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STRENGTHENING GOVERNMENT SUPPORT
SERVICES IN THE NON-METALLIC MINERALS SECTOR

DP/ZIM/83/006/11-02

(R) ZIMBABWE

Technical report: Technology development and product
diversification for the ceramic industry

Prepared for the Government of Zimbabwe
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Gyorgy Lenkei,
expert in production and technology of ceramics

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United Nations Industrial Development Organization
Vienna

Explanatory notes

The monetary unit in Zimbabwe is the dollar (\$Zim).

References to tonnes (t) are to metric tonnes.

Mention of the names of firms and commercial products does not imply endorsement by the United Nations Industrial Development Organization (UNIDO).

ABSTRACT

Within the context of the large-scale project "Strengthening of government support services in the non-metallic minerals sector" (DP/ZIM/83/006), an expert in production and technology of ceramics was assigned to the project for a period of six months, starting in June 1985.

The purpose of his mission was to evaluate existing ceramic products and to recommend measures for quality improvement and product diversification; to train local counterparts in ceramic technologies and lecture on certain topics; and to identify training needs and recommend suitable training programmes for local staff.

The expert appraised the raw material situation and the existing fine-ceramic industry, established the properties of local raw materials in a series of tests and, based on the results, developed and tested new compositions of bodies and glazes for sanitary ware, porcelain insulators and wall tiles.

The examined raw materials were found to be suitable for product diversification and an expansion of the ceramic industry, and the expert makes detailed recommendations to that end. To substitute the import of glazes for wall tiles and tableware by locally produced glazes, he recommends to continue research and to provide equipment for frit firing as well as additional equipment for the ceramic laboratory.

The expert trained the local laboratory staff in the testing of fine-ceramic raw materials, bodies and glazes, gave a series of training sessions in ceramic technology and recommends the further training of at least one staff of the laboratory through a fellowship.

CONTENTS

	<u>Page</u>
INTRODUCTION	6
RECOMMENDATIONS	7
<u>Chapter</u>	
I. ACTIVITIES AND OUTPUTS	8
A. The raw material situation	8
B. Evaluation of the existing fine-ceramic industry	8
C. Technological research work	9
D. The status of the pilot plant	13
E. Ad-hoc assistance to local manufacturers	14
F. Training in ceramic technology	15
II. UTILIZATION OF THE RESULTS	16
III. CONCLUSIONS	17
<u>Annexes</u>	
I. Job description	19
II. Equipment listed in the project document	20
III. Equipment received under the project	21
IV. Tested raw materials	22
V. Properties of casting slips, bodies and glazes for sanitary ware	23
VI. Properties of bodies and glazes for porcelain electric insulators	25
VII. Properties of porcelain bodies fired to different temperatures	26
VIII. Properties of wall-tile bodies	27
IX. Properties of wall-tile bodies fired to different temperatures	28
X. Properties of Zimbabwean wall-tile bodies and glaze	29
XI. Properties of frits glazes	30
XII. Additional equipment for the pilot plant	31
XIII. Spare parts for the pilot plant equipment	32
XIV. Sample log sheet	33
XV. Curriculum of training in ceramic technology	40
XVI. Additional laboratory equipment	41

INTRODUCTION

The Zimbabwean authorities are aware of the wealth of different non-metallic minerals that exist in the country. The UNIDO project "Processing of ores of light non-ferrous metals" (SI/ZIM/82/801) drew the attention of the Zimbabwean Government to the possibility of industrially exploiting those resources which might be utilized not only in the existing industries as substitutes for imported raw materials, but also as a basis for new industrial activities in Zimbabwe.

The immediate objectives of the present project, "Strengthening government support services in the non-metallic minerals sector" (DP/ZIM/83/006) the project document of which was signed in June 1984, were to assist in:

- (a) The further strengthening of the already developed capability of the laboratories of the Department of Metallurgy in the qualitative and quantitative testing of non-metallic minerals;
- (b) The immediate utilization of locally available resources for an increased and diversified production of the existing industries, specifically the ceramics industry;
- (c) The establishment of an information base for the industries concerned, and the creation of a systematic non-metallic minerals inventory for that purpose.

In order to achieve those objectives, the inputs by UNDP and UNIDO included various services and equipment, and the assignment of the expert in production and technology of ceramics (post No. 11-02) was one of the expert services rendered. During his mission of six months, from June to December 1985, the expert was attached to the Department of Metallurgy, Ministry of Mines. His job description is reproduced in annex I.

The original UNDP input for equipment as indicated in the project document is given in annex II. However, in the project budget the cost of only a part of the equipment (see annex III) could be accommodated. Consequently, the pilot plant received only three pieces of equipment, which are, in part, suitable for body and glaze preparation. There is no possibility for shaping, drying, glazing and firing.

RECOMMENDATIONS

1. The examined local raw materials should be used by the existing ceramic industry as well as in new plants.
2. The existing ceramic industry should diversify its products, based on current processes, namely:
 - (a) Sanitary ware production at Clay Products Ltd.;
 - (b) Extruded tile production at Willsgrove Bricks and Potteries Ltd.;
 - (c) Technical porcelain production at Morbel Potteries Ltd.
3. The experimental production of white glaze for sanitary ware from domestic raw materials should be undertaken at the Department of Metallurgy. If the results of those trials are positive, it might be possible to stop the importation of white sanitary glaze.
4. If trials on an industrial scale are positive, existing wall-tile manufacturers should introduce local brick clays in the composition of wall-tile bodies to reduce the costs for raw materials and energy.
5. The possibility of producing kiln furniture locally at Clay Products Ltd. should be examined. To that end, the suitability of domestic talc and fire clay ought to be clarified.
6. Research on glazes for wall tiles and tableware should be continued with a view to reducing their importation. This requires the creation of suitable conditions for frit firing, at laboratory level, at the Department of Metallurgy and the verification of the possibility of industrial production at Zimglass Ltd.
7. The possibility of utilizing various waste materials (e.g. fly ash) in the ceramic industry should be investigated.
8. To improve the performance of the ceramic laboratory further equipment, listed in annex XVI, should be provided.
9. The future of the pilot plant should be discussed at the next tripartite review meeting.
10. At least one staff of the local laboratory should be trained for one month in the production of bodies and glazes for sanitary ware, porcelain insulators and wall tiles.

