

CTT TYPE 8 OXIDE MILLS

INTRODUCTION

The CTT Type 8 Oxide Mill has been designed to give a nominal output of 50 tonnes per week (300 kg/hr) of consistent quality oxide at 65% oxidisation level.

The Mill is driven by a 37kW motor driving a reduction gearbox through a belt drive, this in turn drives a pinion and ring gear system to give a final Mill speed of 27 turns per minute. As the drum rotates the lead pig sections chopped from full size 42kg lead pigs cascade inside the Mill and the lead is oxidised by an exothermic reaction.

The drum is enclosed in a steel shell; the upper half is fitted with a flanged hood to which is attached ducting and a cooling fan. The Mill drum temperature is automatically controlled using an infrared non-contact pyrometer which will monitor the temperature and the readings will be processed by the PLC control system. The cooling of the Mill drum is achieved by a two-stage water cooling system, the first stage is normal process cooling and the second is an emergency water flow rate.

The plant is semi-automatic in operation and is most suited for continuous operation.

When running in a steady state condition the installation requires little attention apart from the loading of lead pigs to the pig feed storage conveyor, product quality checks should be taken with hourly checks of the Pbo content and HMI trend readings.

The plant requires little maintenance apart from scheduled of plant items.

Equipment is provided for the automatic chopping and feeding of lead pigs to the Mill where it is then ground by an attrition process into fine particles and oxidised by the Mill temperature and amount of air drawn into the system. As the oxidised powder reaches the correct level of oxidisation its weight is reduced allowing it to be drawn off via a ducting system into a dust filter. Oxidised particles which are less oxidised and therefore too heavy will not be able to rise out of the Mill and will fall back down the outlet ducting and returned to the Mill by the oversized return conveyor.

A dual air filter exhaust system is fitted to ensure safe working conditions. The oxidised lead powder is collected by the dust filter before passing through a rotary airlock and discharged via the vacuum conveying system where it will be sent to the selected storage Silo.

The electrical controls are split across two control panels, one panel designated the motor control centre (MCC) is for the motor drive units and is positioned within the main Mill room and allows all Mill and conveying motors to be selected in Hand/off/Auto mode.

In hand mode, the motors can be controlled directly from the MCC manually, if selected to auto then they can be operated from the ICA control unit using the HMI in the Mill control room in either fully automatic mode or as a manual operation. All the motors controlled from the MCC are protected by emergency stop functions with push button stations positioned on the



Mill itself, if any push buttons are operated then the appropriate section of the MCC will require resetting using the MCC mounted reset facility.

All the control circuits are from the ICA control panel located in the Mill control room.

CTT TYPE 8 OXIDE MILL - INSTALLATION AND START-UP

LIST OF CONTENTS

	<u>PAGE</u>
• FOUNDATIONS	3
HOLDING DOWN BOLT DETAILS	4
PLANT ITEMS	4
• SERVICES	
 WATER COMPRESSED AIR ELECTRICAL ITEMS DUCTING 	5 5 6 7
TESTING BEFORE START-UP	8



INSTALLATION

FOUNDATIONS

The foundations for the whole plant are to be in compliance with the loadings and the layout shown on drawing No. OMP 001 REV C.

Before the installation of the plant is commenced, the building and foundations for the Mill must be completely finished and checked, ensuring that the floor is level and has a good surface for packing and setting the equipment.

The Mill must be mounted on the TICO anti vibration mounting pads as supplied.

Care should be made to the correct positions of the drainage system with special attention being made to the trough falls.



HOLDING DOWN BOLT DETAILS - TYPE 8 OXIDE MILL

EQUIPMENT	BOLT TYPE	<u>QTY</u>	SUPPLIER
Pig feed	M12 x 150 HSA Stud Anchor	4	Client
Pig storage conveyor	M12 x 150 HSA Stud Anchor	14	Client
Mill base	M10 x 140 HSA Stud Anchor	10	Client
Dust filter	Fitted onto platform	As required	Client
Absolute filter	Fitted onto platform	As required	Client
Exhaust fan	Fitted onto platform	As required	Client
Control panels	M10 x 108 HSA Stud Anchor	4	Client

PLANT ITEMS

The main items of plant should be positioned in accordance with the general arrangement drawings;

EP 01 001 B, Provisional Layout Mill Area.

EP 01 005 A. Plant layout showing filters, absolute filters and fan sets.



SERVICES

Water

A supply of clean water is required for cooling purposes at the following points: -

Mill Cooling Water (control water)

A minimum flow rate of 180 litres per hour is required to the Flowrator on the Mill water control panel.

Mill Cooling Water (emergency water)

An emergency supply of cooling water with a guaranteed minimum flow rate of at least 250 litres/hr is controlled by a solenoid valve connected to mains water supply with a manual isolating valve upstream of the solenoid. The isolating valve should be such that it cannot be used except by direct action.

Both the control and emergency water solenoid valves are mounted within an enclosure on the Mill body, the flow adjustment device for the control cooling water is mounted externally to this enclosure.

Care should be taken that the water is clean and no impurities are allowed to enter the system which may cause the solenoids to remain open and cause uncontrolled cooling of the Mill. If this occurs then instability in operation will occur with reducing Mill temperature and oxidisation.

Miscellaneous

Provide hosing down points close to the Mill plant for cleaning purposes.

Compressed Air

A minimum supply of 620 litres/min of dry air at a line pressure of $4.0 - 6.0 \text{ Kg/cm}^2$ (57 - 85 psi) is required for operating the dust filter and cooling the temperature measurement pyrometer.

The air supply line to the dust filter and pyrometer are to be fitted with their respective filter and pressure regulator.



ELECTRICAL WIRING AND INSTRUMENT CONNECTIONS

Electrical equipment remote from the panel should be connected to the appropriate outgoing terminals as indicated on the electrical schematic diagram. These connections are all motors supplies from the main MCC and all instrumentation and control circuits from the ICA control panel mounted within the Mill control room.

The seller supplies all the necessary cabling and cable support systems to carry out the installation.

The electrical main supply is to be a TP-N-S system as defined by the UK electrical regulations and is to be connected direct to the shrouded side of main isolator with the neutral conductor connected to the main neutral terminal block. All earth connections are to be made at the common earth bar.

The supply cable size is to be calculated by the client using the loadings supplied by the seller which shall be 160kW of connected load and no diversity should be supplied.

The expected power factor is estimated to be 0.88 lagging and the client is advised to allow for this on the entire plant loading, no harmonics are expected to be made during the plants operation which would require harmonic correction.

Provision is made in the MCC panel for a Mill emergency stop operation, the opening of the pig feed guards will only stop the pig feed operation.

On an emergency stop or Mill guard opening the appropriate reset button will need to be operated.

Drawings series numbers C14765 1 - 26 shows location of all connection points in the motor control centre (MCC) and drawing series numbers C14765 sheets 101 - 116 are for the Mill ICA Control panel.

