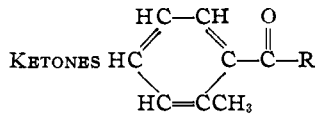
TABLE III

KETONES



R	B.p. °C.	Mm.	d^{20}_4	$n^{20}D$	2,4-DNP[f] M.p., °C.	2,4-DNP[f] Nitrogen, % Calcd.	2,4-DNP[f] Nitrogen, % Found	Semicarbazone M.p., °C.	Semicarbazone Nitrogen, % Calcd.	Semicarbazone Nitrogen, % Found
Methyl[a]	59–60	1.5	1.0114	1.5325	161–162	...	...	205–206	...	...
Ethyl[b]	72–73	1.5	0.9967	1.5250	106–107	17.07	16.78	172–173	...	...
n-Propyl[c]	84–85	2	.9774	1.5181	92–93	16.37	15.74	185–186	19.16	19.18
n-Butyl	97–98	2	.9645	1.5141	76–78	15.72	15.43	151–152	18.01	18.04
i-Butyl[d]	85–86	3	.9578	1.5104	91–92	15.72	15.57	171.5–172	18.01	18.06
s-Butyl[e]	79–80	4	.9639	1.5115	73–74.5	15.72	15.66	.....	...	...
i-Amyl	108–109.5	2.5	.9544	1.5090	105–106	15.13	14.94	143–144	16.99	16.84

[a] E. Huntress and S. Mulliken, "Identification of Pure Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1941, p. 390, gives d^{20}_4 1.014; $n^{20}D$ 1.5320; 2,4-DNP, m.p. 159°; semicarbazone, m.p. 206°. [b] J. Senderens, *Ann. chim.*, **28**, 332 (1913), reports semicarbazone, m.p. 169°. [c] *Ibid.*, **28**, 332 (1913), reports semicarbazone, m.p. 176°. [d] *Ibid.*, **28**, 333 (1913), reports semicarbazone, m.p. 166°. [e] *Anal.* Calcd. for ketone: C, 81.77; H, 9.15 Found: C, 81.79; H, 9.39. [f] 2,4-Dinitrophenylhydrazone.

TABLE IV

CATALYTIC REDUCTION RATES AND POLAROGRAPHIC HALF-WAVE POTENTIAL

R	*K*[a]	Relative rate	Half-wave potential Ketimine	Half-wave potential Ketone
Methyl	1.67	5.97	−1.67	−1.68
Ethyl	0.93	3.32	−1.72	−1.71
n-Propyl	.74	2.64	−1.90	−1.71
n-Butyl	.66	2.36	−1.91	−1.70
i-Butyl	.43	1.54	−1.91	−1.68
i-Amyl	.41	1.47	−1.94	−1.73
s-Butyl	.28	1.00	−2.02	−1.76

[a] K = milliliters of hydrogen per minute per gram imine.

Germany. Tetramethylammonium chloride was used as supporting electrolyte in a concentration of 0.15 *M* in ethanol. The organic compounds were freshly distilled and samples were dissolved in the 0.15 *M* tetramethylammonium chloride to produce solutions 5×10^{-3} *M* in the compound being run. A large constant area mercury pool was used as the reference electrode. The drop time of the dropping mercury electrode was 2.5 seconds. All solutions were degassed prior to determining the polarogram. Nitrogen was bubbled through alkaline pyrogallol to remove oxygen, through absolute alcohol to saturate it with alcohol vapor and minimize solvent loss from the sample, and then through the sample. It was found that ten minutes bubbling was sufficient to remove dissolved oxygen from the sample. Each wave exhibited a maximum except the *o*-tolyl *s*-butyl ketimine. These maxima could not be eliminated by addition of gelatin or methyl red, by changing galvanometer sensitivity or by varying concentration of electrolyte or test compound. Half-wave potentials are listed in Table IV.

