

Organic Polarography

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THIS REVIEW covers articles which have appeared in *Chemical Abstracts* and readily available journals during the two-year period ending November 1, 1965. As in the past, duplications and reinvestigations of previous work appear widely, and the tendency for authors to publish the same work in more than one journal has continued.

This period has seen the publication of a special issue of *Talanta* honoring the 70th year for Professor Izaak M. Kolthoff (411). This issue is comprised entirely of invited contributions of his former students and associates and a list of Professor Kolthoff's past publications. Best wishes go to Professor Kolthoff for many more years of fruitful labor.

Polarographic advances in Japan (704, 800, 801) and in Prague (429) were reviewed.

The use of electron spin resonance spectrometry in evaluating electrode reactions in which free radicals are formed has continued to grow (3, 25). Books dealing with "Polarography of Polymers" (64) and "Organic Polarographic Analysis" (947) have been published. Two books containing a listing of literature covering polarography through 1964 also became available (340, 341).

Numerous reviews have appeared on the applications of polarography to pesticide analysis (98, 224, 225, 524), lignin chemistry (627), cytostatically active compounds (297), smoke analysis (700), proteins (114, 557, 893), pharmaceuticals and drugs (174, 242, 249, 329, 659, 917), petroleum analysis (171, 318, 942), polymers (63), biochemical analysis (661), feed medicament analysis (9), clinical chemistry (104), and enzyme activity (435). Other reviews have covered organic analysis (119, 169, 330, 331), organic polarography (496, 543, 583, 773, 805, 897, 914, 946) and application to electrochemical synthesis (285).

Specialized polarographic reviews were concerned with alkaloids and their analysis (706), the effect of pH on half-wave potentials (176), utilization in assigning ring and chain structures and following the rate of tautomerism (344), polarography of enediols (109), substituted 3-phenyl-sydnone (767), carbon disulfide and derivatives (639) and organic peroxides (269), the study of electron and proton transfer (6), the

progress and problems in electrode kinetics (84) and the techniques for studying kinetics of oxidation (210).

Reviews on adsorption of organic compounds at electrode surfaces (13, 218, 443) and effect on reduction (493), a theoretical treatment of adsorption (136), and studies in adsorption isotherms (148) have appeared. Oscillographic and alternating current polarography were used to study the effects of anionic, cationic, and neutral surface active agents on polarographic analysis (337). Additional studies on adsorption are included under specific compounds.

Studies in nonaqueous media have been reviewed (199, 253, 412). Other studies, however, were concerned with the effects of different solvents on polarographic active compounds (311, 719).

Work on the correlation of half-wave potentials with various constants continues. The relationship between half-wave potential and organic structure (774) and Hammett and Taft substituent constants (948) were reviewed. Other studies in this area are mentioned under specific compounds.

Applications and techniques of oscillographic polarography were reviewed (90, 108, 356, 691). Other recently developed polarographic methods were also reviewed. These included the utility of square wave polarography (585), oscillographic polarography with alternating current (358, 360), newer developments in alternating current polarography (33, 46, 101, 103) and application of a titration technique to alternating current polarography (102).

The use of electrodes other than mercury were the subject of several reviews. The application of the carbon paste electrode (2) and the graphite electrode (43, 177) in polarography were reviewed. Oxidation at Pt in acetonitrile (35) and reduction of azobenzene, hydrobenzene, nitrosobenzene, and hydroxylamine at the rotating graphite disk and pyrolytic graphite (213) were described. The use of boron carbide was reviewed, and it appears to be most useful in the anodic range where the noble electrodes fail (553).

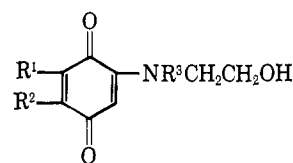
The applications and design of a special cell to follow photolytic reactions of hydrocarbons, chlorophyll, and other biochemical compounds were described (59).

REVERSIBLE SYSTEMS

The quinone-hydroquinone system continues to be used to test new techniques, solvents, electrodes, and calculations in polarography. Data are given for this system at the rotating Pt disk electrode (675, 925), Pt disk of different shapes (923), unshielded planar Pt electrodes in a theoretical development of chronopotentiometry and chronoamperometry (472), rotating Ge disk electrode (645), in 10-16*M* H₂SO₄, where five distinct anodic waves for *p*-hydroquinone were found and were due to four new sulfonated species (518), and in pyridine solvent at pyrolytic graphite electrode (841, 843, 845). In other instances, anthraquinone was used. Data for this system are reported for dimethylformamide solvent (465), related to electron spin spectrum (685), and studied at the carbon paste electrode (450).

Correlations of half-wave potentials with constants were reported. Ultra-violet spectra of quinones and ketones (61), vibrational spectra of proton donor complexes between phenol and quinones (214), and spectra for molecular complexes of *p*-benzoquinones containing Cl, F, and CN substituents with hexamethylbenzene (630) were correlated to half-wave potentials.

The acid hydrolysis of bis(ethyleneimino) benzoquinone (47), hydrolysis of 23 different quinones of the general structure, I, to the quinone and the amine (200), and the consummation of



I

14 microorganisms (906) were followed polarographically. The method was used for the identification of chloroquinones in chlorination reactions of quinone (175), quinone, 2-amino-*p*-quinone, and 2,5-diamino-*p*-quinone in N-fixation studies (470), and 3-hydroxy-*p*-quinone in a reaction mixture (670).

Reduction of 2-ethylanthraquinone (400) and nitro derivatives of anthraquinones (465) were studied in dimethylformamide. Pyridine was recommended on the basis of hydroquinone

studies as a very useful solvent for studying electrolytic oxidation (843, 845). Data for a variety of quinones and quinone-like derivatives in liquid $\text{LiNO}_3\text{-NH}_3$ (964) were reported.

Mechanistic studies have been reported. The compounds, 1,4- and 2,7-hydroxyanthraquinone sulfonic acid, were studied to show the existence of monomolecular semiquinone free radicals (105). Reduction studies and electron spin resonance study of the anion radical of benzocyclobutadienoquinone indicated a one-electron reversible wave in acetonitrile (238). 12,15-Dihydro-12,15-dioxo-13,14-benzotryptycene gave reversible two one-electron waves in dimethylformamide and one two-electron wave in ethanol-benzene mixture (398, 399). Polarographic data for diquinone was used to predict conditions for radical production for electron resonance studies (769).

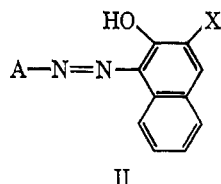
Camphorquinone reduction was studied at pH 4-14 (89). Polarographic data for 1,8-dihydroxyanthraquinone (648) and monomethylhydroquinone at rotating Pt disk electrode (676) were reported. Alternating current polarographic studies of shikonin (709) and quinone and quinone derivatives (827) were described. Data for hydroquinones and derivatives were related to photographic properties (824).

Procedures for the analysis of trimethylhydroquinone (812, 814), anthraquinone (69), monomethyl ether of hydroquinone at carbon paste electrode (139), mono- and di-nitroanthraquinones after chromatographic separation (582), and anthraquinone at carbon paste electrode (450) were reported. Methods for the determination of 1-aminoanthraquinone (440), quinone in air (439), homogentisic acid (2,5-dihydroxyphenyl acetic acid) by anodic polarography (27), Menaphthone (2-methyl-1,4-naphthoquinone) in presence of vitamins (112), and Frangula Emodin (1,3,8-trihydroxy-6-methylantraquinone) in pharmaceutical products (217) were described.

The polarographic behavior of several hydroxy and sulfonic acid substituted alizarines in water-acetone and water-methanol mixtures as a function of pH (413) was described. Chronopotentiometric measurements of alizarin red S at hanging mercury drop (453) and a study of alizarin and its complex with cobalt (924) were reported.

Data are given for the polarographic behavior of Solochrome Violet R. S. and thorium complexes in tartrate solution (847), Eriochrome Violet B and Eriochrome Violet B Red and their Cr(III) complexes (34), Eriochrome Black T (740-742), and complexes of Eriochrome Black T with Mg and Ca and applied to analysis (484), and of Suprachrome Garnet Y, sodium salt of 5-sulfo-2-hydroxybenzeneazo- β -naphthol, and its

use in Al analysis (427). Redox potentials of Variamine Blue and derivatives were measured (39). The reversibility in reduction of Methyl Orange was examined by alternating current polarography (48). Effects of Cu(II) on several azo type compounds (764) and surface active agents on *p*-aminoazobenzene (367) in reduction studies were reported. Data for 34 azo compounds in liquid $\text{LiNO}_3\text{-NH}_3$ were measured (965). Six azo dyes used in the textile industry were analyzed by oscillopolarography (636). Data were reported for 13 different 1-(arylozo)-2-naphthol dyes of the general formula, II (663). Half-



wave potentials for dyes of the structure $\text{XC}_6\text{H}_4\text{N:NC}_6\text{H}_3(\text{CO}_2\text{H})\text{OH}$ -3,4 and 1,2,4- $\text{HO}(\text{HO}_2\text{C})\text{-C}_{10}\text{H}_5\text{N:NC}_6\text{H}_5\text{X}$ were correlated to Hammett values (903). Studies on 32 triphenylmethane derivatives in liquid $\text{LiNO}_3\text{-NH}_3$ (966) and cyclic voltammetric studies of similar dyes at carbon paste and Pt electrodes (229) were reported.