The control panels are both top entry and therefore the termination gland plates shall be removed prior to any cutting of holes for cable entry. Serious damage to the internal electrical components can occur if this is not carried out.

The control panels must be mounted on the TICO anti vibration mounting pads as supplied.

The signal cabling from the Infrared pyrometer and dust filter thermocouples are to be run separate from other electrical systems in metal conduits to eliminate EMF interference.

Wiring details to the Mill are to be as per the schedule in appendix 5.



DUCTING TO ATMOSPHERE

Two exhaust ducts from each Mill emit to atmosphere, one from the absolute filter and the other from the Mill cooling duct.

The two ducts are to be connected to final extraction system provided by the client.



TESTING BEFORE START-UP

The following tests are to be carried out by the Commissioning Engineer before lead is loaded into the system: -

- Thorough inspection of all plant items.
- Check that voltage and current ratings are correct, all gearboxes contain the correct oil and quantity of oil, all lubricating points are greased or oiled as specified.
- Test each motor in turn by manually switching on and off quickly. When the correct direction of rotation has been obtained the motors can be run for longer periods.
- When the Mill motor direction has been checked (the Mill should rotate in a clockwise direction when viewed from the feed inlet) run the Mill for a short time and check that the gears are fully lubricated.
- Before switching on the main exhaust fan, fully close the main slide valve as the motor is sized to drive the fan when the air is at working temperature. When the air is at ambient temperature the motor could be overloaded on start up.
- The cooling water system can be checked by adjusting the temperature set point above and below the control and alarm temperatures. When the indicated temperature is above the control set point a flow of cooling water of at least 180 litres/hr should be available, when above the set alarm temperature, it should be possible to obtain an emergency cooling water flow of at least 250 litres/hr. The cooling water must spray onto the Mill from all of the water jets. Also check that the emergency water system and high temperature alarm operate simultaneously.
- Run the plant continuously for 8 hours to ensure that all items are functioning correctly.
- Check the air flow ventilation system from the oxide Mill.
- Test all pneumatic equipment for correct functioning and check air flow. The automatic dust filter air pressure regulator should be adjusted to give 6.0 kg/cm² (85 psi) whilst the pyrometer pressure regulator is pre-set at 1.4 kg/cm² (20 psi).



CTT TYPE 8 OXIDE MILL INSTALLATION - OPERATING INSTRUCTIONS

LIST OF CONTENTS

			<u>PAGE</u>	
•	START-UP PROCEDURE AND SCREEN FUNCTIONS		10 - 22	
•	NORMAL RUNNING PROCEDURE		22	
•	NORMAL SHUT-DOWN PROCEDURE		24	
•	EMERGENCY SHUT-DOWN PROCEDURE		25	
•	CONTROL OF PRODUCT		26 - 28	
•	RUNNING FAULTS AND REMEDIES		29 - 31	
•	MILL NORMAL CHARGE LEVEL		APPENDIX 1	
•	LUBRICATION SCHEDULE		APPENDIX 2	
•	LEAD CHOPPING TIMER SETTING		APPENDIX 3	
•	LEAD PIG FEED CALCULATIONS	APPE	NDIX 4	
•	ELECTRICAL INSTALLATION SCHEDULE		APPENDIX 5	
•	Pbo DETERMINATION GUIDE		APPENDIX 6	



START-UP PROCEDURE

Both the Oxide Mills and conveying system can be started manually or automatically using either the hand/off/auto functions on the MCC or by the HMI mounted on the ICA panel in the Mill control room.

In normal operation the controls on the MCC should all be in the "Auto" position and all control functions should be from the ICA panel.

All items can be selected on the HMI for "Auto Control" or "HMI Control", in normal operation all items should be selected for "Auto Control" and this function is automatically set after a cold start to ensure no item is not left in "HMI Control" after a manual operation.

A facility for changing the screen language is available by selecting the appropriate "Flag" on the screen, this will change all the screens text.

A Mill cannot be started unless the clients conveying system is running and is indicated by a change of status on the main overview screen.

A detail of the screens is explained on the following pages.



THE MAIN OVERVIEW SCREEN



This screen is the initial screen after a cold control panel start and can be accessed from all the screens available, this screen is designed to be present on the system during normal operation of the plant as it gives an overview of the Mills and conveying system status.

It will show the Mill status for Mill sequence during start-up, Mill current, Mill temperature, dust filter temperature, amount of pigs processed, pig feed sequence status, extract to silos status using the vacuum conveying system, clients conveying system status and alarm indication.

From this screen each Mill and the conveying system can be accessed for additional information using the navigation buttons.



THE CONVEYING OVERVIEW SCREEN



This screen is for status indication and for the manual control or any item. To select an item for manual control is by touch contact on the item and then switching to "HMI control". Once HMI control is selected the option for open / close, start / stop is available. Please note that it is required to revert to "Auto Control" after finishing with any "Manual" operation.

This screen will show the status of the oxide transfer system from the Mill outlet rotary valves into vacuum conveying system, items detailed are;

- Rotary valves
- Air inlet valve position
- Wrapper solenoid
- Vacuum pump
- Differential filter pressure
- Clients conveying system

All motors detailed will show a change of status for when unavailable (Yellow), stopped or closed (Red) or running or open (Green), the status is also available as a text status.





THE MILL SCREEN

This screen will give an overview of the Mill and also allow any manual operation to be carried out, it will also detail the Mill operating conditions and running sequence status which are mirrored on the main overview screen.

This screen also gives the details of the pig feed timers for high and low rate feeding of the lead pigs into the Mill, it will also show the status of the clients conveying system which is a prerequisite for operation of the Mill.

From this screen there is access to the "Filter Screen" and "Pig Feed" screen to allow for monitoring or manual control.

To start or stop a Mill has to be carried out from this screen, selecting "Start Mill" will start the Mill in a full auto start sequence, selecting "Stop Mill" will stop the Mill in a full auto stop sequence. Selecting any motor or valve can place the item in a manual mode.

On selecting "Mill Start" the status should be watched until the "Mill Stable" status is shown indicating the Mill has started correctly and is operating correctly.



If any item is left in a manual operation control then an automatic start will stop when trying to automatically start an item that has been left in manual control.

All alarm conditions are detailed on this screen.



PIG FEED SCREEN

The screen for the pig feed will show the status of all the items operating within the pig feed and is accessed from the Mill screen, these are;

- Pig flap sensor
- Pig pusher position
- Bed position
- Blade position
- Hydraulic pump operation

All the above are indicated by text and colour as to the operation status.

A facility to place the pig feed in the "Home" position is available which should be used whenever the plant may have stopped in an emergency shutdown or power failure, this will place all items in the home position.



All items can also be placed in a manual operation by selecting the item by touch and then entering the "HMI Control" which will place the item in manual operation.

All alarm conditions are detailed on this screen.

