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JOS, NIGERIA

Technical Report: First Visit of Mission

Prepared for the Government of Nigeria
by the United Nations Industrial Development Organisation
acting as executing agency for the
United Nations Development Programme

Based on the work of P. Knowles
Consultant in Instrumental Analysis

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SUMMARY

This report describes a visit of a Consultant in Instrumental Methods of Analysis to the National Metallurgical Centre in Jos, Nigeria, during October and November 1993. The visit was the first of two to be undertaken to complete the mission. The mission concerns the training of local staff in the use of various instruments such as atomic absorption spectroscopy, flame photometry, X-ray diffraction and fluorescence, ultra-violet/visible spectrometry, etc.

At the start of the mission the Consultant reviewed the facilities, equipment and staff and agreed a training programme for the mission with the counterpart officers. Unfortunately much of the equipment was not working properly or awaited installation and the Consultant directed his attention first to this area. At the end of the visit most of the equipment was working satisfactorily and basic training had been given in many of the techniques. The instruction was to be continued during the second visit.

Arising from the visit the Consultant made a number of recommendations concerning building facilities, equipment installation and the purchase of instrument spares and consumeables. It was hoped that many of the deficiencies could be corrected in the break between the two visits and the Consultant agreed to assist the Centre in the sourcing and pricing of essential items where these were not available locally. In addition it was further recommended that overall management of the laboratories needed to be upgraded and the Consultant proposed the preparation of a Laboratory Control Manual. This work would require an extension to the mission.

Finally it was concluded and agreed that the second visit should take place starting about mid-January 1994.

CONTENTS

		PAGE NO.
	SUMMARY	1
	CONTENTS	2
	INTRODUCTION	4
1	SAMPLE PREPARATION	7
	1. Introduction 2. Description of the present facilities 3. Discussion of the facilities 4. Recommendations	
II	INSTRUMENTAL LABORATORY FACILITIES	10
	1. Introduction 2. Water Services 3. Electrical Services 4. Laboratory fitments 5. Recommendations	
III	ATOMIC ABSORPTION SPECTROSCOPY	14
	1. Training Programme 2. Instruments at NMDC 3. Consumeable Supplies 4. Recommendations	
IV	ATOMIC EMISSION SPECTROSCOPY	20
	1. The Training Programme 2. The NMDC Flame Photometer 3. Consumeable Items 4. Recommendations	
V	ULTRAVIOLET/VISIBLE SPECTROMETRY	22
	1. The Training Programme 2. UV/Vis Instruments at NMDC 3. Consumable Items 4. Recommendations	
VI	CARBON AND SULPHUR ANALYSIS	25
VII	X-RAY DIFFRACTION ANALYSIS	26
	1. The Training Programme 2. The NMDC Instrument 3. Recommendations	

VIII X-RAY FLUORESCENCE ANALYSIS

1. Introduction
2. Method
3. Recommendations

IX COAL ANALYSIS 31

1. Introduction
2. Carbon, Hydrogen and Nitrogen Determinations
3. Forms of Sulphur
4. Coal Ash Analysis
5. Recommendations

X EQUIPMENT SERVICING 34

1. Introduction
2. Basic services
3. Equipment Installation
4. Equipment Maintenance
5. Recommendations

SUMMARY OF RECOMMENDATIONS 40

ANNEXES

1. Job Description 42
2. Training Programme 44
3. Memo Re. Laboratory Control Manual 47
4. Lists of Required Equipment 49
5. Senior Counterpart Staff and Trainees 53

INTRODUCTION

The mission of Consultant Chemist Mr. Patrick Knowles, (the Consultant) to the National Metallurgical Centre, Jos, Nigeria, (NMDC) involves the training of local operaters in instrumental methods of analytical chemistry (Job Description - Annex 1). The mission was split into two parts of six weeks each; the first during October and November 1993 is the object of this report. The second visit will take place early 1994 at dates to be mutually agreed by all parties.

At the beginning of the mission the consultant received the status of equipment and facilities and, in conjunction with the Head of the Analytical Unit, prepared a training programme which covered the first visit in detail and the second visit in outline. In accordance with the job description and the review the programme covered the following topics;

a. Sample Preparation
b. Atomic Absorption Spectroscopy (AAS)
c. Atomic Emission Spectroscopy - Flame Photometry (AES-FP)
d. Ultra - Violent/Visible Spectrometry (UV/Vis)
e. Leco Carbon-Sulphur Analyser
f. X-Ray Diffraction (XRD)
g. X-Ray Fluorescence (XRF)
h. Coal Analysis
i. Equipment Servicing.

The agreed programme is reproduced as Annex II and it generally includes lectures on the theoretical aspects of each topic, instrumental designs with particular reference to the equipment held by NMDC, standard preparation, methods for sample analysis, calculations and equipment maintenance.

Some of the equipment was quite old whilst others such as XRD and XRF had been purchased 5 years previously, having been kept in store in the intervening period prior to being installed recently in rehabillitated buildings. In many ways the age of equipment and the delays have been unfortunate, because in some cases, essential items such as operating manuals, spare parts, etc, seem to have been lost or were not supplied originally. After this time it is not possible to dispute missing items with suppliers. A second important point is that the original order specifications were incomplete resulting in essential items necessary to using the instrument for analyses not being considered and ordered along with the equipment.

Other points affecting the first visit of the mission were;

a. Most of the equipment was inoperative and required service maintenance before it could be used, whilst the XRF remained to be installed.

b. Some of the laboratory facilities were incomplete.

c. Some essential items, such as glassware, chemicals, standard samples, etc, necesary to operating a laboratory were not available.

d. Considering the training aspect, only one of the candidates had received previous training in the techniques, whilst the others had none. The training programme was tailored to cover the two situations.

Whilst the mission remains incomplete until the second visit, interim recommendations are given under each chapter heading. They generally concern laboratory facilities, equipment servicing and spares, essential capital items, essential consumeable items and further training requirements.

