

In the last experiment, 5 grams of sodium acetate were added in order to increase the conductivity of the solution and also to ascertain what effect an excess of this salt would have, because, if the acetic acid were used to acidify the alkaline solution obtained by the decomposition of molybdenite, a great deal of this salt would be present. The concordant results justified the next step, which was to decompose weighed amounts of pulverized molybdenite with sodium carbonate and nitrate, then take up the fusion with water, filter out the insoluble oxides, acidify with acetic acid, boil off the carbon dioxide, and electrolyze. The liquid poured off from the deposit of the sesquihydroxide was heated to boiling and precipitated with a hot solution of barium chloride.

RESULTS OBTAINED FROM MOLYBDENITE.

	Molybdenite in grams.	Molybdenum found in per cent.	Sulphur found in per cent.
1	0.2869	57.37	38.28
2	0.1005	57.15	38.33
3	0.1388	56.83	37.87

MOLYBDENUM-SULPHUR RATIO.

(1).....	I : 2.004
(2).....	I : 2.012
(3).....	I : 1.998

From several experiments, which will not be recorded here, there is a possibility of separating molybdenum electrolytically from tungsten, the latter, of course, being present as an alkaline tungstate. Further work, however, will be necessary to fully establish this hope.

The experience detailed above proves conclusively that molybdenum must also be included in the list of metals which can be determined with the aid of the current and the analysis of molybdenite, as outlined, is vastly better than the usual procedures.

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A COMPARISON OF THE SOLUBILITY OF ACETYLENE AND ETHYLENE.

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NO satisfactory or convenient method for the separation of acetylene and ethylene has yet been devised. The two gases exhibit such a similarity in their solubility that very few

reagents are known which will make a quantitative separation. The necessity for this separation arose while the authors were preparing ethylene which was generated simultaneously with acetylene from certain products produced in the electric furnace.

The reagents known to be good solvents for acetylene are water, ammoniacal or acid cuprous chloride, ammoniacal silver chloride or the nitrate, acetone, and alcohol; and those commonly mentioned as dissolving ethylene are bromine and sulphuric acid.

Wherever any mention is made of a separation of acetylene and ethylene it is generally taken for granted that the latter gas is completely insoluble in ammoniacal cuprous chloride, and the analysis is based upon this false assumption, Hempel being the one authority who takes a different view. The method suggested by him for the determination of acetylene although accurate was found undesirable owing to the length of time it takes for the analysis. This method is not recommended by the author for the separation of the two gases but merely as a means of determining acetylene. However, owing to the fact (stated later) that ethylene dissolves in cuprous chloride to form a soluble compound, this method is applicable to the separation of the two gases.

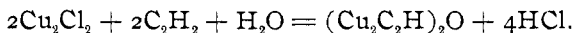
The method as used by us was carried out as follows: The cuprous chloride was contained in a train of five 5-inch U-tubes containing about 20 cc. each of the absorbent. To prevent the mechanical lifting of the precipitate into the connecting tubes, it was found desirable to use U-tubes with bulbs blown on each limb.

The mixed gases were collected over mercury in a pair of ordinary Hempel burettes, and from these delivered very slowly through the train of tubes. After all the gas was delivered from the burette, a T opening into the delivery tube was used for the introduction of nitrogen or hydrogen gas which acted as a sweep.

The red precipitate which was found in the tubes corresponds to $(\text{Cu}_2\text{C}_2\text{H})_2\text{O}$. This was removed from the tubes, collected on a filter, washed to remove soluble copper compounds, dissolved in hydrochloric acid, converted to sulphate, and the copper deposited electrolytically. The quantity of acetylene in the original gas is easily calculated from this weight of copper. This method was tried repeatedly with good results, the only source of error being the possibility of loss through too rapid passage of the gas, and the only objection the amount of time consumed. In fact it is out of the question to make a rapid separation of the gases by

this method. Consequently an attempt was made to find a reagent which would absorb the acetylene and leave the ethylene undissolved.

