



# XXIX. On the use of hydriodic salts as photographic agents

Mr. Robert Hunt

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XXVIII. *Mineralogical Notices.* Communicated by W. H. MILLER, Esq., Professor of Mineralogy in the University of Cambridge.

[Continued from p. 105.]

ANALYSIS OF MONAZITE.

[From Poggendorff's *Annalen*, vol. xlvii. p. 385.]

ONE hundred parts of Monazite, the Mengite of Mr. Brooke, analysed by M. Carl Kersten, gave

Oxide of cerium .....	26·00
Oxide of lantanum .....	23·40
Thorina .....	17·95
Oxide of tin .....	2·10
Protoxide of manganese .....	1·86
Lime .....	1·68
Phosphoric acid .....	28·50
Traces of potash and titanac acid.	

ANALYSES OF OCTAHEDRAL COPPER PYRITES. BY M. PLATTNER.

[From Poggendorff's *Annalen*, vol. xlvii. p. 351.]

From Condurrow Mine near Cam- borne, in Corn- wall.	From the Woitzki Mine near the White Sea.	From the Mår- tan mountain in Dalarne in Sweden.
Sulphur... 28·238	25·058	25·804
Copper ... 56·763	63·029	56·101
Iron ..... 14·843	11·565	17·362
	Silica...	0·120
From Eisleben.	From Sange- hausen.	Unknown locality. (Analysed by M. F. Varrentrapp.)
Sulphur... 22·648	22·584	26·981
Copper ... 69·726	71·002	58·199
Iron ..... 7·539	6·406	14·845

XXIX. *On the Use of Hydriodic Salts as Photographic Agents.*

By Mr. ROBERT HUNT.\*

To the Editors of the *Philosophical Magazine and Journal*.

GENTLEMEN,

I HAVE been engaged more than twelve months in studying the peculiarities of the salts of hydriodic acid, when used for the production of those photographic pictures which are formed by one operation, having their lights and shadows correct as in nature; and the results of my very numerous

\* Communicated by the Author, whose former papers on Photography will be found in vol. xvi. p. 138, 267: see also our report of the proceedings of the Royal Society for the past session.

experiments are, the establishment of fixed principles, which remove most of the uncertainty attending the use of the hydriodates, an explanation of many of the anomalous results they give, and the discovery of some very remarkable properties not before noticed.

The uncertainty attending the application of the hydriodates, has greatly circumscribed their use, and it is the desire of forwarding the progress of a beautiful art, which now makes me solicit a few pages of your valuable *Journal*.

Sir John Herschel, in his very excellent memoir "On the Chemical Action of the Rays of the Solar Spectrum," &c.\* particularly notices the inconstancy of the effects exhibited by the hydriodates. "Nothing," says that talented and indefatigable inquirer, "can be more variable and capricious than the results obtained according to the different intensities of the solutions applied; the qualities of the paper; the degree of darkening induced on the paper before the application of the ioduretted solution, the state of the paper as to moisture or dryness, and other circumstances."

That the various positions I wish to establish may be completely understood, and to ensure the same results in other hands, it will be necessary to enter into a somewhat detailed account of several kinds of paper which have been used, and to give tolerably full directions for successfully using the same, either in the camera obscura, or for drawings by application.

1. *The preparation of the paper*.—The variable texture of even the finest kinds of paper occasioning irregularities of imbibition, is a constant source of annoyance, deforming the drawings with dark patches, which are very difficult to remove; consequently my first endeavours were directed to the formation of a surface on which the photographic preparations might be spread with perfect uniformity.

2. A variety of sizes were tried with very variable results. Nearly all the animal glutens appear to possess a colorific property, which may render them available in many modifications of the processes published by Mr. Talbot, but they all seem to protect the darkened silver from the action of the hydriodic solutions. The gums are acted on by the nitrate of silver and browned, independent of light, which browning considerably mars the effect of the finished picture. It is a singular fact that the tragacanth and acacia gums render the drawings much less permanent. I therefore found it necessary for general practice to abandon the use of all sizes, except such as enter into the composition of the paper in the manufacture.