A further topic, which does not form part of the job description is the lack of a laboratory control manual, which should describe the facilities and capabilities of the laboratory and its staff and how it is managed and operated. Following the completion of the mission the consultant would be in a position to prepare such a document and then visit the laboratory to supervise its implementation. The consultant prepared an appropriate proposal for the Director of NMDC (Annex III).

The whole of the first visit of the mission overlapped with the parallel mission of Mr. Bin Fu which concentrated on Wet Chemical methods.

I. SAMPLE PREPARATION

1. Introduction

Sample preparation concerns the crushing, grinding and dividing of samples in preparation for analysis. It is important to consider the following factors of any facility;

a. Sample preparation and storage,
b. Drying of samples,
c. Coarse crushing,
d. Fine crushing,
e. Grinding,
f. Sample division,
g. Sample tracking and stores,
h. Dust control and avoidance of contamination.

Proper sample preparation is extremely important. The value of the analytical information depends on good techniques for handling and dividing samples and the avoidance of contamination.

2. Description of the Present Facilities

The sample preparation for the chemical analysis section is handled by the mineral processing section. The section has a jaw crusher for coarse crushing, roll crusher for fine crushing and disc mills for grinding. Sample division is carried out by cone and quartering

or riffling. The laboratory has no facilities for dust control and relies on strict cleaning procedures but, however, the whole area is covered in a thick layer of dust. Sample drying facilities are shared with mineral processing laboratory products. There are no separate areas for chemical analysis sample handling or temporary stores whilst the samples are in the preparation area.

In addition to the facilities described above a separate building has been fitted with new jaw and roll crushers, as yet unused. Both machines lack drive motors, but that for the roll crusher is kept in the stores. A new motor is required for the jaw crusher. There is ample space in this building to establish a separate preparation facility for chemical analysis samples.

3. Discussion of the Facilities

A conflict exists between the preparation needs of a mineral processing laboratory and those of chemical analysis. Mineral processing usually needs to prepare and handle bulk samples of ore and minerals. Separation processes can give rise to intermediate and concentrate products containing high levels of the metals under test. The sample size is usually quite large in comparison to those for chemical analysis. At any one time the laboratory will be dealing with single or a very small number of ore types. Chemical analysis, on the other hand, usually requires the preparation of a larger number of small individual samples. Each sample is discrete and must not be contaminated with earlier samples or extraneous material. It is difficult to see how the present facilities can

meet this requirement especially at the drying and grinding stages.

4. Recommendations.

Ideally, completely separate facilities should be provided for the preparation of samples for chemical analysis. The existence of the separate building with the two crushers already installed provides a good solution. The following programme is proposed:

a. Clear out all extraneous material from the new building and renovate to include glass to windows, closing of holes, necessary electric and water supplies, locks to doors, benching and sample storage shelving, etc.

b. Install dust extractor and ducting, ring mill grinder, drying oven, dust enclosures and vacuum cleaner.

c. Purchase necessary consumeable items.

d. Train a technician in sample preparation techniques.

During the break in the mission the Consultant will obtain quotations for all the necessary equipment and consumeable items. At the next visit he will advise on the fittings required to be fixed locally and assist in identifying a suitable technician for training. This laboratory could be implemented during the extension to the mission if this is agreed.

II INSTRUMENTAL LABORATORY FACILITIES.

1. Introduction.

The instrument laboratory facilities are housed in two refurbished individual office blocks, one (laboratory 1) for XRD and XRF and the other (laboratory 2) for the other instrumental techniques. The space available is adequate for the purpose and there is unassigned space remaining in both blocks for future expansion. The second block contains many files or other material left over from the previous occupants.

2. Water Services

The instrument laboratories, in common with the most of the site, lacked a reliable water supply because of frequent failures of the mains supply. A good water supply is essential to the efficient operation of any laboratory. It is understood that it is planned to drill two boreholes connected to large storage tanks in association with the development of the Mineral Beneficiation Pilot Plant. Such a system designed to meet the substantial requirements of this plant, should have ample spare capacity to supply the much lesser needs of the laboratories. In the meantime it will be necessary to provide a temporary supply to laboratory 2.

In laboratory 1, the only water requirements are for the cooling of the X-ray equipment and this is provided by a recirculating chiller

needing only occasional top up with distilled water.

3. Electrical Services

Like water, a good electrical supply is essential but that supplied to NMDC has a variable voltage and is subject to frequent interuptions. Most instruments can cope with sudden power failures and voltage variations may be smoothed by in-line voltage regulators fitted to sensitive instruments. However for the XRD and XRF instruments sudden power failures may cause instrumental damage because of the large voltages involved. A new generator has been installed near the laboratory which could be used whenever the two instruments are operating. It is not sufficient for it to cut in when power is interrupted because the built-in delay will allow the cessation of supply to the instruments.

Laboratory 2 lacked any reasonable power distribution within the building. It is understand that wiring instructions were given sometime ago but, so far, these have not been carried out.

4. Laboratory Fitments

A) Laboratory One

This building comprised a large central room with five smaller rooms leading off. An annex of two rooms was attached to one side of the building with a separate entrance. The X-ray diffraction (XRD) instrument had been installed in one of the smaller rooms and

it was planned to install the X-ray fluorescence (XRF) instrument in the adjacent room. Both these rooms were next to the annex and were connected to it through a hatch for passing samples, etc, and had air conditioning. The central room and the other smaller rooms had no fitments but will need a selection of desks and tables for office and interpretation work. One room of the annex contained the water chiller for the instruments and the other had been fitted out with benches for sample preparation facilities.

B) Laboratory 2.

