[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF DUKE UNIVERSITY]

Weston Standard Cells with Buffered Electrolytes[1]

BY RUDOLF J. PRIEPKE AND WARREN C. VOSBURGH

Lack of constancy of electromotive force of the Weston standard cell has been considered to be connected with hydrolysis of the mercurous sulfate.[2] It has been suggested also that the hysteresis often observed after a temperature decrease may sometimes be connected with the presence of basic mercurous sulfate.[3] The fact that the addition of sulfuric acid to the electrolyte decreases or eliminates hysteresis and favors constancy of electromotive force[4] is in harmony with this conclusion.

However, there is a lack of agreement as to how much acid is desirable and even as to whether or not any should be added. Shaw, Reilley and Clark[5] prepared the electrolytes for their acid cells by saturating 0.05 molar sulfuric acid solution with hydrated cadmium sulfate.[6] They reported that more of the acid cells were rejected for erratic behavior than of the neutral cells.

Von Steinwehr[7] considers that such a concentration of acid is objectionable. The acid would be expected to react with the cadmium of the amalgam, resulting in change of acidity and electromotive force. Von Steinwehr has not taken into account the hydrogen overvoltage at a mercury surface.

Vosburgh[4] found that the presence of as little as 0.004 mole of sulfuric acid per liter of saturated cadmium sulfate solution gave cells that were more reproducible and less variable than cells made without acid. Larger quantities of acid made a further small improvement, especially in hysteresis, and a concentration of 0.01 molar was quite satisfactory.

Hulett[2] seems to question the advisability of the addition of acid, but he and his co-workers obtained good results with the use of "equilibrium water," a solution prepared by digesting mercurous sulfate with water, as the solvent for the cadmium sulfate.

Shaw and Reilley[8] obtained good results when they added a little basic cadmium sulfate to the solution. In view of the above-mentioned opinions as to the bad effects of hydrolysis of the mercurous sulfate, this is surprising. However, Vosburgh and Elmore[9] found that cells in which an excess of basic cadmium sulfate was present were more nearly constant

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(1) A thesis presented by Rudolf J. Priepke in partial fulfilment of the requirements for the degree of Doctor of Philosophy in the Graduate School of Arts and Sciences of Duke University.

(2) Hulett, *Trans. Am. Electrochem. Soc.*, **58**, 303 (1930).

(3) Vosburgh and Elmore, THIS JOURNAL, **53**, 2831 (1931).

(4) Vosburgh, *ibid.*, **47**, 1262 (1925).

(5) Shaw, Reilley and Clark, *Trans. Roy. Soc.* (London), **A229**, 125 (1930).

(6) See Shaw and Reilley, *Can. J. Research*, **3**, 486 (1930).

(7) Von Steinwehr, *Z. Instrumentenk.*, **51**, 530 (1931).

(8) Shaw and Reilley, *Proc. Roy. Soc. Canada*, **13**, 171 (1919).

(9) Ref. 3, p. 2826.

in electromotive force and had much less hysteresis than neutral cells to which no basic or acidic material was added.

One difficulty in the use of very small acid concentrations is the difficulty of keeping them constant, except perhaps at the transition point for basic and normal mercurous sulfates, with both forms present. In order to study the effect of acidity of the electrolyte, a series of cells with buffered electrolytes was prepared.

Materials

Normal Mercurous Sulfate.—A 1 molar solution of recrystallized mercurous nitrate 0.1 molar in nitric acid was added slowly, with stirring, to a large excess of 3 molar sulfuric acid. The precipitate was digested hot for six hours under two portions of 1 molar sulfuric acid.

Basic Mercurous Sulfate.—A 0.4 molar mercurous nitrate solution 0.1 molar in nitric acid was added slowly to a 0.3 molar solution of sodium sulfate in 0.01 molar acetic acid. Sodium bicarbonate solution just sufficient to neutralize the nitric acid was added at the same time. The precipitate was white at first, but turned to greenish yellow in a few minutes. It was washed with 0.001 molar sulfuric acid solution and digested for ten hours under two portions of the 0.001 molar acid. A second similar preparation, undigested, was analyzed.

Anal. Calcd. for $Hg_2(OH)_2 \cdot Hg_2SO_4$: SO_4, 10.30. Calcd. for Hg_2SO_4: SO_4, 19.31. Found: SO_4, 17.20, 17.12.

