

(d) Each owner or operator who elects to comply with §60.646(e) shall keep, for the life of the facility, a record demonstrating that the facility's design capacity is less than 150 LT/D of H<sub>2</sub>S expressed as sulfur.

(e) The requirements of paragraph (b) of this section remain in force until and unless EPA, in delegating enforcement authority to a State under section 111(c) of the Act, approves reporting requirements or an alternative means of compliance surveillance adopted by such State. In that event, affected sources within the State will be relieved of obligation to comply with paragraph (b) of this section, provided that they comply with the requirements established by the State.

**§60.648 Optional procedure for measuring hydrogen sulfide in acid gas—Tutwiler Procedure.<sup>1</sup>**

(a) When an instantaneous sample is desired and H<sub>2</sub>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 H<sub>2</sub>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 starts 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 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,

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

(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 H<sub>2</sub>S-free gas or air, is required.

<sup>1</sup>Gas Engineers Handbook, Fuel Gas Engineering Practices, The Industrial Press, 93 Worth Street, New York, NY, 1966, First Edition, Second Printing, page 6/25 (Docket A-80-20-A, Entry II-1-67).

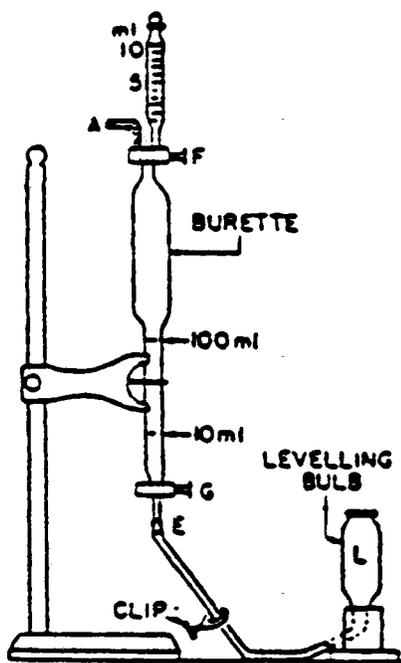


Figure 1. Tutwiler burette (lettered items mentioned in text).

#### Subpart MMM [Reserved]

#### Subpart NNN—Standards of Performance for Volatile Organic Compound (VOC) Emissions From Synthetic Organic Chemical Manufacturing Industry (SOCMI) Distillation Operations

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

#### § 60.660 Applicability and designation of affected facility.

(a) The provisions of this subpart apply to each affected facility designated in paragraph (b) of this section that is part of a process unit that produces any of the chemicals listed in § 60.667 as a product, co-product, by-product, or intermediate, except as provided in paragraph (c).

(b) The affected facility is any of the following for which construction, modification, or reconstruction commenced after December 30, 1983:

(1) Each distillation unit not discharging its vent stream into a recovery system.

(2) Each combination of a distillation unit and the recovery system into which its vent stream is discharged.

(3) Each combination of two or more distillation units and the common recovery system into which their vent streams are discharged.

(c) Exemptions from the provisions of paragraph (a) of this section are as follows:

(1) Any distillation unit operating as part of a process unit which produces coal tar or beverage alcohols, or which uses, contains, and produces no VOC is not an affected facility.

(2) Any distillation unit that is subject to the provisions of Subpart DDD is not an affected facility.

(3) Any distillation unit that is designed and operated as a batch operation is not an affected facility.

(4) Each affected facility that has a total resource effectiveness (TRE) index value greater than 8.0 is exempt from all provisions of this subpart except for §§ 60.662; 60.664 (d), (e), and (f); and 60.665 (h) and (l).

(5) Each affected facility in a process unit with a total design capacity for all chemicals produced within that unit of less than one gigagram per year is exempt from all provisions of this subpart except for the recordkeeping and reporting requirements in paragraphs (j), (l)(6), and (n) of § 60.665.

(6) Each affected facility operated with a vent stream flow rate less than 0.008 scm/min is exempt from all provisions of this subpart except for the test method and procedure and the recordkeeping and reporting requirements in § 60.664(g) and paragraphs (i), (l)(5), and (o) of § 60.665.

[NOTE: The intent of these standards is to minimize the emissions of VOC through the application of best demonstrated technology (BDT). The numerical emission limits in these standards are expressed in terms of total organic compounds (TOC), measured as TOC less methane and ethane. This emission limit reflects the performance of BDT.]