X. On the Properties of Electro-deposited Antimony. By G. Gore, Esq. Communicated by Dr. Tyndall, F.R.S.

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- 1. While engaged in depositing antimony by the electro-process in October 1854, I observed a remarkable phenomenon of development of heat in the deposited metal when gently struck, and published a brief account of it in the Philosophical Magazine for January 1855; since that period I have investigated the phenomenon, and have now the honour of laying the results before the Royal Society.
- 2. In depositing this metal by the electro-process several solutions may be successfully used, consisting of compounds of the metal either with hydrochloric or tartaric acid; but those I have generally operated with have consisted,—1st, of ordinary chloride of antimony (as prepared for pharmaceutical purposes) nearly saturated with antimony, by suspending a plate of that metal in it as an anode, and passing an electric current from several pairs of zinc and silver batteries through it until a copious deposit of bright metal occurred, and until its yellow colour had nearly disappeared; and 2nd, of five parts of tartar-emetic and five parts of tartaric acid, dissolved in a mixture of two parts of hydrochloric acid and thirty parts of water; each solution being filtered before A most excellent solution in lieu of the first may quickly be formed by saturating ordinary chloride of antimony with tartar-emetic, using about three or four parts of the former to one of the latter; or by dissolving two parts of tartar-emetic in three parts of hydrochloric acid. I have also in place of that liquid occasionally used hydrochloric acid saturated with antimony by the battery process; and sometimes a liquid composed of seven parts of tartar-emetic dissolved in a mixture of eight parts of hydrochloric acid and four parts of water. Substituting pure acid, distilled water, and pure antimony, for those of the ordinary quality, made no material difference in the results. Each solution requires to be electrolysed a short time before it yields a good and uniform deposit.
- 3. There are two distinct kinds of antimony deposit produced in these liquids, which I shall distinguish by the terms amorphous and crystalline; dark and grey: for the special production of the former I use the first liquid, or better that composed of chloride of antimony and tartar-emetic; and for the latter I use the second: the first solution will yield both varieties, but the second gives the crystalline variety alone. The line of demarcation between the two kinds of deposit is rigorously distinct, and there appears to be no transitional or intermediate gradation, at least not by this mode of procedure.
- 4. There are a great many circumstances which influence the production of those two MDCCCLVIII. 2 c

kinds of metal in the first liquid; but the most important are, the temperature and density of the solution, the strength of the battery, the state of the depositing liquid, and the physical condition of the receiving surface; if the battery is nearly exhausted in power, the solution unequal in density from long-continued working and neglected stirring, and the receiving surface rough or unclean, the deposited metal will be amorphous and smooth in the lower and denser part of the liquid, and crystalline and warty in the upper and lighter portion. If the liquid is boiling hot, or if it is very dilute or contains much free acid, the deposit will be grey throughout.

- 5. The production of either of these deposits is so easy that the simplest of conditions will suffice. To produce the amorphous variety, take a small battery consisting of one or two pairs of zinc and platinized silver plates, charged with a mixture of one measure of sulphuric acid and twenty measures of water, and having prepared a small quantity of the mixture of chloride of antimony and tartar-emetic, connect with the terminal silver a piece of commercial antimony ("best French regulus") of the same amount of immersed surface as one of the battery-plates, and immerse it in the liquid as an anode, and for a receiving surface immerse a piece of clean polished silver or copper of about one-half the amount of surface of the antimony; connect it by a silver or copper wire with the negative pole of the battery; suspend it vertically in the lower part of the depositing liquid at a few inches from the anode, and protect the connecting wire from receiving a deposit near the surface of the liquid by a piece of gutta-percha tube, glass tube, or by a layer of varnish formed by dissolving pure gutta percha in bisulphide of carbon to the consistence of treacle. A perfectly bright deposit of amorphous antimony will immediately be formed, and will, if the electric power is rather great, exhibit a scaly surface after half an hour's action; but by further continuance the scaliness will cease, and a firm coherent deposit of perfect brightness, and of similar appearance to highly polished steel, will be produced. If the electric power is too great, the deposit will be granular and loose. Much impure matter will appear upon the anode during the process, especially if "common bowl regulus" is used, and may be removed by means of a hard brush and dilute hydrochloric acid. There is no depositing liquid with which I am acquainted, of any metal, which will bear so great a variation of electric power without deteriorating the quality of the deposit, as that composed of tartaremetic dissolved to saturation in hydrochlorate of terchloride of antimony; good amorphous deposits have been obtained in it with batteries varying from one to thirty-six pairs in intensity.
- 6. To produce a crystalline deposit, prepare a similar quantity of the second liquid, and proceed in exactly the same manner, except that a much larger receiving surface should be used in consequence of the greater tendency to a sandy deposit; and the protecting tube or varnish may be omitted. In this case the deposit is grey, and frequently of a dull aspect, or even a dark loose granular powder if the power is too great; but when properly produced it has a silky lustre or semi-iridescent silver-grey appearance, especially when viewed within the liquid; and if the process is continued several days,