This building was the same as laboratory one but without the annex. One AAS instrument had been fitted in one room, with an extraction fan. A start had been made on a second extraction system but this remained incomplete throughout the visit. This room should have the second AAS instrument, if it can be made to work, and the flame photometer. Because of the heat generated during operation and the ambient temperatures, air conditioning is required for the room. A second room was identified by the consultant for use as a balance room, whilst the carbon sulphur instrument was installed in a third room, one room will be needed for an office, calculation area. etc. The central room had been allocated for solution preparation and a fume cupboard had been ordered. This was installed during the visit. The Consultant recommended also the installation of a laboratory sink and table bench working areas. Regarding the fume cupboard, it was the Consultant's opinion that it would have insufficient fume extraction capability for the work which will be carried out. Escape of fumes is not only injurious to health but

can initiate corrosion of sensitive instrument components. It appears that the suppliers of the fume cupboard were not given any specification regarding efficiency requirements, i.e, face velocities for the installation. The fume cupboard had several other, less serious defects which affect its efficiency and life.

5. Recommendations.

The following recommendations arise from the above discussion:

A) Laboratory One

i) Checking the efficiency of the air conditioning for both the XRD and XRF instrument rooms.

ii) Connection of both instruments to the new generator.

iii) Provision of desks/tables as a working area.

iv) Installation of the mill and powder press in the annex.

B) Laboratory Two.

i) Installation of a reliable water supply.

ii) Installation of good electrical wiring to all rooms.

iii) Provision of sink and building of the solution preparation table.

iv) Completion of the second fume extraction system in the AAS room.

v) Fitting of Air conditioning to the AAS room.

vi) Checking efficiency of the fume cupboard.

vii) Removal of all material left over from the previous occupants.

III. ATOMIC ABSORPTION SPECTROSCOPY (AAS).

1. The Training Programme.

The programme for AAS consisted of a number of distinct phases:

A) Supplied Material

The trainees were given photocopies detailing the theory of atomic spectroscopy. This was for self learning and was intended to revise and expand upon lectures received at Univerisity or College.

B) Lectures

i) The pertinent theoretical aspects of AAS necessary to a thorough understanding of the technique.
ii) Description of the main components of AAS instruments, the lamp, the detector and monochromator, with a demonstration of these features on the NMDC instruments.
iii) Description of solution preparation, the function of the nebuliser, burner design and safety and the formation of atoms.
iv) Units of concentration, calibration solutions and calculations in AAS.
v) A detailed presentation of a standard method for the determination of Cu, Pb, Zn, Ag, Fe, Co, Ni, and Mn by AAS in soil, rock and mineral samples, the necessity for pure water and reagents, techniques and calculation of results

C) Practical Work.

i) Application of the standard method to the analysis of selected samples for Cu, Pb, Zn, Fe and Ag, setting up and use of the instrument for analysis.
ii) Safety, routines and preventative maintenance.
iii) Use of standard data for instrumental conditions and the operations manual.

2. AAS Instruments at NMDC

NMDC has two AAS instruments, a Perkin Elmer 303 (nearly 20 years old) and a Pye Unicam SP9 (about 8 years old). At the time of arrival of the Consultant neither instrument was working.

A) Perkin Elmer 303

This was completely inoperable and no service engineer was known in Nigeria. The Consultant inspected the instrument and concluded that the fault lay in the electronic circuitry. The problem could only be solved by an experienced service engineer. In view of the age of the instrument, the fact that the supplier's no longer maintain spares for this model and the lack of a local engineer, it is recommended that this instrument be scrapped. It has a substantial number of lamps but these do not readily fit the Pye Unicam instrument. The Consultant will investigate the possibility of purchasing a suitable adaptor.

B) Pye Unicam SP 9

This instrument had been recently serviced, however it was reported that an acetylene leak rendered the it unsafe to use. The operating manual was unavailable, but the Consultant was able to organise a copy from the Nigerian Mining Corporation laboratory which was copied for future use at the NMDC. The Consultant inspected the instrument and found that the source of the acetylene leak was so serious that it was considered lucky that NMDC had not had an explosion causing damage to the instrument or injury to the operators. Also certain minor but critical parts had been wrongly fitted at installation which meant that the instrument had not ever been operated correctly. Partway through the visit the service engineer arrived to service the instrument under the supervision of the Consultant. Following this work, the fitting of temporary parts by the Consultant to correct the original installation faults and fine tuning of the instrument, the Consultant considered the instrument to be working close to the quoted specifications. It was obvious that the previous service history of the equipment was entirely unsatisfactory.

The range of lamps for this instrument was small (only 7 in total) and it is recommended that this be extended. The possibility of using the Perkin Elmer lamps will be investigated, but if this is not possible other lamps need to be purchased.

3. Consumeable Supplies

It is very important for AAS to have pure chemicals available for use to avoid contamination of samples and standards during preparation for analysis. For most uses "Analar" grade chemicals may be used but for very low level determinations "Aristar" grade must be used. The availability of "Analar" grade chemicals in the laboratory was limited and no "Aristar" grades were on site.

Similarly water for dilution of samples must be pure. It is usual to use a distilled or deionied supply. The laboratory tap water was of poor quality and contained a high level of suspended and dissolved solids. The laboratory had a water still but no deioniser. (It is unlikely that a deioniser would operate efficiently considering the poor quality water supply). A high level of solids had built up in the still and it is expected that single stage distilled water in the laboratory would be inadequate for accurate AAS work. The use of double distilled water or single stage distilled water passed through a deioniser may be necessary. A second still existed in the laboratory, but this had never worked properly. The Consultant pointed out that it was installed incorrectly and recommended correct installation. (Subsequently, later during the visit, the first still failed completely and the second still was re-installed correctly).

The range of laboratory glassware, beakers, flasks, pipettes, etc. is inadequate to support an instrumental and wet chemical laboratory. Other items considered essential to a laboratory did not exist. These were such items as, reagent dispensers, weighing

pans, beaker tongs, safety glasses, etc. Many of the standard stock solutions were very old and of doubtful integrity. No standard samples containing elements which were suitable for AAS analysis were available. The Consultant prepared a list of consumeable items which were recommended for purchase for the instrument laboratory.

