after forming, the recovered trinitrobenzene was light raspberry colored, although even two days of standing had little effect on the melting point of the recovered material. The identification of any further products was not undertaken as the material obtained from long standing or from heating the trinitrobenzene in excess piperidine consisted of brown crystallized gums, completely soluble in concentrated hydrochloric acid but not responding to any simple method of purification.

1-Chloro-3,5-dinitrobenzene (20).—This exhibited the same color phenomena with piperidine as *sym*-trinitrobenzene, but no crystalline addition product could be prepared although various ratios of piperidine to chloronitrobenzene were tried and the thick red liquids cooled in dry-ice until they hardened to glasses. This addition product does not form as readily, for although 20 deliquesces in saturated piperidine vapor, it will not produce color with aqueous piperidine. The same deep-seated reactions occur, but no pure product could be isolated from the brown powders by fractional precipitation from either the concentrated hydrochloric acid solution or from acetone solution.

Relative Reactivities.—A qualitative determination of the relative reactivities of the various compounds was made both very roughly by observing the phenomena occurring when piperidine is added to the solids and more accurately by determining the mildest conditions for complete replacement of each halogen atom as well as by certain comparative experiments not discussed. The following list is in order of decreasing reactivity in respect to monopiperidination only: A. Compounds producing sputtering as simultaneous precipitation of the piperidine hydrohalide and boiling of the red or orange liquid occurs (sp) or boiling of the red or orange solution without salt precipitation (b), (1) reactions instantaneous in cold alcohol solution [10 (sp), 7 (sp), 8 (sp), 14 (sp), 28 (sp), 24 (sp), 9 (sp), 21 (b) and 13 (b)], (2) reactions not instantaneous in cold alcohol solution, 19 (b), 25 (b), 27 (sp), [16 (sp) and 31 (sp)], 26 (b, sp), 32 (sp); B. Compounds forming clear yellow solutions from which piperidine hydrohalide precipitates in the cold in a few minutes (Xm) generally accompanied by liberation of heat, [36(0m), 37(0m) and 18(0m)], 15(0m), 17(0-1m), 22(1m), 29(1-2m), 30(2$^1/_2$m), 12(5m), 33(15m); C. Compounds dissolving in piperidine with absorption of heat and producing clear yellow solutions, 2, 11, 1, 6, 5, 23, [3, 4, 34 and 35].

Summary

1. Two standard procedures for the preparation of piperidyl derivatives of aromatic halogenonitro compounds have been developed.

2. All possible piperidyl derivatives of the compounds studied have been isolated and the most favorable conditions for their preparation determined.

3. The structures of certain derivatives have been proved.

CAMBRIDGE, MASS. RECEIVED NOVEMBER 28, 1939

[CONTRIBUTION NO. 210 FROM THE RESEARCH LABORATORY OF ORGANIC CHEMISTRY, MASSACHUSETTS INSTITUTE OF TECHNOLOGY]

The Deepening of Color of Sodium Nitrophenolate Solutions with Elevation of Temperature

BY TENNEY L. DAVIS AND JOSEPH L. RICHMOND

The yellow color of solutions of the salts of *m*-nitrophenol is of significance in discussions of theories of color and constitution. Satisfactory quinoid formulas may be written for the colored salts of *o*- and *p*-nitrophenol, as Armstrong[1] pointed out, and the parent substances may be supposed to exist in tautomeric quinoid forms. Hantzsch[2] accepted the view of Armstrong and suggested formulas for the colored salts of *m*-nitrophenol. Later he[3] succeeded in preparing colored and colorless esters of *o*- and *p*-nitrophenol, but not of the *m*-compound, and concluded that the free *m*-quinone is not capable of existence; or, as we should say, that *m*-nitrophenol is not capable of tautomerizing to a quinoid form, but that the colored salts of *m*-nitrophenol are nevertheless quinoid, the *m*-

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[1] Armstrong, *J. Chem. Soc.*, 101 (1892).

