THE TRANSMUTATION OF ELEMENTS.

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bу

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The first scientifically established transmutation of a chemical element, performed by artifical means, is due to Rutherford who showed in 1919 that it is possible to disintegrate nitrogen atoms by bombarding them with high velocity \propto -particles. These projectiles form a very powerful energy agent, and when they hit a nitrogen nucleus under suitable conditions, they knock out a hydrogen nucleus. Later investigations have shown that a number of elements can be disintegrated by this procedure.

But there had already been announced other ways of transmuting elements, where the products were rare gases and hydrogen, and rather recently it has been claimed that an artificial disintegration of heavier elements is possible. In these cases, however, there is no powerful energy agent present like the α -particles, as far as we know. This leads to the assumption that if the reality of these transmutations could be established, one would have to look for a different mechanism of the process than in the case where α -particles are the active agent. This makes the reports about these transmutations rather doubtful, but if the importance of such a process is taken into consideration it seemed to be worth while to check up on these experiments.

Before entering into a very brief review of this kind of transmutations, it must be pointed out as a general feature by almost all of them, that the amount of transmuted material is exceedingly small, so that even if the analytical detection is rather certain, there is always a great danger that the transmutation product is present in the various materials used in the experiment.

In the case where the products are gases, the experiments go

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back to 1912, when Ramsay announced the finding of He and Ne in

discharge tubes. A continuation of this work, carried out by Collie

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and Patterson gave He, Ne, and H. These exepriments were, however,

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repeated by several workers with negative results. In the mean
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time, Collie, Patterson, and Masson got more positive results.

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Recently experiments of Riding and Baly seemed to show that the

transmutation was due to a disintegration of films of nitrides on

the electrodes, as suggested to them by Masson.

In the class of transmutations of heavier elements, the experi6)
ments of Miethe have attracted much attention. Miethe and his
assistant Stammreich had been working with a new kind of mercury
arc lamp, but had been bothered by the formation of a dark layer on
the inside of the tube. A chemical analysis of the layer showed
traces of gold, which according to Miethe's opinion could not
originate from the mercury. Miethe and Stammreich then performed
numerous experiments, both with this mercury lamp and with other
kind of discharges, and got gold in most of them. The amounts in
question are rather small, under favorable conditions he got 1/10 mg
to the kilogram of mercury in the lamp.

A number of workers have tried to repeat Miethe's experiments, but none have had success. Miethe himself has been quite unwilling to admit the possibility of errors in his work and has attacked his

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assailants rather vigorously . The argument which always comes back is, that it is necessary to have quite definite working conditions, e.g. a special kind of discharge and a definite shape of the tube, which the other workers in the field have not been able to reproduce. If one assumes that such special conditions are essential for the success of the transmutation, it is extremely difficult to refute Miethe. The most plausible explanation for the occurrance of gold is contamination of the materials, especially the mercury beforehand. Haber, Jaenicke, and Matthias have especially pointed out, how extremely careful one must be to avoid dust and vapors which contain the substances one is looking for.

Nagacka reports that he has performed a transmutation by passing high potential discharges between a tungsten electrode and a mercury surface in a vessel filled with liquid paraffin. The power was furnished by an induction coil, capable of giving a 120 cm spark, and the current in the primary was 25-30 amp.

The most remarkable among the numerous investigations that

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Miethe's work gave rise to, is the experiment of Smits and Karssen,
if it could be checked. Smits has for a number of years been
interested in the idea of allotropy, which in his terminology means
that a socalled simple substance as for instance one of the metallic
elements, consists of electrons, atoms, and ions in different stages
of ionization, forming a dynamic equilibrium. By applying violent
means, for instance an electric arc, in order to disturb the equilibrium, deeper layers of electrons in the atom, perhaps even the
nucleus, could be affected. Smits has tried several different
constructions of electric arc lamps where lead vapor acted as the

conductor, in analogy with the conditions in a mercury arc lamp, and got strong positive evidence of a transmutation. His results are in a class by themselves for the following reasons: The transmutation products, mercury and thallium, are found by taking the spectrum of the arc, keeping the lead in the same tube in which the experiment had been performed, without touching it. The other remarkable thing is that the mercury is so abundant, that Smits and Karssen think that if the lamp had a long enough life time, a point could be reached when the lead spectrum would have disappeared entirely.

