (1956) was followed. Acetonitrile (Eastman Kodak Practical Grade) was used as solvent, the titrant 0.1*N* perchloric acid in *p*-dioxane. [b] The purity of the anilines was 97–100%. [c] The instrument used was a Precision-Dow Recordomatic Titrimeter.

In the reaction of the chloroanilines, we found that *m*- and *p*-chloroaniline each formed a bis product (XVII and XVIII, Table II) at 90°. Formation of XVI from *o*-chloroaniline required more rigorous conditions. According to the values in Table I, if reactivity is dependent on base strength, it would be expected that those amines above *p*-chloroaniline would form bis products on treatment with ethylene oxide at 90°. However, our results show that as the -OR group in the *ortho* position increased in bulk, bis addition became increasingly difficult (compare VI, VIII, IX, XII in Table II). When some of these same groups were in the *para* position, normal reaction under our standard conditions took place (see VII, XI, XIII in Table II). Steric effects were also noted in the reaction of *o*-methylmercaptoaniline with ethylene oxide. While less basic than its oxygen analog, *o*-anisidine, it is of the same order as *m*-chloroaniline and more basic than *m*- and *p*-trifluoromethylaniline, each of which reacted with ease. The *o*-fluoro compound, which is slightly more basic than either of the trifluoromethyl compounds listed in Table I, also required longer heating and higher temperature.

It would appear then from the results of this study, that in the reaction of ethylene oxide with such a resonating system as an aromatic amine steric effects as well as electronic effect must be taken into consideration.

EXPERIMENTAL[5]

The nitrophenyl ethers used as intermediates in this work were commercially available or prepared by the known literature method.[6]

2-Aminophenyl cyclohexyl ether. Thirty grams (0.136 mole) of 2-nitrophenyl cyclohexyl ether (b.p. 155–158° (0.3 mm.), n_D^{23} 1.5310) dissolved in 75 cc. of ethanol was hydrogenated under 2.5 atm. in the presence of 1.5 g. of 5% palladium on activated carbon. Reaction was exothermic and was complete in about 30 min. After the solvent was removed, benzene was added to the residue and the mixture distilled to remove water azeotropically. The base crystallized on standing and was recrystallized from pentane. Yield of product melting at 57.5–58° was 57%.

Anal. Calcd. for $C_{12}H_{17}NO$: C, 75.35; H, 8.96; N, 7.32. Found: C, 75.50; H, 9.12; N, 7.50.

A *hydrochloride salt* was obtained; m.p. 179–181°.

Anal. Calcd. for $C_{12}H_{18}ClNO$: C, 63.28; H, 7.96; N, 6.15. Found: C, 63.10; H, 8.04; N, 6.17.

This procedure gave high yields for the preparation of 2-phenoxyaniline (93%), 4-phenoxyaniline (quantitative), 2-butoxyaniline (87%),[6] 3-butoxyaniline,[6] and 4-butoxyaniline[6] (almost quantitative). The reduction of 2-nitrophenyl allyl ether could not be carried out catalytically with palladium, platinum, or nickel catalysts. In every case, hydrogenolysis occurred with *o*-aminophenol and *o*-nitrophenol as resultant products. Chemical reduction yielded the desired amine.[7] 2-Fluoroaniline, 4-fluoroaniline, and 2-methylmercaptoaniline are commercially available.[8]

p-Trifluoromethylnitrobenzene. A mixture of 83.5 g. (0.5 mole) of *p*-nitrobenzoic acid and 215 g. (2.0 moles) of sulfur tetrafluoride was heated for 7 hr. at 175° in a 1 l. stainless steel bomb equipped with a platinum gasket. After cooling and venting the gases, the mixture in the bomb was treated with chloroform and filtered from the insoluble product.[9] The chloroform solution was extracted with aqueous sodium bicarbonate to remove any nitrobenzoic acid and was then washed with water and dried. After removal of solvent, the product weighed 75.0 g. (78.5% yield) and melted at 47–48° (lit. m.p. 41–43°).[10]

Anal. Calcd. for $C_7H_4F_3NO_2$: N, 7.33. Found: N, 7.28.