4.Recommendations

A) Training

i) Lectures will be given to demonstrate the use of alternative flameless samples presentation systems such as furnace, hydride and mercury cell, the use of back ground correction techniques and the method of standard additions.

ii) Further sample analysis will be undertaken to improve techniques and accuracy. Other methods for analysis will be introduced.

iii) Further instructions will be given in preventative maintenance.

B) Instruments

i) The instrument should be equipped with further lamps either by adapting the Perkin Elmer lamps or purchase of new lamps. (Annex IV).

ii) The instrument requires a set of spares for further use.

iii) Servicing beyond that described in the manual should be undertaken by a qualified services engineer (who exists).

iv) No further money should be spent on the Perkin Elmer instrument.

C) Consumables Supplies

i) The recommended list of consumables items should be purchased. (Annex IV)

ii) The water quality from a single distillation will be monitored by the Consultant to assess the need for further water purification.

IV ATOMIC EMISSION SPECTROSCOPY (AES)

(with particular emphasis on Flame Photometry (FP)

1 The Training Programme

A) Lectures

i) Revision of the theory of Atomic Spectrocopy with particular emphasis on AES. Methods of AES including FP, inductively coupled plasma (ICP), spectrography (arc and spark) and use of AAS instruments for AES.
ii) A description of the Flane Photometer instrument, the main components, limitations and a practical demonstration of the features of the NMDC instrument.
iii) Calibrations, solutions and calculations.
iv) Standard method for the determination of Na, K and Li.

B) Practical Work

i) The use of the standard method using FP to determine Na, K and Li on selected samples
ii) Safety, routine and preventative maintenance.
iii) Use of the AAS instrument in the AES mode.

2. The NMDC Flame Photometer

The instrument was the Fison Flame Photometer of unknown age and

appeared to be in good condition. The Consultant carried out routine maintenance which required ıttention to rust and corrosion on the atomiser and burner. The instrument then seemed to be working satisfactorily but the performance could not be checked because the operating manual was missing.

3. Consumable Items

These have been covered under the AAS Chapter (III.3).

4. Recommendations

A) Further samples will be tested to improve technique and accuracy.

B) Further training will be given on routine and preventative maintenance.

V ULTRA VIOLET/VISIBLE SPECTROMETRY (UV/Vis)

1. The Training Programme

A) Lectures

i) Theoretical aspects of UV/Vis, Lambert's Law , Beer's Law, the electromagnetic spectrum, absorption and transmission, deviations from Beer's Law.

ii) Factors involved in the selection of a procedure, reagents, interferences, separations, control of pH, time, etc.

iii) Description of the main features of UV/Vis instruments and demonstration of NMDC instruments.

iv) Typical methods of analysis. (The detailed procedures arise from Mr Bin Fu's training programme).

B) Practical Work

i) Setting up of instrument, checking responses and interferences.

ii) Producing an absorption curve.

iii) Reading solutions produced in Mr Bin Fu's programme.

iv) Routine and preventative maintenance.

2. UV/Vis Instruments at NMDC

NMDC has two instruments, Pye Unicam, SP-200 and SP-550, both at least 8 years old. They are located in the balance room of the wet chemistry laboratory, which is not ideal because of possible

contamination of the balances but acceptable with careful use. The room is large and is well situated close to laboratory where the solutions will arise. The instruments, therefore, should not be moved to the instrument laboratory. The Consultant was advised that neither instrument was working. On his detailed examination of the instruments, the problems with the SP-200 seemed to be confined to a disconnected wavelength drive belt, incorrectly positioned source lamp and a faulty shutter mechanism. When these faults were corrected the instrument was working close to specification.

For the SP-550 the Consultant concluded that the main problem lay with failure of one of two photomultiplier tubes. During the visit the service engineer arrived and replaced the faulty tube. He then carried out a full service under the supervision of the Consultant following which it was concluded that the instrument was working close to specification. One area of doubt existed for the engineer engineer concerning the operation of the alternation Deuterium source lamp. However the Consultant adviced NMDC that this was unlikely ever to be required for their work and replacement was not necessary.

It was apparent that previous service work carried out by other companies was of doubtful quality causing more problems that those solved. It also seemed that some parts had been rifled. For a more efficient operation is recommended that a minor item, a manual cell changer, some spare parts and a wider selection of samples cells be purchased.

3. Consumable Items

Since the work concerns the wet chemical laboratory any required items will form part of Mr. Bin Fu's report.

4. Recommendations

A) The UV/Vis instruments should remain in their present location because that is close where the samples will arise. The chemist in charge of the wet laboratory will take responsibility for the instruments.

B) A manual cell changer should be purchased. (Annex 4)

C) Some spares and additional cells should be purchased (Annex 4)

D) Servicing beyond that described in the manual (quite detailed) should be left to the qualified service engineer.

E) Further practical training is use of the instruments should arise for application of procedures laid down by Mr. Bin Fu.

VI CARBON AND SULPHUR ANALYSIS

The instrument, a Leco Model, was very old and has been superceded several times by the manufacturer's. It was not possible to assess the condition of the instrument because of the lack of power supply. However if this can be installed the necessary tests will be carried out during the next visit of the mission.

The Consultant strongly recommends that NMDC should consider the purchase of a modern instrument. Because of the association with the Steel Industry such an instrument is essential to enhance NMDC's image. However such equipment is not cheap so that the amount of expected future business must be considered also.

VII X-RAY DIFFRACTION ANALYSIS

1. The Training Programme

During the Consultant's visit a previously arranged training programme organised by the installer's of the instrument was carried out by staff of Zaria University. The Consultant attended this programme, assisted the lecturers and later was able to expand an special features and answer questions arising from the earlier course. The programme below combines the main features from both the external and the Consultant's programme.

A) Lectures

i) Theory of X-rays - lectures and distribution of notes for self study.
ii) Theory of X-ray powder diffraction.
ii) Main components of XRD instruments, practical demonstration on the NMDC instrument.
iv) Sample preparation for XRD.
v) Interpretation of results.
vi) Safety aspects in the use of X-Rays.

