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Standard Test Method for Oil from Oil Shale (Resource Evaluation by the Fischer Assay Procedure)¹

This standard is issued under the fixed designation D 3904; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the amount of oil, water, and spent shale obtained upon prescribed heating of a representative oil shale sample that has been crushed to an appropriate size (2.36 mm or 8 mesh). The amount of gas evolved can be calculated from data obtained in this procedure. This test method is applicable to oil shale, which upon heating evolves liquid and gaseous products (Note 1). It has been tested over the range 19 to 227 L/Mg (5 to 60 gal/ton) of oil.

NOTE 1—The test method does not include procedures for identifying the nature of the gas.

1.2 The test method neither yields quantities that necessarily duplicate yields in experimental or commercial oil-shale processes nor does it yield products that necessarily duplicate in physical or chemical nature the products that may be obtained in such experimental or commercial processes.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Notes 4 through 7.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 346 Test Methods for Collection and Preparation of Coke Samples for Laboratory Analysis²
D 1250 Petroleum Measurement Tables³
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)³
D 2013 Test Method for Preparing Coal Samples for Analysis²
E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke, and Oil Shale.

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² Annual Book of ASTM Standards, Vol 05.05.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

3.1.1 *assay*—the determination of the potential yield of a specific component from a resource material.

3.1.2 *gas-plus-loss*—a term covering evolved gases not condensed at $0 \pm 3^\circ\text{C}$ plus experimental error. In addition to experimental error, gas-plus-loss values arise from organic material and from minerals present in the oil shale that may release hydrogen sulfide (H_2S) or carbon dioxide (CO_2). An example of these minerals are: dolomite ($\text{CaMg}(\text{CO}_3)_2$) and pyrite (FeS_2).

3.1.3 *oil shale*—a sedimentary rock containing insoluble organic matter that upon heating yields an oil similar to petroleum.

3.1.4 *resource evaluation*—evaluation of an oil shale resource is the determination of the approximate oil resource present in the deposit. Oil yield assay results combined with stratigraphic and geographic distribution permit estimation of a shale oil resource.

3.1.5 *shale oil*—the organic liquid-obtained from the retorting of oil shale.

4. Summary of Test Method

4.1 Crushed raw shale is sampled by riffle-splitting. The determination of the amount of oil available from oil shale is made by heating the raw shale from ambient temperature to 500°C in cast aluminum-alloy retorts. The vapors distilled from the sample are cooled and the condensed fraction is collected. The oil and water fractions are separated, the water volume (converted to weight equivalent) is measured and subtracted from the oil plus water weight. The weight of uncondensable gases evolved (gas-plus-loss) is then calculated by difference.

5. Significance and Use

5.1 This test method provides standardized data useful for oil-shale resource evaluation and is useful as a basis of comparing the oil-producing capabilities of oil shales.

5.2 Because the U.S. Department of Energy, Laramie Energy Technology Center (formerly a U.S. Bureau of Mines Laboratory) has developed an extensive record of data concerning the properties of oil shales from various regions by the use of this test method, the test method provides a means of comparing oil shale deposits.

5.3 The extensive use of the procedure makes it a basis for standardizing other evaluation methods for oil shale.

6. Interferences

6.1 An erroneously high gas-plus-loss value will be obtained if the system is not gas-tight. If smoke or vapor is detected rising from the system, or a pungent odor is present, a leak is evident and the sample must be discarded.

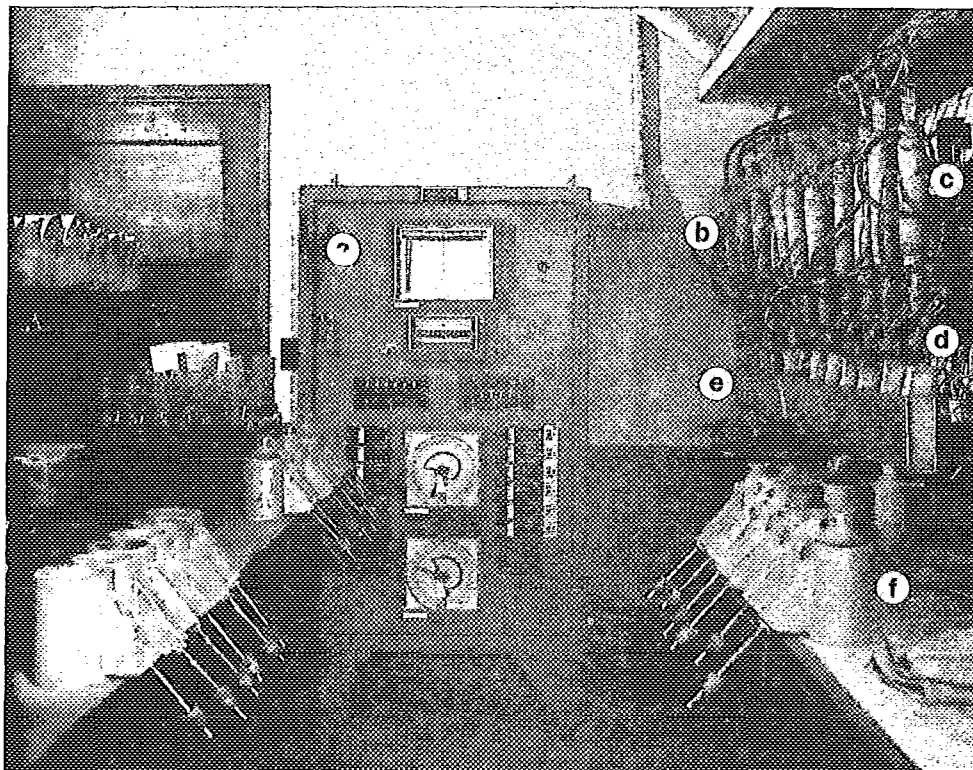
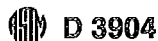


