

CONCLUSION.

A satisfactory method for the determination of camphor in camphor liniment by the use of the vacuum oven has been proposed.

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ALKALOIDAL REAGENTS.* II. AROMATIC MONOHOMOCYCLIC DERIVATIVES.¹

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The behavior of seventeen reagents (21, 45), has been determined with a series of the simpler benzene derivatives. Detailed precipitate and color responses are recorded in the accompanying tables. To avoid the confusion of recording the threshold dilutions which just produced color or precipitate reactions with these reagents in terms of numerical values of Molar or Millimolar concentrations, it was decided to use a code, as follows:

a represents a concentration of $M/10$ or 100 Millimolar of a compound which proved to be the threshold concentration producing a reaction. The particular letter or letters designate the color of the solution or of the precipitate.

a represents a threshold concentration of $M/100$ or 10 Millimolar.

a represents a concentration of $M/500$ or 2 Millimolar.

A represents a concentration of $M/1000$ or 1 Millimolar.

A represents a concentration of $M/5000$ or 0.2 Millimolar.

A represents a concentration of $M/10,000$ or 0.1 Millimolar.

In general the standard procedure previously described has been followed; however, much of this work was completed before the necessity for more accurate definition of colors was appreciated. Most of the tests gave negative results for color as well as for precipitate formation, but they were made to be certain that there would be no interference in subsequent work. Those tests which are positive, and which later prove characteristic and definite, will be repeated and color findings reported in accordance with the Mulliken nomenclature (44).

1. BENZENE AND HYDROCARBON DERIVATIVES.

Benzene, toluene, *m*-xylene and phenyl-benzene (diphenyl) were tested. Since none of these substances was soluble in water to the extent of 100 millimols per liter, solutions saturated at room temperature were used. All of these substances gave negative results with all of the reagents.

2. PHENOLIC DERIVATIVES.

Detailed results upon the compounds investigated are given in Tables I and II. Pertinent comments found in the literature follow:

* Alkaloidal Reagents. I, 20 (1931), 1037.

¹ Scientific Section, Miami meeting, 1931.

PHENOL.

Reagent 1: Pollaci (50) reported that an aqueous phenol solution, stratified upon concentrated sulphuric acid, developed a red color at the contact zone.

Reagent 4: Pougnet (51) recommends a mixture of sulphuric acid and formaldehyde as a group reagent for phenols. Thoms (9, 63) observed a purple-red color with phenol.

Reagent 9: Autenrieth (3) reports that a very dilute solution of ferric chloride imparts a blue-violet color to 1:1000 phenol solutions (1000 mg. per liter). In our hands, this test was less sensitive. This test is also known as Deville's or Hirsch's test.

Reagent 12: Results with potassium permanganate are difficult to interpret and too much stress should not be placed upon its apparently great sensitivity.

Reagent 17: Autenrieth (3) heated Millon's reagent with the test solution and reported a distinct red color at a dilution of 1:100,000 (10 mg. per liter). Stronger solutions were reported to give a positive reaction at room temperature. Almen (2) obtained a yellow precipitate, soluble in nitric acid with a red color, from a solution containing as little as 2.5 mg. of phenol per liter. In our hands, no color was obtained with 100 millimolar solutions at room temperature (approximately 10,000 mg. per liter). This test is also called Hoffmann's test.

Other Reagents: (1) An excess of bromine water produces a yellowish white crystalline precipitate of tribromophenyl hypobromite with a solution containing as little as 20 mg. of phenol per liter. This test is also positive with cresols and polyphenols (3, 29, 36, 40, 41).

(2) Gently warming a mixture of ammonium hydroxide and calcium or sodium hypochlorite solution with phenol gave a blue color. Acidification converted this to red, and basification restored the blue color (2, 3, 5, 6, 16, 26, 33, 47, 56). Ortho- and meta-cresol give a blue color; para-cresol a dirty brownish color.