[\* An abstract of Sir J. Herschel's paper appeared in vol. xvi. p. 331.]

3. It occurred to me that it might be possible to saturate the paper with a metallic solution, which should be of itself entirely uninfluenced by light, on which the silver coating might be spread without suffering any material chemical change. The results being curious, and illustrative of some peculiarities to be explained when the hydriodates come under our examination (65.), I shall record a few of them.

4. *Sulphate and Muriate of Iron*.—These salts, when used in certain proportions, overcame many of the first difficulties, but all the drawings on papers thus prepared faded out in the dark.

5. *Acetate and Nitrate of Lead*.—The salts of lead I have since perceived have been used by Sir John Herschel with success in some of his *negative* processes. I found a tolerably good result when I used a *saturated* solution of the above-named salts; but papers thus prepared required a stronger light than other kinds to give good results; when I used weaker solutions the drawing was covered with black patches. On these a little further explanation is required. When the strong solution has been used, the hydriodic salt which has not been expended in forming the iodide of silver, which, it is well known, is the lights of the photograph, goes to form an iodide of lead. This iodide is soluble in boiling water, and is thus easily removed from the paper. When the weaker solution of lead has been used, instead of the formation of an iodide, the hydriodate exerts one of its peculiar functions in producing an oxide of the metal (65—67.).

6. *Muriate and Nitrate of Copper*.—These salts, in any quantities, rendered the action of the hydriodates very quick, and when used in small portions appeared to promise much assistance in quickening the process; but experience has shown their inapplicability, the edges of the parts in shadow being destroyed by chemical action.

7. *Chloride of Gold*.—I did not anticipate much from the use of this salt. On trial it was found to remain inactive until the picture was formed, when a very rapid oxidation of the gold took place, and a consequent darkening of all the bright parts (5.) (65—67.).

8. Chloride of platina was found to act in all respects similarly to the chloride of gold, the re-darkening of the lights being much more rapid and intense (5. 7. 67.).

9. A very extensive variety of preparations were tried with like effects, and I was at length convinced, that the only plan by which a perfectly equal surface could be obtained, without impairing the sensitiveness of the paper, was careful manipulation with the muriated and silver solutions.

By attention to the following directions, simple in their character, but arrived at by a long series of inquiries, any one may prepare photographic papers, on which the hydriodic solutions shall act with perfect uniformity.

10. Soak the paper for a few minutes in a muriated wash, removing with a soft brush any air-bubbles which may form on it. The superfluous moisture must be wiped off with very clean cotton cloths, and the papers dried at common temperatures. When dry, the paper must be pinned out on a board, and the silver solution spread over it boldly but lightly, with a very soft sponge-brush. It is to be instantly exposed to sunshine, and, if practicable, carried into the open air; as the more speedily evaporation proceeds, the less does the silver penetrate the paper, and the more delicate it is. The first surface is very irregular, being made up of blue streaks, which are parts on which a true chloride is formed; and of brown ones, which appear to be the chloride of silver combined with a portion of undecomposed nitrate. As soon as the surface appears dry the silver solution must be again applied as before, and the exposure repeated. It must now be exposed until a fine chocolate-brown colour is produced equally on all parts of the surface, and then, until required for use, carefully preserved from the further influence of light.

11. In darkening these papers, the greatest possible attention must be paid to the quantity of light to which they are submitted, everything depending on the rapidity of the blackening process. The morning sun should be chosen, it being very evident that some portion of the violet rays are absorbed by the atmosphere after the sun has passed the meridian, which permeated it freely before he had arrived at that point.

A perfectly cloudless sky is of great advantage. The injurious consequence of a cloud obscuring the sun during the last darkening process, is the formation of a surface which has the appearance of being washed with a dirty brush. This is with difficulty removed by the hydriodates, and the resulting pictures want that clearness which constitutes their beauty. Papers darkened by the diffused light of a cloudy day are scarcely, if at all acted on by these salts.

12. The kind of paper on which the silver is spread, is an object of much importance. A paper known to the trade as satin-post, double-glazed, bearing the mark of J. Whatman, Turkey Mill, is decidedly superior to every other kind I have tried.

