

# **EXPERIMENTAL**

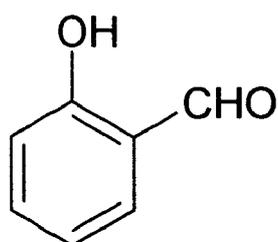
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## 2. EXPERIMENTAL

### 2-1. MATERIALS:

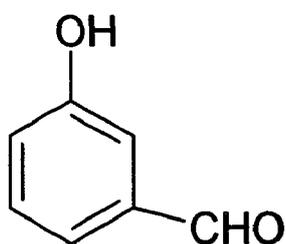
#### 2-1-1. Aromatic Aldehyde

##### a. o- Hydroxy benzaldehyde (Salicylaldehyde)



It was obtained from WINLAB Ltd (UK) with the following specifications: Clear, colorless, M.wt. 122.12, M.p 1-2 °C, B.P 197 °C, d =1.146

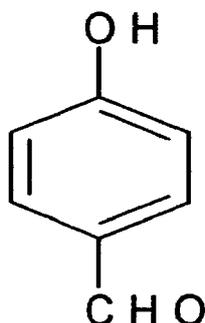
##### b. m- Hydroxy benzaldehyde



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It was obtained from MERCK-SCHUCHARDT. It has the following : characteristics: M.wt. 122.12, M.p 103-105 °C, B.P 191 °C

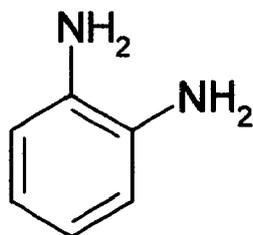
**c. p- Hydroxy benzaldehyde**



It was obtained from BDH, ENGLAND. It has the following characteristics: M.wt. 122.12, M.p 117-119 °C

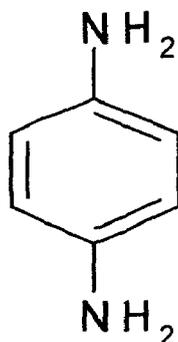
**2-1-2. Amines**

**a. o-Phenylene diamine**



It was obtained from ALDRICH – GERMANY with the following specification:

M.wt 108.14, M.p 100-102 °C, B.p 256 - 258 °C

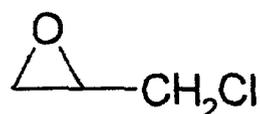
**b. p -Phenylene diamine**

It was obtained from ALDRICH – GERMANY with the following specification: M.wt 108.14, M.p 143-145 °C, B.p 267 °C

**c. Pentaethylenhexamine (PEHA)**

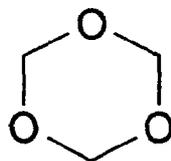
Formula  $H_2N-(CH_2-CH_2-NH)_4-CH_2-CH_2-NH_2$

It was obtained from Fluka GMBH with the following specification: M.wt 232.38

**2-1-3. Reagents****a. Epichlorohydrin (EC)**

It was obtained from FERAk LABORAT GMBH BERLIN (WEST) with the following specification: Clear, colorless, M.wt 92.53, B.p 92-93 °C, viscosity :1.12cps at 20 °C

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**b. 1, 3, 5 - Trioxane**

It was obtained from Fluka GMBH with the following specification: M.wt 90.08, M.p 59-62 °C, B.p 112-115 °C

**c. Acetic acid glacial**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics M. wt= 60.05, Sp.gr.1.05, Assay 99 %

**d. Sodium hydroxide**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. M.wt 40.07

**e. Sulphuric acid**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics: M. WT= 98.07, Sp.gr.1.834 – 1.835, Assay 97 – 99 %

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## **2-1-4. Solvents**

### **a. Ethyl alcohol absolute**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics: M.wt = 46.07, density = 0.793 g / ml

### **b. Methyl ethyl ketone (MEK)**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics: M. WT= 72.11, B.p 80 °C, d = 0.805 Min. Assay 99 .5 %

### **c. Acetone**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics: M. wt= 50.08, Sp.gr.0.789 - 0.791, B.p, 56-57 °C

### **d. Diethylether (ethoxy ether)**

It was obtained from MAY & BAKER LTD. with the following specification:

M.wt 74.12, B.p 34-36 °C, Sp.gr.0.714 - 0.716

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**e. Petroleum ether (60/80)**

It was obtained from ELNASR PHARMACEUTICAL Co.