(3) Heating a mixture of sulphuric acid and benzaldehyde with phenol produces a dark red color and precipitate; basification produces a violet-blue color, which is soluble in ether (3, 39). This test is reported to be positive with a solution containing 500 mg. of phenol per liter. Of the three cresols, only ortho-cresol is reported to give this test.

(4) Freshly diazotized sulphanilic acid, or a 1:60 solution of diazobenzenesulphonic acid (48) gives a yellow color with phenol (20, 22). Ortho- and meta-cresol similarly give a yellow color, para-cresol a red color by this test.

(5) Defacqz heated tungstic acid with sulphuric acid containing a trace of potassium bisulphate, then added enough sulphuric acid to prevent solidification upon cooling. Trituration of this reagent with phenol developed a very intense red color (11).

(6) A pine shaving moistened with hydrochloric acid gave a blue color with phenol (55). Tommasi (64) and Hoppe-Seyler (25) use a mixture of hydrochloric acid and potassium chlorate.

(7) Upon adding ammonium hydroxide and tincture of iodine to an alcoholic solution of phenol, a green color develops which persists on heating or on acidification with hydrochloric acid (38).

(8) On adding phenol to a mixture of molybdic and sulphuric acids, a dark olive-green color appears, which changes to blue, then to violet (10).

(9) Stratification of phenol upon sulphuric acid containing nitric acid produces a blue color (34), Linde (35) states red color at the contact zone; if spirit of nitrous ether is used instead of nitric acid, a red color zone forms (15).

DIHYDROXYBENZENES.

Reagent 8: With formaldehyde and sulphuric acid resorcinol gave a yellow color, turning to red and finally to orange (63).

Reagent 9: Brieger (8) obtained an emerald-green color on mixing dilute ferric chloride with pyrocatechol; basification produced a violet color, which returned to green upon re-acidification (13).

Reagent 17: Millon's reagent (also known as Plugge's reagent (3)) gives an orange color with hydroquinone, and a yellow color with resorcinol, upon heating (42).

Other Reagents: (1) Upon adding ammonium hydroxide and tincture of iodine to an alcoholic solution of pyrocatechol, a "catechu" color is reported; with resorcinol, "old cognac;" with hydroquinone, a reddish brown color (38).

(2) Tungstic acid-sulphuric acid mixture gave a violet-black color with pyrocatechol; with resorcinol, red-brown; with hydroquinone, a very intense amethyst-violet color (11).

(3) Pyrocatechol reduces silver nitrate and Fehling's solution upon warming (46). Hydroquinone reduces Fehling's solution at room temperature (60).

(4) Fusion of resorcinol with phthalic anhydride, in the presence of zinc chloride, produces fluorescein (17).

(5) Seliwanoff (57) detects fructose by the development of a red color and precipitate upon heating with a hydrochloric acid solution of resorcinol. The precipitate dissolves in alcohol, giving a striking red color. Borchardt (7) showed that an alkaline solution of this precipitate in acetic ether was yellow.

(6) On heating an alkaline solution of resorcinol with chloroform, an intense crimson color is reported, which changes to pale yellow upon acidification with hydrochloric acid (14, 53, 65).

(7) With sodium hypochlorite solution, resorcinol gave a violet color, which changed to yellow on standing (6). Heating caused a change to yellowish red, then to brown. On adding ammonium hydroxide, a violet color developed, which changed to yellow at room temperature, and to dark green on boiling.

(8) A mixture of sodium hydroxide and formaldehyde boiled for half a minute with resorcinol, produced a distinct red color. The limit of delicacy for detection of formaldehyde was reported to be 1:10,000,000 (0.1 mg. per liter); the limit for resorcinol was not stated. This has been called Lebbin's test (31).

(9) An alkaline solution of 2,6-dibromoquinonechloroimide gave a red color with resorcinol (18, 53).

TRIHYDROXYBENZENES.

Reagent 4: Formaldehyde and sulphuric acid gave a red color with phloroglucinol (63).

Reagent 17: Millon's reagent gives a brown color (42).

(1) An alcoholic solution of phloroglucinol and vanillin, heated with free hydrochloric acid, gives a purplish red color (22, 43, 59). This reagent has been called Günzberg's reagent.