The demy printing papers are many of them bleached by chlorine, after an artificial substance has been given them by lime. These reverse the photographic process, and the parts

on which the light acts with the most power become the darkest of the drawing, while the shaded parts are whitened.

The dark specks which abound in some kinds of paper must be avoided, and the spots made by flies very carefully guarded against. These are of small consequence, indeed are not noticed during the darkening action; but when the hydriodic wash is applied they form centres of chemical action, and the bleaching process goes on around them independent of light, deforming the drawing with small rings, which are continually extending their diameters.

19. *The Muriated Solutions.*—These saline washes may be considerably varied, and combined to an indefinite extent with a continued change of effect, which is singularly interesting. In their application I am invariably guided by the combining proportion of the salt; for having tried solutions of all strengths, I am at length satisfied no other proportions give such certain results; consequently I always work with my scale of equivalents at hand. The following is a list of the salts I most frequently use, selected from upwards of seven hundred combinations which I have tried. They are placed in the order of sensitiveness they appear to maintain, when used under as nearly as possible the same circumstances.

Colour of Picture.

a. Muriate of ammonia ...	{ Red, changing to black in sunshine.
b. Chloride of sodium ....	Ditto.
c. Muriate of strontia ....	Brown, changes but slightly.
d. Muriate of baryta.....	{ A rich brown inclining to purple, darkens slightly.
e. Sol. chloruret of lime .	Very red.
f. Sol. chloruret of soda .	Red, changes a little.
g. Iodide of potassium ....	Yellow brown.
h. Chlorate of potassa ....	{ Variable, sometimes yellowish, often of a steel blue.
i. Phosphate of soda.....	Mouse colour.
k. Urate of soda.....	Yellow brown.
l. Muriate of iron .....	Deep brown, blackens.
m. Bromide of sodium ....	Red brown.

The change I mention in the colour of the finished picture is that which arises from a fresh exposure to the solar rays; where no change is mentioned, it is too slight to be worth notice. This phenomenon will, however, occupy our attention presently (38.). In addition to the salts named I sometimes use

## Colour of Picture.

- |                                  |                        |
|----------------------------------|------------------------|
| <i>n.</i> Hydrochloric acid .... | Red which blackens.    |
| <i>o.</i> Hydrochloric æther ... | Black.                 |
| <i>p.</i> Aqueous chlorine ..... | Red, deepens a little. |
| <i>q.</i> Phosphoric acid .....  | Very variable.         |

14. When papers prepared with any of the above, except *i* and *q*, are soaked for a little time in water, and dried in the sunshine, the picture produced—it matters not what hydriodate is used—is rendered peculiarly red, and does not change by re-exposure: washing either of the papers *b*, *c*, or *d* with weak solution of ammonia, occasions this peculiarity in a striking manner.

15. *The Solution of Silver.*—Take of crystallized nitrate of silver 120 grains, distilled water 12 fluid drachms; when the salt is dissolved, add of alcohol 4 fluid drachms, which renders the solution opaque. After a few hours a minute quantity of a dark powder—oxide of silver?—is deposited, and must be separated by the filter.

16. The addition of the alcohol to the solution was adopted from an observation I made of its influence in retarding the chemical action of the hydriodates on the salt of silver, which goes on in the shade. Its use is therefore to make the action depend more on luminous influence than would be the case without it.

17. Nitric æther and acetic æther not only check the bleaching process in the shade, but actually act with the hydriodic salts in exalting the oxidation of the silver. In copying lace or feathers, they are very valuable agents, but for any other purposes they are useless, as all the faintly lighted parts are of the same tint.

18. The hydrochloric æther, which I use as the solvent of the silver, and apply without any saline wash, has a similar property to the nitric; but as it is readily affected by faint light, it is of greater value. However, papers prepared with it must be used within twenty-four hours, as after that they quickly lose their sensitiveness, and soon become nearly useless.

19. *The Hydriodic Solutions.*—To fix with any degree of certainty the strength of the solution of the hydriodic salts, which will in all cases produce the best effect, appears to me impossible; every variety of paper, either as regards its composition, or the intensity of light to which it has been exposed to darken, requiring a solution of different specific gravity.