NORMAN, OKLAHOMA

[CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY, UNIVERSITY OF ILLINOIS]

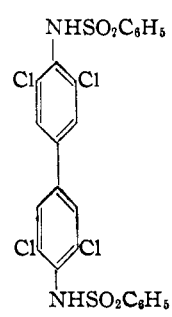
Quinone Imides. XXXI. 3,3-Dimethoxydiphenoquinonedibenzenesulfonimide and its Reaction Products

BY ROGER ADAMS, RICHARD R. HOLMES AND JOHN W. WAY[1]

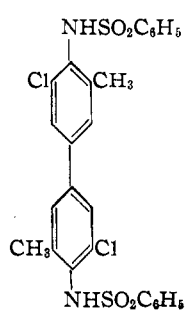
RECEIVED JUNE 17, 1953

The diimide obtained by the oxidation of the dibenzenesulfonyl derivative of *o*-dianisidine was subjected to hydrogen chloride addition. Oxidation of the resulting monochlorodiamide and addition of hydrogen chloride to the diimide thus formed gives N,N′-dibenzenesulfonyl-5,5′-dichloro-3,3′-dimethoxybenzidine. The structure was established by showing its non-identity to the other two symmetrical isomeric dichloro-3,3′-dimethoxybenzidines and by the similarity of the infrared absorption spectrum to that of N-benzenesulfonyl-2-chloro-6-methoxyaniline.

The addition of hydrogen chloride to the diphenoquinonedibenzenesulfonimides formed by lead tetraacetate oxidation of the N,N′-dibenzenesulfonyl derivatives of benzidine and tolidine has been described in previous papers.[2] The process involves 1,8-addition. The products derived from benzidine and tolidine on repeated oxidation and addition of hydrogen chloride were established as 3,3′,5,5′-tetrachlorodiphenoquinonedibenzenesulfonamide (I) and 3,3′-dichloro-5,5′-dimethyldiphenoquinonedibenzenesulfonamide (II).



I

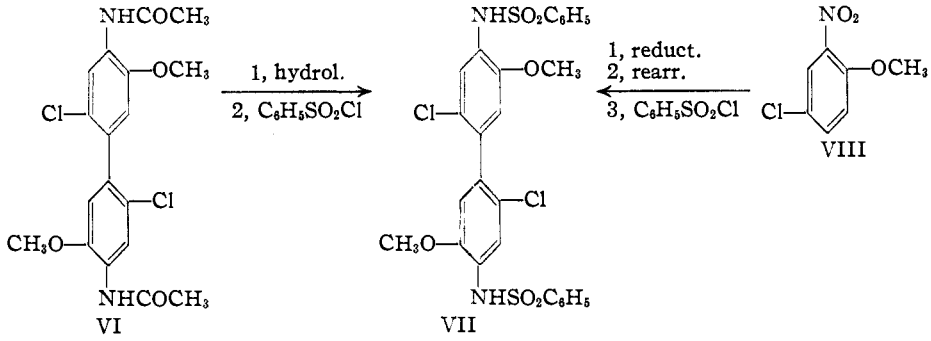


II

(1) An abstract of a thesis submitted by John W. Way to the Graduate College of the University of Illinois, 1953, in partial fulfillment of the requirements for the Degree of Doctor of Philosophy. Minnesota Mining and Manufacturing Company Fellow, 1951–1952; Allied Chemical and Dye Corporation Fellow, 1952–1953.

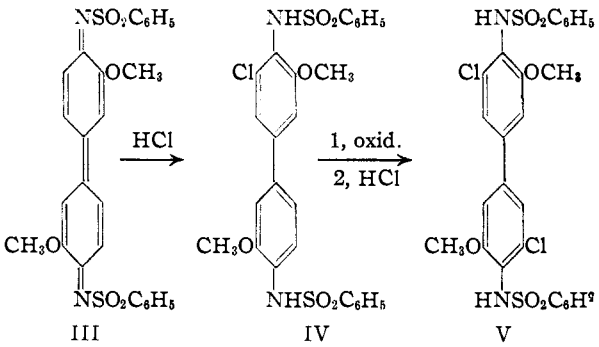
(2) R. Adams and R. R. Holmes, THIS JOURNAL, **74**, 3033 (1952); **74**, 3038 (1952).

To explain the orientation of groups, certain resonance forms of the intermediate ions were postulated as being those primarily responsible for the additions occurring in the manner observed. If these speculations were correct, the diimide from the N,N′-dibenzenesulfonyl derivative of dianisidine should add hydrogen chloride in a similar manner with the chlorines entering ortho to the benzenesulfonamido and meta to the methoxyl groups. This reaction has now been investigated.