Azodicarbonitrile, NCN=NCN , was found to be reversibly reduced in a one-electron process to NCN=NCN^- (523).

Reduction data for azobenzene (720, 735, 736) were reported. Reversibility for *p*-nitroazobenzene and irreversibility for other *p*-substituted azobenzenes was found by conventional and alternating current polarography (288).

Reduction behavior of diazonium salts of *N,N*-dimethylbenzidine (532) was reported. The reaction of benzoic acid with diphenyldiazomethane was followed polarographically (799). Amperometric titrations involving coupling reactions were performed by using the reduction wave of the diazonium salt of 2-naphthol or pyrazolone derivatives (531).

The behavior of tetracyanoquinodimethan (165) and 2-benzhydryl-7,7,8,8-tetracyanoquinodimethan (267) in acetonitrile was reported with the latter showing two well defined one-electron reversible waves. Polarographic data for dicyanomethylene-2,2,4,4-tetramethylcyclobutane and the bis derivative were determined in order to facilitate selection of conditions for radical formation in EPR studies (345). Molecular complexes and their stability of tetracyanoethylene, tetracyanobenzene, tetracyanoquinodimethane, and dichlorodicyanoquinone with hexamethylbenzene or pyrene were studied (628).

Conventional and oscillopolarographic study of borazines in dimethyl-

formamide demonstrated a reversible one-electron reduction to form the radical anion (738).

The stable radical, diphenylpicrylhydrazyl, was suggested as a good test compound for electrochemical studies (748).

Acyclic voltammetric study of *p*-aminophenol (270) and a chronopotentiometric study and analysis of *p*- and *o*-aminophenol at Pt (462) was reported. A rotating Pt disk was used in oxidation studies of *p*-aminophenol (675).

Data were given for the oxidation of *p*-, *m*-, and *o*-phenylenediamines at the rotating Pt electrode (295) and for *p*-phenylenediamine (675) and *o*-phenylenediamine (676) at a rotating Pt disk.

The graphite paste electrode was used in the conventional and alternating current polarographic oxidation study of *N,N*-dimethyl-*p*-phenylenediamine (339). Oxidation of diethyl-*p*-phenylenediamine was measured at the rotating Pt disk electrode (677).

Data for *p*-phenylenediamine and derivatives were related to photographic properties (824). The sensitivity of the analysis of *o*-phenylenediamine in a medium containing Ni(II) was found to be improved upon the addition of LiI (485).

Oxidation of tetrakis (dimethylamino) ethylene (TDE) was shown to proceed in two steps with the formation of TDE^+ and then TDE^{+2} (451). The graphite electrode was used in studies with methylene blue (632).

Procedures for the analysis of riboflavin (599) in vitamins and antibiotic supplements (342) were described. Adsorption study of riboflavin in H_2SO_4 solution by chronopotentiometry (272) and of the leuco form (82) were reported.

IRREVERSIBLE SYSTEMS

Alcohols, Glycols, and Phenols.

The oxidation of simple aliphatic alcohols (468) and ethyl alcohol (684) at Pt electrode has been studied. Data were reported for the oxidation of methyl alcohol at Pt in unbuffered media (875) and as a function of chloride ion which inhibits the oxidation (100).

Analysis of polyhydric alcohols by oxidation at Pt (653), poly(ethylene glycol) analysis in polymers by oscillopolarography (606) and polyvinyl alcohol and carboxymethyl cellulose analysis in textiles by oscillopolarography (237, 260) were described. Polyvinyl alcohol was determined by measuring its effect on the Cu(II) wave (78). The α -glycol structure was shown to be absent in a characterization study (14).

A rotating Pt electrode was used to study the oxidation of all possible

methoxybenzenes (955) and pyrogallol (672). Half-wave potentials in the former case were correlated to charge transfer bands. The rotating graphite electrode was used for oxidation studies of tetra substituted (aryl or/and cyano groups) phenol (166). Data for phenol oxidation at graphite (781), several hydroxybenzenes in pyridine at pyrolytic graphite (843) and for pyrocatechol oxidation at rotating Pt disk electrode (676) were reported. Cyclic voltammetry with carbon paste electrode was used for oxidation studies of *p*-methoxyphenol in 2.0M H₂SO₄ (268).

Half-wave potentials for the oxidation of 13 4-substituted 2-chlorophenols (739) and 18 *o*-, *m*-, and *p*-substituted phenols (894) were correlated to Hammett values. Extensive studies on the oxidation of 2,6-di-*tert*-butyl-*p*-cresol were reported, and data for this and several other phenols were correlated to Hammett values (885). Data for substituted diphenyl ethers were correlated to Hammett values (93).

Oscillopolarography was used for the analysis of naphthol and derivatives (607, 608). Analysis of phenol in vat residues in phenol synthesis was carried out after nitration of the sample (410).

Numerous studies on surface activity of alcohols and their effect on polarographic activity were reported. Alternating current polarography was used to study the effect of *n*-amyl alcohol and *o*-cresol (257, 258). The effects on double layer properties by nonionic surface active agents (908) and by *n*-amyl and benzyl alcohol in a theoretical study (548) were reported. The influence of 19 simple alcohols on reduction in oscillographic square wave polarography (586) was described. Drop time studies in the presence of a variety of aromatic and aliphatic alcohols (32) and Triton X-100 (44) were measured, and their effect on alternating current polarographic peak heights was observed. Adsorption of polyvinyl alcohol in a theoretical study (446), of polyoxyethylene lauryl ether (802), and of *n*-amyl alcohol and its effect on reduction (882) was reported.

The tensammetric wave of cyclohexanol was used to study the adsorption of dodecylbenzenesulfonic acid (364). The order of increasing adsorption in 1M H₂SO₄ at the DME was found to occur in the order naphthyl > phenyl > aliphatic hydroxy compounds (542). Surface active agents were the subject of other studies (367, 368, 370, 401, 404, 464, 504).

Methyl cellulose and polyvinyl alcohol (808, 891) and compounds containing sulfonic acid group, ether linkage, or quaternary nitrogen (266) were examined for their ability to suppress polarographic maxima.

Changes in the oxygen wave have been used to study the effect of dextrin (476)

and surfactants (613), for the analysis of surfactants (419, 420) and caprolactam, urea, and derivatives (919), and to determine the enzyme, 3-hydroxyanthranilic oxidase (223) and enzyme activity in hydrolysis studies of potato starch (701).

Aldehydes. Most of the studies concerning aliphatic aldehydes were devoted to analysis. Procedures were suggested for the determination of formaldehyde (666, 810, 828, 838) in plastic utensils (20), in waste water (436), and in amino resins (467). DL-Glyceraldehyde was determined in blood (21).

Methods of analysis for acetaldehyde (390, 747, 803, 838) in presence of vinyl cyanide (402, 403) and in ethanol (547) were reported. Aldehyde content in anesthesia ether (231) and waste water (436) was determined.

Other studies with formaldehyde included using its reduction wave in amperometric titration of active amino groups (143), reduction studies at a single crystal of zinc (568), study of its reaction with ammonia (849), and an oscillopolarographic study in buffered media (874).

Data for the protonation and its effects on reduction for semi- and thio-carbazones of aliphatic aldehydes (394) were reported. Polarographic studies have been carried out with phosphorylated aldehydes of the general structure RR'P(=O)CH₂CHO (669) and α -amino aldehydes (713).

Procedures for the analysis of crotonaldehyde by conventional (390, 618, 619, 747) and oscillographic (803) polarography and the analysis of (CH₃CH₂-OOC)₂CHCH:CHCHO in a reaction mixture (234) were reported.

Oxidation of formaldehyde was studied at Pt electrodes (468).

Methods of analysis for vanillin (811), streptomycin and dihydrostreptomycin (144), *p*-aldehydobenzoic acid, its methyl ester, and *p*-tolualdehyde in mixtures (334), and terpene aldehydes as intermediates in vitamin A synthesis (406) were reported. Furfural was analyzed (809, 810) in waste water (86), in soil (87), in hydrogenation products (248), and in pulp hydrolysis products (762, 763). Hydroxy and methyl derivatives of furfural were analyzed (36).

Reduction behavior of a variety of aromatic aldehydes (88, 362, 622), benzaldehyde (500), aldehyde derivatives of imidazole and thiazole (457), 5-nitrosalicylaldehyde in presence of several metal perchlorates (487), and furfural in buffered media (778) was reported. The effect of acetal formation on the reduction of benzaldehyde, *p*-chlorobenzaldehyde, and phenylacetaldehyde (77) in methanol was studied. Chronopotentiometric studies with benzaldehyde gave hydrobenzoin in the

first step and benzyl alcohol in the second step and served as a model for studies of a system in which the product of first step is not reducible to product of the second step (181, 182).

Degradation products of heated lactose in milk (562) and heated sucrose (652) were detected by polarography. Reduction behavior and analysis of 3,6-anhydrogalactose and agarobiose (222), saccharin in nickel plating solutions (690), and lactulose (884) were reported.

Ketones. Data are given for the reduction of croconic acid (4,5-dihydroxy-4-cyclopentene-1,2,3-trione) (204), Girard T derivatives of steroids (312), γ -piperidone derivatives (495), methylphenylphenacyl sulfonium bromide and diethylphenacylsulfonium bromide (712), tropone and 2,7-dimethylbenzotropone (657), and *N*-tropenyl amino acids (938).

