MAX LE BLANC'S STUDIES ON ELECTROLYTIC POLARIZATION

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In 1800, William Nicholson (1753-1815) and Anthony Carlisle (1768-1840) showed that water can be decomposed into its elements by the passage of an electric current (1). This discovery naturally encouraged similar experiments. For example, $CuSO_4$ solution was found to deposit copper on a platinum cathode; iodine was liberated at the anode when KI solution was electrolyzed. A most striking example was the liberation of elemental potassium or sodium by the electrolysis of the slightly moist hydroxides (2).

The first "battery" was the Volta "pile", described in 1800 (3). This and its immediate successors were based on the cell system copper (or silver)/dilute electrolyte/zinc. Dilute H_2SO_4 was the usual electrolyte. Such batteries became *polarized* when called upon to supply more than a little current. The emf dropped and gas (hydrogen) accumulated on, or rose from, the copper electrodes.

Although these facts of electrolysis are simple, their interpretation raised many questions. On the assumption that the decomposition of water is a *primary* electrolytic process, is the deposition of copper also primary, or does hydrogen, as the primary product, react with $CuSO_4$ to give copper as a *secondary* product? Although Michael Faraday (1791-1867) had introduced terms such as *electrolyte, anion,* and *cation* in 1834 (4), the "ionic theory" of Svante August Arrhenius (1859-1927), postulating that electrolytes are ionized, often extensively, in solution, was not published until 1887 (5). Incidentally, Faraday was convinced that the electrolytic deposition of metals from aqueous solution was a secondary process. He therefore turned to the much
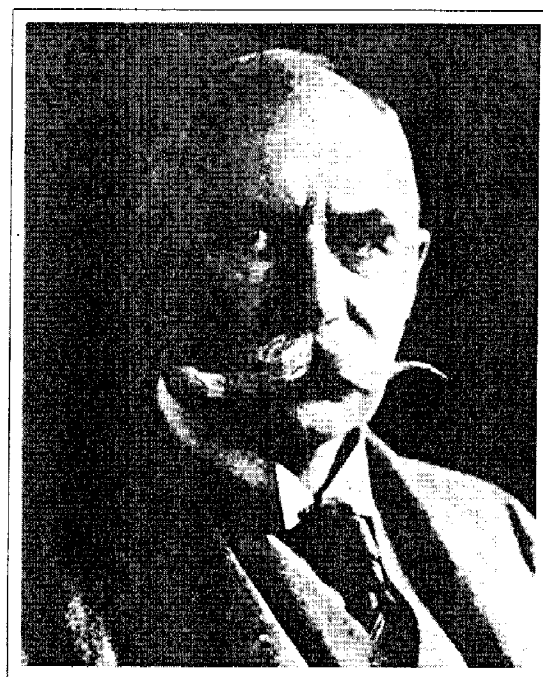

Figure 1. Max Le Blanc (1865-1943)

more difficult area of quantitative fused-salt electrolysis as a basis for the enunciation of the second law of electrolysis.

Commonly, a certain minimum applied emf is needed to produce visible electrolysis. Under comparable conditions, do electrolytes of the same type (*e.g.*, strong acids) require similar minima for electrolysis? In an electrolysis, reactions occur both at the anode and at the cathode. What are the individual contributions of potential that make up this minimum applied emf? Is

there more to the phenomenon of polarization than the mere formation of films of gas on the electrodes? The path to the answers to such questions was by no means simple and direct. It was Max Julius Louis Le Blanc (1865-1943) who surveyed the entire area and placed it on a firm experimental and theoretical foundation.