VI VII

The formation of 3,3′-dimethoxydiphenoquinonedibenzenesulfonimide (III) from the corresponding diamide by lead tetraacetate oxidation proceeded smoothly. The product, like the benzidine and tolidine derivatives, forms black platelets which are difficult to crystallize from solvents without considerable loss. The hydrogen chloride addition to III was rapid and in good yield but the product had a wide melting point and was impure. The character of the impurities was not determined but apparently a certain amount of reduction of the diimide occurred as judged by analysis of the crude product and an isomeric monochloro compound might also have been present. A pure monochloro adduct was isolated by crystallization followed by chromatography. The oxidation of the monochloro compound, to which structure IV was assigned, proceeded smoothly to give black platelets of the corresponding diimide.



III IV V

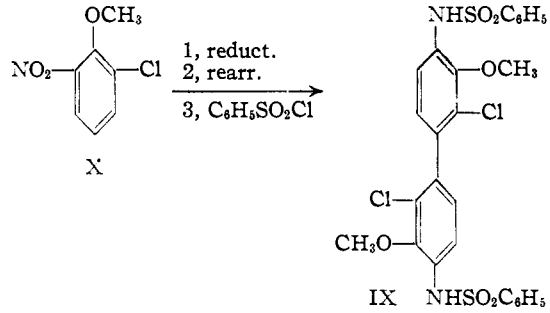
Upon addition of hydrogen chloride to the diimide a dichloro diamide V formed which like the corresponding monochloro diamide IV had a wide melting point and was difficult to purify. It was necessary to resort to chromatography to obtain the product free from by-products.

Since several attempted methods for an unequivocal synthesis of a compound of structure V failed, the other two symmetrical isomeric dichloro compounds of analogous structure were synthesized and shown not to be identical with V.

The diacetyl derivative of dianisidine upon treatment with sulfuryl chloride results in formation of VI which may be hydrolyzed and benzenesulfonated to VII.

The product VII also was formed by reduction of the known 5-chloro-2-methoxynitrobenzene (VIII) to the corresponding hydrazo compound which was then subjected to a benzidine rearrangement and subsequent benzenesulfonation.[3]

The other isomer, N,N′-dibenzenesulfonyl-2,2′-dichloro-3,3′-dimethoxybenzidine (IX), was prepared from the known 3-chloro-2-methoxynitrobenzene (X) by reduction to the hydrazo compound, rearrangement to the benzidine derivative, and benzenesulfonation.



X IX

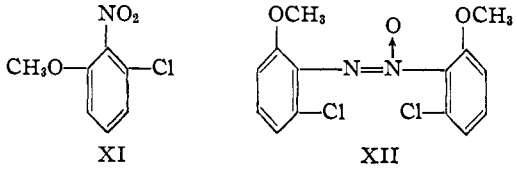
Compounds VII and IX were both different from the dichloro compound V obtained from the dibenzenesulfonyl derivative of dianisidine through successive oxidations and additions of hydrogen chloride. The postulated structure V was thus strongly indicated.

The infrared spectra of compounds V, VII and IX were all different. Those of VII and IX exhibited a relatively strong band at 950 cm.$^{-1}$ which is not in the spectrum of V. The spectrum of N-benzenesulfonyl-2-chloro-6-methoxyaniline derived from compound XI was very similar to that of V and was devoid of bands in the region from 900 to 1038 cm.$^{-1}$. On the other hand, the spectrum of N-benzenesulfonyl-3-chloro-2-methoxyaniline derived from compound X was very similar to that of IX and exhibited an absorption band at 947 cm.$^{-1}$. Likewise the spectrum of N-benzenesulfonyl-5-chloro-2-methoxyaniline derived from compound VIII was very similar to that of VII and exhibited an absorption band at 930 cm.$^{-1}$.

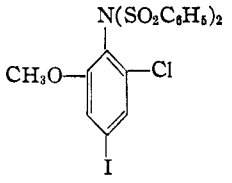
These data have been interpreted to mean that molecules in which the chlorine atoms are meta to the benzenesulfonamido group show an absorption band between 930 and 950 cm.$^{-1}$ not found when the chlorine atoms are in the *ortho* positions. Hence the structure of the dichloro isomer from the benzenesulfonyl derivative of diamisidine prepared through its diimide must be V.