It has the following characteristics: Sp.gr.0.675 - 0.695, B.p 60-80 °C

**f. Methyl alcohol**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics M. wt= 32.04, Sp.gr.0.790 - 0.793, B.p 64 °C

**g. Chloroform**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics M. wt= 119.38, B.p 60.4 °C

**h. Ethyl acetate**

It was purchased from EDWIC, Laboratory chemical. It was packed and distributed by ELNASR PHARMACEUTICAL Co. It has the following characteristics M. wt= 88

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## **2-2. METHODS AND TECHNIQUES:**

### **2-2-1. Synthesis of Schiff Base of Monomers:**

The reactions were completed in three-necked flask (0.5 L capacity) equipped with a condenser, magnetic stirrer, thermometer, dropping funnel and nitrogen atmosphere inlet as represented in *Figure 2.1*. In this respect, the flask was charged with Phenylenediamine (27gm, 0.25 mole) and 200 ml absolute ethanol, with stirring well with magnetic stirrer on hot plate. Hydroxy benzaldehyde derivatives (61gm, 0.5 mole) were dissolved in 200 ml absolute ethanol and the solution was transferred to dropping funnel to add into three necked flask within one hour with stirring in presence of nitrogen gas. The temperature of reaction was gradually increased up to reflux for 3 hours. The reaction was cooled to filtrate the solution. The reaction yields were determined as well as physicochemical characteristics such as color and melting point.

### **2-2-2. Synthesis of Schiff Base (5, 5'-Methylene -bis-salicylaldehyde):**

The reaction was carried out in reaction flask which represented in *Figure 2.1*. Salicylaldehyde (80gm, 0.655 mole), glacial acetic acid (50ml) and trioxane (7gm) were charged in reaction flask with stirring well with magnetic

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stirrer and hot plate. Mixture of 0.5ml conc. sulphuric acid and 2.5 ml of glacial acetic acid was added slowly using dropping funnel and the mixture was stirred. The reaction temperature was increased up to 90 – 95 °C under nitrogen atmosphere. The temperature was maintained for twenty two hours with stirring. The reaction temperature was cooled and the mixture was poured into 3 l of ice water and allowed to stand overnight. The solid product was filtered and extracted twice with 100 ml of petroleum ether to remove water phase. The isolated solid was recrystallized from 150 ml acetone.

### **2-2-3. Synthesis of Polymer of Schiff Base:**

A three- necked flask (0.5 L capacity) equipped with a condenser, magnetic stirrer, thermometer and dropping funnel was charged with 5,5'-methylene -bis-salicylaldehyde (15gm) , absolute ethanol (50ml) and glacial acetic acid (200ml). The reaction mixture was stirred well with magnetic stirrer and hot plate. A solution of o-phenylenediamine (6.66gm) in glacial acetic acid (100ml) was added slowly using dropping funnel. After 10 seconds a bright yellow precipitate was formed. Methanol (50 ml) was added with stirring .The solid product was isolated by filtration and washed with methanol. The product was examined for structure characterization.

