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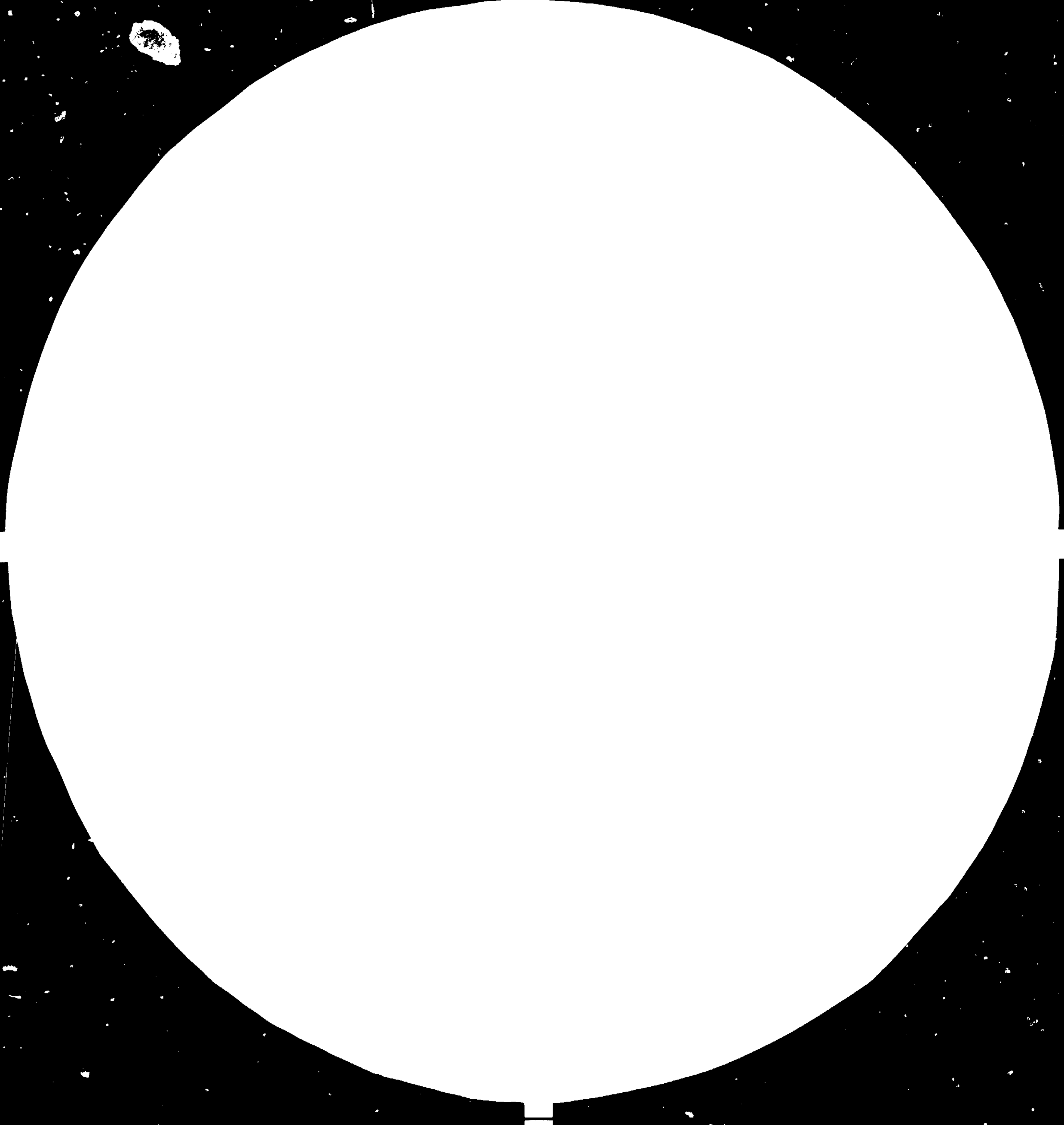
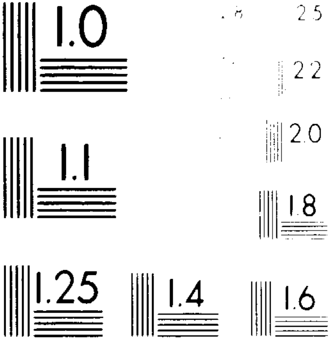
UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

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30 January 1984

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India.

COAL GASIFICATION .