I. ACTIVITIES AND OUTPUTS

A. The raw material situation

For the production of fine ceramic products (sanitary ware, porcelain insulators, tiles etc.) non-metallic plastic and non-plastic raw materials are needed for the bodies, and various metallic oxides and chemicals for the glazes. The following of those raw materials are available in the country, i.e. they are being mined and sold:

(a) Plastic materials: ZT kaolin, Beit Bridge ball clay, Hwange fire clay, Gwaai ball clay;

(b) Non-plastic materials: Mistress silica, Bikita silica, silica from Gweru, Mistress feldspar, Bikita feldspar, dolomite, limestone, talc, kyanite, Bikita petallite, manganese ore, chrome ore;

(c) Metallic oxides and other chemicals: ZnO, SnO₂, PbO, Na₂CO₃, borax.

The plastic raw materials are milled but not washed. The non-plastic raw materials, except the ores, are crushed and milled. In addition, there are some raw materials mined and used locally such as Rutendo refractory clay, Chiredzi flint clay and various brick clays (Willsgrrove, Mt. Hampden), and a few others mined only from time to time (Kadoma kaolin, Bude kaolin, new kaolin).

On examination of these raw materials two aspects were of main interest:

(a) The quantity and quality of the domestic and imported raw materials used by the existing ceramic industry, considering the possibility of reducing importation;

(b) Further possible applications of available domestic raw materials in the fine-ceramic industry, over and above the ones already being used.

The answers by six main manufacturers to a questionnaire indicate that the importation of ceramic raw materials is insignificant. Only some 100 t/year of plastic and non-plastic raw materials are imported, while the consumption of domestic raw materials is about 4,000 t/year, including the requirements of the refractory industry. The value of glazes imported by the existing ceramic industry amounts to \$Zim 150,000-200,000 per year. This sum ought to consist of several smaller constituents considering the wide range of colours used in the production of tableware.

To investigate further applications of raw materials, those listed in annex IV have been tested for composing sanitary-ware, porcelain-insulator and wall-tile bodies. However, the tested raw materials were only such which are regularly mined and prepared to some degree. There are still many occurrences of other ceramic raw materials in the country, the applicability of which should be investigated in the future.

B. Evaluation of the existing fine-ceramic industry

G. and W. Industrial Minerals, Harare

This company is practically the only one which buys, prepares and sells various ceramic raw materials. Present annual sales are about 7,000-7,200 t,

of which less than 100 t are sold to the ceramic industry, while the main part is consumed by the paper, rubber, paints and cosmetics industry. The equipment of G. and W. is suitable for crushing, dry-milling, classification and packing of plastic and non-plastic raw materials.

Clay Products Ltd., Bulawayo

This company produces mainly normal-duty refractories, but also earthenware pipes, fittings and flower pots.

Recently they started to produce sanitary ware on an experimental basis, using domestic raw materials in the casting slip. The glaze is imported. They employ a few skilled workmen for shaping and have one electrically heated top-hat kiln for firing.

The laboratory is basically suitable for performing raw material tests, production and product control. By manufacturing sanitary ware it is intended to increase the output of the kiln step by step. There is a possibility to improve the casting properties of the slip and to substitute the imported white glaze by a domestic one.

Willsgrove Brick and Potteries Ltd., Bulawayo

At this company there are more profiles for producing bricks, tableware and extruded tiles. Domestic raw materials are used for the bodies, but the glazes and the kiln furnitures are imported. Electrically heated top-hat kilns are used for firing. The company started a trial production of various technical ceramics in order to substitute imports.

The technological potentiality of increasing the production of tableware and extruded tiles exists, but the shortage of imported glazes makes it impossible. The local raw materials are suitable for transparent and opaque basic glazes, but unfortunately no suitable kilns are available for firing frits to a temperature of 1,380-1,420 °C.

Norbel Potteries Ltd., Harare

The company manufactures tableware using domestic raw materials for the bodies, and imported glazes and kiln furniture. For firing they have electrically heated top-hat kilns. The possibilities of substituting the import of glazes and kiln furniture are the same as mentioned for Willsgrove Ltd. Some equipment and the professional skills of the staff would permit product diversification, e.g. the production of technical porcelain.

Stewart Tiles Ltd., Harare

That small company produces wall tiles. The raw tiles are manufactured by manually driven fly-presses, dried and fired in electrically heated driers and kilns. The press powder, the imported glaze and the kiln furniture came from a manufacturer who recently closed down. At present a drum mill, a filter press and an edge-runner mill are being put into operation to manufacture press powder. A glazing conveyor belt and a chamber kiln are under construction to increase the production. The possibilities of producing glazes and kiln furniture locally are the same as mentioned for Willsgrove Ltd.

C. Technological research work

The raw materials listed in annex IV, to be used in bodies and glazes for sanitary ware, porcelain electric insulators and wall tiles, have been tested, using the following testing methods and equipment:

- (a) Chemical analysis;
- (b) Mineralogical tests by DTA (Netzsch);
- (c) Grain size analysis of the non-plastic raw materials by laboratory sieving machine, 53-100 μm ;
- (d) Particle size analysis of the plastic raw materials by a Warman cyclosizer, 0-75 μm ;
- (e) Viscosity measurement of clay and casting slips for sanitary ware by the so-called tube system and by Brookfield's viscometer;
- (f) Determination of casting rate (wall thickness) of plastic raw materials and slips for sanitary ware;
- (g) Determination of plasticity by the Pfefferkorn method;
- (h) Determination of water vapour absorption of plastic raw materials;
- (i) Determination of linear shrinkage after drying and firing;
- (j) Determination of bending strength of dried and fired bodies by bending strength tester (Netzsch);
- (k) Thermal expansion of various ceramic bodies and glazes by a dilatometer (Netzsch);
- (l) Determination of water absorption;
- (m) Determination of bulk density;
- (n) Autoclave tests on glazes.

Bodies and glazes for sanitary ware

Sanitary appliances such as wash-basins, water closets, urinals, bidets and others are made from a mixture of clays and other minerals and rendered impervious to water by firing to high temperatures. Vitreous china sanitary ware is coated on all surfaces exposed to view in normal use with an impervious glaze giving a smooth, shiny, white or coloured finish which is durable and easy to clean.

The following process sequence is typical: preparation of casting slip, shaping by casting in plaster moulds, drying, glazing and firing.

The tests were aimed at achieving the following:

- (a) Usage of only domestic raw materials in body and glaze;
- (b) A litre-weight of the casting slip of 1,740-1,780 g/l;
- (c) A casting rate of the slip of 8-9 mm/90 seconds;
- (d) A water absorption after firing to 1,240-1,260 °C of 0.5-1%.