As will be seen from the foregoing, it is very important if one chooses to work in the field of transmutations, to select experimental conditions where there seems to be ample and indisputable evidence of transmutation, and where the chances of containnation are the least.

An agent which might be active in the reported transmutations is the electron, and Dr. R. A. Millikan suggested to try the X-ray tube, where high velocity electrons impinge upon a target. If any appreciable amount of matter was transformed, it would show up in the characteristic X-ray spectrum. The evidence one might get here is indisputable, and as the tube remains closed all the time, the danger of accidental contamination should be rather small.

The experiment was carried out in the following way: An unused, large deep-therapy X-ray tube from Victor Corporation was put to our disposal by the kindness of Dr. A. Soiland in Los Angeles. To ascertain that no other lines except those of tungsten were present to start with, the characteristic K-spectrum was photographed, applying the Bragg principle: same distance between crystal and slit as between crystal and film. The crystal used was a calcite cleavage

plate which was turned very slowly by means of a clockwork, a very important precaution when one does not have a very good crystal. If this is omitted, the background of the lines does not become uniform, and pseudo-lines will appear. The current used was taken from a transformer and rectified by one kenotron tube. An autotransformer and resistances were used for regulation.

An exposure with 25 cm distance between crystal and film using 83 kV and 2 mA gave a fairly good film; a new exposure under the same conditions, but with 83-85 kV gave a better film. No other lines than those of tungsten could be detected. The last exposure was taken October 16, 1925, and the tube returned to Dr. Soiland and run by him for about 80 hours at 2-3 mA and 207 kV peak voltage. A new picture was then taken on February on February 3, 1926 under the same conditions as before. No lines that indicated a transmutation could be found. Later, the author has had the opportunity of seeing a number of spectral photographs, taken with a Zeeman spectrograph of an X-ray tube which had been running for several hundred hours at around 200 kV. No foreign lines could, however, be detected.

As the next experiment was chosen that of Smits and Karssen for the reasons mentioned above. Their first construction consisted of a quartz tube shaped like a Y, turned upside down. Through the two legs were stuck the electrodes, and the arc was maintained through the connecting tube. Such a tube was also built by the author, but failed to work satisfactorily. In the meantime, Smits and Karssen had changed their construction, and the tube which the author built and ran successfully, was as nearly as possible a copy of the tube applied by them. The size, however, was rather much smaller due to the difficulty

of judging the size from their drawings and photographs. The justification for copying their tube as closely as possibly lays in their statement, that small changes in the experimental conditions may almost or entirely prevent the transmutation from taking place.

The tube used by the author is made of transparent quartz and has the following shape (see fig. 1)

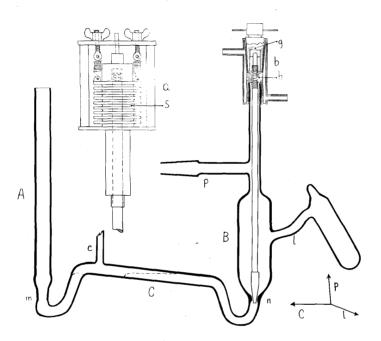


Figure 1

It consists of the two vertical legs A and B, and the connection C between them, in which the arc burns. In the two legs are inserted the rod-shaped electrodes, fastened to the holders a (drawn separately) and b, which close the tube on top. The side tube 1 on B carries a container from which the lead used in the experiment can be poured over in the main tube. Another side tube p connects the tube to the oil pump by means of a glass tube (not drawn), which is attached to the quartz tube by a cooled ground joint sealed with wax. On this

glass tube are two stopcocks, and between them is a good sized charcoal tube. In the drawing, the side tubes p and l are bent so that they lie in the plane of the paper; in reality they protrude as in the end view as sketched on the side. C has a vertical side tube c which is closed by a window at the top.

