INTERNATIONAL

**Designation: ~~G205 – 16~~ G205 – 23**

# Standard Guide for Determining Emulsion Properties, Wetting Behavior, and Corrosion-Inhibitory Properties of Crude Oils[1]

This standard is issued under the fixed designation G205; 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 guide ~~presents~~covers some generally accepted laboratory methodologies that are used for determining emulsion forming tendency, wetting behavior, and corrosion-inhibitory properties of crude oil.

1.2 This guide does not cover detailed calculations and methods, but rather covers a range of approaches that have found application in evaluating emulsions, wettability, and the corrosion rate of steel in crude oil/water mixtures.



1.3 Only those methodologies that have found wide acceptance in the industry are considered in this guide.

1.4 This guide is intended to assist in the selection of methodologies that can be used for determining the corrosivity of crude oil under conditions in which water is present in the liquid state (typically up to ~~100°C).~~100 °C). These conditions normally occur during oil and gas production, storage, and transportation in the pipelines.

1.5 This guide is not applicable at higher temperatures (typically above ~~300°C)~~300 °C) that occur during refining crude oil in refineries.



1.6 This guide involves the use of electrical currents in the presence of flammable liquids. Awareness of fire safety is critical for the safe use of this guide.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety and health~~safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[1] This guide is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

~~D96 Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure) (Withdrawn 2000)[3]~~
D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method
D665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water
D724 Test Method for Surface Wettability of Paper (Angle-of-Contact Method) (Withdrawn 2009)[3]
D1125 Test Methods for Electrical Conductivity and Resistivity of Water
D1129 Terminology Relating to Water
D1141 Practice for Preparation of Substitute Ocean Water
~~D1193 Specification for Reagent Water~~
D4006 Test Method for Water in Crude Oil by Distillation
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
~~D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (Withdrawn 2020)[3]~~
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G31 Guide for Laboratory Immersion Corrosion Testing of Metals
G111 Guide for Corrosion Tests in High Temperature or High Pressure Environment, or Both
G170 Guide for Evaluating and Qualifying Oilfield and Refinery Corrosion Inhibitors in the Laboratory
G184 Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using Rotating Cage
G185 Practice for Evaluating and Qualifying Oil Field and Refinery Corrosion Inhibitors Using the Rotating Cylinder Electrode
G193 Terminology and Acronyms Relating to Corrosion
G202 Test Method for Using Atmospheric Pressure Rotating Cage

2.2 *ISO Standard:*[4]

ISO 6614 Petroleum products—Determination of Water Separability of Petroleum Oils and Synthetic Fluids

2.3 *NACE Standard:*[5]

TM0172 Standard Test Method Determining Corrosive Properties of Cargoes in Petroleum Product Pipelines

## 3. Terminology

3.1 *Definitions*—The terminology used herein, if not specifically defined otherwise, shall be in accordance with terminologies in Guide G170, Terminology G193, and Terminology D1129. Definitions provided herein and not given in terminologies in Guide G170, Terminology G193, and Terminology D1129 are limited only to this standard.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *emulsion, n*—a two-phase immiscible liquid system in which one phase is dispersed as droplets in the other phase.

3.2.2 *emulsion-inversion point, n*—the volume percentage of water at which a water-in-oil (W/O) emulsion converts into oil-in-water (O/W) emulsion.

3.2.3 *wettability, n*—tendency of a liquid to wet or adhere on to a solid surface.

3.3 *Acronyms:*

| | | |
|---|---|---|
| $CO_2$ | = | Carbon dioxide |
| EIP | = | Emulsion inversion point |
| $H_2S$ | = | Hydrogen sulfide |
| ~~KOH~~ | = | ~~Potassium hydroxide~~ |
| NaCl | = | Sodium chloride |
| ~~$Na_2CO_3$~~ | = | ~~Sodium carbonate~~ |
| ~~$NaHCO_3$~~ | = | ~~Sodium bicarbonate~~ |
| ~~NaOH~~ | = | ~~Sodium hydroxide~~ |
| ~~$Na_2S$~~ | = | ~~Sodium sulfide~~ |

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from the American National Standards Institute, 25 W. 43rd St., New York, NY 10036.

[5] Available from the National Association of Corrosion Engineers, 1440 S. Creek Dr., Houston, TX 77084-4906.

