CHAPTER (3)
EXPERIMENTAL WORK

The purpose of the experimental work is to examine and evaluate experimentally a new approach to rehabilitate exhausted transformer oils instead of its disposal. Figure 3.1 shows a typical schematic diagram for the work strategy. The rehabilitate treatment was based mainly on using unfriendly environmental material which is the cement kiln dust. The rehabilitation process and experimental testing is described in the following.

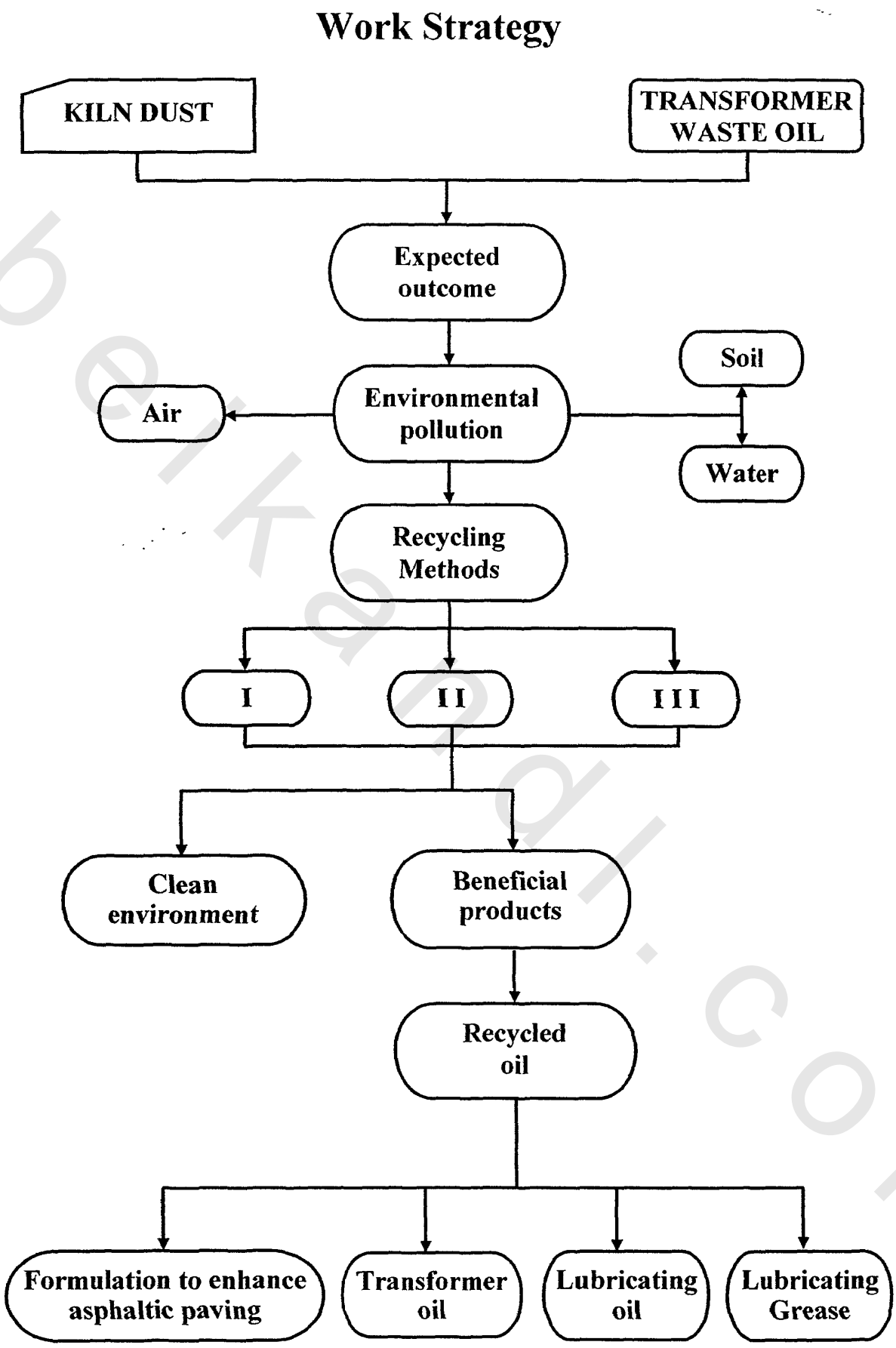
3-1 Raw Materials

3-1-1 Used Oil Samples

Two samples of Egyptian used transformer oils were collected from two electrical step-down transformer stations, namely; Egyptian Petroleum Research Institute (EPRI) station and Quaha station. The service durations of the transformer oil samples were 18 and 30 months for the EPRI and Quaha stations, respectively. The oil samples are given code letters which are UTO1 and UTO2. Figure 3.2 shows a typical schematic diagram for the above mentioned electrical stations. The sub-transmission system is typically at 66 kV while the distribution system is at 11 kV. A huge number of distribution transformers are spread to step down the medium voltage from 11 kV to the normal consumer voltage 380/220 V. The samples for treating were taken from two different types of transformers; one is 66/11 kV and the other 11/0.4 kV.

3-1-2 Cement Kiln Dust

By-pass cement kiln dust was supplied by the Tourah Portland Cement, Helwan. This cementations by-product is very cheap and largely available and its chemical composition gave attraction to use for reclamation of the used transformer oils.



