VI. *On the Decomposition of the Acid of Borax or Sedative Salt.* *By* Lawrence de Crell, *M. D. F. R. S. Lond. and Edinb. and M. R. I. A. Translated from the German.*

Read February 7, 1799.

THE salt called Borax, so useful in various manufactures and arts, and hitherto imported only from Thibet and Persia, or, in small quantities, from Tranquebar,* has ever excited the attention of natural philosophers. This attention was principally directed to the acid (called sedative salt) contained in it; its other component part, the alkaline salt, (soda or natron,) being better known, and found in many other natural productions, either alone, or in conjunction with other acids. The acid abovementioned has hitherto been discovered only by HÖFER, in the lagone of Castelnuovo; by MARTINOVICH, in the petroleum of Gallicia,† mixed with alkaline earth; and by Mr. WESTRUMB, near Luneburg. The scarcity of this acid, and its being found only in the substances and situations abovementioned, occasioned a supposition, in the minds of those who minutely observe and examine the course of nature, that it is not a simple substance, but is formed afresh from a variety of other substances, previously decomposed, by a singular coincidence of operative causes; and, consequently, that it belongs to compounds.

* DEMACHY's *Laborant im Grossen*, Part 2, page 89.

† CRELL's *Annalen*, 1791. T. I. p. 162.

Numerous have been the experiments made by chemists, who supposed they had formed this salt by composition: some described experiments, which they declared to have succeeded with them, though they always failed, when attempted by others;* from which, LEONHARDI concludes, that nothing more can be expected from any similar attempts to produce sedative salt.†

I was surprised that these chemists had never (so far as I knew) examined the subject by the way of analysis, and endeavoured to decompose the sedative salt already formed by nature. Indeed, no great hopes of success could be entertained, as daily experience shows, that though this salt be kept fluid, in the hottest fire, for many hours together, till it becomes a vitrified substance, yet, when it is afterwards dissolved in distilled water, the solution is complete, without any residuum, and it then shoots into crystals of precisely the same salt as before. Notwithstanding all this, when I reflected, that borax is generated only in certain climates of the East, and that its acid is found only in particular substances and situations, as has been already mentioned, I could not but suppose the latter to be the produce of a new formation. This being premised, I considered maturely in what manner the decomposition of this new and extraordinary compound might be attempted. Admitting the composition to be formed by the coalition of a number of different substances, it seemed not improbable but that an acid, penetrating into and dissolving the whole mass, would rather

* See FUCHS *Geschichte des Boraxis.*

† MACQUER's Dictionary, translated by LEONHARDI, 2d edit. Vol. V. p. 588.

associate with some than with others of its various component parts, and thus produce a separation or change of the latter. Besides, as the sedative salt, strong as its operation is (in a high degree of heat) upon almost all neutral salts, has but a faint taste of acid, it might be supposed, that its acid is contained within some unknown species of earth, intimately combined; or within some sort of inflammable matter; or, according to a phrase used in the new system, there might be a deficiency of acid matter; that, therefore, some more powerful acid would probably separate and dissolve the earthy particles, destroy or change the inflammable matter, or impart the acid it might be supposed to want.

My choice, among the different acids, was fixed upon that particular one which, though not always quick in its operation, never fails to penetrate deep into all soluble substances, is nearly related to all inflammable bodies, and possesses an abundance of acid matter; I mean the oxygenated muriatic acid, prepared with manganese. In the application of this menstruum, I resolved to follow the practice established by the constant experience of both ancient and modern chemists; which has taught us, that difficult decompositions of parts closely united, are more easily effected by a gentle, long continued, digestive heat, and repeated distillation of the same menstruum, than by a heat which is more violent, and operates more quickly.

I first made some preliminary experiments, in order to judge what probability there might be of success.

Experiment 1. I poured an ounce and a half of the above-mentioned acid upon two drams of sedative salt, in a retort, to

which I adapted a proper receiver, and then placed the mixture in a gentle digestive heat, of from 140° to 200° of FAHRENHEIT. The fluid was distilled over very slowly, and the salt was dry on the third day. The salt in the retort seemed unchanged; nor had the marine acid lost any thing of its usual smell.