The electrodes were first made of invar which was later exchanged for steel, as invar dissolves in the lead. The lower ends carry carbon points, which are turned in the shape of cones to fit the ground restrictions m and n in the quartz tube. To lift or lower the electrodes, the following arrangement was used. In the lower end of the ground brass cone g on b is drilled a hole, along which two parallel grooves are cut. The top of the electrode is pierced by a removable pin and can slide in the hole. The pin fits in the grooves and thus the electrode is compelled to follow when the cone is turned. This brings about a vertical displacement of the electrode by means of the threaded part h. The holder itself which is water cooled, is attached to the tube by means of black sealing wax. This arrangement was originally used for both holders but the strong heating of the electrode in A brought about some evaporation of the grease on the cone, and this holder was modified in the following way, as shown in the figure: The threaded part inside the holder was taken away, the electrode exchanged for a longer one which is screwed into a water cooled brass cylinder on top of the syphon s. The syphon itself is supported by two rods connected by a yoke and can, together with the cylinder and the electrode be lifted and lowered by means of an arrangement of two screws and two winged nuts.

On figure 2 can be seen the tube mounted on a rectangular frame which is fastened to a heavy tripod by means of a bearing a and an

arm 1. This arm is excentrically connected to a pulley b, the axle of which is rigidly attached to the tripod. When the pulley is turned the frame is set in a rocking motion. The tube is fastened to the upper side of the frame and arranged so that it can be heated by means of 5 bunsen burners screwed on to a main tube which turns the lower side of the frame. To the frame are also fastened two copper tubes (not drawn), each having a row of fine jets directed against the quartz tube, through which compressed air is blown to cool the tube. Under the upper part of the frame is fastened a sheet of asbestos to protect the sealing wax on the electrode holders from the heat of the gas burners.

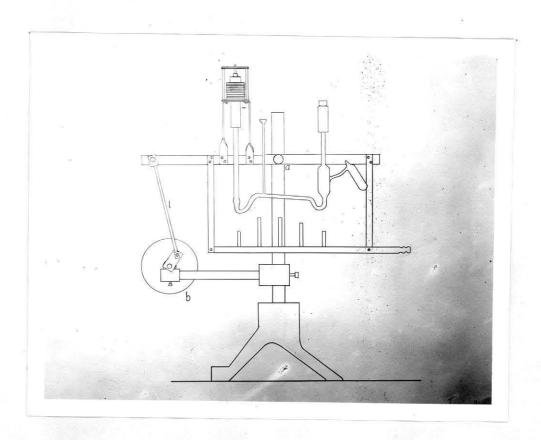


Figure 2

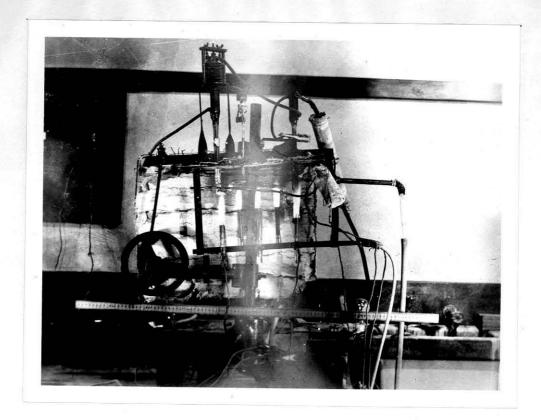


Figure 3 is a photograph of the tube mounted.

For the photographing of the spectrum is used a big Hilger quartz spectrograph. The light is either taken out directly and by means of of a quartz lens focussed on the slit, or the light coming through the window on the vertical side tube is reflected by means of a stainless steel mirror, and then prought to focus by the lens. Panchromatic plates from Wratten and Wainwright were used.

Great difficulties were experienced in preparing a pure lead.