[2] Hantzsch, *Ber.*, **39**, 1095 (1906).

[3] Hantzsch, *ibid.*, **40**, 339 (1907).

quinone structure being stable in electronegative radicals present in an electrolyte along with positive ions of the metal. Hantzsch's *m*-quinoid formulas have not met with general acceptance, for they represent atomic arrangements which are thought to be unstable. The problem has been complicated further by the absence from the literature of any clear cut definition of quinones and of an understanding or an agreement as to what the structure of *m*-quinones, if they exist, would be.[4]

Hirsch[5] reported that solutions of the sodium salts of *o*- and *p*-nitrophenol become more deeply colored when they are warmed, and stated wrongly that the colors of the solutions on cooling revert only slowly to the original shades. We find that the changes of color intensity are as rapid as the changes of temperature, and we find, contrary to the observation of Hirsch, that solutions of sodium *m*-nitrophenolate behave in the same general way as those of the *o*- and *p*-compounds. The change appears to be solely a change in color intensity, for the color of a short column of warm solution matches perfectly with the color of a longer column of cold solution.[6]

The colors of solutions of the three sodium nitrophenolates are sensibly the same to the eye. Measurements of the thermotropic color intensifications have shown that the three substances differ from one another in respect to this property no more than substances of similar but not wholly identical structure would be expected to differ. The results of our experiments with 0.1 *N* solutions of the sodium nitrophenolates are plotted in Fig. 1, where each curve summarizes the results from two or from three runs, and the spread of the data indicates the approximation or error with which we have been able to match the colors.

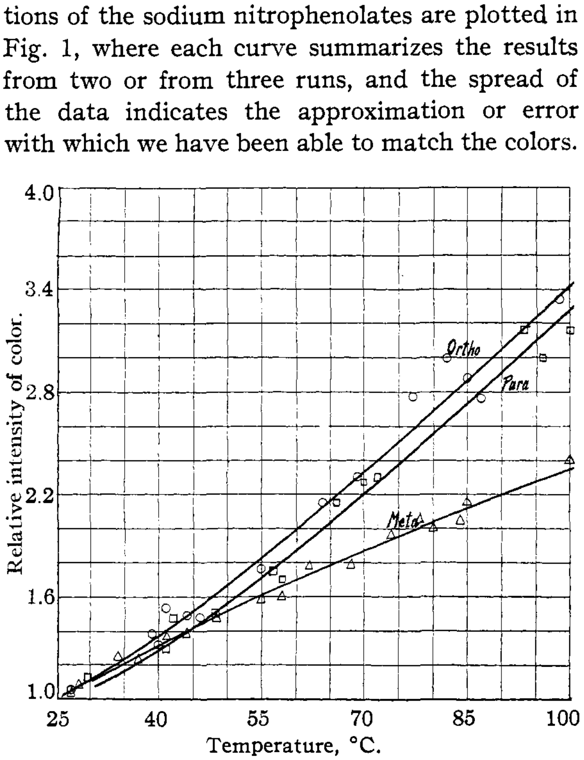

Fig. 1.—Intensity of color of warm 0.1 *N* sodium nitrophenolate solutions compared with the color of the same solutions at 25°: ○, ortho; □, para; △, meta.

The curves for the *o*- and *p*-compounds are close together; the color of the solution of the ortho compound is about 3.4 times as intense at 100° as at 25°; that of the para compound about 3.3 times. The curve for the *m*-compound slopes upward less sharply but is of the same general form as the curves for its isomers; the color of 0.1 *N* sodium *m*-nitrophenolate is about 2.3 times as intense at 100° as at 25°. In other experiments to determine the effect of alkalinity, sodium carbonate was added to 0.1 *N* *m*-nitrophenol solution in amounts corresponding to 0.02, 0.2, 1.0 and 2.0 *N* concentration of sodium ion. The results, plotted in Fig. 2, show that the thermotropic color intensification is lessened by increasing amounts of alkali, hence, since the alkali may be presumed to reduce the ionization of the salt, that the effect is due to a property of the ion.

