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SUPPORT TO NATIONAL METALLURGICAL DEVELOPMENT CENTRE

DP/NIR/87/031/11-68

JOS, NIGERIA

Technical Report: Second 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

Substantive officer: C. Beinoff, UNIDO

United Nations Industrial Development Organisation
Vienna

SUMMARY

This report describes the second half of a mission to the National Metallurgical Development Centre, Jos, Nigeria, for the Consultant to complete a training programme in instrumental methods of analysis. The first half of the mission was reported in November 1993.

It had been hoped that certain items such as the supply of essential ancillary equipment and chemicals, the completion of facilities and the installation of the X-ray fluorescence instrument would have been completed in time for this second visit but for a variety of reasons this was not the case. These factors determined that the objectives of the mission were not fully attained in two main areas of X-ray diffraction and fluorescence.

To compensate the Corsultant allocated more time to Atomic Absorption analysis and included work on Environmental projects. This latter aspect lies within the capability of the laboratory and involves the substantial use of instrumental techniques.

It must be concluded that the full aims of the mission cannot be achieved within the allocated time frame and it is recommended that an extension to the mission be arranged when all the required items have been received and all installation work is complete.

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INTRODUCTION

The mission of Consultant Chemist, Mr. Patrick Knowles, (the Consultant) to the National Metallurgical Development Centre, Jos, Nigeria, (NMDC) involved the training of local operators in instrumental methods of analytical chemistry, (Job Description - Annex I). The mission was split into two parts of six weeks each; the first visit took place during October and November 1993 and was described in a report dated 16 November 1993. The second visit took place during January and February 1994 and is the subject of this report.

At the start of the second visit the Consultant reviewed the activities of the first visit and progress during his absence from mid-November 1993 to mid-January 1994. A programme for the second visit was then agreed (Annex II). Briefly this concentrated on the following topics.

(a) Atomic Absorption Spectroscopy (AAS)
(b) X-ray Fluorescence Analysis (XRFA)
(c) Coal Analysis
(d) Environmental Projects.

In addition, quotations for various items recommended in the first report which the consultant had obtained and submitted to NMDC were discussed and prioritised for the placing of orders. In general it has to be concluded that only a little progress had been achieved during the break in the mission. Much of the work recommended to NMDC to improve the facilities had not been carried out and, for various reasons, certain key items necessary to the operation of some of the equipment were not able to be ordered and supplied. The installation of the XRF instrument was only completed just before the end of the visit. Where possible,

time was spent during this second visit to progress all these items as much as possible.

This report must be read in conjunction with the first report which set out the situation at the start of the mission and progress during the first visit. This report updates the mission from the end of the first visit to the end of the second visit. Certain discrete topics of the first report are not repeated here. This report has the same chapter headings as the first report, in the same order, but an additional chapter on environmental projects is included.

I SAMPLE PREPARATION

1. Introduction

The first report discussed the essential requirements for sample preparation, the current facilities at NMDC, and recommended that a laboratory separate from mineral processing be established under the control of the analytical section. Many of the components for this already existed but were unused. Renovation of a building, the purchase of a dust extractor, a grinding mill and consumable items were required with training of preparation staff.

2. Present Facilities

The jaw and roll crushers in the mineral processing section remained dismantled through until just before the end of the second visit and until then laboratory had no working sample preparation facilities.

3. Proposed Facilities

No renovation work had been carried out on the proposed new facility and this did not progress during the visit. It was agreed that a ring grinding mill should be purchased as soon as possible, but other items such as the dust extractor and consumable materials were given a lower priority. In the absence of the new facilities no personnel training was possible.

4. Recommendations

The recommendations of the first report are still advised to NMDC.

II INSTRUMENTAL LABORATORY FACILITIES

Two laboratory blocks are provided, one for the X-ray instruments and the other for AAS and other instrumental techniques. The first report recommended a number of improvements required to complete the blocks, and by the end of the second visit the following work had been carried out.

1. In Laboratory one
    a. A generator had been installed intended to supply the X-Ray laboratories as part of the circuit, but the wiring and connections necessary to allow control from the X-ray rooms remained to be installed.
    b. The water cooler had been flushed with distilled water.
    c. Both X-ray rooms had working air conditioning units.
    d. The grinding mill and powder press had been moved to the X-ray sample preparation annex and were connected to the power supply. The press required top up with hydraulic oil for correct operation. The mill was missing an essential part to allow its proper operation. The consultant will obtain a quotation for this part.
    e. Orders had been placed for 3 tables/desks.
2. In Laboratory Two
    a. The sink and working bench had been installed
    b. The AAS room had an air conditioning unit.