[redacted]

DP/IND/80/004

Technical Report*

Commissioning of
a Thermogravimetric Analysis Apparatus
November/December 1983

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

Based on the work of Alfred Sulimma,
consultant on instrumentation and for
analytical support in coal research

United Nations Industrial Development Organization
Vienna

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Summary

A thermogravimetric analysis apparatus for kinetic studies of coal gasification (TGA) was delivered by Bergbau-Forschung, Essen/FRG, in September 1983 to Regional Research Laboratory, Hyderabad (RRL). From 8 November to 9 December I was assigned to install and commission the TGA, to perform test runs and to train RRL staff in operation and maintenance of the TGA. I was accompanied by Mr. Nottebaum from Bergbau-Forschung, who assisted me in installation and commissioning.

The first three days had to be spent for preparing the room. After this the installation was completed within two weeks and 13 test runs were done, most of them under conditions requested by RRL. During installation Mr. Prasad and Mr. Narasimhan from RRL Coal Division and Mr. Maria Das, head of the RRL Instrumentation Division, and other members of the Instrumentation Division and the workshop of RRL were trained in repairing and maintaining the TGA. During test runs the functioning and handling of all parts of the unit, including the TGA computer, were explained to Mr. Prasad and Mr. Narasimhan in detail, so that they were able to carry out successfully one run by themselves. (Moreover Mr. Narasimhan has been with BF for training for 4 weeks in September/October 1983.)

Some of the controls were not functioning properly, possibly by undetected damages during shipping, so that they had to be replaced or repaired by the suppliers. A test program for the weeks after commissioning was proposed and accepted by Dr. Vaidyeswaran, Director of RRL Coal Division.

1. Background

An autothermic fixed bed pressure coal gasification pilot plant based on the Lurgi process has been erected at the Regional Research Laboratory in Hyderabad to improve the process and to study the gasification characteristics of Indian coals. The fixed bed or moving bed reactor is operated in counter current flow of coal and gasification agent, so that the coal is subsequently preheated, dried and devolatilized, and the so formed char is gasified and finally combusted.

The properties of the coals play very different roles in the different zones of a moving bed reactor. The volatile matter content has great importance in the devolatilization zone, where mainly tar, higher hydrocarbons, and CH_4 can be formed. Also the kinetic of pyrolysis has to be taken into account and secondary reactions of the pyrolysis products with the raw gas from the gasification zone.

In the gasification zone mainly the conversion with steam, including its inhibition by the reaction products H_2 and CO, and with hydrogen takes place whereby the catalytic influence of ash components on the gasification reactions can be of great importance. The reactivity of the solid, i.e. the rate of reaction with the gasifying agent, only affects the process at temperatures up to 1000°C, when the chemical reaction itself determines the rate of the conversion. At higher temperatures the pore diffusion becomes decisive for the gasification rate.

In the combustion zone the oxygen is completely converted in a very fast reaction with the remaining carbon. The resulting CO_2 reacts only very slowly with the carbon as far as the temperature is in the range of 800-1000°C.

The thermogravimetric analysis apparatus (TGA) delivered by Bergbau-Forschung, Essen, FRG, is designed for studying the kinetic of all reactions which occur in the gasification zone of a pressurized fixed bed reactor. The data of the TGA tests can be used for comparison of coals of different origin or different pretreatments (charring) and also to feed a mathematical model of the gasification reactor which is a basis for designing large plants.

2. Description of the TGA

The equipment is designed for the determination of the reactivity of solid fuel like coal and coke with steam, pure gases, and steam/gas mixtures at pressures of up to 100 bar within a temperature range of 300 and 1100°C (Fig. 1). For analysis a micro balance housed in a pressure resistent casing is used. By this micro balance changes of the sample weight in time are measured.

Since low product gas concentrations already do influence the reactivity of the feedstock with pure gasification agents the reactor was designed as differential type reactor. This characteristic is assured if the product gas concentration of the gasification agent is of less than 1 %.

The reactor pressure vessel is made of stainless steel as any other pressure vessels and mains. Inside, an Incoloy 800 tube is mounted around which a helicoidal heating element is wound. Underneath the heating coil which can reach temperatures of up to 1100 °C, a thermocouple for heating control is arranged. A temperature program control assures linear heating rates of up to 100 °C/min.

Underneath the sample basket 3 thermocouples are arranged in a spacing of a few centimeters relative to each other for measuring the gas temperature from which the not directly measurable sample temperature is deduced.