Exp. II. I poured the distilled fluid out of the receiver upon the same salt, and exposed them to the same degree of heat as before. The salt again became dry on the third day, but there was yet no appearance of any change.

Exp. III. I repeated the same process a third time. I now perceived, during the distillatory digestion, several bright yellow spots upon the salt, as it ascended the sides of the retort, resembling well-formed ammoniacal flowers of iron; more of which I discovered, after the entire exhalation of the fluid.

Exp. IV. The above change induced me to repeat the distillation; and I then perceived, not only as many, but a much greater number of bright yellow spots, some of which were even much darker in colour, and approaching to brown. A change had now evidently taken place, which change increased upon every repetition of the process; I therefore judged I might follow this direction with confidence. But, with a view to use the greatest accuracy and precaution in my proceedings and observations, I resolved to begin my work over again.

First, I procured some ounces of sedative salt, which had been obtained from borax by means of vitriolic acid; and then prepared two quarts of the abovementioned oxygenated muriatic acid, by distilling three parts of muriatic acid with one part of the purest manganese, in the usual manner; this I preserved in a cool dark place. Thus, the substances used in the following experiments, were always of the same nature.

Exp. v. I poured three ounces of the oxygenated muriatic acid upon half an ounce of the sedative salt, in a white glass tubulated retort. I used such a retort, that (in frequently pouring back the distilled fluid) I might not have to lute afresh the several vessels, after every distillatory digestion. For the same reason also, I chose a tubulated receiver, the tube of which gradually terminated in a point, in shape of a funnel. This tube passed into a phial, placed in such a manner that all the fluid passing into the receiver dropped immediately into the phial, the joinings of which were closed with bladder. To close the tube of the retort, I did not think it right to use a waxed cork, (though it closes very tight,) because it might be corroded; and also, because the vapours, dropping from the cork, might carry some fat and oily matter back into the retort. For the same reason, I would not use any greasy lute; but closed the joints of the glass stopper (which fitted remarkably close) with a ring of fine sealing-wax, closely pressed upon it, but which could easily be disengaged, after my work was done, while the retort was still warm: and, as I was even afraid of an oily lute about the joints of the receiver, I closed them up with a ring of very fine white clay, which I fitted to them as exactly as possible, by pressure; letting it stand several days, to dry, and then carefully filling up all the cracks. Having made this previous arrangement, and put the abovementioned ingredients together, I suffered them to remain cold for twenty-four hours; at the end of which, the salt was not entirely dissolved, but, upon the application of heat, the whole became a clear fluid.*

* This appeared to me so striking, that I endeavoured to obtain a confirmation of it. I made a similar mixture, in the same proportions, which was not dissolved so long as it remained cold; but was dissolved by heat. When the solution cooled, a

The degree of heat in the sand was from 180° to 240°, by which the fluid evaporated very slowly. During this operation, there ascended, or rather crept up the sides of the retort, a considerable quantity of salt, in very loose flowers, rising pretty high above the fluid, increasing by degrees, and chiefly occupying that half of the retort which received a greater degree of heat than the other; but never the opposite or colder half. In four days, the fire being extinguished towards the evening of the last, the fluid had evaporated, so as to leave the salt apparently dry. After cooling for some time, the bladder upon the phial was moistened by water, and the vessels were separated; the sealing-wax also having been removed, and the stopper taken out, the distilled fluid was poured back, through a glass funnel, upon the salt, without disturbing the lute.

Exp. VI. As soon as the fluid was added, the salt at the bottom began by degrees to dissolve: that on the sides of the retort did the same, after it was heated, but soon began to form again: the solution appeared of a yellowish hue. In general, however, the whole experiment took the same course as in *Exp.* V. and the smell, both of the salt and the fluid, seemed to be unchanged. The only difference was, that the former did not appear like salt, (the crystallization on the sides excepted,) and in single detached crystals, but something like a white, uniform, spongy, and, as it were, earthy mass. The fluid was now again taken from the phial, as in *Exp.* V. and poured back upon the salt.

Exp. VII. VIII. and IX. During the third distillation, bright

small part of the salt (and a larger as the cold increased) precipitated, which was dissolved again, by a fresh application of heat. But, with the degree of heat I employed, no more than one part of salt would dissolve in six parts of the acid.

yellow spots began to appear upon the white flowers; and, after the salt at the bottom had become dry, similar spots appeared upon it, particularly upon the lower surface. The fluid was again, for the fourth time, poured upon the salt, and distilled; when the yellow spots and flowers increased in number. This was also the case in the fifth distillation.