An hypothesis to account for the behavior of the three nitrophenolates must assign similar structures to the ions of all of them, and will have additional plausibility if it relates the structure of the *m*-nitrophenolate ion to the known

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(4) The common practice in textbooks appears to be to describe the production of *p*-benzoquinone by the oxidation of hydroquinone, to discuss the peculiar chemical and structural properties of this substance, and then to use these properties as a basis for outlining, but not for defining, the class of quinones. The definition of quinones as oxidation products of benzene derivatives is not satisfactory; quinone dioxime, an undoubted quinone, is oxidized by strong nitric acid to yield *p*-dinitrobenzene. For a number of years T. L. D. has been accustomed in his classes to define quinones as substances in which two atoms attached to an aromatic ring (or ring system) are the terminal atoms of a conjugate system through the aromatic ring (or ring system). (An aromatic ring is conceived and defined as a six-membered carbon ring, each carbon atom of which has one residual valence.) This definition is believed to include all substances which are recognized to be quinones, and to exclude all those which are not now classed as such. It has the advantage that it unifies the reactions of quinones, in which the side chain conjugated into the ring acts in many cases to make the ring accessible to reagents which do not affect the ring of other aromatic substances. It excludes the possibility of *m*-quinones, but leaves open for study the question of what structures result when two hydrogen atoms are removed from *m*-diphenols.

(5) Hirsch, *Ber.*, **36**, 1893 (1903).

(6) Hantzsch, *ibid.*, **39**, 1084 (1906), reported observations on the colors of the dry salts, that they intensified on warming and after cooling later faded again. Goddard, *J. Chem. Soc.*, 54 (1922), found that the hydrated sodium salts of the nitrophenols lose water to form the anhydrous compounds which are darker in color, and that these on standing in the air take up water to form the lighter hydrated salts.

facts relative to the existence, or non-existence, of *m*-quinone.

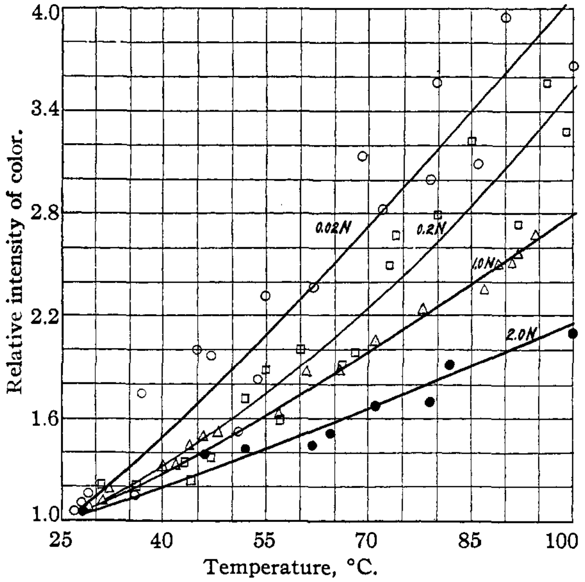


Fig. 2.—Effect of sodium carbonate upon thermotropic color intensification of 0.1 *N* *m*-nitrophenol solutions: ○, 0.02 *N* sodium carbonate; □, 0.2 *N*; △, 1.0 *N*; ●, 2.0 *N*.