(3) E. Harrison, *Chemistry and Industry*, **54**, 213 (1935).

Attempts to prepare structure V are described briefly below. When 2-chloro-6-methoxynitrobenzene (XI) was subjected to zinc and alkali reduction or to the action of lithium aluminum hydride, no azo compound was obtained. The corresponding azoxy derivative XII was isolated by the action of zinc and ammonium chloride followed by zinc dust and alkali but the yield was so low as to discourage attempts to reduce this product to the corresponding hydrazo derivative.



XI XII



XIII

N,N-Dibenzenesulfonyl-2-chloro-4-iodo-6-methoxyaniline (XIII) was synthesized by iodination of 2-chloro-6-methoxyaniline to 2-chloro-4-iodo-6-methoxyaniline followed by benzenesulfonation. No biphenyl derivative could be isolated by treatment of XIII with copper powder with or without solvent.

Acknowledgment.—The authors are indebted to Miss Helen P. Miklas for the determination and interpretation of the infrared spectra and to Miss Emily Davis, Mrs. Katherine Pih, Mrs. Jean Fortney, Mrs. Esther Fett, Mrs. Lucy Chang and Mr. Joseph Nemeth for the microanalyses.

Experimental[4]

N,N'-Dibenzenesulfonyl-*o*-dianisidine.—A solution of 25 g. of *o*-dianisidine and 40 g. of benzenesulfonyl chloride in 250 ml. of pyridine was heated at 100° for 12 hr. It was then poured into 500 ml. of water with stirring. The gray solid which separated weighed 54 g. (quant.). The material was recrystallized from glacial acetic acid (25 g. per l.) (Darco) from which it separated as white needles. After recrystallization from ethanol and then from glacial acetic acid pure material resulted, m.p. 211–213°.

Anal. Calcd. for $C_{26}H_{24}N_2O_6S_2$: C, 59.52; H, 4.61; N, 5.34. Found: C, 59.64; H, 4.74; N, 5.22.

3,3'-Dimethoxydiphenoquinonedibenzenesulfonimide (III).—Into a solution of 10 g. of lead tetraacetate in 200 ml. of glacial acetic acid was stirred 10 g. of finely-powdered diamide at room temperature for 4 hr. Oxidation was rapid, and the black diimide separated from the solution in a short time.

The product was removed by filtration and stirred with 200 ml. of glacial acetic acid containing 10 ml. of ethylene glycol. The solid was removed and stirred with 200 ml. of water and finally with 150 ml. of acetone. After filtering and drying, the black crystalline diimide weighed 7.1 g. (71%).

The diimide III was more soluble than the diimides derived from benzidine and *o*-tolidine, but was as easily destroyed by prolonged boiling with almost any solvent. It had no melting point but decomposed somewhat below 200° when heated in a capillary. A small sample was successfully recrystallized from hot ethylene dichloride. The pure product was then obtained as glittering black platelets with a metallic luster.

Anal. Calcd. for $C_{26}H_{22}N_2O_6S_2$: C, 59.75; H, 4.24; N, 5.36. Found: C, 59.64; H, 4.20; N, 5.13.

N,N'-Dibenzenesulfonyl-5-chloro-3,3'-dimethoxybenzidine (IV).—A suspension of 5.0 g. of the diimide III in 100 ml. of chloroform was treated with a stream of dry hydrogen chloride. In a few seconds the black solid had disappeared and a colorless solution resulted. A few minutes later white needles began to appear. The product weighed 8.0 g. This material was recrystallized from glacial acetic acid, from which it separated as short white needles, m.p. 170–184° and weighing 4.5 g. A second crystallization did not change the melting point appreciably. Although the product had an analysis close to that of a monochloro compound, the wide melting point indicated the presence of impurities.

Anal. Calcd. for $C_{26}H_{23}ClN_2O_6S_2$: C, 55.86; H, 4.15; N, 5.01. Found: C, 56.53; H, 4.34; N, 5.00.