Exp. x. The fluid obtained by the last experiment, which had changed a little in smell, and had acquired a particular scent, almost as if some sebacic acid had combined with the muriatic, was poured upon the salt, as before. The number of yellow spots, which had also become of a darker hue, was considerably increased. The salt had now been exposed, ever since the vth. *Exp.* for thirty-two days, to the digestive distillation; and the intermediate time between each distillation, had been longer or shorter, in proportion to the degree of heat, and to the time of kindling and extinguishing the fire. As I now found, that business of importance would prevent me from continuing my labours for some months, I poured two other ounces of the muriatic acid upon the salt, besides the fluid so often drawn off by distillation, and left the mixture at rest.

Exp. xi. xii. xiii. xiv. When my business was finished, I again undertook the distilling of the mixture, which had been so long digesting in the cold, for the seventh time, and obtained the same results as in *Exp.* x. Nor was there much difference observed in the xiith. xiiith. and xivth. *Experiments.*

Exp. xv. I now poured the fluid obtained by the xivth. *Exp.* upon the salt, (which had acquired more and more yellow spots, brighter in hue,) and then proceeded as before, till the salt became dry; upon which, when the retort was cool, I poured one

ounce and three drams of the muriatic acid, in addition, and allowed the mixture to digest gently for some days.

Exp. XVI. In this twelfth distillation, there appeared a large quantity of flocculent sublimate, looking almost like branches, hanging down, and in many places of a yellow colour; it extended even into the neck of the retort, and almost covered the interior aperture of the tube.

Exp. XVII. The thirteenth distillation produced the same phænomena. Upon the lowermost surface of the mass of salt, many light-brown spots appeared, as soon as the fluid was so much evaporated that no more of it could be seen upon the salt.

From all these circumstances, I now believed the mass of salt, by a digestion of twenty-two days, and seven distillations, from *Exp.* XI. to XVII. (that is, by a digestion of fifty-four days, and thirteen distillations, in the whole,) to be so far decomposed, as to admit of a separation of some of its constituent parts. I therefore supposed I might leave off applying only a digestive warmth, and proceed to a greater degree of heat.

Exp. XVIII. Having poured out the fluid obtained by *Exp.* XVII. and replaced the phial, I increased the degree of heat. By this, the retort became quite obscured, first by fumes, and afterwards by a quantity of white sublimate, attaching itself to all its sides, which, however, had not the appearance of common sedative salt. As I increased the heat, the sublimate grew darker in colour; afterwards became black and frothy; and, at length, ran down the sides of the retort, in different places, like thick oil of hartshorn, the retort being almost wholly blackened by it.

Exp. XIX. While the retort was still warm, I poured into it

the fluid obtained by *Exp.* XVII. having first warmed it a little; when, almost in the same instant, a very agreeable phænomenon took place. Crystals, perfectly white, shot forth suddenly, and all at once, from every part of the black mass covering the sides of the retort. The distillation being continued, these crystals were at length dissolved, and entirely removed. The supernatant fluid was, as usual, almost colourless. When the mass of salt appeared dry, the fire was increased, as in *Exp.* XVIII. and the same appearances as above related took place: first, the sublimate appeared white, then black, frothy, and flowing down the sides.

Exp. XX. I proceeded, as in *Exp.* XIX. to pour back the distilled fluid. Instantly a number of the whitest crystals shot forth from the black ground, forming small groups; but the retort was cracked.

Exp. XXI. I therefore took all the vessels asunder, and shook the retort well, till whatever hung upon its sides was dissolved; then distilled the fluid in another retort, till the mass of salt appeared quite dry. I now put the retort into a crucible, surrounded it with sand, fitted another receiver to it, and placed the crucible in an open fire. First, some sublimate was produced, towards the neck of the retort, (but which vanished as the heat increased,) and then a small portion of fluid, (hardly more than a dram, or a dram and a half,) which appeared to smell a little of the sebacic acid. At the bottom of the retort was a blackish mass, *a*, and likewise some sublimate, *b*, which, by its varied appearance, seemed to be of a two-fold nature.