Experimental Results

A colorimeter was constructed in which two beams of light from a single source were made to pass respectively through two tubes, one of which contained a column of solution at room temperature the length of which could be varied while the other contained a column of constant length enclosed in a jacket through which water was circulated into which steam or warm water or cold water was injected as required. The temperature of the second column of solution was determined by means of a thermocouple which, it was found, did not interfere with the optical path. The light after passing through the two tubes was reflected upon a single target as two semicircular spots so placed as to form a complete circle with each other. The length of the column of liquid at room temperature was then adjusted until the two halves of the circular spot were indistinguishable. The runs were started with the same solution in both tubes in columns of equal length. The temperature of the jacketed column was then raised a convenient amount. When the potentiometer readings showed that the temperature had become constant, the length of the column in the other tube was adjusted until the colors matched and the ratio of the length of the cold column to the length of the warm column was recorded as the relative intensity of the color of the solution at the temperature in question as compared with the color at room temperature. Similar readings were made at intervals while the temperature was raised to about 100°, then while the temperature was lowered to that of tap water, and the relative color intensities were calculated in comparison with the color at 25°. There was no perceptible lag in the intensification of the color on warming or in the fading of the color on cooling; the relative intensities of color were found to be the same whether measured while going up or while going down the temperature scale. At each concentration two or generally three runs were made, and the results were plotted in a single curve. The results at the even temperatures read from such curves, for 0.1, 0.02, 0.01 and 0.005 *N* solutions of the three nitrophenolates are summarized in Table I. From these data it is evident that the difference between the isomers is less in the more dilute solutions.

In another series of experiments sodium carbonate in amounts corresponding to 0.02, 0.2, 1.0 and 2.0 *N* concentration was added to 0.1 *N* solutions of *m*-nitrophenol, and the thermotropic color intensifications of these solutions, containing, respectively, one-fifth, twice, ten times, and twenty times the equivalent amount of sodium ion, were determined. The data lay closest to smooth curves in the experiments in which the more sodium carbonate was used. Other experiments were carried out with 0.01 *N* solutions of *m*-nitrophenol containing one-fifth, twice, ten times and twenty times the equivalent amount of

TABLE I

INTENSITY OF COLOR OF WARM SODIUM NITROPHENOLATE SOLUTIONS COMPARED WITH THE COLOR OF THE SAME SOLUTIONS AT 25°

	o-Nitrophenolate, *N*				*m*-Nitrophenolate, *N*				*p*-Nitrophenolate, *N*			
Temp. °C.	0.1	0.02	0.01	0.005	0.1	0.02	0.01	0.005	0.1	0.02	0.01	0.005
25	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
30	1.12	1.11	1.06	1.10	1.10	1.04	1.06	1.08	1.05	1.04	1.04	1.02
35	1.24	1.23	1.15	1.22	1.21	1.11	1.13	1.17	1.16	1.09	1.16	1.06
40	1.37	1.35	1.25	1.34	1.32	1.20	1.22	1.28	1.28	1.14	1.25	1.11
45	1.52	1.48	1.36	1.47	1.42	1.30	1.33	1.40	1.42	1.20	1.36	1.16
50	1.67	1.60	1.49	1.60	1.52	1.39	1.44	1.51	1.56	1.28	1.47	1.23
55	1.83	1.74	1.63	1.75	1.61	1.51	1.55	1.62	1.71	1.38	1.60	1.32
60	1.99	1.90	1.79	1.89	1.70	1.66	1.68	1.74	1.87	1.50	1.75	1.43
65	2.16	2.06	1.85	2.06	1.78	1.80	1.82	1.88	2.03	1.63	1.90	1.59
70	2.33	2.25	2.11	2.21	1.87	1.93	1.96	2.03	2.20	1.78	2.08	1.76
75	2.50	2.45	2.28	2.37	1.95	2.07	2.12	2.19	2.37	1.98	2.27	1.87
80	2.68	2,64	2.46	2.44	2.03	2.21	2.32	2.35	2.55	2.17	2.48	2.16
85	2.86	2.86	2.65	2.63	2.12	2.38	2.51	2.49	2.73	2.38	2.70	2.40
90	3.05	3.10	2.90	2.82	2.19	2.55	2.70	2.64	2.90	2.64	2.98	2.62
95	3.23	3.38	3.18	3.12	2.27	2.72	2.87	2.81	3.10	2.89	3.20	2.84
100	3.41	3.66	3.46	3.32	2.35	2.90	3,06	2.98	3.28	3.20	3.43	3.10