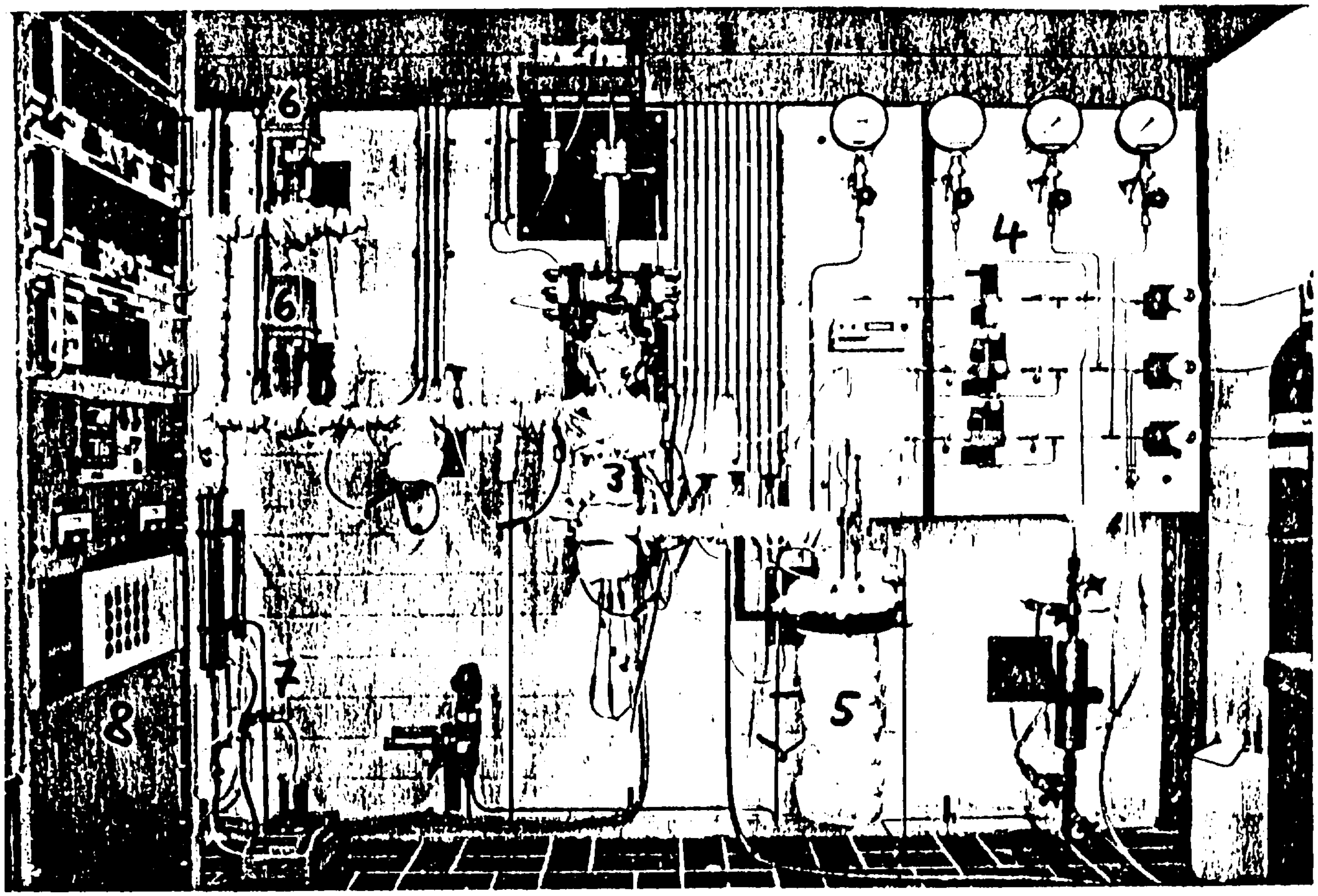


Fig. 1: Thermogravimetric analysis apparatus for kinetic studies of coal gasification during testing at Bergbau-Forschung

1 microbalance
2 sample lock
3 reactor
4 pressure and flow control board
5 steam generator
6 expansion valves
7 condensate measurement
8 control rack

Between thermobalance and reactor a sample lock is arranged. By means of an electric winch system housed in the pressure vessel the sample carrier can be hoisted into the lock system and after filling be lowered into the reaction tube and softly released.