(2) Phloroglucinol, heated with hydrochloric acid and lignin, arabinose or a pentose, gives a red color (22, 43, 62).

(3) Phloroglucinol and pyrogallol reduce Fehling's solution and ammoniacal silver nitrate at room temperature (46, 49).

(4) Phloroglucinol added to toluidine and potassium nitrate solution developed a light yellow, opalescent solution which turned to orange, with the development of a cinnabar-red precipitate (67). This test is claimed to be sensitive at a dilution of 1 : 200,000 (5 mg. per liter).

(5) With tungstic acid-sulphuric acid mixture, pyrogallol gave a red-black color (11).

(6) Upon adding ammonium hydroxide and tincture of iodine to an alcoholic solution of pyrogallol, a black color is produced (38). An aqueous solution of pyrogallol is reported to give a lemon-yellow color (66).

(7) Pyrogallol gave a yellowish red color with potassium cyanide solution (19).

CRESOLS.

Some notes have been given under Phenols.

Reagent 8: Thoms (61) states that ortho-cresol gives a combination containing two mols of cresol and three mols of picric acid as orange-yellow needles. Meta- and para-cresols failed to form picrates.

(1) Upon adding ammonium hydroxide and tincture of iodine to an alcoholic solution of creosote or of the cresols, a brownish green color is produced (38).

(2) Upon complete sulfonation of a mixture of cresols and phenols (containing less than 10% of phenol) and subsequent nitration, meta-cresol quantitatively forms the trinitro derivative; ortho- and para-cresols form oxalic acid (44, 52).

(3) A mixture of tungstic acid and sulphuric acid produces an intense red-brown color with para-cresol (11).

(4) Sodium nitroprusside and potassium hydroxide produce a reddish yellow color with para-cresol; the addition of acetic acid converts this to light pink (27).

3. AMINO DERIVATIVES.

The compounds tested and results obtained are given in Tables III and IV.

ANILINE.

Reagent 2: With fuming nitric acid, aniline gave a deep blue solution, changing to yellow and to red on heating (24).

(1) Crystalline benzene- or toluene-sulfoneamides are obtained by mixing benzene- or toluene-sulphonyl chloride with an amine, in alkaline solution. Derivatives of the primary amines are soluble in alkalis (23, 44).

(2) Heating aniline or the toluidines with chloroform in the presence of a strong alkali produces an isonitrile, which has a characteristic disagreeable odor (3, 17, 24, 49).

(3) On mixing calcium or sodium hypochlorite solution with aniline, a violet or purplish red color develops, changing on standing to dirty red. Addition of a trace of phenol or ammonium hydroxide yields a stable blue color. The limit of sensitivity is reported to be 1 : 66,000 (16 mg. per liter) (3, 37, 44, 58, 63). Runge (55) claims that ammonium chloride must be absent. Jacquemin (26) treated a solution of aniline with calcium hypochlorite, followed by ammonium sulphide, obtaining a rose-red color, and reported a threshold concentration of 1 : 250,000 (4 mg. per liter).

(4) Aniline gives a flesh-colored precipitate with bromine water. The limit of sensitivity of this test is reported to be 1 : 66,000 (16 mg. per liter) (3, 49).

(5) Concentrated sulphuric acid mixed with a drop of aqueous potassium dichromate solution and aniline gives a blue color on standing. The addition of water produces an immediate blue color (3, 49). Using a crystal of potassium dichromate, Beissenhirtz (4) obtained a red color, which turned to blue and then disappeared.

(6) Aniline reduces gold chloride, precipitating metallic gold (58).

(7) Aniline heated with powdered mercuric chloride, gives a rich crimson dye (58).

(8) The addition of lead or manganese dioxide to a solution of aniline in sulphuric acid gives a green solution (12, 32).

(9) A solution of sodium phosphomolybdate in sulphuric acid or oxalic acid gives a yellow precipitate with aniline, which turns to blue and dissolves in ammonium hydroxide with the formation of a blue solution (24).