Scheme 3.1 A typical schematic diagram for the work strategy

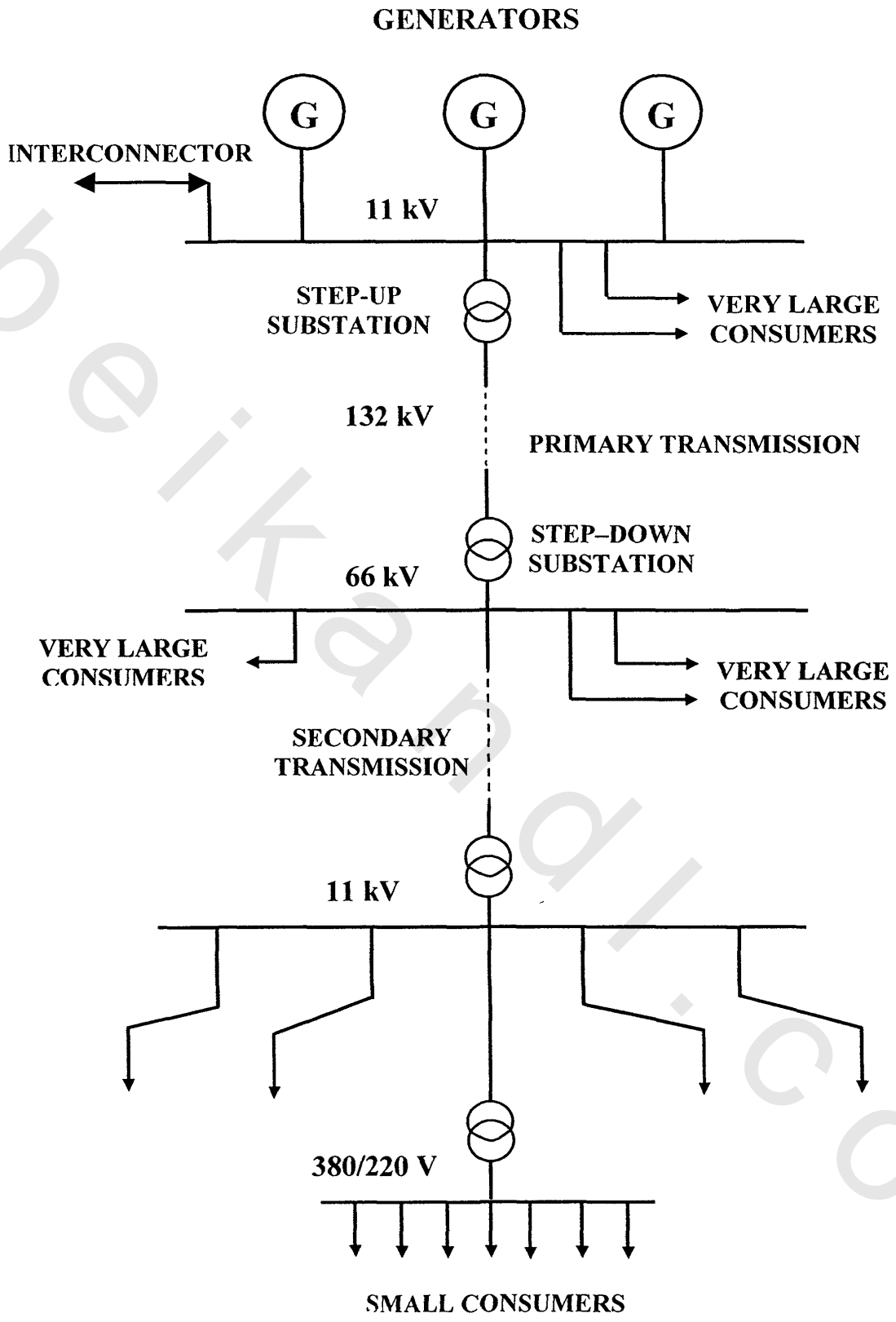


Figure 3.2 A typical simplified power system.

3-2 Determination of Physico-Chemical Characteristics

The used transformer oils (UTO1 and UTO2) were filtrated through calcium chloride then dehydrated under reduced pressure at 20 mm Hg. The physicochemical characteristics of the used transformer oils before and after treatment were carried out according to ASTM [95] and/or IP [96] standard test methods.

3-2-1 Preliminary Distillation

Preliminary distillation of the oils was carried out according to the IP24 preliminary distillation standard method [96]. In this method 100 ml of the oil sample were distilled under reduced pressure.

3-2-2 Density

The density of the used transformer oils was determined according to the IP190 capillary-stoppered pyknometer method [97]. In this method, the weights of equal volumes of the sample and of the water are compared. Equal volumes are ensured by placing the filled pyknometer in a bath at the test temperature until equilibrium is reached.

The density of the oils was determined at 25°C. Specific gravity at 60°F of the oils were calculated using the ASTM-IP Petroleum Measurement Tables, according to the ASTM D 1250-IP 200-API 2540 methods.

3-2-3 Kinematics Viscosity

The kinematics viscosity of the oils was determined according to the ASTM D 445-IP 71 glass capillary viscometer method.

In this method, the time is measured in seconds for a fixed volume of oil to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematics viscosity is the product of the measured flow time and the calibration constant of the viscometer.

3-2-4 Pour Point

The pour point of the oils was determined according to the ASTM D 97-IP15 method.

In this method, after preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C (5°F) for flow characteristics. The pour point is recorded as the lowest temperature at which movement of the sample is observed.