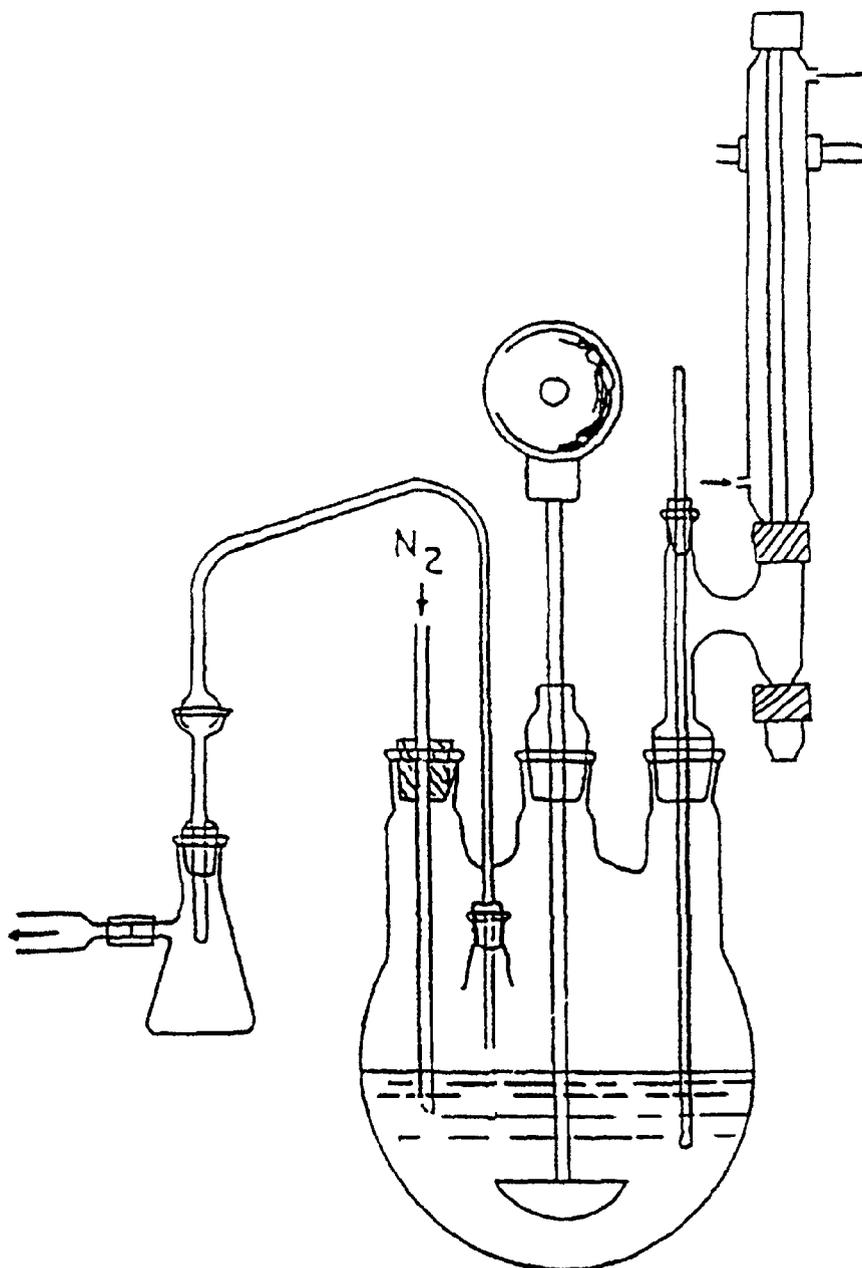
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#### 2-2-4. Synthesis of Epoxy Binders from Schiff Base

##### Monomers:

A three-necked flask (0.5 L capacity) equipped with a condenser, magnetic stirrer, thermometer and dropping funnel was charged with Schiff base monomers (31.6gm, 0.1 mole) and epichlorohydrin (92gm, 1 mole). The reaction mixture was stirred well with magnetic stirrer on hot plate at 40 °C under nitrogen atmosphere for 1 hour. The reaction temperature was raised up to 90 – 100 °C. Sodium hydroxide (8 gm, 0.2 moles in 16 gm water) 50 % concentrated solution was transferred to dropping funnel and added to the reaction flask within two hours with stirring.

The reaction temperature was raised to reflux for one hour at 90 – 100 °C. The reaction temperature was cooled to filtrate the solution. The obtained resins were isolated under pressure using rotary evaporator and they were examined for structure determination. The same reaction condition was used to obtain epoxy binder based on Schiff base polymer.



**Figure(2.1): Diagrammatic Kettle Sketch Used for Polycondensation Reaction**

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### **2-2-5. Curing of the Prepared Epoxy Resins**

Pentaethylenehexamine (PEHA) and p- phenylenediamine (PDA) were used as curing agents for the curing of Schiff base monomers and polymer. Epoxy binders were mixed with (PDA) as curing agents in methyl ethyl ketone (MEK) as solvent to prepare 90% solution of the reaction mixture. After evaporation of the solvent at room temperature, the mixture was dried perfectly in vacuum oven and stored at  $-10\text{ }^{\circ}\text{C}$  before use to prevent the curing reaction of the mixture. The weight ratios between epoxy binders were ranged between 4:1, 3:1, 2:1 and 1: 1 as weight of epoxy: weight of curing agent. Cured network of epoxy binders was prepared after curing at  $130\text{ }^{\circ}\text{C}$  for 2 h and post curing at  $145\text{ }^{\circ}\text{C}$  for 1h.