Le Blanc (Fig. 1) was born on May 26, 1865, in Barten, East Prussia. He studied in Tübingen, Münich, and then Berlin, where he became assistant to August Wilhelm Hofmann (1818-1892). He obtained his doctorate in organic chemistry under Hofmann's direction in 1888 (6). Le Blanc was not impressed by this branch of chemistry. In fact, he even thought of leaving chemistry for geology (7). However, he encountered, and was captivated by, the physical chemical writings of Wilhelm Ostwald (1853-1932). Le Blanc moved to Leipzig in
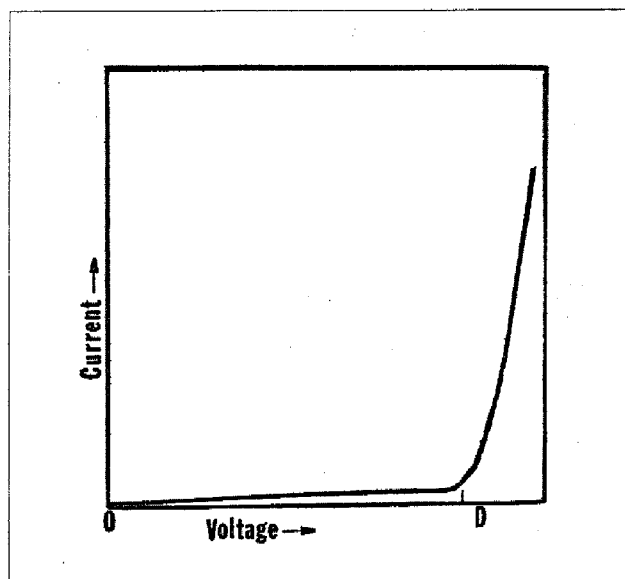

Figure 2. Current-voltage curve showing "decomposition point"

1890 and became assistant to Ostwald. Here Le Blanc began to develop his life work in physical chemistry, especially electrochemistry.

It is interesting to contrast Le Blanc's career with that of Robert Behrend (1856-1926), a colleague at Leipzig (8). After one paper and his dissertation, Le Blanc left organic chemistry. Behrend, after publishing one paper (outstanding, because it described the first potentiometric titrations) on electrochemistry (9), then spent the rest of his career as an organic chemist.

Polarization concerns phenomena occurring at the boundaries between the electrode surfaces and the electrolyte when a current passes through them. Although

Ostwald knew that the term "polarization" had been used since the mid 1830's, he could not trace its origin (10). Nowadays, an electrode is said to be polarized when it adopts a potential impressed upon it with little or no change in the current passing through it. Our understanding of polarization can be traced to the pioneering work of Le Blanc.

Suppose that an increasing emf is applied to a pair of small platinum electrodes immersed in dilute H_2SO_4 and the current that passes through the solution is measured. The resulting current-voltage curve is shown diagrammatically in Fig. 2. In the voltage region 0D, almost complete polarization of one or both of the electrodes occurs. In this region, only a small *residual current* is observed (11). Beyond D, the *decomposition point*, the current increases rapidly with further increase in applied emf. The "point" is really a narrow voltage region; the observer merely looked for a sudden jump in current.

Early workers attributed polarization to the formation of a poorly conducting layer, such as a film of gas, on the electrodes. By the early 1840's, it was realized that a more general effect was "galvanic polarization", *i.e.*, the generation of an emf that opposed the emf applied to the system. Various investigators examined the effects of applied emf and of the nature of the electrodes and electrolyte. Because of differing methodologies and interpretations, individual reports on similar systems often differed widely (12).

In 1851, William Thomson (1824-1907), later Lord Kelvin, proposed his "mechanical theory of electrolysis (13). This theory implied that, if the heats of reaction of the substances involved in an electrochemical system were known, then the "intensity" (emf) of the system could be calculated. In fact, Thomson made use of the data of James Prescott Joule (1818-1889) to calculate the "intensity" of the Daniell cell. The conclusions of some later workers tended to support Thomson's theory. However, the investigations of (Karl) Ferdinand Braun (1850-1918) cast doubt on this theory and then refuted it (14). The agreement between the measured "intensity" of the Daniell cell and the value calculated from the heats of reaction was a coincidence, because the emf of this cell has only a very small temperature coefficient (15).