The first preparation must have contained less of the normal salt than is indicated by the analysis of the second, because it was digested under a solution dilute enough in sulfuric acid to hydrolyze normal mercurous sulfate.

A third preparation was made like the first except that carbon dioxide was bubbled through the sodium sulfate solution while the two reagents were being added. The precipitate was bright yellow and after standing appeared distinctly orange. Later it changed to pale yellow. It was washed twice with 0.001 molar sulfuric acid solution and digested three hours under a third portion of the acid. After this treatment the crystals appeared under the microscope to be cubic in shape whereas before digestion they had looked like needles. They were stored under water to which had been added 0.5 cc. of 1 molar sulfuric acid solution and 1 cc. of 6% sulfurous acid solution per liter, the latter to reduce mercuric ion.

Anal. Calcd. for $Hg_2(OH)_2 \cdot Hg_2SO_4$: SO_4, 10.30; Hg, 86.06. Found: SO_4, 11.55 11.51, 11.27; Hg, 86.96, 86.36, 86.48, 86.07.

Other Materials.—Mercury was purified by washing and distillation. Cadmium amalgam, 12.5% cadmium, was prepared electrolytically. Cadmium acetate was recrystallized from 90% alcohol. Acetic acid was distilled from chromic acid.[10]

Preparation of the Cells

Buffer solutions were prepared by mixing 0.05 molar cadmium acetate and 0.1 molar acetic acid solutions in the desired proportions. The P_H values of three of these buffer solutions were measured by means of a hydrogen electrode. Table I gives the compositions of the buffer solutions. These buffer solutions were saturated with hydrated cadmium sulfate to prepare the electrolyte solutions for the cells.

The cells were prepared essentially as described by Wolff and Waters.[11] Air trapped in the vessels by the mercury and the amalgam was removed by evacuation. The tops

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[10] Orton and Bradfield, *J. Chem. Soc.*, **125**, 960 (1924).

[11] Wolff and Waters, *Bur. Standards, Bull.*, **4**, 30 (1907).

TABLE I

COMPOSITION OF BUFFER SOLUTIONS

Buffer no.	1	2	3	4	5	6	7	8	9
$Cd(C_2H_3O_2)_2$, 0.05 molar, cc.	0	33.3	36	40	44	50	55.5	75	41
$HC_2H_3O_2$, 0.1 molar, cc.	100	66.6	64	60	56	50	44.5	25	59
P_H		4.1				4.4		4.8	

of the vessels were flushed out with nitrogen before sealing. The mercurous sulfate was exposed to air during washing, but care was taken to prevent any prolonged exposure. The mercurous sulfate was washed six times with 5-cc. portions of the electrolyte by placing it in a glass-stoppered bottle and rotating the bottle mechanically, after which the wash solution was decanted.

Electromotive Force Measurements.—The cells were kept in an oil-bath at 25 ± 0.02°. When the temperature of the room was favorable, the temperature did not fluctuate more than 0.005°. A second bath at 35 ± 0.02° was used in hysteresis tests

The electromotive forces were measured by means of a Leeds and Northrup Type K potentiometer. Saturated Weston Cells 353–6 and 496 were used as reference standards.[12] Errors in cell comparisons were probably not larger than 0.01 mv.[13] Hysteresis tests were made as described by Vosburgh and Eppley.[14] The cells were made in duplicate, and the mean values of the electromotive force are given in the tables.

Experimental Results

The behavior of buffered cells made with normal mercurous sulfate is shown in Tables II and III and that of similar unbuffered cells in Table IV. Cells made with Buffers 1 to 5 compared favorably with acid cells in constancy and reproducibility.[4,12b] Only two of the cells changed in electromotive force more than 0.01 mv. after the first month, neglecting some measurements at 30°, which after calculating to 25° differed by 0.02 mv. from some of the others.