The other recommendations remained incomplete. Whilst it was stated during the first visit that the electrical wiring of the block had been ordered, it was agreed during the second visit that the Consultant would prepare an electrical layout diagram for the engineering division to progress the installation.

III ATOMIC ABSORPTION SPECTROSCOPY

1. Introduction

The programme was continued with further lectures and more practical work.

A) Lectures

i) The method of Standard Additions for sample analysis.
ii) Solvent Extraction Techniques.
iii) Ionisation Suppressors.
iv) Releasing and Protection Agents
v) Background Correction
vi) Limitation of Flame Atomisation and the use of flameless Atom Cells
vii) Electrothermal Atomisation - the Graphite Furnace
viii) The Hydride method
ix) The Cold Vapour Mercury Cell
x) The Delves Cup, the Slotted Tube Atom Cell and Atom Trapping AAS.

B) Practical Work

i) Further analysis using the standard method
ii) Analysis of water samples using solvent extraction and evaporation procedures
iii) Determination of lead in roadside dust samples
iv) Determination of pyritic iron in the analysis of coal samples for the forms of sulphur analysis
v) Determination of magnesium in limestone samples by the method of standard additions.

2. AAS Instruments at NMDC

Unfortunately it was concluded that the Perkin Elmer lamps could

not be adapted to work with the Pye-Unicam instrument and further lamps need to be purchased. As stated in the first report further expenditure on the Perkin Elmer instrument is not recommended. However many of its lamps are unused and the sale of these and the instrument as spare parts to a current users of Perkin Elmer elsewhere in Nigeria would release useful funds to help maintain the Pye Unicam instrument in good working order.

3. Consumable Supplies

The purchase of consumable supplies and spare parts was allocated as second priority in the assessment of the needs of the laboratory.

4. Recommendations

a) The staff of the laboratory should continue to implement the techniques demonstrated during the mission at every opportunity.

b) The sale of the Perkin Elmer instrument as spare parts together with the supply of lamps should provide useful funds to NMDC.

IV ATOMIC EMISSION SPECTROSCOPY

This aspect of the training programme was completed during the first visit. The techniques was used for the analysis of water samples during the present visit.

V ULTRA VIOLET/VISIBLE SPECTROMETRY

This aspect of the training programme was completed during the first visit. The purchase of spares, etc was allocated to the second priority purchase list.

VI CARBON AND SULPHUR ANALYSIS.

Unfortunately the power supply remained unconnected to this laboratory and it was not possible to check or operate the instrument nor to carry out any training.

The purchase of a new instrument was allocated to the second priority list.

VII X-RAY DIFFRACTION ANALYSIS.

The necessary and recommended items for the analysis of samples by XRD, had not been purchased but were allocated to the first priority list. Additional training in the practical aspects of this technique was not possible during the second visit. Some theoretical aspects were covered during the lectures on XRF. (Chapter VIII)

This aspect of the mission can only be completed when the necessary items have been received and by an extension to the mission.

VIII X-RAY FLUORESCENCE ANALYSIS.

I. Introduction

The installation of the instrument was only completed just before the end of the second visit so that no practical training was possible. The NMDC staff observed all stages of the installation to become familiar with the instrument and training from the service engineer on the basic operation of the instrument was scheduled. Lectures were given by the Consultant on the theoretical aspects of the technique and consideration was given to the type of samples to be analysed using the instrument. It was also concluded that application software would be necessary for routine operations and this was allocated to first priority purchases.

2. Lectures

a. A definition of XRF analysis.
b. The generation of X-rays and the theory of X-ray production.
c. The form of X-ray spectra - relationship of energy to wavelength.
d. X-ray absorption and fluorescence.
e. Main components of X-ray instruments.
    i. The tube.
    ii. The crystal and Bragg's law.
    iii. The detectors.
f. Qualitative analysis and the use of X-ray tables.
g. Quantitative analysis.
    i. Spectral interferences and how to overcome them.
    ii. Matrix effects - primary and secondary absorption and enhancement.
    iii. Calibration curves.
    iv. Method of standard additions.

v. Mathematical methods, empirical coefficients and fundamental parameters.

h. Advantages and disadvantages of XRF analysis.

i. Sample preparation - physical effects, surface roughness, particle size and packing density.

j. Preparation and analysis of solid, liquid and gas samples.

k. Measurement of metal plating thickness.

3. Sample Preparation Equipment

A grinding mill is required for fine grinding, the preparation of standard samples and the mixing in of binding agents. The powder mix is then pressed into a pellet prior to the X-ray analysis using a pelletising press.

