tempt is made to exhaust the system to a very high vacuum. Great difficulty is experienced and there appears to be a slight leak, but if the liquid air is removed and shortly afterwards replaced, this difficulty at once disappears and the system can then be exhausted to a very high vacuum with ease.

Summary.

1. When a tungsten wire is heated to a temperature between 1300° and 2500° K. in hydrogen at very low pressure (0.001–0.020 mm.) the H_2 slowly disappears.

2. With N_2 or CO such disappearance never occurs below about 2200° K. and seems to be an electrical effect, while with H_2 it seems purely thermal.

3. There is a distinct fatigue effect, but the substitution of a new section of wire does not restore the action.

4. The H_2 is not absorbed by the wire, but is deposited on the glass, especially where the latter is cooled by liquid air. If the wire is allowed to cool and the liquid air is then removed, ordinary H_2 is set free which will not recondense when the liquid air is replaced.

5. If this gas is pumped out and oxygen is admitted, the oxygen will disappear and in its place a small quantity of hydrogen will appear.

6. With platinum and especially with palladium wires these effects are much more marked.

7. These effects are not due to the presence of finely divided metal on the surface of the glass.

8. When phosphorus is present on the bulb and H_2 is introduced and the wire heated, the H_2 disappears and PH_3 is formed (apparently the first direct synthesis of PH_3).

9. These effects are accounted for by the theory that the H_2 dissolves in the material of the wire in the atomic condition and that some of these atoms, leaving the wire, do not meet other atoms (because of the low pressure), but diffuse into the tube cooled by liquid air or become adsorbed by the glass, and thus remain in the atomic condition, retaining all of the chemical activity of the atoms.

The author wishes to express his appreciation of the careful work of Mr. S. P. Sweetser, who has carried out most of the experimental part of this investigation.

RESEARCH LABORATORY, GENERAL ELECTRIC COMPANY.
July 27, 1912.

RESEARCHES UPON THE COMPLEXITY OF TELLURIUM.

BY WILLIAM R. FLINT.

Received August 21, 1912.

In continuation of the research upon the tellurium problem,[1] the following experiments have been carried out:

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[1] *Am. J. Sci.*, 28, 112, 347; 30, 209.

I. Ignition of the orange colored, crystallin material obtained in the previously described fractionation.

Since Harcourt and Baker[1] have suggested the possibility that the presence of tellurium trioxide, formed by the oxidizing action of chlorine in the hydrochloric acid used in the fractionation process, might have accounted for the low atomic weights found, it appeared desirable to determin whether the orange colored precipitate, noted in the preceding paper,[2] consists either wholly or in part of the trioxide. The authors mentioned state[3] that they obtained an orange colored product which they proved to be tellurium trioxide.

About 1.5 grams of the precipitate above referred to was subjected to ignition in platinum, over a porcelain radiator, the source of heat being a Bunsen flame:

Weight before ignition	1.5164 g.	Color, orange
0.5 hours at 100°	1.5164	
2 hours at 110–115°	1.5164	
2 hours at 190–195°	1.5162	
2 hours at 200–210°	1.5162	Color, orange Weight constant
1 hour below red heat	1.5159	
1 hour below red heat	1.5159	Color, nearly black
5 minutes, red heat	1.5143	
5 minutes, red heat	1.5141	Completely fused
3 minutes, red heat	1.5140	
3 minutes, red heat	1.5140	Color, orange Weight constant

Excepting the loss in weight, part of which, at least, may have been due to the expulsion of a trace of ammonium salt, the behavior of this material upon ignition does not appear to correspond in any regard with that of the trioxide. The trioxide when heated evolves oxygen, forming the dioxide, the process being accompanied by a change of color from orange to pure white. It will be observed that in the progressive ignition recorded above the substance changed color from orange to nearly black, when heated for two hours at a temperature just below red heat; whereas, upon an increase of the temperature to redness in the final ignitions, the original orange color was reassumed. Furthermore, the unignited substance goes into solution in concentrated hydrochloric acid without any detectable evolution of chlorine.

The dioxide obtained by the water precipitations, in the course of the fractionation previously described, was in every case pure white in color, and it was each time dissolved in hydrochloric acid with the aid of heat,

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[1] *Trans. Chem. Soc.*, 99, 1311 (1911).

[2] *Am. J. Sci.*, 30, 218.