TABLE II

ELECTROMOTIVE FORCES OF BUFFERED CELLS AT 25°

		Electromotive force minus 1018.00 mv.						
Cells	Buffer	Initial mv.	1 week	1 month	2	4	6–7	11–16
659–660	1	−0.005	0.00	0.005	....	0.00^a	0.005^a	0.00^a
677–680	2	.025^a	.01^a	− .01^a	0.00^a	.00	.00	− .01^a
689–690	4	− .03	.02	.005	− .005	.00	− .02^a	....
691–692	5	.005	.01	− .01	.00	− .005	− .02^a	....
661–662	6	.06	.05	.04	....	.005^a	.00	− .02^a
697–698	7	.025	.01	.01	.01	.01	− .01^a	....
665–666	8	.00^a	.00^a	− .005^a	.00^a	.005	.005	− .01^a

^{a} Calculated from measurements at 30°.

Two determinations, four months apart, were made of the hysteresis of these cells, and the results are given in Table III. The cells made with the most acid buffers, Nos. 1 and 2, and those made with the most basic buffer, No. 8, showed little hysteresis in either test. The hysteresis of the other groups increased noticeably with age.

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(12) Vosburgh, (a) Ref. 4, p. 1257, (b) THIS JOURNAL, **49**, 85 (1927).

(13) See Ref. 3, p. 2822.

(14) Vosburgh and Eppley, THIS JOURNAL, **46**, 109 (1924).

TABLE III

HYSTERESIS OF BUFFERED CELLS

Cells	Buffer	Hysteresis I Age, mo.	Hysteresis I 1 hr. mv.	Hysteresis I 6 hr. mv.	Hysteresis II Age, mo.	Hysteresis II 1 hr. mv.	Hysteresis II 6 hr. mv.
659-660	1	8	−0.005	0.015	12	−0.01	0.01
677-680	2	3	.02	.02	7	0.02	.025
689-690	4	1	.03	.04	5	.06	.06
691-662	5	1	.025	.025	5	.06	.05
661-662	6	7	.20	.15	11	.25	.20
697-698	7	4	.05	.03	..	..	...
665-666	8	4	.01	.025	8	.03	.03

In order to have a good basis for comparison of the buffered cells with neutral or acid cells, some of the latter types were prepared, as shown in Table IV. The acid cells served also as duplicates of the standards.

TABLE IV

UNBUFFERED CELLS

Cells	Acid concn. molar	E. m. f. minus 1018.00 mv., 25° 1 week, mv.	1 month, mv.	4-5 mv.	6-8 mv.	Hysteresis Age. mo.	1 hr. mv.	6 mv.
711-712	None	0.085	−0.01	−0.20[a]	...	1	0.75	0.70
713-714	None	.28	.08	− .04	...	0.5	.30	.27
657-658	0.01	.04	.05	.05[a]	0.05	8	.02	.03
707-708	.01	.05	.05	.05	.05	2	.02	.01
663-664	.02	− .06	− .015	.03[a]	.04	7	.005	.005

[a] Calculated from measurements at 30°.

The two pairs of neutral cells[15] were respectively one month and half a month old at the time of the hysteresis test, but they showed more hysteresis than any of the buffered cells when the latter were five to eight months old. The acid cells showed as little hysteresis as the best of the buffered cells.

The lack of constancy of the neutral cells is not surprising and was probably not accidental.[16] To set up neutral cells that are constant in electromotive force requires special conditions that were not maintained for these cells. The washing of the mercurous sulfate was doubtless excessive[17] and the mercurous sulfate itself was large grained. Wolff[18] observed that most of his cells that showed hysteresis were made with large grained, white mercurous sulfate. The neutral cells are comparable with the buffered cells, which were made by the same procedure and, excepting the buffer, with the same materials.

To compare the hydrogen-ion activities of the buffered cadmium sulfate solutions with those of neutral solutions and of solutions acidified with

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(15) Cells to which no acid or basic material was added are designated as neutral cells.

(16) Compare with the results of (a) Gardiner and Hulett, *Trans. Am. Electrochem. Soc.*, **56**, 135 (1929); (b) Vosburgh and Elmore, Ref. 3; (c) Elliott and Hulett, *J. Phys. Chem.*, **36**, 2085 (1932); (d) Hagar and Hulett, *ibid.*, **36**, 2098 (1932).

(17) See footnote 20 and the text to which it refers.