### **2-3. CHARACTERIZATION**

#### **2-3-1. Measurements:**

The hydroxyl value is defined as the number of milligram of potassium Hydroxide equivalent to the hydroxyl content of one gram of sample.

The hydroxyl values were determined by the conventional acetic anhydride/ pyridine method [181]. In this respect, 2g of resin was weighted to the nearest milligram and dissolved in 25ml of acetic anhydride and pyridine mixture (1: 4 by volume). The solution was refluxed for 1h the condenser is then washed out by the addition of 5ml water, and the heating

is continued for 5 min. The heat is then removed and the condenser tube and washed with 25 ml of methanol. When the mixture has cooled to room temperature, it is titrated with approximately 0.1 N standard KOH aqueous solution to phenolphthalein end point. The volume of KOH was determined to calculate hydroxyl values (mg KOH /g of resin) from the following relation.

$$\text{Hydroxyl value} = \frac{\text{Volume of KOH} \times 56.1 \times N \text{ of KOH}}{\text{Weight of resin}}$$

The epoxy content of resins is frequently expressed as weight per epoxide or epoxide equivalent weight (EEW), which is defined as the weight in grams which contains one gram equivalent epoxide. In this respect, 0.5 g of the sample was dissolved in 20 ml HCl / pyridine solution (1L.pyridene +16ml.concenterated HCl) and boiling in flask with reflux condenser for 30 minutes. The excess of HCl is titrated against 0.5 N KOH. In the same time a control (blank) experimental is carried out.

$$\text{The epoxy group content} = \frac{(a - b) \times K \times 0.028 \times 100}{g}$$

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Where (a) = quantity of KOH in ml taken for titration the acid in the control experiment (blank), (b) = quantity of KOH in ml taken for titrating the acid in the sample,  $k = 0.998$  and  $g$  is weight of the sample.

### **2-3-2. Color of Resins:**

Color measurement [182] was conducted using the "Gardner 1953 standard colors". The method determines the color by comparison with standards of definite color compositions. The material in which color is matched was inserted inside clear glass color tubes 10.65 +0.25 mm inside diameter and 114 +1 mm out side length. Gardner color standards, consists of 18 colors numbered from 1 to 18. The transparent sample solution was poured (at temperature of 25 + 5 °C) into an empty color tube. The tube was placed next to the standard it most nearly approaches in light intensity. The rack containing the standards was held the standard and sample was reported in terms of one of the 18 Gardner standards. If a sample falls between two standards, it will be reported as " + " or " - ", depending upon which standards, it most closely approaches. If it closer to the lower of the two standards, it will be reported as " + ". If it is closer to the higher of the two standards, it will be reported as " - ". Any sample darker than 18 was reported as ">18

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### 2-3-3. Infrared (IR)

IR spectra was carried out in the laboratories of Egyptian Petroleum Research Institute. Recorded on Mattson –Infinity series FTIR Bench Top 961(in KBr). Infrared (IR) radiation refers broadly to that part of the electromagnetic spectrum between the visible and micro wave regions of greatest practical use to the organic chemist is the limited portion between 4000 and 400  $\text{cm}^{-1}$ . These has been same interest in the near –IR. [14,290- 4000  $\text{cm}^{-1}$ ] and the far –IR regions 700-200  $\text{cm}^{-1}$ . Band intensities can be expressed either as transmittance (T) or absorbance (A). Transmittance is the ratio of the radiant power transmitted by a sample to the radiant power incident on the sample. Absorbance is the logarithm, to the base 10, of the reciprocal of the transmittance  $A = \text{Log } 10 (1/T)$ . Usually report intensity in semi quantitative terms (S = Strong, M = Medium, W= Weak).

### 2-3-4. $^1\text{H}$ NMR Analysis

$^1\text{H}$ NMR analysis was carried out for the prepared resins in the laboratories of Cairo university. Recorded on a 270 MHz spectrometer W-P-270 &Y Bruker. The solutions for  $^1\text{H}$ NMR analysis were prepared by dissolving the prepared resins in  $\text{CDCl}_3$ .

