[CONTRIBUTION FROM THE DEPARTMENT OF EXPERIMENTAL MEDICINE, NORTHWESTERN UNIVERSITY MEDICAL SCHOOL]

Polarographic Studies of Organic Mercury Compounds[1]

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A series of organic mercury compounds, *i.e.*, simple phenylmercuric compounds, *p*-chloromercuribenzoic acid, Merthiolate and mercurial diuretics of the mercurated allylamide type, has been investigated polarographically. Their behavior differs markedly from that of inorganic mercury compounds, since they are reduced irreversibly in two single-electron steps at the dropping mercury electrode. The effects of *p*H and concentration on the half-wave potentials, as well as the general chemical properties of these compounds, suggest that the reduction leads first to the formation of organomercury free radicals which are further reduced to the corresponding hydrocarbon and metallic mercury. The evidence in favor of this mechanism is presented and discussed. The polarographic reduction of these compounds provides a simple and specific method for their analytical determination and suitable conditions for analytical measurements are described. In the case of the mercurial diuretics the method shows promise for application to biological fluids.

The polarographic behavior of mercuric and mercurous salts[2,3,4] has been studied by several investigators and the waves obtained have been satisfactorily interpreted. However, little attention has been paid to the polarographic reduction of compounds in which mercury forms one or more covalent bonds with carbon. The first reference to a reduction of a compound of this type at the dropping mercury electrode was made by Sullam.[5] He observed that the decomposition potential of compounds obtained by the interaction of mercuric salts with ethylene ($CH_2OH{\cdot}CH_2HgX$) differs from that of inorganic mercury compounds, but did not report any numerical values. Recently Page and Waller[6] have shown that the polarographic reduction of Merthiolate may be used for the determination of this compound in pharmaceutical preparations. It was demonstrated that Merthiolate as well as phenylmercuric acetate and nitrate are reduced at more negative potentials than free mercury ions, but no studies on the mechanism of the reduction are reported.

The present investigation was started in view of the wide use of monovalent organic mercury compounds in the study of biologically occurring thiols.[7–11] This paper deals with the behavior of some of these compounds at the dropping mercury electrode and the mechanism of their reduction. Polarographic studies on the interaction of these compounds with thiols are in progress and will form the subject of a subsequent report.

Experimental

Materials.—Phenylmercuric chloride, acetate and borate, pharmaceutical grade, Metalsalts Corp., Hawthorne, N. J. Phenylmercuric nitrate (basic), pharmaceutical grade, Hamilton Laboratories, Asheville, N. C. Phenylmercuric hydroxide, prepared from basic phenylmercuric nitrate as described by Schramm.[12] *p*-Chloromercuribenzoic acid highly purified sample prepared by the method of Whitmore and Woodward.[13] Salyrgan (sodium *o*-(γ-hydroximercuri-β-methoxypropylcarbamyl)-phenoxyacetate), pharmaceutical grade, Winthrop–Stearns Inc., Rensselaer, N. Y. Salyrgan/Theophylline, approximately two parts by weight of Salyrgan to one part by weight of Theophylline, pharmaceutical grade, Winthrop–Stearns Inc., Rensselaer, N. Y. Mercurin (sodium salt of β-methoxy-γ-hydroxymercuripropylamide of trimethylcyclopentanedicarboxylic acid) was kindly donated by Dr. D. L. Tabern of Abbot Laboratories, North Chicago, Ill. Mercurophylline, approximately equal molecular proportions of Mercurin and Theophylline, pharmaceutical grade, Campbell Products Inc., New York, N. Y. Mercuhydrin Sodium (3 moles of the sodium salt of methoxyoximercuripropylsuccinylurea and two moles of Theophylline), pharmaceutical grade, Lakeside Laboratories Inc., Milwaukee, Wis. Merthiolate, Eli Lilly and Co.

Apparatus.—The instrument used in these investigations was a Sargent Model XXI automatically-recording polarograph. An H cell[14] with a saturated calomel electrode as the anode was used for all measurements with the exception of those where chloride had to be excluded. In this case connection between the saturated calomel electrode and the electrolysis cell was made through a junction vessel with a 13% potassium nitrate agar bridge. Oxygen was removed from the test solutions by the passage of oxygen-free nitrogen. All measurements were carried out at 25 ± 0.5°. The capillary had the following characteristics: $m = 2.254$ mg. mercury per second, $t = 3.55$ seconds per drop at -0.20 volt.