(18) Wolff, *Bur. Standards Bull.*, **5**, 337 (1908).

sulfuric acid, some indicator experiments were made. A solution made by saturating 0.0028 molar sulfuric acid solution with cadmium sulfate had a larger acidity than the solution made by saturating 0.1 molar acetic acid with cadmium sulfate. Neutral cadmium sulfate solution had a somewhat smaller acidity than the solution made by saturating buffer 8 with cadmium sulfate. Thus, the buffered cells lay between the neutral cells and the acid cells with respect to the acidity of the solution from which they were prepared. However, in the case of the neutral solution, only a little hydrolysis of the mercurous sulfate within the cell would bring the hydrogen-ion activity within the range of the buffers.

It was of interest to inpuire as to whether or not any of the buffered cadmium sulfate solutions were capable of hydrolyzing normal mercurous sulfate. For this purpose some buffered cells were made with basic mercurous sulfate instead of normal.

The electromotive force of such cells should depend on the sulfuric acid activity in the electrolyte, because the acid enters into the cell reaction. The sulfate-ion activity is qractically fixed, so the electromotive force should depend on the hydrogen-ion activity.[19]

At one particular hydrogen-ion activity, designated below as the transition point, basic and normal mercurous sulfates can both exist in equilibrium with the solution. With an electrolyte of this hydrogen-ion activity, a cell made with basic mercurous sulfate should have the same electromotive force as the normal cell. With a higher hydrogen-ion activity the electromotive force should be higher and with a lower one it should be lower.

Cells containing basic mercurous sulfate were made by the same procedure as cells with the normal salt, and with the same buffer solutions. The electromotive forces at different ages are given in Table V. The values

TABLE V

ELECTROMOTIVE FORCES OF CELLS MADE WITH BASIC MERCUROUS SULFATE AT 25°

			Electromotive force minus 1018.0 mv.			
Cells	Prepn. no.	Buffer	Initial, mv.	10 days, mv.	100 mv.	200 mv.
673–674	1	2	2.9[a]	2.3[a]	2.1	1.9
685–686	1	3	2.4[a]	2.2[a]	2.0	1.7
681–682	1	4	1.3[a]	1.0[a]	0.6	0.3
715–716[b]	3	9	....	− 0.4[a]	0.3	....
687–688	1	5	− 0.6[a]	− .8[a]	− 1.1	− 2
669–670	1	6	− 5.3[a]	− 5.4[a]	− 6.0	− 6.3
701–702	3	7	− 8.3	− 5.2	− 7.6	....
667–668	1	8	−12.7[a]	−13.1[a]	−13.5	−13.9
703–704	3	8	−14.0	−21.4	−15.9	....

[a] Calculated from measurements at 30°. [b] The authors are indebted to Dr. Grady Tarbutton for the preparation of these cells.

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[19] Hulett, Ref. 2, p. 300, has overlooked this point in giving the electromotive force of such a cell. The most obvious reason for the decrease in electromotive force of his basic cell is a decrease in the hydrogen-ion activity.

at the even intervals were obtained from curves expressing the change of electromotive force with time.

The electromotive forces of the cells of Table V are somewhat uncertain because they were not constant. The variation, however, was smaller than the differences between groups. The electromotive force decreased in the different groups with decrease in hydrogen ion activity. The first three groups had higher electromotive forces than the normal, and the last five groups lower ones. This indicates that the transition point is in the vicinity of the hydrogen-ion activity of buffer 9.

If the first three groups represent an unstable condition, that is, basic mercurous sulfate in contact with a solution capable of converting it into the normal salt, the variation in electromotive force is understandable. It may represent an approach toward the lower electromotive force of the normal cell. In this case the initial values of the electromotive force are the most reliable.

The decrease in the other cells is less easily understood. It may have been connected with the normal mercurous sulfate that must have been present as an impurity.

All the cells of Table V showed hysteresis after a temperature decrease.

Several cells were made using a mixture of about one-third basic mercurous sulfate and two-thirds normal salt. Such cells made with buffers 2 and 4 showed a high electromotive force, but not as high as for cells with basic salt only and they showed some hysteresis. Cells made with buffer 6 had the normal electromotive force and showed very little hysteresis. The lack of hysteresis in the latter case is in contrast to the behavior of the similar cells made with normal mercurous sulfate only, which showed the largest hysteresis of any of the buffered cells. Table VI shows the electromotive forces at 25° and Table VII the hysteresis of these mixture cells.