or better for one week, the edges of the deposit assume a partly nodular form. The cleaning of the anode in this case may be of less frequency, and with water alone.

- 7. A speed of deposition, varying from 0.75 to 3.0 grains per square inch of receiving surface per hour, has repeatedly yielded a good bright deposit with the first-named solution; and from 0.25 to 2.25 grains per square inch per hour has often given a good crystalline deposit with the second liquid. By keeping the cathodes in constant motion, much more rapid action may be successfully ventured.
- 8. By similar means to those described, and without the aid of motion, a solid bar of dark bright antimony, upwards of 1 inch in diameter and 3 inches long, was formed upon a piece of copper wire $\frac{1}{10}$ th of an inch thick and 2 inches long, in a period of twenty-eight days, and with the aid of motion in about half that time.
- 9. In depositing antimony from these solutions a variety of circumstances occur, a knowledge of which will greatly assist in obtaining perfect specimens of the two varieties. In using the ordinary chloride, after the process has been progressing one or two days with continuous bright deposit, warty excrescences of the grey variety will nearly always occur upon portions of the receiving surface, the localities of their appearance differing according to the shape of the cathode and its depth of immersion; if it be within half an inch of the surface of the liquid, rough grey metal will appear upon its upper parts. The presence of these excrescences appears in nearly all cases to depend upon accumulations of partly exhausted solution at the upper part of the liquid, and at different parts of the receiving surface. The solution, partly deprived of its metal by the action, and thus rendered specifically lighter, ascends in a layer against the cathode, and accumulates at its over-hanging parts and at the surface of the liquid, and yields in those exhausted or acid portions only the grey deposit; similar effects, but in a much less degree, occur in the solution of chloride of antimony and tartar-emetic. To obviate these effects, the solution should be frequently stirred; and to entirely prevent them, the cathode should be immersed at least 1 inch below the surface of the liquid, and be kept in a constant state of gentle swinging motion by attachment to the moving pallets of an ordinary clock. Roughness or want of cleanliness of the receiving surface also seems to favour the production of those excrescences.
- 10. On every occasion with the first liquid, when the battery power became nearly exhausted grey nodules appeared, and when once commenced there was a strong tendency in them to continue; but by increased electric power, stirring the liquid, and persistent action, they became covered with amorphous deposit.
- 11. If during the process of deposition the temperature of the first solution was gradually raised, the deposited metal suddenly changed from amorphous to crystalline at about 205° Fahr., and less suddenly resumed its amorphous character on cooling at about 79° Fahr.; on interposing a galvanometer the conductivity of the circuit was found to be gradually increased during the rise of temperature, and gradually decreased during the fall. These phenomena were repeatedly produced by reheating the same portion of liquid; and similar effects were obtained with the solution composed of

tartar-emetic and dilute hydrochloric acid. The first liquid exhibited no material irregularity in its rate of cooling, indicative of molecular change, between 220° and 100° Fahr. By previously evaporating the same solution to one-half its original bulk, its tendency to yield a grey deposit by heat was considerably reduced; and by diluting it either with pyroxylic spirit, dilute hydrochloric acid, or solution of table-salt, its aptitude to give grey metal at ordinary temperatures was increased and its depositing quality injured.