[3] *Loc. cit.*

a precaution which must have reduced any trace of trioxide, supposing it to have been present. And lastly, this orange colored material was obtained at that end of the series of fractions at which, in the preliminary trial[1] of the method, a high atomic weight was found. This fact Harcourt and Baker appear to have overlooked.

It is impossible, therefore, to attribute the low atomic weights to contamination of the basic nitrate preparations by tellurium in the higher condition of oxidation.[2]

II. In the course of the preparation of a very large quantity of tellurium for the purpose of further investigations, it became convenient to make a series of hydrolytic precipitations using crude material. Twelve successive precipitations were performed, with the idea of ascertaining whether a separation could be effected upon unpurified tellurium, similar to that previously obtained with the purified substance. The series began with about 1.5 kilos of crude tellurium dioxide, contaminated with selenium, silica, copper, etc., the source of material being electrolytic residues from the Baltimore Copper Smelting and Rolling Company. The hydrochloric acid solution of this dioxide was boiled and then poured into boiling water and the whole allowed to cool. The precipitate came down instantly upon dilution and was flocculent. After filtration and washing, it was redissolved in hydrochloric acid and the process repeated until the twelfth precipitation had been made. Long before this point was reached, it became apparent that a large portion of the precipitate consisted of antimony, and the process was discontinued when it was judged that the proportions of antimony and tellurium in the final precipitate were about equal. The tellurium was next twice precipitated from hydrochloric acid solution by sulfur dioxide and recrystallized as basic nitrate, after which it was subjected three times to distillation in hydrogen, in quartz, using the electric current as a source of heat. After the third distillation the quartz boat contained no appreciable residue. The final product consisted of 114 grams of the metal.

Fifty grams of this preparation were dissolved in nitric acid (sp. gr. 1.25) and crystallized as basic nitrate. Four ignitions, in platinum, and using electric heaters gave the following results (the observed weights are recorded):

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[1] *Loc. cit.*

[2] Since the above was written, a report has been received of a spectroscopic examination of the purified tellurium used in the work to which Harcourt and Baker have taken exception. This material gave an atomic weight of 127.45. Dr. Uhler, of the Sloane Physical Laboratory, Yale University, who made the examination, reports that the sample of elementary tellurium is absolutely pure, as far as spectroscopic examination would show any impurity. The low atomic weights found in the author's previous work cannot, therefore, be ascribed to the presence of any known elements, as has been suggested by Harcourt and Baker.

	Wt. $2TeO_2.HNO_3$. Grams.	Wt. TeO_2. Grams.	Atomic wt. of Te.
1	3.2059	2.6766	127.4
2	5.1043	4.2620	127.4
3	4.6762	3.9046	127.4
4	6.3670	5.3160	127.4
	19.3534	16.1592	127.4 (Mean)

Conclusion.

III. The fractionation of crude material, with purification of the final fraction, is thus shown to be ineffective in securing any separation, as was quite to be expected. If the substance known as tellurium really is a complex body, the experiences which investigators too numerous to mention have had with it prove beyond question that the properties of the intermixed substances must be very closely alike, else a separation would long since have been obtained. A reaction, therefore, which if applied a sufficient number of times, might be capable of producing a separation upon purified tellurium, could hardly be expected not to be adversely affected by the presence of considerable quantities of various other elements.

It appears most probable to the writer that the chief cause of the failure of others,[1] to get a separation has been a lack of practical appreciation of the closeness of the similarity between tellurium and its homolog. It will probably be found, if a complete separation is ever brought about, that in most reactions tellurium and its homolog behave identically, and in those reactions in which they differ, the differences will be slight. The problem is further complicated by the probability that the small differences which would be exhibited by the separated substances are either masked or nearly obliterated by their mutual influence, each upon the other, in the mixture.

The successful solution of the problem evidently depends primarily upon two factors: first, the securing of such a reaction as is most favorable to the separation of the similar constituents; and second, the continued application of this reaction for a sufficient length of time to produce an appreciable separation. In the writer's opinion, the hydrolytic method is at least as well suited to the purpose as any that have been tried and he is now engaged in giving it an exhaustive test. It is hoped that the work will be ready for publication within a reasonable time.

Chemical Laboratory of Throop Polytechnic Institute.
August 1, 1912.

AN ELECTRICALLY HEATED MICROSCOPE SLIDE.

By F. G. Cottrell.

Received June 18, 1912.

At the request of Dr. H. C. Biddle, and primarily to accompany his

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[1] Cf. also Pellini, *Atti accad. Lincei,* 21, I, 218–25.