Several different methods were tried out. The best one was found to be to recrystallize lead nitrate, c.p., two times, heat it in a quartz tube to form lead oxide, and reduce this in the same tube by means of hydrogen. Still, this did not give lead that was free from mercury. Finally the lead was sent for which the firm C. A. F. Kahlbaum in Germany had prepared for Smits and Karssen, who had found it to be almost free from mercury. A spectrum of this lead was taken up, but gave strong lines of mercury and thallium, stronger than any of the

homemade preparations. As a matter of fact, this spectrogram was used as a reference spectrum when looking for thallium and mercury lines. The preparation of homemade lead was then resumed, and by distilling off 10-30% in high vacuum at 1000-1400° C the remaining lead would be spectroscopically free from mercury and thallium.

The procedure of starting the experiment was now as follows: The tube was first cleaned very carefully, and heated while an air current was passed through. The electrodes were also carefully cleaned, the carbon points initially by heating them at 700-800° C in a vacuum for several hours in a quartz vessel. The tube was then mounted, the electrode holders and the connection to the pump sealed on, and lead broug t into the side tube, usually in molten condition, and kept liquid there all the time by means of a little. electric furnace. The side tube was then sealed off and the pumping started, while the charcoal tube was heated. During the period of pumping, both electrodes were hoisted up, and in the latter part, the gas burners were lit, and the lead heated to 700-800° C. Then the further stopcock was closed, the charcoal tube cooled by liquid air for 1-2 hours, the long electrode lowered down so as to prevent the lead from getting into the leg A. The liquid air was now removed and at the same time the tube tipped so as to make the lead run into the main tube. The volume was usually regulated so that the lead filled up the tube as shown in fig. 1. A big direct current generator furnished the power, and after fastening on the electric leads, so that b is the positive pole, the arc can be started.

As has been mentioned before, the construction and the mounting of the tube is in as close agreement with the work of Smits and Karssen as could be judged at that time (fall 1926). According to

their statement, a low current density has no influence upon the spectrum, while a high current density may bring about a transmutation. They proved this by taking spectra before and after a run of 10 hours, at about 10 amp. No change in the lead spectrum could be observed. But by running the tube at 30-35 amp and about 80 volts for 6 hours, the mercury lines to appear, and after 10 hours burning, the strongest mercury lines could be seen very clearly, and also the characteristic thallium lines. This and other experiments showed, that high current density was very favorable for bringing about a transmutation, and they tried currents up to 60 amp. This was, however, rather hard on the quartz tube. Consequently they changed their working conditions, and the construction described above is very similar to theirs. idea is that the arc does not burn continuously, but "sparks" of high current density are formed when the lead surfaces are brought together by rocking of the tube. At make, the current in the tube was 60-100 amp, and at the break considerably more. When this procedure is applied, it is necessary to take the light out through the side tube, as a black film appears inside the tube that cuts down the intensity of the light. Working by this procedure, Smits and Karssen got strong evidence for a transmutation. Already after 9-1/4 hour of sparking all the mercury lines, even the very weak ones were present on the plate.

The author first made a run for 10 hours with continuous arc and low current density. The arc burns very nicely with a length of 5-7 cm at 12-14 amp, and 23-26 volts, and with some heating by the gas burners. No change in the spectrum was observed, in accordance with Smits and Karssen.

Then comes the question of running the tube with higher current densities. This means that the gas burners must be put out, and cooling

applied, so as to get rid of the heat developed in the arc. But the cooling must not be too strong, the lead must be kept liquid all the time. If it solidifies, it will mostly by cooling and invariably by reheating break the quartz tube. The temperature of the lead must then always be higher than 327° C, but not very much more, as a high temperature of the lead makes the arc too hot, and it will melt the quartz tube. High current density will then require strong cooling, but this can very easily bring about a breakdown if the current for some reason is interrupted. Below dull red heat (525°C) it is very difficult to judge the temperature, and it also must be taken into consideration that the temperature around the electrodes usually is lower than in the middle of the tube, due to loss of heat through the electrodes and the greater distance from the arc. After several trials, a run of 35-1/4 hours was completed. At the make of the arc the current was 60-75 amp for 14 hours, and 80-90 for 21-1/4 hours. Of this time around 20% was effective burning time for the short flashes. The amount of lead used in this experiment is only 180-200 grams, and it was found to be spectroscopically pure before the actual run was started. The spectra throughout the experiment were mostly photographed with 5 hours intervals under standard conditions with 12-14 amp and 25 volts in vacuum, and showed no mercury or thallium lines. On trying to run the tube with continuous current, it broke down, due to too strong cooling.

