plate to about 5 mL. Cool, repeat the oxidation with 3 g of potassium chlorate and 30 mL of nitric acid, and evaporate to about 5 mL. Add 25 mL of hydrochloric acid, and again evaporate to about 5 mL. Add 100 mL of water, heat to boiling, filter, and wash well. To the hot filtrate add 25 mL of barium chloride TS, and heat on a steam bath for 1 hour. Collect the barium sulfate on a previously ignited and tared filtering crucible, wash, dry, and ignite, then cool, and weigh. Each g of barium sulfate is equivalent to 137.4 mg of S.

Ichthammol Ointment

» Ichthammol Ointment contains an amount of Ichthammol equivalent to not less than 0.25 percent of ammonia (NH_3).

Ichthammol	100 g
Lanolin	100 g
Petrolatum	800 g
to make	1000 g

Thoroughly incorporate the Ichthammol with the Lanolin, and combine this mixture with the Petrolatum.

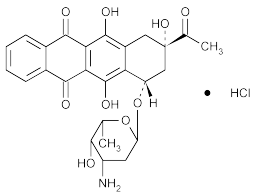
Packaging and storage—Preserve in collapsible tubes or in tight containers, and avoid prolonged exposure to temperatures exceeding 30°.

Assay—

Assay preparation—Transfer an accurately weighed portion of Ointment, equivalent to about 2 g of ichthammol, to a 250-mL beaker, and add about 70 mL of boiling water. Mix with a glass rod, heat on a steam bath, with frequent agitation, for 10 minutes, cover with a watch glass without removing the stirring rod, and allow to stand at room temperature for 15 to 20 minutes. Place in a refrigerator to cause the upper layer to congeal, form a second opening through the congealed layer with the glass rod, and transfer the dark-colored aqueous extract to a funnel containing a pledget of cotton, collecting the filtrate in a 500-mL volumetric flask. Repeat the extraction of the portion of the Ointment several times in the same manner until the aqueous extract is practically colorless, passing each extract through the same cotton filter into the flask containing the main extract. Dilute with water to volume, and mix.

Procedure for ammonia—Transfer 100.0 mL of the *Assay preparation* to a suitable distillation flask, add 3 g of paraffin, and add 20 mL of sodium hydroxide solution (4 in 10). Connect the flask to a condenser by means of a spray trap, and immerse the lower outlet tube of the condenser in 30.0 mL of 0.05 N sulfuric acid VS. Distill slowly, collect about 50 mL of distillate, and then titrate the excess acid with 0.05 N sodium hydroxide VS, using methyl red TS as the indicator. Perform a blank determination, and make any necessary correction. Each mL of 0.05 N sulfuric acid is equivalent to 0.8515 mg of NH_3.

Idarubicin Hydrochloride



$C_{26}H_{27}NO_9 \cdot HCl$ 533.95

5,12-Naphthacenedione, 9-acetyl-7-[(3-amino-2,3,6-trideoxy-α-L-*lyxo*-hexopyranosyl)oxy]-7,8,9,10-tetrahydro-6,9,11-trihydroxyhydrochloride, (7*S*-*cis*)-.

(1*S*,3*S*)-3-Acetyl-1,2,3,4,6,11-hexahydro-3,5,12-trihydroxy-6,11-dioxo-1-naphthacenyl 3-amino-2,3,6-trideoxy-α-L-*lyxo*-hexopyranoside, hydrochloride [57852-57-0].

» Idarubicin Hydrochloride contains not less than 960 µg and not more than 1030 µg of $C_{26}H_{27}NO_9 \cdot HCl$ per mg, calculated on the anhydrous basis.

Caution—Great care should be taken to prevent inhaling particles of Idarubicin Hydrochloride and exposing the skin to it.

Packaging and storage—Preserve in tight containers.

Labeling—The amorphous form is so labeled.

