was heated at 190–195° for 12 hours. The reaction mixture was cooled, diluted with water and worked up to give 51 mg. (quantitative from phenylcyclohexanone) of phenylcyclohexane as a mobile liquid, identical with an authentic sample as shown by infrared absorption and vapor phase chromatography.

[CONTRIBUTION FROM THE BOUND BROOK LABORATORIES, AMERICAN CYANAMID CO., BOUND BROOK, N. J.]

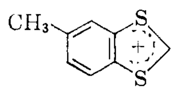
The 1,2-Dithiolium Cation. A New Pseudoaromatic System. II. Condensation of Phenyl-1,2-dithiolium Salts with Tertiary Aromatic Amines[1]

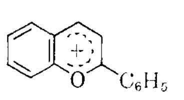
BY ERWIN KLINGSBERG AND ANN M. SCHREIBER

RECEIVED JANUARY 26, 1962

Phenyldithiolium salts condense with tertiary aromatic amines, giving rise to a new family of basic dyes, which may also be obtained from 1,2-dithiole-3-ones and 1,2-dithiole-3-thiones. Spectral properties are discussed.

Electrophilic substitutions by pseudoaromatic cations constitute an interesting class of carbonium ion reactions, recently investigated by Wizinger and by Shriner. Thus dimethylaniline is attacked by the methylbenzo-1,3-dithiolium (I) and flavylium


I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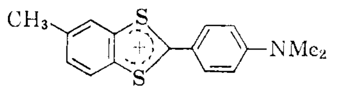
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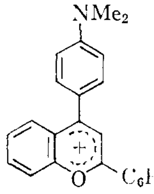
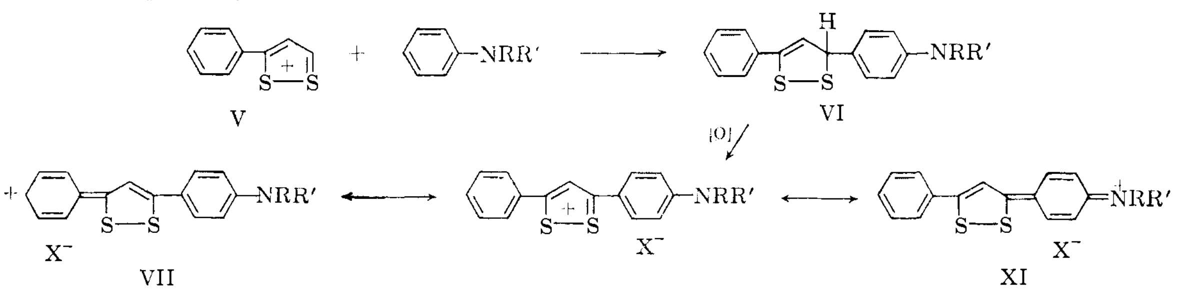

IV

(II) cations to give the highly colored salts of III[2] and IV,[3a,b] respectively.

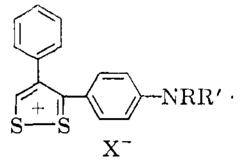


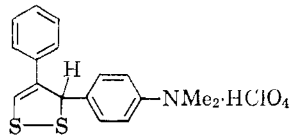
The recently discovered phenyl-1,2-dithiolium salts[4] also show electrophilic activity. Reaction occurs in a few minutes in warm ethanol with dimethylaniline or other tertiary aromatic amines, giving intense violet dyes readily isolated as perchlorates. Assignment of structure VIII and XII to these products is based on elementary analysis and the following considerations: Substitution in the phenyl ring *para* to the dimethylamino group was confirmed by the finding that N,N-dimethyl-*m*-toluidine reacted much like dimethylaniline, giving a very similar product, while N,N-dimethyl-*p*-toluidine did not react under the same conditions. As for the dithiolium ring, the natural expectation that substitution would occur at the positive 3- (or 5-) position rather than the neutral 4-position[4] is borne out by the finding that the 3- and 4-phenyldithiolium salts react under similar conditions to give products of similar character; the 4-phenyl isomer obviously cannot react at the 4-position.

Two additional syntheses of VIII provide further proof of structure and further analogies between the chemistry of the 1,2-dithiolium system and the older benzo-1,3-dithiolium (I) and flavylium (II) systems. Since flavone condenses with dimethylaniline in phosphorus oxychloride to give IV,[3a] it was of interest to demonstrate that under these conditions 5-phenyl-1,2-dithiole-3-one (XIV) gives VIII.