A grinding mill and pelletising press existed on site having been used in a previous X-ray laboratory. These machines were moved to the new laboratory. It was found that the mill was missing a minor but essential part for which the consultant will obtain a quotation for NMDC. The powder press required top up with hydraulic fluid and it is not known whether it is in proper working order.

Both machines are essential to the operation of the XRF analysis laboratory.

4. Analytical Systems

4.1 Introduction

It was necessary for the Consultant to consider the probable future use of the instrument for analysis. For each type of sample analysed it is necessary to have a series of standards or accurately analysed samples for calibration purposes or in some cases it is possible to prepare synthetic standards from pure

chemicals. Although NMDC staff had some ideas on the required analyses the Consultant gave some attention to how this might be achieved practically.

Discussions were held with appropriate heads of department to assess their analytical requirements and consideration was given to the possible needs of external clients. The following analytical systems were identified:

a. Iron ore samples - both as low grade ores and concentrates.
b. Clay, rock and refractory materials.
c. Limestone, dolomite, magnetite and cements.
d. Chromite and chrome-magnesites.
e. Bauxites.
f. Phosphates.
g. Major elements in coal ash, sulphur in coal.
h. Tin, tungsten, tantalum, niobium and other ores and concentrates.
i. Steel and other metal samples.

It was decided to prepare list of possible standards to cover the above requirements, assess the costs and allocate priorities to meet the allowed budget.

4.2 Standard Samples

a. Iron ore samples - an excellent range of standards are available from a number of sources. A total of 14 were identified, seven for each of the ranges of total iron 24-40% and 50-70% respectively with corresponding matrix elements. The sources were;

i. Bureau of Analytical Samples, UK (B.A.S) - 6 standards
ii. Canada Centre for Mineral and Energy Technology (CANMET) - 4 standards

iii. Standards Association of Australia (SAA) -1 standards
iv. Bundesanstalt for Materialforschung, Germany, (BAM) - 1 standard.
v. National Institute of Standards and Technology, USA, (NIST) - 2 standards

b) Clay, rock and refractory materials - again a very wide range of samples and sources are available. Standards were selected to cover the range, silica 30-75% and alumina 10-60% and a variety of other elements as follows

i. British Ceramic Research, (CERAM) - 12 standards
ii. B.A.S. - 8 standards.

c) Limestone, dolomite, magnesite and cements - a fair range of standards are available and these were selected to cover the range 1-90% magnesium oxide and 1-70% calcium oxide with appropriate impurity elements.

i. CERAM - 12 standards
ii. B.A.S. 4 standards
iii. South African Bureau of standards (SBS) - 1 standard.

d) Chromite and chrome-magnesites - a fair range of standards exists and a selection for the range of chromic oxide 20 -50% was made.

i. B.A.S. - 4 standards
ii. CERAM - 5 standards
iii. SBS - 1 standards
iv. British Geological Survey (B.G.S.) - 1 standard.

e) Bauxites - a fair range of standards are available and these were selected in range, alumina, 40 - 50% or up to 90% for calcined bauxite.

i. BAS - 2 standards
ii. CERAM - 2 standards
iii. NIST - 4 standards.

f) Phosphates - only a limited range is available and those were selected to cover the range for phosphorus peroxides of 7 - 40 %

i. BAS - 3 standards
ii. SBS - 1 standard
iii. BAM - 1 standard
iv. NIST - 1 standard

g) Coal and Coal ash - NMDC have a number a standards for sulphur and other samples were selected for the limited range available as follows;

i. S.B.S - 3 standards
ii. S.A.A. - 2 standards

Other standards are available from NIST but these are very expensive and were rejected on cost grounds. Additional standards may be prepared from synthetic chemicals.

h) Tin, tungsten, tantalum, niobium ores, etc. - only limited range of suitable standards exist and these will be used in combination with synthetic standards

i. CANMET - 2 standards
ii. BGS 6 standards
iii. BAS 2 standards

i) Steel and other metal samples- NMDC already has a selection of standards. However the certificates of analysis are missing and the Consultant will try to obtain further copies of these for

NMDC. A decision on whether further purchases are necessary depends on the range of the present standards and the expected work requirements.

4.3 Availability Cost and Source of Standard Samples

There are many sources of standards samples, some of which are named in the proceeding section, but there are others. Where possible the selections have been made to cover the expected ranges of the main and impurity elements likely to be met with Nigerian materials but this is not possible in all cases. Additional ranges will have to be covered by the preparation of synthetic standards or by samples previously analysed accurately by the Wet Laboratory. As far as possible the selections have been based on the most economical sources of which BAS, CERAM and BGS are the cheapest and these have been used wherever possible. The other sources used are medium to high cost but the selections are necessary to cover the expected range of concentration.