3-2-5 Carbon Residue Content

The carbon residue content of the oils was determined according to the ASTMD 524-IP 14 Ramsbottom carbon residue method.

In this method, the sample after being weighed into a special glass bulb having a capillary opening is placed in a metal furnace maintained at approximately 550°C. The sample is thus quickly heated to the point at which all volatile matter is evaporated out of the bulb with or without decomposition, while the heavier residue, remaining in the bulb, undergoes cracking and coking reactions. In the latter portion of the heating period, the coke or carbon residue is subjected to further slow decomposition or slight oxidation due to the possibility of breathing air into the bulb. After a specified heating period, the bulb is removed from the bath, cooled in desiccators, and again weighed. The residue remaining is calculated as a percentage of the original sample, and reported as Ramsbottom carbon residue.

3-2-6 Ash Content

The ash content of the oils was determined according to the ASTM D 482-IP 4 method.

In this method, the sample contained in a suitable vessel, is ignited and allowed to burn until only ash and carbon remain. The carbonaceous residue is reduced to an ash by heating in a muffle furnace at 775°C, cooled, and weighed.

3-2-7 Water Content

The water content of the oils was determined according to the ASTM D 95-IP 74 distillation method.

In this method, the sample is heated under reflux conditions with a water-immiscible solvent which co-distils with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settles in the graduated section of the trap, and the solvent returns to the distillation flask.

3-2-8 Hydrocarbon Components Analysis

The used transformer oils were separated into their hydrocarbon components by using silica gell column chromatography. Thus, the tested oil sample (10 g), dissolved in the least amount of petroleum ether 40-60°C was transferred to the column and packed with activated silica gell (150 g). The elution was carried out with petroleum ether 40-60°C (300 ml) to separate the saturated hydrocarbon fraction and 200 ml of mixture of 95% petroleum ether plus 5% benzene to remove the monocyclic aromatic hydrocarbons followed by elution with 200 ml of benzene to separate the dicyclic aromatic fraction and finally eluted with 300 ml of ethyl alcohol to desorbs the polycyclic aromatic - polar compound fraction.

In this method 25 ml of the eluted fraction were collected in the receiving flask and the solvent were distilled off on the water bath and then dried in drying oven till constant weight. The oil was separated into its hydrocarbon components according to refractive index.

Hydrocarbon components	Refractive index, at 20°C
Saturates	up to 1.48
Monocyclic aromatic	1.48 –1.53
Dicyclic aromatic	1.53 –1.59
Polycyclic aromatic	Above 1.59
Resins	Dark

3-2-9 Structural Group Analysis

The structural group analysis of the oils was determined by using the n-dM method [104].

This method gives the percentages of carbon in aromatic, naphthenic and paraffinic structures ($\%C_A$, $\%C_N$ and $\%C_P$) and total aromatic and naphthenic rings per molecule (R_T, R_A, and R_N).

3-3 Analysis Techniques

3-3-1 Gas Chromatographic Analysis

The used transformer oils (UTO1 and UTO2) were analyzed using Agilent 6890 plus HP gas chromatograph, equipped with flame ionization detector (FID), using fused silica capillary column HP-1 of 30 meter length and 0.35 mm internal diameter. The elution of the transformer oils was achieved with temperature programming from 80 to 300°C at a temperature rate of $5^{\circ}Cmin^{-1}$. Nitrogen (oxygen-free) was used as a carrier gas, flow rates were measured uncertain marker was used to correct the dead volume from the end of the column with a soap bubble flow rate. Methane as a column in the case of FID, the injector and detector temperatures was mentioned at 250 °C and 300°C, respectively. The integration of the area under the resolved chromatographic profile is obtained using the computer software chemstation.

3-3-2 High Performance Liquid Chromatography (HPLC) Analysis

The polyaromatic hydrocarbons of the used transformer oils before and after treatment were analyzed using HPLC model Waters 600E equipped with auto sampler Waters 717 plus and dual wavelength absorbance detector Waters 2487 set at 254 nm. The conditions of operation were as follows:

Column: Supelcosil LC-PAH, 15 cm × 4.6 mm ID, 5 μm particles size.

Mobile phase: Acetonitrile: water HPLC grades, gradient from 60:40 to 100% acetonitrile.

Flow rate: Gradient program, 0-2 minute, 0.2 ml/min and 2-45 minute, 1.0 ml/min.

Detector: Set at 254 nm.

3-3-3 Infrared Spectrophotometer

Infrared spectra of the used transformer oils were recorded on a FT-IR spectrophotometer, Model 960M000g, ATI Mattson Infinity Series, USA. The samples were analyzed in the pure phase using KBr pellets. The spectra of the all studied samples were measured in the range of 4000-400 cm^{-1} by summing 32 scans at 4 cm^{-1} resolution.

3-4 Procedures

3-4-1 The first Technique (Acid/kiln dust treatment)

Definite weight (250 g) of dehydrated used transformer oil was treated with different weight percent (3 wt%, 5 wt% and 7 wt%) from sulphuric acid (98%). These treatments were carried out at different mixing times (30, 60, 90 and 120 minutes) with stirring in a glass extractor of conical shape having a tap at the end of its bottom. After settling for one hour the sludge was removed and the raffinte was collected. The acid treated raffinte (oil layer) was treated with by-pass kiln dust at different weight percent (1, 3, 5, 7 and 9 wt%) at room temperature. The predetermined quantities of the acid treated oil and kiln dust were introduced into the extractor and stirred thoroughly for one hour to ensure perfect mixing. After settling, the treated oil was freed from kiln dust using centrifuge (Biofuge with open lid and rotor put into place) at 3000 rpm for ten minutes and then washed with distilled water several times. The recovered oils obtained were dried over anhydrous calcium chloride.