- 12. Upon examining the electric relations of the depositing liquids with platinum plates in the apparatus described at page 1 of the Philosophical Magazine, January 1857, hot platinum was found to be increasingly electro-negative to cold platinum up to 210° Fahr. in the tartaric acid solution, in accordance with the general rule there stated; but with the first liquid an unusual variation occurred, hot platinum being increasingly negative to cold platinum up to 110° Fahr., then decreasingly to 145° Fahr., and above that temperature increasingly positive to 210° Fahr. Similar effects were obtained with small square columns of Duboscq's carbon (made by him for electric light apparatus) immersed in those liquids in V-tubes. In each case the solution was previously boiled, and repeated experiments gave uniform results.
- 13. By separating the first and second solutions from each other by means of a porous diaphragm, and immersing two perfectly similar pieces, either of platinum or of Dubosco's carbon, one in each liquid, and connecting them with a galvanometer, that in the chloride solution was in each case found to be rather strongly electro-positive to that in the tartaric acid liquid.
- 14. In depositing from either of the solutions which yield the amorphous metal, especially ordinary chloride of antimony nearly saturated with antimony by the battery process, there is a great tendency in the deposit to extend itself in the form of a thin sheet of grey metal upon the surface of the liquid; this lateral spread of the metal is unique, and increases rapidly, and like the grey excrescences formed upon other parts of an amorphous deposit, its speed of increase is much greater than that of the dark metal near it (see 10.); the reason of this appears to be that crystalline antimony is more electro-negative than the amorphous variety, and therefore receives the electric current with greater facility. I have found with perfect specimens of grey and dark antimony deposited upon perfectly similar copper wires, that the amorphous variety was electro-positive to the crystalline in the following liquids:—dilute phosphoric, sulphuric, hydrochloric, and nitric acids; in aqueous solutions of ammonia, potash, and carbonate of soda; also in the first-mentioned chloride solution. This difference of electric condition was in some instances of deposition so great as to generate a separate electric current, and completely redissolve some of the dark portions. The phenomenon of lateral surface deposit did not occur in the tartaric acid solution. Antimony trees, consisting of branches of amorphous and crystalline nodules, were easily formed by suspending a horizontal copper wire as a cathode upon the surface of the terchloride solution.
 - 15. Heat was perceptibly evolved in the first-named liquid during deposition; on one

occasion with a feeble battery, when the external air was at 66° Fahr., the coldest part of the liquid, viz. behind the anode, was at 67° 5 Fahr., and the warmest part in front of the anode 71° Fahr. And on another occasion, with a stronger battery, consisting of twenty-four pairs of zinc and silver plates, weakly excited, with an anode of moderate size, a thermometer in contact with the front of the anode rose from 74° 5 to 87° 5 Fahr.

- 16. Faint crackling sounds, audible at about 4 or 5 feet distant, frequently occurred in the depositing liquid during the process; they issued from the receiving surface, and appeared to be due to alterations in the cohesive state of the metal, being generally attended by the production of cracks, and occasionally of complete rents in the metal; they most frequently occurred when the temperature of the deposit was changed by removal from the liquid or by reimmersion. In one instance metallic sounds were definitely traced to bubbles of gas suddenly expelled with force from a small spot of a very firm deposit formed upon a bar-magnet in the tartaric acid liquid, but no fissure or orifice was observable at the part; they were unattended by evolution of light.
- 17. In common with electro-deposits generally, the inner and outer surfaces of these deposits are in unequal states of cohesive tension, frequently in so great a degree as to rend the metal extensively and raise it from the receiving surface during the process in the form of a curved sheet with its concave side towards the anode. This separation is very apt to occur in depositing upon extended flat surfaces, and does not often occur when the form of the surface is unfavourable, as with wires, rods, bars, &c., where the deposit extends around the mass, or where it passes round projections, edges, &c., and retains a hold thereby. This state of tension is most manifest in thin deposits, and especially in the first-formed layer; for by depositing a thin layer of amorphous antimony upon gold-leaf attached by varnish to a tube of glass, the film of gold was quickly raised from the surface, and curled into fantastic shapes by the cohesive action. Both varieties of the metal exhibit this phenomenon.
- 18. The fracture of amorphous antimony is conchoidal, smooth, and wavy; that of the grey variety is crystalline, radiating like hematite. The texture of the former is rather soft and weak, that of the latter is quite hard and strong. The amorphous metal files easily, the grey with more difficulty. The grey metal, if struck by a hard substance, emits a clear metallic sound, whilst the dark variety gives a more dull tone. The crystallization lines of grey antimony are at right angles to the receiving surface; and in several experiments of depositing upon magnets, the direction of those lines did not seem to be altered by the magnetism.
- 19. The amorphous variety, if gently struck by a hard substance, undergoes a rapid and intense change throughout its mass, attended by development of considerable heat; the crystalline kind undergoes no such change, apparently under any circumstances. The change appears to be molecular in its character; and according to this view the crystalline variety of metal must be regarded as being in a state of comparatively stable molecular equilibrium.
 - 20. The following are a few selected instances of this phenomenon. Antimony was

deposited to $\frac{1}{10}$ th of an inch thick upon a permanent bar-magnet immersed vertically in the solution of tartar-emetic and dilute hydrochloric acid; the deposit was well washed, and after remaining in this state two days, I struck it gently with a small piece of iron; it at once shattered all to pieces with great evolution of heat and with a small cloud of vapour, emitting an odour of burnt tartrates and hydrochloric acid.

