Fluid **S**ealing **A**ssociation

STANDARD

FSA-NMG-201-02

RECOMMENDED PRACTICES
FOR DETERMINATION
OF EXTRACTABLE SULFUR
FOR NON-METALLIC GASKETING MATERIAL

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RECOMMENDED PRACTICES FOR DETERMINATION OF EXTRACTABLE SULFUR FOR NON-METALLIC GASKETING MATERIAL

1. SCOPE

1.1 This method covers the measurement of Extractable Sulfur in Gasket Material.

2. SIGNIFICANCE

2.1 This method is designed to compare related material under controlled conditions for Extractable Sulfur.

3. GENERAL

3.1 This procedure is applicable to all types of elastomer products and product using elastomer as a binder such as beater addition or compressed gasket material. The sulfur determined by this recommended practice consists of any uncombined extractable elemental sulfur plus some of the sulfur contained in extracted accelerator fragments. Sulfur combined with the elastomer is not determined. Procedure is based on the oxidation of sulfur to sulfate with bromine followed by precipitation of the sulfate by addition of barium chloride solution.

4. REAGENTS

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society. Other grades may be used provided these are of sufficient high quality to permit the use without affecting the accuracy of the method. Reagents include:

Barium Chloride, Hydrate
Acetone
Hydrochloric Acid, Concentrated
Bromine

WARNING: Contact with bromine causes burns and blisters to the skin. Keep this reagent sealed with glass stoppers. Always keep an ammonia-water solution within reach to neutralize any spills.

5. PREPARATION OF REAGENTS

5.1 <u>10% Barium Chloride</u> - Dissolve 40 grams of $BaCL_2*2H_2O$ in distilled water and make to 400 mls. in a graduated cylinder. Insure it is mixed well and pour into plastic screw cap bottle for storage.

6. TEST SPECIMEN

6.1 Specimen shall consist of not less than 5 grams of material. The specimens shall be cut into appropriate square pieces (1/16"-1/8"). For gasket material thicker than 1/16", split the pieces in half with a knife to obtain thinner cross sections.

6.2 No conditioning of the specimen is necessary however precautions should be taken to insure no contamination is allowed to affect the outcome of the test.

7. PROCEDURE

7.1 Prepare two samples of 2.0 - 3.0 grams each and weigh to the nearest .001 gram.

7.2 Install in soxhlet apparatus, rubber extraction apparatus or other suitable extraction apparatus.

7.3 Extract for 16 - 24 hours with 100 - 125 ml acetone.

7.4 Evaporate acetone in weighed glass evaporating dishes on steam bath. (Dishes 80 mm in top diameter and 45 mm deep are suggested.)

7.5 When solvent has evaporated, dry dishes in circulating air oven at 100-110°C for about 10 minutes.

7.6 Cool in desiccator and weigh if percent of acetone soluble matter is desired. If not, the weighing may be omitted.

7.7 Fill the dishes about one half full with distilled water. Under a hood, add sufficient bromine to at least cover the residue in the bottom of the dish.

7.8 Cover with a watch glass and allow to stand under the hood for about fifteen minutes.

7.9 Place, covered, on top of steam bath and heat until all bromine is gone. Water will be colorless or have a yellow tint.

7.10 Remove from steam bath, add bromine again to cover bottom of the dish and repeat Steps 7.8 and 7.9.

7.11 Filter dish contents into 400 ml beaker. Rinse off watch glass and rinse out dish with water from a wash bottle.

7.12 Add 3 ml concentrated hydrochloric acid and adjust volume to about 200 ml.

7.13 Heat to boiling over a burner. Hold at boiling 2 - 3 minutes.

7.14 Rapidly add 10 ml of 10% barium chloride solution with continuous, vigorous stirring of the boiling solution.

7.15 Stir and heat an additional 30 seconds. Let stand overnight on bench.

7.16 Filter on #40 paper (or finer), rinse beaker well with distilled water and use policemen on bottom and sides.

7.17 Wash precipitate several times on paper with distilled water.

7.18 Place paper in weighed #0 porcelain crucible, burn off paper carefully over a burner flame.

7.19 Ignite crucible and contents for 1 hour at 1600°F in muffle furnace, cool in desiccator and weigh.

8. CALCULATION

8.1 Calculate percent extractable Sulfur as follows:

A = weight of barium sulfate in crucible.

B = weight of sample taken.

$$\frac{A \times 0.137}{B} \times 100\% = \% \text{ Extractable Sulfur}$$

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