The physicochemical characteristics, hydrocarbon type analysis and AC-breakdown voltage of these oils were determined. The recovered transformer oils of EPRI and Quaha electrical stations are given codes RTO1 and RTO2,

respectively. The sludge which results from acid treatment was processed. In addition, the product produced from acid sludge treatment and kiln dust would be the subject for many forthcoming investigations.

3-4-2 The Second Technique For Treatment Acid Sludge

The kiln dust, sodium chloride, sodium hydroxide, sodium silicate and sodium carbonate are used as commercial grade. Different formulations (S1 to S6) of the used salts were prepared as indicated in Table 3.1. The acid sludge which produced from the treatment of used transformer oil by sulphuric acid was treated by different formulations S1 to S6. It was carried out by adding the formulation gradually to a sample of 100 g acid sludge diluted with 100 ml of water while stirring at a temperature range of 90-100°C. The aqueous phase was separated by decantation and its pH was measured.

The processes were repeated several times until the pH of aqueous phase was nearly neutral. The hydrocarbon phase which separated was called multi-component resin (MCR). The identification and hydrocarbon type analysis of the MCR was carried out according to IP 134/190 [96].

Table 3.1 formulation of the component used in the treatment of acid sludge

Sample of formulation / Basic constant gm/L	S1	S2	S3	S4	S5	S6
By-pass kiln dust	35.0	30.0	25.0	20.0	15.0	10.0
Sodium hydroxide	0.0	1.0	2.0	3.0	4.0	5.0
Sodium chloride	3.0	5.0	7.0	9.0	11.0	13.0
Sodium silicate	2.0	2.0	2.0	2.0	2.0	2.0
Sodium carbonate	5.0	5.0	5.0	5.0	5.0	5.0

3-5 Dielectric Test Equipment and Methods

The tests on the samples were carried out at two different high voltage (HV) laboratories which are: the faculty of engineering, Ain Shams

University and Al Sabteia HV labs, Ministry of Electricity.

There were approved using Hiptronic device as the proceedings follow applied to the ASTMD 877 standard presented hereinafter the testing apparatus procedure and methodology where the testes results are presented in the next chapter.

3-5-1 The Test Apparatus

A Hiptronic test apparatus, Photos 3.1 and 3.2, was used to determine the break down voltage of the oil. The apparatus is capable of increasing the voltage in steps of 500 V, 2000 V and 3000 V. Oil dielectric tests are usually used for making dielectric tests on oil. Units with a variable high voltage of 60 kV between the Bakelite electrodes and test cups are considered satisfactory. Figure 3.3 shows a schematic diagram of the test cell with spherical surfaced electrode.