The tube was then tepaired, and a new run started with new lead, which, however, on very good spectrograms taken under the standard conditions, showed faint traces of mercury and thallium. Since Smits and Karssen had reported that they also had had success when the tube was filled with nitrogen, such an experiment was tried. The tube was first thoroughly evacuated and pure nitrogen let in, so that the

pressure was 1/5 - 1/7 of an atmosphere. The length of the arc was regulated by means of cooling and heating the charcoal tube, which then took up or gave off nitrogen, and by this change in pressure the level of the lead could be controlled.

First, an experiment with continuous current was performed for 25 hours, with current densities of from 15 to 25 amp., the latter value being maintained for 10 hours at about 38 volts. No appreciable change in the intensities of the mercury and thallium lines could be observed. In this experiment the lead has to be at a higher temperature to get the arc started, than when the arc is started in a vacuum. The discharge did not, as in a vacuum fill up the whole tube, but had a more definite arc-like form.

with the same tube, the method of sparking was again taken up. Accidentally it had been found that an oscillatory motion of the lead in the short leg of the tube can be established by a cercertain pressure. If the two inner lead surfaces are joined, an arc is formed which heats up and expands the nitrogen so that the distance between the two surfaces increases very rapidly, pulling the arc out to a length of 12 cm, where it breaks. Now the nitrogen between the two lead surfaces cools off, which brings about a new contact and a new arc is formed. These conditions were made use of in the following run, and did away with the rocking device.

The pressure in the tube was again regulated by means of the charcoal tube and the stopcock between them. The actual burning time of the intermittent arc was a little less than half of the t total time, and each flash lasted around half a second. After a run for 10 hours with a current of from 65 - 95 amp. in the make was performed, the scheme of connections was changed, putting a condenser over the terminals and a big inductance in series with

with the tube to protect the generator against transients. With this arrangement a twelve hour run was performed, raising the current to 120 amp. in the make, but no increase in the strength of the mercury and thallium lines could be detected. On increasing the current to 150 amp. the tube broke after $2\frac{1}{2}$ hours run, but there is no doubt that a new tube would stand the current for 5 - 10 hours.

As will be seen, the currents compare with and even exceed those used by Smits and Karssen, and as the diameter of the tube. where the arc is burning is only 3/8 in. the actual current densities are higher. On account of the smaller amount of lead used, (180 - 200 g, against \pm 900 g.) the expected products of transmutation should be more easily detected.

It has been mentioned before that Smits and Karssen have found that smaller changes in the experimental arrangements may destroy the transmutation. They have, however, also reported upon a second method of producing a transmutation which is analogous to Nagaoka's, and they state that by this method it is possible to get reproducible results. The process, according to Smits and Karssen's publication in Zeitschr.f. El. Chem. is as follows. (see fig. 4)

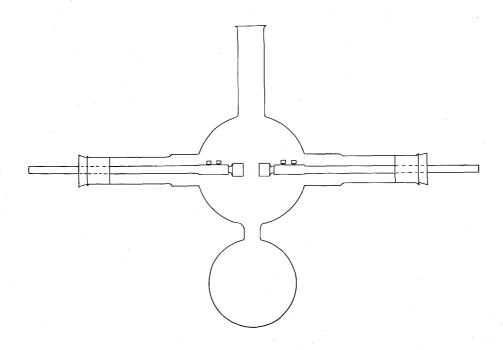


Figure 4

Two lead electrodes in steel holders are placed horizontal and opposite one another in a Pyrax flask. The holders are stuck through cork stoppers and are, on the other side, connected to the the electric leads. These stoppers close the two side tubes which are blown onto the flask. The neck is closed by a cork stopper, through which is stuck a long glass tube, which acts as an air cooler. The flask itself is filled with carbon bisulphide. High potential discharges are passed between the lead electrodes, the surface layers of which are torn off and go out in the solution as a very fine suspension which is precipitated by and by along the sides of the tube from where it flakes off and collects in the bottom. Besides, carbon is also formed by the disintegration of the carbon bisulphide. The precipitated material is collected and analysed chemically, and mercury is found. As will be seen, this methid is much less clean-cut than the previous ones, the as system is not quite closed, and the mercury is found by a rather complicated chemical analysis. Another objection to this method