By meeting those requirements, production according to international standards would be ensured.

Based on the results of the chemical analysis the following raw materials were used in testing the composition of bodies and glazes for sanitary ware: ZT kaolin, Hwange fire-clay, Beitbridge ball clay, Mistress silica, Mistress feldspar, Chimbaranga feldspar, limestone, ZnO and SnO₂.

Prior to the preparation of the casting slips, the casting properties of the plastic raw materials i.e. the change in fluidity with various deflocculants, and the casting rates were to be determined. The litre-weight of the basic slips was in the range of 1,300-1,500 g/l. As deflocculants Na₂CO₃, Na₂SiO₃ and sodium tannate (prepared by the expert) were tested. The sodium tannate proved to be the most effective deflocculant for all clays. The optimal quantities to be added to the various clays were established and a litre-weight of 1,570-1,660 g/l was achieved.

The casting rates of these clays were tested in plaster moulds prepared by the expert, applying casting times of 30, 60 and 90 minutes, and the thickness and the moisture content of the walls were recorded. On the non-plastic raw materials the particle size distribution and the melting behaviours were examined by a firing test.

On the basis of these results, eight combinations were tested by examining the following properties: residue after milling, litre-weight, thixotropy, casting rates, moisture content of the bodies, linear shrinkage, water absorption and rupture after firing. The values of shrinkage and rupture were measured on extruded test pieces. The firing temperature was 1,250 °C, the soaking time 0.5 hours and the firing time 6 hours. After evaluating the results based on the average, control tests were made on the two best recipes. The results were satisfactory and met the general technological requirements.

The opaque effect in glazes for sanitary ware can be achieved by application of tin oxide or zirconium silicate, but none is produced commercially in Zimbabwe.

The first recipe which contained, besides the domestic materials, imported tin oxide to about 10 per cent, yielded a good result. The possibility of producing SnO₂ from flue dust and from metallic tin coming from the local metal industry has been examined. The flue dust is a by-product in the production of metallic tin. By appropriate handling of the flue dust, based on the results of DTA and TG examinations, suitable SnO₂ has been prepared. Other SnO₂ samples made from metallic tin are also suitable for sanitary glazes. The results obtained correspond to international standards in terms of brilliance, whiteness and good thermal shock resistance.

As a consequence of those tests local manufacturers can be advised on how to compose bodies and glazes for sanitary ware containing exclusively domestic raw materials. The various compositions and test results are detailed in annex V.

Bodies and glazes for porcelain electric insulators

Porcelain electric insulators, such as low- and high-tension insulators, are made from a mixture of kaolins, clays and other minerals and rendered non-porous to water by firing to high temperatures. The appropriate composition of the body guarantees proper mechanical and electrical properties. By using suitable glazes, the insulator operating properties can be improved.

The following process sequence is typical: forming of slip, dehydrating by filter press or spray-drier, extruding or forming press powder, shaping by throwing or pressing, drying, glazing and firing.

The tests were aimed at achieving the following:

- (a) Usage of only domestic raw materials in body and glaze;
- (b) A flexural strength after drying of $2-3\text{N/mm}^2$;
- (c) A water absorption after firing to $1,300-1,320\text{ }^\circ\text{C}$ of 0.0%;
- (d) A minimum flexural strength after firing (unglazed) of 50N/mm^2 .

This would ensure production according to international standards.

Based on the results of their chemical analyses, the following raw materials were used: ZT kaolin, Beitbridge ball clay, Chiredzi flint clay, Mistress feldspar, Mistress silica, kyanite, Bikita feldspar, dolomite, clacite, manganese ore, chrome ore, ZnO, fired porcelain debris, porcelain biscuit. The kyanite was calcined at $1,450\text{ }^\circ\text{C}$ because of its volume increase of 16-18% during firing. The kyanite and Chiredzi flint clay were used to achieve higher flexural strength after firing.

Four different recipes were tested (see annex VI), by examining the following properties: residue after milling; plasticity; linear shrinkage and rupture after drying; linear shrinkage, rupture and water absorption after firing. The values of shrinkage, rupture and water absorption were measured on extruded test pieces. The firing temperature was $1,320\text{ }^\circ\text{C}$, the soaking time 0.5 hours and the firing time 6.5 hours.

The values of bulk density, rupture after firing and water absorption meet the requirements of international standards. Other technological characteristics such as plasticity, shrinkage after drying and firing and rupture after drying satisfy the general technological requirements. The whiteness of the bodies is below the international level, but this does not preclude their practical application. The slightly different colour is partly due to the properties of the used raw materials, but mainly a result of the fact that with the kiln at our disposal reducing conditions could not be achieved.

It is known that reducing conditions are necessary during a certain period of porcelain firing to achieve an appropriate colour and a high density. In spite of this problem, it is certain that the examined raw materials are suitable for producing porcelain insulator bodies.

During research on brown and white porcelain glazes, the shortage of brown ceramic stains and Al_2O_3 presented difficulties. Brown porcelain glazes usually contain 5-10% of brown ceramic stains produced by a few specialized companies (Degussa, Blythe Conlors, Romer etc.). To substitute these ceramic colours, trials have been made with domestic manganese and chrome ores. As for white glaze, the shortage of Al_2O_3 posed a problem, but eventually suitable brown and white glazes were developed. The different compositions and test results are detailed in annexes VI and VII.

There was no possibility to examine the electrical properties.

Bodies and glazes for wall tiles

Wall tiles are made from a mixture of clays and other minerals, and after glazing and firing they should be suitable from a hygienic and aesthetic viewpoint, to cover walls in bathrooms, toilets, kitchens, hospitals, premises in the food industry etc. Tiles are durable, easy to clean and the possibilities of colouring and decorating them are wide.

The usual process sequence is as follows: forming a slip, dehydrating and forming a press powder by spray-drier, shaping by pressing, drying, biscuit firing, glazing and decorating before glaze firing.

The main technological goals to be achieved were:

- (a) Usage of only domestic raw materials in body and glaze;
- (b) A flexural strength after drying of $5-7\text{N/mm}^2$;
- (c) A water absorption after firing to $1,000\text{ }^\circ\text{C}$ of 15-20%;
- (d) A thermal expansion coefficient in the range $20-500\text{ }^\circ\text{C}$ of $7-8.10^{-6}\text{K}^{-1}$.