Acetyl benzoyl (19), *p*-hydroxybenzophenone (127), fluorescein (246), and acetophenone (499) reduction was studied as a function of pH. The position and number (three or four) of methyl groups on acetophenone reduction (72) was reported. Oscillopolarographic studies on aromatic carbonyls (362), steroids (553), and cardenolides in strong acid and base (550) and alternating current polarography of 2,6-disubstituted cyclohexanones (708), benzoylpyridine (899), and cyclohexanone (833) were carried out. Data were collected for the reduction of 2-aryl and 2-acyl derivatives of 1,3-indandione (149, 150, 153), derivatives of Δ^2 -cyclohexanone (230), 18 carbonyl compounds which included methoxy derivatives of chalcone, dibenzalacetone, and cinnamalacetophenone (459), acetophenone (500) and other aromatic ketones (622), α,β -unsaturated carbonyl compounds in dimethylformamide (910), and amurine and 10 other cyclohexadienones (536). Data for the reduction of acetophenone and 4-fluoroacetophenone in dimethylformamide and dimethyl sulfoxide were determined to facilitate anion radical formation for EPR studies (760).

Several mechanistic studies were reported. Benzophenone reduction indicated a one-electron reversible process to an anion free radical which dimerizes or hydrolyzes (134). Three reduction waves for diazoacetophenone were found at pH 6 to 8; the first involving six-electrons to C₆H₅COCH₂NH₂ and NH₄⁺, the second involving two-electrons to C₆H₅COCH₃ and NH₄⁺, and the third involving two-electrons to C₆H₅CHOHCH₃ (137). Two mechanisms for the reduction of Δ^2 -cyclohexanone depending on whether protonation occurred (321) were postulated. Reduction studies on seven aryl diketones in aqueous ethyl alcohol, two one-electron waves at low pH and one two-electron

wave at high pH with the formation of the enediol, and in dimethylformamide, one two-electron wave to the enediol (640), and the effect of Li ion (641) were reported. Benzil in acid underwent a two-electron reduction to two isomeric stilbenediols (890).

Half-wave potentials for 10 aromatic α,β -unsaturated ketones (70) and 1,3-indandiones substituted in the 2-position by *o*- and *p*-substituents (772) were correlated to Hammett values. Correlations were made between half-wave potentials of 22 diones of the variety, 1,3-indandione and 4,5,6,7-tetrahydro-1,3-indandione and energy of the lowest vacant electron orbital (776).

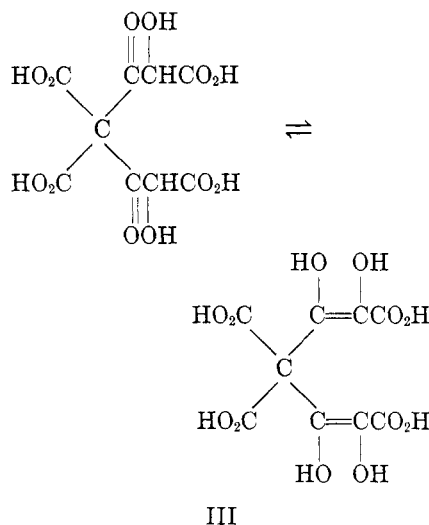
Reduction studies of thiosemicarbazones of ethyl acetoacetate and ethyl methyl ketone (904), a variety of hydrazines after reaction with acetone (38), Girard-T and -D hydrazones of aliphatic and aromatic ketones (529), and thiosemicarbazones of 4'-formyl acetanilide (26), were reported. Protonation effects on semi- and thio-carbazones were considered (393).

Irreversible reduction behavior of several corticosteroids was determined by conventional and alternating current polarography (301, 302). Oscillographic study of steroids (553) and 10 steroids with an olefin in conjugation with the carbonyl (303) was reported. Reduction studies on 9- α -thiocyanato-hydrocortisone 21-acetate (798), ninhydrin and derivatives (292), vitamin K (407), and the following of iodination and acetolysis reactions of progesterone and other steroids (85) were done polarographically.

Polarography was used to elucidate the structure of oxycolchicine (142), detect acid degradation products of oxytetracycline (131) and ketone group in ferrocene derivatives (232), study adsorption properties of aromatic carbonyls (290), follow changes in benzophenone upon exposure to ultraviolet radiation (431), detect triquinoyl (cyclohexanehexone) (205) and enediols (206) in reaction mixtures, follow oxalacetate concentration in biochemical reactions (442), and detect the presence of Bindone and its nitro derivative in synthesis procedures (930).

The reduction of α -oxime of *o*-aminobenzoylpyruvic acid and its ethyl ester (54) and kinetics of cyclization (55) were reported. The hydrolysis of β -morpholinopropiophenone to phenylvinyl ketone and morpholine (118), reaction between chalcone and phenylhydrazine (460), hydrolysis of Girard hydrazone of cyclopentanone (528), decomposition of $C_6H_5NH:CHC(OH):CHCH:CHNHC_6H_5$ (539), kinetics of reactions involving ketones (912) and changes in the equilibrium, III (754), were followed by polarography.

Procedures for analysis of diacetyl in fat (646), benzanthrone and bromo-



benzanthrone mixtures (387), flavonoids in wine (515), α,β -unsaturated ketones (230, 746), acetoacetic acid ester via its iron complex (829), *p*-, *m*-, and *o*-methyl- α -bromopropiophenone mixtures (797), 5-aminobenzophenone at the carbon paste electrode (450), and 1-piperidino-2-methyl-3-(*p*-tolyl)-3-propanone hydrochloride (152) were reported. Derivatives of 1,3-indandiones were analyzed in tablets or blood serum (151). Terpene ketones as intermediates in vitamin A synthesis (406), benzanthrone and mono- and di-bromo derivative mixture (68), and 4-ketopimelic acid isonicotinoylhydrazones, $RCONHN:R'$, where R is 4-pyridyl and R' is pimelic acid, and which is reduced by four-electrons to $RCH_2NHN:R'$ (8) were determined.

The drugs, Diazepam, 2-(methylamino)-5-chlorobenzophenone, after hydrolysis (580) and Methadone, 4,4-diphenyl-6-dimethylamino-3-heptanone, after nitration (743) were analyzed polarographically.

Tetracycline in urine (281) and its chloro- and oxy-derivatives were analyzed by conventional (117) and oscillographic (184, 274) polarography.

Polarography was used to analyze 20 different carbonyls as they appeared in a gas chromatographic separation (922).

Acids and Their Derivatives. A rotating Pt anode was used in a study of the Kolbe reaction involving several carboxylic acids (10). Oxidation of oxalic acid and oxide formation at the anode was measured by chronopotentiometry (556). A rotating Pt disk was used in the oxidation of (ethylene-dinitrilo) tetraacetic acid (673, 674).

The polarographic behavior of ascorbic acid and hydroascorbic acid (273, 304) was used to follow autoxidation of ascorbic acid (635), analyze for ascorbic acid (889) in potatoes (918, 920), illustrate the utility of square wave polarography (901), and analyze for ascorbic acid derivatives (905).

Phthalic acid was analyzed in pres-

ence of linear polyesters (193). Procedures for the determination of terephthalic acid (325) and its potassium salt in presence of phthalic, toluic, and benzoic acid and salts (52), isophthalic acid in polyester fibers (56), dimethylaminoterephthalate and dimethyl nitroterephthalate (853), pyro and trimellitic acid, tetrachlorophthalic acid, and 1,4-endo-methylene-1,4,5,6,7,7-hexachloro-5-cyclohexane-2,3-dicarboxylic acid (405) and trimellitic acid (854) were reported.

Conventional and oscillograph polarography were used for the analysis of citraconic, mesaconic, maleic, fumaric, and phthalic acid in polyesters (571-573). Mandelic acid was determined in urine (42, 198). Analysis of maleic and fumaric acid (521, 810) and their isomerization was studied by polarography (397). Chlorosuccinic acid was analyzed by conversion to fumaric acid (15).

α -Keto acids were determined by reaction with *o*-phenylenediamine and then measuring the quinoxaline product (106). Indirect determination of alkylbenzenesulfonates was carried out by measurement of the sulfonate-methylene blue complex by alternating current polarography (363). Procedures for the analysis of DL and MESO dibromosuccinic acids in mixtures (16), α -iminopropionic and pyruvic acid (522), carnitine by conversion to crotonic acid betaine (770), benzactyzine, 2-(diethylamino)ethyl ester of benzoic acid, after nitration (867), and 7-aminoheptanoic acid after reaction with acetone (934-936) were reported.

Methyl methacrylate in polymers (732) and intermediates in its manufacture (343) were analyzed polarographically. Amino derivatives of ethyl methacrylate in polymers (74) were determined. Analysis of methacrylic acid, methyl ester, and sodium salt in a study of the polymerization (452) was reported.

The theoretical approach was taken in the discussion of catalytic hydrogen waves for organic compounds (490). Proton exchange between molecules of alcohol and water was studied by polarography (848). Proton recombination rates in α -keto acids in different buffers were examined (49). Maleic and fumaric acid reduction was studied as a function of pH (779). Phenylphosphonic dichloride-chlorobenzene solvent mixture was reported to be a useful medium for analysis of active hydrogen compounds and water (259). A conventional and alternating current polarographic study of terephthalaldehydic acid in buffered media showed one pre-wave due to adsorption of undissociated molecules, one reduction wave due to the carboxyl group and pH dependent, and two reductions due to the carbonyl and pH independent (570).