TABLE II

EFFECT OF SODIUM CARBONATE ON THERMOTROPIC COLOR INTENSIFICATION OF *m*-NITROPHENOL SOLUTIONS AT VARIOUS CONCENTRATIONS

m-Nitrophenol, *N*	0.1	0.1	0.1	0.01	0.01	0.01	0.01	0.01	0.001	0.001
Na_2CO_3, *N*	0.02	0.2	1.0	2.0	0.002	0.02	0.1	0.2	0.01	0.02
Temp. °C.										
25	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
30	1.15	1.10	1.08	1.06	1.08	1.06	1.04	1.05	1.06	1.04
35	1.32	1.21	1.18	1.13	1.16	1.10	1.09	1.10	1.11	1.08
40	1.50	1.33	1.28	1.20	1.22	1.18	1.16	1.17	1.16	1.12
45	1.69	1.47	1.38	1.27	1.34	1.28	1.23	1.25	1.22	1.16
50	1.89	1.60	1.50	1.35	1.44	1.40	1.30	1.34	1.28	1.20
55	2.19	1.75	1.61	1.42	1.56	1.52	1.38	1.42	1.34	1.25
60	2.30	1.90	1.74	1.50	1.69	1.66	1.45	1.51	1.40	1.32
65	2.51	2.07	1.86	1.58	1.83	1.80	1.53	1.61	1.46	1.38
70	2.73	2.24	1.99	1.66	1.97	1.92	1.62	1.73	1.53	1.46
75	2.95	2.43	2.11	1.74	2.11	2.08	1.74	1.86	1.62	1.55
80	3.17	2.63	2.25	1.82	2.26	2.22	1.85	2.00	1.70	1.65
85	3.40	2.85	2.38	1.90	2.41	2.38	2.00	2.12	1.79	1.76
90	3.62	3.07	2.51	1.98	2.57	2.54	2.18	2.29	1.88	1.87
95	3.84	3.30	2.65	2.07	2.74	2.68	2.40	2.42	1.98	1.99
100	4.05	3.54	2.79	2.15	2.91	2.82	2.65	2.59	2.07	2.10

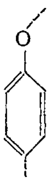
sodium ion, and with 0.001 *N* solutions containing ten and twenty times the equivalent, respectively. The results read from the curves are summarized in Table II. The data from the experiments with the more dilute solutions are probably the least precise because of the difficulty of matching the fainter colors.

m-Nitrophenol, prepared by the method of Adams and Wilson,[7] was purified by vacuum distillation and finally by sublimation in vacuum, almost white crystals melting sharply at 96°. The sodium salt, prepared in the usual way and recrystallized from alcohol and from water, consisted of orange colored small crystals containing two molecules of water of crystallization. *Anal.* Sodium found, 11.4, 11.5. Calcd. for $C_6H_4O_3NNa{\cdot}2H_2O$: Na, 11.6.

Sodium *o*-nitrophenolate, recrystallized from alcohol and from water, consisted of anhydrous bright red needles. *Anal.* Sodium found, 14.6, 14.5. Calcd. for $C_6H_4O_3NNa$: Na, 14.3.

Sodium *p*-nitrophenolate crystallized from water in bright canary-yellow crystals containing two molecules of water of crystallization. *Anal.* Sodium found, 11.6.