O/W = Oil-in-water
W/O = Water-in-oil

## 4. Summary of Guide

4.1 This guide describes methodologies for determining three properties of crude oils that are relevant to corrosion processes caused by the presence of water in hydrocarbon transport and handling: *(1)* the emulsion of the oil and water, *(2)* the wettability of the steel surface, and *(3)* the corrosivity of water phase in the presence of oil.

4.2 Conductivity of emulsion can be used to determine the type of emulsion: oil-in-water (O/W) or water-in-oil (W/O). The conductivity of O/W emulsion (in which water is the continuous phase) is high. The conductivity of W/O emulsion (in which oil is the continuous phase) is low.

4.3 The wettability of a steel surface is determined by either contact angle methodology or spreading methodology.

4.4 The corrosiveness of water phase in the presence of crude oil can be determined using several methodologies.

## 5. Significance and Use

5.1 In the absence of water, the crude oil is noncorrosive. However, trace amounts of water and sediment have the potential to create corrosive situations during crude oil handling or transport if such materials accumulate and persist on steel surfaces. Test Methods ~~D96,~~ D473~~,~~ ~~D4006, and~~and D4006 ~~D4377~~provide methods for determination of the water and sediment content of crude oil.



5.2 The potential for a corrosive situation to develop during the handling and transport of crude oil that contains water can be determined by a combination of three properties (Fig. 1) (**1**)[6]: the type of emulsion formed between oil and water, the wettability of the steel surface, and the corrosivity of water phase in the presence of oil.

5.3 Water and oil are immiscible but, under certain conditions, they can form emulsion. There are two kinds of emulsion: oil-in-water (O/W) and water-in-oil (W/O). W/O emulsion (in which oil is the continuous phase) has low conductivity and is thus less corrosive; whereas O/W (in which water is the continuous phase) has high conductivity and, hence, is corrosive (**2**) (see ISO 6614). The percentage of water at which W/O converts to O/W is known as the emulsion inversion point (EIP). EIP can be determined by measuring the conductivity of the emulsion. At and above the EIP, a continuous phase of water or free water is present. Therefore, there is a potential for corrosion.

5.4 Whether water phase can cause corrosion in the presence of oil depends on whether the surface is oil-wet (hydrophobic) or water-wet (hydrophilic) (**1, 3-5**). Because of higher resistance, an oil-wet surface is not susceptible to corrosion, but a water-wet surface is. Wettability can be characterized by measuring the contact angle or by evaluating the tendency of water to displace oil from a multi-electrode array by measuring the resistance (or conductors) between the electrodes (spreading methodology).

5.4.1 In the contact angle methodology, the tendency of water to displace hydrocarbon from steel is determined by direct observation of the contact angle that results when both oil and water are in contact with the steel. Although this contact angle is determined by the interfacial free energies of the phases involved, there is no standard method to determine the steel-oil or steel-water interfacial free energies.

5.4.2 In the spreading methodology of determining wettability, the resistance between isolated steel pins is measured. If a conducting phase (for example, water) covers (wets) the distance between the pins, conductivity between them will be high. If a non-conducting phase (for example, oil) covers (wets) the distance between the pins, the conductivity between them will be low.

5.5 Dissolution of ingredients from crude oils may alter the corrosiveness of the aqueous phase. A crude oil can be classified as ~~corrosive, neutral,~~ corrosive or inhibitory based on how the corrosivity of the aqueous phase is altered by the presence of the oil. Corrosiveness of aqueous phase in the presence of oil can be determined by methods described in Test Method D665, Guide G170, Practice G184, Practice G185, Test Method G202, and NACE TM0172.

[6] The boldface numbers in parentheses refer to a list of references at the end of this standard.