Polarographic data for *N*-bromosuccinimide (304), several betaine esters of the type $(\text{CH}_3)_3\text{N}^+\text{CH}_2\text{CO}_2\text{R}^- \text{Cl}^-$ where R is alkyl (475), *o*-, *m*-, and *p*-substituted cinnamic acids in 75% dioxane (915), and 3-aminophthalic acid hydrazide, 3-nitrophthalic acid hydrazide, and maleic hydrazide (725) were reported. Idonic and gluconic acid reaction with periodate ion (29) and the effect of acetylcarnitine on succinate oxidation (771) were followed polarographically.

A mechanistic study and analysis of CO_2 at Au or Pt electrodes (686) and mercury electrode (716) in dimethyl sulfoxide was reported.

Effect of surface active substances on polarographic behavior of diethylenetriaminepentaacetic acid and (ethylenedinitrilo)tetraacetic acid (324) was reported. Citric acid reduction was used to study the adsorption effect of cyclohexanone and Triton X-100 (834). Adsorption of *n*-valeric acid (882) and the electrocapillary behavior of unsaturated acids (421) were described. The surface active properties of lauryl sulfate (419, 420) were used for its analysis.

The polarographic behavior of hydroxamic acids was reported (929). The half-wave potentials of 20 aliphatic amino hydroxamic acids were correlated to their dissociation constants (538).

Studies of benzonitrile in 0.1M H_2SO_4 were shown to involve a four-electron reduction to the amine (510). Polarographic behavior of pyromellitonitrile was determined in acetonitrile (958). Half-wave potentials of several aromatic and aliphatic nitrile anion radicals were correlated to calculated π -electron energies (681).

Polarography was used for the analysis and study of hydrocyanic acid polymers (591). These were shown to be a tetramer (592, 593).

Procedures were reported for the analysis of acrylonitrile (461, 687) in polymeric food-packaging materials (140) and as residues in copolymers (141). Reduction of acrylonitrile was found to be irreversible with the addition of one electron and one proton to form $\cdot\text{CH}_2\text{CH}_2\text{CN}$ which reacts with itself to form $\text{NC}(\text{CH}_2)_4\text{CN}$ (194).

Data for 18 meta derivatives of phenyl isothiocyanate (366) and the analysis of α - and β -naphthylisothiocyanates (759) were reported.

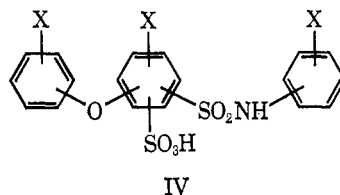
Organic Halogen Compounds. Procedures for the analysis of carbon tetrachloride and chloroform (58), iodoacetate in biochemical reactions at a vibrating Pt electrode (135), dichlorodiphenyltrichloroethane (711), β -bromo- β -aryl-acrylic acid derivatives and esters (941), dl and meso dibromosuccinic acid mixtures (16) were reported. Chlorosuccinic acid was analyzed by

conversion to polarographically active fumaric acid (15). Mono- and dichloroanthraquinones (566), 2,3,4,6-tetrachlorotoluene (569), and 2,6-dichloro-4-nitroaniline (895) were analyzed.

Procedures for the analysis of DDT and other halogenated insecticides (188) in plants and plant products (426) were described. Oscillopolarography was used for the analysis of DDT and analogs (226). The insecticides, *N*-trichloromethylthiotetrahydropthalimide and *N*-trichloromethylthiophthalimide, were analyzed (563).

Dimethylformamide was used as solvent in the analysis of *p*-chloroacetanilide in the presence of phenacetin (346). Medicinal compounds containing the dichloroacetamide group were analyzed by polarography (371). Analysis of chlorpromazine after bromination was reported (654).

Oscillographic analysis of Larvilan, IV, where X is halogen, was reported (605).



The bromopyrogallol-uranyl complex was used for analysis of uranium (832).

Polarographic data for the bromo derivatives of imidazoles and thiazoles (456, 457), *p*-chloro-, *p*-iodo-, and 2,4-dichlorophenylisothiocyanate (694), iodopropionitrile (823), and hexabromocyclohexane and *p*-diiodobenzene (937) were reported. Reduction data for $\text{ICH}_2\text{CH}_2\text{Cl}$ and $\text{CH}_3\text{CH}_2\text{I}$ (190) and dichloro- and trichloroacetic acid (860) were measured to better understand the electrolysis of these compounds. The polarographic behavior of 34 compounds of the type, $\text{RN}(\text{CH}_2\text{CH}_2\text{Cl})_2$, where R is aliphatic, aromatic, and heterocyclic groups, was reported under hydrolysis conditions (642).

The reduction of α -bromopropionic diacetyl morphine was used to study the effects of surface active polyvinyl alcohol (444). Benzyl chloride and bromide reduction data were used in a theoretical adsorption study (807). The effect of electrolytes on the polarographic behavior of diisopropylamine dichloroacetate was reported (876).

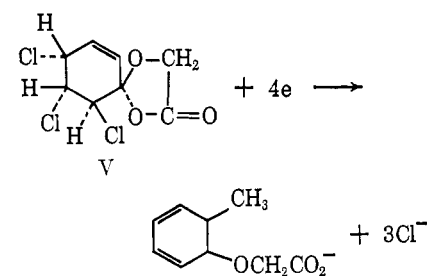
The reduction behavior of 20 different halogen derivatives of cyclohexane which included mono- and 1,2-di- and 1,4-di-chloro, bromo, and iodo, cis and trans derivatives, was reported. The half-wave potential in 50 and 90% ethyl alcohol was found to be dependent on a steric effect due to the large size of the halogen atom, particularly when adjacent, the dipole moment which tended to strengthen adsorption, and chemi-

sorption, importance of which decreased in the order $\text{I} > \text{Br} > \text{Cl}$ (133).

A polarographic study of methyl esters of 2-bromo-*n*-alkyl acids from C_2 to C_{22} showed that half-wave potential decreases to a constant value at C_{14} , while for *n*-alkyl esters of the acids, no such trend was observed (642).

Molecular complexes of tetrachlorophthalic anhydride and chloranil with hexamethylbenzene or pyrene were studied (628). Half-wave potentials for *m*- and *p*-substituted benzyl chlorides in dimethylformamide were correlated to Hammett values (782).

Methylpentachloroethyl ketone was reported to give five- and four-electron waves in acidic and basic solution, respectively (5). Chlorination of *o*-cresoxyacetic acid produces the chlorinated cyclohexane derivative, V, which was reduced according to the following equation (480). Studies on β -iodo-



and bromo-propionitrile revealed two one-electron waves for the iodo compound and one two-electron wave for the bromo compound. The effect of different alkali metal salts on half-wave potential was also noted (191).

Discussion of the reduction of alkyl and benzyl halides in terms of mechanism has continued. Reduction studies on benzyl halides, benzyl mercuric iodide, and α -chloro- and α -bromoethyl acetate in acetonitrile and dimethylformamide have been reported. The mechanism for benzyl derivatives, which depends on electrolyte, involves the formation of benzyl mercuric halide which is finally reduced to toluene (909). Polarographic reduction behavior of alkyl, cycloalkyl, and bridgehead bromides (455, 721) has been reported. In one case the mechanism suggested is that displacement of the bromine takes place to give an alkyl free radical and a bromide ion with the latter located immediately next to the mercury surface (721). On the other hand, it is suggested that the bromide is away from the electrode and a backside attack at the mercury drop takes place (455).

Nitro Compounds. Polarographic studies have been reported for 2,2-dinitropropane (907), $\text{RCH}(\text{NO}_2)\text{CO}_2\text{CH}_2\text{CH}_3$ where R is H, $-\text{CH}_3$, $-\text{CH}_2\text{CH}_3$, and $-\text{CH}_2\text{CH}_2\text{CH}_3$ (530), nitroethane in the presence of a variety of solvents, organic acids, and amides (463), and the catalytic wave of the C-nitrohydroxyamine complex (116).

The reaction between sodium hydroxide and nitroglycerin was followed polarographically (31). Examination of tautomerism in α -nitro ethyl acetate (51) and nitrocyclohexane (93) is reported. The method was used to follow the hydrolysis of several amino derivatives of alkyl nitrates (574) and α -hydroxyisobutyric acid nitrate (693).

The reduction mechanism for hydrocarbons with a tertiary nitro group was suggested to proceed to an unstable anion radical which cleaves to nitrite ion and another free radical (284).

Procedures for the analysis of nitrocyclohexane and *N*-cyclohydroxylamine (30), nitroglycerin (469), compounds containing a primary or secondary nitro group (852), and α -nitroethyl acetate in acetonitrile (815) were reported.

Tetranitromethane in the presence of nitromethane was analyzed by oscillographic polarography (279).

A wide variety of nitro alkanes were separated by column chromatography and polarography used to analyze the effluent (372, 374, 375, 383).

The reduction of aromatic nitro compounds continues to be used to study the applicability of new and modified techniques, medium effects, and different electrodes. A new chronopotentiometric set-up was described and tested with *p*-nitro- and *p*-nitrosophenol (271). The use of oscillographic polarography with rectangular alternating current was demonstrated by *p*-nitrophenol reduction (359).

Various studies on medium effects were reported. The effect of the type of buffers on nitrobenzene reduction was considered (4). Alternating current polarography of nitrobenzene derivatives was used to study the effect of pH buffers, buffer capacity, and solvent (254, 256).