Exp. XXII. The residuum taken out of the broken retort had a spongy appearance, and swam upon water; it had a blackish colour, and weighed three drams and ten grains.

Being exposed to the air, the blackish colour became lighter, and inclining to grey. When digested in sixteen parts of distilled water, in the usual temperature, for two days and a half, it did not all sink to the bottom; and, after being digested with heat for twenty hours, it was not entirely dissolved: that part which sank, was of a blackish brown. More water was then added, and it was made to boil for two hours; it was afterwards placed upon a paper filter, (the weight of which was previously ascertained,) and edulcorated with boiling distilled water, till at last a proportion of twenty-six parts of water to the substance had been used. After all the fluid, α, had passed through, and the filter, with the residuum, had been dried in a heat of 212°, for an hour and a half, the residuum, β, weighed, exclusive of the filter, nineteen grains.

Exp. XXIII. The fluid, α, obtained by *Exp.* XXI. was suffered to evaporate gradually, and yielded three drams and ten grains of a white transparent salt.

Exp. XXIV. This salt (obtained by *Exp.* XXIII.) was put into a small retort, and exposed, in a crucible filled with sand, to an open fire. It became of a blackish-brown colour, yielded some sublimate, *a*, (about five grains,) a small portion of fluid, *b*, and a blackish-brown residuum, *c*, which grew lighter in colour, on being exposed to the air.

Exp. XXV. The fluid, *b*, (of *Exp.* XXIV.) smelt like marine acid, and precipitated nitrate of lead.

Exp. XXVI. The residuum, *c*, (of *Exp.* XXIV.) by the addition of some water, became whiter, and was dissolved; more water having been added, it was digested with heat, by which the matter was dissolved. The solution being afterwards filtered, I

obtained two drams and four grains of white salt: the residuum upon the filter weighed four grains.

Exp. XXVII. This salt (*Exp.* XXVI.) I again exposed to the fire; when it yielded from twenty to thirty drops of acid liquor, four grains of sublimate, and a residuum, which, being dissolved, yielded one dram and thirty-three grains of salt, and left two grains and a half, *c*, upon the filter.

The same salt, (obtained by *Exp.* XXVI.) being distilled, became of a brownish-grey colour; and, besides a few drops of fluid, yielded not quite two grains of sublimate. On treating the residuum with water, it yielded sixty-eight grains of salt, and there were not quite two grains left upon the filter.

Exp. XXVIII. On treating these sixty-eight grains of salt in the same manner, they yielded a few drops of fluid, and two grains of sublimate: after filtration, there remained forty-eight grains of salt, and a residuum of hardly one grain and a half.

Exp. XXIX. The same salt, treated in the same manner, yielded a few drops, and a little sublimate; and, after filtration, thirty-five grains of salt, and a residuum of hardly one grain.

Exp. XXX. On treating these thirty-five grains of salt in the same way, they yielded, besides a very small quantity of fluid and of sublimate, twenty-four grains of salt, and about three quarters of a grain of residuum.

As I now discovered that the quantity of salt was continually decreasing, and some coal separating from it, I thought it superfluous to endeavour to decompose the above twenty-four grains any farther.

Exp. XXXI. The residuum, β, of *Exp.* XXII. was light, blackish,

and like coal. I now poured common concentrated muriatic acid upon three grains of it, and digested the mixture, for forty-two hours, in a considerable degree of heat, but no dissolution was apparent. I then added smoking nitrous acid, and digested it for twenty-four hours, till it boiled, without any apparent dissolution. I added some sugar, (about two grains,) but without effect, except that its colour grew yellowish. I now boiled the fluid, till it all evaporated in reddish-yellow vapours: there remained a very black, thick, glutinous mass, smelling like burnt sugar. Having added three ounces of water, the greatest part of the blackish matter rose to the surface, and the water appeared only a little tinged. The fluid part, indeed, became brown by boiling; but, after rest and subsidence, it again grew clear. I filtered it, *a;* then poured two ounces more distilled water upon the residuum, and, after digesting, boiling, and filtering, added the filtered fluid, *b,* to the former, *a.* After this treatment, there remained two grains of residuum, *c.*

Exp. XXXII. Having caused the fluid *a, b,* of *Exp.* XXXI. to evaporate, it yielded a salt greyish-yellow mass, which very quickly attracted the moisture of the air. Being again dissolved in water, and saturated with potash, a considerable quantity of whitish earth was precipitated, very much resembling talc.