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### **2-3-5. GPC Technique**

The synthesized epoxy binders were characterized in terms of weight, number average molecular weight ( $M_w$  and  $M_n$ ), polydispersity and molecular weight distribution using GPC technique. The molecular weight of binders were measured by GPC (Waters model 510) at the following condition:-

Solvent : THF HPLC grade

Column : Ultrastyrigel 500; 1000; 10.000; 100.000 °A

Temperature : 25°C

Flow speed : 18 cm<sup>3</sup>/min

GPC measurements were completed using polystyrene molecular weight standards.

### **2-3-6. Differential Scanning Calorimetric (DSC):**

Transition temperature of epoxy binders was measured with DuPont 2100 differential scanning calorimeter at heating rate of 10 °C/min. Dynamic curing was performed in DSC to observe the curing behavior of epoxy binders. Dynamic curing was performed at a heating rate of 20 °C/ min.

### **2-3-7. Thermal Gravimetric Analysis (TGA):**

Thermal stability of epoxy binders was determined by Perkin Elmer TGA 7 system under N<sub>2</sub> gas flow at heating rate of 10 °C /min. The start and final temperatures were 25 and 600°C,

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respectively. The weight of the samples was always about 1 mg.

## **2-4. COATING EVALUATION:**

### **2-4-1. Preparation of Steel Panels for Testing:**

Before blast cleaning and application of the coating system, the panel shall be free of all surface contaminates. Oil, grease, and other contaminates shall be removed by suitable solvent, detergent or potable water. The treatment involved the following steps [183]:

1. Remove the rust by hand tool cleaning (wire brush or sanding paper) to achieve the requirements of St2, **SIS 05590 and BS 7079**.
2. The rust free panels wiped vigorously on both sides with a cloth wet with methanol.
3. The panels were allowed to dry at room temperature (15 minutes). Then rubbed briskly with a clean, lintless cloth to remove any adhering dirt.
4. Then, the panels were dipped in phosphoric acid (1:1) at 25 °C for one minute and rinsed immediately by distilled water for a minimum 15 sec.
5. The panels were dried at a temperature between 50 °C – 100 °C and coated immediately or stored in desiccators for future use.

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6. The panels should not be touched with the fingers during the process of preparation.
  7. The coated panels should be scribed to bare steel, the scribe line was approximately 10cm

#### **2-4-2. Pinhole Test:**

Test which verifies the continuity of the coating shall be carried out on a coated surface before and after the coat has completely cured. The coating application shall be made on prepared glass panel 1 x 3 inches (25 x75 mm) examination of panel shall be made as follow [160]:

##### **a) Wet film**

Five minutes after application of the coating material, an examination for pinholes in the film shall be made by holding the panel to a strong light source, holding slide over a slot in a container which houses a 100 watt bulb, the bulb being a distance of between 4 and 5 inches ( 100 and 130 mm) from the coating slide. An opaque shield to prevent light interference to observation of the panel being viewed support the panel over viewing slot. The opaque shield shall extend a minimum of 6 inches around all sides of the panel. Any pinhole shall constitute a failure.

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**b) Cured film**

If the coating is acceptable in the wet state above, it shall be held an additional 15 to 30 minutes for air curing and then placed in a circulating warm air oven at 66 to 79 °C for a minimum 30 minutes. Observation to the light source described above shall be repeated. Any pinhole shall constitute a failure.

**2-4-3. Drying, Curing, or Film Formation of Organic Coatings at Room Temperature:**

To determine **Set-To-Touch Time** lightly touch the test film at point not less than 15 mm from the film edges with the tip of a clean finger and immediately place the fingertip against a piece of clean, clear glass. Observe if any of the coating is transferred to the glass. The film is considered to have set-to-touch when it still shows a tacky condition, but none of it adheres to the finger.

**Dry-Hard Time:** With the end of the thumb resting on the test film and the forefinger supporting the test panel, exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film is considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

**Dry-Through (or Dry-To-Handle) Time:** Place the test panel in a horizontal position at a height such that when the thumb is

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placed on the film, the arm of the operator is in a vertical line from the wrist to the shoulder. Bear down on the film with the thumb, exerting the maximum pressure of the arm, at the same time turning the thumb through an angle of  $90^{\circ}$  in the plane of the film. The film is considered dry-through or dry-to-handle when there is no loosening, detachment, wrinkling, or other evidence of distortion of the film [184].