Le Blanc's first paper on polarization began with a discussion of the work of Braun and others, showing that the values of polarization maxima cannot be calculated from the heats of reaction of the chemical processes involved (16). Le Blanc then referred to a paper by Marcellin Berthelot (1827-1907), who retained the

idea of the total convertibility of chemical
energy into electrical energy (17). As po-
larizing source, Berthelot used a battery
of cells of differing voltages to find a com-
bination that reached the "decomposition
point" (i.e., the polarization maximum),
taken as the first appearance of gas bubbles
on the electrodes. From a study of the elec-
trolysis of K_2SO_4 solution, Berthelot con-
cluded that water was decomposed and the
salt was separated into potash (KOH) and
H_2SO_4. Le Blanc declared that these con-
clusions were inadmissible. Having the
great advantage of working in the Leipzig
laboratories, he was exposed to the
Arrhenius ionic theory and to the concepts
of Hermann Walther Nernst (1864-1941)
(18, 19), which are the basis of the famil-
iar "Nernst equation".

Figure 4. Diagrammatic representation of DV arrangement

The appearance of gas bubbles as an indication of
the polarization maximum is open to an obvious objec-
tion. Supersaturation may be needed before actual bubble
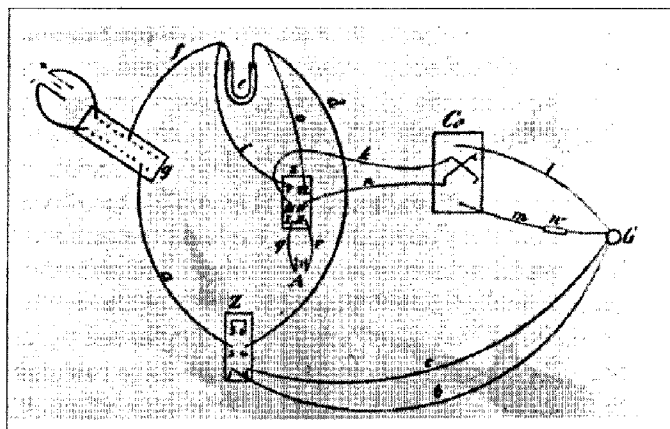formation begins. The effect is analogous to superheat-

Figure 3. Le Blanc's decomposition value arrangement

ing, which may give rise to "bumping" when a liquid is
boiled. Le Blanc therefore decided to locate the "de-
composition value" as that at which a small increase in
applied emf gave a sharp rise in current.

Figure 3, reproduced from Le Blanc's paper, shows
his experimental arrangement. The sketch, not easy to
follow, has been redrawn diagrammatically and
relettered, as shown in Fig. 4. The following descrip-
tion refers to this latter figure.

The polarizing emf E was selected by potential di-
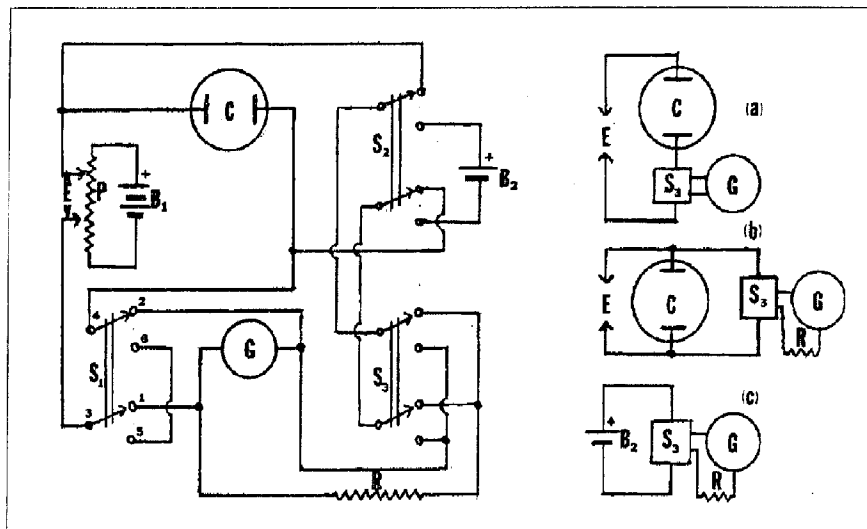vider P, supplied by battery B_1. Le Blanc used as poten-

tial divider a 1000-W resistance box, into which take-
off plugs were suitably inserted. He was thus able to
vary E in steps of 0.02 to 0.03 V. With switches S_1, S_2,
and S_3 in the positions shown, the effective circuit is
that of (a). The polarizing emf was then stepped up until
the deflection of galvanometer G jumped at the decom-
position value (DV). Switch S_3, which reversed the cur-
rent direction through G, was not operated at this stage.