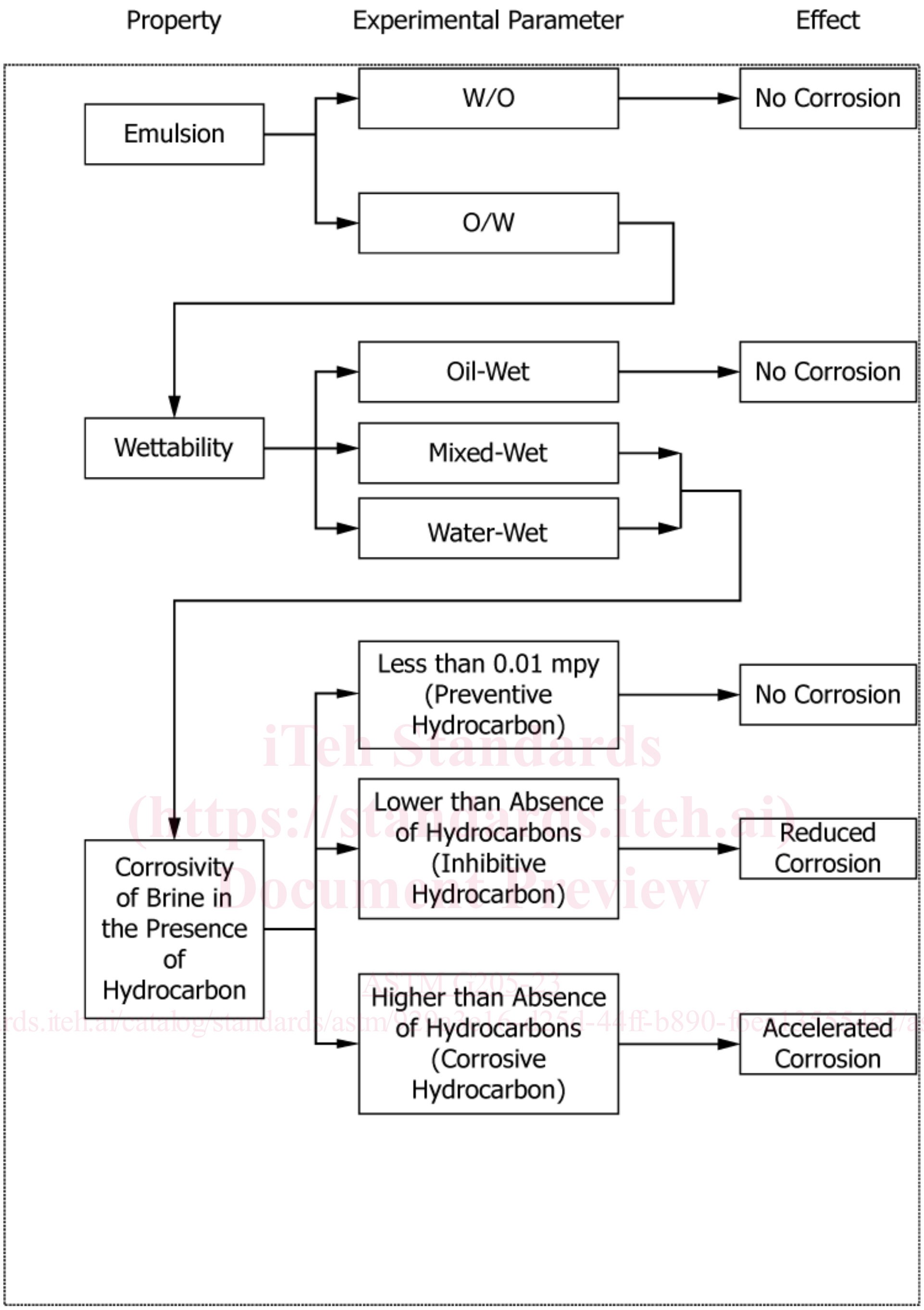



**FIG. 1 Predicting Influence of Crude Oil on the Corrosivity of Aqueous Phase**



## 6. Materials

6.1 Methods for preparing coupons and probes for tests and for removing coupons after the test are described in Practice G1. Standard laboratory glassware should be used for weighing and measuring reagent volumes.

6.2 The coupons/probes should be made of the ~~field material (such as carbon steel) and have the same metallographic structure as that~~ same metallographic material as the carbon steel that is used in the service components. The probes for wettability and EIP measurements should be ground to a surface finish of 600 grit. Preparation of coupons for corrosion measurements is described in Guide G170, Practice G184, Practice G185, and Test Method G202.

## 7. Preparation of Test Solutions

7.1 Oil should be obtained from the field that is being evaluated. Practice D4057 provides guidelines for collecting crude oil. It is ~~important~~preferred that live fluids do not contain externally added contaminants, for example, corrosion inhibitors, biocides, and surfactants to allow measurement of Guide G205 parameters on the "base" crude. A water sample should also be obtained from the field. A synthetic aqueous solution could be used; the composition of which should be based on field water analysis. Alternatively, use of 3 % NaCl aqueous solution composed of purified water and reagent grade sodium chloride or synthetic brine of a composition provided in Practice D1141 (substitute ocean water, note brine stability is approximately one day) may be used. Their composition should be specified in the work plan and recorded in the laboratory logbook. The solutions should be prepared following good laboratory practice.