The steam generator of 5 l volume and an evaporation capacity of 5 to 6 g of water/minute can supply steam over several hours before refill becomes necessary. For feeding water to the steam generator also if the generator is pressurized an additional small pressure vessel is used. The pressure in the steam generator is kept constant by a temperature measuring and control system. For internal heating a heating coil is provided.

In view of tests run on steam/gas mixtures as gasification agents, the steam generator design caters for either the gas being proportioned to the steam generator directly or to the steam conduit downstream generator. Exact gas proportioning is assured by a system of thermal mass flow meters and magnetic control valves. The gas pressure is controlled via a two step pressure reduction.

In order to avoid steam condensation in the non-heated part of the equipment - in particular in the casing which contains the micro balance - inert gas flushing of these parts of the equipment is necessary.

Expansion of the gasification agent is controlled by two valves arranged in parallel. Their k_V-values are graded in a way that defined expansion over the whole pressure range and with respect to use of various gases including steam is assured. After expansion, the steam is condensed in a metallic rapid cooler, and collected. An electrical balance enables continuous monitoring of a condensate produced which is a measure for the steam throughput.

Detailed information of all parts of the TGA are given in tables 1 - 6. The arrangement can be seen from the piping diagram (Fig. 2), the heating diagram (Fig. 3) and the P+I-diagram (Fig. 4).

Data logging and processing is assured by the computer HP9816 with video display and disc memory. A data logger and a printer are connected to this computer. Changes of the sample weight are monitored by multiple measurements, logged together with the temperature near the sample and visualized on the video display.

For monitoring the test conditions further 17 temperature, pressures and flows are measured, logged and displayed alpha numerically on the video scope.

After the test diagrams showing the weight changes as a function of time or temperature can be established, and these values can also be printed out in form of lists.

DETAIL SPECIFICATION OF SECTION 1: Reactor

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
WIR 101	sample weight	microbalance, range 1 g, sensitivity: 0,1 mg	Sartorius	max. loading: 25 g	output: 1 V/1 g
GI 102 H 102	sample lift		Bergbau-Forschung	max. height: 550 mm in 10 turns	chains material: silver and nickel
FO 103	cooling water for the sample lock	flow indicator			flow rate: 0,5 - 2,0 l/min
PIR 104	reactor pressure	pressure transducer	Brandt	range: 0 - 100 bar	output: 0 - 10 V
TIRC 105 KC 105	temperature of the internal heater	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin, sealed by compression screws
		Thermocoax heating element with cold ends ZEZ I 20/50-450-50	Philips	diam.: 2 mm length: 4500 mm cold ends: 50 mm	cover: Inconel 600, sealed by compression screws and welding
		controller, range: 0 - 1200 °C, connected with programmer, thyristor unit and transformer	Eurotherm		Input: mV-signal from thermocouple and 0 - 5 V from programmer Output: 10 V-signal to data-logger, 150 V to heater
TIR 106	temperature near sample	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin, sealed by compression screws
TIR 107	temperature 20 mm below sample	as TIR 106	Künzer	as TIR 106	as TIR 106
TIRC 109	outer heating at the reactor bottom	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin
		heating tape: 350 W	Fleischhacker	length: 1 m	
		controller, range: 0 - 600 °C	Eurotherm		Input: mV-signal from thermocouple Output: 10 V-signal to data-logger, logic output to solid state relay and heater
TIRC 110	outer heating at the reactor middle	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	as TIRC 109
		heating tape: 700 W	Fleischhacker	length: 2 m	
		controller, range: 0 - 600 °C	Eurotherm		as TIRC 109
TIRC 111	outer heating at the reactor top	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	as TIRC 109
		heating tape: 1000 W	Fleischhacker	length: 3 m	
		controller, range: 0 - 600 °C	Eurotherm		as TIRC 109
SV 1	reactor safety valve	full-lift safety valve, spring loaded	Leser	set pressure: 110 bar	