B) Practical Work

i) Preparation of diffractograms of known and unknown samples.
ii) Interpretation of the diffractograms.

iii) Presentation of results.

iv) Limitations and advantages of XRD.

v) Routine and preventative maintenance.

2. The NMDC Instrument

The instrument is a Phillips PW 1800 X-ray powder diffractometer with computer control purchased 5 years ago but only recently installed. Cooling of the instrument is achieved by a recirculating water chiller. During installation the water chiller had been filled with tap water but distilled water must be used to avoid internal corrosion and deposition of solids. The tap water must be drained and thoroughly flushed out with distilled water before filling with further distilled water. The air conditioner should be checked for correct operation and be kept running at all times.

The instrument had been ordered without a chart recorder. This meant it was not possible to produce diffractograms. A print out of readings could be produced but interpretations from this were tedious, inexact and almost impossible for complex mixtures or difficult analyses. Two alternatives exist for interpretation; the chart recorder and manual interpretation or a computerised system.

The Consultant advised the chart recorder method because:

a) Much cheaper cost.

b) A knowledge of manual interpretation is required in application of the computerised system.

c) The capacity of the computerised system would far exceed the present needs of NMDC.

d) Simplicity the computerised system requires a high level of technical knowledge for operation and maintenance.

The laboratory lacked the necessary search and identification manuals essential to the operation of an XRD facility. Only a limited number of types of sample holders had been ordered and further types are required to extend the range of analyses. Similarly a tool to aid packing of powder samples is required also. For sample preparation it is necessary to finally grind small samples and two agate pestles and mortars should be purchased.

3. Recommendations

A) The chiller must be filled with distilled water.

B) The air conditioners should be checked for correct operation.

C) A chart recorder, interface, paper and spare parts should be purchased (Annex IV).

D) The XRD search and data manuals should be purchased. (Annex IV).

E) Additional samples holders, sample preparation kit and agate pestles and mortars should be purchased. (Annex IV).

F) Further training on sample analysis will be carried out during the next visit when all the necessary items are at hand.

VII X-RAY FLUORESCENCE

1. Introduction

At the time of the visit the instrument, a Phillips PW 1404 Automatic Sequential XRF Spectrometer, was not installed although it had been purchased in 1988. The supplier's were insisting that NMDC spend considerable sums of further money before the instrument could operate satisfactorily. The Director requested the Consultant to advise him on the matter.

2. Method

The Consultant made a careful inventory of the supplied items and concluded that the delivery according to the supplier's list was virtually complete (missing only a few minor items reported stolen). After a study of the operations and service manual it was apparent that the instrument could operate satisfactorily provided a keyboard terminal and screen was available. (These NMDC already have). The Consultant studied the quotations of the supplier and found that most of the items listed were already included in the materials supplied or were optional extras not necesary to the basic operation.

The Consultant was concerned that the sample preparation equipment for milling and pelletizing samples before XRF analysis were complete and working. (These had been used with an earlier

instrument). He was assured of this by the Assistant Chief Chemist but there was a need for consumeable items. The Consultant confirmed that the pellets were the correct size for the new instrument. It was found that NMDC lacked any suitable standard samples for the preparation of the standard series necessary to calibrate the instrument before analysis.

3. Recommendations

A) NMDC should arrange the installation of the XRF instrument before the next visit of the Consultant, although the programme could tolerate this work during the early part of the visit.

B) NMDC must move the grinding mill and pelletising machine to the new laboratory as soon as possible and extablish they are both working correctly.

C) NMDC must purchase consumeable items for the pelletising process and standard samples for calibration. The Consultant will source and price these items during the break in the mission.

IX COAL ANALYSIS

1. Introduction

The Consultant received a conducted tour of the Fuel and Energy Division laboratories and observed that there was a good selection of equipment for the physical testing of coal and coke. The laboratories were also equipped with facilities for the 'proximate' analysis of coal, sulphur and calorific values. A discussion took place on the analytical requirements of the Fuel and Energy Division and how the Analytical Section could assist. The following list of analytical requirements was agreed:

a) Carbon, hydrogen and nitrogen in coal samples.
b) Forms of sulphur in coal (sulphate sulphur, pyrite sulphur and organic sulphur by calculation).
c) Analysis of coal ash for major components and deleterious trace elements.

Where possible the appropriate ASTM or BS method would be followed. In addition the Head, Fuels and Energy, sought the Consultant's assistance in sourcing and pricing equipment for the Hardgrove grindability and Gray-King coking methods. The Consultant will do this during the break in the mission.

2. Carbon, Hydrogen and Nitrogen

Both the Analytical Section and the Fuel and Energy Division were under the misapprehension that gas chromatography could be used for these determinations. Unfortunately it is not an appropriate technique and the officers were referred to the ASTM methods manual. All three determinations could be carried out by the analytical section given the right equipment and training in procedures. The Consultant will source and price the equipment during the break in the mission.

3. Forms of Sulphur

These methods are well within the present capability of the Analytical Section, involving a gravimetric method for sulphate and an atomic absorption method for pyrite sulphur. The first would be covered by Mr. Bin Fu and the second by the Consultant.

4. Coal Ash Analysis

This involves the determination of SiO_2, TiO_2, CaO, MgO, Fe_2O_3, Na_2O, K_2O, P_2O_5, SO_3 and the heavy metals Mn, Zn, Pb, Cu, Cd, Cr.
Mr Bin Fu has trained the wet laboratory in SiO_2, TiO_2, Al_2O_3, CaO, MgO, P_2O_5, and SO_3 and the Consultant has trained the instrumental laboratory in Na_2O and K_2O by FES and heavy metals by AAS. The Fe_2O_3 may be determined by wet chemistry or AAS. The Analytical section already has the capability to carry out this work except that a full range of AAS lamps does not exist.