Secondly, Soder and Wizinger[2] recently have shown that III is obtained from the benzo-1,3-dithiole-2-thione (XV) *via* its methylsulfonium salt (XVI).

VII, R = R′ = Me (λ_{max} 557)
VIIIa, X = ClO_4
b, X = I
IX, R = Me; R′ = Ph (λ_{max} 550)
X = ClO_4
X, R = Me; R′ = CH_2CH_2CN (λ_{max} 537)
X = ClO_4




XIIa

XII, R = R′ = Me (λ_{max} 544)
X = ClO_4
XIII, R = Me; R′ = CH_2CH_2CN
X = ClO_4 (λ_{max} 525)

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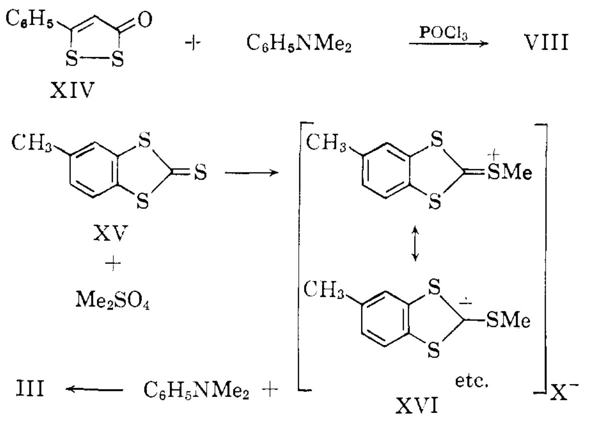
(1) Presented on March 27, 1962, at the 141st National Meeting of the American Chemical Society, Washington, D. C.

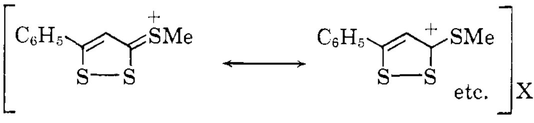
(2) L. Soder and R. Wizinger, *Helv. Chim. Acta*, **42**, 1779 (1959).

(3) (a) R. Wizinger and A. Luthiger, *ibid.*, **36**, 526 (1953). (b) R. L. Shriner, in "The Roger Adams Symposium," John Wiley and Sons, Inc., New York, N. Y., 1955, p. 103.

(4) For the first paper in this series, see E. Klingsberg, *J. Am. Chem. Soc.*, **83**, 2934 (1961).



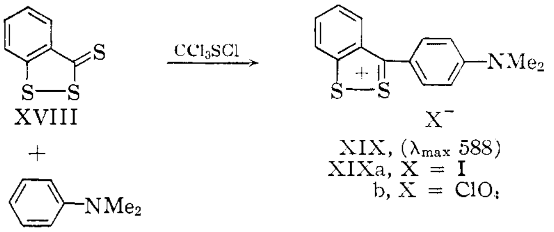
The methylsulfonium salts of the 1,2-dithiole-3-thiones are known as "trithionium salts" (XVII); in them, as in XVI, the charge is distributed between the ring and the side chain[5]



XVII

The extension of Soder and Wizinger's synthesis to a trithionium salt was, in fact, reported[6] during the course of our investigation. We have prepared VIII from XVII and dimethylaniline; this synthesis constitutes further proof of structure.

In the preparation of XIX from XVIII and dimethylaniline, trichloromethanesulfenyl chloride proved to be an effective catalyst; the reaction proceeds very rapidly at room temperature, with instantaneous development of the intense blue color of the product.

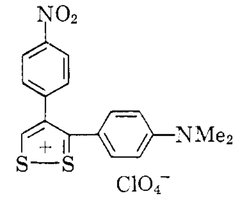


XIX, (λ_{max} 588)
XIXa, X = I
b, X = ClO_4

Presumably the trichloromethanesulfenyl chloride initiates the reaction by attacking the extracyclic sulfur atom to give an intermediate analogous to a trithionium salt but with a more labile side chain, readily eliminated in the condensation with dimethylaniline.