Procedure.—The measurements were made in Britton–Robinson buffers, 0.05 molar in acetate, phosphate and borate.[15] This universal buffer mixture gives a practically linear titration curve over the range of *p*H 3.4 to 10.3 and the concentration used was always at least one hundred times that of the test substance. This is considered to be adequate to maintain a constant *p*H at the dropping mercury electrode.[16] The organic mercurials were dissolved in water or, if necessary, in standard sodium hydroxide. They were then mixed with 10-ml. portions of the buffer acid mixture (0.1 *M*) and sufficient standard sodium hydroxide to give the required *p*H. After the addition of suitable supporting electrolytes, maximum suppressors, etc., the solutions were diluted to a final volume of 20 ml. and polarographed. The supporting electrolytes used were potassium chloride, potassium nitrate and potassium perchlorate in 0.1 *M* concentration. All solutions of the mercury compounds were made up fresh daily from stock solutions which were carefully protected from light.

Results

Unsubstituted Phenylmercuric Compounds.—These compounds are reduced in two steps (Fig. 1). When polarographed in Britton–Robinson buffer solution with potassium chloride as the supporting electrolyte, the half-wave potentials

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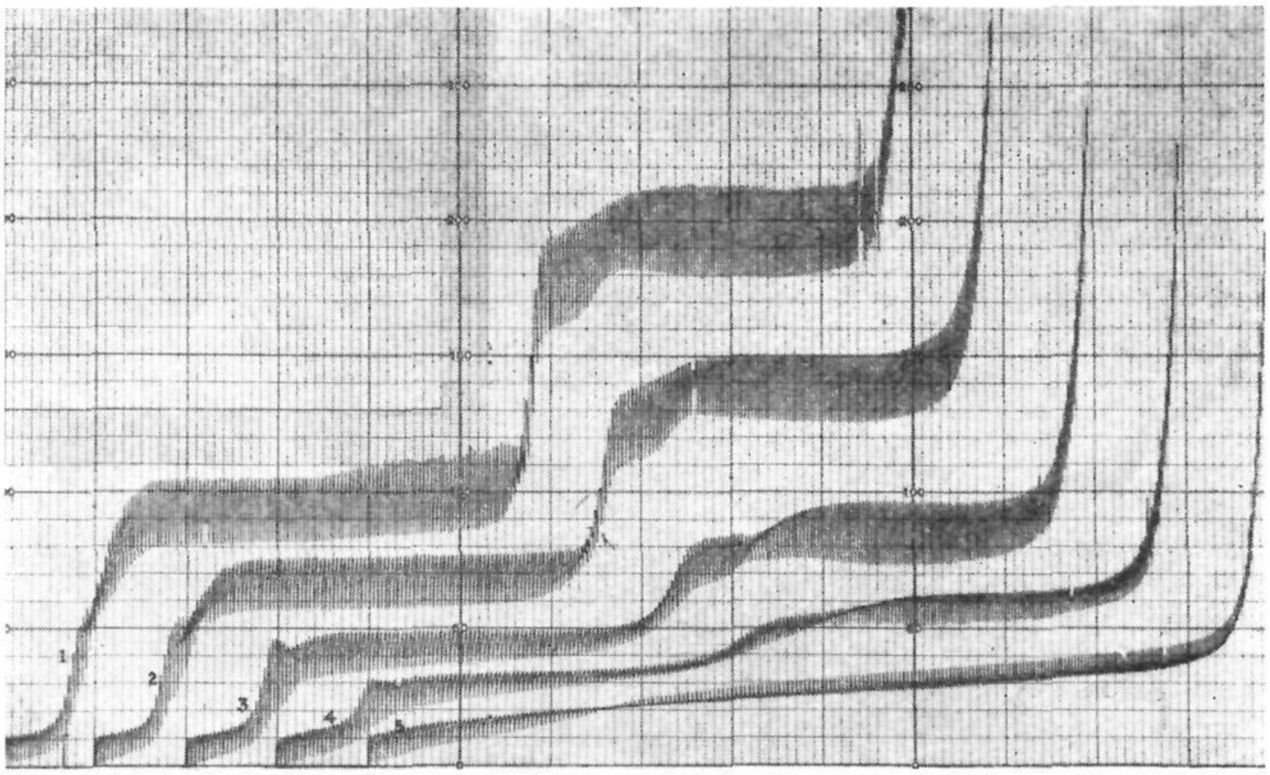

Fig. 1.—Phenylmercuric hydroxide in Britton–Robinson buffer *p*H 9.5 and 0.1 *M* potassium chloride. Each polarogram record from 0 to −2.0 volts and sensitivity = 0.015 μa/mm.: 1 at 3 × 10^{-4} *M*; 2 at 2 × 10^{-4} *M*. 3 at 1 × 10^{-4} *M*; 4 at 0.5 × 10^{-4} *M*; 5 is base line.

versus the saturated calomel electrode range from −0.10 to −0.25 volt for the first wave and from −0.80 to −1.20 volts for the second wave, depending on *p*H and concentration. These waves are quite distinct from those obtained with free or complexed inorganic mercury and it was indeed found possible to determine phenylmercuric nitrate and mercuric nitrate together in 0.1 *M* nitric acid with methyl red as a maximum suppressor. The polarographic behavior of different unsubstituted phenylmercuric compounds was found to be very similar. No significant differences could be detected in the half-wave potentials of phenylmercuric hydroxide, phenylmercuric chloride, phenylmercuric acetate, phenylmercuric borate and phenylmercuric nitrate when these compounds were electrolyzed in the Britton–Robinson buffer solutions.