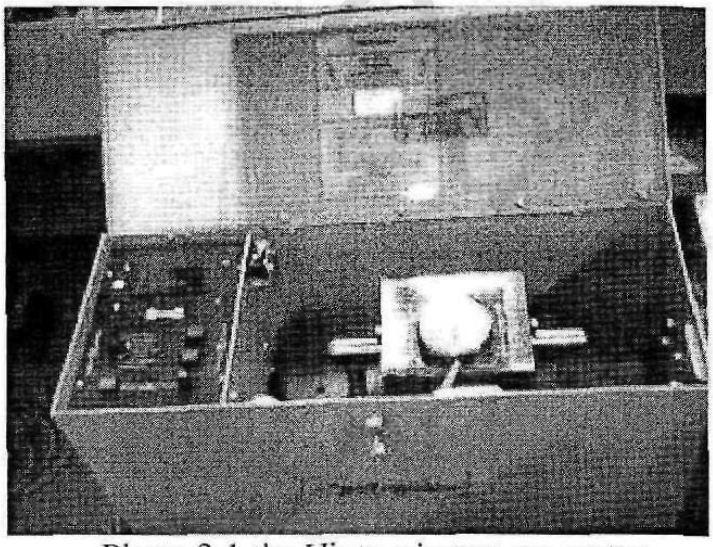


Photo 3.1 the Hiptronic test apparatus

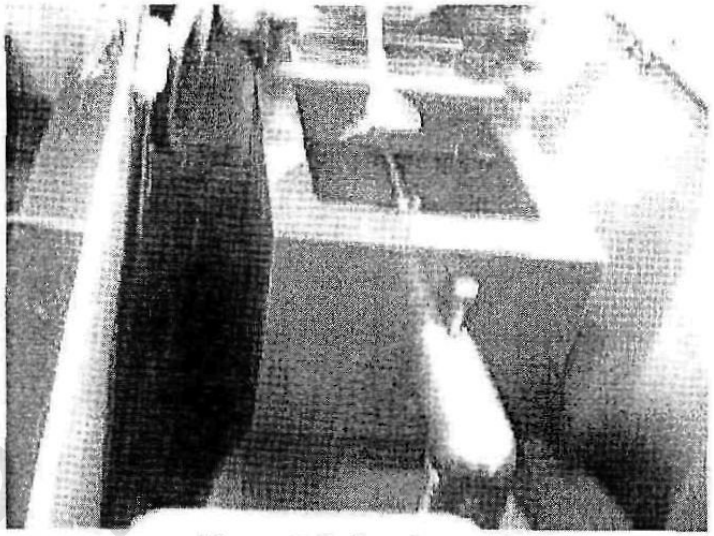


Photo 3.2 the electrodes

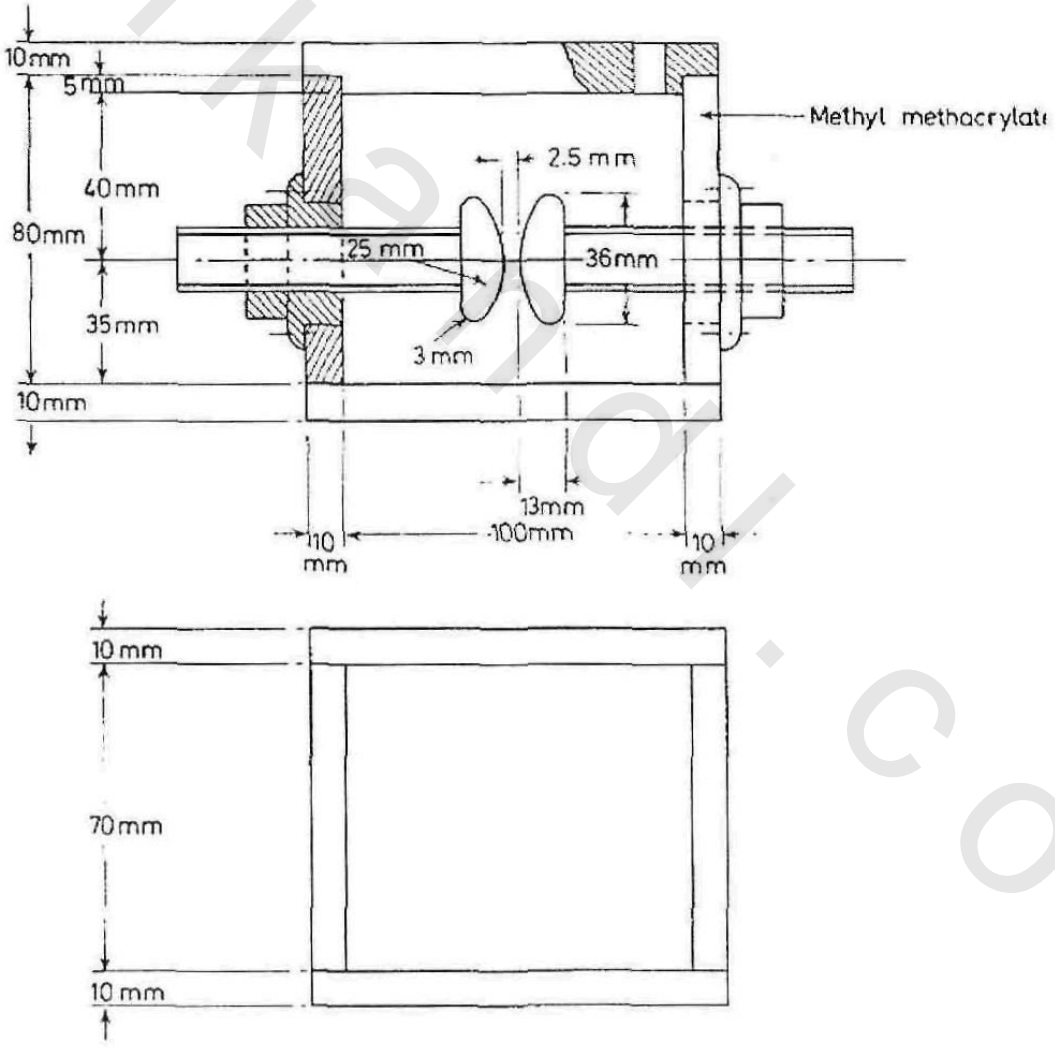


Figure 3.3 Test cell with spherical surfaced electrode.

The following instructions cover the adjustment of the electrodes and test cup.

- Clean the electrodes and the test cups.
- Check the spacing of the electrodes with a standard round gauge having a diameter of 2.54 mm (0.1 in) or with flat steel "go" and "no go" gauges having thicknesses of 2.53 and 2.55 mm (0.0995 and 0.1005 in), respectively.
- Lock the electrodes in position.
- Avoid touching the electrodes or the cleaned gauges with the fingers or with portions of the tissue paper or chamois that have been in contact with the hands.
- Avoid any possible contamination.
- Avoid touching the electrodes or the inside the cups after cleaning.
- After a thorough cleaning, the test cups must be filled with a sample of the cleaning fluid.
- Apply voltage and increase it gradually at a rate approximately between 0.5 and 3 kV/s (rms values) until breakdown occurs.
- If the breakdown is not less than the established value of the oil being tested, the test cups have to be considered in suitable condition for testing.
- If a lower value is obtained, the cups shall again be thoroughly cleaned and the test repeated. A cleaning fluid whose breakdown is not less than the established value of the oil being tested must be used.