Interpretation of Results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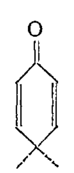
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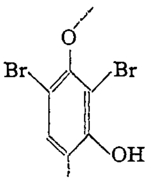
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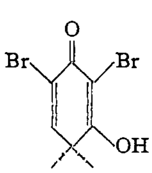
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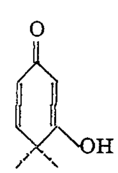
V

The work of Hunter and his co-workers[8] has shown that the oxidation of phenol occurs through the intermediate production of a radical having formulas I and II below (which formulas we regard as equivalent), while that of Davis and Hill[9] has shown that tribromoresorcinol similarly passes through the stage represented by formulas III and IV. Davis and Harrington[10] have shown that the quinyl radical, formula IV, is probably intermediate in many of the reactions of tribromoresorcinol, being formed by the spontaneous splitting off of hydrogen bromide from the tautomeric form of that substance. This formula corresponds to the removal of two hydrogen atoms from dibromoresorcinol and may be regarded, if the language is permitted, as representing dibromo-*m*-quinone. The comportment of *m*-diphenols in general, on oxidation and in other reactions, may be interpreted by supposing the intermediate existence of such *m*-quinones, and formula V may be taken as representing *m*-quinone itself; which is thus a *p*-quinyl radical, probably incapable of an independent existence but ready, according to the environment in which it exists momentarily, to form resorcinol by reduction, hydroxy-*p*-quinone by oxidation, or by doubling-up under conditions which are neither oxidizing nor reducing to form dihydroxydiphenoquinone. If *m*-quinone may hydrate, formula VI, as *o*- and *p*-quinone presumably hydrate, formulas VII and VIII, then the hydrated quinones will exhibit similar unsaturated and resonating structures and will supply a common basis for discussions of color and constitution. The fact that formula VI represents one of the tauto-

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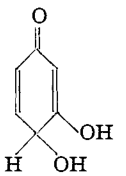
[7] Adams and Wilson, "Organic Syntheses," Vol. 3, John Wiley and Sons, Inc., New York, N. Y., 1923, p. 87.

[8] Hunter, *et al.*, THIS JOURNAL, **38**, 1761 (1916); **39**, 2640 (1917); **43**, 131, 135, 151 (1921); **48**, 1608, 1615 (1926).

[9] Davis and Hill, *ibid.*, **51**, 493 (1929).

[10] Davis and Harrington, *ibid.*, **56**, 129 (1934).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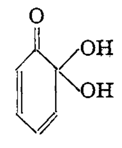
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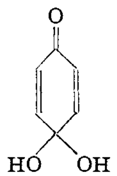
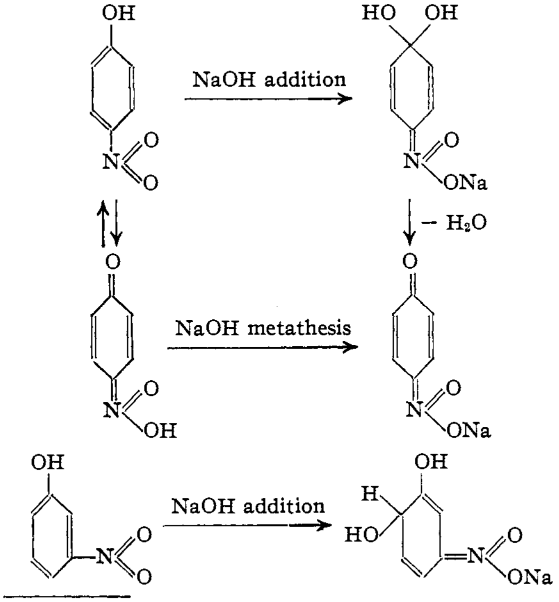
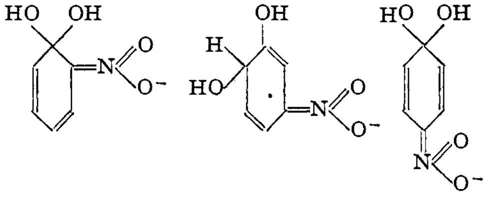