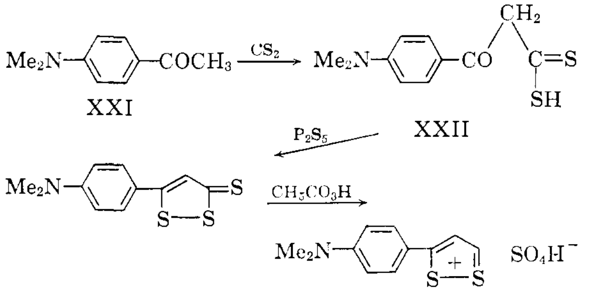
In the formation of VIII from 3-phenyldithiolium salts, the leuco intermediate (presumably VI) sometimes separates from the reaction mixture as a colorless solid unstable in air because of the strong tendency to dehydrogenate. The 4-phenyl isomer gives a somewhat more stable leuco base that has been characterized as a perchlorate believed to be XIIa. Leuco products are also obtained in the condensation of flavylium salts with dimethylaniline.[7]

When the reaction is run without added oxidizing agent, the dehydrogenation of leuco base to dye evidently occurs at the expense of unreacted dithiolium salt, which is itself reduced to hydrogen sulfide, detectable by its odor. Atmospheric oxidation seems to be unimportant, since the reaction runs about as well in the absence of air. The best yields (60–70%) are given with the aid of potassium persulfate as oxidizing agent. Even in its presence, however, results are not fully reproducible and leuco product is sometimes formed, for reasons that are not clear. Thus a mixture of the nitro dye XX and its leuco derivative was obtained in the condensation of 4-(*p*-nitrophenyl)-1,2-dithiolium hydrogen sulfate with dimethylaniline.



XX, (λ_{max} 545)

3-(*p*-Dimethylaminophenyl)-1,2-dithiolium hydrogen sulfate (XXIV), the prototype of this family of basic dyes, was prepared from the dithiolethione (XXIII), which we obtained by an application of the Thuillier–Vialle synthesis[8] to *p*-dimethylaminoacetophenone (XXI), conveniently prepared by reductive methylation of *p*-aminoacetophenone. This and carbon bisulfide gave the ketodithioacid XXII, which reacted with phosphorus pentasulfide to give the dithiolethione XXIII in an over-all yield of 79% from XXI; this compares well with the previously reported crude yield of less than 1% from ethyl *p*-dimethylaminocinnamate.[9]



XXIII XXIV (λ_{max} 535)

The intense color of these dyes evidently is associated with the wide charge separation due to resonance between dithiolium valence bond structures such as VIII and the quaternary ammonium dithiole form XI. Under strongly acidic conditions, protonation of the amino group suppresses this resonance and extinguishes the color. With λ_{max} at 557, VIII is somewhat bathochromic to its isomer XII (λ_{max} 544). This conforms to the pattern set by the ultraviolet absorption of the parent 3- and 4-phenyldithiolium salts and sug-

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(5) J. Teste and N. Lozac'h, *Bull. soc. chim. France*, 437 (1955); A. Lüttringhaus and U. Schmidt, *Chem.-Ztg.*, **77**, 135 (1953).

(6) U. Schmidt, R. Scheuring and A. Lüttringhaus, *Ann.*, **630**, 116 (1960).

(7) R. L. Shriner and J. A. Shotton, *J. Am. Chem. Soc.*, **74**, 3622 (1952).

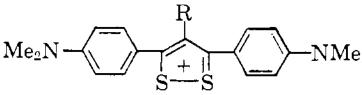
(8) A. Thuillier and J. Vialle, *Bull. soc. chim. France*, 1398 (1959).

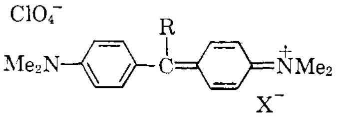
(9) B. Böttcher and F. Bauer, *Ber.*, **84**, 458 (1951).

gests resonance contributions by such forms as VII, which have no counterpart when the phenyl group is at the 4-position. The presence of the basic dimethylamino group naturally limits the importance of such forms, and the difference in λ_{max} between VIII and XII is only 13 mμ, compared to 114 mμ in the isomeric parent cations.[4] The dye XXIV, with no phenyl substituent, has λ_{max} 535 mμ and is thus slightly hypsochromic to XII.