This would meet the basic requirements and ensure production according to international standards.

The possibilities of producing wall tile bodies was investigated because a pertaining request had been made by a manufacturer (Stewart Tiles Ltd.) and also because suitable raw materials are available.

The following raw materials were used in the tests: Mt. Hampden brick clay, Hwange fire clay, Beitbridge ball clay, limestone, Mistress silica and tiles' grog. Three different combinations were tested, examining the following properties: residue after milling, litre-weight of the slip, linear shrinkage and rupture after drying and firing, bulk density, thermal expansion and autoclave tests on glazes. The values of shrinkage, rupture, water absorption and bulk density were measured on extruded test pieces fired to four different temperatures. Compositions and results are given in annexes VIII and IX. The values of rupture after drying and firing, the porosity and the thermal expansion meet the general technological requirements. At present one of the compositions is being tested under industrial conditions.

The main properties of the "old" and "new" tiles of the above-mentioned manufacturer were determined and the results are as shown in annex X. There are remarkable differences between the values of rupture after drying compared with the results in annex VIII.

Trials were made to produce tile glaze. Due to a lack of suitable firing possibilities for frits, an oil-heated kiln fitted with a locally made burner was used, which made a precise control of the firing conditions impossible. The composition of the frit and glaze, as well as some properties of a white glaze, are given in annex XI. One component of the frit, borax, is imported but is readily available.

D. The status of the pilot plant

The equipment for the pilot plant listed in annex III has been installed and used from time to time. Nevertheless, one cannot yet speak of a pilot

plant, because the available equipment will be useful for batch preparation in the fine-ceramic technology only if it is supplemented by additional items, such as tanks, mixers, magnets, screens, pumps etc. The most important equipment which can be produced locally was determined (see annex XII) and its manufacture has started. A list of spare parts for the pilot plant equipment was also compiled and is reproduced in annex XIII (B).

Once these supplementary items will have been put into operation, the pilot plant will still be suitable only for producing ceramic masses. Further equipment for the fine-ceramic technology is needed for shaping, drying, glazing and firing. In accordance with the earlier project proposal, further equipment, listed in annex XII (A), will be needed in the future, the most important item being the $1m^3$ oil- or gas-heated kiln.

It is evident that the pilot plant is far from being fully equipped and this situation should be rectified as soon as some knowledge and experience in laboratory work on ceramic raw materials and technologies has been acquired.

E. Ad-hoc assistance to local manufacturers

During the expert's mission useful contacts have been established with some manufacturers who supplied basic information on the used raw materials and samples for the research work, particularly with G. and W. Industrial Minerals, Norbel Potteries Ltd., Clay Products Ltd., Stewart Tiles Ltd. and Willsgrove Brick and Potteries Ltd., and professional discussions were held with some of them.

During the regular consultations with Stewart Tiles Ltd. it was proposed to:

- (a) Develop a new batch for a wall tile body based on a local brick clay. Semi-industrial trials are in progress with the new body;
- (b) Change the stowing system in the biscuit-firing kiln to reduce waste;
- (c) Alter the so-called "feet" of the tiles to get better surface on them;
- (d) Alter the drying technology (control and change of the humidity in the driers).

The main properties of the tiles produced by that company (see annex X) were determined, evaluated and discussed, and some practical advice was given on the body preparation.

During consultations with Clay Products Ltd., they were informed of the test results with bodies and glazes for sanitary ware (annex V), and it seems that the casting properties of the newly developed bodies are better. Five kilograms of sanitary glaze which was prepared during these activities is now being tested by Clay Products Ltd. on an industrial scale.

Norbel Potteries Ltd. asked the expert to contribute to their research work on glazes, particularly to give advice on firing to a lower temperature and on how to reduce their importation. Because of the difficulties in frit firing, no practical help could be given.

Based on an assessment of existing possibilities, testing and classification methods for ceramic raw materials have been determined. The results of chemical, mineralogical and physical analyses have been systematized and collected on simple log sheets, in co-operation with the expert in testing, classification and evaluation of non-metallic minerals, and the main properties of six plastic and four non-plastic raw materials have been determined in that way. A specimen log sheet is reproduced in annex XIV. The main testing methods needed for fine-ceramic bodies and glazes were also determined.

Five lectures were given to the local laboratory team on the principles of casting-slip preparation for sanitary-ware and wall-tile bodies. Advice was given on wet chemical testing methods used in the laboratories of the five ceramic factories.

As a result of this systematic work, the local laboratory team has acquired basic knowledge in the application of laboratory testing methods and the classification of ceramic raw materials and bodies.

F. Training in ceramic technology

Three training sessions in ceramic technology were given to the specialist selected from the staff by the national project director. The curriculum is given in annex XV.

II. UTILIZATION OF THE RESULTS

The main results of the expert's activity are:

- (a) Bodies and glazes for:
 - (i) Sanitary ware;
 - (ii) Porcelain insulators;
 - (iii) Wall tiles;

were made exclusively from domestic raw materials (with the exception of borax for the frit);

(b) The local laboratory team gained experience in the application of technological testing methods for fine ceramics.

These results can be used as follows:

(a) The production of glazes for sanitary ware may be initiated at Clay Products Ltd., depending on the results of current trials. The necessary prerequisite for a small-scale production of glazes exist at Department of Metallurgy. This glaze could substitute present imports;

(b) The casting properties of the slips for sanitary ware would shorten the present casting time at Clay Products Ltd. and increase their capacity for shaping. A wider use of such casting slips will depend, first of all, on the quantity obtained by trial production;

(c) The test results with bodies and glazes for porcelain insulators cannot be put to immediate use at the present time;

(d) The wall-tile bodies need to be further improved in order to reduce their sensitivity to drying; otherwise the drying technology would have to be modified. Their introduction into manufacturing would significantly cut down raw material and energy costs (the firing temperature would be 140 °C lower than the present one);

(e) There is no possibility of using the developed wall-tile glaze due to a lack of frit firing facilities. Would such facilities exist, the importation of glaze could be considerably reduced and energy costs saved (glost firing is 80 °C lower than at present);

(f) The experience gained by the local laboratory team in the testing of fine-ceramic raw materials, as well as in the testing and composing of bodies and glazes constitutes a sound basis for future research and for quality improvement of fine-ceramic bodies and glazes.

The most important result is that the examined local raw materials were found to be suitable for a diversification and further development of the fine-ceramics industry.