7.2 The solutions (oil and water phases) should be deaerated by passing nitrogen (or any other inert gas) and kept under deaerated conditions. Solutions should be transferred with minimal contact with air. Procedures to transfer the solutions are described in Test Method G202.

7.3 Procedures to deoxygenate and saturate the solutions with acid gases are presented in Test Method G202. To simulate field operating conditions, the solution is often required to be saturated with acid gases such as hydrogen sulfide ($H_2S$) and carbon dioxide ($CO_2$). $H_2S$ and $CO_2$ are corrosive gases. $H_2S$ is poisonous and shall not be released to the atmosphere. The appropriate composition of gas can be obtained by mixing $H_2S$, $CO_2$, and methane streams from the standard laboratory gas supply. Nitrogen or any other inert gas can be used as a diluent to obtain the required partial pressures of the corrosive gases. Alternatively, gas mixtures of the appropriate compositions can be purchased from suppliers of industrial gases. The composition of gas depends on the field gas composition. The oxygen concentration in solution depends on the quality of gases used to purge the solution. The oxygen content of nitrogen or the inert gas should be less than 10 ppm by volume. Any leaks through the vessel, tubing, and joints should be avoided.

7.4 The test vessels should be heated slowly to avoid overheating. The thermostat in the heater or thermostatic bath should be set not more than ~~20°C~~20 °C above the solution temperature until the test temperature is reached. The pressure in the vessel should be monitored during heating to make sure it does not exceed the relief pressure. If necessary, some of the gas in the vessel may be bled off to reduce the pressure. The test temperature should be maintained within ~~±2°C~~±2 °C of the specified temperature. Once the test temperature is reached, the test pressure should be adjusted to the predetermined value. The pressure should be maintained within ~~±10 %~~ ±10 % of the specified value for the duration of the test.



7.5 A general procedure to carry out experiments at elevated pressure and elevated temperature is described in Guide G111. For elevated temperature and elevated pressure experiments using individual gases, first the autoclave is pressurized with $H_2S$ to the required partial pressure and left for ~~10 minutes.~~ 10 min. If there is a decrease of pressure, the autoclave is repressurized. This process is repeated until no further pressure drop occurs. Then, the autoclave is pressurized with $CO_2$ by ~~opening the~~charing with $CO_2$ from a high pressure gas cylinder at a pressure equal to the $CO_2$ + $H_2S$ partial pressure and left for ~~10 minutes.~~ 10 min. If there is a decrease in pressure, the autoclave is repressurized with $CO_2$ gas. This process is repeated until no further pressure drop is observed. Finally, the autoclave is pressurized with an inert gas (for example, methane) ~~by opening the appropriate cylinder at the total gas~~ to the total pressure at which the experiments are intended to be carried out.

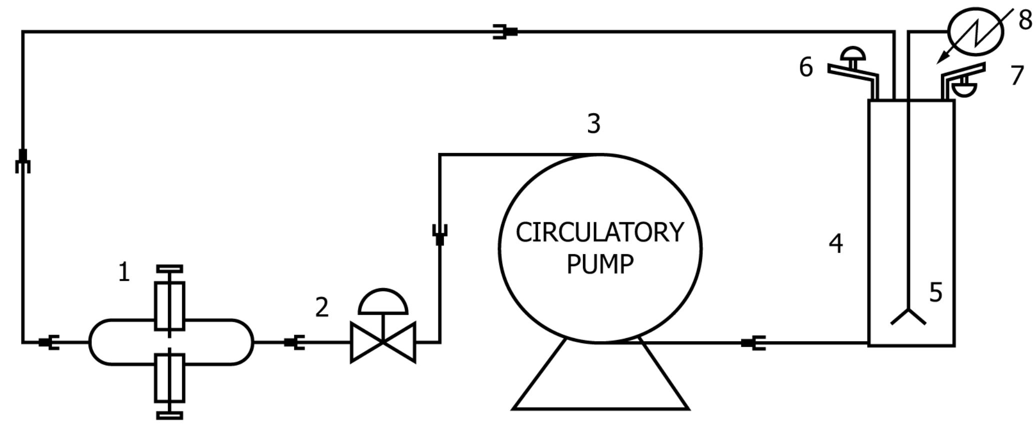
## 8. Laboratory Methodologies

8.1 *Determination of Emulsion Type:*

8.1.1 A schematic diagram of the equipment used for determining the emulsion type is presented in Figs. 2 and 3. The apparatus consists of an experimental section (Fig. 3), a reservoir, a circulating pump, and a flow controller.