USP Reference standards ⟨11⟩**—**
USP Idarubicin Hydrochloride RS

Identification—

A: *Infrared Absorption* ⟨197K⟩.

B: The chromatogram of the *Assay preparation* obtained in the *Assay* exhibits a major peak for idarubicin, the retention time of which corresponds to that in the chromatogram of the *Standard preparation* obtained in the *Assay*.

Crystallinity ⟨695⟩: meets the requirements, except where it is labeled as amorphous, most of the particles do not exhibit birefringence and extinction positions.

pH ⟨791⟩: between 5.0 and 6.5, in a solution containing 5 mg per mL.

Water, *Method I* ⟨921⟩: not more than 5.0%.

Chromatographic purity—Using the chromatogram of the *Assay preparation* obtained in the *Assay*, and disregarding the solvent peak, calculate the percentage of each impurity taken by the formula:

$$100 r_i / r_S$$

in which r_i is the response of each impurity peak; and r_S is the sum of the responses of all the peaks: not more than 1.0% of any individual impurity is found; and the sum of all impurities is not more than 3.0%.

Assay—

Mobile phase—Prepare a mixture of water, acetonitrile, methanol, and phosphoric acid (540:290:170:2). Dissolve 1 g of sodium lauryl sulfate in 1000 mL of this solution, adjust with 2 N sodium hydroxide to a pH of 3.6 ± 0.1, pass through a filter having a porosity of 0.5 µm or finer, and degas. Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Diluent—Prepare as directed for *Mobile phase*, except to omit the sodium lauryl sulfate.

Standard preparation—Dissolve an accurately weighed quantity of USP Idarubicin Hydrochloride RS in *Diluent* to obtain a solution having a known concentration of about 500 µg of idarubicin hydrochloride per mL.

Assay preparation—Transfer about 50 mg of Idarubicin Hydrochloride, accurately weighed, to a 100-mL volumetric flask, dissolve in *Diluent*, dilute with *Diluent* to volume, and mix.

Resolution solution—Prepare an aqueous solution containing 1 mg of Idarubicin Hydrochloride per mL. T o 2 mL of this solution in a test tube, add 20 µL of hydrochloric acid, and heat in an oil bath at 95 ° for about 8 minutes. Mix 1 mL of this solution and 9 mL of *Diluent*. This *Resolution solution* contains a mixture of 4-demethoxydaunorubicinone and idarubicin.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4.6-mm × 25-cm column that contains packing L13. The flow rate is about 2 mL per minute. Chromatograph the *Resolution solution*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.5 for 4-demethoxydaunorubicinone and 1.0 for idarubicin; and the resolution, *R*, between the 4-demethoxydaunorubicinone peak and the idarubicin peak is not less than 9.5. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the capacity factor, *k'*, for the idarubicin peak is not less than 10 and not more than 20; the tailing factor for the idarubicin peak is not less than 0.85 and not more than 1.2; the column efficiency calculated from the idarubicin peak is not less than 3000 theoretical plates; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. Calculate the quantity, in µg, of $C_{26}H_{27}NO_9 \cdot HCl$ in each mg of the Idarubicin Hydrochloride taken by the formula:

$$100(C/M)(r_U / r_S)$$

in which *C* is the concentration, in µg per mL, of idarubicin hydrochloride ($C_{26}H_{27}NO_9 \cdot HCl$) in the *Standard preparation; M* is the quantity, in mg, of Idarubicin Hydrochloride taken to prepare the *Assay preparation;* and r_U and r_S are the responses of the idarubicin peak obtained from the *Assay preparation* and the *Standard preparation*, respectively.

Idarubicin Hydrochloride for Injection

» Idarubicin Hydrochloride for Injection is a sterile mixture of Idarubicin Hydrochloride and Lactose. It contains not less than 90.0 per cent and not more than 110.0 per cent of the labeled amount of $C_{26}H_{27}NO_9 \cdot HCl$.

