roform and diethyl ether (3:2) successively, combine the extracts in a tared vessel, and evaporate the combined extract on a water bath. Dry the residue over silica gel to constant mass: the mass of the residue is not more than 0.050 g.

(5) Nonvolatile residue—Evaporate 20.0 mL of Oxydol on a water bath to dryness, and dry the residue at 105°C for 1 hour: the mass of the residue is not more than 0.020 g.

Assay Pipet 1.0 mL of Oxydol, transfer it to a flask containing 10 mL of water and 10 mL of dilute sulfuric acid, and titrate with 0.02 mol/L potassium permanganate VS.

Each mL of 0.02 mol/L potassium permanganate VS
= 1.7007 mg of H_2O_2

Containers and storage Containers—Tight containers.
Storage—Light-resistant, and not exceeding 30°C.

Oxygen

酸素

O_2: 32.00

Oxygen contains not less than 99.5 v/v% of O_2.

Description Oxygen is a colorless gas, and is odorless.

1 mL of Oxygen dissolves in 32 mL of water, and in 7 mL of ethanol (95) at 20°C and at a pressure of 101.3 kPa.

1000 mL of Oxygen at 0°C and at a pressure of 101.3 kPa weighs about 1.429 g.

Identification (1) Put a glowing splinter of wood into Oxygen: it bursts into flame immediately.

(2) Transfer 1 mL each of Oxygen and oxygen directly from metal cylinders with a pressure-reducing valve to gas-measuring tubes or syringes for gas chromatography, using a polyvinyl chloride induction tube. Perform the test with these gases as directed under the Gas Chromatography according to the conditions of Purity (2): the retention time of principal peak from Oxygen coincides with that of oxygen.

Purity Keep the containers of Oxygen between 18°C and 22°C for not less than 6 hours before carrying out the following tests, and calculate the volume to be used with reference to the gas at 20°C and at 101.3 kPa.

(1) Acid or alkali—To 400 mL of freshly boiled and cooled water add 0.3 mL of methyl red TS and 0.3 mL of bromothymol blue TS, and boil for 5 minutes. Transfer 50 mL of this solution to each of three Nessler tubes marked A, B and C. Add 0.10 mL of 0.01 mol/L hydrochloric acid VS to tube A, 0.20 mL of 0.01 mol/L hydrochloric acid VS to tube B, stopper each of the tubes, and cool. Pass 1000 mL of Oxygen through the solution in tube A for 15 minutes, employing delivery tube with an orifice approximately 1 mm in diameter and extending to within 2 mm of the bottom of the Nessler tube: the color of the solution in tube A is not deeper orange-red than that of the solution in tube B and not deeper yellow-green than that of the solution in tube C.

(2) Carbon dioxide—Pass 1000 mL of Oxygen through 50 mL of barium hydroxide TS in a Nessler tube, in the same manner as directed in (1): any turbidity produced does not exceed that of the following control solution.

Control solution: To 50 mL of barium hydroxide TS in a Nessler tube add 1 mL of a solution of 0.1 g of sodium hydrogen carbonate in 100 mL of freshly boiled and cooled water.

(3) Oxidizing substances—Transfer 15 mL of potassium iodide-starch TS to each of two Nessler tubes marked A and B, add 1 drop of acetic acid (100) to each of the tubes, mix, and use these as solution A and solution B, respectively. Pass 2000 mL of Oxygen through solution A for 30 minutes in the same manner as directed in (1): the color of solution A is the same as that of the stoppered, untreated solution B.

(4) Chloride—Pour 50 mL of water into each of two Nessler tubes marked A and B, add 0.5 mL of silver nitrate TS to each of the tubes, mix, and use these as solution A an solution B, respectively. Pass 1000 mL of Oxygen through solution A in the same manner as directed in (1): the turbidity of solution A is the same as that of solution B.

(5) Nitrogen—Introduce 1.0 mL of Oxygen into a gas-measuring tube or syringe for gas chromatography from a metal hermetic container under pressure through a pressure-reducing valve and a directly connected polyvinyl tube. Perform the test as directed under the Gas Chromatography according to the following conditions, and determine the peak area A_T of nitrogen. Introduce 0.50 mL of nitrogen into the gas mixer, draw carrier gas into the mixer to make exactly 100 mL, and allow to mix thoroughly. Perform the test in the same manner with 1.0 mL of this mixture as directed above, and determine the peak area A_S of nitrogen: A_T is not larger than A_S.

Operating conditions—

Detector: A thermal-conductivity detector.

Column: A column about 3 mm in inside diameter and about 3 m in length, packed with 250- to 355-μm zeolite for gas chromatography (0.5 mm).

Column temperature: A constant temperature of about 50°C.

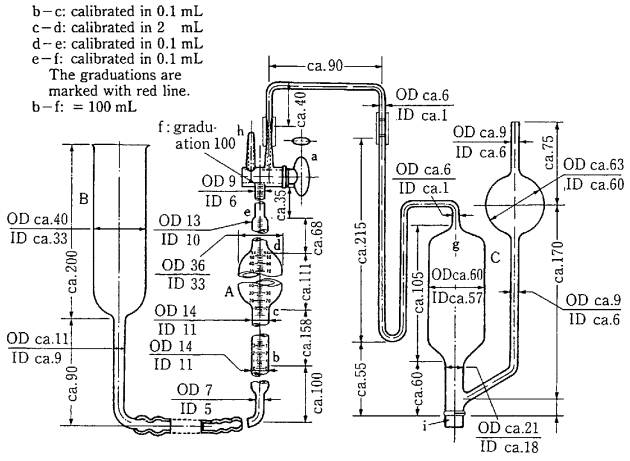
Carrier gas: Hydrogen or helium.