The half-wave potential of the first wave was found to be independent of concentration at all *p*H values investigated (*p*H 4.7 to 10.0). The half-wave potential of the second wave becomes more negative with increasing concentration at *p*H 9 or above, the $-E_{1/2}$ being directly proportional to log *c*. The variation of the half wave potentials of both waves with *p*H is shown in Fig. 2. It can thus be seen that whereas the half-wave potential of the first wave is essentially constant at acid *p*H values and varies at alkaline *p*H values, the converse is true of the half-wave potential of the second wave.

TABLE I

EFFECT OF THE ANION OF THE SUPPORTING ELECTROLYTE ON THE HALF WAVE POTENTIAL OF BOTH WAVES OF UNSUBSTITUTED PHENYLMERCURIC COMPOUNDS

Phenylmercuric borate, 1 × 10^{-4} *M*; Britton–Robinson buffer, 5 × 10^{-2} *M*; supporting electrolyte, 1 × 10^{-1} *M*

*p*H 7.0	Chloride	Nitrate	Perchlorate
$-(E_{1/2})_1$	0.12	0.10	0.06
$-(E_{1/2})_2$	1.02	1.06	1.00
*p*H 4.7			
$-(E_{1/2})_1$	0.12	0.02	0.02
$-(E)_{1/2 2}$	0.86	0.89	0.86

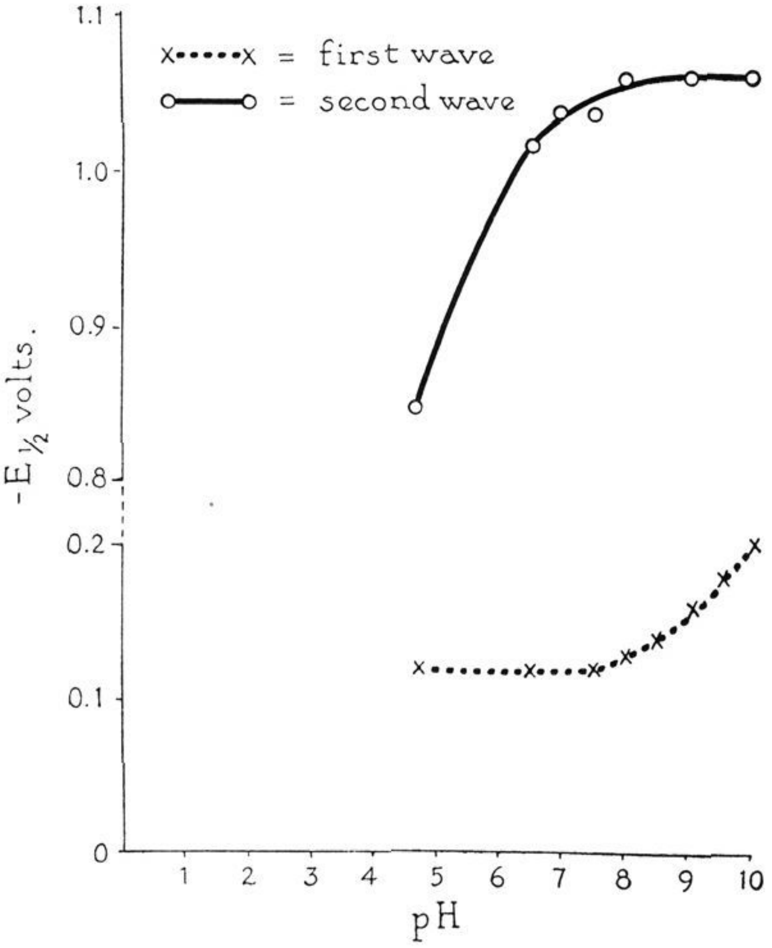

Fig. 2.—Variation of the half wave potentials of unsubstituted phenylmercuric compounds with *p*H: phenylmercuric chloride, 1 × 10^{-4} *M*; Britton–Robinson buffer, 5 × 10^{-2} *M*; potassium chloride, 1 × 10^{-1} *M*.

It is noteworthy that the half-wave potential of the first, but not that of the second wave is affected by the anion of the supporting electrolyte, particularly in acid solution. This is illustrated in Table I. It is evident that the half-wave potential of the first wave is more positive in the presence of nitrate and perchlorate ions than in chloride. This is in agreement with the relative stability of the mercury complexes formed by these anions.