VIII

meric forms of 1,2,4-trihydroxybenzene suggests that the latter substance may perhaps be procurable from resorcinol by oxidation under appropriate conditions. For our present purpose however it is not necessary to suppose that the simple quinones hydrate. The formulas are illustrative. Our hypothesis supposes only that quinoid (not quinone) ions may exist which correspond to the three hydrated quinones.

o- and *p*-nitrophenol are both capable of tautomerizing, and may form sodium salts by the replacement of hydrogen atoms in the tautomers. *m*-Nitrophenol cannot do this. The same sodium salts of *o*- and *p*-nitrophenol, however, may be formed by a different mechanism; it is possible that the sodium hydroxide may add to the benzene derivative to form a quinoid compound[11] from which water may subsequently split off. But there is no evidence that the water does split off in aqueous solution. The colored ions may in fact be hydrated. *m*-Nitrophenol likewise may combine directly with sodium hydroxide to form a *p*-quinoid compound (a hydrated *m*-quinone) from which, however, water cannot split off, and, as far as the formation of hydrated salts, and of colored hydrated ions, is concerned, all three of the nitrophenols are alike. We suppose therefore that the colored ions of the three nitrophenolates are the hydrated quinoid ions indicated. By this hypothesis the structure assigned to the *m*-nitrophenolate ion has the same relationship to the structure of *m*-quinone as that of the *p*-nitrophenolate ion has to the structure of *p*-quinone. The three nitrophenolate ions are supposed to be of similar structure and hence are expected to behave in similar manners. We are not prepared to offer an hypothesis to explain how the quinoid structure accounts for the thermotropic intensification of the colors of the solutions, except by the general supposition that the increase of temperature increases the resonance of the conjugate system of the ions, for we suppose that it is not the quinoid structure merely, but the possibilities of resonance which that structure provides, which make the basis for the quinone theory of color and constitution. The resonating system of the ions extends only into the rings, but not through them and beyond as in many of the dyestuffs, and this circumstance, in some way, may perhaps be associated with the phenomenon of color intensification.





Summary

The colors of warm solutions of the three sodium nitrophenolates have been measured by comparison with the colors of the same solutions at 25°. The thermotropic color intensifications of the three isomers appear to conform to the same general law, but the effect is least in the case of the *m*-compound.

The effect of various concentrations of sodium carbonate in solutions of *m*-nitrophenol has been studied. Alkali retards the color intensification, confirming the opinion that the effect is due to a property of the ion.

An hypothesis has been offered which assigns similar hydrated quinoid structures to the *o*-,

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(11) The addition of alkali to nitro compounds in this manner, the alkali metal to the nitro group and the rest of the adding molecule to the *p*-position of the ring, has been shown to be probable in many instances, particularly by the work of Jackson and his collaborators in their investigations of the reactions of picryl chloride and of trinitroanisole and trinitrophenetole with sodium alcoholates, *Am. Chem. J.*, **20**, 448 (1898); **23**, 294 (1901).

m-, and *p*-nitrophenolate ions, and is, at the same time, in harmony with the known facts concerning *m*-quinone and the oxidation of *m*-diphenols.

CAMBRIDGE, MASS. RECEIVED DECEMBER 28, 1939

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF HARVARD UNIVERSITY]

Cyanates of Silicon, Phosphorus and Boron. Instability of Certain Ternary Boron Compounds

BY GEORGE S. FORBES AND HERBERT H. ANDERSON

Reynolds[1] prepared stable silicon thiocyanate (b. p. 314°) from silicon tetrachloride and lead thiocyanate in benzene. Two years later, Cocksedge,[2] upon treating boron bromide with silver thiocyanate, obtained boron thiocyanate as a white crystalline solid which grew dark even at 100°. Both offered some evidence that sulfur, not nitrogen, was linked to the non-metallic element. Dixon[3] reported the isolation of phosphorous thiocyanate from lead thiocyanate and phosphorus trichloride in commercial benzene as "a reddish-yellow, clear, dense oil which gave the reactions for thiocyanate, thiocarbimide and phosphorus." At 170° the contents of the flask "suddenly changed, without evolution of gas, to a sticky black solid which presently became hard and brittle." Dixon thought that this might be a polymer of $P(NCS)_3$, but was unable to substantiate his opinion.