Polarographic behavior of halonitrobenzenes and methyl nitrobenzenes at 0° C. were reported for $\text{NH}_4\text{SCN}-\text{NH}_3$ solvent (683). Other nitroaromatic compounds were studied in liquid $\text{LiNO}_3-\text{NH}_3$ (962). Reduction of 5-nitrosalicylaldehyde was studied in the presence of a variety of metal perchlorates (487). Solvent effects were evaluated by studying *p*-nitrophenol (433).

A variety of electrodes have been studied. The reduction of 4-nitro-2,6-xenol was used to illustrate the applicability of Au-Hg solid electrode (57). Twenty-one different metallic rotating electrodes were evaluated by using the reduction of sodium *m*-nitrobenzenesulfonate (389). The utility of carbon paste (590) and rotating amalgam electrodes (667) was demonstrated by studying a series of common nitro compounds. Nitrobenzene reduction was studied at Ni (735, 736, 744) and Pd (737) electrode.

Data for the reduction of common nitrobenzene derivatives (264, 638), 1-nitro-2-naphthol and 2-nitro-1-naphthol in dimethylformamide-water (332), nitro derivatives of imidazoles and thiazoles (456, 457), nitro derivatives of anthraquinone, dianthrimide, benzene, and naphthalene in dimethylformamide (465), nitrodiphenylamines and nitroresorcinols in ethyl alcohol (637), and nitroterephthalic and 3- and 4-nitrophthalic acids (872) were reported. Nitro derivatives of heterocyclic compounds (887, 888) and ω -nitroacetophenone and its oxime (161) were studied as a function of pH. Pentachloronitrobenzene and 4,6-dinitro-*o*-cresol were studied by alternating current and conventional polarography (710).

Hydrogenation of β -nitrostyrene on Pd black (211) and nitration of furan and derivatives (883) was followed polarographically. Measurements on the adsorption properties of aromatic nitro compounds (290) and the effect of surface active agents on nitro reduction (367, 370) were reported. Data for aromatic nitro compounds were correlated to their corrosion inhibitor activity (291). Nitrated products were detected in reaction mixtures (864).

Polarographic behavior of the complex between *m*-dinitrobenzene with benzidine (441) and a series of mono-, di-, and trinitrobenzene derivatives with hexamethylbenzene or tetramethyl-*p*-phenylenediamine (629) was reported.

Wave splitting which is suggested to be due to adsorption was seen for a variety of aromatic and heterocyclic nitro acids (633).

Electron spin resonance studies of nitro compound radicals have continued. EPR measurements on the anion radicals and half-wave potentials for *p*-nitrodiphenyl derivatives were reported (160). Half-wave potentials for nitrobenzene derivatives were correlated to nitrogen coupling constants for the corresponding anion radicals (396). Reduction data for nitrobenzene were determined in order to facilitate the production of the anion radical (369). Nitrobenzene (379) and α - and β -nitronaphthalene anion radicals (378, 380, 382) were produced in dimethylformamide polarographically and studied spectrophotometrically. Extensive studies were carried out on the effect of *o*-, *m*-, and *p*-alkyl substituents and steric effect on half-wave potential and EPR spectrum for nitrobenzene, nitroaniline and *N,N*-dimethylaniline derivatives (240).

Half-wave potentials for aromatic nitro compounds in liquid $\text{LiNO}_3-\text{NH}_3$ were correlated to Hammett values (963).

It was reported that *N*-nitroanabasine reduces to *N*-hydroxylaminoanabasine

and then to *N*-aminoanabasine (836, 837). A two-stage reduction of four electrons each with 3-nitrophenylhydroxylamine, the product of the first, and *m*-dihydroxylamine benzene, the product of the second was reported for *m*-dinitrobenzene (322). *o*-Dinitrobenzene was found to be reduced in strongly acidic solution to *o*-quinone-diimine which rapidly converts to 2,3-diaminophenazine and 2-hydroxy-3-aminophenazine. These are further reduced to the corresponding 9,10-dihydrophenazine and 5,6,7,8-tetrahydrophenazine (99).

Procedures for the analysis of dinitrophenols (317), *p*-nitrobenzoylacetate (202), nitro aromatic compounds (203), nitrobenzene in aniline (940), 2,6-dichloro-4-nitroaniline (895), picric acid and mono- and dinitrophenols (855), and 2-nitro- and 3-nitro-4-acetamidophenetole (581) were reported. Nitration followed by polarographic analysis of the resulting nitro compound has been used for the determination of thiophene (753), phenol in vat residues (410), DDT (711), carbazole (69), and strychnine (173). Analysis of tricalcium salt of 5-ethyl-*N*-[*N*-(*p*-nitrobenzoyl)- γ -glutamoyl]- γ -glutamoyl-glutamate in presence of the *p*-aminobenzoyl analog was reported (192).

Phenicol was analyzed in blood (658), in suppositories (696, 697), decongestants (698) and in pharmaceutical products (699, 794).

Analysis of *m*-dinitrophenolic type pesticides in biological materials (132, 241) and insecticides, *o,o*-dimethyl *o*-(3-methyl-4-nitrophenyl) thiophosphate and other nitro and S-alkyl derivatives (430) was reported. Dinitro-*o*-cresol analysis on leaves, twigs, and soils was performed (869).

Separation of mixtures of mono- and dinitroanthraquinones (209, 582), nitrotoluenes (376, 377), nitroanilines and nitrophenols (384), dinitrophenyl amino acids (868) by chromatography were carried out and analyzed by polarography. Partitioning of aromatic and aliphatic nitro compounds between solvents was measured by polarography (374, 375). Analysis of 5-nitrofurfural oxime isomers in column effluent was reported (385).

Nitroso and Azoxy Compounds. Polarographic data for nitrosobenzene (735, 736) in dimethylformamide (381), *N*-nitrosoephedrine and *N*-nitroso-pseudoephedrine (506, 507), and dimethyl-, diethyl-, dipropyl-, and diisopropyl nitrosamine (916) were reported.

The advantages of cyclic stationary electrode polarography were illustrated by studying the reduction of nitrosophenol (567).

The graphite electrode was used in studies of azoxybenzene, azobenzene, and hydrazobenzene (126, 130). The same compounds were examined at solid

stationary electrodes (129) and at the rotating graphite disk and pyrolytic graphite electrode (213).

Chronopotentiometric studies with *p*-nitrosophenol suggested a mechanism that involved reduction to *p*-hydroxylaminophenol which then loses water to *p*-benzoquinoneimine and finally reduced to *p*-aminophenol (183). Alternating current and conventional polarography of azobenzene and several different sulfonated azobenzenes at very low and high pH indicated near reversibility (207).

Reduction behavior of aliphatic nitroso compounds were studied as a function of pH (23). Half-wave potentials of *ortho* substituted nitrosobenzene derivatives were correlated to *ortho* steric hindrance (294).

The effects of gelatin on the reduction of hydrazobenzene (286) and of surface active agents on the oxidation of hydrazobenzene (289) were reported. The azoxybenzene wave (287) and azobenzene wave (293) were used to study the adsorption properties of carboxymethyl cellulose, triphenyl phosphine oxide, and gelatin.

The wave for neocupferron and benzo-hydroxamic acid were used for the amperometric titration of Ti(IV) and Hf(IV) (228).

Nitrosation followed by polarographic analysis was used for monomethyl and dimethyl ether of hydroquinone (926, 927), Dienestrol (4,4'-diethylideneethylenediphenol) in pharmaceutical products (795), *m*-diethylaminophenol (409), and flavanoids (153).

Amines, Amino Acids, and Derivatives. Oxidation of *N,N*-dimethylaniline and *N*-methylaniline at carbon paste electrode (2) and *N,N*-dimethylaniline and methoxy derivatives at the rotating Pt electrode in acetonitrile (956) were reported. Half-wave potentials in the latter case were correlated to the first charge transfer band for complexes with *p*-chloranil (956). Anodic studies of tetraalkylammonium halides in liquid sulfur dioxide at stationary Pt disk were described (705). Acetonitrile and Pt electrode was used for the oxidation study of 19 different aliphatic amines (508). Applications of the graphite electrode were demonstrated by studying aniline and derivatives (555). Phenylhydrazine was studied at rotating Pt (672).

Polarographic data for the two polymorphic forms of α -anisaldoxime (856), aromatic *N*-substituted oximes and aromatic *O*-alkyl oximes (949), nickel (II)-dimethylglyoxime in strongly basic media (95), and benzamidoxime, *N*-phenyl- and *N,N*-diethylbenzamidoxime and *N*-phenylbenzhydrazidoxime (549) were reported. Procedures for the analysis of cyclohexane oxime (850), hydroxylamine (851), and heterocyclic oximes (865, 866) were suggested. Reduc-

tion studies of α -furildioxime in strongly acidic and basic solution were carried out (233). Polarography was used in studies on the equilibria between hydroxyamidoxime and nitrosolic acid (24).

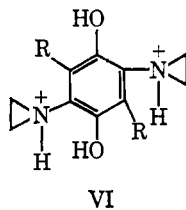
The catalytic hydrogen wave for *N*-methylanabasine, *N*-methyl- α -aminoanabasine, *N*-acetylanabasine, and *N*-methyl- α' -aminoanabasine was used for analysis (527). Catalytic wave for fibrinogen was used to follow its reaction with NaIO₄ (880). The prewave that occurs when Ni(II) is added to a solution of *o*-phenylenediamine was used for the analysis of the diamine in presence of isomeric phenylenediamines (517). The Ni(II)-ethylene-diamine mixture gave a similar wave and was used for the analysis of the diamine (519).