#### **2-4-4. Measurement of Film Thickness:**

There are four methods of determining the dry film thickness of paint:

- a- Non-destructive test gauge
- b- Destructive test gauge
- c- Test panels
- d- Calculation

Measuring DFT directly with non-destructive test is the most widely used method [185]: The instrument used for measuring the dry film thickness (DFT) was the " Minitest microprocessor coating thickness gauge, Model 2100 ", manufactured by ELECTRO – PHYSIK West Germany. The measuring range of the instrument is 0-1.25 mm and its accuracy  $\pm 5 \mu\text{m}$  (1 mm = 1000 micron).

The instrument should be calibrated before use as per ISO 2808 as follow:

- 1- Mount the probe to the instrument and turn on the instrument
- 2- Place the probe onto unpainted smooth steel and hold it there, The instrument shall display 0  $\mu\text{m}$ . If the instrument displays other values than 0, the instrument must be adjusted. Do this while you hold the probe onto the steel)
- 3- Take a non- magnetic shim with a defined thickness e.g. 100  $\mu\text{m}$  and place it into the smooth steel surface. Place the probe onto the shim and hold it there. (The instrument shall display 100  $\mu\text{m}$ . If the instrument displays other values than 100, the instrument must be adjusted. Do this while you hold the probe onto the steel)
- 4- You have now adjusted the instrument for measurements in the area of 0-100  $\mu\text{m}$ . But for your own sake check that you actually get 0  $\mu\text{m}$  on smooth steel and 100  $\mu\text{m}$  on the shim. If you still do not have the correct values displayed do step 2 and 3 again until you do.

#### **2-4-5. Film Hardness by Pencil Test:**

The purpose of this test is to assess the ability of the surface coating to withstand scratching. A coated panel is placed on a

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firm horizontal surface. The pencil is held firmly against the film at a  $45^{\circ}$  angle (point away from the operator) and pushed away from the operator in a  $\frac{1}{4}$ -in. (6.5-mm) stroke. The process is started with the hardest pencil 6 H and continued down the scale of hardness until a pencil found that for scratch hardness (the hardest pencil that will not scratch the surface of the film). Make a minimum of two determinations scratch hardness for each pencil [186].

#### **2-4-6. Stripping Test:**

The panel shall be placed on a flat surface with the coated side up. A sharp blade, held at approximately  $60^{\circ}$  to the surface should be pushed so that the blade has a tendency to lift the coating. The coating shall not be removed from the test panel in strips, but shall flake off. The flakes when rolled between the thumb and the forefinger shall produce powder particles [160].

#### **2-4-7. Adhesion: The Cross Hatch Test:**

Adhesion is a very important property of a paint system, and some useful empirical information can be obtained by the cross hatch test. If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for expected service life. These test methods cover procedures for assessing the

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adhesion of coating films to substrates by applying and removing pressure-sensitive tape (25 mm wide semi transparent tape) over cuts in the film. In this test method a lattice with six cuts in each direction is made in the film to the substrate (space the cuts 1 mm), pressure-sensitive tape is applied over the lattice and then removed, and adhesion of the film is assessed from the amount of the coating removed. The adhesion degree is reported in *Table 2.1*. This test method for coatings having a DFT up to and including 50  $\mu\text{m}$ , For coatings having a DFT between 50  $\mu\text{m}$  and 125  $\mu\text{m}$ , the space cuts 2 mm.

- For films thicker than 125  $\mu\text{m}$ , this test method is not considered suitable. An X-cut is made in the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0A to 5A scale [187]. The adhesion performance is reported in *Table 2.2*

#### **2-4-8. Pull- Off Strength of Coating:**

This test method covers a procedure for evaluating the pull – off strength (commonly referred to as adhesion) [188].