It can be seen (Fig. 1) that a maximum-like irregularity appears after the second wave. This is most pronounced in alkaline solution and almost disappears at lower *p*H values. It does not usually interfere with the measurement of the second wave. The concentration range over which exact measurements can be obtained was, however, found to be limited because of severe irregularities which appear in the region of the electrocapillary maximum at concentrations depending on the sample of phenylmercuric compound used. They appeared, for instance, at 3×10^{-4} *M* with our sample of phenylmercuric chloride, but only at 4×10^{-4} *M* with our phenylmercuric hydroxide. These irregularities were eliminated in the presence of 0.01% gelatin.

Experiments on the relation between wave height and concentration at various *p*H values in the presence of chloride are limited by the insolubility of phenylmercuric chloride. Figure 3 shows the decrease in wave height with decreasing *p*H at higher concentrations due to the formation of this compound. It is, therefore, evident that analytical proportionality between wave height and concentration can only be expected at *p*H 9 or above. The wave heights were obtained by measuring the distance between the projected base line and the diffusion current plateau at the half-wave potential. Table II shows the linear relationship between the height of the first and of the second wave and concentration, at *p*H 9.5, up to a concentration of 2×10^{-4} *M*.

TABLE II

RELATION BETWEEN WAVE HEIGHT AND CONCENTRATION FOR BOTH WAVES OF UNSUBSTITUTED PHENYLMERCURIC COMPOUNDS

Britton–Robinson buffer *p*H 9.5, 5×10^{-2} *M;* potassium chloride, 1×10^{-1} *M*

	First wave		Second wave	
Concn., mM.	i_d (μa.)	i_d/c	i_d (μa.)	i_d/c
0.04	0.16	4.0	0.17	4.2
.05	.22	4.4	.22	4.4
.06	.27	4.5	.28	4.7
.08	.36	4.5	.38	4.7
.10	.45	4.5	.44	4.4
.20	.86	4.3	.87	4.4
.30	1.26	4.2		
.40	1.74	4.3		

***p*-Chloromercuribenzoic Acid.**—This compound was investigated as an example of a substituted phenylmercuric compound in view of its wide use as a thiol group reagent. It resembles the unsubstituted phenylmercuric compounds by being reduced in two steps. The half-wave potential of the first wave was −0.28 volt *versus* the saturated calomel electrode in Britton–Robinson buffer solution (*p*H 9) with potassium chloride as supporting electrolyte. It was found to be independent of concentration as in the case of the simple phenylmercuric compounds. Measurement of the second wave was difficult because of the large flat maximum which it shows. In addition, a large catalytic maximum occurs at about −1.2 volts. The top of this maximum is about fourteen times as high as the diffusion current of the first wave at the same concentration. Studies of the variation of the half-wave potential with *p*H could not be undertaken, in view of the insolubility of this compound in neutral or acid solution. In solutions of *p*H 8 or above, the height of the first wave was found to be proportional to concentration in the range studied, *i.e.*, 3 to 10×10^{-4} *M*.

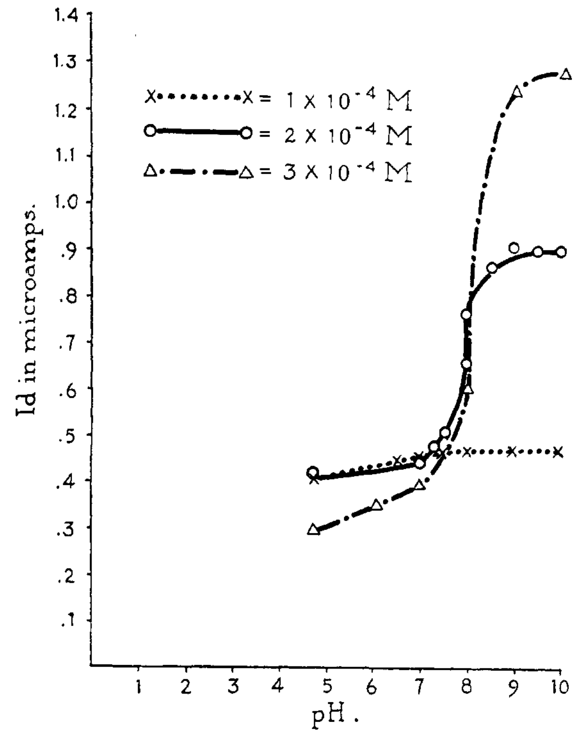

Fig. 3.—Decrease in the wave height of unsubstituted phenylmercuric compounds with decreasing *p*H in the presence of chloride: Britton–Robinson buffer, 5×10^{-2} *M;* potassium chloride, 1×10^{-1} *M*.