SIEMENS 12/31/2000:10:59:397 Mill 4 Filter Overvieu Filte l ách á from Mill 4 Extract to Si Start Mill Stop Mill Entract to Sil Back Mill:40 MillS MELLE Setpoints Logi

MILL FILTER SCREEN

This screen details the operation of the Mill filter and will allow for manual operation of motors and valves associated with the filter and is accessed from the Mill screen.

To start or stop a Mill can be carried out from this screen if required but its recommended to use the "Mill Screen". If required to start the Mill here selecting "Start Mill" will start the Mill in a full auto start sequence, selecting "Stop Mill" will stop the Mill in a full auto stop sequence.

All items can also be placed in a manual operation by selecting the item by touch and then entering the "HMI Control" which will place the item in manual operation.

On selecting "Mill Start" the status should be watched until the Mill Stable status is shown indicating the Mill has started correctly and is operating correctly.



All alarm conditions are detailed on this screen.

MANUAL SELECTION OF ITEMS FROM ANY SCREEN





This item shows a the status of any pneumatic or hydraulic item within the pig feed unit.

Status text for "Extend or Retract" "Raised or Lower" with indicators against each item.

H indicates the item is in manual control.



Advises this item is interlocked with other items of plant.



MANUAL CONTROLS

CAR TO BE TAKEN WHEN CARRYING OUT MANUAL CONTROL OPERATIONS AS MOST ITEMS ARE INTERLOCKED WITH OTHER ITEMS AND DAMAGE MAY OCCUR IF OPERATED INCORRECTLY.

This screen will detail the manual operation of the pig feed bed for raise and lower. This is typical for all non-motor operated items whether raise or lower, extend or retract, open or close.

The screen is in auto control in this description and to enter manual select "Select HMI Control" this box will then change to "Select Auto Control" With this indicated you are now in manual control and can operate any item manually.

Should any item be in alarm mode it can be reset here.

Z #	Mill 4 -	Bed	LIACODE	
		1		
	Select HML			
	Raise Be	4 		
	Lover B			
	Reset Bed A	larm		
Back Alar	na Sull'4 Mull S Mull	6 Estrac(lo Silo Setp	eints	Login



This screen will detail the manual operation of a motor driven drive. This is typical for all motor operated items.

The screen is in auto control in this description and to enter manual select "Select HMI Control" this box will then change to "Select Auto Control" With this indicated you are now in manual control and can operate any item manually.

Should any item be in alarm mode it can be software reset here, har resets must be in the MCC.

Hours run indicators are shown for use by the maintenance departments.

					S PANE
7 533	Mill 4 - Mill	Drive Control	Page	112/01/00/07/07/07/07/04 00/01/07/07/07/07/07/07/07/07/07/07/07/07/07/	200
	2 	Ret Poter Alars			-
Herrs Rim Count	568.4	NIS	X00 Setpoints	Lege	

ADJUSTABLE SETPOINTS



(<u></u>		
Overview		
		Yill Average Current - Number of Samples Samples taken every 10 milliseconds
		Current - Give 🗰 👔 Current-Average
		Mill Current Setpoint 000.00 A
		Low Current High Rate Feed Timer
		High Current * Low Rate reed Timer 00000 ;scs
		Pig Chop Interval Timer 0000 ;ecs
		Mill Cooldown Temperature
		Emergency Cooling Temperature 000.00 Deg C
		Hopper Yibrator Off Time 0000 secs
		Homer Vibrator On Time
		Destine III des tights DIV Carded
		Moli Sbell' Lemperatore: Setpoint:
		Mill Shell Temperature PID Controller- Gain 000.0
		Mill Shell (temperature PH) Controller - Ti 000.0 secs
	11.000.000	
1,1,1,1559 12:00:00 14	· · · · · · · · · · · · · · · · · · ·	
1,1,1000-12,00:00-04	Manage Text . Manage	e fest oo steaarje fest oo steaarje fest oo steaarje fest.
Rack	Alaems	MGILA MGILS MGILE Extract to Sile Saturints

This screen is for all the adjustable setpoints for operating the Mill. To adjust any setpoint tapping the setting will bring up a setting screen where the new setting can be entered.

All settings on this screen are set during commissioning and it should not be necessary to adjust them during normal operation.

The alarms are accessible on this screen as well are returning back to other screens.



TRENDS



Trends are available for viewing for Mill motor current, Mill temperature, Filter temperature, Filter differential pressure, air damper position.

When Pbo checks are made it is advisable to note the Mill current and temperature.

To access any trend this is made by touch on the present reading on the HMI

To access the Mill current trend, touch the section for current on the Mill screen or overview screen.

To access the Mill temperature trend, touch the section for temperature on the Mill screen or overview screen.

The same method is also used for the trend for the filter temperature, differential pressure and air damper position.





1. <u>START-UP PROCEDURE</u>

- Apply drops of oil on the felt rubbing pad; also, at a minimum 8-hour intervals to avoid oxide leaks.
- Ensure main outlet air valve is closed, this is controlled by a set pot on the ICA control panel and should be set to "0" upon Mill start-up.
- Ensure the compressed air and water are turned on.
- All emergency stops are unlocked, pig feed doors are shut and emergency stop circuit resets.
- Lead pigs are loaded onto the storage conveyor.
- Load controller operating amps is set correctly on the HMI parameter set point page set for the required loading. Note that once set it should be the same for all future operating conditions.
- Check the control water and emergency water set points on the temperature controller. Note that once set it should be the same for all future operating conditions
- Ensure that all personnel are clear of the machinery.

The plant can now be started either by automatic start-up using a predefined system built into the PLC operating system or by manually starting each item in hand control. The former is recommended for normal operation and will follow the operational sequence as below;

- Ensure all items are selected in auto mode.
- Ensure clients conveying system is running.
- Press Mill start from the Mill screen or conveying screen.

The system can be started if required in manual but must be carried out in a correct sequence, this must be as follows and note that any items left selected in automatic will automatically start if are in the sequence normally done during an automatic start.