Generally there are two field tests for the adhesion of coatings, the first is pull – off adhesion and the second is Hydraulic adhesion test which is carried out as follow:

- 1- Sand rough the dollies and area of the painted surface with a sandpaper

**Table (2.1): Adhesion Degree of the Coated Films**

<b>CLASSIFICATION</b>	<b>PERCENT AREA REMOVED</b>
5B	0 % None
4B	Less than 5 %
3B	5-15 %
2B	15-35 %
1B	35-65%
0B	Greater than 65 %

**Table (2.2): Adhesion Degree of the Coated Films**

<b>CLASSIFICATION</b>	<b>PERCENT AREA REMOVED</b>
5A	No peeling or removal
4A	Trace peeling at their intersection
3A	Jagged removal up to 1.6 mm on either side
2A	Jagged removal up to 3.2 mm on either side
1A	Removal from most of the area of the X under the tape
0A	Removal beyond the area of the X

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- 2- Degrease the dollies and the painted surface using a suitable solvent and dry them before use.
  - 3- Mix the 2 pack solvent less epoxy in the correct ratio before applying it on to the dollies, press the dollies to the surface and turn them  $360^{\circ}$  in order to ensure that most of the glue is squeezed out
  - 4- Before the pull – off test is accomplished cut through the cured adhesive and the paint coating to the substrate , round the circumference of the test cylinder using a cutting device , extreme care is required to prevent micro – cracking in the coating, since such crack may cause failure at diminished strengths.
  - 5- The instrument measures the force in  $\text{Kg} / \text{cm}^2$ , PSI or MPa

#### **2-4-9. Flexibility (Bend) Test:**

The test was performed to determine the adhesion power of the varnish to the substrate by the bending apparatus. 5x15 cm tin panel was coated by the varnish, and dried. The panel was placed into the bending apparatus (0.3 cm diameter) in such away that the surface of the panel was directed out side. The apparatus was then opened and the panel was examined carefully to find out any cracking or dislodging of the film as criterion of loss of consistency of the film. The varnish was

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considered satisfactory if no marks for cracking or dislodging are observed after bending [189].

**2-4-10. Resistance to Mechanical Damage (Impact Resistance):**

The impact resistance of the coating on the steel panels shall be tested by means of dropped steel ball of two pounds (890 gm) from a 1, 00 m height. After impact no cracks shall be visible by a magnification of ten times. When the area of the impact and the area of 5 mm around the impressed area shows flakes after scratching with a sharp needle the coating failed [190].

**2-4-11. Acetone Rub Test:**

Cotton tissue was drained with acetone and then rubbed 100 times forwards and backwards over one spot of cured coating. A complete cross – linked polymer should show no changes on the film surfaces after this treatment [191].

**2-4-12. Water Resistance:**

The coated glass panels (3 x 5 in) were sealed by paraffin wax from their edges and placed in a beaker containing about 7 cm (2.5 in) of distilled water at room temperature and allowed to remain in the water for 24 hours or other suitable period. The

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panel was then removed from the water, wiped carefully and allowed to dry at room temperature. The panel was tested for any change. For longer testing period, the test liquid was changed at least every 72 hours to avoid contamination. To accelerate the test, the same procedure was applied with the exception that water was gently boiled throughout the immersion period, which may be from 15 minutes to several hours. The panels were removed and examined. The films were considered not affected when no film defects were observed after 24 hours immersing or 15 minutes in boiling water [192].

#### **2-4-13. Alkali Resistance:**

The coated glass panels (3 x 5 in) were sealed by paraffin wax from their edges then immersed in an aqueous solution containing 5 grams of anhydrous sodium carbonate per 100 ml. of water, for four hours or any specified time intervals. The panels were removed, washed and the films examined immediately after drying for one hour for any defects [193].

#### **2-4-14. Acid Resistance:**

The panels were prepared in the same manner as in the previous items and immersed to half the length in solution containing 20 grams of sulphuric acid (sp.gr.1.84), per 100 ml of water and allowed to stand for 24 hours at room

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temperature. The panels were then removed and washed with cold water and allowed to dry in vertical position for two hours at room temperature and the immersed portion of the films was examined for any defects [194].

#### **2-4-15. Solvent Resistance:**

The coated panels were immersed in benzene / mineral turpentine solvent mixture (1:3 by volume) for 15 minutes at room temperature. The panels were removed from the test solvent followed by drying in vertical position for one hour. The panels were then tested for any defects [195].

#### **2-4-16. Corrosion Resistance:**

The coated steel panels to be tested were exposed to Salt Spray (Fog) Corrosion Cabinet. Manufactured by Sheen Instruments LTD. England. for a period of 500 hours. This cabinet is used to measure the corrosion resistance of materials and components. This cabinet is designed to expose test pieces to aggressive environments which simulate prolonged exposure in natural environment. It gives better correlation between accelerated laboratory tests and “real-world” exposure [196]. The salt solution is pumped to the atomizer via a variable speed peristaltic. This ensures accurate control and monitoring of the salt fog out rates.