Sodium Ethyl Mercuri Thiosalicylate (Merthiolate).—Page and Waller,[6] who studied this compound, reported that it gave two steps, the position and height of which varied considerably with *p*H and that the first step was not formed in strongly acid solution.

Upon investigation of this compound, we again found it to be reduced in two waves of equal height (Fig. 4). A catalytic maximum also occurs at potentials more negative than those included in the figure. These two waves are only apparent in alkaline or neutral solution. At more acid *p*H values the catalytic maximum becomes more positive thus obscuring the second wave. This second reduction wave was not described by Page and Waller. The heights of both waves are proportional to concentration between 1×10^{-4} *M* and 10×10^{-4} *M* but the first wave can be used for analytical measurements over a wider *p*H range.

The half-wave potential of the first reduction wave varies approximately linearly with *p*H according to the equation: $-E_{1/2} = 0.42 + 0.05\,p\text{H}$. The theoretical variation for the participation of one hydrogen ion would be 0.059 volt per *p*H unit. The values calculated from this equation agree well

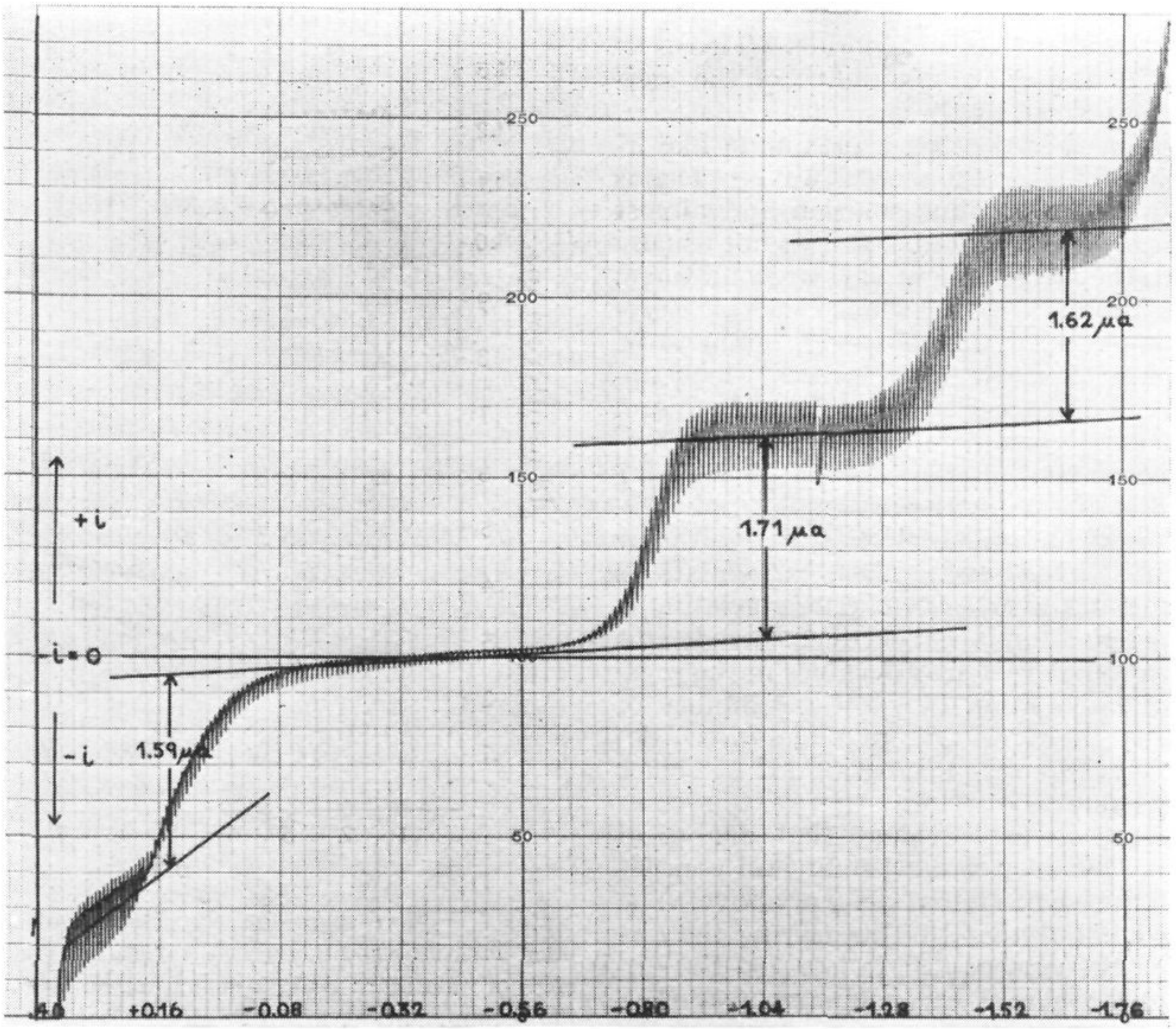

Fig. 4.—Polarogram of Merthiolate: 5 × 10^{-4} M; potassium nitrate, 1 × 10^{-1} M; gelatin, 0.01 %.; sensitivity, 0.030 μa/mm.

with those reported by Page and Waller for their "second step." The position of the second reduction wave was found to be independent of pH, the half-wave potential in Britton–Robinson buffer and 0.1 M potassium chloride being −1.2 volts *versus* the saturated calomel electrode.