It occurred to us to inquire whether these weakly electronegative elements would form stable cyanates, or isocyanates or both. Preparations were undertaken to obtain evidence bearing upon this issue.

Silicon Isocyanate and Cyanate

Preparation.—Eighty grams of silicon chloride was added with vigorous shaking, over a period of fifteen minutes to 250 cc. of pure benzene in which 290 g. of finely powdered silver (iso)cyanate was suspended. The flask was then heated on the steam-bath for half an hour. After filtration, and several washings with benzene, the solvent was distilled off at atmospheric pressure. The yield of crude liquid product, 72 g., was 78% referred to cyanate. Potassium and lead cyanates were much less reactive and led to much smaller yields even after forty hours of refluxing. A second preparation, starting with excess of silicon chloride, afforded a yield of 86%. After expulsion of silicon tetrachloride and benzene, the colorless liquid product was distilled under 25 mm. pressure. The boiling point rose sharply at the end, and the residue, a very pale yellow liquid, was found to be 2.5% of the whole.

Anal. Weighed portions of each fraction were converted into silica by hydrolysis, evaporation and ignition to constant weight all in the same crucible. The results follow.

Fraction of b. p. 185.6°, silicon found 14.22, 14.27, 14.46, 14.47%, average 14.35%.

Fraction of b. p. 247.2°, silicon found 14.06 and 14.56%, average 14.31%.

Calculated for $Si(NCO)_4$ and $Si(OCN)_4$, 14.32%.

The molecular weight of each fraction was determined in Dumas bulbs of about 50 cc. capacity, with the usual precautions.

186° fraction, mol. wt. found 202.5, 205.7, average 204.1.

247° fraction, mol. wt. found 211.9.

Calculated for $Si(NCO)_4$ and $Si(OCN)_4$, 196.1.

In addition, the molecular weight of the 186° fraction was determined by the freezing point method in benzene solution to be 204.1 and 194.4, average 199.2. Not enough of the higher boiling fraction for this determination remained.

Properties.—The boiling points were measured at various pressures by a dynamic method described, among others, by Mack and France.[4] Rubber connections were avoided by fused joints. The thermometers for these and other definitive measurements were standardized in the same apparatus with water, aniline and benzoic acid as primary standards, also with bromobenzene and benzoyl chloride. The plot of $\log_{10} p$ (mm.) for each liquid, against $1/T$ was a satisfactory straight line, and the logarithmic equations predicted the data within three millimeters, assuming the correctness of the temperatures. Densities at 20° were determined only by weighing the contents of a pipet. The index of refraction (white light), also at 20°, was measured with an

TABLE I

	$Si(NCO)_4$	$Si(OCN)_4$
B. p., °C., 760 mm.	185.6 ± 0.3	247.2 ± 0.5
M. p., °C.	26.0 ± 0.5	34.5 ± 0.5
$\log_{10} p$ (mm.)	$9.0198-2816/T$	$9.8211-3611/T$
λ_v, cal.	12,900	16,500
λ_v/T, cal./°	26.5	31.7
d, g./cc.	1.409, 1.413 ± 0.005	1.414 ± 0.005
n	1.4610 ± 0.0003	1.4646 ± 0.0003

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[1] Reynolds, *J. Chem. Soc.*, **89**, 397 (1906).

[2] Cocksedge, *ibid.*, **93**, 2177 (1908).

[3] Dixon, *ibid.*, **79**, 541 (1901).

[4] Mack and France, "Laboratory Manual of Physical Chemistry," D. Van Nostrand Co., Inc., New York, 1934, p. 77.