

- 1 liter.
- 5.5 Ascorbic acid solution: Dissolve 60 g of ascorbic acid in about 600 mL of distilled water. Add 2 mL of acetone and dilute to 1 liter.
 - 5.6 Diluent water: Dissolve 40 g of NaCl in about 600 mL of distilled water and dilute to 1 liter.
 - 5.7 Sulfuric acid solution, 4%: Add 40 mL of conc. sulfuric acid to 800 mL of ammonia-free distilled water, cool and dilute to 1 liter.

6.0 Procedure

Digestion

- 6.1 To 20 or 25 mL of sample, add 5 mL of digestion solution and mix. (Use a vortex mixer).
- 6.2 Add 4-8 Teflon boiling chips. Too many boiling chips will cause the sample to boil over.
- 6.3 With Block Digester in manual mode set low and high temperature at 160°C and preheat unit to 160°C. Place tubes in digester and switch to automatic mode. Set low temperature timer for 1 hour. Reset high temperature to 380°C and set timer for 2 1/2 hours.
- 6.4 Cool sample and dilute to 25 mL with distilled water. If TKN is determined the sample should be diluted with ammonia-free water.

Colorimetric Analysis

- 6.4.1 Check the level of all reagent containers to ensure an adequate supply.
- 6.4.2 Excluding the molybdate/antimony line, place all reagent lines in their respective containers, connect the sample probe to the Sampler IV and start the proportioning pump.
- 6.4.3 Flush the Sampler IV wash receptacle with about 25 mL of 4% sulfuric acid (5.7).
- 6.4.4 When reagents have been pumping for at least five minutes, place the molybdate/antimony line in its container and allow the system to equilibrate.
- 6.4.5 After a stable baseline has been obtained, start the sampler.

7.0 Calculations

- 7.1 Prepare a standard curve by plotting peak heights of processed standards against concentration values. Compute concentrations by comparing sample peak heights with the standard curve.

8.0 Precision and Accuracy

- 8.1 In a single laboratory (EMSL) using sewage sample containing total P at levels of 0.23, 1.33, and 2.0, the precision was ± 0.01 , ± 0.04 , and ± 0.06 , respectively.
- 8.2 In a single laboratory (EMSL) using sewage samples of concentration 1.84 and 1.89, the recoveries were 95 and 98%, respectively.

Bibliography

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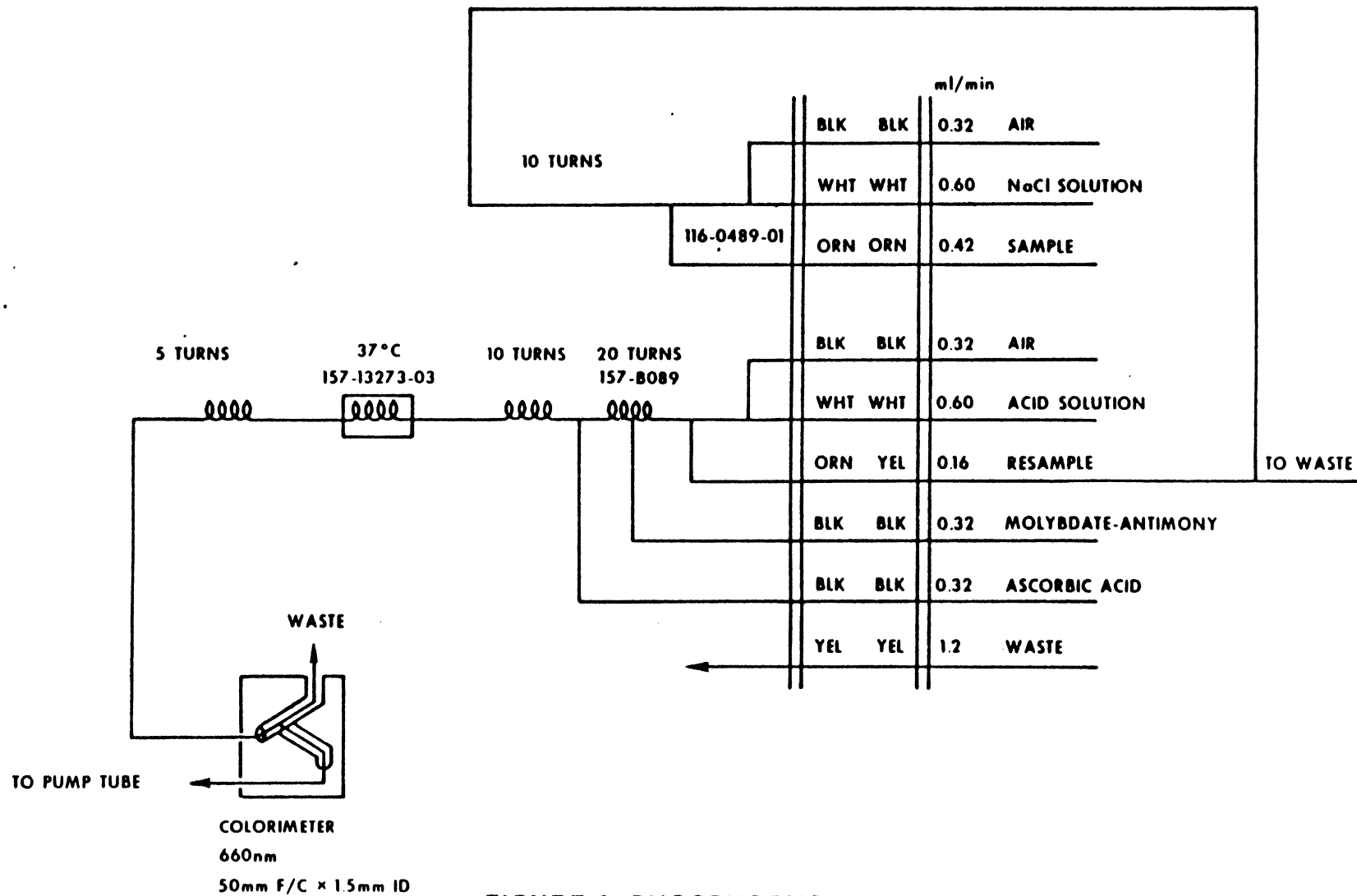