- 21. On another occasion a deposit $\frac{1}{8}$ th of an inch thick formed upon one side of a copper medallion in the same liquid had been removed from the solution and lain in water thirty-six hours, I then wiped it dry and momentarily applied the flame of a candle to a very small portion of its edge; the whole at once changed with exceedingly great evolution of heat.
- 22. Once having by accident feebly struck a deposited bar $\frac{5}{8}$ ths of an inch thick against the glass containing vessel beneath the surface of the liquid, the metal was immediately shattered throughout; many pieces fell to the bottom of the vessel, and a powerful suppressed bubbling noise as of heated metal plunged into water occurred.
- 23. At another time, when examining the thermo-electric properties of a bar $4\frac{1}{2}$ inches long and $1\frac{1}{4}$ inch thick, on bringing a heated brass wire near to a very small portion of one end of the bar, a sudden change or commotion of particles took place throughout the mass, passing rapidly from the heated part to the opposite end, attended by the production of a few very minute cracks, and with great evolution of heat, rapidly boiling a few drops of water placed upon it.
- 24. And on another occasion, on accidentally touching a cathode covered with perfect amorphous deposit against the anode, the change with considerable hissing noise occurred, the electric spark or current from the battery being in this case the apparent exciting cause of the phenomenon.
- 25. And finally, with a rapidly formed and perfect specimen of dark antimony deposited upon a swinging helix of fine copper wire, on washing it first in dilute hydrochloric acid and then in water, and gently striking it with a piece of glass, it at once shattered into small particles with considerable force, projecting some of them to a distance of 10 feet, apparently by converting into steam the adhering particles of water.
- 26. This thermic property of amorphous antimony is retained apparently unimpaired upwards of $2\frac{1}{2}$ years; a piece deposited in October 1854 evolved considerable heat by slight percussion in May 1857. The change appears to be similar to the molecular changes of sulphur, selenium, iodide of mercury, &c., observed by RITTORF, REGNAULT, WEBER, and others. I have never found that the phenomenon could be repeated with the same specimen.
- 27. The only observed exciting causes of the phenomenon have been ordinary heat, concentrated solar heat, friction, percussion, fracture, and the voltaic spark. Heat appears always to determine it, whilst with thin deposits, slight percussion and even fracture have frequently failed. Thick pieces will bear gentle filing, but not violent fracture at ordinary temperatures without the change occurring.

- 28. The production of the change and the rapidity of its action are considerably dependent upon the temperature of the mass; pieces which would easily fracture without further change in cold water would not break without change in the air at 60° Fahr.; the certainty and quickness of the action were also manifestly greater when the metal had been previously warmed.
- 29. Immersing the metal in water and gradually heating it to 212° Fahr. did not cause the change, destroy the property, or have any material effect upon the rate of heating of the liquid. Nor did sudden immersion in boiling water, or partial dissolving of the metal by warm aqua-regia, cause it, or destroy the property; but slight percussion on removing it from the hot water caused it powerfully.
- 30. At the suggestion of Mr. Faraday, and with the kind assistance of him and Dr. Tyndall, I have been enabled to reduce pieces of the metal of moderate thickness (\frac{1}{10}\text{th} of an inch) to a state of fine division without exciting the change or destroying the thermic property, by carefully triturating them in a mixture of broken ice and salt. Pieces of the thickness of an address card were reduced to powder without the change occurring by careful pulverization in water at 60° Fahr.; the most ready plan adopted was by gently pressing the pieces in small quantity, under water, in a mortar with a rolling motion of the pestle (not by blows or rubbing) until they attained the size of a pin's head, listening all the time to detect molecular changes, then reducing the particles to complete powder by gentle rubbing.
- 31. By placing small quantities of the dried active powder upon thin sheets of metal and gradually applying heat until the change occurred, sufficient heat was evolved by the molecular force to commence oxidation, which spread at a much lower speed throughout the mass, producing true combustion, and raising it to a temperature of dull redness visible in daylight. The molecular change in the powder is attended by a visible commotion amongst the particles. Portions of the grey variety reduced to powder and treated similarly, exhibited the same combustion.
- 32. It would appear from these experiments that amorphous antimony is susceptible of three different, distinct, and independent changes:—1st, the cohesive change of its mass, consisting of extensive fractures and bending; 2nd, the peculiar change, attended by evolution of heat; and 3rd, a chemical change, consisting of oxidation and true combustion; whilst the crystalline variety is susceptible of only two of these changes. Antimony, deposited in the state of a black powder upon a small cathode, by rapid action in the solution of tartar-emetic and chloride of antimony, exhibited no perceptible change of the second kind, but manifested rapid combustion by application of heat; and similarly with antimony powder, deposited from a solution of antimonic oxide in pyrophosphate of soda. Deposits of metal, obtained from solutions of tersulphide of antimony in caustic potash, exhibited the phenomenon of unequal cohesion, but not the peculiar change. Fused terchloride of antimony, free from water, with an anode of antimony and cathode of copper, did not conduct, even in a small degree, the electric current from five pairs of zinc and silver batteries. By rotating a horizontal silvered