5. Recommendations

The Analytical Section should be trained in the forms of sulphur and ash methods and that consideration be given to the economic viability of purchasing equipment and introducing the C, H and N determinations.

X EQUIPMENT SERVICING

1. Introduction

The Director asked the Consultant to consider how the centre could improve its ability to service equipment and the programme was extended to include this additional item. Whilst the Consultant was concerned mainly with equipment in the analytical section and became fully aware of the situation there, it was apparent from observation and discussion that similar conditions existed across the site. It is an unfortunate fact that much of the NMDC's equipment is inoperative and that much fails far too quickly, note the high number of furnaces awaiting repair. It is acknowledged that there is a lack of suitable external service engineers and, where they exist their charges are expensive, but it is important to consider why NMDC suffers so many failures in the first place. Modern instruments are generally reliable and will last many years with proper attention.

2. Basic Services

This concerns mainly water and electricity supplies and their distribution round the site. The external supplies of water and power are unreliable, often subject to shut down and variations in pressure or voltage and poor quality water with high suspended and dissolved solids content. Sensitive instruments are difficult to operate under such circumstances.

NMDC plans to install boreholes and water tanks to overcome the water supply problems and this may result in a higher quality supply. If not, then distilled water must be used for sensitive situations - note the discussion on X-ray instruments (chapter 7). NMDC has diesel generators for periods of power failure but in some cases, again note the X-ray chapter, sudden failure may cause serious damage which a generator will not prevent. Other instruments may withstand power failures but be sensitive to voltage fluctuations. These require connection through in-line voltage regulators.

The quality of electrical wiring, fitting of electrical plugs, use of the correct fittings and fuses leaves much to be desired. Not only may this be dangerous, but wrongly specified wiring, poor connections and earthing can affect equipment leading to poor operation and rapid failure.

3. Equipment Installation

The Consultant was faced with several cases where equipment had been incorrectly installed. It was notable in two instances; a muffle furnace and a water distiller, two very simple pieces of equipment. In the first case failure occured after one month and in the second the equipment never worked. The problems were more subtle in the case of the AAS instrument, wrong parts fitted on installation 8 years ago and not detected until the present visit meant that the instrument had never worked correctly. The use of tap water instead of distilled water for the XRD instrument would

have led to rapid failure if it had not been detected by the Consultant.

It is important that installation instructions, usually provided by the suppliers are read and understood by all concerned before installation commences. All suppliers will send further copies of instructions if they are not found with the instrument. Failure to carry out the installation instructions properly usually invalidates guarantees and warranties.

Whilst the maintenance section will have the responsibility for fitting and connection to services, the responsible officer is the user, the laboratory chemist or whoever, and they must ensure that the work meets the installation specifications, understand the operation of the instrument and supervise the installation to ensure that all is correct.

4. Equipment Maintenance

Instrument operating manuals usually lay down instructions for routine maintenance, fault finding, specification checks, etc. Again, if these are not available further copies should be requested before operation is attempted. In too many cases at NMDC, manual copies are not available with the instrument resulting in incorrect operation and no routine maintenance, with all the associated problems. Again it is the responsibility of the senior staff to ensure that equipment is operated correctly and routinely maintained. This means that these people must make great efforts to

become technically aware of the instruments under their control.

The manuals lay down fault finding charts or tables which allow the users to check the operation of certain components. The manuals give clear instructions on what may or may not be replaced by the user. How many instruments remain in operative because of interference by inexperienced staff? In the Consultant's inspection of balances, some had multiple faults many of which could not have been caused by even harsh operation.

It is the responsibility of the person in charge of a particular instrument to carry out the work laid down in the manual. Of course he may need the assistance of the maintenance section in some cases but he must supervise all such work and take ultimate responsibility.

Manuals are very clear at which stage work must be left to a trained service engineer. Because of the wide variety of equipment and its specialised nature it is unlikely that NMDC staff could ever be trained to carry out this work unless they attended the maintenance training courses run by the instrument supply companies. The use of external service engineers then becomes essential.

Fortunately for the analytical laboratory the engineer for Unicam can be recommended. He is well-trained, carried a comprehensive tool kit and spare parts selection and worked efficiently and well. Also he is accredited to Unicam to whom representation can be made

in case of unsatified complaint. This accreditation is too valuable to the engineer to risk losing it. Because of his training and the lack of competition the engineer's charges may seem expensive, however it has been found that cheaper alternatives cannot do the work, cause additional problems and may rifle parts. It is recommended that such trained engineers be used wherever possible. It is likely that their charges for spare parts will be high and much cheaper alternatives may be bought overseas. A judicious selection of suitable spares may be recommended.

5. Recommendations

A) NMDC should attempt to address the poor water and power supplies by;

i) providing boreholes and storage tanks,

ii) using distilled or filtered water supplies to sensitive equipment,

iii) using generator power for equipment where sudden failure may cause damage,

iv) using in-line voltage regulators for sensitve equipment.

B) NMDC Maintenance section should ensure that their staff are trained to high standards with respect to laboratory wiring, proper earthing, good connections and appropriate components. The knowledge should be used to ensure quality installations around the site.

C) NMDC senior laboratory staff responsible for equipment must ensure that they are familiar with all factors, such as

installation requirements, operational condition and routine maintenance according to the intruments manuals and ensure that all staff using the equipments are properly trained in these procedures.

D) The responsible officer must carry out, direct or supervise all non routine maintenance and pay due attention to the work of all external engineers.

E) Qualified and accredited service engineers, when available, must be used for maintenance beyond the scope of the operating manuals.

F) Where such engineers do not exist in Nigeria, NMDC should consider external training for their own staff for specialised work and ensure this person(s) is equipped with all necessary spare parts and tools.

G) Staff responsible for an instrument must ensure the they can carry out all maintenance operations listed in the manuals and only they or staff trained by them carry out this work.

H) Unqualified persons or companies must not be engaged to service equipment beyond the scope of the manual.