Table 1

DETAIL SPECIFICATION OF SECTION 2: Steam Generator

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
PI 201	steam pressure	pressure gauge, bourdon tube, range: 0 - 100 bar	Armaturenbau	diam.: 160 mm	sensitivity: 0,6 % of range
V 21	steam pressure	shut-off valve, connected with pressure gauge 201	Armaturenbau	max. pressure: 600 bar	material: 1.4571 connections: R 1/2"
TIRC 202	temperature of the internal heater	Pt 100-resistance thermometer	Steffen	diam.: 3 mm length: 1 m	cover: 1.4541, sealed by compression screws
		thermocoax heating element with cold ends ZEZ I 20/50-700-50	Philips	diam.: 2 mm length: 700 mm cold ends: 50 mm	cover: Inconel 600, sealed by compression screws and welding
		controller, range: 0 - 400 °C, connected with thy-ristor unit and transformer	Eurotherm		input: Pt 100, three wire thermometer output: 10 V-signal to data-logger, 150 V to heater
TIRC 203	outer heating of the steam generator	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	two thermocouples: one at one third of height, one at the top flange, cover: Incoloy 800, + pole on pin
		heating tape: 2 x 1000 W	Fleischhacker	length: 3 m	two heaters: at the upper and the lower part
		controller, range: 0 - 600 °C	Eurotherm		input: mV-signal from thermocouple output: 10 V to data-logger, logic output to solid state relay and both heaters
LA - 204	upper water level indication	resistance measurement	Bergbau-Forschung		lamp is on, if level contact is free; lamp is off, if level contact is immersed
LA - 205	lower water level indication	resistance measurement	Bergbau-Forschung		as LA - 204
V 22	water refilling, gas inlet	shut-off valve	Hofer	through-hole: 4 mm max.pressure: 240 bar max.temp.: 400 °C	material: 1.4571 connections: R 1/2", cone screwing, packing: graphite
V 23	water refilling, gas outlet	shut-off valve	Hofer	as V 22	as V 22
V 24	water refilling, gas outlet	shut-off valve	Hofer	through-hole: 4 mm max.pressure: 400 bar max.temp. 250 °C	material: 1.4571, connections: R 1/2", cone screwing
V 25	water refilling, water inlet	shut-off valve	Hofer	as V 24	as V 24
V 26	water refilling, emptying of the steam generator	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 100 bar max.temp.: 350 °C	material: 1.4571, connections: compres-sion screws for 6 mm-tubes, packing: graphite
TIRC 206	tube heating from steam generator outlet to reactor inlet	double-thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin
		heating tape: 700 W	Fleischhacker	length: 2 m	
		controller: 0 - 600 °C	Eurotherm		input: mV-signal from thermocouple output: 10 V-signal to data-logger, logic output to solid state relay and heater
V 27	tube from steam generator outlet to reactor inlet	shut-off valve	Hofer	as V 22	as V 22
V 28	tube from steam generator outlet to reactor inlet	shut-off valve	Hofer	as V 22	as V 22
V 29	tube from steam generator outlet to reactor inlet	shut-off valve	Hofer	as V 22	as V 22
SV 2	steam generator safety valve	full-lift safety valve, spring loaded	Leser	set pressure: 110 bar	

Table 2

DETAIL SPECIFICATION OF SECTION 3: Reaction Gas
(for saturation with steam)

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
PI 301	cylinder pressure reducing	pressure gauge	Deutsche L'Air Liquide		connections for burnable gases
PIC 302	cylinder pressure reducing	pressure reducing valve with diaphragm	Deutsche L'Air Liquide		
PC 303	cylinder pressure reducing	spring-loaded pressure reducing valve	Hochdruck-Reduziertechnik	max. supply pressure: 250 bar	
PI 304	gas flow	pressure gauge, range: 0 - 100 bar	Armaturenbau	diam.: 160 mm	sensitivity: 0,6 % of range
V 31	gas flow	shut-off valve, connected with pressure gauge 304	Armaturenbau	max. pressure: 600 bar	material: 1.4571 connections: R 1/2"
FIRC 305	gas flow	thermal mass flow meter / control valve	Wittig / Bronkhorst	max.pressure: 200 bar flow range: 0 - 10 l/min	calibrated at 25 bar with H_2, see calibration certificate, output: 0 - 5 V dc
V 32	gas flow	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 250 bar max.temp.: 150 °C	material: 1.4571 connections: compression screws for 6 mm-tubes
V 33	gas flow	shut-off valve	Hofer	as V 32	as V 32
V 34	gas flow	shut-off valve	Hofer	as V 32	as V 32

Table 3

DETAIL SPECIFICATION OF SECTION 4: Reaction Gas (with or without blending with steam)