If the plant must be operated in manual then select the required plant items and start as follows:

- Ensure all items are selected in auto mode.
- Ensure clients conveying system is running.
- Vacuum pump in manual, start vacuum pump
- Check the main air inlet valve to the conveying system is open, this valve is powered • to close when the vacuum pump is not running.
- Vacuum pump in auto, proceed to oxide collector dust filter rotary valve start. •
- Dust collector rotary valve in manual, start rotary valve.
- Dust collector rotary valve in auto, proceed to Mill filter rotary valve start. •
- Mill filter rotary valve in manual, start filter rotary valve •
- Mill filter rotary valve in auto, proceed to exhaust fan start.
- Mill exhaust fan in manual, start exhaust fan.
- Mill exhaust fan in auto, proceed to oversize return conveyor start. •
- Oversize return conveyor in manual, start oversize return conveyor. •
- Oversize return conveyor in auto, proceed to Mill start.
- Mill motor in manual, start Mill motor •
- Mill motor in auto, proceed to cooling fan start •
- Cooling fan in manual, start cooling fan. (Note this operates in conjunction with cooling • water)
- Cooling fan in auto, proceed to billet conveyor start. •
- Select the pig conveyor for home position •
- Billet conveyor in manual, start billet conveyor •
- Billet conveyor in auto or manual operation check pig feed function •

Operation of the pig feed in manual is carried out as follows from a home position of the pig feed:

Run billet conveyor to place a pig against the pig flap position and align the pig Raise the bed

- Extend the pig pusher to push the pig to the cut position
 Lower the blade
- 3. Raise the blade
- 4. Repeat 1-3 until the pig is completely cut.
- 5. Lower the bed
- 6. Retract the pig pusher

The cooling water and emergency water valves should never be selected for manual operation except for testing purposes



The dust filter pulse timer is automatically started along with the wrapper solenoids when the exhaust fan is running.

Once the plant is in running the system shall be brought on line and to a stable operating condition by careful increasing of the airflow using the positioning pot adjustment device located on the ICA panel below the HMI.

Allow Mill drum to attain working temperature, (this figure will be determined by the Commissioning Engineer). When the Mill temperature is reached after approximately 1 1/4 hours) open the main valve gradually and set its position.

The lead feed timers for both the high rate feed and low rate feed will be set during commissioning to attain the correct loading of the Mill. In general, the high rate setting will provide a lead feed rate slightly higher than the Mill can handle during a normal air flow rate and therefore normal operation. A low feed rate will be set after the ammeter has reached the current set point as indicated by the setting on the setting page of the HMI, the system will now switch to a low feed rate which will be slightly less than the system can handle. This will give the cyclic effect as shown below.





2. NORMAL RUNNING PROCEDURE

As the Mill is semi-automatic the only attention required under normal running conditions is as follows: -

- Place lead pigs on the storage conveyor.
- Take samples of oxide for Pbo evaluation (frequency to be determined on site but generally every hour and recorded against the trend readings for the Mill temperature and motor current).
- Record running conditions every hour, noting the following;
 - Mill temperature.
 - Motor load reading.
 - Lead used.
 - Pbo content.
- Keep Mill area clean.

Initial start-up settings are: -

Control temperature	126°C
Emergency temperature	126°C + 5°C
Mill ampere loading	53 Amps



3. NORMAL SHUT-DOWN PROCEDURE

The Mill can be stopped in a fully automatic mode were the system will be monitored for the correct sequence, it can also be stopped in manual if required but the former is preferred.

Which method is used the following sequence is used, the first stage being to stop the feeding of lead into the Mills.

- Stop the Mill Motor, this automatically stops the lead feed when in automatic, if in manual the billet conveyor must be stopped by hand
- Wait 5 minutes then proceed as follows.
- Stop oversize return conveyor.
- Stop Cooling fan.

Wait until the Mill temperature is below 80°C then

- Stop Extractor Fan.
- Switch off control panel isolator if necessary but this is preferred to be left on as the Mill temperature is constantly monitored for thermal build-up.
- Switch off compressed air and water supply, only is control system isolated.
- Stop oxide transfer system.

NOTES



4. EMERGENCY SHUT-DOWN PROCEDURE

In an emergency the plant can be completely shut-down by switching off the control panel or by pressing the remote emergency stop button.

There are 3 emergency stop positions for each Mill and activating any one will stop the Mill completely up to the outlet from the dust filter. The rotary valve and conveying system will be operated by the oxide transfer panel and the Mill emergency stop system will not affect these items.

If possible after an emergency stop procedure and after all safety checks have been carried out you should attempt to reset the system, re-start the exhaust fan and run for a few minutes to clear the oxide dust from the system and reduce the temperature below the control set point.

The Mill should have a standard charge level which should exist when the drum is stationary, (see appendix 1). The standard charge is what is defined as constantly resident within the Mill at all times. If this charge is allowed to fall then the Mill will require to make this weight up on restarting and thus attaining the correct current loading.

NOTES



5. <u>CONTROL OF PRODUCT</u>

The most consistent quality lead oxide is attained when the plant is run during 24-hour continuous operation. This will reduce the effect of low Pbo levels during the start-up sequence.

The mean Pbo may be varied by adjusting the control temperature.

The particle size and water absorption analyses of the product are a characteristic of this plant, and can only be marginally varied independently.

S.G. Determination method shown in Appendix 6

5.1 Control of Pbo by the Temperature Controller

A pre-requisite of control by temperature adjustment is that the plant is operating normally in all other aspects, and preferably has been for a considerable time before adjustment is made.

Any difference in the product will not be seen for approximately two hours after making the adjustment.

To increase the Pbo, the control temperature should be raised and vice versa to lower the Pbo.

Any temperature adjustments should be in general done in 2 - 3° C increments.

5.2 Correcting a Gradual Drift of Pbo

A 2° C adjustment in the control temperature should be adequate.

At least two hours should elapse before making a further adjustment in the same direction. Adjustment may be made in the opposite direction after two hours if the effect on Pbo has been too severe.

5.3 Correcting Larger Discrepancies in Pbo Level

Determine reason for variation in Pbo level. Check temperature, air flow, Mill load and make necessary adjustments.

At least two hours should elapse before making a further adjustment in the same direction. Adjustment may be made in the opposite direction after two hours if the effect Pbo has been too severe.



5.4 Special Instructions if Pbo is Very High

Some of the more common abnormal conditions which can result in high Pbo level are: -

- Operating temperature too high caused, for example, by inadequate supply of cooling water on to the Mill shell. After several years of operation, the Mill shell may become coated with lime scale impurities from the water. If this happens then the cooling effect of the water will be dampened.
- Opening the main valve too late on plant start-up.
- Running Mill charge up to working temperature twice or more without a period of normal operation between.
- Running Mill at too low air flow.

5.5 Special Instructions if Pbo is Very Low

Some of the more common abnormal conditions which can result in low Pbo level are:

- Operating temperatures too low caused, for example, by too much water onto the Mill shell. Normal cooling flow should reduce the temperature slowly and not cause the Mill to lose temperature quickly.
- Opening the main valve too soon on plant start-up.
- Air flow too great and/or very cold ambient temperatures.
- When the Pbo is very low, the following procedure should be followed.
- Close the main valve to about half way position. This will reduce the air flow, and increase the residence time of the oxide in the Mill drum.
- During this period, continue to monitor the Pbo content every 30 minutes until it is within the desired range and then open the main valve to normal running position.
- 5.6 Special Instructions if the Mill temperature rises and cannot be controlled by the control and emergency water

Some of the more common conditions which can cause loss of control are: -

- Inadequate supply or distribution of cooling water onto the Mill shell.
- Opening the main valve too late on plant start-up.