It is believed that adherence to these recommendations will result in a considerable reduction in equipment failures, equipment will remain serviceable for much longer periods and the need to engage external engineers will be much reduced.

SUMMARY OF RECOMMENDATIONS

Detailed recommendations have been given under the different chapter headings. The main points arising from these are given below.

Introduction - A Laboratory Control Manual is required to improve the overall management of the laboratories.

Sample Preparation - A new facility for the preparation of chemical analysis samples should be developed using the building containing the new, but as yet, unused crushing equipment and additional equipment to be purchased. Training.

Instrument Laboratories - Completion of the rehabillitation, installation of milling and pressing equipment to check operation.

AAS Instruments - Additional training, purchase of equipment spares and consumeables, checking of water quality.

AES Instruments - Additional training.

UV/Visible Instruments - Additional training, purchase of spares.

Carbon and Sulphur - Check of instrument and consider purchase of modern update.

XRD Analysis - Further training, purchase of chart recorder, the Search and Data Manuals, spare parts, change to distilled water in the chiller.

XRF Analysis - Installation of the instrument, purchase of standard samples.

Coal Analysis - Training and consider purchase of C,H,N equipment.

Equipment Servicing - Improve water and electrical supplies, improve services installation quality, senior laboratory staff must familiarise themselves with instrument installation, operation and routine maintenance procedures. Use only accredited service engineers whenever possible and closely supervise their work.

ANNEX I

JOB DESCRIPTION	**DP/NIR/87/031**	**11-68**

POST TITLE:	Consultant in Instrumental Analysis
DURATION:	Six weeks (first part of split mission)
DATE REQUIRED:	As soon as possible,
DUTY STATION:	Jos, Nigeria.
PURPOSE OF THE PROJECT:	Strengthening of the National Metallurgical Development Centre (NMDC), Jos, to develop metallurgical technologies. In the long run, this will lead to improved productivity in the industry as a whole, to substitution of imported raw materials, and to better use of mineral resources.
DUTIES:	The expert will be responsible for training the local operators to carry out elemental and mineralogical determination on metallic and non-metallic ores, coal, metals, etc using XRF, XRD, Atomic absorption, Pye Unicam Spectrophotometers, etc. Therefore, training should include sample preparation, operation and data interpretation, some knowledge of preventive maintenance, etc,

QUALIFICATIONS: Chemical Engineer, Chemist or Geologist, with about 15 years of experience in instrumental analysis, especially of XRF, XRD, Atomic absorption, etc. He or she should possess working experience in Chemical, Mineral, or Metallurgical research institutions or industrial plant.

LANGUAGE: English.

BACKGROUND INFORMATION:

Nigeria is facing enormous challenges in the development of its metallurgical industry. It has to reduce its dependence on imported raw materials and must provide locally made components for advanced industries. The National Metallurgical Development Centre of Jos works in line with the above objectives. Therefore, within the research institution, the analytical service laboratory has been equipped with the above-mentioned instruments in order to support research work by providing elemental and mineralogical determinations.

The local operators require additional training in order to maximize their performance. Some of them have received a certain amount of training.

As a research Centre, NMDC is involved in the analysis of metallic and non-metallic ores, coal, and metals in general.

ANNEX II

WORK PROGRAMME

MR. P. KNOWLES - UNIDO CONSULTANT FOR INSTRUMENTAL ANALYSIS

FIRST VISIT - 5TH OCTOBER - 15TH NOVEMBER, 1993.

SECOND VISIT - EXPECTED JANUARY-FEBRUARY 1994.

PROVISIONAL PROGRAMME

A. First Visit

1. 6th-8th October 1993

   a) Introduction and familiarisation

   b) Preparation of provisional programme

   c) Review of sample preparation laboratory and procedures.

2. 11th-15th October 1993

   a) Theory of spectroscopy and atomic absorption (AAS). This will be a distribution of notes for self study by the participants. The Consultant will answer any queries which arise. The notes are intended to revise University or College lectures with special application to AAS.

   b) Main components of AAS instruments lecture and demonstration using both Perkin Elmer and Pye Unicam Instruments.

   c) Preparation of Standard Solutions and calculations in AAS. Lecture and Practical exercises.

   d) Preparation and analysis of samples for Pb, Zn, Cu.

3. 18th-22nd October 1993

a) Revision of theory of spectroscopy to extend to flame emmission and UV/Vis spectroscopy. (Testing of learning of notes sent out in 2a above).

b) Main components of FES Instruments, lectures and practical demonstration with the flame photometer.

c) Preparation of standards for FES.

d) Analysis of samples for Na, K, Li.

e) Main components of UV/Vis Spectrometers.

f) Preparation of standards and analysis of samples. (This aspect will be coordinated with Mr. Fu-Bin's programme).

4. 25th-27th October

Carbon/Sulphur analysis - check working of instrument.

5. 28th October - 5th November

a) Theory of X-rays

Lecture and distribution of notes for self study

b) Theory of X-ray powder diffraction.

c) Main components of X-ray diffratometers.

Lecture and practical demonstration using the Phillips XRD.

d) Sample preparation for XRD.

e) Analysis of selected samples by XRD

f) Interpretation of results.

g) Safety aspects of X-ray Instruments.

Lecture and notes for self-study.

h) Clay analysis.

6. 8th-11th November

a) Visit to Coal Laboratory

Review of procedures requiring any assistance. Assess need for gas chromatography.

b) Review of present visit and verbal report to necessary officials.

c) Confirmation of time-table and programme for the second visit.

d) Completion of report.

B. Second Visit

1. Theory and Practice of XRF
2. Statistics in Analytical Chemistry.
3. Instrumental Laboratory Management and Maintenance
4. Update and extension to the material of the first visit.