The fused ring dye XIX shows a major bathochromic shift, with λ_{max} at 588; thus it is also highly bathochromic to the 1,3-dithiolium analog III (λ_{max} 536), with which it is isomeric except for the Bz methyl group.[2]

Despite the reduction in electrophilic activity caused by the presence of the dimethylamino group, we find, somewhat unexpectedly, that XXIV and XII are able to condense with a second mole of dimethylaniline to give XXV and XXVI, respectively. These are dithiolium counterparts of





XXV, R = H(λ_{max} 588) XXVII, R = H (λ_{max} 604)
XXVI, R = C_6H_5 (λ_{max} 586) XXVIII, R = C_6H_5 (λ_{max} 616)

classical basic dyes: Michler's hydrol blue (XXVII) in the diphenylmethane series and malachite green (XXVIII) in the triphenylmethane series. In the latter two dyes, the phenyl substituent exerts an appreciable bathochromic influence, attributable to its electron affinity in favoring the release of electrons from the terminal nitrogen atoms.[10] In the dithiolium dyes XXV and XXVI, on the other hand, the phenyl group at the 4-position cannot conjugate with the system and has almost no effect on the position of the absorption maximum.

Experimental

Melting points are corrected; they sometimes vary with the rate of heating.

3-Phenyl-5-(*p*-dimethylaminophenyl)-1,2-dithiolium Perchlorate (VIIIa).—Potassium persulfate (2.0 g., 7.4 millimoles) was added to a solution of 2.00 g. (7.25 millimoles) of 3-phenyl-1,2-dithiolium hydrogen sulfate and 1.50 ml. (1.43 g., 11.8 millimoles) of dimethylaniline in 100–125 ml. of ethanol. The reaction mixture was stirred and refluxed for 19 hours, filtered, and cooled. Treatment with 2.0 ml. of 70% perchloric acid gave 0.85–1.70 g. (30–60%) of metallic-green product, m.p. 226–229° dec. Crystallization from methanol raised the dec. temperature to 246–247°. In 0.1 *N* alcoholic HCl (concn. 10 mg./liter) it had λ_{max} 557 mμ (ε 38,000).

Anal. Calcd. for $C_{17}H_{16}ClNO_4S_2$: C, 51.4; H, 4.0; Cl, 8.9; N, 3.5; S, 16.1. Found: C, 51.5; H, 3.9; Cl, 9.1; N, 3.5; S, 16.3.

Under the same conditions N,N-dimethyl-*m*-toluidine gave 3-(*p*-dimethylamino-*o*-methylphenyl)-5-phenyl-1,2-dithiolium perchlorate, crystallizing from methanol as green needles, m.p. 257–258° dec.

Anal. Calcd. for $C_{18}H_{18}ClNO_4S_2$: C, 52.4; H, 4.4; Cl, 8.6; S, 15.6. Found: C, 52.5; H, 4.2; Cl, 8.7; S, 15.8.

Preparation of VIIIa from Trithionium Salt.—5-Phenyl-1,2-dithiole-3-thione was methylated in benzene solution by refluxing for 2 hours with excess methyl sulfate, then filtering and washing with a little benzene. One gram of the product (XVII, X = $MeSO_4$) and 1.0 ml. of dimethylaniline were stirred and refluxed for 20 minutes in 10 ml. of glacial acetic acid and 1.0 ml. of pyridine. The purple solution was cooled and treated with 0.6 ml. of 70% perchloric acid, yielding 1.1 g. (92%) of green product. Recrystallization from methanol gave 0.55 g. (46%) of bright green needles identical with VIIIa by m.p., mixed m.p. and other properties.

Condensation of 5-Phenyl-1,2-dithiole-3-one with Dimethylaniline.—A mixture of 5.0 g. (0.026 mole) of 5-phenyl-1,2-dithiole-3-one, 5.0 ml. of dimethylaniline and 20 ml. of phosphorus oxychloride was refluxed gently for 15 minutes, cooled, diluted with 15 ml. of acetic acid, decomposed with ice, diluted to 2 liters with warm water, and filtered, giving a recovery of 3.7 g. of starting material. One-half of the purple filtrate was precipitated with perchloric acid, giving 0.45 g. (1.1 millimole) of 3-phenyl-5-(*p*-dimethylaminophenyl)-1,2-dithiolium perchlorate (VIIIa), m.p. 245°. Precipitation of the other half of the filtrate with potassium iodide gave 0.48 g. (1.1 millimole) of 3-phenyl-5-(*p*-dimethylaminophenyl)-1,2-dithiolium iodide (VIIIb), dec. 236–238°. Crystallization from butanol gave green-violet needles, dec. 245°.