20. *Hydriodates of Potassa and Soda.*—The former of these salts being more easily procured than any other of the

hydriodates, is the one generally employed. The strength at which I use these salts for most kinds of paper is thirty grains to an ounce of water. The following results will exhibit the different energies manifested by these solutions at several strengths, as tried on the same paper by the same light.

120	grains of salt to an ounce of water	} 12 minutes.
	took to whiten .....	
100	do. to do.	10 —
80	do. to do.	9 —
60	do. to do.	7 —
40	do. to do.	6 —
30	do. to do.	4 —
20	do. to do.	6 —
10	do. to do.	12 —

The other hydriodic salts correspond nearly with these in their action; a certain point of dilution is necessary with all.

21. Hydriodate of ammonia, if used on unsized paper, has some advantage as to quickness over either the salts of potassa or soda. This preparation is, however, so readily decomposed, that the size of the paper occasions a liberation of iodine, and the consequent formation of yellow-brown spots.

22. *Hydriodate of Iron*.—This metallic hydriodate acts with avidity on the darkened paper; but even in the shade its chemical energy is too great, destroying the sharpness of outline and impairing the middle tints of the drawing. It also renders the paper very yellow.

23. *Hydriodate of lime* acts similarly to the iron, but less energetically, and the paper is not rendered yellow by it.

24. *Hydriodate of manganese* answers remarkably well when it can be procured absolutely free of iron. When the manganetic solution contains it, even in the smallest quantities, light and dark spots are formed over the picture, which give it a curious speckled appearance.

25. *Hydriodic acid*, if used on paper which will not decompose its aqueous solution, acts readily on the darkened silver. It is difficult, however, to procure a paper which does not liberate the iodine. A portion of hydriodic acid, free, in any of the saline solutions, greatly quickens the action.

26. *Hydriodate of baryta* possesses advantages over every other simple hydriodic solution, both as it regards quickness of action, and the sharpness of the outline in the photograph.

27. I find, however, the quickness of this solution may be much increased. Forty grains of the hydriodate of baryta being dissolved in one ounce of distilled water, thereto should be added five grains of pure sulphate of iron, and allowed slowly to dissolve. Sulphate of baryta is precipitated, which



should be separated by filtration, when the solution is composed of the hydriodate of baryta and iron. By now adding a drop or two of very dilute sulphuric acid, more baryta is precipitated and hydriodic acid left free. The clear solution must be decanted off, as the filtering through paper decomposes the acid. By this means a photographic fluid of great value is formed. It should be prepared in small quantities, as it suffers decomposition under the influence of the atmosphere and of light. It is always easy to set hydriodic acid free by precipitating sulphate of baryta.

28. *Directions for taking Photographs.*—For drawings by application less care is required than for the camera obscura. With a very soft flat brush apply the hydriodic solution on both sides of the prepared paper until it appears equally absorbed, place it in close contact with the object to be copied, and expose to sunshine. The exposure should continue until the light parts of the picture (iodide of silver (54.)) are seen to brown. The observance of this simple rule will be found of very great advantage in practice. Immersion for a short time in soft water removes the brown hue, and renders the bright parts of the picture more clear than they would otherwise have been.

29. If the paper is intended to be used in the camera, it is best to soak it in the hydriodic solution, until a slight change is apparent from the chemical action on the silver; it is then to be stretched on a frame, and not allowed to touch in any part but at the edges; placed in the dark chamber of the camera at the proper focus, and submitted to luminous influence.

If the wetted paper is placed upon any porous body, it will be found, owing to the capillary communication established between different points, that the solution is removed from some parts to others, and different states of sensitiveness induced. Another advantage of the frame is, the paper being by the moisture rendered semi-transparent, the light penetrates and acts to a greater depth, thus cutting out fine lines which would otherwise be lost. However, if the camera is large, there is an objection to the frame; the solution is apt to gather into drops, and act intensely on small spots to the injury of the general effect. When using a large sheet, the safest course is to spread it out when wetted upon a piece of very clean wet glass, great care being taken that the paper and glass are in every part in close contact. The picture is not formed so quickly when the glass is used as when the paper is extended on a frame, owing to the evaporation being slightly retarded; the additional time required, about one-

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sixth longer, is however in most cases of small consequence. It is somewhat singular that if the glass plate is interposed between the paper and the lens, the action is not more retarded than if it had been placed behind it. The interference of a transparent plate is little felt in the hydriodic process.