When conditions described by Hasek[10] were used, yields were 56–70%.

3-Trifluoromethylnitrobenzene. In a similar experiment with *m*-nitrobenzoic acid at 160° for 7 hr., a 68.5% yield was ob-

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(4) G. O. Gabel, *Ber.*, **58**, 577 (1925).

(5) Microanalyses by E. F. Shelberg and O. Kolsto and infrared examination by W. Washburn of Abbott Laboratories.

(6) C. F. H. Allen and J. W. Gates, Jr., *Org. Syntheses*, **Coll. Vol. III**, 140 (1955). Of the less frequently encountered ethers 2-nitrophenyl allyl ether [b.p. 80° (0.3 mm.), n_D^{26} 1.5632] was obtained in 75% yield.

(7) J. von Braun and O. Braunsdorf, *Ber.* **54**, 702 (1921). Dr. J. H. Short of this laboratory will offer an improved chemical reduction in a later publication.

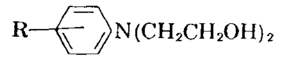
(8) Aldrich Chemical Co., Milwaukee, Wis.

(9) This material melts at 145–146°. It is probably *p*-nitrobenzoyl fluoride. We were never able to get a satisfactory analysis and did not pursue its identification any further.

(10) W. R. Hasek, W. C. Smith, and V. A. Engelhardt, *J. Am. Chem. Soc.*, **82**, 543 (1960).

TABLE II

BIS(2-HYDROXYETHYL)ANILINES



	R	Reaction Conditions^{a}	Yield, %	B.P.	Mm.	n_D^{25}	Formula	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Nitrogen, % Calcd.	Nitrogen, % Found
I	2-CH_3	A	91	166^{i}	1	1.5448	$C_{11}H_{17}NO_2$	67.88	67.41	8.78	8.79	7.13	7.10
II	4-CH_3	A	76	162–163^{i}	0.75–0.85	1.5667	$C_{11}H_{17}NO_2$						
III	2-OCH_3	A	94	165^{i}	1.3	1.5515	$C_{11}H_{17}NO_3$	62.53	62.40	8.11	8.58		
IV	3-OCH_3	A	78	195	0.3	1.5718	$C_{11}H_{17}NO_3$	62.53	62.39	8.11	8.22	6.63	6.67
V	4-OCH_3	A	83.5	^{j}			$C_{11}H_{17}NO_3$						
VI	2-OCH_2H_5	B^{b}	93	168–170	0.8	1.5363	$C_{12}H_{19}NO_3$	63.97	63.49	8.59	8.60	6.22	6.12
VII	4-OCH_2H_5	A	94.5	182	0.3	1.5583	$C_{12}H_{19}NO_3$	63.97	63.98	8.50	8.64	6.22	6.05
VIII	2-$OCH_2CH=CH_2$	B	77.5	195	0.3	1.5516	$C_{13}H_{19}NO_3$	65.80	65.17	8.07	8.20	5.90	6.33
IX	2-OC_4H_9	B^{c}	90.8	161–163	0.3	1.5273	$C_{14}H_{23}NO_3$	66.37	66.08	9.15	9.01	5.53	5.63
X	3-OC_4H_9	A	75.5	195–206	0.3	1.5492	$C_{14}H_{23}NO_3$	66.37	66.24	9.15	9.41	5.53	5.62
XI	4-OC_4H_9	A	90	195	0.3		$C_{14}H_{23}NO_3$	66.37	66.43	9.15	9.22	5.53	5.71
XII	2-OC_6H_5	C^{d}	67	207–216	0.4–0.5	1.5870	$C_{16}H_{19}NO_3$	70.30	70.30	7.01	6.89	5.13	5.06
XIII	4-OC_6H_5	A	86	^{k}			$C_{16}H_{19}NO_3$	70.30	70.49	7.01	6.96	5.13	5.05
XIV	2-OC_6H_{11}	D	67	194–196	0.3	1.5422	$C_{16}H_{25}NO_3$	68.78^{q}	68.62	9.02	9.18	5.01	5.09
XV	2-SCH_3	E^{e}	88.5	172–178	0.4–0.5	1.5848	$C_{11}H_{17}NO_2S$	58.11	57.51	7.54	7.57	6.16	5.75
XVI	2-Cl	E^{f}	90	162–168	0.3	1.5576	$C_{10}H_{14}ClNO_2$	55.68	55.91	6.54	6.45	6.50	6.46
XVII	3-Cl	A	97	^{l}			$C_{10}H_{14}ClNO_2$	55.68	55.93	6.54	6.68	6.50	6.48
XVIII	4-Cl	A	94	^{m}			$C_{10}H_{14}ClNO_2$	55.68	55.73	6.54	6.52	6.50	6.39
XIX	2-F	B^{g}	81.4	172–176	1.3–1.5	1.5440	$C_{10}H_{14}FNO_2$	^{r}				7.03	6.81
XX	4-F	A	90	^{n}			$C_{10}H_{14}FNO_2$	60.28	60.15	7.08	6.99	7.03	7.01
XXI	2-CF_3	E^{h}	45	165–172	1.0	1.4933	$C_{11}F_{14}F_3NO_2$	^{r}				5.62	5.46
XXII	3-CF_3	A	67	163^{o}	0.3	1.5150	$C_{11}H_{14}F_3NO_2$	53.00	53.29	5.66	5.72	5.62	5.55
XXIII	4-CF_3	A	95	^{p}			$C_{11}H_{14}F_3NO_2$	53.00	53.52	5.66	5.58	5.62	5.27