disc of thin brass, $1\frac{1}{4}$ inch diameter, in the solution of tartar-emetic and chloride of antimony, at a speed of 3500 revolutions per minute for thirty-three minutes, and simultaneously passing a current from five zinc and silver batteries through it by means of a disc of antimony $2\frac{1}{4}$ inches diameter, a scaly deposit of bright black metal was obtained, possessing the usual thermic property.

- 33. In every case where the deposited metal was dark and shining, it evolved heat by the exciting causes, and under the conditions named; and in every case where it was grey and crystalline, it would not evolve heat.
- 34. The physical properties of the metal appear to be closely connected with the condition of the liquid; for in every case where the latter was sufficiently changed, whether by alteration of chemical composition, of density, of temperature, or of the electric power, a change occurred in the colour and brightness of the deposit; at the same time, by experimentally examining the influence of each of these circumstances separately, I did not find the peculiar state of the metal to be invariably connected with any one of them.
- 35. On immersing a piece of the unchanged metal in mercury, and applying heat very gradually, it suddenly changed with great force at a temperature of about 250° FAHR.
- 36. A deposit, consisting of 279.5 grains of amorphous antimony and 101.5 grains of the grey variety, was formed upon a spherical bulb of a thermometer, and after well washing and drying, was wrapped in cotton-wool and touched with a small heated wire; it at once changed, and the mercury rose from 75° ·2 Fahr. to 275° ·2 Fahr. in twenty-eight seconds. The diameter of the glass bulb was $\frac{15}{32}$ of an inch.
- 37. A second deposit, consisting of 504·49 grains of dark antimony, formed upon the same bulb, and treated in a similar manner, raised the temperature of the mercury from 75° Fahr. to 445°·0 Fahr. in one minute forty seconds. The thickness of the layer of metal was ¼th of an inch, and by the great heat evolved it acquired a dull grey aspect, similar to the grey variety.
- 38. A cylindrical bar of the amorphous metal, about $\frac{1}{2}$ an inch in diameter, formed upon a rod of grain tin $\frac{1}{8}$ th of an inch thick, when changed by the momentary application of a heated wire, evolved sufficient heat to melt the tin completely, which ran out through a crack in the antimony, and remained fluid a short time. A bar of bismuth, $\frac{3}{16}$ ths of an inch thick, coated to about $\frac{3}{4}$ ths of an inch in diameter, did not melt by the change.
- 39. By immersing a perfect bar of the dark variety, weighing 456.9 grains, in 836.6 grains of distilled water, contained in a large thin glass tube wrapped in cotton-wool, immersing the bulb of a thermometer in the water, and allowing the whole to acquire a uniform temperature, then inducing the change by momentary contact of a heated wire, and stirring the water to render it uniform, the temperature of the water rose from 63° to 82° Fahr. The cooling influence of the water, in this and in several similar experiments, appeared to prevent the metal undergoing its full amount of change.

