§ 60.648 Optional procedure for measuring hydrogen sulfide in acid gas—Tutwiler Procedure.1

(a) When an instantaneous sample is desired and \( \text{H}_2\text{S} \) concentration is ten grains per 1000 cubic foot or more, a 100 ml Tutwiler burette is used. For concentrations less than ten grains, a 500 ml Tutwiler burette and more dilute solutions are used. In principle, this method consists of titrating hydrogen sulfide in a gas sample directly with a standard solution of iodine.

(b) Apparatus. (See Figure 1.) A 100 or 500 ml capacity Tutwiler burette, with two-way glass stopcock at bottom and three-way stopcock at top which connect either with inlet tubulature or glass-stoppered cylinder, 10 ml capacity, graduated in 0.1 ml subdivision; rubber tubing connecting burette with leveling bottle.

(c) Reagents. (1) Iodine stock solution, 0.1N. Weight 12.7 g iodine, and 20 to 25 g cp potassium iodide for each liter of solution. Dissolve KI in as little water as necessary; dissolve iodine in concentrated KI solution, make up to proper volume, and store in glass-stoppered brown glass bottle.

(2) Standard iodine solution, 1 ml=0.001771 g I. Transfer 33.7 ml of above 0.1N stock solution into a 250 ml volumetric flask; add water to mark and mix well. Then, for 100 ml sample of gas, 1 ml of standard iodine solution is equivalent to 100 grains \( \text{H}_2\text{S} \) per cubic feet of gas.

(3) Starch solution. Rub into a thin paste about one teaspoonful of wheat starch with a little water; pour into about a pint of boiling water; stir; let cool and decant off clear solution. Make fresh solution every few days.

(d) Procedure. Fill leveling bulb with starch solution. Raise (L), open cock (G), open (F) to (A), and close (F) when solutions start to run out of gas inlet. Close (G). Purge gas sampling line and connect with (A). Lower (L) and open (F) and (G). When liquid level is several ml past the 100 ml mark, close (G) and (F), and disconnect sampling tube. Open (G) and bring starch solution to 100 ml mark by raising (L); then close (G). Open (F) momentarily, to bring gas in burette to atmospheric pressure, and close (F). Open (G), bring starch solution to 100 ml mark by raising (L); then close (G). Open (F) momentarily. To bring gas in burette to atmospheric pressure, and close (F). Open (G), bring liquid level down to 10 ml mark by lowering (L). Close (G), clamp rubber tubing near (E) and disconnect it from burette. Rinse graduated cylinder with a standard iodine solution (0.00171 g I per ml); fill cylinder and record reading. Introduce successive small amounts of iodine thru (F); shake well after each addition; continue until a faint permanent blue color is obtained. Record reading; subtract from previous reading, and call difference D.

(e) With every fresh stock of starch solution perform a blank test as follows: introduce fresh starch solution into burette up to 100 ml mark. Close (F) and (G). Lower (L) and open (G). When liquid level reaches the 10 ml mark, close (G). With air in burette, titrate as during a test and up to same end point. Call ml of iodine used C. Then,

\[
\text{Grains H}_2\text{S per 100 cubic foot of gas}=100 \frac{(D-C)}{C}
\]

(f) Greater sensitivity can be attained if a 500 ml capacity Tutwiler burette is used with a more dilute (0.001N) iodine solution. Concentrations less than 1.0 grains per 100 cubic foot can be determined in this way. Usually, the starch-iodine end point is much less distinct, and a blank determination of end point, with H2S-free gas or air, is required.

FIGURE 1. TUTWILER BURETTE (LETTERED ITEMS MENTIONED IN TEXT)

Subpart MMM [Reserved]


SOURCE: 55 FR 26942, June 29, 1990, unless otherwise noted.