The adsorption of *N*- and *N,N*-substituted alkyl anilines (959, 960) and alkyl amines (961) in aqueous H₂SO₄ was studied. The effect of the adsorption properties of tribenzylamine (444), tribenzylamine and tetrabutylammonium ion (446), alkyl ammonium ions in alkaline solution (123), tetraalkylammonium ions (857), and aniline, toluidine, and xyldine (671) on reduction of inorganic and organic compounds was reported. Photopolarography was used to study the surface active properties of tetraethyl ammonium iodide (62).

Polarographic data for α -aminonitriles (951), dipeptides (351, 352), and glycoalkaloids (643) were reported. Oscillopolarography was used to study DNA (600-602) and to follow changes in DNA upon exposure to ultraviolet radiation (348, 350). Changes in the enzyme, α -amylase, were followed as it participates in a reaction (551). Interactions between proteins and dyes were studied (353).

Decomposition of azines was followed polarographically (733). Eight aromatic azines of the type, RCH:NN:CHR, were found to give two two-electron reduction waves in dimethylformamide. In highly acidic medium one four-electron reduction wave, which is a result of protonation of both *N*-atoms, was observed (79).

Two two-electron waves were found for ethyleneiminoquinones, VI, which was the result of opening of each *N* ring (296). Benzohydrazides were studied



by alternating current and conventional polarography (395). The polarographic behavior of ethylenimino and haloid

ethylamino derivatives of D-mannitol were reported (327).

Alternating current polarography was used in the study of Bromothymol Blue (255) and Methylene Blue, Neutral Red, and Indigo Carmine (873). The reduction behavior of Methyl Green and Crystal Violet in aqueous solution and in dimethylformamide was reported (373). Aggregate formation for several dyes was measured by polarography (283). Methylene Blue and Brilliant Green was analyzed after reaction with tungstosilicic acid (516). A reduction wave was detected for fuchsin (rosaniline) and, therefore, care should be exercised when using it as a maximum suppressor (236).

Aniline and *p*-toluidine were analyzed by oxidation at a rotating Pt electrode (50). Analysis of Thalidomide, *N*-phthalylglutamic acid imide, (277, 278, 280) and melamine in polymers (606) was completed by oscillographic polarography. *p,p'*-Oxybis benzenesulfonyl hydrazide in polyethylene blends was converted to the double hydrazone by reaction with acetone and then analyzed (107). Methods for the direct and indirect polarographic estimation of antibiotics were described (432). Albumin was separated from the original sample and analyzed by oscillopolarography (361). Procedures for the analysis of *m*-diethylaminophenol after nitration or nitrosation (409), dodecylammonium chloride in flotation agents (649), intermediates in the oxidation of 1-(*p*-tolylamino)-8-naphthalenesulfonic acid (822), and tetramethylammonium nitrate and ethylenebis(trimethylammonium) dinitrate (40) were reported.

Dimethylaniline oxide was shown to have two reduction waves with the first wave due to reduction of the cation and the second due to the hydrated neutral molecule (154). Similar studies are reported for aliphatic amine oxides (155), alkyl aryl amine oxides (156), and substituted aryl alkyl amine oxides (157). Adsorption and reduction studies, which can be applied to analysis, for dimethyldodecyl amine oxide were carried out (125).

Unsaturated Hydrocarbons. In the aliphatic series cyclooctatetraene underwent reduction in two one-electron steps to anion radicals in dimethylformamide and dimethyl sulfoxide (11).

Investigations in the aromatic series are more numerous and have dealt with reduction and oxidation.

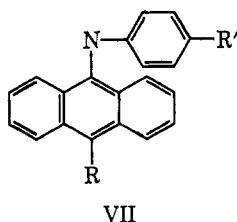
Reduction data for aromatic hydrocarbons in dimethylformamide and methyl cellosolve were correlated to solvation energy and electron affinities (124). Other correlation studies were unsaturated hydrocarbon data to the energy of the lowest unoccupied orbital (525), data for 35 olefinic and aromatic

hydrocarbons in acetonitrile to Huckel coefficients and photoionization potentials (564, 565), aromatic hydrocarbon and diphenyl polyene data to energies of molecular orbitals (610), data for all trans β -carotene, vitamin D₂ and D₃, all trans retinol, and 13-cis retinol to number of conjugated double bonds (445), fluoranthrene and benzenoid-like hydrocarbon data to energies of lowest free molecular orbitals (932), and methylnaphthalene and methylphenanthrene data to the calculated energy of lowest vacant orbital (94).

Reduction data for 4-acetylbiphenyl derivatives (67), 4-acetylbiphenyl and 2-acetylfluorene derivatives in 92% methyl alcohol (71), 2-acetyl- and 5-fluoroacetyltetrahydronaphthalenes (73), and azulene, 2,7-dimethylbenzotropone, and tropone (657) were reported.

Oxidation studies at rotating Pt and in acetonitrile for 3,4-dimethoxypropenylbenzene were carried out (578). Oxidation of the tetraethylammonium salt of cyclononatetranecide anion in acetonitrile was suggested to be a one-electron irreversible one (454).

Polarographic behavior of cinnamic acid (729) and 9-arylaminanthracene, VII, in acetonitrile (120) was reported.



Two one-electron steps if R is H, and one two-electron step if R is C₆H₅ were found in the latter case. Oxidation of tetrakis (dimethylamino)ethylene was shown to proceed in two one-electron steps (451). The anion radical of 9,10-diphenylanthracene in dimethylformamide was studied at Pt and hanging mercury drop (707).

Polymerization of derivatives of 4-vinylbenzene and styrene (66), change in structure of stilbene and naphthalene upon exposure to ultraviolet radiation (431), and addition reactions with unsaturated systems (243) were followed polarographically. Reduction kinetics for several aromatic hydrocarbons were reported (110). The adsorption of benzene on mercury in aqueous and alcoholic potassium chloride was measured (170).

The polarographic behavior of aromatic hydrocarbons, such as benzene, naphthalene, anthracene, and other polynuclear aromatic compounds was correlated to semiconductor behavior (631).

The method has been used for the analysis of styrene in alkyl resins (128), α -methylstyrene in manufactured prod-

ucts (623), and partially oxidized styrene sulfonic acid and its salts in polymerization mixtures (877). Styrene and α -methylstyrene were reacted with mercuric acetate and the products analyzed by alternating current polarography (75). Styrene could also be analyzed by conversion to a diazonium compound and then polarographed (688).

Analysis of vitamin A in synthetic procedures (491, 502, 503), pseudoionone (514), and fulvenes in synthetic procedures (546) was carried out. The monomers, 2-methyl-5-ethynylpyridine and 2-ethynylpyridine, was determined (65). Diphenic acid was analyzed after converting it to 9-oxo-fluorene-4-carboxylic acid (1). Acenaphthene, fluorene, and diphenylene were determined in anthracene oil (655).

Peroxides and Hydroperoxides. The polarographic determination of peroxides has been reviewed (138). Peroxide content in anesthesia ether (231), antioxidants (263), oxidized fat (488), and petroleum (944, 945) was determined.

Hydroperoxides were analyzed in fat (647) and benzene-methanol solvent mixture was used for the analysis of other hydroperoxides (172). Polarographic behavior of organic hydroperoxides and peroxides was described and applied to analysis (928). Diethyl ether peroxides (319) and alkyl hydroperoxides and dialkyl peroxides (689) were analyzed.

The method has been used for the analysis of cyclohexyl peroxides (18), dicyclohexyl peroxydicarbonate in polystyrene (167), and for peroxy carbonates (660, 879).

In other studies the method has been used to determine benzoyl and acetyl benzoyl peroxide and cumene and *tert*-butyl hydroperoxide (424) and for *tert*-butyl perbenzoate, *tert*-butyl peracetate, lauryl peroxide, and *tert*-butyl hydroperoxide (624). Catalase was determined by following its reaction with hydrogen peroxide (323). Oscillopolarography was used to follow the reaction between cyclohexanol or cyclohexanone with hydrogen peroxide (826). Peroxides were detected by polarography after exposing the solution to γ -radiation (931).

Half-wave potentials for alkyl substituted peroxides were correlated to length of alkyl groups (717) and data for *o*-, *m*-, and *p*-substituted dibenzoyl peroxides were correlated to Hammett values (718).

A two-step reduction with each involving two electrons for acetone diperoxide was found and applied to analysis (423).

Sulfur Compounds. Polarographic data are reported for a variety of sulfonium cations having the general structure $RR'R''S^+Z^-$ and $RR'SYSR''$

$R''Z^-$ (477) and sulfonium cations with a carbonyl in the α -position (712, 952). Oxidation of sodium thioglycolate (625) and sodium 2,3-dimercaptopropane-1-sulphonate (751, 752) at the rotating Pt electrode, 1-phenyltetrazole-5-thiol (766), ethyl xanthate and diethyl dioxanthate (730), butyl xanthate (749, 750), benzodithione and -trithione and ethyl di- and tri-thione-5-carboxylate (768), dithiodimalic acid (820), 2-thio-barbituric acid (821), and potassium ethylxanthate (862) was reported. Other studies have dealt with 2,4-dithiobiuret and 1-phenyl-2,4-dithiobiuret (861), 2-amino-ethane thiosulfuric acid (783), and a series of alkyl and aromatic sulfonic acids (745). The half-wave potentials in the latter study were about the same for the alkyl compounds and varied with the substituent for the aromatic ones.

