

tribution mains. It is well, however, as a precautionary measure to take both hydrocarbon and water vapor dew points and if the hydrocarbon vapor dew point is found to be higher than the water vapor dew point, the latter should be taken as representing the minimum temperature.

Again, it is possible that the gas after having reached its minimum temperature may warm up in contact with water vapor which has been introduced into the gas, as for example in purifiers where steam is admitted for manufacturing reasons. Under such conditions the calcium chloride scrubber (H) must be used and the dew point of the hydrocarbon vapors taken as the minimum temperature.

It is a very simple matter to test the gas in all three ways, *i. e.*, direct, through the rubber scrubber, and through the calcium chloride scrubber, and it has been found advisable in order to get a correct idea of what is taking place or what has taken place in the mains to frequently check the direct readings with readings made after scrubbing the gas.

Such observations also enable the gas engineer to judge whether the gas is being scrubbed by tar or heavy drips deposited in the mains or whether it is picking up hydrocarbons from the mains. If the gas has been cooled to a low temperature as it might be in passing through an exposed main as under a bridge and afterwards warmed up in the ground, as previously stated, we would expect the hydrocarbon vapor dew point to be higher than the water vapor dew point. If the hydrocarbon vapor dew point is lower than the water vapor dew point, it would indicate contact of the gas with a deposit of tar or heavy drip oil.

The practical application of this instrument in gas distribution practice is well defined. Owing to its low specific heat, the gas flowing from the works quickly reaches the temperature of the surrounding earth. The gas engineer knowing by this instrument to what extent the hydrocarbon vapors have been dropped and what the consequent fall in candle power will be, is capable of anticipating the reduction in candle power by raising the candle power of the gas going into the holder accordingly.

LABORATORY OF THE
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TECHNICAL METHOD FOR THE DETERMINATION OF LEAD IN ORES, ETC.

By A. H. Low.

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The following scheme is the result of many attempts on my part to improve the methods wherein lead is separated as oxalate and subsequently titrated with permanganate. I have used the method as described below for several months and have found it more satisfactory

for technical work than any other with which I am acquainted. Duplicate assays usually check within one-tenth of one per cent. A test of five portions of pure lead, weighed so as to approximately represent 3, 12, 20, 45 and 60 per cent. of lead in an ore, and put through the entire process, showed errors of -0.01 , -0.06 , $+0.01$, $+0.05$ and $+0.05$ per cent., respectively, the average error being 0.036 per cent. The permanganate was standardized on about 0.200 gram of lead.

Chromate-Oxalate Method.—Take 0.5 gram of ore and treat in a 6-oz. flask by the usual methods to obtain the washed lead sulphate, etc., on a 9 cm. filter. Dissolve the lead sulphate on the filter by stirring it up repeatedly with a jet of hot sodium acetate solution contained in a wash-bottle, receiving the filtrate in the original flask. Prepare the solvent by diluting a cold saturated solution of commercial sodium acetate with an equal bulk of water and adding 40 cc. of 80 per cent. glacial acetic acid per liter. To test if the extraction of the lead sulphate is complete, the flask may be replaced by a small beaker, the washing continued and a little potassium dichromate added to the filtrate. If any lead chromate is produced, the mixture may be added to the clear solution in the flask. Agitate the extract in the flask and heat it if necessary to redissolve any separated precipitate. Add 10 cc. of a 5 per cent. solution of commercial potassium dichromate, heat to boiling and boil gently for a few minutes to render the precipitate basic and easily filtered. The change is shown by its becoming reddish yellow in color. Filter hot, wash out the flask with hot water and then wash the precipitate only once, simply to clean the upper edge of the filter. Place a wide-necked funnel in the flask, open the filter and spread it against the wall of the funnel and wash off the lead chromate with a jet of hot oxalic acid solution, using from 25 to 40 cc. Rinse down any adhering chromate in the flask with hot water. The oxalic acid solution consists of a cold saturated solution of commercial acid 1 part, water 3 parts. Heat nearly to boiling in a wash-bottle. To the mixture in the flask add grain alcohol and then boil until the chromic acid is all reduced and the lead converted to oxalate. Remove from the heat, add 30 cc. of cold water and cool thoroughly, best under the tap or in cold water. When cold, filter through an 11 cm. filter, wash out the flask thoroughly with cold water and then wash filter and precipitate 10 times with cold water. Place 5 cc. of strong sulphuric acid in the flask, dilute first with a little cold water and then with hot water to about 125 cc. Add the filter and precipitate and titrate to the usual pink tinge with standard potassium permanganate solution. The solution used for iron will serve, although too strong for the best work. Theoretically, the oxalic acid ($C_2O_4H_2 \cdot 2H_2O$) value of the permanganate multiplied by 1.642 will give the lead value, but owing to slight losses as sulphate, oxalate, etc., and

the fact that the lead oxalate is not perfectly pure, the factor 1.669 will give a closer approximation. On this basis, for 0.5 gram of ore taken for assay, the solution should contain 1.5185 grams of potassium permanganate per liter, in order that 1 cc. may equal 1 per cent. lead. It is best to standardize on about 0.200 gram of pure lead dissolved in a little 1:2 nitric acid and put through the entire process.

Notes.—The lead oxalate formed in the above process is not pure white, but yellowish, and still contains about 1 per cent. of lead chromate. It appears to be sufficiently uniform in its nature to give accurate results.

Instead of dropping filter and precipitate into the flask for titration, as described above, a neater method of procedure is as follows: Place the flask under the funnel and pour through and over the filter about 75 cc. of hot dilute acid, containing 5 cc. of strong sulphuric acid, and then wash the filter and residue well with hot water, so that the final bulk of the filtrate will be about 125 cc. Titrate the hot liquid as before. This method takes a little longer and gives practically the same results as the simpler way, but it has the advantage of a permanent end-point, there being no organic matter present to slowly decolorize the pink tinge.

Calcium does not interfere with the method, nor does antimony. Bismuth in small amounts is without material influence. Ten per cent. of bismuth added to a mixture containing about 23 per cent. of lead raised the result 0.36 per cent., most of the bismuth being removed as sulphate and chromate.

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THE ELECTROLYTIC DETERMINATION OF BISMUTH.

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Much has been written about the determination of bismuth in the electrolytic way and many electrolytes have been proposed. Without attempting any detailed discussion of the numerous publications which have appeared on the subject, the difficulties with which one almost invariably has to contend may be briefly summed up as follows:

When a bismuth solution is electrolyzed there is deposited not only metallic bismuth on the cathode, but frequently there is a simultaneous deposition of peroxide on the anode and it has been suggested that both anode and cathode be weighed for each determination. Again, the deposited metal is nearly always black and spongy, and in cases where accurate results have been obtained it has been necessary to exercise the greatest care in washing and drying in order to prevent loss mechanic-