FIG. 1 View of a Typical Fischer Assay Laboratory Showing the Following Components: (a) Temperature Controller; (b) Condensors; (c) 2000-W Ovens; (d) Assembled Glass Adapters and 100-mL Centrifuge Tube; (e) Cooling Bath; (f) Cast Aluminum Alloy Retorts

7. Apparatus

7.1 Assay Bench⁵ consisting of the following components (Fig. 1):

7.1.1 Temperature Control Unit⁶ (Fig. 1).

7.1.2 Cooling Bath capable of holding a constant temperature through condensers of $0 \pm 3^\circ\text{C}$ (Figs. 1, 2, and 3).

7.1.3 Constant-Temperature Bath capable of maintaining $40 \pm 5^\circ\text{C}$.

7.1.4 Electric Heater (Figs. 4 and 5).

7.1.5 Programmer⁶ (Fig. 1).

7.1.6 Recorder (Fig. 1).

7.1.7 Thermocouple (Fig. 4).

7.1.8 Centrifuge (large enough to accommodate receiver) (Fig. 6).

7.2 Retorting Apparatus consisting of the following:

7.2.1 Condenser (Allihn style, 300-mm length) (Figs. 1, 2, and 3).

7.2.2 Glass Adapter (Figs. 1, 2, and 7).

7.2.3 Receiver⁸ (wide-mouthed, calibrated centrifuge tubes) (Figs. 1, 2, and 6).

7.2.4 Retort⁹ (cast aluminum alloy) (Figs. 1, 2, 4, and 8).

7.3 Miscellaneous:

7.3.1 Balance, with capacity of 3000 g capable of weighing to 0.1 g.

7.3.2 Balance capable of weighing pycnometers to ± 0.1 mg.

7.3.3 Balance capable of weighing adapters and receivers containing oil and water (250-g maximum) with ± 0.01 -g capability.

7.3.4 Pipet or Pycnometer (approximately 1-mL capacity).

8. Reagents and Materials

NOTE 2—For hazardous or harmful effects, consult a reference such as *Dangerous Properties of Industrial Materials* by N. I. Sax.¹⁰

8.1 Calcium Chloride (CaCl_2), reagent grade.

8.2 Technical Grade Ethylene Glycol ($\text{HOCH}_2\text{CH}_2\text{OH}$) or equivalent coolant such as commercial antifreeze.

8.3 Lead Acetate ($\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_3 \cdot \text{H}_2\text{O}$).

NOTE 3—See section on lead compounds in *Dangerous Properties of Industrial Materials* by N. I. Sax, pp. 861.

8.4 Solvent capable of dissolving shale oil residues from glassware and retorts, for example, technical grade methylene chloride (CH_2Cl_2) (Warning—See Note 4) or technical grade acetone ($(\text{CH}_3)_2\text{C}=\text{O}$) (Warning—See Note 5).

NOTE 4: Warning—Harmful if inhaled, high concentrations can cause unconsciousness or death. Precaution—Avoid prolonged or repeated breathing of vapor. Use only with adequate ventilation. Do not

⁵ Available from Electric Power Equipment Co., E. 56th Ave., Commerce City, CO 80022.

⁶ Available from the following: Gammaflux, Inc., 1821 Michael Faraday Dr., Reston, VA 22090 or Leeds and Northrup, 4800 Hoppy Canyon Rd., Denver, CO 80237.

⁷ *Annual Book of ASTM Standards*, Vol 05.02.

⁸ Available from Kontes of CA, 3045 Teagarden St., San Leandro, CA 94577.

⁹ Available from Arapahoe Aluminum Foundry, 3130 S. Platte Dr., Englewood, CO 80110.

¹⁰ Sax, N. I., *Dangerous Properties of Industrial Materials*, Van Nostrand Reinhold Co., 1975.