The anodic wave of thiourea has been used for the amperometric titration of Pd (41). Other applications of this type reported were the 2,4-dithiobiuret anodic wave for Hg(II) (793), thionalide for Hg(II), Sb(IV), and Bi (863), and the anodic wave for either diphenyl- or dimethyl dimercaptopyrrole for Tl(III) (449). Biuret was analyzed by the fact that the addition of Ni ion causes a decrease in the diffusion current (145). Anodic studies on the Ni(II)-cysteine complex were also reported (45).

Dodecylbenzenesulfonate and dodecylsulfonate were evaluated for their ability to act as maximum suppressors (505). Adsorption of β -naphthalene sulfonate salts at mercury was studied (857). Theoretical studies of the effect of surface active agents such as dodecylsulfonate were reported (446, 447). The adsorption effect of cyclohexanone and Triton X-100 on the reduction of cystine was studied (834). Alternating current polarography was used to study the adsorption of dibenzyl sulfide and camphor on the reduction of other organic and inorganic substances (336, 337).

Half-wave potentials for 13 methylthio aromatic compounds were correlated to the first charge transfer band for complexes with *p*-chloranil (957). Molecular orbital energies and half-wave potentials for 20 different thio-carbonyls were correlated (540).

Irreversible reduction was found for diphenyl disulfide with the electron transfer occurring in two steps of one electron each (365). Thiosulfinate underwent a four-electron change, while thiosulfonate underwent a two-electron change (471). Two reduction waves and one small anodic wave were observed for mercury diethyldithiocarbamate (221). Dithiodiglycolic dianilide gave an irreversible four-electron wave which split into two two-electron waves when benzene was decreased in

the solvent mixture (186). Reduction studies of 29 aromatic and aliphatic thiosulfonic acids (RSO_2SR) were found to have two waves in almost all cases (91).

Polarographic studies were carried out on insulin and other proteins (122) and as a function of pH and temperature (354). The reaction between -SH sites on protein parts of virus and aliphatic mercury compounds was followed polarographically (245). Mold metabolites were detected in peanut butter (227).

Reaction between aromatic disulfide and triphenyl phosphine which produces a thiol (307, 308), hydrolysis of S-alkyl thiourea (187), the decomposition of 2,2'-dithiodiorescinol and 2-mercaptorescinol (790), and acetylthiocholine hydrolysis in presence of the enzyme cholinesterase (195, 197) was followed by polarography. The thiocholine which was produced in the latter reaction was also an indirect estimation of enzyme activity and was applied to enzyme analysis in blood (196).

Applications of the Brdicka test have been applied to proteins (320, 355, 498), amino acids (886), cystine and cysteine (727, 734), analyses in a gynecological laboratory (722), gluten and thiosulfol-gluten (533), and albumin in spinal liquids (650). The catalytic wave for an ammonical Zn(II) solution containing insulin was used for the analysis of insulin (896).

Methods for the analysis of mercaptothiamine (416), sulfhydryl group (703), sodium or antimony dimercaptosuccinate in urine (682), ethionamide and ethionamide S-oxide (81), proteins (726, 761), sulfur in chloroprene latex (859), tetraethylthiuram disulfide (806), enzyme activity (313, 953, 954), protein analysis in cow's milk (559, 560), sodium dibutylphthalenesulfonate (614, 615), sodium dimethyl dithiocarbamate (616, 617), *o,o*-diisopropyl dithiobisthiolate in styrene (620), albumins in blood serum (621, 626), xanthates and dithiocarbamates in flotation agents (649), antibiotics (432), 4,4'-dimethoxy-2,2'-dinitrodiphenyl disulfide (201), and carbon disulfide in benzene (656), were reported. Analytical procedures for 17 aliphatic and aromatic sulfides, 8 thiophene derivatives, 4 aliphatic and aromatic mercaptans, diphenyl disulfide, dibenzyl disulfide, and sulfur in oil by anodic polarography (668), 7 different aromatic and aliphatic disulfides in crude oil (576), and 11 different aliphatic and aromatic mercaptans (577) were described.

Cystine disulfoxide (208) and cysteine, glutathione, and methionine (535, 911, 935) were analyzed. Protein changes in meat upon exposure to gamma rays were followed by polarography (575).

The determination of Thimerosal (sodium salt of [*o*-carboxyphenyl]thio]-

ethyl mercury) in vaccines (83), Sevin, 1-naphthyl-*N*-methyl-carbamate, after nitration (179), pyrogenic material in water used in injections (185), Meproba-mate, 2-methyl-2-propyltrimethylene ester of carbamic acid, in drugs and bio-chemical material (309), and in urine (310), pangamic acid (876), and the in-secticides, *N*-trichloromethylthiotetra-hydrophthalimide and *N*-trichloro-methylthiophthalimide (563), was re-ported.

Oscillopolarography was used for the analysis of mercaptans, sulfur, and hy-drogen sulfide (391).

Thioxolone and derivatives were de-composed to 2,2'-dithiorescinol and then analyzed (791, 792).

Organometallic Compounds and Phosphonium Salts. Polarographic studies are reported for compounds of the type $(\text{R}^1\text{R}^2\text{R}^3\text{R}^4\text{P})\text{X}$ and $(\text{R}^1\text{R}^2\text{R}^3\text{R}^4\text{As})\text{X}$ (298), benzylchromium (408), 1,2-bis (diphenyl) borate ion (486), iodomethyltrialkyl (aryl) silanes (501), 26 organic mercurials (579), 2-amino-ethaneselenosulfuric acid (783), a variety of ferrocene ketones and their oximes (818), and mercuric dipropion-itrile and propionitrile mercuric chloride (823). Benzylmercuric iodide and di-benzylmercury in dimethylformamide and acetonitrile (909) and cobalticenium ion and cobaltocene in water and alcohol (892) were studied.

The oxidation of tetraphenylborate was carried out at rotating Pt (840, 844) and pyrolytic graphite (842). The utility of the carbon paste electrode was demonstrated by applying it to ferro-cene analysis (450).

Two waves were found for the reduc-tion of cyclopentadienylalkylpalladium and derivatives (252). A one-electron reduction for $\text{RhCl}(\text{CO})[(\text{C}_6\text{H}_5)_3\text{P}]_2$ and three waves for dicyclopentadienylti-tanium which are a main, adsorption, and catalytic wave (425) were observed.

Oscillographic data for ferrocene derivatives (414) and chronopotenti-ometric studies of 49 *o*-, *m*-, and *p*-sub-stituted phenyl ferrocenes (473, 474) were correlated to Hammett and Taft values. Half-wave potentials for bi-phenyl derivatives substituted in the 4 and 4' positions by trimethyl group IV elements were correlated to energies of lowest unoccupied orbitals (146).

The adsorption of tributyl phosphate in presence of gelatin (603, 604) and tri-ethyl phosphate effects on double layer capacity (335-337) were studied.

Complexes between alkyl mercury and thiocyanate ion or thiourea were ex-aminated (825).

Procedures for the analysis of tributyl-tin chloride and dibutyltin dichloride (541) and tetraphenyltin in polystyrene and polyvinyl chloride (76) were de-scribed. The analysis of the former compounds was improved by modifying

alternating current procedure (338).

Ethylmercuric chloride in fungicides (665), phosphoric acid ester type insec-ticides (561), *p*-toluenearsonic acid in flotation agents (649) tetraethyl lead in petroleum (943), and organometallic stabilizers in polyvinyl chloride (422) were determined. Analysis of tetra-phenyl borate salts of primary and secondary alkyl amines was described (261).

Phenylmercuric acetate derivatives of barbituric acid (220) and theophylline (219) were used for analysis. Thimersol, sodium salt of [*o*-carboxyphenyl]thio]-ethyl mercury, in vaccines was deter-mined (83).

Oxygen-, Sulfur-, and Selenium-Containing Heterocyclic Compounds. Polarographic studies are reported for tocopherols (407), 2,4,6-trimethylpyr-lyum perchlorate and tetrachloroferrate and hexaphenylbipyran (37), fluorescein in strong base (358), xanthidrol and xan-thone (275, 276), thiophene, furan, *o*-hy-droxychalcone, chromone, and chro-manone and derivatives (816, 817), *S*-substituted furfuryl thiocyanates (777), and isobenzopyrylium salts and sub-stituted derivatives (870). The data in the latter case were correlated to the spectra for the compounds (871).

Data for xanthene phthalein deriva-tives (634) and the effects of alkyl sub-stitution on half-wave potentials for 3-aryl-4-hydroxycoumarin (878) were re-ported.

An oscillopolarographic study of 3-hydroxy- and 3-methoxy-4-pyrone and derivatives showed one irreversible re-duction wave (244) in acid. Adsorp-tion measurements for coumarin and 6-substituted coumarin are described (251).

Furfural was analyzed in pulp hy-drolysis products (762, 763), in soil (87), and studied in presence of different buffers (778).

Furan after nitration (883) and flavo-noids after nitrosation (158) were ana-lyzed polarographically. Procedures for the analysis of flavonoids in the food industry (159) furocoumarin-like com-pounds in drugs (595) tocopheronolac-tone (715), and khellin and furobenzo-pyranones after separation (594, 596) were described. Glycosides of cardiac importance were analyzed polaro-graphically (163, 902).

The complexes between Co(III) and Tl(III) with rhodamine B were studied (313-315).