A solution of 6 g. of crude IV (m.p. 172–185°), which had been once recrystallized from acetic acid, in 200 ml. of ethyl acetate was passed through a column (2 × 20 cm.) of alumina (Alcoa F-20). The column was eluted with 500 ml. of ethyl acetate. The eluates were collected in 50-ml. portions, and each portion was evaporated to dryness. The first 250 ml. contained nothing, the next 100 ml. contained 1.7 g. of dissolved solid, which, after recrystallization from glacial acetic acid, melted at 168–170°. The next 200 ml. of filtrate contained 3.1 g. of solid which, after crystallization from acetic acid, melted at 185–190°. This material was dissolved in ethyl acetate and rechromatographed as above. The first portions of eluate contained 0.6 g. of solid, which, after crystallization from acetic acid, melted at 185–190°. The latter portions of eluate contained 2.1 g. of solid which, after crystallization from acetic acid, melted at 188–191°. Five recrystallizations of this last material from acetic acid gave 1.2 g. of white crystals of pure N,N'-dibenzenesulfonyl-5-chloro-3,3'-dimethoxybenzidine, m.p. 193–195°.

Anal. Calcd. for $C_{26}H_{23}ClN_2O_6S_2$: C, 55.86; H, 4.15; N, 5.01. Found: C, 55.80; H, 4.25; N, 5.02.

5-Chloro-3,3'-dimethoxydiphenoquinonedibenzenesulfonimide.—A mixture of 1.0 g. of pure chlorodiamide IV and 1.0 g. of lead tetraacetate in 20 ml. of acetic acid was stirred at room temperature for 1 hr. The tiny black crystals which formed were removed and shaken with 10 ml. of acetic acid containing 5 drops of ethylene glycol. The diimide was then slurried successively with water and acetone. It weighed 0.8 g. (81%). The crude diimide was used directly for the addition of hydrogen chloride.

This diimide formed a deep purple-red solution in hot ethylene dichloride. Prolonged heating with this or other solvents destroyed it with the formation of colorless, soluble products.

N,N'-Dibenzenesulfonyl-5,5'-dichloro-3,3'-dimethoxybenzidine (V).—Dry hydrogen chloride was bubbled into a suspension of 0.7 g. of the crude monochlorodiimide in 5 ml. of chloroform. Decolorization was rapid. The clear solution was evaporated to dryness and the white solid after crystallization from a minimum of glacial acetic acid weighed 0.4 g. This material was dissolved in 100 ml. of ethyl acetate and the solution poured through a column (2 × 10 cm.) of alumina (Alcoa F-20). The column was eluted with 250 ml. of ethyl acetate. The last 150 ml. of eluate contained the bulk of the solid (0.25 g.). This was recrystallized 3 times from glacial acetic acid; small white prisms of the pure N,N'-dibenzenesulfonyl-5,5'-dichloro-3,3'-dimethoxybenzidine, m.p. 257–259°, were obtained.

Anal. Calcd. for $C_{26}H_{22}Cl_2N_2O_6S_2$: C, 52.63; H, 3.74; N, 4.72. Found: C, 52.86; H, 3.64; N, 4.82.

N,N'-Dibenzenesulfonyl-2,2'-dichloro-5,5'-dimethoxybenzidine (VII).—A solution of 7.0 g. of 2,2'-dichloro-5,5'-dimethoxybenzidine[3] and 7.0 g. of benzenesulfonyl chloride in 100 ml. of pyridine was heated at 100° for 12 hr. The solution was then poured into water and the precipitate removed and recrystallized several times from glacial acetic acid. The pure dichlorodiamide had a m.p. 213–215°.

Anal. Calcd. for $C_{26}H_{22}Cl_2N_2O_6S_2$: C, 52.63; H, 3.74. Found: C, 52.69; H, 3.93.

(4) All melting points corrected.