Anal. Calcd. for $C_{17}H_{16}INS_2$: C, 48.0; H, 3.8; I, 29.9; N, 3.3. Found: C, 48.3; H, 4.0; I, 29.5; N, 3.2.

3-[*p*-(N-Methyl-N-phenylamino)-phenyl]-5-phenyl-1,2-dithiolium Perchlorate (IX).—A mixture of 2.00 g. (7.25 millimoles) of 3-phenyl-1,2-dithiolium hydrogen sulfate, 2.0 g. (7.4 millimoles) of potassium persulfate and 1.58 g. (8.6 millimoles) of N-methyldiphenylamine in 125 ml. of ethanol was stirred and refluxed for 2.5 hours and then filtered. Treatment of the filtrate with perchloric acid gave 1.91 g. (57%) of product, m.p. 221–223°. It formed dark green crystals from propanol, unchanged in m.p. (λ_{max} 550).

Anal. Calcd. for $C_{22}H_{18}ClNO_4S_2$: C, 57.5; H, 3.9; Cl, 7.7, N, 3.0; S, 13.9. Found: C, 57.4; H, 3.6; Cl, 7.5; N, 3.1; S, 13.8.

3-[*p*-(N-Cyanoethyl-N-methylamino)-phenyl]-4-phenyl-1,2-dithiolium perchlorate (XIII).—A mixture of 2.00 g. (7.25 millimoles) of 4-phenyl-1,2-dithiolium hydrogen sulfate, 1.0 g. (3.7 millimoles) of potassium persulfate and 1.20 g. (7.50 millimoles) of N-cyanoethyl-N-methylaniline in 50 ml. of ethanol was stirred and refluxed for 2 hours, and then filtered. Treatment of the filtrate with 2.0 ml. of 70% perchloric acid gave 2.1 g. (66%) of product, m.p. 224–227° dec. Crystallization from ethylene glycol monomethyl ether gave green needles, m.p. 238–239° dec. (λ_{max} 525).

Anal. Calcd. for $C_{19}H_{17}ClN_2O_4S_2$: C, 52.2; H, 3.9; Cl, 8.1; N, 6.4; S, 14.7. Found: C, 52.5; H, 4.0; Cl, 8.2; N, 6.4; S, 14.9.

The isomeric 5-phenyl compound X crystallized as green needles from ethylene glycol monomethyl ether, m.p. 255–257° dec. (λ_{max} 537).

Anal. Found: C, 52.2; H, 4.0; Cl, 8.2; N, 6.4; S, 15.0.

Under these conditions 4-phenyl-1,2-dithiolium hydrogen sulfate and dimethylaniline gave 3-(*p*-dimethylaminophenyl) 4-phenyl-1,2-dithiolium perchlorate (XII), crystallizing as green needles from methanol; m.p. 236–237° dec. (λ_{max} 544 mμ, ε 31,600).

Anal. Calcd. for $C_{17}H_{16}ClNO_4S_2$: C, 51.4; H, 4.0; Cl, 8.9; N, 3.5; S, 16.1. Found: C, 51.6; H, 4.2; Cl, 9.1; N, 3.6; S, 16.2.

The leuco form XIIa of this dye was obtained as follows: Two grams (0.0073 mole) of 4-phenyl-1,2-dithiolium hydrogen sulfate and 1.5 ml. (1.4 g., 0.012 mole) of dimethylaniline were stirred and refluxed for 1 hour in 50 ml. of ethanol and then filtered at boiling. Addition of 2.0 ml. of 70% perchloric acid to the filtrate gave 2.0 g. of pale purple solid, which was recrystallized repeatedly from methanol to give 70 mg. of yellow needles, m.p. 179–182° dec.

Anal. Calcd. for $C_{17}H_{18}ClNO_4S_2$: C, 51.0; H, 4.5; Cl, 8.9; N, 3.5; S, 16.0. Found: C, 51.1; H, 4.2; Cl, 8.7; N, 3.6; S, 16.4.

4,5-Benzo-1,2-dithiole-3-thione (XVIII).[11]—Twenty grams (0.065 mole) of 2,2′-dithiodibenzoic acid and 20 g. of phos-

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(10) M. J. S. Dewar, "The Electronic Theory of Organic Chemistry," Oxford Press, London, Eng., 1949, p. 305.