- 40. The peculiar change is attended by alterations in the colour and fracture of the metal; from a bright steel colour and glassy fracture, it passes to a dull grey colour and granular fracture, as if it progressed a stage towards the crystalline modification: this is particularly manifest in thick pieces gradually heated in the air until the change occurs. It is also attended by a change in the form of the metal; bars which were nearly straight before the action, were considerably curved afterwards, and the direction of the curvature was such, that the outer side, or that last deposited, invariably became more con-Upon applying heat to a thin layer of dark antimony, formed upon a thin sheet of silver, it immediately underwent the change, and became exceedingly curved in a similar direction. This direction of curvature appears to be due to a cause already mentioned (see 16.17), the unequal cohesion of the inner and outer surfaces of the metal.
- 41. By applying heat to the ends of deposits formed upon heliacal copper wires, the action was gradually propagated to the opposite ends at a speed varying from 12 to 30 feet per minute; and its rapidity of progress appeared to be dependent upon the temperature of the metal and the cooling influence of the enclosed wire, also upon the absence of grey nodules; the warmer the antimony, the greater its thickness, and the more free it was from grey metal, the more rapid was the action; and in cases where the deposit was thin in places, or had occasional grey nodules, the action was impeded at those parts, and sometimes totally arrested. Grey nodules invariably impeded the action, and caused it to progress by sudden starts. The progress of the change was rendered more or less visible by the evolution of a cloud of vapour at its advancing point; also in the case of fine copper wires, by a peculiar snake-like movement in the coils at that part, apparently produced by the cohesive alteration already mentioned (see 16. 17. 40.). After the change, the layer of antimony contained numerous transverse and longitudinal fissures, as if it had contracted in those directions.
- 42. In a number of instances, by connecting the ends of the copper wires with a galvanometer, and inducing the change in the coating by a heated wire, a feeble electric current occurred in the enclosed wire whenever, from thinness of the coating, its termination, or other causes, the action ceased; and in every case passed in a direction opposite to that of the peculiar action. By touching the deposit with a heated wire some time afterwards at the point where the action ceased, a current of ordinary thermoelectricity was in each case generated, and passed in a similar direction.
- 43. By exciting the peculiar change in two pieces, weighing upwards of 600 grains each, placed in the vicinity of the poles of delicately suspended magnetic needles, no deflection of the latter in either case occurred.
- 44. By taking two bars, each about 3 inches long, one of amorphous antimony and one of the crystalline variety, connecting their extremities with a galvanometer, and cautiously bringing a small flame of a spirit-lamp within half an inch of their remote and touching ends, the dark bar was found to be feebly thermo-electro-positive to the grey A thin rod of changed antimony was found, in a second experiment, to be weakly 2 p

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thermo-positive to a large lump of grey metal. And in a third trial, a large changed bar was positive to a bar formed by melting and casting deposited antimony.

- 45. Both the grey, the changed, and the unchanged metal acquired a coating of dendritic crystals of silver by immersion in a solution of nitrate of silver.
 - 46. The following are some determinations of the specific gravity of the deposits:—

Three specimens of unchanged antimony.

No. 1. 5.739 at 70° FAHR.

No. 2. 5.924 at 62° FAHR.

No. 3. 5.944 at 62° FAHR.

Five bars of unchanged antimony.

No. 4. 5.766 at 60° FAHR.

No. 5. 5.769 at 62° FAHR.

No. 6. 5.782 at 62° FAHR.

No. 7. 5.785 at 62°.5 FAHR.

No. 8. 5.814 at 61°.5 Fahr.

The bar No. 4 weighed 467·00 grains; and by immersing it in 750 grains of distilled water at 60° Fahr. and inducing the change, it evolved a little chloride of antimony, and acquired a density of 5·818 at 60° Fahr. The bar No. 7 weighed 456·9 grains, and by changing it in 836·6 grains of water at 63° Fahr. (see 39.), it acquired a density of 5·862 at 62°·3 Fahr. And the bar No. 8, changed in the air, acquired a density of 6·186 at 61°·5 Fahr.

Four specimens of changed antimony.

No. 1. 5.748 at 75° Fahr.

No. 2. 5.818 at 60° FAHR.

No. 3. 5.880 at 62°.5 Fahr.

No. 4. 6.029 at 64° Fahr.

Ten specimens of grey antimony.

No. 1. 6.369 at 75° FAHR.

No. 2. 6.444 at 72° FAHR.

No. 3. 6.497 at 72° Fahr.

No. 4. 6.522 at 75° Fahr.

No. 5. 6.557 at 75° Fahr.

No. 6. 6.616 at 72° Fahr.

No. 7. 6.616 at 75° Fahr.

No. 8. 6.662 at 75° Fahr.

No. 9. 6.671 at 75° FAHR.

No. 10. 6.673 at 75° FAHR.

Nos. 1 and 2 of the grey specimens were deposited from the upper part of the solution, composed of tartar-emetic and dilute hydrochloric acid; the remainder were deposited from the tartaric acid liquid, Nos. 9 and 10 being formed very slowly. The greater specific gravity of the crystalline metal appears worthy of consideration in connexion with similar differences of density of amorphous and crystalline sulphide of antimony*.