TABLE VI

ELECTROMOTIVE FORCES OF MIXTURE CELLS

		E. m. f.$_{25}$ − 1018.00 mv.					
Cells	Buffer	Initial mv.	1 wk., mv.	1 mo., mv.	2 mv.	4 mv.	6 mv.
675–676	2	0.15[a]	0.08[a]	0.09[a]	0.10[a]	0.12	0.13
683–684	4	.28[a]	.22[a]	.20[a]	.20[a]	.20	.17
671–672	6	−.02[a]	− .01[a]	− .01[a]	.01[a]	.00	.00

[a] Calculated from measurements at 30°.

TABLE VII

HYSTERESIS OF MIXTURE CELLS

		Hysteresis I			Hysteresis II		
Cells	Buffer	Age, mo.	1 hr. mv.	6 hr. mv.	Age, mo.	1 hr. mv.	6 hr. mv.
675–676	2	3	0.07	0.06	7	0.04	0.04
683–684	4	3	.12	.09	7	.19	.15
671–672	6	3	.03	.02	7	.01	.01

That cells made with normal mercurous sulfate and with a buffer that is capable of hydrolyzing the normal salt are constant in electromotive force, at least for a period of several months, is in agreement with the results of Vosburgh and Elmore,[3] and also with the method of setting up cells of Shaw and Reilley.[8] An explanation can be based on the assumption that the rate of diffusion of acid from the mercurous sulfate paste is very slow. This assumption is in harmony with the facts as to the long life of Weston Cells;[5] diffusion of the mercurous sulfate to the amalgam electrode is very slow. If a cell is made with an electrolyte solution capable of hydrolyzing mercurous sulfate, a little hydrolysis must take place to bring the concentration of acid in the vicinity of the mercurous sulfate paste to the equilibrium value. After that, a very slow hydrolysis will balance diffusion and keep the concentration at the equilibrium value.

If the assumption concerning the slowness of diffusion is correct, cells made in such a way as to favor a more rapid diffusion might be different from those in which the diffusion is slow, provided the acidity of the electrolyte is below the transition point. If the rate of diffusion became large enough to be appreciable as compared to the rate of hydrolysis, the local excess of acid in the vicinity of the mercurous sulfate paste would decrease. With a decreased hydrogen-ion activity, and in the presence of some basic mercurous sulfate, the mercurous-ion activity could not be maintained as high as before, and the electromotive force would decrease. The electromotive force would depend on a steady state of change rather than equilibrium and would not be very reproducible. Hulett[2] found that cells with a thin layer of mercurous sulfate showed marked irregularities and tended to decrease in electromotive force below the normal. The experiments described below show that this is the case when the hydrogen-ion concentration is below the transition point, but above that point easy diffusion has much less effect.

Four cells were made with a layer of normal mercurous sulfate paste about 1 to 2 mm. thick. Only large crystals of cadmium sulfate were placed in the mercury leg of the cell. Two of the cells were made with a buffer above the transition point. These had about the same electromotive force for two months as similar cells made according to the standard procedure. They decreased a little after that. The hysteresis was considerably larger than for cells with more mercurous sulfate. The other two cells were made with the most basic buffer. They had a low and continuously decreasing electromotive force and the hysteresis was very large.

Comparison at 35° of the cells of Table VIII with those of Table II indicated that cells 705–706 were below the transition point at 35°, though not at 25°. The experiments do not show, therefore, whether or not the hysteresis of an acid cell would be increased by making the mercurous sulfate layer thin.

TABLE VIII

CELLS WITH A THIN LAYER OF MERCUROUS SULFATE

		E. m. f. − 1018.00 mv., 25°				Hysteresis[a]	
Cells	Buffer	Initial, mv.	1 mo., mv.	2 mv.	6 mv.	1 hr., mv.	6 hr., mv.
705–706	4	0.05	−0.005	−0.01	−0.03[b]	0.21	0.16
709–710	8	− .03	− .12	− .17	− .33[b]	.71	.38

[a] The age was two months at the time of the hysteresis test. [b] Calculated from measurements at 30°.