In addition to these two reduction waves, an anodic wave also occurs at more positive potentials (Fig. 4). This wave presumably corresponds to the "first step" of Page and Waller. It is best studied in the absence of chloride, *e.g.*, in 0.1 M nitric acid or potassium nitrate, when its half-wave potential is about +0.1 volts *versus* the saturated calomel electrode. The height of this wave is equal to that of each of the reduction waves up to a concentration of 5 × 10^{-4} M, above which it begins to decline.

Mercurial Diuretics.—These compounds are formed by the mercuration of substituted allylamides. Salyrgan, a typical compound of this class, was subjected to detailed investigation. This compound was again found to be reduced in two steps, followed by a large catalytic maximum. The half-wave potential of the first wave is −0.32 volt *versus* the saturated calomel electrode, in Britton–Robinson buffer with potassium chloride as supporting electrolyte, and is independent of pH. The second wave becomes more positive with increasing hydrogen ion concentration (Table III) until it merges with the first wave and only a single wave is obtained at pH 4. The height of this wave is equal to the sum of the heights of the two waves observed in neutral or alkaline solution and was found to be strictly proportional to concentration between 0.5 × 10^{-4} M and 5 × 10^{-4} M. This pH is therefore very suitable for analytical measurements, but greater acidities must be avoided, since the carbon–mercury bond in this type of compound is split in more acid solution.

The waves obtained in neutral solution are too closely spaced for exact measurement. In alkaline solution the second wave is difficult to measure because of the proximity of the catalytic maximum. Analytical measurements can, however, be carried out in alkaline solution on the first wave, since its height is proportional to concentration. At concentrations greater than 4 × 10^{-4} M this wave splits up into two waves, but the sum of the two wave heights remains proportional to concentration. This splitting could be largely suppressed by 0.1% gelatin.

Since Salyrgan and other mercurial diuretics are commonly dispensed in combination with theophylline, with which they form complexes, it was thought of interest to examine some of these compounds. From Table III it is evident that theophylline has little effect on the polarographic behavior of these compounds, except in alkaline solution where the half-wave potential of the first wave is more negative in the case of the theophylline complexes (compare Salyrgan with Salyrgan/Theophylline and Mercurin with Mercurophylline). This is presumably a reflection of the greater stability of the theophylline complexes in alkaline solution.

TABLE III

EFFECT OF *p*H ON THE HALF-WAVE POTENTIALS OF MERCURIAL DIURETICS

Mercury compounds, 3×10^{-4} *N;* Britton–Robinson buffer, 5×10^{-2} *M;* Potassium chloride, 1×10^{-1} *M*

	Salyrgan		Salyrgan/theophylline		Mercurin		Mercupurin		Mercuhydrin	
*p*H	$-(E_{1/2})_1$	$-(E_{1/2})_2$	$-(E_{1/2})_1$	$-(E_{1/2})_2$	$-(E_{1/2})_1$	$-(E_{1/2})_2$	$-(E_{1/2})_1$	$-(E_{1/2})_2$	$-(E_{1/2})_1$	$-(E_{1/2})_2$
4.0	0.31	0.31	0.31	0.31	0.37	0.37	0.37	0.37	0.36	0.36
5.0	.33	..								
6.0	.3[a]	.5[a]								
7.0	.3[a]	.6[a]	0.4[a]	0.6[a]	0.3[a]	0.6[a]	0.4[a]	0.6[a]	0.4[a]	..
8.0	.32	.80								
9.0	.33	.98	0.46	1.00	0.33	0.88	0.46	0.90	0.47	0.95

[a] Approximate values.

Discussion

Mechanism of the Reduction.—The reduction of the organic mercury compounds under discussion must be regarded as irreversible. Several lines of evidence lead to this conclusion: (1) The slopes of the waves varied with conditions such as concentration and *p*H and no simple relationship between potential and current (such as a linear plot of E *versus* $\log i/(i_d - i)$ was found to describe the waves. (2) With most of the waves irregularities (Fig. 1) typical of irreversible reactions were observed. (3) The variation of the half-wave potential of the second wave of the unsubstituted phenylmercuric compounds with concentration would be difficult to understand if the reaction were reversible. Although a number of criteria is necessary to prove the reversibility of a reaction, any of the above points would suffice to rule out reversibility.

