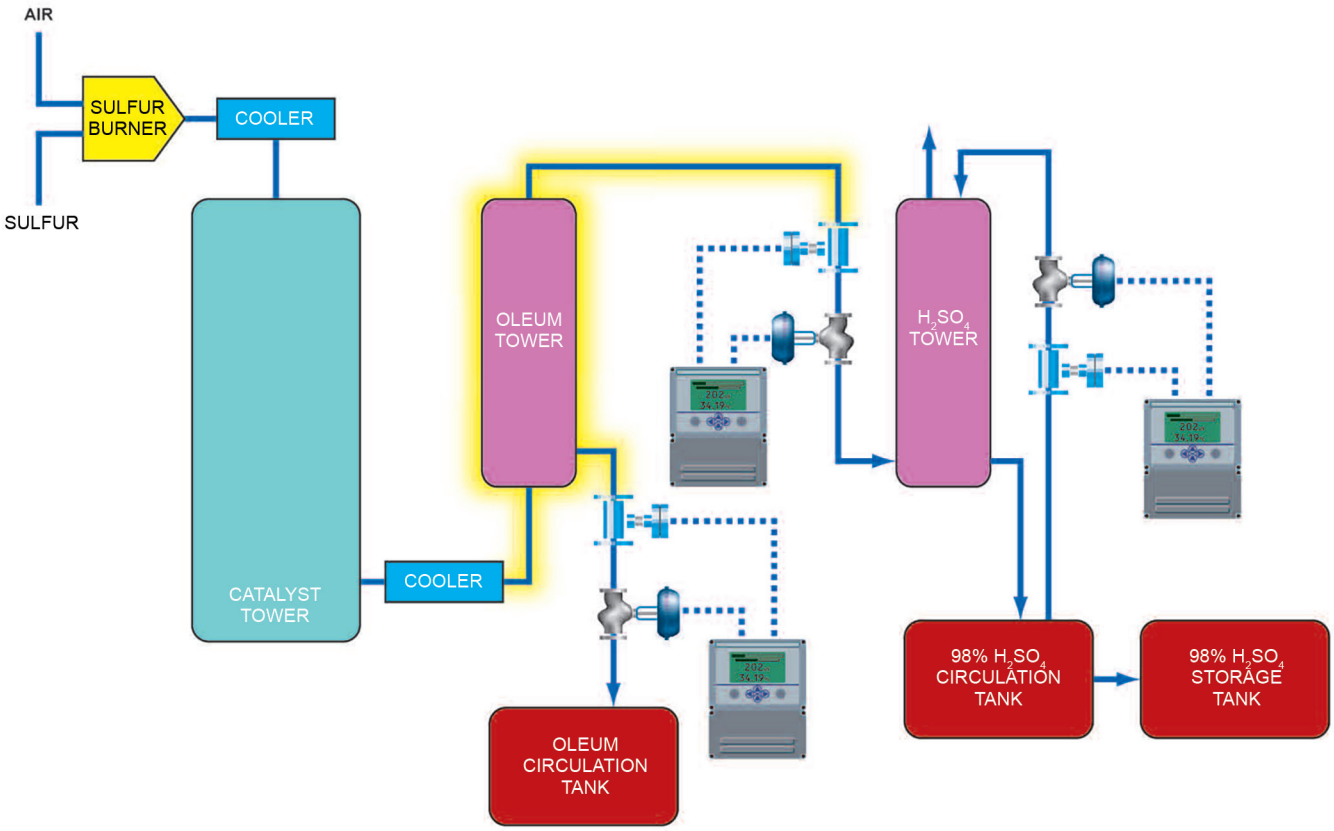
Oleum Acid Measurement



Introduction

Oleum (fuming sulfuric acid, $H_2S_2O_7$) is corrosive and commonly made in various strengths, consisting of SO_3 dissolved in 100% H_2SO_4. Therefore, 20% oleum contains 20% SO_3 and 80% H_2SO_4 by weight. Contingent on strength, vapor pressure is such that escaping SO_3 fumes combine with moisture in the air to form sulfuric acid mist particles. These sulfuric acid mist particles are typically visible and can create dense white clouds of fumes.