3-5-2 Procedure

3-5-2-1 Preparation of Sample

The dielectric strength of oil dielectrics may be markedly altered by the migration of impurities through the oil. In order that representative test specimens may be obtained, the sample container gently tilted or inverted and the oil swirled several times before each filling of the test cup, in such a way

that any impurities present will be thoroughly mixed with the dielectric. Too rapid agitation is undesirable, since it introduces an excessive amount of air into the oil. Immediately after agitating, the test cup filled with oil to a height of not less than 20 mm (0.787 in) above the top of the electrodes. In order to permit the escape of entrapped air, the container rocked gently a few times and the oil allowed standing in the cup for 3 minutes before voltage is applied.

3-5-2-2 Test Temperature

The temperature of the sample when tested was the same as that of the room, but not less than 20°C (68°F). Oil testing at a temperature lower than that of the room is likely to give variable results, which may be misleading.

3-5-2-3 Application of Voltage

Voltage was applied and increased from zero at a uniform rate of 500 V/s until breakdown occurs as indicated by a continuous discharge across the gap. Occasional momentary discharges which do not result in a permanent arc may occur; they disregarded.

3-5-3 Number of Tests

It is desirable to determine the dielectric breakdown voltage of new oil for referee purposes, one breakdown made on each of five successive fillings of the test cup. The breakdown voltage thus obtained subjected to the criterion for statistical consistency. If the five values meet this criterion, their average reported as the dielectric breakdown voltage of the sample. If they do not meet this criterion, one breakdown on each of 5 additional cup fillings made, and the average of the 10 breakdowns reported as the dielectric breakdown voltage of the sample. No breakdown discarded.

3-6 Test Method for Power Factor of Electrical Insulating Oils

This method covers new electrical insulating oils as well as oils in service or subsequent service in transformers.

This method provides a procedure for making referee tests at a commercial frequency of approximately 60 Hz.

3-6-1 ASTM Standards

The following Applicable Documents list the *ASTM Standards* used in tan(δ) and power factor tests.

- D 150 Tests for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials.
- D923 Sampling Electrical Insulating Oils.
- E 145 Specifications for Gravity Convection and Forced-Ventilation Ovens.

3-6-2 General Considerations

Definitions, theory and measuring equipment pertaining to this method shall be in accordance with Method D 150.

3-6-3 Sampling

Oils for use in this test sampled in accordance with Method D 923. Samples to be subjected to the test preferably obtained through a closed system. The quantity of the sample taken for this test was sufficient for at least three separate power factor determinations.

3-6-4 Test Cells

3-6-4-1 Design of the Cell

A cell for the purpose of measuring power factor and dielectric constant of electrical insulating oils, figure 3.4, meet the following general requirements:

- To facilitate easy and thorough cleaning of its component pans permit the

use of the cell in a suitable temperature bath and provide means for measuring the temperature of the oil under lest.

- The materials used in constructing the cell are nonporous and capable of satisfactorily withstanding the temperature to which the cell will be subjected under lest. The alignment of the electrodes not influenced by this temperature or by the operation of filling the cell with the test oil.
- The electrodes or their surfaces made of a metal capable of resisting attack by mild acids such as are found in oils of petroleum origin, Plated surfaces that may be satisfactory for testing oils having low acidity and for short periods of time are gold, platinum, nickel, chromium over nickel, or rhodium.
- When most accurate power factor or dielectric constant determinations are to be made, a guard electrode provided which adequately shields the measuring electrode. A shielded wire or coaxial cable used to connect the guard and measuring electrodes either directly or by a plug to the bridge.
- The solid insulation used to support the guard electrode relative to the measuring electrode is not extended into the portion of the sample being tested.

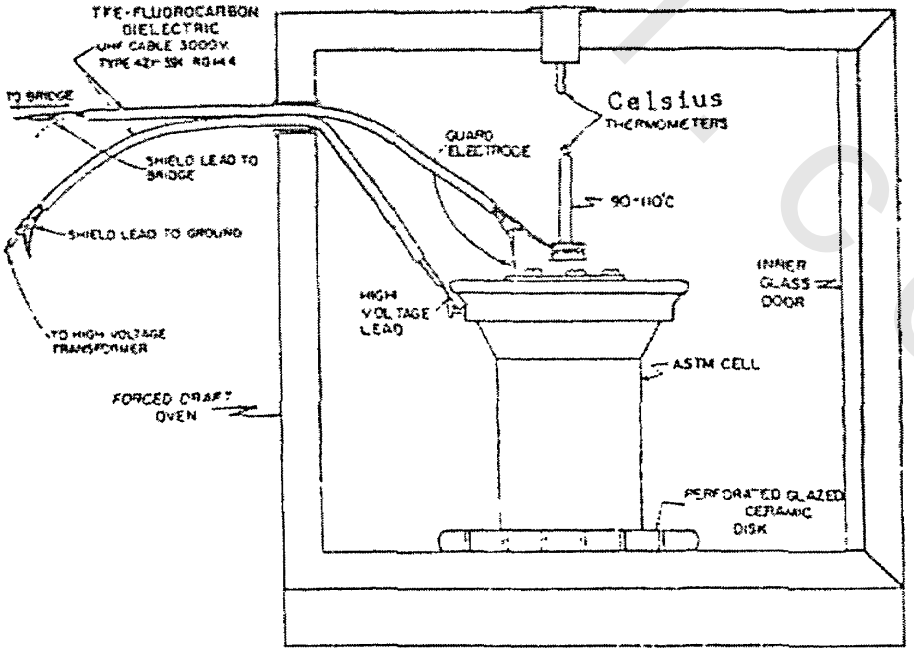


Figure 3.4 Test Set-up for Power Factor Measurements at Elevated Temperature Using Three-Electrode Test Cell.