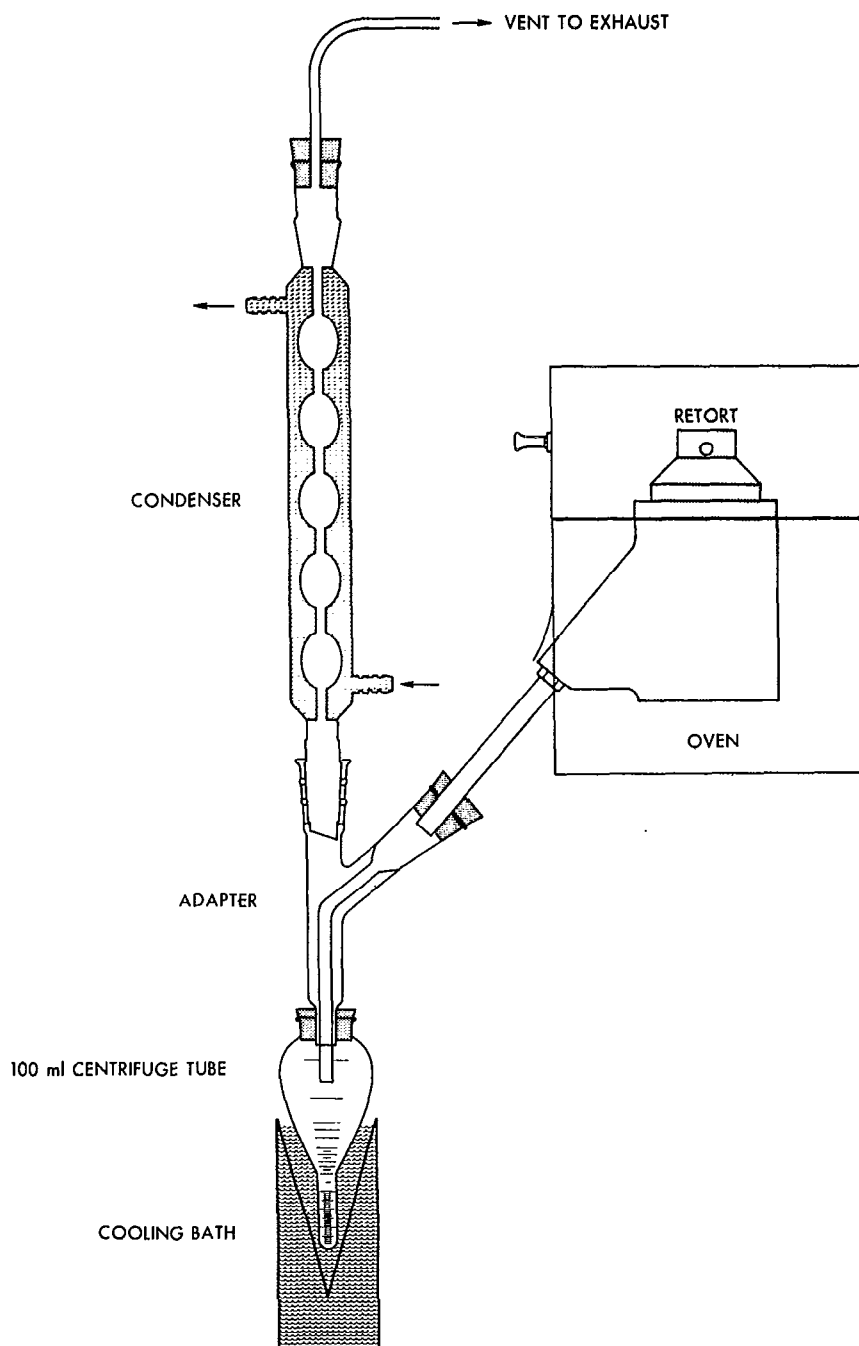
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FIG. 2 Apparatus Assembly

take internally. Avoid prolonged or repeated contact with skin. Do not get in eyes. Do not store in open or unlabeled or mislabeled containers. Do not store degrease, clean-out sludge in tightly sealed containers. Store containers out-of-doors away from combustible materials. Do not use cutting or welding torches, open flames, electric arcs, or glowing surface on empty or full drums that contain trichloroethylene.

NOTE 5: Warning—Danger, extremely flammable. Vapors can cause flash fire. **Precaution**—Keep away from heat, sparks, and open flame. Keep container closed. Use with adequate ventilation. Vapors may spread long distances and ignite explosively. Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion proof electrical apparatus and heaters. Avoid prolonged breathing of vapor or spray mist. Avoid contact with eyes and skin.

9. Sampling

9.1 Sample size and sample preparation procedures shall comply with Test Methods D 346 and D 2013, and Specification E 11.

9.2 Use a minus 2.36-mm particle size (8 mesh, U.S. Standard) (see Specification E 11) for better representation in this test method.

9.3 Because of the nonhomogeneous nature of oil shale, take a 95 ± 10 -g sample by riffle-splitting only.