[a] No solvent used except where indicated. A, 5 hr., 90–100°; B, 18 hr., 125°; C, 26 hr., 180° in benzene; D, 24 hr., 200°; E, 15 hr., 150°. [b] When reaction of *o*-phenetidine and ethylene oxide was carried out at 90–100° for 5 hr., a 63% yield of VI was obtained in addition to 22% of a product which was a 1:1 mixture of VI and the monohydroxyethyl derivative. [c] When reaction was carried out as in A, 41% of IX was obtained plus 33% of the monohydroxyethyl derivative. B.p., 140–150° (0.5–0.3 mm.), m.p. 61–62° from cyclohexane. *Anal.* Calcd. for $C_{12}H_{19}NO_2$: C, 68.86; H, 9.15; N, 6.69. Found: C, 69.07; H, 9.32; N, 6.80. [d] Fifteen hours heating at 150° gave the monohydroxyethyl product in 36% yield, b.p. 139–146° (1 mm.), m.p. 44–46°. *Anal.* Calcd. for $C_{14}H_{15}NO_2$: N, 6.11. Found: 6.38. Subsequent fractions were mixtures. [e] Under reaction conditions A 80% of starting material was recovered. [f] When *o*-chloroaniline was treated with ethylene oxide for 18 hr. at 90°, a 45% yield of monohydroxyethyl compound was obtained boiling at 143–149° (1.7 mm.). *Anal.* Calcd. for $C_8H_{10}ClNO$: N, 8.16. Found: 7.86. [g] At 90–100° for 5 hr., 64% of starting material was recovered. The monohydroxyethyl derivative, b.p. 172° (30 mm.), n_D^{25} 1.5500 was obtained in 12% yield. *Anal.* Calcd. for $C_8H_{10}FNO$: N, 9.02. Found: N, 8.97. [h] Recovered 90% of starting material using condition A. With B, 22% of XXI was obtained in addition to 24% of monohydroxyethyl derivative, b.p. 106–117° (0.9–1.2 mm.), n_D^{25} 1.5003. *Anal.* Calcd. for $C_9H_{10}F_3NO$: N, 6.82. Found: N, 6.62. [i] W. C. J. Ross, *J. Chem. Soc.*, 183 (1944) gives only melting point. [j] Isolated as a solid, m.p. 70–71°. *See ref*[i]. R. M. Anker, A. H. Cook, and I. M. Heilbron, *J. Chem. Soc.*, 917 (1945) report 73°. [k] M.p. 98–100° from benzene and petroleum ether (b.p. 60–68°). Described, m.p. 98°. J. L. Everett and W. C. J. Ross, *J. Chem. Soc.*, 1972 (1949). [l] M.p. 90–92°. Prepared by J. B. Dickey, U.S. Patent 2,618,631 by treating *m*-chloroaniline and ethylene oxide for 12 hr. at 185–190°. M.p. 88–92°. [m] M.p. 96–97° from isopropyl alcohol–pentane. Described, m.p. 95–96°. *See ref*[i]. [n] Solid, m.p. 54–55° from benzene–petroleum ether (b.p. 60–68°). [o] Solidifies and melts at 95–96°. [p] Product polymerizes on distillation. Analysis reported on undistilled product. [q] Also, analyzed for oxygen. *Anal.* Calcd., 17.19. Found: 17.50. [r] Identified by infrared examination.