Caution—Great care should be taken to prevent inhaling particles of Idarubicin Hydrochloride and exposing the skin to it.

Packaging and storage—Preserve in *Containers for Sterile Solids* as described under *Injections* ⟨1⟩.

USP Reference standards ⟨11⟩**—**
USP Endotoxin RS
USP Idarubicin Hydrochloride RS

Constituted solution—At the time of use, it meets the requirements for *Constituted Solutions* under *Injections* ⟨1⟩.

Identification—The chromatogram of the *Assay preparation* obtained in the *Assay* exhibits a major peak for idarubicin, the retention time of which corresponds to that in the chromatogram of the *Standard preparation* obtained in the *Assay*.

Bacterial endotoxins ⟨85⟩—It contains not more than 8.9 USP Endotoxin Units per mg of idarubicin hydrochloride, a solution of Idarubicin Hydrochloride for Injection containing 0.07 mg of idarubicin hydrochloride per mL being used in the *Test Procedure*.

Sterility ⟨71⟩—It meets the requirements when tested as directed for *Membrane Filtration* under *Test for Sterility of the Product to be Examined*.

pH ⟨791⟩: between 5.0 and 7.0, in a solution constituted as directed in the labeling, water being used as the diluent.

Water, *Method I* ⟨921⟩: not more than 4.0%, the *Test Preparation* being prepared as directed for a hygroscopic specimen.

Other requirements—It meets the requirements for *Uniformity of Dosage Units* ⟨905⟩ and for *Labeling* under *Injections* ⟨1⟩.

Assay—

Mobile phase, Diluent, Standard preparation, Resolution solution, and *Chromatographic system*—Proceed as directed in the *Assay* under *Idarubicin Hydrochloride*.

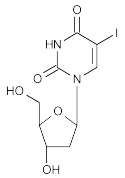
Assay preparation—Dilute the contents of 1 container of Idarubicin Hydrochloride for Injection quantitatively with *Diluent* to obtain a solution containing about 0.5 mg of idarubicin hydrochloride per mL.

Procedure—Proceed as directed for *Procedure* under *Idarubicin Hydrochloride*. Calculate the quantity, in mg, of $C_{26}H_{27}NO_9 \cdot HCl$ in the container of Idarubicin Hydrochloride for Injection taken by the formula:

$$(C / 1000)(L / D)(r_U / r_S)$$

in which *C* is the concentration, in µg per mL, of idarubicin hydrochloride ($C_{26}H_{27}NO_9 \cdot HCl$) in the *Standard preparation; L* is the labeled quantity, in mg, of idarubicin hydrochloride in the container; *D* is the concentration, in mg per mL, of idarubicin hydrochloride in the *Assay preparation* on the basis of the labeled quantity in the container and the extent of dilution; and r_U and r_S are the responses of the idarubicin peak obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Idoxuridine



$C_9H_{11}IN_2O_5$ 354.10
Uridine, 2'-deoxy-5-iodo-.
2'-Deoxy-5-iodouridine [54-42-2].

» Idoxuridine contains not less than 98.0 per cent and not more than 101.0 per cent of $C_9H_{11}IN_2O_5$, calculated on the dried basis.

Packaging and storage—Preserve in tight, light-resistant containers.

USP Reference standards ⟨11⟩**—**
USP Idoxuridine RS

Identification—

A: *Infrared Absorption* ⟨197M⟩.

B: *Ultraviolet Absorption* ⟨197U⟩—

Solution: 35 µg per mL.

Medium: pH 12.0 buffer (prepared from 7.46 g of potassium chloride and 24 mL of 1 N sodium hydroxide dissolved in 2000 mL of water).

Absorptivities at 279 nm, calculated on the dried basis for the test sample only, do not differ by more than 2.0%.

Loss on drying ⟨731⟩—Dry about 500 mg, accurately weighed, in vacuum at 60 ° for 2 hours: it loses not more than 1.0% of its weight.