The above condition may be accompanied by a loss of noise from the Mill shell.

• Under these circumstances stop the Mill immediately to prevent a thermal runaway, and leave the extractor fan running to bring down the charge temperature.



• Wait about 1/2 hour and re-start according to normal start-up procedure and carefully monitor Pbo content until within desired range.

It is always worth remembering the design concepts of the Oxide Mill.

- That lead is the fuel
- The airflow is to extract oxide out of the Mill and also cause Mill cooling
- Water is used to cool the reaction, i.e. hold it place

Problems occur when the medium for cooling takes away more heat than the fuel can provide, i.e. increased airflow or cooling water will require more load and therefore more fuel to try and maintain the temperature and therefore the Pbo.

The Mill is rated at a certain loading which will maintain the specified Pbo if the cooling is correct.

<u>NOTES</u>

6. <u>RUNNING FAULTS AND REMEDIES</u>

The instruments alarms on the control panel HMI will provide an initial indication of faults.

6.1 Temperature Control – HMI Setpoint

This controls the temperature of the Mill shell by actuating solenoids which allow cooling water to be sprayed onto the shell surface.

Mill Shell Temperature

The Mill shell temperature is used to control the Pbo as described previously.

If this temperature does not remain constant and cannot be controlled the following points should be examined: -

- Is the water flow correct?
- Is the water switched on and off correctly by the temperature control? Check for correct operation of the solenoid valve which controls the cooling water flow to the Mill shell.
- Is the water being sprayed onto the Mill shell correctly?
- The spray pipe should distribute water over the full length of the Mill shell. When the water used on the Mill is hard, it is necessary to remove the pipe and clear the jets periodically to ensure that an even spray is obtained.
- Is the temperature control reading correctly?



- Temperature measurement is by a radiation pyrometer and malfunction of this instrument or connections will give an inaccurate temperature reading. It is also important that the signal cable is run correctly, i.e. a minimum of 300mm away from the Mill main drives cables.
- If the temperature becomes erratic, check the above items.

6.2 <u>Flowrator – Water flow controller</u>

This instrument measures the cooling water flow rate onto the Mill shell. During normal running the float should rise and fall as the control water is switched on and off. If the float indicates continuous flow of water onto the Mill or no flow, first ensure that the temperature controller is working correctly. Second, examine the solenoid valve on the Mill panel to ensure that the valve is actuated by the temperature controller. If both the temperature controller and the solenoid valve are working correctly, and there is no flow of water then a blockage in the pipe line or spray pipe may have occurred.

6.3 <u>Differential Pressure gauges</u>

Readings on these instruments vary directly because of the amount of static pressure/air flow in the system. The operators should familiarise themselves with the normal readings and investigate any prolonged deviations as this will affect the consistency of the product.

6.4 Dust Filter Differential Pressure gauge.

The initial reading on the DPG when the dust filter bags are new is normally less than 100 mm. This reading will slowly rise over the months as the bags become partially blinded. When the maximum allowable figure is attained, a new set of bags should be installed.

If the DPG reading falls quickly this indicates a hole in one or more of the bags. In this case, the Mill should be stopped immediately and new bags installed.

6.5 <u>Absolute Filter Differential Pressure gauge</u>

The initial reading of the manometer when the absolute filter element is new is normally less than 20 mm. This reading slowly rises during use. If, however, the reading starts increasing quickly (e.g. 10 mm or more in a day), this indicates a hole in one or more bags in the dust filter. In this case, the Mill should be stopped immediately and new bags installed. When the maximum allowable reading, see is attained, the elements should be renewed.

6.6 Mill Load Ammeter Setting - HMI.

This indicates the Mill motor current. When the current to the Mill motor is less than the set point on the PLC the high feed rate timer will be started, when the current is higher than set point the low feed rate timer will be selected.

The two timers control settings control the lead pig feed conveyor and hence the lead input to the Mill. If the conveyor fails to stop after feeding a lead pig examine the detector plate mechanism and reset if necessary and/or examine the timers for correct operation.



6.7 <u>Alarm System</u>

High Temperature Alarm

If the temperature controller high set point is exceeded, an alarm bell rings and emergency water is sprayed on the Mill. The cause of the high temperature must be determined as described in the notes on Running Faults in Section 6. The emergency water is as stated for emergency conditions and if it is activated then the cause must be investigated.

6.8 <u>Dust Filter</u>

If the differential pressure indicator reading across the dust filter bags has increased rapidly then check that the air cleaning pulses are working correctly. Examine the timer unit mounted on the Dust filter and the air valves on the Dust filter.

There are 5 solenoid valves and 5 diaphragm air valves. Check the diaphragms which may be worn or damaged. The timer operates on a continuous cleaning cycle, purging each compartment with air approximately every 4 minutes.



CTT TYPE 8 OXIDE MILL INSTALLATION

GENERAL DESCRIPTION OF PLANT

LIST OF CONTENTS PAGE 33 - 35 LEAD FEED SYSTEM 36 **OXIDE MILL** OVERSIZED PARTICLES RETURN CONVEYOR SYSTEM 37 DUST FILTRATION SYSTEM 37 DUST FILTER PNEUMATICS 37 MILL CONTROL PANEL 38 ACOUSTIC ENCLOSURE 38 **COOLING FAN** 38 LOAD CONTROLLER 38 TEMPERATURE CONTROLLER 39 MAINTENANCE 40 - 42



1. <u>LEAD PIG FEED SYSTEM</u>

Lead pigs are transported to the Mill work area in stacks of about 1 tonne, with each layer in alternate directions and positioned on the floor within reach of the lifting hoist.

The Pigs are then lifted by the hoist whose vertical movements are controlled by a motorised hoist with pendant activated control (this is to be provided by Enersys); the lateral movements are controlled by hand, and placed on the storage conveyor.

The lead pigs are positioned on the storage conveyor with the ears upper most so the rounded edge of the pig touches the slats, this is important for the ease of movement. As and when the oxide Mill requires lead the conveyor operates by the use of two timers, these set the amount of dwell time between feeding.

The lead is then fed into an automatic pig chopping system. This is pre-set to suit the various sizes of pig that can be used.

At each stage of the pig chopping process, proximity sensors detect the lead position, confirming the signals back to the PLC system.

A rod less cylinder pushes the lead beneath the guillotine blade prior to chopping.