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
PI 401	cylinder pressure reducing	pressure gauge	Deutsche L'Air Liquide		connections for noble gases and CO_2
PIC 402	cylinder pressure reducing	pressure reducing valve with diaphragm	Deutsche L'Air Liquide		
PC 403	cylinder pressure reducing	spring-loaded pressure reducing valve	Hochdruck-Reduziertechnik	max. supply pressure: 250 bar	
PI 404	gas flow	pressure gauge, range: 0 - 100 bar	Armaturenbau	diam.: 160 mm	sensitivity: 0,6 % of range
V 41	gas flow	shut-off valve, connected with pressure gauge 404	Armaturenbau	max.pressure: 600 bar	material: 1.4571 connections: R 1/2"
FIRC 405	gas flow	thermal mass flow meter / control valve	Wittig / Bronkhorst	max.pressure: 200 bar flow range: 0 - 10 l/min	calibrated at 20 bar with CO_2, see calibration certificate, output: 0 - 5 V dc
V 42	gas flow	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 250 bar max.temp.: 150 °C	material: 1.4571 connections: compression screws for 6 mm-tubes
V 43	gas flow	shut-off valve	Hofer	as V 42	as V 42
V 44	gas flow	shut-off valve	Hofer	as V 42	as V 42
TIRC 406	preheater temperature	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	coer: Incoloy 800, + pole on pin
		heating tape: 350 W	Fleischnacker	length: 1 m	
		controller	Eurotherm		input: mV-signal from thermocouple output: 10 V to datalogger, logic output to solid state relay and heater
V 45	gas flow	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 100 bar max.temp.: 350 °C	material: 1.4571 connections: compression screws for 6 mm-tubes packing: graphite

Table 4

DETAIL SPECIFICATION OF SECTION 5: Flushing Gas

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
PI 501	cylinder pressure reducing	pressure gauge	Deutsche L'Air Liquide		connections: for noble gases and CO_2
PIC 502	cylinder pressure reducing	pressure reducing valve with diaphragm	Deutsche L'Air Liquide		
PC 503	cylinder pressure reducing	spring-loaded pressure reducing valve	Hochdruck-Reduziertechnik	max. supply pressure: 250 bar	
PI 504	gas flow	pressure gauge, range: 0 - 100 bar	Armaturenbau	diam.: 160 mm	sensitivity: 0,6 % of range
V 51	gas flow	shut-off valve, connected with pressure gauge 504	Armaturenbau	max.pressure: 600 bar	material: 1.4571 connections: R 1/2"
FIR 505	gas flow	thermal mass flow-meter	Mättig / Bronkhorst	max.pressure: 200 bar flow range: 0 - 20 l/min	calibrated at 15 bar with He, see calibration certificate; output: 0 - 5 V dc
V 52	gas flow	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 250 bar max.temp.: 150 °C	material: 1.4571 connections: compression screws for 6 mm-tubes
V 53	gas flow	shut-off valve	Hofer	as V 52	as V 52
V 54	gas flow	shut-off valve	Hofer	as V 52	as V 52
V 55	gas flow	dosaging valve	Deutsche L'Air Liquide	through hole: 1,19 mm max.pressure: 210 bar max.temp.: 230 °C	material: brass connections: compression screws (gyrolok)