(10) A mixture of sulphuric acid and potassium ferricyanide gives a blue to purple color with aniline (32).

(11) Aniline salts produce a yellow color on pine shavings (55).

DIAMINES.

Reagent 9: Paraphenylene diamine gives a green color with 10% ferric chloride, which rapidly changes to brown, and after a minute or two, to red-violet (44).

(1) Bromine water gives a violet precipitate with metaphenylene-diamine (44).

(2) Nitrites give a deep yellow color with metaphenylenediamine; the detection of traces of nitrites is possible with this reagent (49).

(3) An alkaline solution of *a*-naphthol in 50% alcohol gives a purple solution with paraphenylene diamine (49).

(4) Paraphenylene diamine, mixed with blood and hydrogen peroxide, slowly develops an olive-green color. Upon adding a few drops of acetic acid, the color changes to brownish red (49).

TOLUIDINES.

(1) Acetyl chloride gives characteristic crystalline compounds, which serve to differentiate ortho- from para-toluidine. The meta-derivatives must be brominated for use (44).

(2) In a mixture of sulphuric acid and nitric acid, paratoluidine gives a blue to violet color, changing to red and finally to brown (30, 54).

DIAMINOTOLUENES.

No reactions were found for the two compounds used.

For 1-CH₃,3,5-diaminobenzene, Mulliken (44) reported a characteristic diacetyl derivative; for 1-CH₃,2,6-diaminobenzene, a characteristic diacetyl derivative, also a deep blue color with ferric chloride are reported.

AMINODIPHENYLS.

(1) The 1-NH₂ compound is reported to form a characteristic acetyl derivative, also a chlorplatinate (44).

(2) The 3,8-diamino compound, benzidine, gives a voluminous deep blue precipitate with potassium dichromate (28). It also forms a white precipitate with sulfates, which is very slightly soluble in water.

(3) Benzidine, mixed with blood and hydrogen peroxide, gives a blue or blue-green color (1, 3, 43, 49). The limit of sensitivity for blood has been reported as 1 : 100,000 (10 mg. per liter) although we have obtained positive tests with dilutions of 1 mg. per liter (1 : 1,000,000) as well as with weaker solutions under proper conditions.

4. AMINOPHENOLS.

The compounds used and the results observed are recorded in Tables V and VI.

(1) Meta-aminophenol forms a deep red picramide with picryl chloride (44).

(2) Para-aminophenol combines with benzaldehyde to form a pale yellow crystalline compound (44).

(3) The 2,4-diamino compound is precipitated from aqueous solutions of hydrochloric acid by sodium sulfite as leaflets. The picrate forms lemon-yellow needles. The solution in ammonium hydroxide becomes deep blue upon exposure to air (44).

CONCLUSION.

The behavior of seventeen alkaloidal reagents has been determined with substituted benzenes, in which the substituent groups have been alkyl, hydroxyl, phenyl and amino groups, alone or in combination. In addition, characteristic reactions for these compounds found in the literature have been reported.

TABLE I.—PRECIPITATE FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
Benzene																	
1-OH				<i>rw</i>													
Dihydroxy																	
1,2 OH			<i>dg</i>	<i>p</i>	<i>A</i>					<i>db</i>						<i>g</i>	<i>b</i>
1,3 OH				<i>r</i>								<i>B</i>				<i>blw</i>	<i>W</i>
1,4 OH				<i>b</i>	<i>B</i>							<i>b</i>				<i>b</i>	
Trihydroxy																	
1,2,3 OH				<i>b</i>	<i>B</i>					<i>B</i>						<i>b</i>	
1,3,5 OH				<i>ry</i>								<i>b</i>				<i>w</i>	<i>YW</i>
Cresols:																	
1-OH, 2-CH ₃		<i>ry</i>		<i>w</i>								<i>b</i>				<i>w</i>	
1-OH, 3-CH ₃				<i>w</i>												<i>w</i>	
1-OH, 4-CH ₃		<i>ry</i>		<i>w</i>												<i>w</i>	