- The insulating materials used in constructing the cell not absorb or adversely affected by the test oils or cleaning solvents. The power factor of these insulating materials must necessarily be low, particularly that between the guard and measuring electrodes; otherwise difficulty may be experienced in balancing the guard circuits at elevated temperatures. Insulating materials that have proven satisfactory are borosilicate glass, quartz, steatite, and TFE-fluorocarbon. Thermoplastic materials such as hard rubber and polystyrene, although having good electrical properties, arc net suitable as they soften below 130 °C. Insulating materials of the molded mica-dust type have been found to absorb solvents and therefore arc not considered as satisfactory in the measurement of low power factor oils.
- In designing the cell, the distance across the surface of the test specimen and across the solid insulating material between the guard and the measuring electrode is long enough to withstand the applied test potential. Leakage across these paths has been found to produce an unsteady bridge balance on some bridges.
- The surface area of the measuring electrode and the gap spacing between the measuring electrode and high-voltage electrode is such that the ratio of surface area to thickness of the test sample is large enough to provide sufficient current for adequate operation of the measuring equipment. No minimum capacitance is therefore suggested, It is required, however, that the precision of the measurement meets the intended accuracy of this method
- The design of test cells that conform to the general requirements is considered suitable for use in making these tests.
- There are many types of guarded electrode test cells that conform to these requirements and that have been found suitable for measuring the power factor and dielectric constant of insulating oils, but a three-terminal cell

intended for making referee tests and research investigations arc shown in Figure 3.4.

3-6-5 Test Chamber

When the tests are carried out above room temperature below 300°C, a forced-draft, thermostatically controlled oven must be used as the test chamber and conform to Specification E 145.

For tests at room temperature the un-energized oven can be conveniently used as the test chamber.

The test chamber has an opening in the wall through which two lengths of TFE-fluorocarbon-insulated shielded cable pass to make electrical connection from the measuring equipment and high-voltage transformer to the test cell. A perforated ceramic plate or disk is used to insulate the test cell from the metal flooring of the oven when the flooring is not insulated from the oven. A safety Interlock was provided on the door of the oven so that the electrical circuit supplying voltage to the test cell will be broken when the oven door is opened.

A cross-sectional view of the test chamber with a three-electrode test cell in place and with test cables connected are shown in figure 3.5.

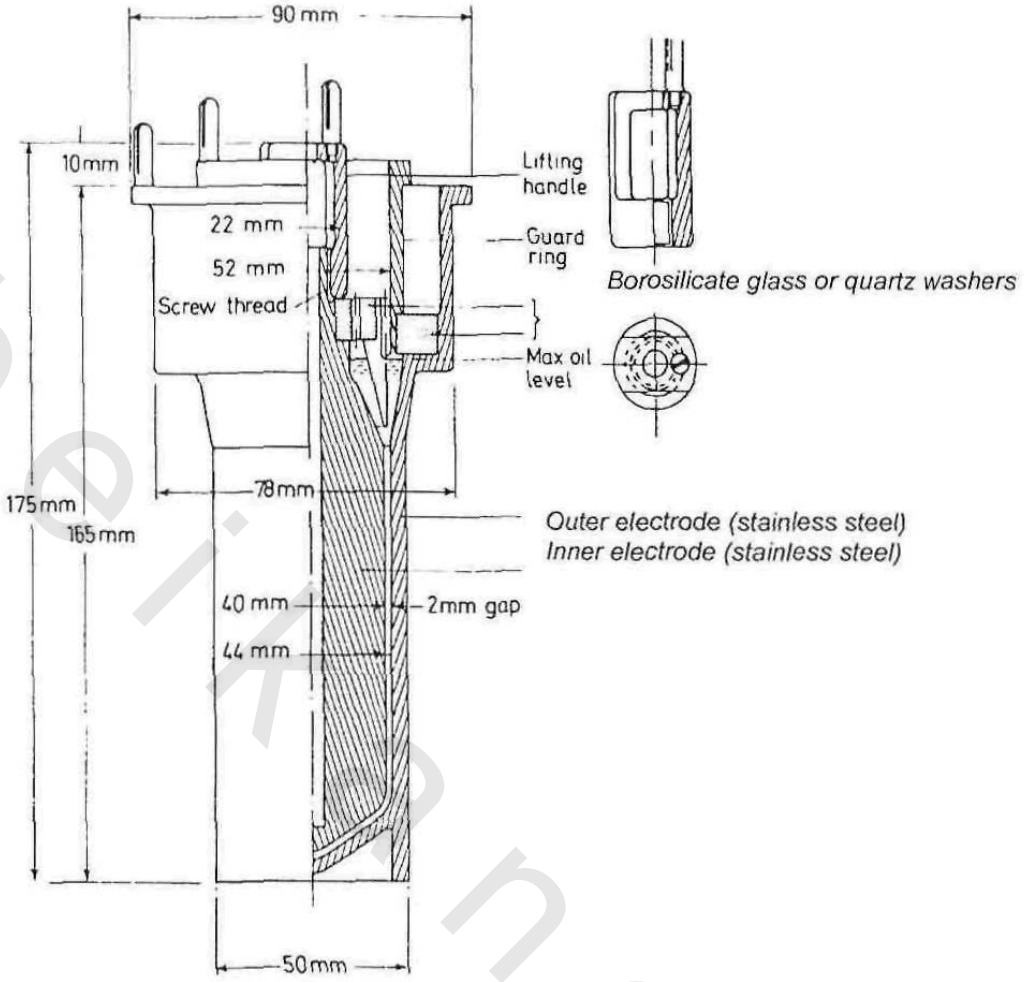


Figure 3.5 Loss tangent test cell recommended by CIGRE
(quantity of oil required to fill cell 45 ml, approx.)