- 47. The densities of both varieties changed to that of ordinary antimony by fusion; several specimens were fused under tersulphide of antimony, also under cyanide of potassium, and their specific gravities were as follows:—6.67; 6.694; 6.71 at 76° Fahr.; 6.75 at 73° Fahr.; and 6.83 at 75° Fahr.
 - 48. I have endeavoured to determine the electro-chemical equivalent, or equivalents,

^{*} Vide GMELIN'S Handbook, vol. iv. p. 337-349. Cavendish Society.

of the two deposits; and by repeated experiments with two perfectly similar batteries, one for depositing the metal, and the other for determining the loss of zinc by ordinary chemical action, and deducting the average loss of zinc in the cells of the idle battery from that occurring in those of the working one, and excluding, as far as possible, all other sources of error, I found in one experiment that for every 32.6 parts of zinc dissolved, 43.38 parts of amorphous antimony were deposited in the solution of tartar-emetic and dilute hydrochloric acid; in a second experiment 43.61 parts of antimony were deposited; and in a third experiment 43.71 parts; and of grey antimony, from the tartaric acid solution, 41.1 parts.

To exclude the possibility of difference of local action in the two batteries, I arranged three depositing liquids in succession, viz. 1st, the saturated solution of chloride of antimony (i. e. saturated with antimony by means of a battery); 2nd, a solution composed of 4 parts of sulphate of copper, dissolved in a mixture of 1 part of pure sulphuric acid and 20 parts of distilled water; and 3rd, the tartaric acid solution of tartar-emetic. current from twelve pairs of zinc and silver batteries was passed through these liquids by suitable electrodes of copper and antimony, and the weights of the respective deposits, after washing and drying, ascertained; and I found in the following experiments that for 31.7 parts of copper deposited in the sulphate solution, the weight of perfect crystalline deposit varied from 40.41 to 40.797 parts, and of perfect amorphous deposit, from 42.304 to 43.813 parts.

Crystalline deposit.		Amor	Amorphous deposit.	
No. 1.	40·41 parts.	No. 1.	42·304 parts.	
No. 2.	40.612 parts.	No. 2.	43·112 parts.	
A No. 3.	40.659 parts.	No. 3.	43·160 parts.	
B No. 4.	40.707 parts.	A No. 4.	43·170 parts.	
No. 5.	40.797 parts.	No. 5.	43·199 parts.	
		B No. 6.	43·428 parts.	
		No. 7.	43.813 parts.	

Nos. 3 of grey deposit, and 4 of amorphous deposit, were formed by the same current; and Nos. 4 of grey and 6 of amorphous deposit, were also formed by one current. Nos. 1 of grey, and 3 and 7 of amorphous deposit, were formed upon copper wire helices, kept in a state of constant motion; No. 7 being formed by very slow action.

- 49. In a separate experiment, with a two-pairs battery, the equivalent of crystalline metal obtained was 40.728, and of amorphous metal 43.989; and in a very reliable experiment with a moving helix, the equivalent obtained of amorphous antimony was 43.284. With a one-pair battery, an equivalent of 44.95 parts of amorphous metal was obtained.
- 50. One source of variation in the resulting numbers of the dark variety appeared to arise from particles of the depositing liquid being mechanically enclosed within the depositing metal, chiefly by the formation of minute cracks and scales; these were, as far as possible, prevented by using cathodes formed of wire, maintaining a uniform tem-

perature and an uninterrupted electric current, by suitable adaptation of the electric power, and by keeping the cathodes in constant motion. The adhering liquid was removed by washing in dilute hydrochloric acid, distilled water, and drying.