Switch S_1 was then shifted, yielding the effective
circuit (b), so that G now measured emf E. The 70,000-
W resistor R limited the current through G. The deflec-
tion of G was noted, and effectively doubled by shifting
S_3. Finally, S_2 was shifted to yield the effective circuit
(c) and the deflection of G produced by the standard
Clark cell B_2 was observed. Because the deflections were
small, their ratio could be taken as the ratio $E:B_2$.

Le Blanc's electrolysis cell was a 7-cc capacity U-
tube, mounted in a waterbath held at 20° C. Each 6-mm
long, 0.7-mm diameter platinum wire electrode was
sealed into the end of a glass tube. Unless otherwise
specified, 1N solutions were used. Le Blanc found that,
with some electrolytes, the DV was not easy to locate;
sometimes he made 8 to 10 runs. His first set of experi-
ments showed that, for a given anion, the DVs of so-
dium and potassium salts were equal.

Table 1 lists the DVs of some of the acids and bases
examined by Le Blanc. Except for the halide acids, the
values clustered around 1.7 V. This suggested a com-
mon electrolytic process, the decomposition of water.
In acid solutions, hydrogen obviously came from the
discharge of the sole cation. Analogously, discharge and
breakdown of OH⁻, the sole anion in the basic solutions,
was the source of oxygen. Hydrogen from these solu-

Table 1
Decomposition Values (DVs) of Acids and Bases

Acids	DV, V	Bases	DV, V
H_2SO_4	1.67	NaOH	1.69
HNO_3	1.69	KOH	1.67
H_3PO_4	1.70	NH_3	1.74
$HClO_4$	1.65	*$MeNH_2$	1.75
Tartaric	1.62	**Et_2NH	1.68
Hcl	1.31	***Me_4NHOH	1.74
HBr	0.94		
HI	0.52	*0.25N; **0.5N; ***0.125N	

tions *might* have arisen from cation discharge, followed by immediate reaction with water. Similarly, oxygen from acid solutions *might* have resulted from anion discharge, followed by immediate reaction with water. The obvious liberation of halogen from the halide acids, together with their lower DVs, indicated that processes other than the decomposition of water were involved. An alternative explanation for the similarity of results for both acid and alkaline solutions (excepting the halides) involved the assumption that the primary electrolytic process was the decomposition of water. Although the OH⁻ concentration in the acid is only about 10^{-14}, the discharge of this ion must occur at a potential lower than that needed to discharge the acid anion. Simi-

Table 2
Decomposition Values of Inorganic Salts and
Sodium Salts of Organic Acids (1.0N)

Inorganic Salts	DV, V	Sodium Salts	DV, V
Na_2SO_4	2.21	Acetate	2.10
K_2SO_4	2.20	Butyrate	2.05
$NaNO_3$	2.15	Malonate	2.05
KNO_3	2.17	Tartrate	1.98
$LiNO_3$	2.11	Succinate	2.06
$Ca(NO_3)2$	2.11	Benzoate	2.00
$Sr(NO_3)_2$	2.28	Monochloracetate	2.17
*$Ba(NO_3)_2$	2.25	Dichloracetate	2.23
NH_4NO_3	2.08	**Oxalate	1.13

*0.5N **Potassium Salt

larly, in alkaline solutions, hydrogen ion must discharge more readily than the base cation. Le Blanc found that the DVs of H_2SO_4, NaOH, etc., were essentially constant over a 60-fold concentration change. However, the DVs of HCl and HBr rose as the concentration was decreased.