Reagent 1: H ₂ SO ₄	Reagent 10: K ₂ Cr ₂ O ₇	Key to Code:
..... 2: HNO ₃ 11: K ₄ FeCN ₆	<i>M</i> /10 or 100 Millimol. . . . <i>a</i>
..... 3: Froehde's 12: KMnO ₄	<i>M</i> /100 or 10 Millimol. . . . <i>a</i>
..... 4: Marquis 13: K ₂ PtCl ₄	<i>M</i> /500 or 2 Millimol. . . . <i>a</i>
..... 5: Mayer 14: Schiebler	<i>M</i> /1000 or 1 Millimol. . . . <i>A</i>
..... 6: Dragendorff 15: Uranium acetate	<i>M</i> /5000 or 0.2 Millimol. . . . <i>A</i>
..... 7: Wagner 16: SeO ₂	<i>M</i> /10,000 or 0.1 Millimol. . . . <i>A</i>
..... 8: Picric Acid 17: Millon	
..... 9: FeCl ₃		

A denotes amber; b, brown; bl, blue; d, dark; g, green; p, purple; r, red; v, violet; w, white; y, yellow.

TABLE II.—COLOR FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
Benzene																	
1-OH		y	r						p			B					
Dihydroxy																	
1,2 OH		rY	B						g	B		B			rb		
1,3 OH		Y							p								
1,4 OH												B					
Trihydroxy																	
1,2,3,OH		bY	bY		Y				By			B		RB	R		
1,3,5,OH			rY						v			B					
Cresols:																	
1-OH, 2-CH ₃		Y							g			B					r
1-OH, 3-CH ₃		r							p			B					r
1-OH, 4-CH ₃									bl			B					r

TABLE III.—AMINO DERIVATIVES PRECIPITATE FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
Benzene:																	
1-NH ₂							w					B					
Diamino:																	
1,2-NH ₂							B	Y	r	RB				r			r
1,3-NH ₂							W		r			b					W
1,4-NH ₂							b	y		b				w			w
Aminotoluenes:																	
1-CH ₃ , 2-NH ₂									y								w
1-CH ₃ , 3-NH ₂						w	b	y				b					w
1-CH ₃ , 4-NH ₂												B					w
Diaminotoluene:																	
1-CH ₃ , 2,5-NH ₂																	w
1-CH ₃ , 2,4-NH ₂																	w
Aminodiphenyl:																	
1-NH ₂			w	w	w		wb	y		g	w	r	w	w	w		w
3-NH ₂																	Y*
3,8-NH ₂	w	w	w	W		w		Y		P	W	b	W	w			w

TABLE IV.—AMINO DERIVATIVES COLOR FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
Benzene:																	
1-NH ₂														RB			
Diamino:																	
1,2-NH ₂			R							RY							
1,3-NH ₂			rY							R			bY				
1,4-NH ₂			bY							p	B		by				
Aminotoluenes:																	
1-CH ₃ , 2-NH ₂			Y										bl				
1-CH ₃ , 3-NH ₂													B				
1-CH ₃ , 4-NH ₂													rb				

TABLE IV.—Continued.

Product.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
Diaminotoluene:																	
1-CH ₃ , 2,5-NH ₂								g		G		g					
1-CH ₃ , 2,4-NH ₂												rb					
Aminodiphenyl:																	
1-NH ₂		Y										bl					
3-NH ₂		Y*															
3,8-NH ₂		Y	Y						rb			G					

TABLE V.—AMINOPHENOLS PRECIPITATE FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
1-OH, 2-NH ₂									r								
1-OH, 3-NH ₂																	Y
1-OH, 4-NH ₂																	
1-OH, 2,4-NH ₂																	r

TABLE VI.—AMINOPHENOLS COLOR FORMATION:

Product.	Reagent No.																
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	17.
1-OH, 2-NH ₂		rY							rY	rY				g			
1-OH, 3-NH ₂										r							
1-OH, 4-NH ₂		r							p								
1-OH, 2,4-NH ₂		R							R	R		R					R

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