3-6-6 Test Temperature

Although the temperature at which a referee test is made has to be mutually agreed upon between the purchaser and the seller. In this investigation, power factor measurements are made at many different temperatures. For acceptance tests, it is generally made, at a temperature of 100°C.

3-6-7 Test Voltage

The average stress to which the specimen is subjected was less than 200 V/mm (rms). Tests at higher stresses are desirable but not reach such values that electrical discharges across the cell insulating surfaces occur or that internal ionization of the specimen may be expected. Stress ranges in normal

usage for referee tests are: 200 - 1200 V/mm (rms).

3-6-8 Test Cell Cleaning

The cleanliness of the test cell is of paramount importance when making power factor measurements because of the inherent susceptibility of most insulating oils to contaminating influences of the minutest nature. For this reason, the cell was cleaned and dried immediately prior to making the test. The cell is disassembled completely and all the component parts washed thoroughly with a technical grade of a suitable solvent *(such* as tri-chlorotrifluoroethane, petroleum ether or pentane). The component parts were washed with a mild abrasive soap or detergent, then all parts were thoroughly rinse with hot tap water, then with cold tap water, followed by several rinsing with distilled water. Extreme care is taken during the washing and rinsing of the test cell shown in figure 4.4 to prevent any moisture from entering the thermometer well in the inner electrode. As a precaution against this eventuality, a suitable stopper is used to plug this opening prior to starting the cleaning operation.

After the surface of the electrodes and guard has been washed care is taken not to touch these surfaces during the rinsing or any subsequent operation.

The component pans of the test cell are placed in an oven maintained at 110°C for a period of not less than 60 min. a care is taken not to dry the test cells at the elevated temperature for more than 90 min as oxidation will take place causing erroneous results because they are made of Monel. Further, care is taken that the surfaces on which the component pans of the cell are placed in the oven are clean.

At the expiration of the drying period, the cell is assembled in the oven, using clean cotton gloves as protection for the hands.

The assembled test cell was quickly transferred to the test chamber maintained at a temperature of 5°C above the desired test temperature and

allowed the cell to attain temperature equilibrium.

3-6-9 Preparation of Specimen and Test Cell Filling

A three-terminal cell is used for these tests. When insulating oils are heated to elevated temperatures, some of their characteristics undergo a change with time and the change, even though of the minutest nature may be reflected in the power factor results. It is therefore desirable that the elapsed time necessary for the test specimen to attain temperature equilibrium with the test cell be held to a minimum. For optimum procedure it is essential this time is not to exceed 20 minutes.

In order that representative test specimens may be obtained, the sample container was gently tilted or inverted and the fluid swirled several times. After mixing the sample, a quantity of fluid sufficient for four fillings of the test cell was immediately poured into a chemically clean dry beaker and heated on a hot plate to a temperature 2°C below the desired test temperature. The fluid frequently is stirred during the heating period.

The cell was removed from the test chamber, lift out the inner electrode, but avoiding not rest it on any surface, and the cell was filled with a portion of the heated sample. The beaker was replaced with the remainder of the heated sample on the hot plate. The inner electrode was inserted and the two electrodes rinse twice by raising and lowering the inner electrode. The inner electrode was removed and held suspended in air, then decant the rinsing fluid and the cell was immediately filled by the remainder of the heated sample. The inner electrode was replaced.

A mercury thermometer, graduated in 0.25°C increments was inserted in the thermometer well provided in the inner electrode. The filled cell was immediately returned to the test chamber (adjusted to a temperature of 5°C above the desired test temperature) and the necessary electrical connections to the cell were made.

3-6-10 Procedure of Power Factor Test

The loss characteristic is commonly measured in terms of dissipation factor (tangent of the loss angle) or of power factor (sine of the loss angle). For values up to 0.05, dissipation factor and power factor values are equal to each other within about one part in one thousand. Since in general good insulating oils have values below 0.0005, the two terms may be considered interchangeable.

The exact relationship between dissipation factor *(D)* and power factor (PF) is given by the following equations:

$$PF = \frac{D}{\sqrt{1+D^2}} \quad \therefore D = \frac{PF}{\sqrt{1-PF^2}}$$

The power factor measurements were made while the temperature of the inner electrode was within ±0.5°C of the desired test temperature.

On completing the initial measurement, the inner electrode was removed and the oils poured off, and the cell refilled. The same procedure was followed and the same precautions exercised in order to make another filling, before taking another measurement.

A precaution was taken such that if the difference in the power factor measurements of the two specimens is equal to or less than 0.0001, plus 10% of the higher of the two values, no further tests are required, and the power factor of the sample was taken as the average of the two measurements. If the difference in the power factor measurements of the two specimens is greater than 0.0001, plus 10% of the higher of the two values, refill the cell and take a third measurement. If the difference between the third measurement and either the first or second is not within 0.0001, plus 10% of the higher of the two values used in this computation, discard these results, re-clean the cell, obtain another sample, and repeat the procedure until two measurements from a sample arc obtained that do meet the prescribed limits.