FIGURE 1. PHOSPHORUS MANIFOLD AA11

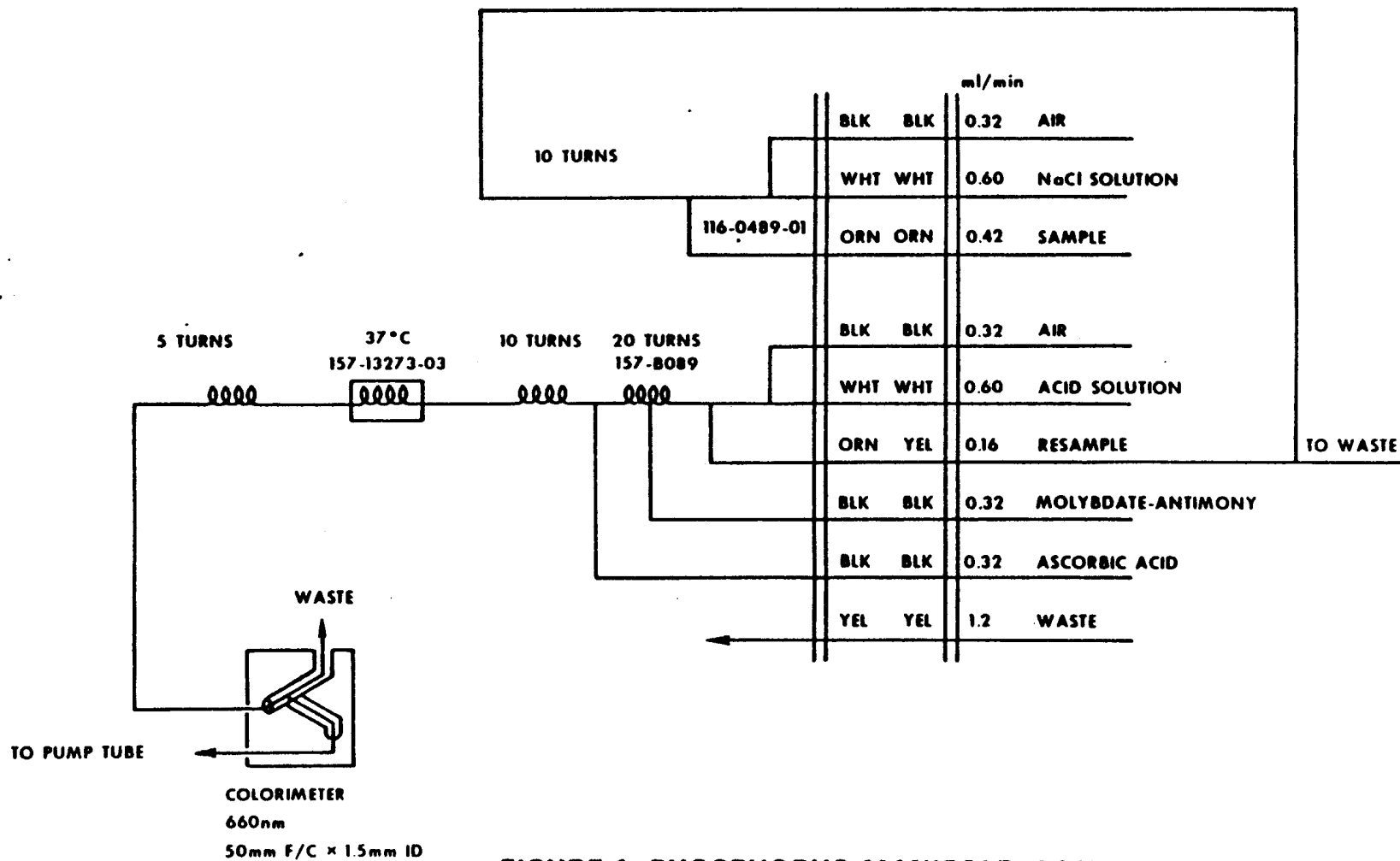


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