III. CONCLUSIONS

The following conclusions can be drawn from the experience gained during the activity:

1. The examined domestic raw materials are suitable for the production of bodies and glazes for sanitary ware and porcelain insulators, as well as wall tile bodies.
2. The properties of bodies and glazes for sanitary ware can be improved further by:
 - (a) Determination of the exact firing range and soaking time;
 - (b) Using for the body non-plastic raw materials of correct grain size;
 - (c) Using nepheline syenite in the body and wollastonite and nepheline syenite in the glazes.
3. Body and glaze for sanitary ware are suitable for fast-firing as indicated by their thermal expansion coefficients.
4. The properties of porcelain insulator bodies and glazes can be improved further by:
 - (a) Determining and using non-plastic raw materials of correct grain size in the body;
 - (b) Applying the exact conditions for porcelain firing;
 - (c) Using calcined kyanite to increase the mechanical strength should this be needed for special products.
5. There are good possibilities for the production of wall tiles. Cheap brick clays can be used in the bodies but good homogeneity of the clay material has to be ensured to achieve acceptable linear shrinkage.
6. The laboratory can carry out basic tests on ceramic raw materials, bodies and glazes. To increase its range of research work, the laboratory needs some additional equipment which is listed in annex XIV.
7. The pilot plant is only partly suitable for body and glaze preparation. It should therefore be determined which purpose it is to serve, and, in accordance with this, the required technology should be selected.
8. The experience gained by the local laboratory staff in the testing of fine-ceramic raw materials, bodies and glazes is a good basis for further research.
9. More investigation would be useful on glazes for wall tiles and tableware to reduce the importation and increase the existing production.

Annex I

JOB DESCRIPTION

- Post title: Expert in production and technology of ceramics
- Duration: Six months
- Date required: As soon as possible
- Duty station: Harare, with travel within the country
- Purpose of project: To provide technical assistance to the Department of Metallurgy, Ministry of Mines, in technology development, identification and diversification of existing ceramic industries, including their expansion.
- Duties: The expert will be attached to the Department of Metallurgy, Ministry of Mines, and will be expected to:
- (a) Evaluate existing ceramic products and recommend measures for quality improvement and product diversification;
 - (b) Advise local authorities on composing bodies and glazes for porcelain electric insulators;
 - (c) Advise local authorities on composing bodies and glazes for sanitary ware;
 - (d) Inspect and advise on pilot tests for sanitary ware and porcelain electric insulators;
 - (e) Train local counterparts in basic ceramic technologies applied in Zimbabwe;
 - (f) Lecture on selected ceramic technologies;
 - (g) Identify training needs and recommend programmes for the training of local personnel.
- In co-operation with the economic adviser and the expert in testing, up-grading and evaluation of ceramic raw materials, the expert will also be expected to prepare the final report setting out the findings of his mission and his recommendations to the Government for follow-up actions which might be taken.
- Qualifications: Silicate engineer, experienced in ceramic production and technology.
- Language: English

Annex II

EQUIPMENT LISTED IN THE PROJECT DOCUMENT

A. Laboratory equipment

1 densometer
1 apparatus for breaking-strength determination
1 dilatometer for ceramic glazes and bodies
1 torsion viscometer for ceramic slips
1 laboratory autoclave
1 laboratory hydraulic press
7 Baume densimeters for glaze and slip
1 centrifuge for classification of clay particles
2 Andreason apparatuses
1 kiln for determination of refractories
1 kiln for determination of refractories under load
1 DTA apparatus
1 TGA apparatus
1 gradient kiln
1 device for electric resistance measurement

B. Pilot plant equipment

1 drum mill, capacity 100 litres
1 jet spray drier, output up to 200 kg per hour
1 edge runner mill (mixer)
1 technical balance, 50 kg capacity
2 apparatuses for fast measurement of moisture
1 vacuum extruding machine with mixer
1 drier, 1 m³
1 kiln, 1 m³, temperature up to 1,300 °C
1 kiln, 0.24 m³
1 rotating machine for the shaping of insulators
1 turning table for hand shaping
1 turning machine for shaping into plaster moulds
10 plaster moulds for sanitary ware
1 semi-industrial glazing device
1 spray gun
3 hydrocyclones, 50, 150 and 350 mm diameter
1 kiln, 0.5 m³, temperature up to 3,600 °C
1 complete set of Seger cones

Annex III

EQUIPMENT RECEIVED UNDER THE PROJECT

A. Laboratory equipment

- 1 DTA apparatus for mineralogical research
- 1 TGA apparatus for mineralogical research
- 1 dilatometer for thermal expansion measurements
- 1 laboratory sieving machine for grain size measurements between 100 and 200 μm
- 1 ultrasonic microsieving apparatus for grain size analysis between 5 and 100 μm
- 1 bedding strength tester for modulus of rupture measurements
- 1 Brookfield's rotating viscometer for viscosity measurements
- 1 laboratory furnace for temperatures up to 1,700 °C
- 1 gradient furnace ($\Delta T = 300$ °K) for firing up to 1,340 °C
- 1 high-pressure autoclave for various ceramic tests
- 1 Lange colourimeter for colour measurements
- 1 magnetic separator for de-ironing of slips

B. Pilot plant equipment

- 1 drum mill, capacity 100 litres, for preparing ceramic batches
- 1 Boulton filter press, capacity approximately 200 kg per pressing cycle, for de-watering of ceramic slips
- 1 vacuum extruding machine, capacity approximately 250 kg per hour, for de-airing of ceramic masses

Annex IV

TESTED RAW MATERIALS

Plastic materials

ZI kaolin
Beitbridge ball clay
Kwange fire-clay
Chiredzi flint clay
Mt. Hampden brick clay

Non-plastic materials

Mistress silica
Mistress feldspar
Bikita feldspar
Chimbaranga feldspar
Kyanite
Dolomite
Calcite
Chrome ore
Manganese ore
Metallic oxides and chemicals (SnO₂, ZnO, borax)