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**2. Salt Solution:** The salt solution prepared by dissolving  $5 \pm 1$  parts by weight of sodium chloride in 95 parts of water.

**3. Position of Specimens During Test :** The position of the specimens in the salt spray chamber during the test shall be supported or suspended between  $15$  and  $30^\circ$  from the vertical and preferable to the principal direction of horizontal flow of fog through the chamber.

**4. Air Supply:** The air supply to the nozzle for atomizing the salt solution shall be free of oil and dirt and maintained between  $69$  and  $172 \text{ kN/m}^2$  ( $10$  and  $25 \text{ psi}$ )

**5. PERIOD OF TEST:** The test method does not prescribe the type of the test specimen or exposure periods to be used for a specific product. The period of test shall be as designated by the specifications covering the material or product being tested, but exposure periods of multiples of  $24 \text{ h}$  are suggested.

6. The films were examined for any defects such as blistering and rusting.

#### **2-4-17. Evaluating Degree of Blistering of Paints:**

This test method employs photographic reference standards to evaluate the degree of blistering that may develop when paint systems are subjected to conditions which will cause blistering. While primarily intended for use on metal and other nonporous surfaces, this test method may be used to evaluate

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blisters on porous surfaces, such as wood, if the size of blisters falls within the scope of these reference standards. A quantitative physical description of blistering would include the following characteristics determined by actual count:

**SIZE:** Reference standards have been selected for four steps as to size on a numerical scale from 10 to 0, in which No.10 represents no blistering. Blistering standard No.8 represents the smallest size blister easily seen by the unaided eye. Blistering standards Nos.6, 4 and 2 representatively larger sizes [197].

#### **2-4-18. Evaluation of Painted or Coated Specimens Subjected To Environments (Scribe Failure):**

This test method covers the treatment of previously painted or coated specimens for accelerated and atmospheric exposure tests and their subsequent evaluation in respect to corrosion and blistering associated with corrosion ,loss of adhesion at scribe mark and other film failure. Painted or coated specimens subjected to a corrosive environment are evaluated by record the average (mean) maximum and minimum creepage from the scribe mark. Record creep values in millimeters, inches,or rating numbers [198] as represented in *Table (2.3 )*

Table (2.3) Rating of Failure at Scribe

Millimeters	Inches (Approximate)	Rating Number
Zero	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0.5 to 1	1/64 to 1/32	8
Over 1 to 2	1/32 to 1/16	7
Over 2 to 3	1/16 to 1/8	6
Over 3 to 5	1/8 to 3/16	5
Over 5 to 7	3/16 to 1/4	4
Over 7 to 10	1/4 to 3/8	3
Over 10 to 13	3/8 to 1/2	2
Over 13 to 16	1/2 to 5/8	1
Over 16 to more	5/8 to more	0

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## **2-4-19. Evaluating Degree of Rusting on Painted Steel Surfaces:**

This test method covers the evaluation of the degree of rusting on painted steel surfaces using visual standards. These visual standards were developed in cooperation with the Steel Structures Painting Council (SSPC) to further standardization of methods. The amount of rusting beneath or through a paint film is a significant factor in determining whether a coating system should be repaired or replaced. Visually compare the surface with photographic reference standards to determine the percentage of the area rusted [199]. The following *Table (2.4)* represents the scale and verbal description.

**Note:** The coated panels were immersed in Acetic acid glacial / Ethyl acetate solvent mixture (1 : 1 by volume) as a paint remover.

**Table (2.4): Scale and Description of Rust Grades**

<b>Rust Grades</b>	<b>Description</b>
10	No rusting or less than 0.01 % of surface rusted
9	Minute rusting ,less than 0.03 % of surface rusted
8	Few isolated rust spots, less than 0.1 % of surface rusted
7	Less than 0.3 % of surface rusted
6	Extensive rust spots but less than 1 % of surface rusted
5	Rusting to the extent of 3 % of surface rusted
4	Rusting to the extent of 10 % of surface rusted
3	Approximately one sixth of the surface rusted
2	Approximately one third of the surface rusted
1	Approximately one half of the surface rusted
0	Approximately 100 % of surface rusted