tained. The product distilled at 93–96° (18 mm.),[11] n_D^{24} 1.4718 and solidified and melted at 59–61°.

4-Trifluoromethylaniline. A solution of 30 g. (0.157 mole) of 3-trifluoromethylnitrobenzene was hydrogenated under 2.5 atm. in the presence of Raney nickel. After hydrogen uptake was complete (1.5 hr.), the solution was filtered from the catalyst. After removal of solvent, the residues from this experiment and a second run were distilled at 84–86° (18 mm.), n_D^{25} 1.4775. The combined yield amounted to 48.0 g. (95%).[12]

In a similar manner, 4-trifluoromethylaniline b.p. 102–102.5° (30 mm.), n_D^{22} 1.4831 was obtained in 80% yield; lit. b.p. 117° (40 mm.).[12]

When palladized charcoal was used as catalyst in these reductions, residual poisons (possibly from sulfur tetrafluoride) appeared to retard hydrogenation. Several portions of catalyst had to be added in order for hydrogen uptake to be completed.

N,N-Bis(2-hydroxylethylated) aromatic amines. N,N-Bis-(2-hydroxyethyl)-2-methoxy-1-naphthylamine. Two-tenths mole (34.6 g.) of 2-methoxy-1-naphthylamine[13] and 21 cc. (0.42 mole) of ethylene oxide were placed in a stainless steel rocker type bomb and heated and shaken for 5 hr. at 90–100°. After cooling, the viscous mass was treated with pentane after which it solidified. It was filtered and dried and weighted 46.4 g. (89% yield). After recrystallization from absolute alcohol and pentane it melted at 75–76°.

Anal. Calcd. for $C_{15}H_{19}NO_3$: C, 68.94; H, 7.33; N, 5.36. Found: C, 69.14; H, 7.26; N, 5.30.

Acknowledgment. The authors are greatly indebted to Mr. D. C. Wimer of the Analytical Research unit of Abbott Laboratories for the results described in Table I.

NORTH CHICAGO, ILL.

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[11] F. Swarts, *Bull. acad. roy. sci. Belg.* [3] 35, 375 (1898) reports b.p. of 201.5° at atmospheric pressure.

[12] Prepared in 74% yield by N. L. Drake, *et al.*, *J. Am. Chem. Soc.*, 68, 1602 (1946) by high pressure reduction of the nitro compound with Raney nickel. B.p. 74–75° (10 mm.).