Annex V

PROPERTIES OF CASTING SLIPS, BODIES AND GLAZES
FOR SANITARY WARE

A. Casting slips and bodies

1. <u>Preparation</u>	<u>C5</u>	<u>C8</u>
<u>Composition (%)</u>		
ZT kaolin	24.0	24.0
Beitbridge ball clay	19.0	20.0
Hwange fire-clay	10.0	9.0
Mistress feldspar	26.0	27.0
Mistress silica	21.0	20.0
 <u>Milling</u>		
Residue above 200 µm	1.1	0.7
Sodium tannate deflocculant (as % of solids)	0.10	0.18
 <u>Blending</u>		
Sodium tannate deflocculant (as % of solids)	0.75	1.25
 2. <u>Results</u>		
Litre-weight (g/l)	1 699	1 768
Solid content of the slip (%)	65.5	72.5
Thixotropy (T2/T1)	1.09	1.27
Casting rate (mm)		
after 30'	3.4	3.3
after 60'	6.4	6.2
after 90'	7.2	9.1
Moisture content (%)		
after 30'	19.5	20.2
after 60'	20.3	20.6
after 90'	20.4	20.8
Shrinkage after drying (%)	3.8	4.0
Rupture after drying (N/mm ²)	5.77	7.0
Shrinkage after firing (%)	6.7	7.0
Rupture after firing (N/mm ²)	41.1	41.6
Water absorption (%)	1.5	1.1
Thermal expansion (K ⁻¹ , 20 to 500 °C)	-	5.65 x 10 ⁻⁶
Firing range (°C)	1 230-1 260	1 230-1 260

B. Glazes

<u>Composition (%)</u>	<u>SG4/SG5</u>
Mistress silica	27.0
Chimbaranga feldspar	35.0
CaCO ₃	19.0
ZT kaolin	6.0
ZnO	3.0
SnO ₂	10.0

SnO_2 can be prepared from tin-processing flue dust (SG4) or from metallic Sn (SG5)

Residue above 53 μm	0.1%
Litre weight (g/l)	1 560-1 580
Firing range ($^{\circ}\text{C}$)	1 230-1 260
Autoclave test (at 3.5 bar/2 hours)	Good
Thermal expansion (K^{-1} , 20 to 500 $^{\circ}\text{C}$)	6.15×10^{-6}

Annex VI

PROPERTIES OF BODIES AND GLAZES FOR PORCELAIN ELECTRIC INSULATORS

A. Porcelain electric insulator bodies

<u>Composition (%)</u>	<u>P1</u>	<u>P2</u>	<u>P3</u>	<u>P4</u>
ZI kaolin	25.0	25.0	25.0	25.0
Beitbridge ball clay	25.0	25.0	25.0	25.0
Mistress feldspar	30.0	30.0	30.0	30.0
Mistress silica	20.0	10.0	-	-
Chiredzi clay	-	10.0	30.0	-
Fired kyanite	-	-	-	20.0
Residue above 75 μm (%)	2.5	1.5	1.7	1.5
Moisture content at extrusion (%)	22.8	23.3	22.9	23.0
Pfefferkorn number	24.9	28.5	26.5	-
Shrinkage after drying at 110 °C (%)	4.0	4.9	4.8	4.0
Rupture after drying (N/mm^2)	2.2	2.1	2.5	1.3
Shrinkage after firing to 1,300 °C (%)	12.6	13.3	13.9	12.5
Rupture after firing to 1,300 °C (N/mm^2)	70.0	56.5	50.4	68.1
Water absorption after firing to 1,300 °C (%)	0.0	0.0	0.0	0.0
Bulk density (g/cm^3) after firing to 1,300 °C	2.28	2.25	2.35	2.40
Thermal expansion 20-600 °C ($10^{-6} \times \text{K}^{-1}$)	5.59	4.96	5.32	5.43
Firing range (°C)	1 290- 1 320	1 290- 1 320	1 300- 1 350	1 300- 1 400

B. Porcelain electric insulator glazes

White glaze composition (%)

Mistress silica	37.0
P1 biscuit (1,000 °C)	16.5
Dolomite	11.5
Calcite	3.0
Chimbaranga feldspar	23.5
ZI kaolin	5.0
ZnO	1.0
SnO ₂	2.5
Residue above 53 μm (%)	max. 0.1
Litre weight (g/l)	1 450

Brown glaze composition (%)

Mistress silica	30.0
P1 fired porcelain (1,320 °C)	18.0
P1 biscuit (1,000 °C)	17.0
Dolomite	10.0
Chimbaranga feldspar	10.0
ZI kaolin	5.0
Cr-Mn spinal	10.0
Residue above 53 μm (%)	max. 0.1
Litre weight (g/l)	1 450

Annex VII

PROPERTIES OF PORCELAIN BODIES FIRED TO DIFFERENT TEMPERATURES

<u>Shrinkage (%)</u>	<u>P1</u>	<u>P2</u>	<u>P3</u>	<u>P4</u>
1 250 °C	12.6	13.4	13.0	11.9
1 300 °C	12.6	14.4	14.8	12.5
1 350 °C	11.6	12.8	13.6	12.4
1 400 °C	10.7	12.2	12.2	12.2
<u>Rupture (N/mm²)</u>				
1 250 °C	62.0	51.6	54.7	60.5
1 300 °C	70.0	56.5	50.4	68.1
1 350 °C	60.0	49.1	51.3	77.7
1 400 °C	44.6	47.5	50.1	77.4
<u>Water absorption (%)</u>				
1 250 °C	1.0	1.1	1.4	1.5
1 300 °C	0.0	0.0	0.0	0.0
1 350 °C	0.2	1.4	0.7	0.0
1 400 °C	5.0	4.1	1.5	0.0
<u>Bulk density (g/cm³)</u>				
1 250 °C	2.31	2.20	2.31	2.35
1 300 °C	2.26	2.19	2.20	2.41
1 350 °C	2.07	2.06	2.35	2.36
1 400 °C	2.11	2.06	2.36	2.31

Firing programme: (a) To 510 °C: 1.5 °C/min; soaking time: 15 min;
(b) To top temperature: 3.0 °C/min; soaking time: 30 min.