The electrolysis of K_2SO_4 solution, like that of the parent acid and base, yields hydrogen and oxygen. If the fundamental process is the decomposition of water, the salt, acid, and base might be expected to have the same DVs; yet Le Blanc found that DVs for salts were significantly greater. Some typical values that he found are listed in Table 2. He recognized that the radicals of oxalic and some other organic acids might undergo anodic oxidation and thus give anomalous results. Le Blanc found that the DVs for the salts of sodium and potassium were not sharp. He recognized that, in these electrolyses, formation of alkali at the cathode and of acid at the anode introduced new unknowns. These salt solutions have negligible buffer capacity, so that even slight electrolysis could produce a hydrogen-ion concentration cell. The back emf of this would raise the DV above that of the constituent acid or base. To demonstrate his recognition of the localized formation of acid and base, Le Blanc measured the DVs of the system 1N NaOH/1N acid (the slash represents a paper or clay diaphragm). For H_2SO_4, $HClO_4$, H_3PO_4, and HNO_3, the values ranged from 2.41 to 2.46 V.

Having examined solutions of various salts that gave metal deposits on the cathode, Le Blanc concluded that metal deposition is a primary process. He noted that salts of lead, cobalt, and nickel gave dull-red "superoxide" deposits on the anode. In experiments with electrodes of zinc, cadmium, or silver in solutions of the corresponding salts, he observed that the slightest applied emf caused dissolution from one electrode and deposition of metal on the other.

When the electrolysis of a potassium salt is carried out at a mercury cathode, hydrogen does not appear immediately. Le Blanc attributed this well-known fact to amalgam formation, i.e., dissolution of potassium in the mercury, so that K⁺ is discharged more readily than the hydrogen ion. The interpretation of the fact gave rise to considerable controversy. [It was not until 1899 that it was definitely shown that discharge of hydrogen ion at a mercury cathode required a potential much more negative than at a platinum cathode (20)]. A mercury cathode thus exhibits a high *hydrogen overpotential*. Polarography, introduced much later by Jaroslav Heyrovsky (1890-1967), makes use of the high hydrogen overpotential of a dropping mercury cathode (21).
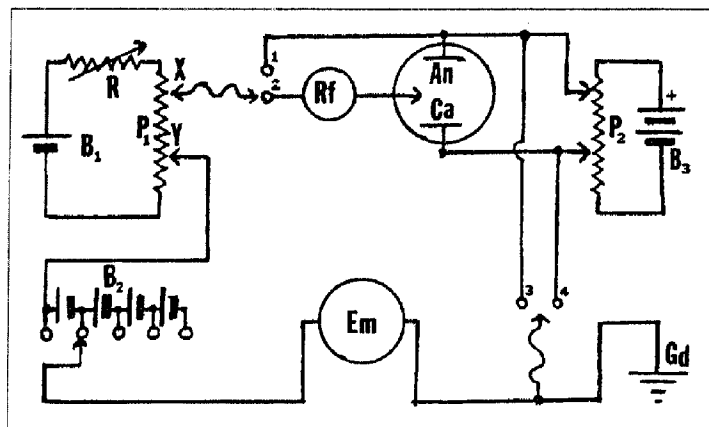
Figure 5. Diagram of arrangement for the measurement of individual electrode potentials.

Following the appearance of Le Blanc's paper (16), Arrhenius critically examined the electrolysis of solutions of alkali metal salts between a large platinum anode and a small mercury cathode (22). He measured the time between closure of the circuit and the first appearance of hydrogen bubbles. He noted that if the circuit was opened *before* this time had elapsed, pronounced hydrogen evolution occurred. He attributed this to the attack on water by the metal amalgam that had formed.

Arrhenius concluded that hydrogen was produced by the same mechanism when the circuit remained closed. This implied that the deposition of the alkali metal is a *primary* process, giving hydrogen as a secondary step. Arrhenius felt that the same mechanism held for a platinum cathode; here hydrogen evolution began rapidly because deposited alkali metal could not penetrate the solid electrode. Analogously, oxygen liberation was attributed to attack on water by the primarily-discharged acid anion.