The reduction was, however, shown to be diffusion controlled, since the diffusion current constants ($i_d/cm^{2/3}t^{1/6}$) obtained with two capillaries of widely different drop time, *i.e.*, 3.40 and 1.42 seconds, were almost identical, *i.e.*, 2.1 and 2.2, respectively. Therefore, the Ilkovic equation was used to calculate the number of electrons involved in each reduction step. For the purpose of this calculation it was thought reasonable to use as a first approximation the diffusion coefficient of 0.9×10^{-5} cm.² sec.⁻¹ which is the value reported by Kolthoff and Miller[2] for mercurous ion. The results are shown in Table IV.

TABLE IV

	First wave		Second wave		Anodic wave	
Compound	$\frac{i_d}{cm^{2/3}t^{1/6}}$	n	$\frac{i_d}{cm^{2/3}t^{1/6}}$	n	$\frac{i_d}{cm^{2/3}t^{1/6}}$	n
C_6H_5HgX	2.10	1.16	2.10	1.16	..	..
$COOHC_6H_4HgCl$	1.71	0.94	..	..	..	..
Merthiolate	1.63	.90	1.63	0.90	1.63	0.90
Salyrgan (alkaline)	1.54	.85	1.54	0.85	..	..
Salyrgan (acid)	3.05	1.68	..	..	..	..

As might be expected, the calculated values of n reflect the trend in the molecular weights of these compounds.

The irreversibility of the electrode reaction makes it difficult to postulate an exact mechanism for the reduction process. However, the polarographic as well as other evidence discussed below makes the following mechanism appear feasible

$$RHgX + e = RHg\cdot + X^- \qquad \text{(1a)(acid solution)}$$

$$RHgOH + H^+ + e = RHg\cdot + H_2O \qquad \text{(1b)(alkaline solution)}$$

$$RHg\cdot + H^+ + e = RH + Hg \qquad (2)$$

The considerations which lend support to this scheme are:

(1) The reduction proceeds in two single-electron steps (Table IV).

(2) The observed variation of the half-wave potential of the first wave with *p*H (Fig. 2) is in agreement with the reactions 1a and 1b above.

(3) Reaction (2) involves a split of the carbon–mercury linkage. As is well known, the stability of this linkage decreases with increasing hydrogen ion concentration. This should cause a shift of the second wave to more positive potentials as the *p*H is decreased and this is indeed the case (Fig. 2, Table III). Moreover, the greater lability of the carbon–mercury linkage in compounds of the mercurated allylamide class, as compared with that in the phenylmercuric compounds, is reflected by the observed fact that the half-wave potential of the second wave of the former was more positive than that of the latter at all hydrogen ion concentrations studied (*cf.*, Fig. 2 and Table III).

(4) Reaction (1), above, involves the formation of an organomercury free radical. The existence of such radicals has been known since Kraus[17] found methylmercury free radicals to be deposited on the cathode during the electrolysis of methylmercuric chloride in liquid ammonia. At higher temperatures these radicals dimerize to form dimethylmercury. This was later confirmed by other workers.[18] Since diphenylmercury was found to be irreducible at the dropping mercury electrode (in 50% ethanol) and is also known to be very resistant to chemical reduction, the existence of a second reduction step argues in favor of the intermediate formation of RHg· free radicals. It is clear that the second reduction step can only occur if the rate of dimerization to R_2Hg, of the RHg· free radicals formed as the result of the first step, is slow compared to the rate of their reduction to RH and mercury. Two factors tend to favor dimerization:

(a) Increase in concentration, since the rate of dimerization would be expected to increase with the square of the concentration ($2\,RHg\cdot = (RHg\text{-}HgR) = R_2Hg + Hg$). This explains the shift of the half-wave potential of the second wave of the phenylmercuric compounds to more negative values with increasing concentration. In this connection it is also of interest to report that a good yield of diphenylmercury (m.p. 123°) was obtained when a solution 2×10^{-3} *M* in phenylmercuric acetate and 1×10^{-1} *M* in potassium nitrate was electrolyzed with vigorous stirring at a large mercury cathode,

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[17] C. A. Kraus, THIS JOURNAL, **34**, 1732 (1913).

[18] F. O. Rice and B. L. Evering, *ibid.*, **56**, 2105 (1934).

at a potential of −1.4 volts *versus* the saturated calomel electrode, since complete dimerization of the radicals would be expected under these conditions.

(b) Decrease in hydrogen ion concentration, since compounds of the type RHgX are converted to R_2Hg by reducing agents such as sodium stannite in alkaline solution, but no R_2Hg is obtained in acid solution, where only free mercury is liberated.[19] This is in agreement with the effect of *p*H on the half-wave potential of the second wave already referred to (Fig. 2, Table III).