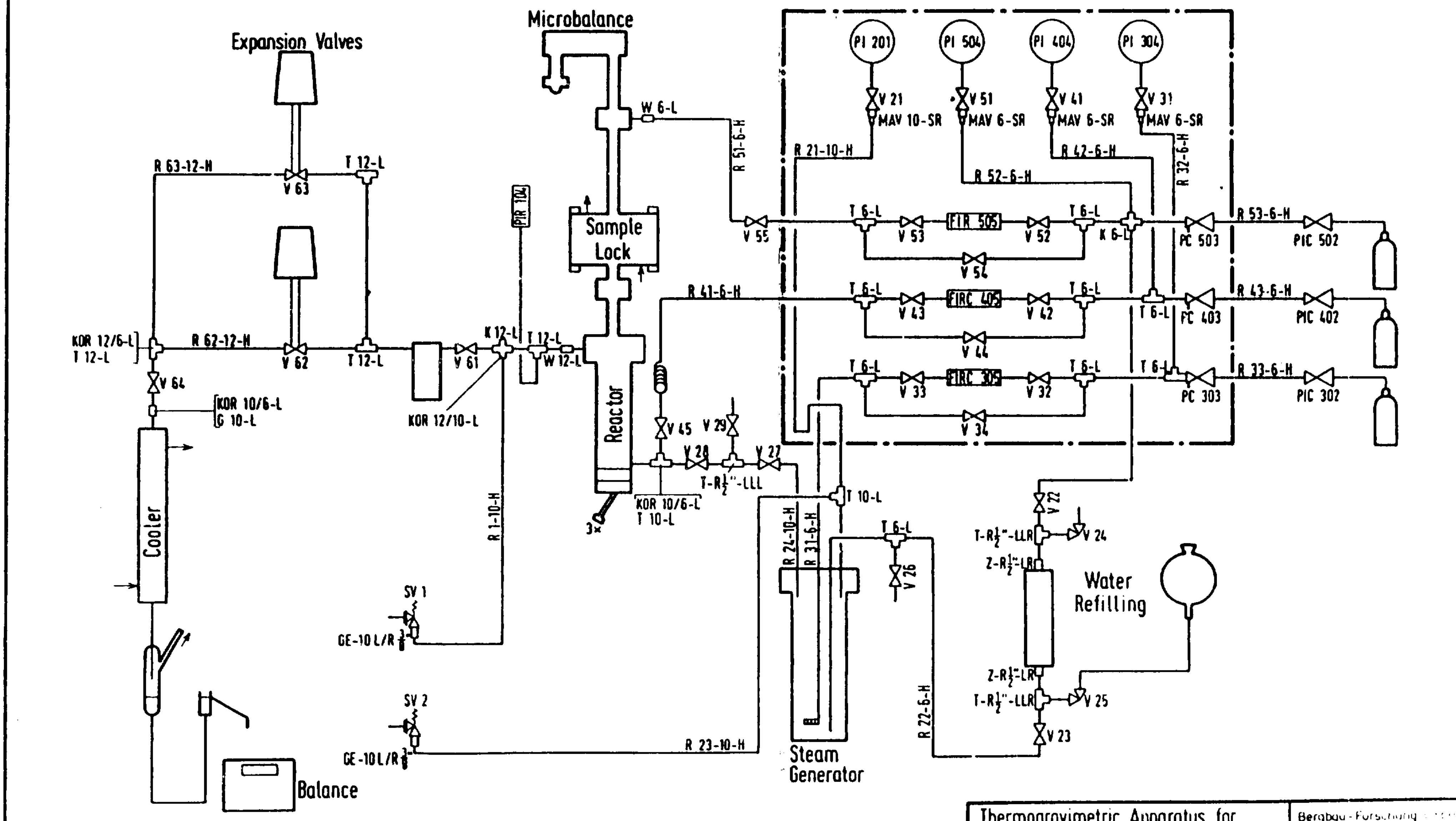
Table 5

DETAIL SPECIFICATION OF SECTION 6: Filter, Expansion, Cooling

Tag No.	Measuring Circuit	Type	Supplier	Dimension	Notes
V 61	filter	shut-off valve	Hofer		
TIRC 601	filter outer heating	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin
		heating tape: 1000 W	Fleischhacker	length: 3 m	
		controller, range: 0 - 600 °C	Eurotherm		input: mV-signal from thermocouple output: 10 V-signal to data-logger, logic output to solid state relay and heater
V 62	expansion	control valve	Kämmer	max.pressure: 100 bar max.temp.: 350 °C k_v: 0,025	material: body 1.4571 cone stellite seat stellite
G 603 H 603	expansion	valve gear: motor drive	Kämmer	220 V, 50 cycles, 5 W	
		step controller and position indicator	Joens	220 V, 50 cycles for 1000 Ω check-back potentiometer	
V 63	expansion	control valve	Kämmer	max.pressure: 100 bar max.temp.: 350 °C k_v: 0,0004	material: body 1.4571 cone stellite seat 1.4122
G 604 H 604	expansion	valve gear: motor drive	Kämmer	as G 603	
		step controller and position indicator	Joens	as G 603	
TIRC 602	expansion valves outer heating	double thermocouple NiCr-Ni	Künzer	diam.: 2 mm length: 500 mm	cover: Incoloy 800, + pole on pin, two thermocouples at V 62 and V 63
		heating tape: 700 W (V 62)	Fleischhacker		
		heating tape: 1000 W (V 63)	Fleischhacker		
		controller, range: 0 - 600 °C	Eurotherm		input: mV-signal from thermocouple output: 10 V to data-logger, logic output to solid state relay and both heaters
V 64	expansion	shut-off valve	Hofer	through-hole: 3 mm max.pressure: 100 bar max.temp.: 350 °C	material: 1.4571, connections: compression screws for 6 mm-tubes, packing: graphite
FO 605	cooling	flow indicate			flow rate: 0,5 - 2 l/min
WIR 606	cooling	balance for condensate measurement	Precisa	range: 4700 g	output: 1 V/1 g