Responding to a claim to have demonstrated the constancy of the minimum emf required to bring about the electrolysis of alkali salt solutions (23), Le Blanc summarized his results, pointing out his priority (24). This elicited comments from Berthelot (25), who accused Le Blanc of forgetting that his work agreed with that described by Berthelot in 1882 (17). Le Blanc replied vigorously (26), pointing out that his 1891 paper (16) acknowledged Berthelot's memoir. Subsequently, Berthelot had stated that, for the electrolysis of an alkali salt, the minimum emf was effectively governed by the sum of the heat absorbed in the separation of acid and base, and the heat of decomposition of water into hydrogen and oxygen. Berthelot described this as an *experimental* law, *independent of all theory* (25). Le Blanc proceeded to demonstrate that this "law" did not exist

(26)! Nevertheless, Berthelot persisted, stating that his own experimental results were "independent of theory, fundamental or illusory, to which a clever physicist, such as Le Blanc, perhaps would attribute an immoderate importance" (27).

Le Blanc's second paper on polarization appeared in 1893 (28). Now convinced that the electrolytic decomposition of water was a primary process, he stated his intention to deal with this and with the opposing view of Arrhenius. This was covered in a separate paper (29). Fully aware that the DVs reported in his first paper (16) were the joint contributions of the anodic and the cathodic potentials, Le Blanc planned the separate determination of these potentials.

Le Blanc's experimental arrangement is diagrammed in Figure 5 . Adjustable resistor R enabled Leclanché cell B_1 to produce 1.00 V across the measuring potential divider P_1. When needed, the Helmholtz cells (30) in battery B_2 provided additional 1-V steps. The polarity of the B_1—B_2 combination was set as required. The two Leclanché cells in battery B_3 and potential divider P_2 provided the adjustable polarizing emf.
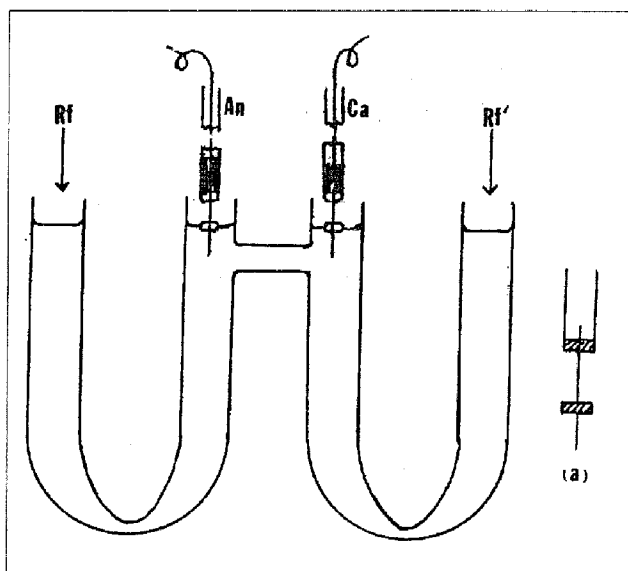

Figure 6. Le Blanc's electroysis cell

Capillary electrometer Em (30) was used for balance detection. Arrangements were such that contact X of P_1 could be connected to the anode, An, of the cell, to its cathode, Ca, or to the normal calomel electrode, Rf. Also, either An or Ca could be grounded.

The 9-cm long limbs of the electrolysis cell were narrowed at the bottom, as shown in Fig. 6. The aim was to prevent KCl in the reference electrode from reach-

ing the platinum electrodes. One of these is shown at (a); the glass bead on the wire is a guide for depth of immersion. The reference electrode was inserted at Rf for anode potential measurement, or at Rf' for cathode

Table 3
Cathode Potentials at the Decomposition Point*

Cation	1N Salt	Cathode potl., V	Electrode potl., V**
Cadmiun	$CdSO_4$	0.16)	0.19
"	$CdCl_2$	0.18	
Zinc	$ZnSO_4$	0.515	0.51
Cobalt	$CoSO_4$	0.17	–
Copper	$CuSO_4$	-0.56	-0.60
Silver	$AgNO_3$	-1.055	-1.01