Flow rate: Adjust the flow rate so that the retention time of nitrogen is about 5 minutes.

Selection of column: Introduce 0.5 mL of nitrogen into a gas mixer, add Oxygen to make 100 mL, and mix well. Proceed with 1.0 mL of the mixture under the above operating conditions. Use a column giving well-resolved peaks of Oxygen and nitrogen in this order.

Assay (1) Apparatus—The apparatus is shown diagrammatically in the accompanying figure. A is a 100-mL gas buret having a two-way stopcock a, b - c, d - e and e - f are graduated in 0.1 mL, and c - d is graduated in 2 mL. A is properly connected with a leveling tube B by a thick rubber tube. Fill ammonium chloride-ammonia TS up to the middle of A and B. Place in the absorption ball g of the gas pipette C a coil of copper wire, not more than 2 mm in diameter, which extends to the uppermost portion of the bulb, add 125 mL of ammonium chloride-ammonia TS, and stopper with a rubber stopper i. Connect C with A using the thick rubber tube.

(2) Procedure—Open a, set B downward and draw the liquid in g to the stopcock opening a. Then close a. Open a to the intake tube h, and fill A and h with ammonium chloride-ammonia TS by lifting B. Close a, connect h with a container of Oxygen, open a, set B downward and measure accurately 100 mL of Oxygen. Open a toward C, and transfer the Oxygen to g by lifting B. Close a, and rock C gently for 5 minutes. Open a, draw the residual gas back into A by set-



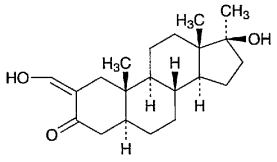
ting B downward, and measure the volume of the residual gas. Repeat the procedure until the volume of residual gas is constant, and designate this as V (mL). With fresh ammonium chloride-ammonia TS in C, repeat the procedure at least four times, and measure the volume of residual gas. Calculate V and the volume of Oxygen used as the sample with reference to the gas at 20°C and at 101.3 kPa.

Volume (mL) of oxygen (O_2)
= calculated volume of the sample (mL)
− calculated volume of V (mL)

Containers and storage Containers—Metal cylinders.
Storage—Not exceeding 40°C.

Oxymetholone

オキシメトロン



$C_{21}H_{32}O_3$: 332.48
17β-Hydroxy-2-hydroxymethylene-17α-methyl-5α-androstan-3-one [*434-07-I*]

Oxymetholone, when dried, contains not less than 97.0% and not more than 103.0% of $C_{21}H_{32}O_3$.

Description Oxymetholone occurs as a white to pale yellowish white, crystalline powder. It is odorless.

It is freely soluble in chloroform, soluble in 1,4-dioxane, sparingly soluble in methanol, in ethanol (95) and in acetone, slightly soluble in diethyl ether, and practically insoluble in water.

It is gradually colored and decomposed by light.

Identification (1) Dissolve 2 mg of Oxymetholone in 1 mL of ethanol (95), and add 1 drop of iron (III) chloride TS: a purple color develops.

(2) Dissolve 0.01 g of Oxymetholone in methanol to make 50 mL. To 5 mL of the solution add 5 mL of sodium hydroxide-methanol TS and methanol to make 50 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) Determine the infrared absorption spectrum of Oxymetholone as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

Optical rotation $[\alpha]_D^{20}$: +34 – +38° (after drying, 0.2 g, 1,4-dioxane, 10 mL, 100 mm).

Melting point 175 – 182°C

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Oxymetholone in 25 mL of 1,4-dioxane: the solution is clear, and shows a colorless to pale yellow color.

(2) Other steroids—Dissolve 0.050 g of Oxymetholone in 5 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add chloroform to make exactly 200 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography, and air-dry the spot. Develop immediately the plate with a mixture of toluene and ethanol (99.5) (49:1) to a distance of about 12 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate, and heat at 100°C for 3 to 5 minutes: any spot other than the principal spot and starting point obtained from the sample solution is not more intense than the spot from the standard solution.

Loss on drying Not more than 1.0% (0.5 g, in vacuum, phosphorus (V) oxide, 4 hours).

Residue on ignition Not more than 0.15% (0.5 g).

Assay Weigh accurately about 0.04 g of Oxymetholone, previously dried, and dissolve in methanol to make exactly 50 mL. Pipet 5 mL of this solution, and add methanol to make exactly 50 mL. To exactly measured 5 mL of this solution add 5 mL of sodium hydroxide-methanol TS and methanol to make exactly 50 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 315 nm, using a solution, prepared by adding methanol to 5 mL of sodium hydroxide-methanol TS to make 50 mL, as the blank.

$$\text{Amount (mg) of } C_{21}H_{32}O_3 = \frac{A}{541} \times 50{,}000$$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.