- Normally turbid, off-white liquid
- Solution of uncombined SO_3 dissolved in H_2SO_4
- Oleum sometimes referred to as greater than 100% H_2SO_4

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Uses

- In sulfonation and nitration processes
- Leading source of sulfonation for powdered/ synthetic laundry products
- High-strength adhesives
- Insecticides
- Oil refining
- Explosives

Production

Typically, oleum is produced as part of the commercial process of manufacturing sulfuric acid. Today essentially all sulfuric acid and oleum produced in the United States is manufactured using the contact method. The contact method incorporates three basic operations, each of which corresponds to a distinct chemical reaction. The basic raw materials for sulfuric acid are sulfur, air, and water.

First — the sulfur feedstock (molten sulfur or sulfur containing ore) is oxidized (burned in air) to sulfur dioxide (SO_2).

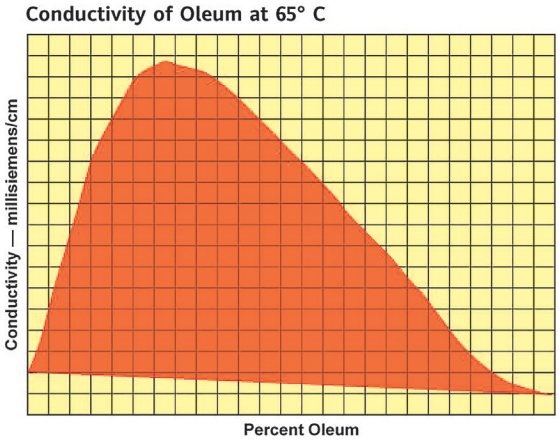
Next — the sulfur dioxide is fed to a process unit called a converter, where it is catalytically (such as vanadium pentoxide catalyst or platinum) oxidized (at 450° Celsius) to sulfur trioxide (SO_3).

Finally — the sulfur trioxide is absorbed in a strong 98% sulfuric acid solution (fuming sulfuric acid or oleum). Final sulfuric acid product is sent to storage tanks. Conductivity measurements are often considered for the oleum tower line and the recirculation line between the tower and tank. Typical percent concentration ranges encountered in industry for oleum are 0 to 10% or 42 to 18% oleum, and for sulfuric acid are from 99.5 to 93% and 0 to 25% H_2SO_4 (occasionally mid-ranges).

Application Challenges – Curve Shape

Conductivity vs. percent by weight vs. temperature

Oleum, occasionally referred to in terms of greater than 100% sulfuric acid concentration. However, like most binary solutions, oleum exhibits a generally bell-shaped conductivity versus concentration by weight curve. It exhibits a front slope (0% to 10% oleum) and a back slope (42% to 18% oleum). Typically, when referring to the back slope of conductivity curve, the concentration ranges are reversed (such as 42% to 18%), because the lowest conductivity is actually the higher percent concentration. As with any conductivity measurement, it is imperative to utilize the most accurate conductivity versus concentration (by weight) and temperature compensation curve set(s) available. Foxboro® offers oleum front slope and back slope percent concentration curve sets as standard. Furthermore, since a single conductivity can report to more than one concentration on many bell-shaped curves, conductivity measurements are typically restricted to a single, linear curve section. In addition, conductivity increases with temperature, so the curve shape(s) can and do change as the process temperature changes.



Per Corrosion Resistance Tables, Fourth edition — Schweitzer

Metals –	Excellent = < 2 mills/yr.	Good = < 2 0 mills/yr.	Satisfactory = < 50 mills/yr.	Unsatisfactory = > 50 mills/yr.
Non-metallic –	R* = Resistant/recommended No = Not recommended PEEK = PolyethereKetone PCTFE = PolyChloroTriFluoro-Ethylene	A = No attack, little or no absorption (per Victex) nr = not rated (Virgin or Glass filled) PVDF = PolyVinylideneDiFluoride (Also was regionally known as Kel-F)	PerFluoroElestomer = Chemraz 505	

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This matrix identifies oleum

a. The standard curves resident in memory in an 875EC Communicator or an 870ITEC Intelligent Transmitter

b. The temperature compensation range and the reference temperature of each standard curve

c. Some typical conductivity values for low-, mid-, and high-end percent concentrations at reference temperature, relative to the standard curve solutions

Temp. Comp. Temp. Range & Ref. Temp.	875EC and 8701TEC Std. Curves	1%	5%	10%	15%	18%	20%	25%	35%	42%	90%	93%	96%	99.50%	99.90%	99.99%
89° – 249°F 65°C	>100% H_2SO_4 Oleum 0 – 10%	39 mS/cm	67 mS/cm	79 mS/cm												
89° – 249°F 65°C	>100% H_2SO_4 Oleum 42 – 18%					78 mS/cm	74 mS/cm	64 mS/cm	34 mS/cm	15 mS/cm						

The conductivity values provided (above) have been gleaned from data published by numerous sources, and are to be considered estimations only. Since no two sources are likely to completely agree on the "conductivity" of a solution, this table should be used as a reference source only. Ultimately, any precise conductivity values required should be determined independently. Often the most representative data is derived on-site. For conductivity or material compatibility information relative to either Standard or Custom curve solutions, contact Foxboro.