*See text
**Metal in 1M metal salt solution; Ostwald (30a)

potential measurement. To measure a DV, contact X (Fig. 5) was connected to point 1, while point 4 was grounded. The DV set up by adjustment of P_2 was measured as the sum E at balance, of the voltage across XY and any supplied by B_2. X was then shifted to point 2, when the new value of E required to restore balance gave the cathode potential. To measure the anode potential, grounding was shifted from point 4 to point 3.

Le Blanc concentrated on the determination of cathode potentials, regarding these as the more important. Some of his findings are summarized in Table 3. Because of accompanying liberation of hydrogen, Le Blanc's results for zinc were at first unsatisfactory. By preliminary electrolysis at an applied emf of about 4.5 V, however, zinc was deposited on the cathode, satisfactory results then being obtained.

Le Blanc conducted experiments with other than platinum cathodes, e.g., $CdCl_2$ solution and a copper cathode, or $AgNO_3$ solution and a gold cathode. This led to the conclusion, *"The polarization value of the cathode at the decomposition point is equal to the potential that the metal concerned shows with respect to the liquid; it is independent of the electrode material"*.

The strange appearance of the data in the tables is partly due to the use of Ostwald's sign convention, which is opposite to that of present-day reduction potential. For example, Le Blanc's value for zinc is +0.515 V; the modern value is -0.763 V. The numerical discrepancy

arises largely from Le Blanc's acceptance of -0.560 V as the potential of his reference electrode (31b). The modern value of this electrode would be +0.280 V.

Le Blanc measured the potentials of both of the platinized platinum electrodes used in his study of the electrolysis of 1N H_2SO_4. He saturated the system with hydrogen and oxygen by a preliminary electrolysis to set up a "Gaskette" (gas chain, i.e., a hydrogen-oxygen cell). He reasoned that the combined electrode potentials of the "Gaskette" should be equal to the applied emf at the balance point, so that the current through the system should be zero. He increased the applied emf in steps, measuring the electrode potentials after each increment. He found that the anode potential increased with increasing applied emf, but that the cathode potential remained essentially constant. In fact, he stated that, with a given pressure of hydrogen, the cathode behaved in acid solution like a metal electrode immersed in a solution containing the metal ions. He is thus regarded as the originator of the *hydrogen electrode*, later to become important in the establishment of standard potentials and in the measurement of pH.

Le Blanc repeated experiments with solutions of $CdSO_4$ and of $ZnSO_4$, now measuring the potentials of both electrodes above and below the "gas chain" value. He found that essentially constant cathode potentials could be obtained after the metal deposit had coated the cathode. Without such coating, discharge of hydrogen must occur, because both metals are electronegative to hydrogen. However, the lack of buffer capacity causes rapid depletion of hydrogen ion in the immediate vicinity of the cathode, with development of alkaline conditions in this vicinity. Deposition of zinc, favored by higher current density, can then accompany the liberation of hydrogen, as both ions migrate or diffuse from the bulk of the solution. Local alkaline conditions could convert Zn^{2+} into the zincate anion; Le Blanc actually considered that "insoluble zinc hydroxide" might influence the cathode process, but regarded this as improbable.

Le Blanc remained in Leipzig until 1896 and then joined the firm of Höchster Farbwerke as director of electrochemistry. In 1901, already known for his researches, as well as for his text on electrochemistry (32), he was appointed Professor of Physical Chemistry in the Technische Hochschule, Karlsruhe. Le Blanc's strong liking for electrochemistry did not prevent the spread of his activities to other branches of chemistry. His numerous publications cover topics as diverse as problems concerning rubber, the preparation of chromium and its

compounds, and the X-ray study of mixed crystals. An excellent experimenter, Le Blanc insisted that his students should become well versed in practical work. He retired in 1933 and died in Leipzig on July 31, 1943.

ACKNOWLEDGMENT

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