(11) F. S. Fowkes and E. W. McClelland, *J. Chem. Soc.*, 187 (1941).

phorus pentasulfide were refluxed for 45 minutes in 150 ml. of pyridine, cooled, and diluted with water. Filtration and subsequent washing with cold alcohol gave an 80–90% yield of orange product, m.p. 92–94°. Crystallization from methylcyclohexane gave short orange needles, m.p. 94–96°.

4,5-Benzo-3-(*p*-dimethylaminophenyl)-1,2-dithiolium Iodide (XIXa).—To a solution of 1.84 g. (0.010 mole) of 4,5-benzo-1,2-dithiole-3-thione (XVIII) and 1.30 ml. (1.26 g., 0.010 mole) of dimethylaniline in 50 ml. of pyridine, stirred in an ice-bath, was added 1.1 ml. (1.9 g., 0.010 mole) of trichloromethanesulfenyl chloride. After 5 minutes the dark blue solid was filtered, dissolved in 40 ml. of propanol, and treated with 2.0 ml. of 57% HI. The dark green product was filtered; yield 2.0 g. (50%), m.p. 230–231° dec. It was crystallized from methanol without change in m.p.

Anal. Calcd. for $C_{15}H_{14}INS_2$: C, 45.2; H, 3.5; I, 31.8; N, 3.5; S, 16.1. Found: C, 45.4; H, 3.4; I, 31.9; N, 3.5; S, 16.1.

In some experiments the m.p. varied between 220° and 240° dec.

The corresponding perchlorate XIXb crystallized from ethylene glycol monomethyl ether as green needles, m.p. 225° dec., with λ_{max} 588 (ϵ 29,000) at a concentration of 10 mg./liter in 0.1 *N* alcoholic HCl.

Anal. Calcd. for $C_{15}H_{14}ClNO_4S_2$: C, 48.5; H, 3.8; Cl, 9.6; N, 3.8; S, 17.2. Found: C, 48.5; H, 3.7; Cl, 9.7; N, 3.9; S, 17.1.

3-(*p*-Dimethylaminophenyl)-4-(*p*-nitrophenyl)-1,2-dithiolium Perchlorate (XX).—A mixture of 3.5 g. (0.0108 mole) of 4-(*p*-nitrophenyl)-1,2-dithiolium hydrogen sulfate,[4] 1.5 g. (0.0055 mole) of potassium persulfate and 1.6 ml. (1.5 g., 0.013 mole) of dimethylaniline in 100 ml. of ethanol was stirred and refluxed for 40 minutes and filtered at boiling. Treatment of the purple filtrate with 1.0 ml. of 70% perchloric acid gave a purple gum, which formed a mixture of purple and yellow crystals from methanol; more of this mixture was obtained by concentration of the mother liquor remaining after removal of the gum. The purple product was extracted with refluxing glacial acetic acid, from which it separated on cooling. Two crystallizations from methanol gave 25 mg. of purple plates, m.p. 240–242° dec., λ_{max} 545 in alcohol.

Anal. Calcd. for $C_{17}H_{15}ClN_2O_6S_2$: C, 46.1; H, 3.4; N, 6.3. Found: C, 46.2; H, 3.9; N, 6.2.

The portion insoluble in acetic acid was crystallized twice from ethanol to give 70 mg. of the hydroperchlorate of the leuco form of XX, yellow needles, m.p. 222–225° dec.

Anal. Calcd. for $C_{17}H_{17}ClN_2O_6S_2$: C, 45.8; H, 3.8; N, 6.3. Found: C, 46.2; H, 4.2; N, 6.3.

p-Dimethylaminoacetophenone (XXI).—A solution of 6.8 g. (0.050 mole) *p*-aminoacetophenone in 40 ml. of ethanol and 2.5 ml. of 37% HCl was cooled externally to 10°. To this were added 8.0 ml. (0.100 mole) of formalin and an ethanol suspension of freshly prepared platinum black. The mixture was reduced in a Parr apparatus under 35 lb. of hydrogen pressure. The catalyst was removed by filtration and the filtrate neutralized with ammonium hydroxide. Dilution with water and ice precipitated 6.5 g. (80%) of product. Crystallization from cyclohexane gave 5.4 g. (66%) of cream-colored plates, m.p. 101.5–103°.

p-Dimethylaminobenzoyldithioacetic Acid (XXII).—To an externally cooled solution of sodium *t*-amylate (0.050 mole) in 45 ml. of benzene was added dropwise a solution of 4.1 g. (0.025 mole) of *p*-dimethylaminoacetophenone and 1.5 ml. (0.025 mole) of carbon disulfide in 35 ml. of benzene. The orange sodium salt of the product precipitated. It was filtered and then dissolved in 150 ml. of water. Neutralization with 5 *N* sulfuric acid precipitated 5.4 g. (90%) of red product that was used in the next step of the synthesis. A specimen was crystallized for analysis from dioxane; m.p. 214–216° dec.

