

in the normal hydrogen atom.<sup>20</sup> These relationships are approximately quantitatively true in a number of varied examples, inasmuch as the energies of formation of  $\text{AgCl}$ ,  $\frac{1}{2} \text{PbCl}_2$  and  $\frac{1}{4} \text{SnCl}_4$  are all less than the electron affinity of chlorine by only about 0.5 volt-equivalent. There are two classes of distortion of the octets. In one the energy of formation is greater than the electron affinity of the halide atom. In the other, which represents the combination of two electronegative atoms, the electron affinities of the two electronegative atoms are not completely satisfied. These relationships, which are derived by a comparison of thermal data with available spectroscopic data, do not permit a choice of the exact dynamic model of these molecules, but perhaps the study of these molecular spectra will lead to a model of these molecules and also to a picture of the electron bond.

### Summary

1. Energies of formation of some diatomic halides from atoms have been calculated from thermal data.

2. The energies of formation of the hydrogen halides from their atoms are substantially equal to the electron affinity of the halide atom.

3. In the case of metallic halides both positive and negative, but not inappreciable, deviations are found to exist, which are discussed from the viewpoint of distortion of the valence octet of electrons.

4. In spite of lack of precision in the thermal data, especially the energies of dissociation of the halogens, and the uncertainty in interpretation of the spectroscopic data on electron affinity, there exists an interesting general relationship between energy of reaction and electron affinity.

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### NOTE

**Permanent Filter Mats in Gooch Crucibles.**—Preliminary work in this Laboratory has shown that a porcelain Gooch crucible fitted with a permanent porcelain mat can be made at a very low cost. Such a crucible has most of the advantages of the more expensive Munroe crucible.<sup>1</sup>

The chemist is often deprived of the convenience of the Monroe crucible because of its great cost. To supply the demand various types of porous crucibles and filter cones have been devised. Those made from sintered aluminum oxide are familiar. The disadvantages of these are that the sides as well as the bottoms are porous and the liquid adsorbed in the sides is not easily removed by washing. Considerable difficulty is often experi-

<sup>20</sup> It may be well to mention that the equations used to represent changes in state are not intended to represent the mechanism but only differences in energy, since it seems that in some cases atoms and molecules must be activated before actual reaction can take place.

<sup>1</sup> THIS JOURNAL, 31, 456 (1909).

enced in drying and weighing them because they absorb moisture rapidly. With the proposed crucibles the method of filtering and removing the precipitate is similar to that used with the Munroe crucible. They will satisfactorily withstand the full heat of the blast lamp.

Various brands and types of crucibles were used for our experiments. Those made from crucibles with flanged, open bottoms fitted with loose perforated disks were most satisfactory.

The porcelain used in preparing the mat for the crucibles was made from the walls of a crucible of the same brand. The size of the interstices in the mat was controlled by crushing the porcelain and grading it with a set of Bureau of Soils sieves: that passing through 130 mesh was designated as Grade I; that through 64 mesh as Grade II; that through 0.5 mm. as Grade III; and that through 1.0mm. mesh as Grade IV. Two general methods were used in building up the mat. In each case Grade IV was used first to fill in the perforations of the crucible. By one method Grades III, II and I were then dusted on in alternate layers and in the other a known proportion of Grades III, II and I was mixed and dusted on in one consistent layer. The mats formed in this way were built up to a depth of 6 to 7 mm. The second method gave the most satisfactory results when the ratio of Grades III, II and I was about 2:2:3; photomicrographs of cross-sections showed that the interstices were fairly uniform.

Of the various materials used for cementing the mat, that found most satisfactory consisted of a 0.5 *N* potassium hydroxide solution saturated with calcium hydroxide. The prepared mat was wetted with this solution, dried in an oven at 110° for about ten hours and heated in the full flame of a Meker burner in a small furnace for from two to eight hours. The furnace was made up of two inverted sand crucibles, with bottoms cut off, set one upon the other. Clay rods were thrust through the walls of the lower crucible to support the upper and also to support the Gooch crucible. By this arrangement the crucible was kept enveloped in the flame of the Meker burner throughout the heating, thus providing a uniform heat, at a high temperature.

After the first period of heating in the furnace the mat was treated with 50 cc. of 6.0 *N* hydrochloric acid, washed and again heated as described above until no further sodium flame was visible. Another treatment with hydrochloric acid (d., 1.2) followed and after no further sodium flame was visible the crucible was dried and weighed. It was then repeatedly treated with aqua regia in 50cc. portions, washed, dried at 110° and weighed; in every case the mat reached constant weight after the first treatment.

The relative porosity of the mats was determined by allowing 50 cc. of water to flow freely through, while the crucible was kept filled during the operation. The flow in cubic centimeters per minute was recorded for each crucible as indicated in the table below.

The application of the porcelain mat to analytical operations was tested by making filtrations of known quantities of precipitated silver as silver chloride and barium as barium sulfate. A solution of silver nitrate of known value was prepared and aliquot parts were treated by the usual gravimetric methods. After the second determination most of the precipitate was removed by mechanical means and the remainder was dissolved out with ammonium hydroxide solution. A barium chloride solution was made up in the same manner and the accepted method of treating the barium sulfate precipitate was adopted. Table I gives the values in grams obtained with each crucible in each determination and also the calculated value for comparison.

Table I contains self-explanatory data for four representative prepared crucibles.

TABLE I  
RESULTS FOR FOUR CRUCIBLES

Cruc. No.	Total hrs. heating in furnace	Free-flow test cc. per min.	AgCl det. (0.1788) (0.3628) G.	BaSO <sub>4</sub> det. (0.3585) G.
VIII	7.75	1.45	0.1790 .3628	-0.3588
IV	3.5	1.94	.1788 .3628	- .3585
VI	3.0	2.63	.1790 .3628	- .3584
III	3.0	2.94	.1789 .3626	

The values in parentheses are the theoretical values in grams for comparison.

In order to obtain definite and permanent record of the structure of the different types of mats microscopic examinations were made and photomicrographs were prepared.

From the above data it is apparent that heating a mat in the furnace for a period of about three hours is sufficient. By the free-flow test Crucibles III and VI showed greater porosity but the determinations recorded above do not indicate any loss of precipitate.

Methods of standardizing the porcelain mats are being studied and, further, the possibility of building mats on silica Gooch crucibles in a manner similar to that described above for porcelain is being investigated in this Laboratory.

It should be added that recently Schott and Genossen have developed a Jena glass crucible with a sintered glass mat for similar purposes.

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