ANNEX 111

MEMO

FROM: PATRICK KNOWLES (UNIDO Consultant)

TO: DR. USMAN M. TURAKI (DIRECTOR/CHIEF EXECUTIVE (NMDC)

DATE: 1ST NOVEMBER, 1993

SUBJECT: LABORATORY CONTROL MANUAL

Dear Dr. Turaki,

Further to our recent discussions, I write to confirm that a laboratory control manual is essential to improve the operation and management of the Analytical Chemistry Laboratory. Briefly such as manual would:

a) Describe the laboratory facilities, equipment and staff and define their responsibilities;

b) Describe how the laboratory is organised and managed;

c) Define the responsibilities of management and staff with a description of their duties;

d) Define the ongoing in-house training programme and future development of the laboratory.

Following completion of my mission (at the end of my second visit) I will have gained sufficient indepth knowledge of the laboratory to enable me to prepare such a document and to assist in its implementation. However, such work is outside the scope and time allocation of my present UNIDO mission and would require further funding.

I estimate that three weeks work in the U.K. to prepare the report plus reproduction costs for 15 copies and a three week visit to NMDC to introduce and implement the manual would be required.

For your consideration of this proposal, I must emphasise that successful implementation will depend on full co-operation of all laboratory staff especially those involved in management.

Yours Sincerely,

PATRICK KNOWLES.

ANNEX IV

ITEMS TO EQUIP THE INSTRUMENT LABORATORY

1. Hotplates (2)

2. Beakers Borosilicate

   48 x 400 ml + 48 watch glasses to fit

   24 x 600 ml + 12 watch glasses to fit

   12 x 250 ml + 12 watch glasses to fit

   6 each 100 ml, 1L

   2 x 2L.

3. Volumetric Flasks all 'A' grade

   36 x 100 mls

   12 each 250, 500, 1000mls

   6 each 10, 25, 50mls

4. Pipettes 'A' Grade

   Bulb 6 each 5, 10, 20, 25mls

   3 each 1, 2, 50, 100mls

   Graduated 6 each 5, 10mls

   3 each 1, 2mls

   12 pipette bulbs

   2 pipette stands.

   Plastic Pipettes 5, 10mls graduated.

5. Measuring Cylinders

   Glass 3 each 10, 25, 50, 100mls

   Polythene 3 each 10, 25mls

6. Dispensers

   3 each tilt 2, 5, 10, 20 mls with bottles

   2 each 250mls dispensing burettes.

7. Burette Stands, Clamps and bosses 3 of each

8. Beaker Tongs, 6 off.

9. Safety glasses 12 pairs

10. Marigold gloves 12 pairs

11. Disposable gloves 2 boxes 100.

12. Wash bottles 24 500mls with tips.

13. Telfon Dishes 12 off

14. Balance scoops.

15. 12 balance brushes

16. 50 stirring rods with bobies.

17. 6 small filling funnels.

18. 6 x 250mls graduated separating funnels and stands.

19. 2 distilled water containers with bottom taps.

20. 4 dozen marker pens (not water soluble)

21. Chemicals

   24 x 2.5l Analar HCL

   24 x 2.5l Analar HNO_3

   6 x 2.5l Analar $HCLO_4$

   6 x 500ml Bromine

   6 x 500ml HF

   3 x 2.5l DIBK

22. 2000 Test tubes and 4 Racks

23. Long and short handled Pt tipped tongs

ANNEX IV (continued)

SPARE PARTS FOR INSTRUMENTS

A. XRD

1. 1 x PW 1770/10 Kit
2. 3 each sample holders PW 1813/26, 1813/32 and 1812/00
3. 1 x Recorder PM 8261/02 and Interface 9190/20,
   6 x Pens - 9920 and paper.
4. Search and data manuals.
5. Two Agate Mortars and Pestles.

B. UV/Vis

1.	Manual Cell Changer	9423-179-05371
	Accessory Lid	9423-179-05321
2.	Cells 4 each	0.5, 2.0, 4.0 cm.
3.	Tungsten Halogen lamp LP2	4013-171-63541
4.	Spare cell clips	4013-163-81231
5.	Beam attenuator	4013-164-91360
6.	Cam Spanner	2613-150-12213
7.	2 each all fuses for	SP6-550
8.	Filter and attenuator set	9423-179-08501
9	Set of spares	

C. AAS

1.	Nebulizer	4013-172-03400
2.	Lamp location mouldings stepped	4013-164-15100
3.	Lamp sleeves	4013-172-04020
4.	Spray chamber renovation kit	4013-172-20260
5.	Over pressure disc	4013-164-13881

6. Sample uptake tube 2m PP44 0813-031-98003 2m
7. Burner 'O' ring 2622-080-91045 2 off
8. Burner setting jig 4013-172-04390 1 off
9. Fuses F51, F52, 2422-086-01026 4 off
10. Selection of lamps
11. Selection of Standard solutions

D) Balances

1. 2 x Transformers Sartorious Balance
2. 6 x bulbs.

E) Tool Kit For Instrument Laboratory.

F) Selection of Standard Samples.

G) Sample Preparation

1) Dust Extractor
2) Ring Mill Grinder
3) Sample Drying Oven
4) Selection of sample bags
5) Vacuum cleaner
6) Riffle boxes
7) Selection of consumeable items

H) XRF - Replacement of flow counter windows lost from the delivery

ANNEX V

SENIOR COUNTERPART STAFF

1. Dr. Usman M. Turaki, Director/Chief Executive NMDC
2. Mr. C.I.C. Nwankwo, Chief Chemist, Head of Scientific Services Department, NMDC.
3. Mr. A.T. Lawal, Head of Chemical Analysis Unit, Scientific Services Department, NMDC.

STAFF PARTICIPATING IN TRAINING PROGRAMME

1. Mr. B. Katsina, Head of Instrument Laboratory.
2. Mr. L. P. Ganchok, Chemist, Instrument Laboratory.
3. Mr. M. M. Chagga, Chemist, Instrument Laboratory.
4. Mrs. F. L. Fabumni, Environmental and Pollution Section.
5. Mr. R. Makam, Fuel and Energy Division.