Sensors for the Lead Chopping Device

INPUT	DESCRIPTION
PR1	Flap Switch
PR2	Bed raised
PR3	Pusher cylinder extended to lead cutting position
PR4	Blade lowered
PR5	Blade raised
PR6	Pusher cylinder extended to fully extended
PR7	Bed lowered
P78	Pusher cylinder retracted





Operating Sequence

PRESS START SIGNAL WILL BE INITIATED ON INTERNAL HIGH / LOW TIMER SYSTEM HIGH / LOW TIMER WILL TIME OUT CONVEYOR WILL RUN FLAP SWITCH HIT CONVEYOR WILL STOP AFTER 1.0 SECOND TO ALIGN PIG CORRECTLY BED WILL RAISE AFTER ADDITIONAL 1.0 SECONDS

- 1. INTERNAL TIMER FOR PAUSE BETWEEN CUTTING WILL COMMENCE (30 45 SECONDS)
- 2. PUSHER CYLINDER WILL ADVANCE
- 3. SWITCH BEHIND CUTTING BLADE WILL BE ACTIVATED
- 4. BLADE WILL LOWER UNTIL LOWERED SWITCH IS ACTIVATED
- 5. BLADE WILL RAISE UNTIL RAISED SWITCH IS ACTIVATED

SEQUENCE 1 - 5 WILL COMMENCE UNTIL PIG IS FINISHED

SYSTEM WILL THEN RECOMMENCE FROM THE BEGINNING

Should the pig not be pushed enough to hit the switch (3) within the 30 second time period then a cut will take place whether or not. This allows for the error which may occur it the pig is stuck in the cutter.

Note: All times are adjustable during commissioning and may differ.

Alarms for the following

- General disturbance if a door opens or lead misfeeds
- If a door is opened during running all outputs will stop, once door is closed the control will recommence
- Reset the system by closing all doors and switching the reset
- See appendix 3 for the standard timings for the chopping sequence.
- Calculations for setting up of the Pig Feed are generally as described in App 4



2. OXIDE MILL

The Mill is driven by a 37-kW motor and geared to give an approximate speed of 27 rev/min.

As the drum rotates, the lead pieces cascade and oxide particles are formed. The drum is enclosed by a sheet casing. The upper half is fitted with a flanged hood to which is attached ducting and the Mill cooling fan.

The Mill drum temperature is controlled by an automatic water-cooling system. An additional emergency water control system is incorporated to deal with any abnormal temperature rises.





3. OVERSIZED PARTICLES RETURN CONVEYOR SYSTEM

A proportion of the oxide particles withdrawn from the Mill by the air stream are oversized.

The design is such that they do not reach the dust filter, but fall back onto a screw conveyor which returns the particles to the Mill for further grinding. The conveyor is chain driven by a 0.37 kW motor geared unit.

4. <u>DUST FILTRATION SYSTEM</u>

Oxide particles from the Mill are conveyed by the flow of air to a dust filter comprising five compartments each with four filter bags through which the air is drawn by an extraction fan driven by a 5.5 kW motor.

An absolute filter is provided downstream of the dust filter and extraction fan to ensure minimum atmospheric dust release and also as a precaution against element failure in the dust filter.

The absolute filter medium, esparto grass-based paper with a proportion of finely corded asbestos fibres, is accessible through removable doors in the filter case.

The duct to atmosphere is to be connected to a final extraction system provided by Enersys.

The dust filter timing unit is mounted on the dust filter and an electrical supply point for this unit is included in the control panel.

The timer unit will be active whenever the oxide is being transferred from the dust filter.

The timer unit also allows for the pulsing of the pneumatic rapper on the filter cone which is operated in conjunction with the filter pulse sequence.

Each of the five compartments is cleaned on a continuous cycle, in sequence by a programme timer. When the cleaning cycle is energized the section, outlet is sealed by a damper and pneumatic cylinder thus isolating the section from the rest of the filter.

A series of high energy pulses are actuated by the pilot operated diaphragm valve at pre-set intervals.

The pulses are exhausted into the clean air chamber, not directly into the bags, thereby ensuring longer bag life.

See separate operation and maintenance manual for the filtration system.



5. <u>MILL CONTROL PANELS</u>

There are two control panels provided. The MCC panel which will control all the electrical motors for the operation of the Mill and conveying. This panel is located within 10 m of the Mill installations. There is also a PLC ICA control panel which is to be located in the Mill control room which will provide all of the controls for both the Mills and the pneumatic conveying system provided.

Circuit diagrams of the Mill electrical system and the PLC ICA panel are supplied.

6. <u>ACOUSTIC ENCLOSURE</u>

The acoustic Mill enclosure assembly is designed to give a maximum noise level of 85 dBA at a distance of one metre. There is a corrosion resistant discharge light fitted with an on/off switch inside the enclosure.

7. <u>COOLING FAN</u>

When the start button is pressed the operation of the fan motor is a function of the actual temperature set point. Below set point the fan is inoperative, and above set point operative.

8. <u>LOAD CONTROLLER</u>

The load controller consists of an averaged ammeter within the PLC unit taking an average of 1000 current samples over 10 seconds. The controller works in conjunction with the lead feed system which has two HMI timers for the high and low lead feeds.

These timers will be used to set the dwell time between the feeding conveyor starting from a stopped position.

When the current is below set point the dwell time will be considerably less than when it is above set point.

These dwell times will be set during commissioning.

The amount of current taken by the Mill motor gives an indication of the charge weight and is detected by the damped ammeter.

When the weight is high and the load is above set point, the control circuit selects a lower feed rate. When the weight is low and the load is below the set point, the controller changes to a higher feed rate.



11. <u>TEMPERATURE CONTROLLER</u>

This Instrument is internal to the PLC and has a proportional time action at the control point and a direct ON/OFF action at the alarm high point. The control water solenoid set point is indicated by set point one and the alarm high emergency water solenoid and alarm bell high set point are indicated by the set point two.

All PID functions are accessed on the setpoint page.

The signal for the temperature control is derived from a radiation pyrometer which detects the surface temperature of the Mill drum.

Maintenance of the pyrometer should be carried out in accordance with the manufacturer's instruction manual supplied separately.

NOTES



CTT TYPE 8 OXIDE MILL INSTALLATION - MAINTENANCE

2. <u>MILLING PLANT MECHANICAL MAINTENANCE</u>

2.1 <u>Weekly</u>

Inspect the following drive belts for signs of wear and ensure correct tension: -

- Main motor drive belts.
- Exhaust fan drive belts.

Change belts and/or tension when necessary.

Oversized Particles Return Conveyor Seal

The felt seal between the oversized particles return conveyor, and the Mill drum outlet trunnion, should be inspected for possible oxide leaks. At the first sign of a leak check compression springs are seating correctly. If components are correctly sealed it will be necessary to change the felt seal. To change the felt seal it is also necessary to remove the oversized particles return conveyor.

Pig Feed

- Inspect the pig feed blade cutting edge.
- Check oil level in hydraulic power pack.

Air-Line Filters: -

Air-line filters are designed to collect impurities from the pneumatic system. The filters are cleaned by opening the drain taps on the transparent bowl. In very humid conditions it may be necessary to drain off daily.

Main Gears

Inspect main gears and check for damage or excessive wear.



2.2 <u>Monthly</u>

Dust Filter

Inspect bag tension and adjust when necessary. See manufacturer's Instruction manual for maintenance details.

