IDENTIFICATION TESTS

FOR

DURACOR TABLETS

PROTOCOL APPROVALS

Norvin Pharma Inc.	Signature and Date
Author Analytical Laboratory	
Approver Analytical Laboratory Group Leader	
Approver Manager Quality Control Chemistry	
Approver Regulatory Compliance Auditor Regulatory Compliance	

1.0 INTRODUCTION

The purpose of identification tests is to uniquely identify an article. We will use three separate Identification Tests to prove the identity of Duracor:

Infrared Spectroscopy (IR)
Melting Point Determination
Ultraviolet Spectroscopy (UV)

One absolute procedure is generally the preferred approach for the identification of a compound. Thus, an infrared spectroscopy (IR) or similar spectroscopic identification tests are preferred over wet chemistry or colorimetric tests, because the spectroscopic procedures provide a conclusive identification. The IR absorption spectrum of a substance, compared with that obtained concomitantly for the corresponding Reference Standard provides perhaps the most conclusive evidence of the identity of a substance that can be realized from a single test.

The determination of melting points is one of the oldest identification methods for organic substances. The melting point of a substance is the temperature at which the material changes from a solid to a liquid state. Pure crystalline substances have a clear, sharply defined melting point.

The UV absorption spectrum on the other hand does not exhibit a high degree of specificity but provides additional evidence in the identity of a compound.

Conformance with the IR absorption test, the UV absorption test specifications, and the melting point specifications leaves little doubt regarding the identity of the compound under observation.

2.0 TEST METHODS

Identification tests in Norvin Pharma monograph “Duracor”, current version.

Sample preparation:

Grind 1 Duracor tablet to a fine powder using a mortar and pestle, transfer the powder to a 50 mL plastic screw-top vial, and add 10 mL of ethyl alcohol. Place the flask on a mechanical shaker for 5 minutes. Pour the mixture into a centrifuge tube and centrifuge for 5 minutes. Filter the supernatant solution through Whatman #1 filter paper. Take 4 drops of the resulting

filtrate, add it to a new 50 mL plastic screw-top vial, dilute to 50 mL with anhydrous ethanol and mix. This solution will be used for the UV determination. Additionally take approximately 2 mL of the filtrate, place in a petri dish and evaporate the solution to dryness on a hot plate. It may be necessary to induce crystallization by scratching the dish with a metal spatula. The resulting solids will be used for the IR and melting point determinations.

3.0 EXPERIMENTAL PROCEDURE FOR IR ANALYSIS

In this experiment you will compare the IR spectra of your sample with the IR spectra of a reference standard.

Equipment and Supplies

Infrared spectrophotometer
Agate mortar and pestle
Kimwipes, spatula, micro transfer pipettes
Duracor reference standard

Reagents

Nujol (mineral oil)
Isopropanol

Preparing a Nujol Mull for the Sample and Reference Standard

1. Using the spatula, transfer a very small amount of your sample to the agate mortar.
2. Grind the sample to a very fine powder using the agate pestle.
3. Add 4 drops of Nujol (mineral oil) and grind to disperse the sample (forms a Nujol mull).
4. Repeat this procedure to prepare the Duracor reference standard mull.

Running a Background IR Spectrum

1. Place one salt plate in the sample holder and lay the holder flat on the bench top.
2. Place a second salt plate over the first.

3. Place the cover over the plates and insert the holder into the IR spectrophotometer.
4. Record the background spectrum.

Running a Nujol IR Spectrum

1. Place one salt plate flat on the bench top.
2. Place 1 drop of Nujol on one the face of the salt plate.
3. Place a second salt plate over the first to spread the Nujol.
4. Carefully place the salt plate sandwich into the sample holder.
5. Place the cover over the plates and insert the holder into the IR spectrophotometer.
6. Record the Nujol spectrum.
7. After obtaining the spectrum, disassemble the holder and wipe both plates clean with a Kimwipe.

Running a Sample and Reference Standard IR Spectrum

1. Gently mix the Nujol mull one last time.
2. Place one salt plate flat on the bench top.
3. Place one drop of the Nujol mull on the face of the salt plate.
4. Place a second salt plate over the first to spread the Nujol.
5. Carefully place the salt plate sandwich into the sample holder.
6. Place the cover over the plates and insert the holder into the IR spectrophotometer.
7. Record the sample spectrum.
8. After obtaining the spectrum, disassemble the holder and wipe both plates clean with a Kimwipe. Place a few drops of isopropanol on each plate and wipe clean with a new Kimwipe.
9. Repeat the above steps to obtain the Duracor reference standard spectrum.

Reviewing the IR Spectra

Look closely and compare the absorbance bands in the entire IR spectra (from 600 cm-1 to 4000 cm-1) for the sample spectra and the reference standard spectra.

Requirement

There should only be maxima at the same wavelenghts for the sample and the reference standard.

4.0 EXPERIMENTAL PROCEDURE FOR UV ANALYSIS

In this experiment you will compare the UV spectra of your sample with the UV spectra of a reference standard.

Equipment and Supplies

UV/Vis Spectrophotometer
Matched quartz cells
Duracor reference standard
Anhydrous ethanol
Kimwipes
50 mL plastic screw-top vials

Reference Standard Solution Preparation

Accurately weigh 8 mg of Duracor reference standard into a 50 ml plastic screw-top vial and add approximately 20 mL of anhydrous ethanol. Place the flask on a mechanical shaker for 5 minutes or until the reference standard is dissolved. Dilute to volume with anhydrous ethanol and mix.

Running a UV Spectrum

1. Clean the quartz cells by rinsing them with anhydrous ethanol
2. Fill both cells with anhydrous ethanol and dry the outsides with a Kimwipe

3. Place the cells in the reference and sample cell holders within the spectrometer with the clear sides of each cell facing the open slots of the cell holder
4. Click the grey start button on the computer monitor
5. When the dialog box comes up for the blank, click OK. A background correction is carried out from the maximum to the minimum wavelength range (400 nm – 200 nm).
6. Wait for the next dialog box to come up
7. Take out the sample cell (front cell) and place its contents into the waste beaker
8. Fill the sample cell until approximately ¾ full with the sample solution and wipe the outside of the cell with a Kimwipe. Place the sample cell back into the spectrometer and click OK to record the sample spectrum
9. Repeat steps 6 – 8 for the reference standard solution

Reviewing the UV Spectra

Look closely and compare the absorbance bands in the entire UV spectra (400 nm – 200 nm) for the sample spectra and the reference standard spectra.

Requirement

The requirements are met if the UV absorbance spectra of the sample solution and the standard solution exhibit maxima and minima at the same wavelenghts and provides a similar profile.

5.0 EXPERIMENTAL PROCEDURE FOR MELTING POINT DETERMINATION

In this experiment you will compare the melting point range of your sample with the melting point range of a reference standard.

Equipment and Supplies

Melting point apparatus
Melting point capillaries
Duracor reference standard

Procedure

1. Dip the open end of a melting point capillary into the pile of sample crystals
2. Invert it and tap on the desk to collect the crystals in the bottom of the tube
3. Repeat steps 1 and 2 for the reference standard
4. Place the capillaries into the melting point apparatus
5. Turn the unit on, set the dial to #2 (approximately 80 ° C) to start and watch the sample and standard through the sight glass as the temperature increases.
6. Record the temperature when the sample and reference standard begins to melt and the temperature when they fully melt.

Evaluation of Results

Compare the melting range of the sample and the reference standard. The melting point range should be approximately 122 to 126 ° C.

Requirement

The requirements are met if the melting range of the sample and the reference standard are comparable.