- 51. The amorphous metal invariably contained a small portion of chloride of antimony, more or less of which was always evolved during the molecular change, according to the temperature to which it was raised; thin pieces, changed under water, still retained the greater portion of the chloride. A thin specimen, formed upon a copper wire helix, lost by change in air 0.337 per cent. of its weight, and a thick one lost 3.556 per cent. The grey variety, when perfectly prepared, evolved scarcely a perceptible odour of burnt tartrates or hydrochloric acid by the application of heat.
- 52. To remove the chloride of antimony, portions of thin deposits were finely pulverized in cold water, then washed either with distilled water, dilute hydrochloric acid, or solution of caustic potash, finally with water and dried; small quantities of the chloride were removed, especially by the dilute acid, but in each case the powder still retained its thermic property, and evolved chloride of antimony during the change. Other portions of the pulverized metal were shaken with boiling hot dilute hydrochloric acid, also boiled in similar acid; the chloride was then found to be more completely removed, and the thermic property destroyed. By digesting the powder in one part of hydrochloric acid and three parts of water, for one week, less chloride was evolved during the change and during the combustion succeeding it.
- 53. A solid piece of amorphous antimony, weighing 774·0 grains, was placed under a bell-glass filled with water; and the change induced in it by a blow; a small quantity of chloride of antimony separated, together with a minute quantity (less than $\frac{1}{20}$ th of a cubic inch) of gas; the water was then strongly acidulated with hydrochloric acid, and allowed to rest five days; the gas still remained unabsorbed.
- 54. By fusing well-washed specimens, either of the grey or of the changed dark variety, perfectly free from laminæ or crevices, in a porcelain crucible, no material alteration occurred in them until at a temperature of incipient fusion; they then swelled to a large pasty mass, evolving volatile matter, and finally contracted to a clear shining globule, covered with downy crystals of oxide of antimony. Similar results were obtained by fusion under tersulphide of antimony or cyanide of potassium, except that in the latter case a minute quantity of black matter separated, which I found by combustion alone, and with nitrate and chlorate of potash, to be carbon. The metal, previously powdered and placed under cyanide of potassium, agglutinated into a pasty mass before fusion or evolution of vapour. Five specimens of grey antimony, fused under the cyanide, lost respectively 0.437, 0.618, 0.81, and 0.88 per cent. by weight. Two thin specimens of amorphous antimony, previously changed under water at 60° Fahr., lost respectively, by fusion under cyanide of potassium, 5.1 and 5.2 per cent. A moderately thin specimen, previously changed in air at 60° Fahr., lost 3.3 per cent.; a thick one, similarly changed, lost 2·1 per cent.; a second ditto lost 2·52 per cent.; and three other specimens of changed antimony lost respectively 3.484, 3.92, and 5.56 per cent.

specimens of antimony, $\frac{1}{10}$ th of an inch thick, changed in the air at 60° Fahr., and then fused under chloride of potassium, lost each 7·0 per cent. by the fusion, while two other portions of the same lost, by fusion under cyanide of potassium, 1·86 and 2·1 per cent. respectively. Five pounds of the changed dark metal, fused in an ordinary crucible, evolved much volatile matter at complete fusion. The antimony, fused under cyanide of potassium, and cooled slowly without disturbance, had strong crystalline markings upon its upper surface, while that fused under chloride of potassium had not, probably because in the latter case the salt solidified before the metal and confined its surface.

55. The combined loss of weight by the change, and by fusion under cyanide of potassium, of three specimens, was as follows:—first specimen, a thin one, changed in air at 65° .0 Fahr., lost $3\cdot31$; by fusion, $2\cdot49$; total loss, $5\cdot8$ per cent.: second specimen, a thin one, by change in air, $3\cdot19$; by fusion, $2\cdot71$; total loss, $5\cdot9$ per cent.: and third specimen, deposited at the upper part of the liquid, by change in air, $1\cdot94$; by fusion, $3\cdot098$; total loss, $5\cdot038$ per cent.

56. Small pieces, either of grey or changed antimony, immersed in fused nitrate of potash, rose to the surface by the aid of the evolved gas when approaching fusion, and burned with great evolution of heat and formation of antimoniate of potash. No particular effect occurred by rubbing together unchanged antimony powder and pulverized chlorate of potash.

57. Contrast of the two varieties:—

Unchanged dark variety.

Specific gravity:

5.739 to 5.944.

Colour:

Polished steel.

Fracture:

Amorphous.

Texture:

Soft and weak.

Relative electric condition:

Positive.

Relative thermo-electric state:

Positive.

Grey variety.

Specific gravity:

6.369 to 6.673.

Colour:

Silver-grey.

Fracture:

Crystalline.

Texture:

Hard and strong.

Relative electric condition:

Negative.

Relative thermo-electric state:

Negative.

58. It would be interesting to inquire, to what force or mode of physical action is the evolution of heat in antimony due? If it is caused by some *new* mode of physical action, then the force causing it will of course differ, in some respects, from all other forces, and will be found to manifest itself in a number of substances and not in antimony alone, like heat, electricity, magnetism, &c.; and will also be found, like each of them, to be correlative with, and convertible into, other natural forces.