is that it would be very difficult to perform a reliable analysis if one wanted to make sure that even small traces of mercury are absent in the carbon bisupphide used. Nevertheless, the experiment was tried.

A tube like the one drawn was made, but instead of steel holders, glasstubes filled with lead were used in the first experiment. The electrodes were cast in glass tubes, and the glass round the stems was simply left on. Instead of the smaller bulb underneath the main tube, in the first experiment a cylindrical container was used, similar to a test tube. Here the spattered lead is precipitated and accumulates. The current used was taken from a set of 3 transformers from the Roentgen manufacturing Co., San Francisco, one insulating transformer and two "boosters" furnished with a mechanical rectifier. Over the tube was put a screen cage for protection in case of fire.

Carbon bisulphide, quality c.p. from Baker, was used in the first experiment, after it had been dried over calcium chloride and re-distilled. In the later experiments Anchor Brand, commercially guaranteed not less than 99.9% CS₂ from Wheeler, Reynolds & Stauffer, San Francisco, was used. This product was distilled one time with a high rectifying column, dried over calcium chloride and again distilled in the same apparatus. The reason for using the commercial product is a statement in the literature that mercury is sometimes used for the preparation of c.p. carbon bisulphide, which then might be contaminated by mercury. In both distillations only 4/5 of the whole colume was driven over, assuming the heavier end fraction to contain possible mercury compounds.

The runs were usually of 5 - 12 min. length, separated by 20 - 40m interval to let the carbon bisulphide cool down. In this and the following experiments the current was from 20 - 40mA. The potential difference also varied considerably.

When measured by a sphere gap with 12.5cm. diameter of spheres, the distance between them was around 5.9 cm. when the discharge was going through the tube, chrresponding to 148kV peak voltage. During the run, when the discharge was going, the sparking distance was 5.8 cm, equal to 147 kV.

The first run lasted, when the intervals were added up, for 1 hour 20 minutes. The precipitated material was brought out of the tube and dried. It amounted to 7 g. but held a fair amount of sulphur. This, together with 3.5 g. lead, cut loose from the rough surface of the electrodes, was subjected to the same kind of analysis as employed by Smits and marssen. The method is maintly puilt upon a procedure described by Stock and Heller 1. The lead was brought into a quartz tube, which was being heated to 800-900°C. The hydrogen takes the mercury along with it to a double U-tube, made of quartz and cooled by liquid air. Here the mercury is condensed. After 4 hours run, the tube is broken off, and the distillate dissolved in a small volume of aqua regia, diluted, four drops of a copper nitrate solution added, neutralised by NH3 and precipitated by HoS.

The precipitate is then separated from the solution by centrifuging, washed with H₂S-water, stirred up in 3 - 4 cm. of water and dissolved by bubbling Chlorine at 80 -90°C through the little test-tube with the precipitate. A current of air is then passed through the solution to drive off the chlorine, the remaining yellowish sulphur filtered off and the dissolved content again precipitated with H₂S. This pracipitate is centrifuged, washed, brought into solution by chlorine, excess of chlorine driven off by air, and the solution filtered. Now, fine powdered ammonium oxalate is added in small portions, until the copper precipitate again dissolves, and a few crystals of ammonium oxalate are left in the bottom of the test-tube. In this clear solution of 4-5 cm