Annex VIII

PROPERTIES OF WALL-TILE BODIES

<u>Composition (%)</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>
Mt. Hampden brick clay	66.0	65.0	65.0
Rwange fire clay	12.0	15.0	12.0
Beitbridge ball clay	11.0	-	-
Limestone	7.7	7.0	8.0
Mistress silica	3.3	3.0	5.0
Biscuit	-	10.0	10.0
 <u>Results</u>			
Residue above 200 µm (%)	0.09	0.09	0.10
Litre-weight of the slip (g/l)	1 419	1 435	1 430
Moisture content at extrusion (%)	23.0	21.8	26.2
Shrinkage after drying at 110 °C (%)	7.68	5.98	6.1
Rupture after drying (N/mm ²)	7.63	6.10	3.0
Shrinkage after firing to 1,000 °C (%)	8.00	6.40	6.7
Rupture after firing (N/mm ²)	19.16	17.97	10.1
Thermal expansion coefficient 20-500 °C (10 ⁻⁶ x K ⁻¹)	6.9	6.5	-
Water absorption after firing (%)	17.81	17.6	-

Annex IX

PROPERTIES OF WALL-TILE BODIES FIRED TO DIFFERENT TEMPERATURES

		<u>T2</u>	<u>T3</u>
Moisture content at extrusion (%)		22.3	26.3
Shrinkage after firing (%)	900 °C	-	-
	950 °C	-	-
	1 000 °C	6.7	6.7
	1 050 °C	5.2	6.2
Rupture after firing (N/mm ²)	900 °C	-	-
	950 °C	13.0	9.5
	1 000 °C	14.8	10.0
	1 050 °C	11.5	9.2
Water absorption after firing (%)	900 °C	-	-
	950 °C	23.5	27.3
	1 000 °C	2.0	26.3
	1 050 °C	19.9	-
Bulk density after firing (g/cm ³)	900 °C	-	-
	950 °C	1.67	1.59
	1 000 °C	1.68	1.71
	1 050 °C	1.71	1.78

Firing programme: (a) To 510 °C: 1.5 °C/min; soaking time: 15 min;
(b) To top temperature: 3.0 °C/min; soaking time: 30 min.

Annex X

PROPERTIES OF ZIMBABWEAN WALL-TILE BODIES AND GLAZE

<u>Wall tiles</u>	<u>Old tiles</u>	<u>New tiles</u>
Moisture content at extrusion (%)	23.7	22.0
Shrinkage after drying at 110 °C (%)	5.4	4.9
Rupture after drying (N/mm ²)	3.6	1.9
Shrinkage after firing to 1,150 °C (%)	7.1	13.0
Rupture after firing (N/mm ²)	28.2	46.1
Water absorption after firing (%)	20.1	7.6
Thermal expansion coefficient 20-500 °C ($10^{-6} \times K^{-1}$)	7.11	6.23
Biscuit firing temperature in the factory (°C)	1 120	1 120
<u>Tile glaze (imported)</u>		
Thermal expansion coefficient 20-500 °C ($10^{-6} \times K^{-1}$)	5.63	
Glaze firing temperature (°C)	1 040	
Autoclave test (3.5 bar/2 hours):	Bad	

Annex XI

PROPERTIES OF FRITS GLAZES

1. Frit

Composition (%)

Borax	33.2
ZnO	1.0
SnO ₂	11.8
Chimbaranga feldspar	25.5
Mistress silica	28.5

Estimated firing temperature (°C) 1 330-1 360

2. Glaze

Composition (%)

Frit	94.0
ZT kaolin	6.0

Residue above 53 µm (%) 0.0
Litre-weight (g/l) 1 560
Thermal expansion coefficient, 20-600 °C
(10⁻⁶ x K⁻¹) 8.44
Glaze firing temperature (°C) 960

Annex XII

ADDITIONAL EQUIPMENT FOR THE PILOT PLANT

A. Equipment to be provided by the project

- 1 oil- or gas-heated kiln (up to 1,400 °C, 1 m³)
- 1 kiln (up to 1,600 °C, 0.5 m³)
- 1 kiln (up to 1,500 °C, 0.25 m³)
- 1 oil- or gas-heated small rotary kiln (up to 1,600 °C)
- 1 drier, 1 m³
- 3 hydrocyclones, 50, 150 and 350 mm diameter
- 1 jet spray drier
- 1 magnetic separator
- 1 edge runner mill
- 1 rotating machine for the shaping of insulators
- 1 turning table for hand shaping
- 1 semi-industrial glazing device for spraying and waterfall application
- 1 device for electrical resistance measurements of low- and high-tension insulators
- 1 device for measurements of thermal conductivity of ceramics

B. Equipment to be produced locally

- 1 screen (BS240), aperture 0.63 mm
- 1 plastic container (200 l) for blunging
- 1 plastic container (200 l) for slow mixing of body slip
- 1 wooden mixer, 30-35 rev/min
- 4 wooden plates (50 x 50 cm) for filter press cakes and extruded blocks
- 1 connection of slow-mixing drum to filter press

Annex XIII

SPARE PARTS FOR THE PILOT PLANT EQUIPMENT

For filter press (William Boulton Ltd., serial No. FS 9754):

4 drive belts;
1 set of spare filter cloth (polypropylene outers and nylon inners);
1 ceramic pump component with packing.

For vacuum extruder (Edwards and Jones Ltd., type 37.B):

Extrusion dies, 7, 12, 20, 38 and 45 mm diameter;
Extrusion bits, one complete set of various shapes and sizes to suit
the above dies.

For ball mill (H. Welte, jug type B6AK/SE No. 84044 - 1984):

Drive belts;
1 set ceramic lining;
100 kg ceramic milling balls each, 35 and 45 mm diameter.

Annex XIV

SAMPLE LOG SHEET g/

SAMPLE DESCRIPTION

ZTK, pink, slightly plastic clay

SAMPLE SOURCE (AREA/DISTRICT)

SAMPLE LOC NUMBER

SAMPLE ENTERED ON

WORK COMPLETED ON

1.1 Spectrography

1.2 Chemical Analysis (%)

pH = 8.5

Solu- ble Salts	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	CaO	MgO	F ₂ O	Na ₂ O	LOI	SO ₃	P ₂ O ₅
0.75	43.37	36.88	3.97	1.07	0.14	0.23	tr	1.15	13.53		

1.3 Rational Analysis

	CONVENTION	
	KAOLINITE	FELDSPAR
Kaolinite	94	36
Feldspar	0	10
Silica	0	1
Illite		
Montmorillonite		

1.4 Particle size analysis (Below 60 μm)

Particle size (μm)	0-9	9-13	13-19	19-28	28-37	37-75	+75
Z	89.26	3.10	3.35	2.05	1.30	0.85	0.1

1.5 MINERALOGICAL ANALYSIS

A. Thermal Analysis

(i) DTA

Reaction Peak Temperature/ °C	Reaction Type	Peak Shape (Intensity)	Remarks
540	N	S(I)	Kaolinite (dehydroxylation) Kaolinite (mullitization)
960	X	S(I)	

(ii) TGA

Temperature range/°C	Mass Change (%)	Remarks
50 - 200	- 1	drying
440 - 710	- 11	Kaolinite (80%) (dehydroxylation)

(iii) Dilatometric Behaviour

Temperature/°C	Remarks

B.