30. *On fixing these Photographs.*—The picture being formed by the influence of light, it is required, to render it unchangeable by any further action of the luminous fluid, not only that the hydriodic salt be entirely removed from the paper, but that the iodide of silver which is formed be also dissolved out of the drawing.

31. By well washing the drawing in warm water the hydriodate is removed, and the pictures thus prepared have been stated to be permanent; and if they are kept in a portfolio, and only occasionally exposed, they are really so; for I shall show presently (54.) that they have the property of *being restored in the dark to the state in which they were prior to the destructive action of light.* I have now before me the first drawing of this kind I ever executed, bearing the date June 17, 1839. This drawing has been kept loosely in my table drawer, and has often been exposed for many successive days to the action of the sun; yet the most delicate venations of the rose leaves are as perfect as at first. Thus prepared, however, these photographs will not bear continued exposure without injury, about three months in summer, or six weeks in winter being sufficient to destroy them.

32. For a long period I was under the impression that two iodides of silver existed, the one sensitive to solar influence, but the other not so; and in my paper published in your Magazine for April, I stated such to be my opinion. I have, however, since that period seen reason sufficient to question the correctness of my conclusion. Under the former impression, not being successful in removing the iodide from the paper without also injuring the oxidized or dark portions, I endeavoured to effect a chemical change in the iodide of silver. Some of the results being curious, I shall give them.

33. By washing the photograph with a hot saturated solution of the acetate of lead, the yellowness of the lights was at first increased, but eventually considerably whitened, and the dark parts assumed a peculiar crimson hue. The drawing faded out entirely by the action of light in three weeks.

34. When these drawings are dipped into a solution of the bichloride of mercury, they fade out in precisely the same manner as Sir John Herschel discovered the photographs on Mr. Talbot's principle were obliterated, and in like manner

are they restored by a liquid hyposulphite; the paper, instead of being completely white, being altogether of a full rich yellow. When these photographs are restored by the hyposulphite, they are even less permanent under the influence of light than those washed with the salt of lead.

35. The ferrocyanate of potassa exerts no action on these photographs in any way remarkable, unless they have been formed by the agency of the hydriodate of iron (22.) or of baryta and iron (27.). They are then obliterated by it, but on exposure, the light parts of the picture are darkened, changing thus to a *negative* photograph, the originally dark parts being now a light blue.

36. With much attention, I have tried the hyposulphites of soda, ammonia and potassa. But I have failed to remove all the iodide of silver, without destroying at the same time the dark parts, and the minute portion which remains in the paper is very soon darkened by light to a tint similar to the lighter shades of Indian ink. When first done the drawing is much improved in appearance, but it is difficult to remove the hyposulphite so completely as is necessary to prevent the formation of the sulphuret of silver.

37. Sulphuretted hydrogen gas, which has the singular property of blackening the iodide of silver, when in that state which is easily darkened by light, *but of bleaching it in the less susceptible state*, acts on these photographs in a manner similar to the hyposulphites; but the oxidized portions of the picture are first destroyed and then restored by light. The light parts are, however, rendered brown.

I have tried a great variety of other agents, diversifying my method of using them in almost every possible way, but as yet I have discovered no material which effectually removes the iodide of silver alone; consequently I satisfy myself with well washing my photographs in hot water.

[To be continued.]

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XXX. On the Anthracite Coal of South Wales. By SAMUEL WOODS, Esq., F.G.S.

To the Editors of the Philosophical Magazine and Journal.

GENTLEMEN,

HAVING received from a friend at Paris the accompanying analysis of some of the anthracite coal found abundantly in this neighbourhood, I transmit it to you in the belief that it may be worthy of record in your pages; and the quality and uses of this coal having lately excited so much