Compatibility

For general manufacturing, sensor selection is a critical aspect of any oleum acid measurement. Typically, there are various wetted materials to consider (such as thermoplastics, O-rings, and metals), where process compatibility decisions are based on maximum process temperature and percent acid concentration range. The most appropriate wetted materials will change relative to these process factors, though different sensor material selections are typically not required for front slope (0% to 10%) versus high-concentration (42% to 18%) oleum applications (as they might for sulfuric acid measurements less than 100%). An exception would be those very atypical applications seeking to measure 65% oleum. Foxboro offers a sensor that routinely provides long life in even this unusually severe environment.

Materials Compatibility

Metals	
316 ss	Rated as Good to ~ 240°F
Hast C	Rated as Excellent to 140°F
C-20	Rated as Good to 110°F
Borosilicate Glass	Rated to ~ 380°F
Thermoplastics	
PEEK	Unacceptable
PVDF	Unacceptable
PFA	Rated to ~ 80°F
Tefzel	Rated to ~ 150°F
O-Ring Material	
Viton	Rated to ~ 180°F
Kalrez	Rated to ~ 200°F
Chemraz	Rated to ~ 380°F

Note: approximations only. Final materials compatibility decision resides with end-user.

Installation

Sensor installation must also be considered as one of the potentially critical application decisions. Whether invasive or flowthrough sensor design, the sensor selection must be made relative to the application parameters (such as maximum process temperature, process line pressure, maximum percent concentration), as the sensor selected may impact frequency and ease of calibration and/or exposure of personnel.

871FT Sensor

EP307G Sensor

Foxboro Product Application Solutions – Curves for 875EC Intelligent Analyzer

The Foxboro 875EC Intelligent Analyzer (four-wire, V ac) and 870ITEC Intelligent Transmitter (two-wire, V dc) offer standard oleum curve sets for 0% to 10% and 42% to 18%, both at 65°C reference temperature (and more than 18 other standard binary solution curve sets). In addition, they offer multiple application capability (up to three distinct application range and temperature compensation curve sets), any combination of which may be standard or custom curves, and can be calibrated into memory. They may also be employed with the capability to autoswitch from one calibrated application to another. These unique-to-Foxboro features essentially permit the accurate measurement of the entire oleum acid concentration range by utilizing either the standard 0 to 10% curve set for front slope measurements, or the standard 42 to 18% (back slope) curve set. Dilutions across the back-to-front slope range are possible by utilizing the Foxboro-unique multiple application capability with the auto-switch feature.

Material Compatibility

Foxboro offers the widest array of electrodeless conductivity sensors in the industry. Among these are invasive sensors and offer a wide selection of wetted materials, including metals, thermoplastics, and O-rings (such as the 871EC series) compatible with typical oleum applications and all thermoplastic sensors (such as the EP307B series) or borosilicate glass sensors (such as EP307G series) for the most aggressive oleum applications.

In addition, the noninvasive Foxboro 871FT Industrial Flowthrough electrodeless conductivity sensor series solves many historically encountered application problems. Instead of installing an insertion-type sensor, the Industrial Flowthrough sensor becomes a section of the process line. A wide selection of bore sizes (0.5, 1.0, 1.5, 2.0, 3.0, or 4.0 inch) permits the selected sensor to match the size of the process line and become a section of the process line. The 871FT Flowthrough sensors may also be selected from a wide list of compatible wetted materials.

Installation

Foxboro 871FT series Industrial Flowthrough sensors permit calibration in-line without exposing personnel or the environment to aggressive and dangerous chemicals, thereby reducing calibration time and expense. Foxboro's EP485A series calibration plugs further increases accuracy and reduces completion time for the calibration.

Reference Documentation

Sensors:

871EC-TF2-V (ref. PSS 6-3C4A)

Analyzer/Transmitter:

875EC Intelligent Analyzer (Ref. PSS 6-3N1C)
870ITEC Intelligent 2-wire Transmitter (Ref. PSS 6-3N2A)

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