Inspect vibrator

2.3 <u>Annually</u>

<u>General</u>

The plant has been designed to operate continuously with a minimum of stoppages for maintenance. It is though recommended to close down the plant annually for a full maintenance procedure.

Inspect the whole plant and replace, or repair as appropriate any items showing signs of excessive wear, deterioration or other signs of failure. Oil in the gearbox and pig feed is to be drained and filled to the correct level with new oil.

2.4 <u>Miscellaneous</u>

Absolute Filter Element

The life of an absolute filter element is approximately 12 months. Each time the filter bags are changed withdraw the element from the casing and inspect. Ensure that the upper sides are oxide free which indicates that the element is performing satisfactorily and has not become blocked with lead oxide dust.

The differential pressure indicator across the absolute filter should normally read less than the figure stated.

When replacing the absolute filter, fit in the case with the rubber seal downwards (ignore the arrow on the side of the filter box). Check the rubber seals on the base of the case. Slide the clamp frame over the top of the filter cartridge and tighten the four clamp bolts down firmly onto the clamp frames, this should compress the rubber seal on the bottom of the cartridge.

Dust Filter

Change the complete set of bag filters if a hole develops in one or more bags. This condition is indicated if the reading on the manometer across the absolute filter increases at a much greater rate than normal and the manometer reading across the dust filter decreases. Regular reading of the manometers is essential to ensure early warning of bag filter failure.

Change the bag filters if the reading on the differential pressure manometer across the dust filter exceeds the maximum. If it is evident that this reading will be reached shortly



after the annual shutdown, it may be more convenient to change the bags during the main plant maintenance period.

However, they should be renewed at least once every 12 months.

Belt Drives

Inspect for signs of wear and ensure correct tension. If belts require changing replace whole set and destroy used belts. Keep spares stocked at correct level by re-ordering immediately belts are used. Specify matched sets.



APPENDIX 1



NORMAL OPERATION MILL CHARGE LEVEL

This shows the standard level of charge which is to remain in the Mill during normal operation.

If the charge is too high then Pbo levels will be high, too low and the Pbo levels will be low. It is therefore important that this level is maintained.

The correct level will be when the Mill loading is at the ammeter setpoint.

NOTES



APPENDIX 2 MILL LUBRICATION DETAILS

Item	Reference	Lubricant / grease	Quantity	Comments
Oxide Mill gearbox	David Brown gearbox TWU12	Mobil gear SHC 632	12.5 litres	Change after first 100 hrs running
Main Mill drive motor	Brook Crompton	Mobil grease HP		As required
Oxide Mill drive gears	Hargreaves Hamilton	Shell malleus OGH viscosity 160 at 100° c	25 litres / case	Check level with dip stick
Conveying system vacuum pump	GM 7 I machine	SAE 5W40	1 litre	Change after first 100 hrs running
Oversized particle gearbox	David Brown gearbox R0322	Mobil gear SHC 632	1.2 litres	Sealed unit oil filled
Pig feed hydraulic tank	Dodd Engineering	ISO 46	60 litres per tank	Hydraulic oil supplied by CTT
Pig feed conveyor gearbox	SEW geared unit R107 R77	Sew gear oil poly 460 WE	Sealed for life	Check oil filled prior to start up
Mill bearings	Cooper bearings	Darina R2 grease		Grease lubrication point as necessary
Oversized particle motor	David Brown	Alvania RI3 grease		Grease lubrication point as necessary
Oversized particle drive chain		Morlina 320 oil		As required
Exhaust fan motor	Awaiting information			
Fan bearings	Awaiting information			
Rotary valve gearbox	Awaiting information			
Cooling fan motor		Alvania RI3 grease		As required
Pig feed blade slides	Dodd Engineering	Alvania RI3 grease		Grease each slide daily
		<i>v</i>		

The above lubricants are manufactured by Shell Limited. Other reputable specialist lubricants may be used, provided prior approval is obtained from Chloride Technical and Trading Ltd.



APPENDIX 3





APPENDIX 4

Sample calculation for automatic lead chopping operation

Based on ingot weight of 40 Kg - cut into 3 pieces

The following calculation can be changed if the lead ingot size is different to 40 Kg, the timing can also be adjusted to give higher or lower feed rates. See Section on programming the P.L.C.

ESTIMATE OF TIMINGS

Start Conveyor Lead to Flapper Switch, Sensor 1 Start Origa, Hit Sensor 3 Blade down & blade up, Sensors 3 & 4

First piece into Mill in

Wait time variable, set at Start Origa, hit sensor 3 Blade down & blade up, Sensors 4 & 5

Wait time variable, set at Start Origa, hit sensor 6

Third piece into Mill in

Withdraw Origa Cylinder, Sensor 8

Estimated Cycle Time

Go to high or low rate timers

High rate time, variable Low rate time, variable Nominal rate, variable Dependent on lead position on conveyor 10 Seconds 20 Seconds

45 Seconds

125 Seconds 10 Seconds 20 Seconds

125 Seconds 5 Seconds

330 Seconds

15 Seconds

345 Seconds

90 Seconds (Set on HMI) 180 Seconds (Set In HMI) 135 Seconds

Start at beginning again for next cycle

As well as the HIGH and LOW rate feed timers which operate between full ingots of lead fed into the Mill, there is a wait time between each piece chopped.

This wait time is adjustable on commissioning in the Programmable Logic Controller, (P.L.C.).



MILL THROUGHPUT - OXIDE PRODUCTION RATE

Based on assumed variable times of: -

Origa cylinder wait time Nominal feed rate High feed rate Low feed rate 125 Seconds 135 Seconds 90 Seconds 180 Seconds

At Constant High Rate

90 seconds off and wait 125 seconds

One ingot processed in 7 minutes 15 seconds (435 secs) 8.2758 ingots per hour x 40 Kg = 331 Kg/hour

= 55.614 TONNES PER WEEK

AT CONSTANT LOW RATE

180 Seconds off and wait 125 seconds

One ingot processed in 8 minutes 45 seconds (525 secs) 6.857 ingots per hour x 40 Kg - 274 Kg/hour

= 46.08 TONNES PER WEEK

AT CONSTANT NOMINAL RATE

135 Seconds off and wait 125 seconds

One ingot processed in 8 minutes 0 seconds (435 secs) 7.5 ingots per hour x 40 Kg = 300 Kg/hour

= 50.4 TONNES PER WEEK

All the above are examples and the time settings need to be adjusted during commissioning, the basis of the settings is to achieve a feed rate to the Mill on high rate of 330 kgs/hr and a low rate feed approx. 280 kgs/hr



Theoretical throughput of automatic lead ingot chopping system

Forgetting entirely the load control facility.

With PLC wait time set to

With high & low rate feed timers set to

Cycle time for processing 1 ingot

0 seconds

5 seconds

100 seconds

- = 36 ingots per hour x 40 kg
- = 1440 Kgs per hour
- = 242 tonnes per week

The above shows a maximum speed of the automatic lead ingot feed system and is a theoretical figure only.