3,3'-Dichloro-2,2'-dimethoxyhydrazobenzene.—To a solution of 5.0 g. of 2-chloro-6-nitroanisole[5] in 35 ml. of methanol, 7.3 g. of sodium hydroxide dissolved in 10 ml. of water was added, and then 10 g. of zinc dust portionwise to the rapidly stirred solution. The heat of reaction caused refluxing. At the end of the addition the gray-red suspension was refluxed one-half hour. It was then filtered into a flask containing 3 ml. of saturated aqueous sodium bisulfite. The precipitate was washed with boiling methanol containing some sodium bisulfite solution. To the filtrate 150 ml. of water was added. After cooling a white crystalline solid separated which was filtered, washed with 1% aqueous sodium bisulfite, and dried in a vacuum desiccator. The product weighed 2.5 g. (59%), m.p. 69–71°. This was used directly in the following experiment.

2,2'-Dichloro-3,3'-dimethoxybenzidine.—A solution of 2.5 g. of the crude dry hydrazo compound prepared above in 40 ml. of ether was added to 100 ml. of 1:1 hydrochloric acid with vigorous shaking. In about 5 minutes a solid began to form. After standing for 12 hr. the white solid was collected and dried. The weight of the dihydrochloride was 1.5 g. A sample was recrystallized 4 times from dilute hydrochloric acid for analysis. The product decomposed above 230°.

Anal. Calcd. for $C_{14}H_{16}Cl_4N_2O_2$: C, 43.55; H, 4.18; N, 7.26. Found: C, 43.32; H, 4.33; N, 7.46.

Treatment of the filtrate with 10% aqueous sodium hydroxide gave 0.5 g. of the free base. Thus a total yield of 68% was realized. The free base crystallized with difficulty from aqueous methanol, m.p. 109–112°.

N,N'-Dibenzenesulfonyl-2,2'-dichloro-3,3'-dimethoxybenzidine (IX).—A solution of 1.0 g. of the dihydrochloride of the dichlorodimethoxybenzidine and 1.0 g. of benzenesulfonyl chloride in 20 ml. of pyridine was allowed to stand for 12 hr. The light yellow solution was poured into 50 ml. of 1:1 hydrochloric acid and a white solid separated. It weighed 1.39 g. (90%). Recrystallization from acetic acid and then from ethanol gave the pure dichlorodiamide, m.p. 175–176°.

Anal. Calcd. for $C_{26}H_{22}Cl_2N_2O_6S_2$: C, 52.63; H, 3.74; N, 4.72. Found: C, 52.72; H, 3.78; N, 4.62.

3-Chloro-2-nitroanisole.—3-Chloro-2-nitrophenol was prepared by the method of Hodgson and Moore[6] and methylated by alternate additions of an excess of aqueous 20% sodium hydroxide and dimethyl sulfate to an aqueous solution of the phenol. Attempts to convert it by means of lithium aluminum hydride[7] to the azo compound failed. Prolonged action of zinc dust and alkali[8] gave only the corresponding amine.

2,2'-Dichloro-6,6'-dimethoxyazoxybenzene (XII).—A solution of 20 g. of 3-chloro-2-nitroanisole in 250 ml. of ethanol was boiled with 10 g. of zinc dust and 3 g. of ammonium chloride for 30 min. Then 50 ml. of 30% aqueous sodium hydroxide was added dropwise over the course of an hour, while the mixture was kept vigorously boiling. The mixture was filtered hot, and water was added to the cloud point. On standing, the dark solution deposited a solid. This was removed and recrystallized from aqueous ethanol (Darco). The pale yellow needles weighed 2.4 g. Two recrystallizations from ethanol gave 1.9 g. of almost white needles, m.p. 137–139°.

Anal. Calcd. for $C_{14}H_{12}Cl_2N_2O_3$: C, 51.39; H, 3.70; N, 8.56. Found: C, 51.86; H, 4.09; N, 8.34.

N-Benzenesulfonyl-2-chloro-6-methoxyaniline.—A solution of 2-chloro-6-methoxyaniline[9] in pyridine was treated with benzenesulfonyl chloride. The product was purified from glacial acetic acid, m.p. 144–145.5°.

Anal. Calcd. for $C_{13}H_{12}ClNO_3S$: C, 52.45; H, 4.03; N, 4.71. Found: C, 52.38; H, 4.02; N, 4.71.

N-Benzenesulfonyl-3-chloro-2-methoxyaniline.—A solution of 0.26 g. of the hydrochloride of 3-chloro-2-methoxyaniline[10] in 10 ml. of pyridine was treated with 0.24 g. of benzenesulfonyl chloride. The product was recrystallized from aqueous acetic acid; white needles, m.p. 124.5–126°.