YPD

C. Optical Methods

2.1 PHYSICAL PROPERTIES
Dilatometric Behaviour

Test piece prefiring temperature ($^{\circ}\text{C}$)	Temperature range	(K^{-1})
1100	20 - 300	4.48×10^{-6}
	20 - 500	4.45×10^{-6}
	20 - 800	4.58×10^{-6}

- water vapour adsorption C.20%
(above sat. NaCl /48 hrs)
- particle size analysis (up to 200 μm)

Sieve aperture (um)	+20)	180-200	160-180	125-160	100-125	-100
%	-	-	-	-	-	100


2.2 RHEOLOGICAL TESTS

(i) Qualitative deflocculation tests

<u>Deflocculant</u>	<u>Remarks</u>
3% Na_2CO_3	poor
Na_2SiO_5 (1.15)	fair
15% Sodium tannate	good

(ii) Lehmann Viscometer

Slip density (g/l)	<u>1384</u>
Deflocculant	<u>Sodium tannate</u>
Temperature (slip)	<u>Room temp $^{\circ}\text{C}$</u>

Time/min	Vol electrolyte/cm ³	Flow Time/Sec	
0	0	102.80	
	1	16.15	
	2	15.70	
	3	15.25	
	4	15.50	
	6	15.70	
	8	15.50	
	30	3	18.00

Thixotropic index (TC) $\frac{18.00}{15.50} = \underline{1.18}$

Remarks : Suitable for casting

Brookfields Viscometer

Slip density (g/l) 1384
 Deflocculant Sodium tannate
 Temperature 24°C

Spindle # 4

<u>Shear rate/RPM</u>	<u>Viscosity/Poise</u>
100	16
50	28
20	59
10	80

Spindle

RPM 100
 Number 1

Vol. of slip 475 ml

Time/min	Vol. electrolyte/cm ³	Apparent Viscosity/Poise
0 ↓ 30	0	16.00
	1	0.39
	2	0.35
	3	0.35
	4	0.36
	5	0.36
	0	16.20

Remarks : Plastic rheological behaviour. Shear thinning.
 Non-thixotropic. Sodium tannate is a very effective deflocculant for ZTK.

2.3 Casting Rate*

Temperature

Room temperature

Time/min	Wall thickness/mm	Moisture (%)
30	4.6	27.36
60	7.0	28.43
90		

* Slip density = 1573 g/l

2.4 Plasticity (Pfefferkon)

Moisture content (Z)	Index E_0/H
15.7	1.1
23.5	3.3
29.4	4.2

This clay had a very good plasticity.

2.5 UNFIRED PROPERTIES

Visual colour	<u>Pinkish</u>
Colour co-ordinates	<u> </u>
Extrusion moisture content	<u>29.4%</u>
Critical moisture content	<u>22.0%</u>
Linear drying shrinkage	<u>5.4%</u>
MOR (material dried 110°/1 hr)	<u>1.5 N/mm²</u>

2.6 FIRED PROPERTIES

FIRING PROGRAM

Initial temperature	=	Room temperature
Initial heating rate	=	1.5°C/min
First holding temperature	=	510°C
Dwell time at 510°C	=	15 min
Second heating rate	=	2°C/min
Temperature maxing	=	1000°C/1100°C/1200°C/1300°C
Dwell time at max. temp	=	30 min

PROPERTIES	Firing Temperature / °C			
	1 000	1 100	1 200	1 300
Linear shrinkage (%)	4.3	5.0	10.1	13.8
Total shrinkage (%)	9.7	10.4	15.5	19.2
Water absorption (%)	32.2	29.3	18.2	9.7
Bulk density (g/cm ³)	2.0	2.5	2.1	2.4
MOR (N/mm ²) -	4.0	4.3	11.8	14.0
Visual colour	Pinkish	Cream	Creamish white	
Colour co-ordinates				

3.0 Remarks

China clay (kaolin)

4.0 Suggested Uses

Sanitary ware, procelain insulators (technical porcelain), glazes, tableware, tiles with white body and in refractories.

Annex XV

CURRICULUM OF TRAINING IN CERAMIC TECHNOLOGY

A. Raw materials and body preparation for fine ceramics

Main groups of fine ceramic products and their properties;

The important technological properties of china clays, ball clays, brick clays, feldspars and silica;

The aims and ways of body preparation:

- (a) Preparation of plastic raw materials;
- (b) Preparation of non-plastic raw materials;
- (c) De-watering of the slips;

Equipment for the preparation of:

- (a) Non-plastic raw materials;
- (b) Plastic raw materials.

B. Technology for sanitary ware production

Definition of sanitary ware;

The main steps in manufacturing:

- (a) Mould making;
- (b) Recipes;
- (c) Slip preparation;
- (d) Casting and casting systems;
- (e) Drying and drying systems;
- (f) Glazes and glaze application;
- (g) Firing and various kiln types.

C. Technology for porcelain production

Definition of porcelain;

The main steps in manufacturing:

- (a) Recipes for body formulation and preparation;
- (b) Various shaping systems and their fields of application;
- (c) Drying and drying systems;
- (d) Glazing;
- (e) Technology of firing;
- (f) Main physical and electrical testing methods.

Annex XVI

ADDITIONAL LABORATORY EQUIPMENT

- 1 XRD apparatus to complete the equipment for mineralogical analysis
- 1 Andreasen apparatus, for grain size analysis of clays between 0-100 μm
- 1 apparatus for fast moisture-content measurement
- 1 Leitz high-temperature microscope, up to 1,600 °C, to test the melting behaviour of feldspar, glazes and frits
- 1 mercury porosimeter to measure the porosity of ceramic bodies
- 1 laboratory hydraulic press (IMM) to prepare test pieces
- 1 high-temperature laboratory furnace (Naber) for refractories (up to 1,700 °C)
- 1 laboratory sieving machine (Retch) with laboratory ball-mill, to prepare samples (XRD, DTA, TG, dilatometer etc.)
- 1 hot-load testing furnace, for determination of rupture under load (RuL) and creep in compression (CiC) of refractories
- 1 sample drilling and one precision grinding machine, for the preparation of samples for RuL and CiC determination
- 1 special kiln (Netzsch), for pyrometric cone equivalent determination