APPENDIX 5

ORIGIN	DESTINATION - MILL No3	CABLE SIZE (Assume 10 Metre RUN)	
PLC Control Panel	Mill Hopper Filter Controller	1mm 3 Core	110 Volt Supply
PLC Control Panel	Mill Hopper Filter Controller	1mm 4 Core	DI & Start Signal
PLC Control Panel	Mill Hopper Filter Controller	1mm 1 Pair Screen	Differential Pressure
Mill Junction Box	Raise Bed Solenoid	1mm 3 Core	4
Mill Junction Box	Retract Pusher Cylinder Solenoid	1mm 3 Core	
Mill Junction Box	Lower Blade Solenoid	1mm 3 Core	
Mill Junction Box	Raise Blade Solenoid	1mm 3 Core	
PLC Control Panel	Water Solenoid Control Panel	1mm 4 Core	
PLC Control Panel	Hopper Wrap Round Solenoid	1mm 3 Core	
PLC Control Panel	Emergency Stop PBS	1mm 4 Core	
PLC Control Panel	Guard Switch No1	1mm 4 Core	
PLC Control Panel	Guard Switch No2	1mm 4 Core	
PLC Control Panel	Guard Switch No3	1mm 4 Core	
PLC Control Panel	Mill Temperature Monitor	1mm 2 Pair Screen	Pyrometer
PLC Control Panel	Air Flow Damper	1mm 4 Core	Belimo
PLC Control Panel	Mill Junction Box	1mm 17 Core	Switches To JB
PLC Control Panel	Mill Junction Box	1mm 17 Core	
PLC Control Panel	Mill Temperature	Type "K" Thermocouple	



APPENDIX 6

DIRECT Pbo DETERMINATION BY GRAVITY

Rise using acetic acid (S.G. 1.030)

GRAVITY READING	<u>% Pbo</u>	GRAVITY READING	<u>% Pbo</u>
1126	49.8	1146	60.2
1127	50.3	1147	60.7
1128	50.8	1148	61.3
1129	51.4	1149	61.8
1130	51.8	1150	62.3
1131	52.3	1151	62.8
1132	52.8	1152	63.3
1133	53.4	1153	63.8
1134	53.9	1154	64.3
1135	54.4	1155	64.8
1136	54.9	1156	65.4
1137	55.5	1157	65.9
1138	56.0	1158	66.4
1139	56.6	1159	66.9
1140	57.1	1160	67.4
1141	57.6	1161	67.9
1142	58.1	1162	68.4
1143	58.7	1163	69.0
1144	59.2	1164	69.5
1145	59.7	1165	70.0



NOTES on S.G. rise method

- 1. The 40g of oxide must be accurately weighed on the balance
- 2. The measuring cylinders must be used, one for measuring out the 1.030 SG and one for measuring the final S.G. of the lead solution. The cylinder used for measuring the final S.G. should not be washed out between each determination unless it is to be left for several hours, in which case the cylinder would be thoroughly rinsed several times with water and allowed to drain before re-use. A cylinder which has lead acetate crystals on the inside of the wall should never be used without first being washed and allowed to drain.
- 3. The object of the mechanical stirring is to eliminate the variation from one operator to another which occurs when hand agitation is used. The residual lead in the oxide should form numerous small pellets and not one consolidated lump. Formation of one lump of lead will give rise to low oxide figures.
- 4. The temperature of the stock acetic acid solution must be 20°C when the Specific Gravity is taken (1.030 @ 20°C). This does not mean that the acid must be 20°C when used for an oxide determination.
- 5. The final S.G. must be taken with the liquid at 20°C.
- 6. The hydrometer must be washed free from acetic acid and lead acetate and wiped dry when not in sue, but several determinations are to be carried out in succession it is only necessary to allow the liquid to drain off between determinations.
- 7. The propeller and shaft of the stirrer should be wiped dry between determinations and washed and dried when determinations are complete. This also applies to beakers.
- 8. Non-laboratory personnel must be properly instructed in the hazards involved.
 - a) An eye shield must be worn at all times when handling acetic acid
 - b) As this method involves handling lead solutions it is even more important than usual to wash the hands free from contamination when the testing is completed.
- 9. The factor, F, used in calculating the results can be influenced by local conditions, particularly by the type and speed of the stirrer used. Once conditions have been established, they must be standardised and rigidly adhered to. For this reason, a stirrer with a scale denoting the position of the speed control knob is essential, particularly with inexperienced operators.



GREY OXIDE ANALYSIS

Lead Monoxide - method suitable for plant operatives

Reagents required

 Dilute acetic acid (S.G. 1.030 at 20°C). Add 920 cm³ of glacial acetic acid to 3,080 cm³ of water and mix thoroughly. If necessary, make minor adjustments (by adding small amounts of acid or water) to bring the S.G. to exactly 1.030 at 20°C.

Equipment Required

- 1. Two measuring cylinders, 250 cm³
- 2. One beaker, 400 cm³
- 3. One balance, accurate to 0.1 g
- 4. One scoop (for weighing oxide)
- 5. One electric stirrer, variable speed complete with impeller
- 6. One accurate hydrometer 1.100 1.200 S.G. with long stems (length of Scale of the order of 16 cm). A suitable hydrometer is one to British Standard 718, Series L100, Medium Category, and Normal Tolerance.

<u>Method</u>

Measure exactly 200 cm³ of the Acetic acid solution in a measuring cylinder and transfer to a 400 cm³ beaker.

Weigh out 40 g of oxide on the scoop provided.

Lower the stirrer into the beaker of acid solution and clamp the motor in a position such that the stirrer blades are in the lower half of the acid.

Connect the motor to the supply and adjust the speed to give good agitation of the acid.

Add the oxide from the scoop, <u>slowly</u>, to the agitated acid by tapping the slide of the scoop with the finger.

Lead granules will form in the acid which will slow down the stirrer. When this occurs the stirrer setting should be increased in order to maintain the original speed.

Note the time when all the oxide has been added and continue stirring for one minute.

Decant the liquid into the 250 cm³ measuring cylinder and cool to 20°C by running cold water onto the outside of the cylinder, taking care to prevent any water splashing into the cylinder, and so diluting the liquid.



When the temperature of the liquid is 20°C note the specific gravity.

Theory of method

The lead monoxide in the grey oxide powder dissolves in the acetic acid to form lead acetate. The resultant increase in S.G. is proportional to the amount of lead oxide present.

Calculation

(Final S.G. at 20°C - 1.030) x F = % Pbo

Where F is a factor calculated from the S.G. rise of grey oxide samples of known lead monoxide content (carefully analysed samples).

At Chloride facility the factor used is 519 (see Note 9) and a table based on this factor is given overleaf.