The purchase of standard samples were allocated as first priority.

5. Recommendations

Because of the delays in the installation of the XRF instrumental and the non-availability of standards and the analytical software, the training programme for this technique could not be completed during the term of the mission. Because of the importance of the instrument it is recommended that consideration is given to a extension to the mission to allow this work to be completed.

IX COAL ANALYSTS

1. Introduction

In the first report the Consultant recommended a number of areas where the analytical section could assist the Fuels and Energy Division. These were;

a) Carbon, hydrogen and nitrogen in coal samples

The Consultant had prepared a costing of the necessary equipment and this was allocated to the second priority list for purchases.

b) Forms of sulphur in coal.

The Consultant prepared a procedure for the laboratory based on the British Standards and ASTM methods. The laboratory carried out the analysis of selected samples under the supervision of the consultant.

c) Analysis of coal ash

In addition to the possibility of wet analysis, the determination of major elements by XRFA is described in chapter VIII. The heavy metals may be determined by AAS given a suitable selection of lamps.

d) Hardgrove Grindability and Gray-King Coking Determination

The Consultant had prepared costings from suppliers quotations and these were allocated to the second priority list for purchases.

e) The Determination of Total Sulphur

During visit the consultant hoped to use the Leco-Sulphur in coal apparatus, held by the Fuels and Energy Division. However this instrument seems to have been subject to rodent attack and now requires a major service.

The consultant prepared a wet chemical procedure for use in the Analytical Section. The use of XRFA will also be investigated.

2. Recommendations

The Head of Fuels and Energy Division should prepare a financial appraisal for the purchase of the carbon, sulphur and nitrogen Analyser, Hardgrove Grindability and Gray-King Coking test, before purchase is approved. The appraisal should consider the number of samples to be tested over a period of time and the relative costs of sub-contract to an overseas laboratory (known to the Consultant) and purchase and operating costs. It should also consider the strategic importance of each equipment to be available in Nigeria. The Management Board of NMDC should only approve the purchase if it is satisfied with the appraisal.

X EQUIPMENT SERVICING

The Consultant considered this aspect at length in the first report. The findings are recommended to NMDC. It was notable during the second visit that extensive problems still existed in water and power supply to the laboratories. The working relationship between the maintenance section and the operating divisions leaves much to be desired with many minor jobs being subject to lengthy delays. This factor has affected the training programme.

XI ENVIRONMENTAL PROJECTS

NMDC has a small environmental section concerned with sampling and analysis. The Consultant believes that the analytical aspects could readily be undertaken by the analytical sections (both wet chemistry and instrumental) and recommends this change for consideration by NMDC. The Environmental Section should then concentrate on identifying suitable projects, carrying out the sampling and preparing the reports. The employment of a specialist environmental scientist is, of course, a necessary pre-requisite as would the purchase of appropriate field sampling and instrumentation equipment.

The Consultant recommends that contact is made with the Federal Environmental Protection Agency which may be persuaded to fund such a development by NMDC in exchange for NMDC becoming an authorised FEPA laboratory, thus providing NMDC with a useful source of future work.

The Consultant prepared a memorandum (Annex III) corsidering the various aspects of Environmental work and suggested two projects within the present capability of NMDC which might be undertaken to gain experience. These were;

a) The sampling of a river to monitor the change in chemistry as it passed through Jos.

b) The sampling of roadside dust samples in a busy part of Jos to assess the lead content.

These projects were undertaken during this visit of the Consultant.

CONCLUSIONS AND RECOMMENDATIONS

1. Following the completion of the second visit it is appropriate to review the progress achieved by the full mission. Unfortunately it has not been possible to achieve all the objectives for five main reasons.

a) At the start of the mission much of the equipment on which training was to be given was not working or not installed.

b) In some cases ancillary equipment or consumable supplies necessary to perform the analysis was not available and needed to be ordered or had not been installed.

c) Many of the laboratory facilities were incomplete.

d) Problems with water and power supplies caused unforseen delays.

e) The level of previous training of some staff was less than expected.

2. a) For AAS., FES and U.V/Visible, the training programmes were completed and the staff need to practice what they nave learnt at every opportunity.

b) It was not possible to achieve any progress at all for the Leco Carbon and Sulphur in Steel Analyser which remained inoperable throughout both visits due to lack of power connections. The instrument, itself, is very old and it was hoped to, at least, check if it was in working order. It was concluded in the first report that NMDC should consider the purchase of a modern instrument which was agreed subject to the availability of funding. The training element for this technique is small and could be achieved in just a few days.

c) The most disappointing progress was in the X-ray methods. The XRD instrument was installed and working but because of the lack of a recorder and the necessary Search and Data Manuals, it was not possible to carry out any practical training. The XRF instrument was not installed until very late during the second visit, but, in any case, practical training would not have been possible because of the non-installation of the sample preparation facilities and the lack of any reasonable standards for calibration purposes.