(5) The over-all reduction, *i.e.*, steps (1) *and* (2), has been effected chemically. Thus phenylmercuric acetate is reduced by sodium amalgam in the presence of water with the formation of mercury, benzene and sodium acetate.[20] It has also been shown that ethylmercuric chloride is reduced by sodium in liquid ammonia with the formation of sodium amalgam and ethane.[21] Furthermore, Sand and Singer[22] have demonstrated that the reduction of ethanol mercuric iodide and isopropanol mercuric iodide by sodium amalgam, or by electrolytically generated hydrogen, leads to the production of pure ethanol and isopropanol, respectively. This is particularly significant since the structure of these compounds is analogous to that of the mercurial diuretics discussed in this paper.

Anodic Wave of Merthiolate.—This wave must be ascribed to the depolarization of the dropping mercury electrode by the thiosalicylic acid formed as a result of the dissociation of Merthiolate. It is probably analogous to the wave described for the anodic reaction of cysteine by Kolthoff and Barnum.[23] The decrease in the relative height of this wave at concentrations greater than 5 × 10^{-4} *M* is therefore probably due to the formation of a film of insoluble mercaptide, as postulated by these authors in the case of cysteine. Below 5 × 10^{-4} *M* the wave does, however, correspond to a one-electron step. This anodic wave, as well as those due to other mercaptides of inorganic and organic mercury compounds, is at present under investigation and will form the subject of a later communication.

In conclusion it should be emphasized that the polarographic method described in this paper provides a simple and rapid means for the determination of organic mercury compounds directly and specifically, which has not been possible by any other method. Preliminary studies have shown that the polarographic assay is applicable to the detection and estimation of mercurial diuretics in biological fluids such as urine.

Acknowledgment.—This investigation was supported by a research grant from the National Cancer Institute, of the National Institutes of Health, Public Health Service. The authors also wish to express their gratitude to Dr. D. D. De Ford for valuable discussion and criticism of this work.

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[19] F. C. Whitmore, "Organic Compounds of Mercury," A. C. S. Monograph Series, New York, N. Y., 1921, p. 62.

[20] *Ibid.*, p. 175.

[21] C. A. Kraus and L. Kurtz, THIS JOURNAL, **47**, 43 (1925).

[22] J. Sand and F. Singer, *Ber.*, **35**, 3172 (1902).

[23] I. M. Kolthoff and C. Barnum, THIS JOURNAL, **62**, 3061 (1940).

CHICAGO, ILL. RECEIVED DECEMBER 20, 1950

[CONTRIBUTION FROM THE RADIATION LABORATORY AND THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA]

The Preparation and Some Properties of Americium Metal[1]

BY EDGAR F. WESTRUM, JR.,[2a] AND LEROY EYRING[2b]

Americium trifluoride prepared by hydrofluorination of the dioxide was reduced to metal on the 40 to 200 microgram scale by reduction with barium metal in a high vacuum micro-furnace at 1100° in various refractory materials. High yields of silvery, very malleable and ductile, metal globules were obtained. Reaction with hydrogen to form a hydride and measurement of hydrogen evolution upon dissolution in acid were further proof of the metallic state. The density was determined as 11.7 ± 0.3 g. $cm.^{-3}$. Its low value compared to that of its preceding elementary neighbors is in striking resemblance to the behavior of its lanthanide analog, europium. The heat of solution of the metal in 1.5 molar aqueous HCl was determined as −160 ± 4 kcal. $mole^{-1}$.

Seaborg, James and Morgan discovered americium in plutonium irradiated in the nuclear fueled reactor.[3-5] In a recent note[6] it is indicated that americium formed by the reaction

$$Pu^{239}(n,\gamma)Pu^{240}(n,\gamma)Pu^{241} \xrightarrow{\beta} Am^{241}$$

may be produced in milligram amounts by intense radiation of large quantities of plutonium. The isolation of americium in a relatively pure state was first achieved by Cunningham who obtained a few micrograms of 99% pure material.[7] The solution chemistry has been studied by Cunningham and others, and the work of Fried[8] at the Argonne National Laboratory has indicated marked formal analogies of the simple crystalline compounds to those of the rare earths as well as isomorphism with some of those of the transuranium elements.

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[1] This research was performed under the auspices of the U. S. Atomic Energy Commission and was reported in AEC Documents MB-IP-96, (July 1946), MB-IP-97 (August 1946), MB-IP-122 (Oct. 1946), MB-IP-165 (Jan. 1947), MB-IP-170 (Feb. 1947). Presented at the 118th National Meeting of the American Chemical Society at Chicago, Sept. 5, 1950.

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