volume was dropped a double bent copper wire which had been heated almost to melting for several hours in an evacuated quartz tube. After 24 hours the wire was taken out, washed, carefully dried in a little tube over Po05, brought over into a little glass tube sealed off in one end. The other end was then drawn out in a capillary, and a moist piece of filter paper wrapped round the tube 3-4 cm from the copper wire. The end of the tube with the .. ire was then heated strongly to drive off the mercury, and finally the tube melted off behind the wire. mercury can come off in different forms. If it appears as fine droplets the amount can be much over estimated, while tubes which show very little on a superficial examination may be found to contain large droplets along the furrows in the glass, when examined under a microscope. To make quite sure that mercury is present, the capillary part is cut off and the tube is put together with a little crystal of iodine in a somewhat bigger tube, closed by a stopper. After 12-18 hours the iodine vapor has combined with the mercury, forming red crystalline mercury iodide, which is very easy to recognise and also permits a better judgment of the amount of mercury present. An amount of 1/100 of a milligram of mercury cannot be overlooked, and amounts of 1/1000 of a milligram of mercury as iodide can be rather easily distinguished under the microscope.

The result of the analysis of the first run was an amount of mercury between 1/10 and 1/100 of a milligram. To make sure that there was no mercury in the electrodes, they were analysed by melting them down and the lead heated to 800-900°C in a stream of hydrogen, proceeding the same way as with the analysis, but no mercury was found. Blind tests showed that no mercury comes in through the chemicals used, and numerous tests with solutions of 1/10 to 1/100 mg of mercury, treated as in the analysis, showed,

as far as could be judged, that these amounts could be recovered.

In the next experiment the above mentioned carbon bisulphide was used. To ascertain the purity of the lead, hydrogen was bubbled through it for 24 hours at 850°C and for 12 hours at 950°C. The purity was tested by heating it in the same vessel as used in the analysis, and proceeding in the same way. No mercury could be detected. Then the actual run was started and a total time of 2 hours was attained. The amount of precipitated material amounted to 4.5 grams, which, together with 3.5 grams from the electrodes was distilled and analysed as before. The result was a very evident positive mercury reaction, but the amount was much less than before.

At the same time, a purification of the German lead from Kahlbaum was carried out, by heating it for twenty hours at 950°C, while hydrogen was bubbled through. Then the previously mentioned double U-tube was melted on and an analysis performed, the difference from the previous analysis of lead for electrodes being that hydrogen actually bubbled through the molten lead. This analysis gave mercury, although not very much. This seemed to show that the procedure of leading hydrogen over the lead was an unreliable process of analysis. The electrodes used in the last experiment were then melted down, and an analysis performed by bubbling hydrogen through the liquid lead. This analysis, however gave no mercury.

Previous experiments had shown that it was possible to get rid of mercury by distilling it in a high vacuum. Consequently the German lead was subjected to such a process, distilling over about half of the amount in the containers. The residue was analysed by leading hydrogen close over the surface of the lead at 900° C. This analysis gave no mercury. With this lead a new run for $3\frac{1}{2}$ hours was performed, yielding about 11 grams of washed

precipitate. This was analysied in the usual way and gave only a slight mercury reaction.

The conclusion from these runs seems to be that with a sufficient purification of the materials used, it is possible to get
down in the amounts of mercury that are found, which again means
that the mercury originates from electrodes, carbon bisulphide
or dust coming in during the experiment. In a physical laboratory
it is very hard to find places which are free from mercury. All
dust must be suspected to contain mercury, which again means an
extremely strict cleanliness when it comes to handling things.
It must also be mentioned that red Dennison sealing wax contains
mercury as cinnabar, and red sealing wax is almost as common in
a physical laboratory as mercury.

If one wanted to make further tests concerning the transmutation of lead, it is the author's opinion that it ought to be done in a way whereby it would be possible to reach, so to speak, a higher order of magnitude of cleanliness. This would mean working in a building where no mercury had ever been spilled, or working in the open, starting from greater amounts of carbon bisulphide and lead and purifying by distilling many times. This would be a long and tedious piece of work but would perhaps for some time kill the reports on transmutation of elements by these processes where the energy seems to be so little concentrated.

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