In the sulfur-containing heterocyclic series, data are reported for the analysis of thiophene after nitration (753, 819) and after oxidation with H_2O_2 (333). A theoretical study of adsorption and its effect on reduction was tested with 5-bromo-2-acetylthiophene (492).

Nitrogen - Containing Heterocyclic Compounds. In the pyridine series, polarographic studies are reported

for 2- and 4-vinylpyridine (12), cyanine dyes (756) differing by the length of carbon chain (260), phenanthridine and derivatives (386), isonicotinic hydrazide (478), isonicotinic acid and *N*-alkyl derivatives (479, 482), 1-acylpyridinium chlorides (731), pyridine and *N*-alkylpyridinium salts (755), pyridine and derivatives (816), 2,2'-dimethyl-5,5'-diethyl-4,4'-azopyridine and its 1,1'-dioxide and 5-ethyl-2-methylpyridine *N*-oxide (554), 1-methyl-3-carbamidopyridinium chloride (113), and derivatives of pyridoxine, pyridoxamine, and pyridoxal (511).

The catalytic wave for pyridine was studied in unbuffered media (404, 678, 679) and in the presence of KCl and CsCl (651). Catalytic wave studies for pyridine, 2,6-lutidine, and anabasine (497) were also reported.

Adsorption and effects on reduction were tested by examining 4-bromopyridine (492). The surface active properties of 2,6-lutidine (494) and cetylpyridinium bromide (419, 420) were studied.

The dimerization process that occurs after reduction of quaternary pyridinium salts was examined (489). *N*-Nitroanabasine was found to reduce in two steps to the hydroxyl and then the amino derivative (836, 837). Complexes between Cr(III) and 2,2'-bipyridine (835) and the reaction of pyridoxal with hydroxylamine (950) were studied. An oscillographic study of the products of the reduction of pyridine, 2-picoline, and 2,6-lutidine proved the presence of dipyridyls, polypiperidyls, free aldehydes, and pyrans (357).

Half-wave potentials for several pyridine-like heterocyclics were correlated to energies of the lowest free molecular orbital (609, 610).

The method has been used to determine 1,1-trimethylenebis (4-formylpyridinium)bromide dioxime (865), 2-pyridine aldoxime methiodide (866), 2-(β-methoxyethyl) pyridine (265), picolin-aldehyde and picolinic acid (513), and Diodrasl in blood and urine (664). 2-Methyl-5-vinylpyridine in polymerization reactions (7) and 2-methyl-3-nitromethoxymethyl-5-cyanopyridine and 2-methyl-3-nitro-4-methoxymethyl-5-cyano-6-chloropyridine as intermediates in pyridoxole synthesis (417, 418) were analyzed.

In the quinoline-like series data are reported for 8-hydroxyquinoline (115, 804), quinoline and derivatives (816, 817), quinoline, isoquinoline, 3,4-hydroxyisoquinoline, papaverine, and 3,4-dihydropapaverine as methiodides (537) and for the analysis of isoquinoline in heavy bases from coal tar after separation (520). The effect of alcohol on the Cu(II) - 8-hydroxy-5-quinoline sulfonic acid complex was measured (60). Molybdenum was titrated amperometrically with 8-hydroxyquinoline

(509). Half-wave potentials for 8-substituted hydroxyquinolines and 8-substituted quinaldines (839) were correlated to Hammett values (846).

Pyrrole and *N*-methyl pyrrole were analyzed after nitration (819). Other pyrrole-like compounds studied were the porphyrins and derivatives (316, 680, 714, 728, 758).

In the piperidine-like series data are reported for the catalytic wave for codeine (881), analysis of codeine (702), and for hydroxylcoctonine as a function of pH (921). Morphine was analyzed (939) after nitration (597) and after chromatographic separations (282). Analysis of morphine and derivatives by oxidation at rotating Pt in 4*N* H₂SO₄ (164), 1-piperidine-2-methyl-3-(*p*-tolyl)-3-propanone hydrochloride (152), and pyrimidinone and 1-piperidinepropanol after nitration (96) was reported.

The polarographic behavior under hydrolysis conditions for 34 compounds of the type RN(CH₂CH₂Cl)₂ where R is a heterocyclic group was reported (662). Oxidation studies on chlorophylls a and b and pheophytins a and b at rotating Pt disk (757), uric acid at graphite electrode (784, 785, 788), and 9-hydrazoacridine (121) were described. Data are reported for isothiocyanatoacridine derivatives (434), alloxan, alloxantin, and dialuric acid (786), parabamic acid (787), azomethine reduction in benzodiazepine derivatives (724), chlorophyll and related compounds (189), and for hemoglobin as a function of pH and temperature (354). Oscillography was used to study a wide variety of *N*-containing heterocyclics (898) while alternating current polarography was used for a series of flavin mononucleotides (723). Degradation of alkaloids (611) and changes in enzymic activity of adenosine diaminase (349) was followed polarographically. Data for monoaza-aromatic derivatives were correlated to orbital energy calculations (17).

Data for riboflavine and nicotinamide (407, 552) and their analysis along with pyridoxal, thiamine, nicotinic acid, B vitamins (162, 180, 813) were reported. The method was used for the analysis of alkaloids (168) after separation and nitrosation (831), dimer of cycloserine (796) lobelins (612), orotic acid (438), adenylic acid in yeast ribonucleic acid (534), glucobrassicin in plant material (448), acetonitrile in blood, urine, and organs (644), allantolactone, isoallantolactone, and dihydroisoallantolactone (539), ajmaline after nitrosation (830), parabamic acid (789), Niamid and Marsilid, derivatives of isonicotinic acid, in tablets (97), furazoline, *N*-(5-nitro-2-furfurylidene-3-amino-5-morpholinomethyl-2-oxazolidinone, in water, urine, and blood plasma (775), and isonicotinic acid hydrazide after reaction with 1,2-naphthoquinone (558).

Data for vitamin B₁₂ (92) and 5-methyl- and 5-hydroxy-derivatives of cytosine (328) were reported. Reduction in the latter case involves splitting off of the amino group bound to C-4 and a reduction of the 3,4 double bond.

Investigations of heterocyclic compounds containing nitrogen and oxygen report data for derivatives of 2-pyrazoline in dioxane and ethanol (53) and for the analysis of nitrofurazone and furazolidone in feeds (212). The reduction of C=N, C=C, and C=O bonds were found to take place in studies with 1,3-oxazole derivatives and 1,3,4-oxadiazole derivatives (80).

Data for heterocyclic compounds containing two or more nitrogen atoms are reported for 2-amino-4-hydroxypteridine derivatives (28), tetrazolium salts (326), 50 mono- and di-substituted phenazines and *N*-oxides (692), quinazoline and derivatives (481, 483), pyrazine and methyl derivatives (262, 900), pyrimidines and purines and their analysis (178, 306), azotetrazolium chloride (598), nitro, aldehyde, and halogen derivatives of imidazole (456, 457), pterin derivatives (392), indazolone and derivatives (247), and acridizinium and phenanthridizinium ions and derivatives (215, 216).

Data for azines such as 9,10-diaza-phenanthrene, 2,2'-bipyrimidine and Δ^{2,2'}-biisobenzimidazolylidene were measured in dimethylformamide in order to facilitate the selection of conditions for anion radical formation for EPR studies (239). Half-wave potentials for substituted 2-amino-3-anilino-5-phenylphenazinium chlorides (428) and substituted sydnones and sydnominines (388) were correlated to Hammett constants.

Compounds produced in decomposition of cytostatic active compounds were found to contain the polarographically active 1-aziridinyl ring (300). Oscillography was used to follow naphthoylbis (benzimidazole) as it participated in a reaction (22) and changes in six pyrimidines and 3 cytosine derivatives upon exposure to ultraviolet light (347). Studies on 2-amino-4-hydroxy-6-methyl pteridine revealed a two-electron reduction that occurs in the pyrazine ring (415).

Methods of analysis are reported for 1,3,5-triphenyl-Δ²-pyrazoline (458), adenine and adenine derivatives in blood (466), 6-azauracil riboside (111), Dimetridazole, 1,2-dimethyl-5-nitroimidazole, in feeds (147), and hydroxyquinoxaline and 2-sulfanilamidoquinoxaline (765).

Investigations of heterocyclic compounds containing nitrogen and sulfur report data for 13 phenothiazine sulfonoxides and sulfones (437), eight *N*-substituted phenothiazines (544, 545), nitro, aldehyde, and halogen derivatives of thiazole (457), 5-benzoylthiamine o-monophosphate (587), 2,2'-azines of

aromatic heterocyclics (305) and for the analysis of furothiazole (780).

Reduction studies of 2-methyl-3-hydroxy-5-(hydroxymethyl)-4-pyridylthiazolidine-4-carboxylic acid as a function of pH revealed a two-electron reduction wave with opening of the thiazolidine ring (512).

Alternating current polarography of 27 S-acyl derivatives of thiamine revealed two waves with the first due to the S-acyl bond and the second due to the pyrimidine ring. The peak potential of the first wave was correlated to Hammett values (584). The effect of phenothiazine and derivatives on the oxygen wave which is attributed to complex formation was observed, and the shift was correlated to Hammett values (526).

Binding between thiazine red-R and fluorescein with proteins was studied (235).

ACKNOWLEDGMENT

The author thanks Sherlock Swann, Jr., and Stanley Wawzonek for their help in scanning *Chemical Abstracts*.

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