The solvent action on ethylene of known absorbents of acetylene was first determined. The usual solvent recommended for acetylene is ammoniacal cuprous chloride. This solution is made up of 42 grams cupric chloride, 32 grams copper turnings, 100 cc. hot water, and 200 cc. commercial hydrochloric acid. The mixture is boiled for two or three hours and then a little concentrated hydrochloric acid added, and the whole boiled until it is pale yellow. This was cooled and an excess of ammonium hydroxide added. Such a solution was found to absorb acetylene rapidly and completely according to the following reaction :¹



The next step was to investigate the solubility of known ethylene, this gas being generated by the action of metallic zinc on ethylene dibromide. Ethylene is so little soluble in water that there was no trouble to apprehend from such a small amount being absorbed. This was found by actual trial, and also the solubility in ammonium hydroxide. Here again a very slight solubility was noticed. Considering these two facts and that ethylene is not usually considered to be soluble in cuprous chloride, it seemed surprising, when either ammoniacal or acid cuprous chloride was added to a sample of ethylene to find that fully 95 per cent. of the gas was absorbed.

The foregoing is apparently not taken into account by Moissan whose results, according to the method of procedure given on pages 313 and 320 of "Le Four Électrique," can hardly be right. To be sure, the method is only spoken of in a vague way, but as stated in the reference given, it is natural to suppose that ethylene was considered as insoluble in the copper salt. If this were the fact, a large portion of the ethylene evolved from his thorium carbide must have been absorbed with the acetylene and counted with the latter.

Thinking that a mixture of ethylene and acetylene might act differently from either taken alone, equal volumes of the two gases were mixed and nearly the total volume was found to be absorbed by the copper solution. Other good solvents for acetylene are alcohol (1 volume of alcohol dissolves 6 volumes acety-

¹ Hempel's "Gas Analysis," p. 183.

lene) and acetone (1 volume of acetone dissolves 25 volumes acetylene). To investigate the possibilities of using either of these to absorb the acetylene and leave the ethylene the solubility of the latter gas was tried. Thirty cc. of pure ethylene were found to be all absorbed by acetone. Similarly when 100 cc. of ethylene were treated with an excess of 95 per cent. ethyl alcohol, a residue of less than 10 cc. was left, showing that both reagents were worthless for the purpose.

Since Nordhausen acid is such a good absorbent for ethylene, it seemed possible that it might first remove the ethylene and leave the acetylene. To determine this 56 cc. of known pure acetylene were passed several times into a Hempel pipette filled with the acid, and after two passages, over 60 per cent. of the acetylene was absorbed, showing that the reagent also failed in the required purpose. Finally the action of ammoniacal silver was investigated with the following results: The solution was made up by dissolving 10 grams silver nitrate in 500 cc. of water, making the solution barely acid with hydrochloric acid and then slightly ammoniacal. The clear solution thus obtained proved to be an excellent solvent for acetylene although it is slower in its action than ammoniacal cuprous chloride. The compound formed by its action is, according to Lewes,¹ Ag_2C_2 and not $\text{C}_2\text{H}_2\text{Ag}_2\text{O}$ as stated by other authorities.

It now remained to try its action upon ethylene. When 26 cc. of known ethylene were treated with an excess of ammoniacal silver chloride, only 0.2 cc. were absorbed, and repeated trials gave the same result; so that, although there is not absolute insolubility of ethylene, the method was admirable for its quickness and, of course, infinitely more accurate than the use of ammoniacal cuprous chloride.

Lastly the effect of the reagent upon known mixtures of pure acetylene and ethylene was tried. 29.2 cc. of ethylene and 37.3 cc. of acetylene were taken, and treated with the silver solution. The residue left corresponded closely with the volume of ethylene taken, thus showing conclusively that a mixture of the two gases may readily be separated by this method. Fractional percentages of ethylene might be lost and for such small amounts there seems nothing better than recourse to the gravimetric determination of the copper corresponding to the acetylene present, but this is not necessary for ordinary work.

¹ Lewes' "Acetylene," p. 154.