Discussion

The assumption that the diffusion of acid away from the portion of the mercurous sulfate paste in the vicinity of the mercury surface is very slow can account for certain features of the observed behavior of Weston cells. When basic and normal mercurous sulfates are both present and the hydrogen-ion concentration is below the transition point, as in cells 671–672 when first made (Tables VI and VII), the normal mercurous sulfate must tend to hydrolyze. This liberates sulfuric acid and tends to bring the acidity to the transition point. If the diffusion of acid is very slow, the hydrogen-ion concentration rises to the transition point, and the cell then has the normal electromotive force. However, if diffusion is appreciable compared with the rate of hydrolysis, the hydrogen-ion concentration is lower than the transition point and the cell tends to act as if made with basic mercurous sulfate. The electromotive force depends on the hydrogen-ion concentration and is lower than the normal, as in cells 709–710, Table VIII.

Thus, cells made with solutions below the transition point in acidity can have the normal electromotive force. However, as Hulett[2] has pointed out, continued hydrolysis would cover the mercurous sulfate crystals with a crust of basic salt, reducing the rate of hydrolysis. The effect of continued hydrolysis at a slow rate probably shows itself in neutral cells that have a low and continuously decreasing electromotive force. Cells with decreasing electromotive force seem to result when the mercurous sulfate paste is washed too thoroughly with a neutral cadmium sulfate solution, thus causing appreciable hydrolysis before the cells are set up.[20] On the other hand, cells in which the mercurous sulfate paste contains considerable basic salt that is present only in independent crystals show no such effect, at the beginning of their life, at least.

The basic cells of Vosburgh and Elmore[3] had low and not very reproducible electromotive forces. In this case, while the layer of mercurous sulfate was thick, the acid concentration gradient from the mercurous sulfate paste to the solution above the paste saturated with basic cadmium sulfate must have been large. This would favor a more rapid diffusion of the acid. Why the electromotive force did not decrease as much as that of neutral

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(20) This was pointed out to one of the authors by Dr. Marion Eppley several years ago. Though definite proof seems to be lacking it seems in accord with experience. See, for example, Ref. 3, p. 2827, and compare with Eppley, *Trans. Am. Electrochem. Soc.*, **53**, 149 (1928). Gardiner and Hulett, Ref. 16a, probably washed their mercurous sulfate too well.

cells not containing the basic cadmium sulfate seems to be explainable only on the assumption that independent crystals of the basic mercurous sulfate were formed in the basic cells and the surfaces of the normal crystals were not covered over, while the opposite was the case in the neutral cells.

Summary

Weston standard cells were prepared with buffered electrolytes of varying acidities. The electromotive forces were practically constant over the period of observation, six to sixteen months. Hysteresis was very small in the most acidic and the least acidic groups.

A similar set of cells made with basic mercurous sulfate instead of normal varied in electromotive force with acidity and made it possible to estimate roughly at what acidity basic and normal mercurous sulfates can exist in equilibrium with the same saturated cadmium sulfate solution. It was shown that some of the buffered cells made with normal mercurous sulfate had acidities above this transition point and some below it.

Cells made with a mixture of basic and normal mercurous sulfates at the mercury electrode had high electromotive forces if the acidity was above the transition point and the normal electromotive force if it was below.

When the layer of mercurous sulfate was unusually thin, 1 to 2 mm. in thickness, the cells were quite variable if the acidity was below the transition point, but not if it was above.

An explanation is proposed for the slow decrease in electromotive force often observed in neutral cells.

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RECEIVED SEPTEMBER 14, 1932
PUBLISHED MAY 6, 1933

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF HARVARD UNIVERSITY]

Equilibria, Complex Ions and Electrometric Titrations. I. Iodine or Bromine in Hydrochloric Acid[1]

BY J. HORACE FAULL, JR., AND GEORGE SHANNON FORBES

Two earlier papers[2,3] from this Laboratory have described, and analyzed mathematically the formation, in four and six normal hydrochloric acid, of the iodine chlorides and of bromine monochloride. Unexpectedly, also, it was found that a fifth or more of the bromine added to such acids oxidized chloride ion because the resulting chlorine was firmly bound as bromine monochloride. Such effects cannot be neglected in a precise study of electrometric titrations of mixed halogens or halides. Incidentally, the great stability of the compounds studied suggested the pos-

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[1] Complete data and calculations are available in the doctoral thesis of J. Horace Faull, Jr., Widener Library, Cambridge, Mass.

[2] Forbes, Glass and Fuoss, THIS JOURNAL, **47**, 2892 (1925).

[3] Forbes and Fuoss, *ibid.*, **49**, 142 (1927).