[13] Obtained from L. Light and Co. Ltd., Colnbrook, Bucks, England.

[FROM THE SCIENTIFIC DEPARTMENT, MINISTRY OF DEFENCE]

A New Amino Acid Reagent, 2,4-Dinitro-5-fluoroaniline

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2,4-Dinitro-5-fluoroaniline and its acetyl derivative give with amino acids well defined derivatives. In the former case, the presence of the aromatic amino group in these derivatives permits the preparation of characteristic azo-dyes.

Since the observation of Sanger[2] that 2,4-dinitrofluorobenzene can be used as a reagent for the amino group in amino acids and peptides, a number of similar substances have been proposed for the same purpose: 2,4-dinitro-1,5-difluorobenzene,[3] 2,4-dinitro-1-fluoro-5-chlorobenzene,[4] ethyl 3-nitro-4-fluorobenzoate,[5] fluorobenzene-2,4,6-tricarboxylic acid[6] and 3-nitro-4-fluorobenzaldehyde.[7,8] In the present investigation, a variant of this method has been studied which permits the introduction of a new amino group together with the 2,4-dinitrophenyl radical. The reagent used was 2,4-dinitro-5-fluoroaniline (or its acetyl derivative). This reagent has two advantages, first that the amino group has a bathochromic effect (the maximum of 350 mμ in Sanger's dinitrophenyl derivatives being shifted to 400–410 mμ), and second that the aromatic amino group can be diazotized and coupled and thereby converted into an azo dye which lends itself to the detection of very small amounts, *e.g.*, on paper chromatograms.

2,4-Dinitro-5-fluoroacetanilide was prepared by the nitration of 3-fluoroacetanilide; it is deacylated easily by means of 50% sulfuric acid to the base which has been prepared before[9] by a different route.

For the preparation of 3-fluoroacetanilide the method developed previously[10] in this laboratory, is superior to the older method based on 3-nitroaniline.[11]

Both 2,4-dinitro-5-fluoroaniline and its *N*-acetyl derivative react with amino acids in the presence of sodium bicarbonate. The products crystallize easily and show characteristic crystal forms (See Tables II and III). The diazotization of the

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[1] This paper forms part of a thesis submitted by M. Bentov to the Hebrew University in fulfilment of the requirements for the degree of Ph.D.

[2] F. Sanger, *Biochem. J.*, 39, 507 (1945).

[3] H. Zahn and A. Wuerz, *Biochem. Z.*, 325, 182 (1953/4).

[4] H. Zahn and R. Kocklaeuner, *Biochem. Z.*, 325, 339 (1953/4).

[5] F. F. Micheel, K. Weichbrodt, and J. Plenikowski, *Ann.*, 581, 238 (1953).

[6] F. Micheel and W. Busse, *Ber.*, 90, 2049 (1957); 91, 985 (1958).

[7] F. Micheel and D. Noffz, *Ber.*, 90, 1586 (1957).

[8] Similar derivatives, *viz.*, 4-diethylamino-3,5-dinitrophenyl derivatives, have been briefly described by H. Edmunds and W. S. Reith, *Biochem. J.*, 57, XVIII (1954) and by A. Drèze and W. S. Reith, *Biochem. J.*, 63, 21P (1956). *Cf.* also M. Justial, E. Scoffone, and P. de la Llosa, *Bull. Soc. chim. France*, 1551 (1959); E. Scoffone, P. de la Llosa, and M. Justial, *Bull. Soc. chim. France*, 1553 (1959); Z. Talik and E. Plažek, *Bull. Acad. Polon. Sci., Chem. Sci. Series*, 8, 227 (1960).

[9] F. Swarts, *Rec. Trav. chim.*, 35, 161 (1915).

[10] E. D. Bergmann and M. Bentov, *J. Org. Chem.*, 20, 1654 (1955).

[11] A. Roe, *Org. Reactions*, 5, 193 (1949).