3. To remedy these shortcomings of the training programme, it can only be recommended that a further visit of the consultant is necessary to complete the programme. It is estimated that a further period of about five to six weeks would be required to satisfactorily complete the process. If agreed this visit can only be of use if all the missing items have been received by NMDC and facilities are working to specification and installed.

4. Outside the strict reading of the Job Description the Consultant has sought to advise NMDC on sources of work for the instrumental (and Wet chemistry) laboratories. The opportunities in environmental work have been discussed specifically in this report, but when all the techniques are working there should be a concerted effort to interest outside clients in using the laboratories. Such work will supplement the internal work and provide income to help maintain and improve the present facilities.

5. The first report discussed another important topic concerning management and quality control procedures for the laboratories and an extension to the mission was agreed to give time to this topic. Good procedures give confidence to clients, encouraging the use of laboratories, possible income to the

laboratories and promote staff morale. It is suggested that the extension necessary to complete the training programme could be accommodated by adding to this extension already agreed.

6. Finally it must be stated that, despite the deficiencies described above, NMDC appear to have benefitted considerably from the training programme. All the instruments except the Leco Analyser were in working order at the end of the second visit. The staff have received a detailed theoretical grounding in all the techniques and, except for XRD and XRF, have undertaken a broad spectrum of practical work and now should be capable of carrying out a fair range of instrumental analytical projects.

ANNEX I

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

POST TITLE:	Consultant in Instrumental Analysis
DURATION:	Six weeks (second 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

MEMO

FROM; MR. P. KNOWLES
UNIDO EXPERT IN MISSION
INTRUMENTAL ANALYSIS

TO: DR. U.M. TURAKI
MR. C.I.C NWANKWO
MR. A.T. LAWAL
MR. B.M. KATSINA

Programme For Visit 19 January - 28 February 1994

(A) 19-23 January - Travel to site and orientation

(B) 20-31 January
- (1) Revision of AAS operation
- (2) Lectures on alternative AAS sources
- (3) Forms of sulphur in coal by wet chemistry and AAS

(C) 1-7 February
- (1) Installation of XRF
- (2) Installation of sample preparation for XRF (Mill and Press)

(D) 8-14 February
- (1) Standardisation and Operation of XRF
- (2) Lectures on Theory of XRF

(E) 15-21 February
- (1) Operation of XRF
- (2) Operation of XRD

(F) 21-28 February
- (1) C and S Analysis
- (2) Discussion of findings of the Visit
- (3) Report presentation
- (4) Travel Home

This programme may be modified by the timing of the visit of XRF installation engineers.

Other matters such as coal testing, refractory testing and discussion of equipment orders will be arranged at appropriate times during the above programme.

ANNEX III

MEMO; FROM: P . KNOWLES
TO: C.I.C. NWANKWO

ENVIRONMENTAL SAMPLING SUGGESTION

I have spoken to Mrs Fabunmi who asked if I could recommend any suitable environmental work within the capability of the present equipment. I considered the main areas of interest, air, water, soil and waste.

(1) AIR - this requires special equipment for measurements, collection of air and dust samples. This is not available at present to NMDC.

(2) WATER - whilst extraneous pollution by mining or other industry may be a problem in certain areas of Nigeria, the general problem will be bacteriological contamination of drinking water supplies. Such work requires specialist equipment, expertise and training not available in NMDC. It might be best undertaken in cooperation with an outside bacteriological laboratory (private, University etc)
I suggest a simple project to start with would be to find a river which passes through Jos and to take a sample upstream and one downstream from the city. These should be analysed for standard water parameters, to check if the city has caused any significant alteration. Any particular problems would be checked by intermediate sampling and repeat. Sampling at regular intervals.

(3) SOIL - these may be contaminated by deposits from chimneys, solid or liquid waste effluents from industry. An important area is lead deposits at roadsides from petrol. I suggest you select a busy section of road in the city and collect road dusts along a small section, and also away from the road and

analyse these for lead. The results will suggest future work.

(4) WASTES - these require knowledge and location of their arisal and disposal and investigations would be difficult without a specific project.

(5) Conclusions

Two projects are suggested to start with;

(1) River sampling,

(2) Roadside lead Testing.

P. KNOWLES