Anal. Calcd. for $C_{11}H_{13}NOS_2$: C, 55.2; H, 5.5; N, 5.9; S, 26.7. Found: C, 55.4; H, 5.4; N, 6.1; S, 26.5.

5-(*p*-Dimethylaminophenyl)-1,2-dithiole-3-thione (XXIII).—To 350 ml. of refluxing pyridine was cautiously added 18.7 g. (0.085 mole) of P_2S_5, followed by 16.7 g. (0.070 mole) of XXII. The resulting solution was refluxed 4.5 hours and then diluted with water and ice, precipitating 15.3 g. (87.5%) of brown product, m.p. 201° dec. Crystallization from butyl acetate yielded 10.0 g. (57%) of purple needles, m.p. 207–210° dec.

Anal. Calcd. for $C_{11}H_{11}NS_3$: C, 52.1; H, 4.4; N, 5.5; S, 37.9. Found: C, 52.4; H, 4.3; N, 5.7; S, 37.8.

3-(*p*-Dimethylaminophenyl)-1,2-dithiolium Hydrogen Sulfate (XXIV).—To a solution of 2.5 g. (0.010 mole) of XXIII in 800 ml. of acetone was added dropwise 5.0 ml. (0.030 mole) of 40% peracetic acid in 50 ml. of acetone during 30 minutes with stirring and external cooling. A gummy red-violet product (3.0 g., 94%) separated. Crystallization from glacial acetic acid gave 2.3 g. (72%) of purple needles, m.p. 209–210° dec. Repeated crystallization raised the m.p. to 214–215° dec. In 0.1 *N* HCl (concn. 5 mg./liter) it had λ_{max} 535 mμ (ϵ 39,000).

Anal. Calcd. for $C_{11}H_{13}NO_4S_3$: C, 41.4; H, 4.1; N, 4.4; S, 30.1. Found: C, 41.0; H, 3.8; N, 4.6; S, 29.8.

3,5-Bis-(*p*-dimethylaminophenyl)-1,2-dithiolium Perchlorate (XXV).—A solution of 2.0 g. (0.0063 mole) of XXIV and 1.0 ml. (1.0 g.; 0.008 mole) of dimethylaniline in 50 ml. of ethanol was stirred and refluxed for 88 hours. Addition of perchloric acid precipitated 0.60 g. (20.6%) of green product of m.p. 231–235° dec. Two crystallizations from methoxyethanol gave 0.15 g. green needles, m.p. 269–271° dec. In 0.1 *N* alcoholic HCl this showed absorption maxima at 588 (ϵ 93,000) and 495 (ϵ 22,000) mμ.

Anal. Calcd. for $C_{19}H_{21}ClN_2O_4S_2$: C, 51.7; H, 4.8; N, 6.4; S, 14.5. Found: C, 51.7; H, 4.9; N, 6.5; S, 15.0.

3,5-Bis-(*p*-dimethylaminophenyl)-4-phenyl-1,2-dithiolium Perchlorate (XXVI).—A solution of 0.50 g. (0.0013 mole) of 3-(*p*-dimethylaminophenyl)-4-phenyl-1,2-dithiolium perchlorate and 1.0 ml. (0.008 mole) of dimethylaniline in 50 ml. of ethanol was stirred and refluxed for 72 hours. Dilution with water precipitated 0.25 g. (37%) of product, m.p. 264.5° dec. Recrystallization from methanol gave 0.12 g. of golden plates, m.p. 263–265° dec. In 0.1 *N* alcoholic HCl this had λ_{max} 586 mμ (ϵ 64,000).

Anal. Calcd. for $C_{25}H_{25}ClN_2O_4S_2$: C, 58.0; H, 4.9; Cl, 6.9; N, 5.4; O, 12.4; S, 12.4. Found: C, 57.8; H, 5.0; Cl, 7.1; N, 5.4; O, 12.3; S, 12.7.

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