METHOD 29

REF: Reg 8-42

DETERMINATION OF ETHANOL IN BAKERY EFFLUENTS

1) PRINCIPLE

1.1 This method applies to the determination of ethanol in bakery effluents. The ethanol in the effluent gases is collected by use of Source Test Procedure ST-32, using three Greenberg-Smith impingers. The concentration of ethanol is then determined by gas chromatography.

1.2 The minimum detectable concentration of ethanol by this method is 0.02 µl **(0.016 mg)** ethanol per ml of sample.

2) APPARATUS

2.1 Gas Chromatograph. This unit is fitted with a flame ionization detector **(FID)**, a glass sleeve injection port, and a compatible integrator or data station.

2.2 Analytical Column.

2.2.1 17' x 1/8" O.D. SS column packed with 20% SP-2100/0.1% carbowax 1500 in Supelcoport, 100-120 mesh.

2.3 10 µl Microsyringe.

2.4 Volumetric Flasks. Assorted Sizes according to needs.

2.5 Pipettes. Assorted sizes according to needs.

2.6 Vari Whirl Mixer.

2.7 Refrigerator.

2.8 Graduated Cylinder. Assorted sizes according to needs.

3) REAGENTS

3.1 Ethanol. Anhydrous, 200% proof **(Sp. Grav. 0.816).**

3.2 Distilled Water.

3.3 Cylinder Hydrogen.

3.4 Cylinder Helium or Nitrogen.

3.5 Air Supply.

4) ANALYTICAL PROCEDURE

4.1 Samples should be processed as rapidly as possible after collection. In any case store in a refrigerator until time for analysis.

4.1.2 Measure and record the total liquid volume (ml) of each impinger, using a graduated cylinder.

4.2 Gas Chromatograph. Column and operating parameters.

4.2.1 Column: 17' x 1/8" O.D. SS column packed with 20% SP-2100/0.1% carbowax 1500 in Supelcoport, 100-120 mesh.

4.2.2 Operating Parameters.

Oven Temperature	**60^{o}C**
Injection Temperature	**250^{o}C**
Carrier Gas Flow	**20 ml/min**
Injection Sample Size	**2 µl**

4.3 Inject 2 µl of the working ethanol standard **(5.2)** into the gas chromatograph using a 10 µl micro syringe. Record the retention time and peak area of ethanol. Retain the chromatogram.

4.4 Inject 2 µl of the sample into the gas chromatograph using a 10 µl micro syringe. Record the retention time and peak area of ethanol. Retain the chromatogram.

4.5 If the concentration of ethanol in the sample exceeds 10 times the concentration of ethanol in the standard, dilute the sample using water as a diluent. Record the dilution factor **(DF)**.

5) PREPARATION OF STANDARD SOLUTIONS

5.1 **Stock Ethanol Standard Solution.** Pipet 1.0 ml of the anhydrous ethanol **(200% proof)** into a 100 ml volumetric flask and dilute to the mark with distilled water. This solution contains 10 µl of ethanol per ml **(8.16 mg ethanol/ml)**. Stopper the flask. Thoroughly mix the solution by inverting the flask several times. The stock standard solution is kept refrigerated and is stable for at least three **(3)** months.

5.2 **Working Ethanol Standard Solution.** Pipet 1.0 ml of the stock ethanol standard solution into a 10 ml volumetric flask and dilute with water to the mark. This solution contains 1.0 µl ethanol per ml **(0.816 mg ethanol/ml).** Stopper the flask. Thoroughly mix the solution by inverting the flask several times. This working standard is always prepared fresh prior to use.

5.3 The distilled water used for standard preparation must be checked for contamination. Inject 2 µl of the distilled water into the gas chromatograph as in Section **4.3.** There should be no responses of any kind.

6) CALCULATIONS

6.1 Compare the chromatograms from **4.3** and **4.4** to identify the ethanol in the sample. Quantitate the ethanol in the sample using the following equations:

6.2

$$\frac{\text{Mg Ethanol}}{\text{Impinger}} = \frac{\text{Conc (STD) x PA (sample) x DF x V}}{\text{PA (STD)}}$$

Where:

PA (Sample) = Peak area of ethanol found in the sample (4.4).

Conc (STD) = Concentration in mg/ml of ethanol in the Standard (5.2)

DF = Dilution factor (5.4). If no dilution is made then DF = 1.

V = Total volume in ml of the sample in each impinger.

PA (STD) = Peak area of ethanol in the standard (5.2).

6.3 Total mg ethanol content in the sample is equal to the summation of the weight (mg) of ethanol found in each impinger.

7) REFERENCES

7.1 **"Resolving Solvent Mixtures and Determining Water in Solvents"**, Supleco, Inc., G. C. Bulletin 7471.

72. Elkins, H.B., **"The Chemistry of Industrial Toxicology"** John Wiley & Sons, Inc., New York, 1951.