8.1.2 The experimental section (Fig. 3) is a 15.2 cm long horizontal pipe section of ~~2.5 cm~~ 1.6 cm in diameter containing two vertically placed electrically isolated measuring pins (typically made from carbon steel). The distances between the pins can be varied with a screw arrangement. For optimal measurements, a pin distance of ~~0.25 cm~~ 1 mm to 2 mm is suggested.

8.1.3 The reservoir (typically ~~7 L~~ 7 L capacity) may be an autoclave (for higher pressure measurements) or a glass container (for atmospheric pressure measurements). The top cover of the reservoir is fitted with an inlet, an outlet, and an impeller. For higher pressure experiments, the reservoir is also fitted with a pressure gauge to monitor the pressure. The impeller should be capable of




1—Experimental section (see Fig. 3)
2—Flow controller
3—Circulatory pump
4—Reservoir (volume = 7 L)
5—Impeller
6—Gas inlet
7—Gas outlet
8—Power source to operate the impeller

**FIG. 2 Schematic Diagram of a Flow Loop of an EIP Apparatus**

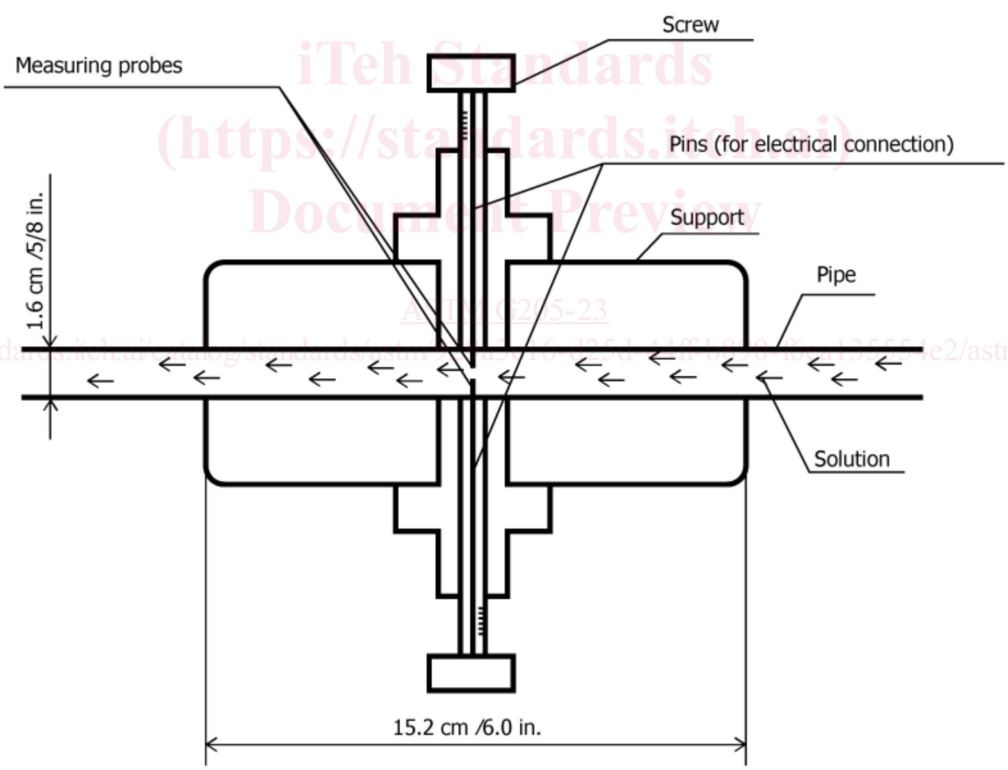





**FIG. 3 Schematic Diagram of the Experimental Section of the EIP Apparatus**

rotating at annular rotation speeds higher than 1000 rpm. A homogenous solution may also be created without an impeller by designing the reservoir inlet with horizontal flow and adjustable height to the top of the liquid level that provides sufficient turbulence to mix the test fluids.

8.1.4 The circulating pump is used to circulate the emulsion between the reservoir and the experimental section. The pump should be capable of pumping fluids ~~up to~~ at a speed of 50 ~~cm/s.~~ cm/s through the experimental section across the measuring pins. The flowrate is achieved with a flow controller or variable speed pump.