Exp. XXXIII. The residuum, *c,* of *Exp.* XXXI. which, besides its insolubility and lightness, had much of the external appearance of coal, was now thrown upon melted nitre, and it deflagrated. I placed a second crucible with melted nitre close to it, and having, at the same moment, thrown into one the above-mentioned residuum, and into the other a quantity of common charcoal pulverized, I could not observe the smallest difference in effect. Very little difference was also apparent, as to the

residuum, β. of *Exp.* XXII. *c*, of *Exp.* XXIV. and that of the following experiments.

Exp. XXXIV. To obviate the objection, that sedative salt alone would perhaps deflagrate with melted nitre, I made that experiment also, but in vain. Not the smallest deflagration took place, even when both were melted together for many hours.

Exp. XXXV. Another objection may be made, namely, that in distilling the muriatic acid from manganese, part of the latter had passed over with the acid; and, in the frequent distillations of the sedative salt, had been deposited upon it, and thus deflagrated. But, on throwing fresh pulverized or solid manganese, either such as is usually sold, or quite pure, heated to redness, into melted nitre, not the smallest deflagration took place.

Exp. XXXVI. to L. Instead of the interrupted heat used in the foregoing experiments, I now exposed half an ounce of the salt, with three ounces of the oxygenated muriatic acid, to a continued heat, of between 200° and 300° of FAHRENHEIT. The fluid had nearly evaporated in twenty-four hours. I changed the phial, towards the close of the operation, for another, that the former might be gently heated, and the fluid by that means be poured back, with the greater safety, upon the warm salt, through the tube of the retort. In this manner, during an uninterrupted fire of fourteen days, the acid was fourteen times distilled, and returned upon the salt. On the third day, yellow spots appeared. On the fourth, some particles of oil or fat were discovered, swimming on the surface of the fluid in the phial; which particles, after cooling and emptying the phial, adhered to its sides, so as to obscure its transparency. More or less of these oily particles were discovered in every successive opera-

tion; and the oily matter, adhering to the inside of the glass, increased considerably.

Exp. LI. When the fluid was distilled, the receiver was changed, and the fire increased. A considerable quantity of sublimate was obtained, pretty white in colour, as was likewise the surface of the mass of salt at the bottom of the retort; but, lower down, it was almost of a light ash-grey. After the sublimate ceased to arise, I diminished the fire.

Exp. LII. Upon the mass of the former experiment, I poured the fluid obtained by *Exp.* XLIX. and continued a gentle digestion. I very soon perceived something rising towards the surface, and swimming upon it: after some hours, it appeared to be a thick wrinkled skin, like fat, or a skin of mould, increasing in size, until it covered the whole surface. White spots of sublimate appeared upon it, but it did not sink. It assumed gradually a fine lemon colour; and some yellow matter, though not in large quantity, ascended the sides of the retort. The fluid having been gently distilled, and the receiver changed, I placed the retort in an open fire; on which, more sublimate soon appeared; but, not long after, it all vanished, and the retort lost its transparency. The mass contained in it began to rise, first gently, and then violently, especially in the centre, in large frothy bubbles. The distillation was finished, after obtaining one dram of fluid, and when the frothy bubbling had ceased. The retort being broken, that part where the bubbling had been strongest, was found to be black; the upper surface being covered with a thin greyish matter, under which a solid, compact, and almost vitrified substance appeared. Upon this I poured water, and dissolved it in the usual manner; filtered it,

let it evaporate, and treated it as described above, *Exp.* XXII.—XXX.

Exp. LIII. I obtained a white salt, *a*, and some coal, *b*, (which deflagrated briskly with nitre,) in nearly the same proportions as throughout the series of experiments described from *Exp.* XXII. to XXXIII. which I will not repeat, on account of the little variety observed in them; one of them, however, deserves to be distinguished from the rest.