Anal. Calcd. for $C_{13}H_{12}ClNO_3S$: C, 52.45; H, 4.03; N, 4.71. Found: C, 52.44; H, 3.93; N, 4.66.

N-Benzenesulfonyl-5-chloro-2-methoxyaniline.—To a solution of 5.0 g. of 4-chloro-2-nitroanisole[11] in 30 ml. of boiling ethanol was added slowly 15.5 g. of sodium hydrosulfite along with 100 ml. of water. The mixture was boiled for an additional 15 minutes and cooled. White plates of the amine separated, weighing 2.56 g. and melting at 81–83°.

A solution of 0.73 g. of the 5-chloro-2-methoxyaniline and 0.90 g. of benzenesulfonyl chloride in 10 ml. of pyridine was heated for 40 minutes on a steambath. The product was purified by recrystallization from glacial acetic acid; colorless prisms, m.p. 153–154°.

Anal. Calcd. for $C_{13}H_{12}ClNO_3S$: C, 52.45; H, 4.03. Found: C, 52.45; H, 4.15.

2-Chloro-4-iodo-6-methoxyaniline.—A suspension of 6.4 g. of 2-chloro-6-methoxyaniline in a solution of 5 g. of sodium bicarbonate in 30 ml. of water was stirred and 10.3 g. of iodine added in small portions. The dark brown solution effervesced vigorously. Stirring was continued for 8 hr. The reaction mixture was placed in a separatory funnel and extracted with ether. The combined ether extracts were washed with 5% aqueous sodium bisulfite and then with water. The purple ether solution was dried over anhydrous magnesium sulfate. After removal of the solvent at atmospheric pressure, the dark residue was distilled under reduced pressure. After a small forerun, 3.5 g. (30%) of product was obtained; yellow oil, b.p. 140–145° (2.4 mm.).

The monobenzenesulfonamide was prepared using benzenesulfonyl chloride and pyridine. It was purified by recrystallization from acetic acid, m.p. 174–175°.

Anal. Calcd. for $C_{13}H_{11}ClINO_3S$: C, 36.85; H, 2.62; N, 3.31. Found: C, 36.58; H, 2.81; N, 3.60.

N,N-Dibenzenesulfonyl-2-chloro-4-iodo-6-methoxyaniline (XIII).—A solution of 3.5 g. of 2-chloro-4-iodo-6-methoxyaniline (XII) in 15 ml. of pyridine and 6.0 g. of benzenesulfonyl chloride was refluxed for 4 hr. After cooling, the solution was poured into 150 ml. of water. The tan oil which formed readily crystallized to a brown solid which was filtered and dried; it weighed 6.9 g. (99%). Several recrystallizations from acetic acid (Darco) gave white needles, m.p. 206–207.5°.

Anal. Calcd. for $C_{19}H_{15}ClINO_5S_2$: C, 40.47; H, 2.68. Found: C, 40.56; H, 2.99.

Sodium ethoxide in ethanol easily converted XIII to the corresponding monobenzenesulfonamide.

Heating XIII at 260° with activated copper powder for 2.5 hours without solvent or in boiling nitrobenzene gave only amorphous products.

URBANA, ILLINOIS

(5) H. H. Hodgson and A. Kershaw, *J. Chem. Soc.*, 2167 (1930). The phenol was prepared by the method of C. K. Ingold and E. W. Smith, *ibid.*, 1693 (1927).

(6) H. H. Hodgson and F. H. Moore, *ibid.*, **127**, 1599 (1925).

(7) R. F. Nystrom and W. G. Brown, THIS JOURNAL, **70**, 3738 (1948).

(8) P. Cohn, *Ber.*, **33**, 3552 (1900).

(9) H. H. Hodgson and A. Kershaw, *J. Chem. Soc.*, 191 (1928).

(10) W. M. Lauer, R. T. Arnold, B. Tiffany and J. Tinker, THIS JOURNAL, **68**, 1269 (1946).

(11) M. Kohn and R. Kramer, *Monatsh.*, **44**, 147 (1928).