Exp. LIV. I put six grains of the coal, *b*, (of *Exp.* LIII.) in three drams of common muriatic acid, and digested them for two days, till the acid had gradually evaporated. I then added one dram of the same acid, with one scruple of nitric acid, and, when they had evaporated, boiled the residuum full half an hour in distilled water. By this process, I obtained a red solution; and, having saturated it with mild alkali, a sort of skin rose to the surface, with some small pieces of a fat slippery substance, *a*. A considerable quantity of loose earth, *b*, was also precipitated, of a light brown colour.

Exp. LV. On throwing the floating pieces, *a*, (*Exp.* LIV.) into a solution of caustic alkali, they dissolved; the solution had a reddish-brown colour.

Exp. LVI. With the same solution of caustic alkali, I covered the light brown earth of *Exp.* LIV. As the solution changed its colour to a reddish brown, the earth gradually became perfectly white.

Exp. LVII. To observe the affinity of other acids to the sedative salt, I poured six drams of nitrous acid upon two drams of the salt, with ten drams of the forementioned oxygenated muriatic acid; digested the mixture, and distilled it, in twenty-four

hours, with a gentle heat. Upon the fluid swam a white compact substance, and some small particles of the same kind lay at the bottom, which however rose, on the application of heat, and swam about with the rest.

Exp. LVIII. to LXIII. I poured the whole distillation back upon the salt, and, by means of a digesting heat, again drew off a fluid, which appeared covered with a thin fat skin. I then poured the fluid back, distilled it again, and thus repeated the process three times more. No phænomenon particularly remarkable appeared, except that the thin fat skin grew more inconsiderable, and at last seemed almost to vanish.

Exp. LXIV. The salt separated from the fluid, by the gentle distillation in *Exp.* LXIII. emitted now, by the force of additional heat, dark red vapours, as is usual in strong nitrous acid. When the distillation was at an end, the retort was exposed to an open fire; but, during this operation, no black matter appeared; nor was any coal separated from the mass, upon dissolving it in distilled water.*

Exp. LXV. I now tried the effect of a mixture of four drams of strong vitriolic acid and twelve drams of the muriatic acid, repeating the usual digestion and distillation six times. I will pass over other circumstances, and only mention, that after the sixth distillation of the fluid, a stronger heat, and at length an open fire, was applied; but hardly any fluid was produced, though the fire was so violent, that the whole mass appeared to be melted down into one uniform compact substance.

Exp. LXVI. The vessels having cooled, the mass was of a

* Here the nitrous acid seemed to destroy, and carry off, the inflammable matter, sooner than it could become coal; as it had before occasioned the oily and fat substance to vanish, in the beginning of this experiment.

light milky colour throughout, without the least mixture of brown or black, or any other indication of coal.* Being some time exposed to the air, it became moist, and for a long time attracted much water, which I caused to run off. At last it remained pretty dry; but the mass seemed to have diminished, by at least one-fourth part.

Here I will stop, for the present, in the description of my experiments, which sufficiently tend to prove, in a general way, the decomposition of sedative salt, and to show, that one of its component parts is inflammable matter, which may be converted into coal. I obtained of true coal, (mixed with some earth, *Exp.* XXXII. and LIV.) according to the above-described experiments, (*Exp.* XXII. XXVI.—XXX.) thirty grains and three quarters, in the whole; and by other experiments, often repeated, in general, one grain and a half, more or less. Every other substance liable to be changed into coal, (as gum, tartar, sugar, &c.) suffers this change by a gentle heat, and deflagrates with nitre, in the degree of heat necessary to melt the former. But sedative salt can bear a red heat for many hours, without shewing any signs of becoming coal, of burning, or of deflagration. Astonishing phænomenon! What menstruum preserves it so securely against the assault of force, in a dissolved state, and yet suffers itself to be separated from it by more gentle means? What power exists here, to protect the inflammable particles (which afterwards turn to coal) so effectually against a degree of heat which nothing else can resist? Of what nature is the

* Perhaps here also the remark contained in the former note holds good: yet I am rather of opinion, that the vitriolic acid did not operate with sufficient strength to separate the component parts.

salt obtained in conjunction with the coal? These are all questions which excite great interest, but which are not easily answered. How far I have been successful in resolving them, some subsequent Essays will show; which I shall have the honour to lay before the Royal Society, as soon as I shall have sufficiently repeated the experiments I have already made.