Table 6

Fig. 2

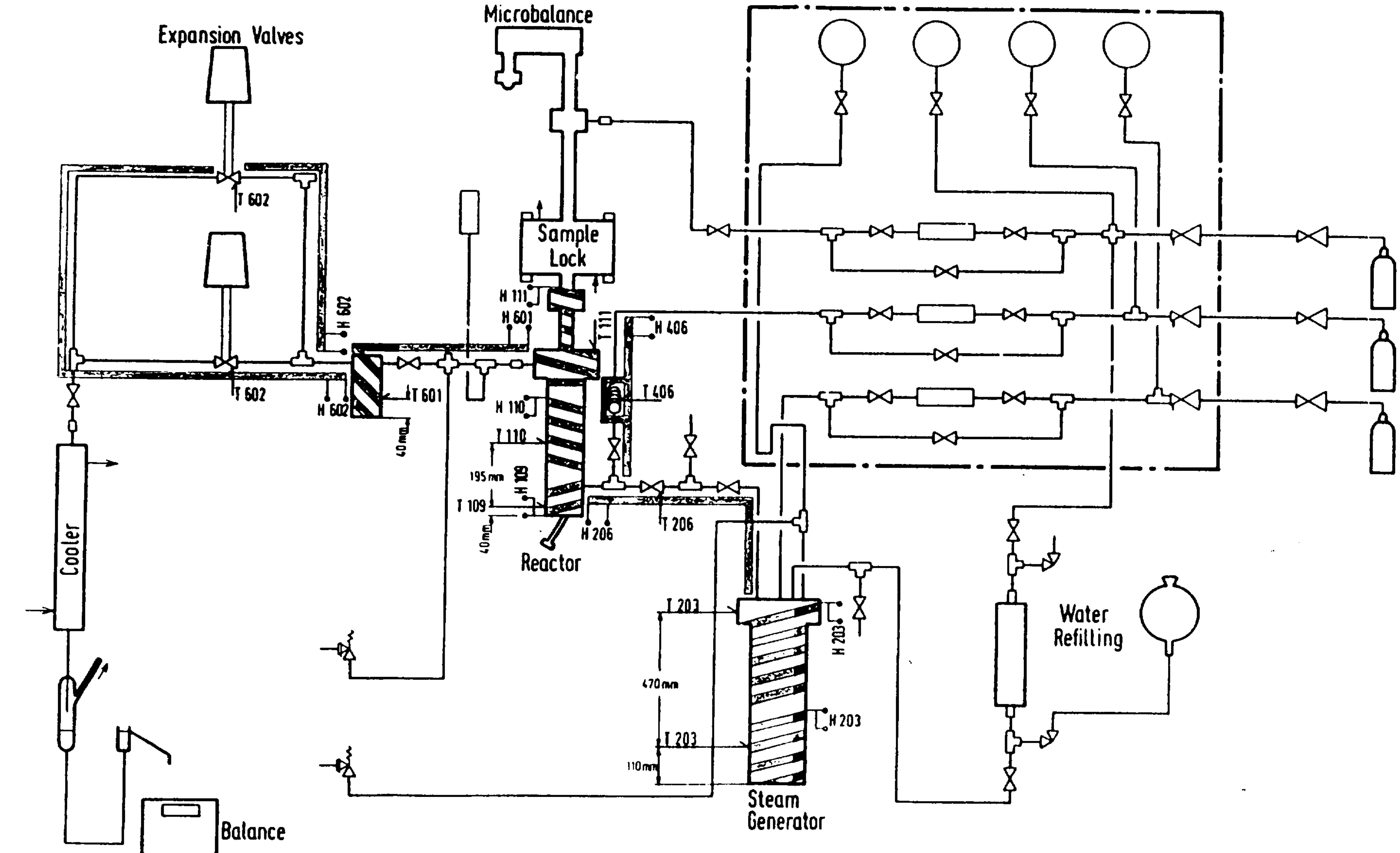


Thermogravimetric Apparatus for Kinetic Studies of Coal Gasification
Project: DP / IND / 80 / 004
-Piping Diagram-

Bergbau-Forschung GmbH
Essen-Kray

31061

Fig. 3

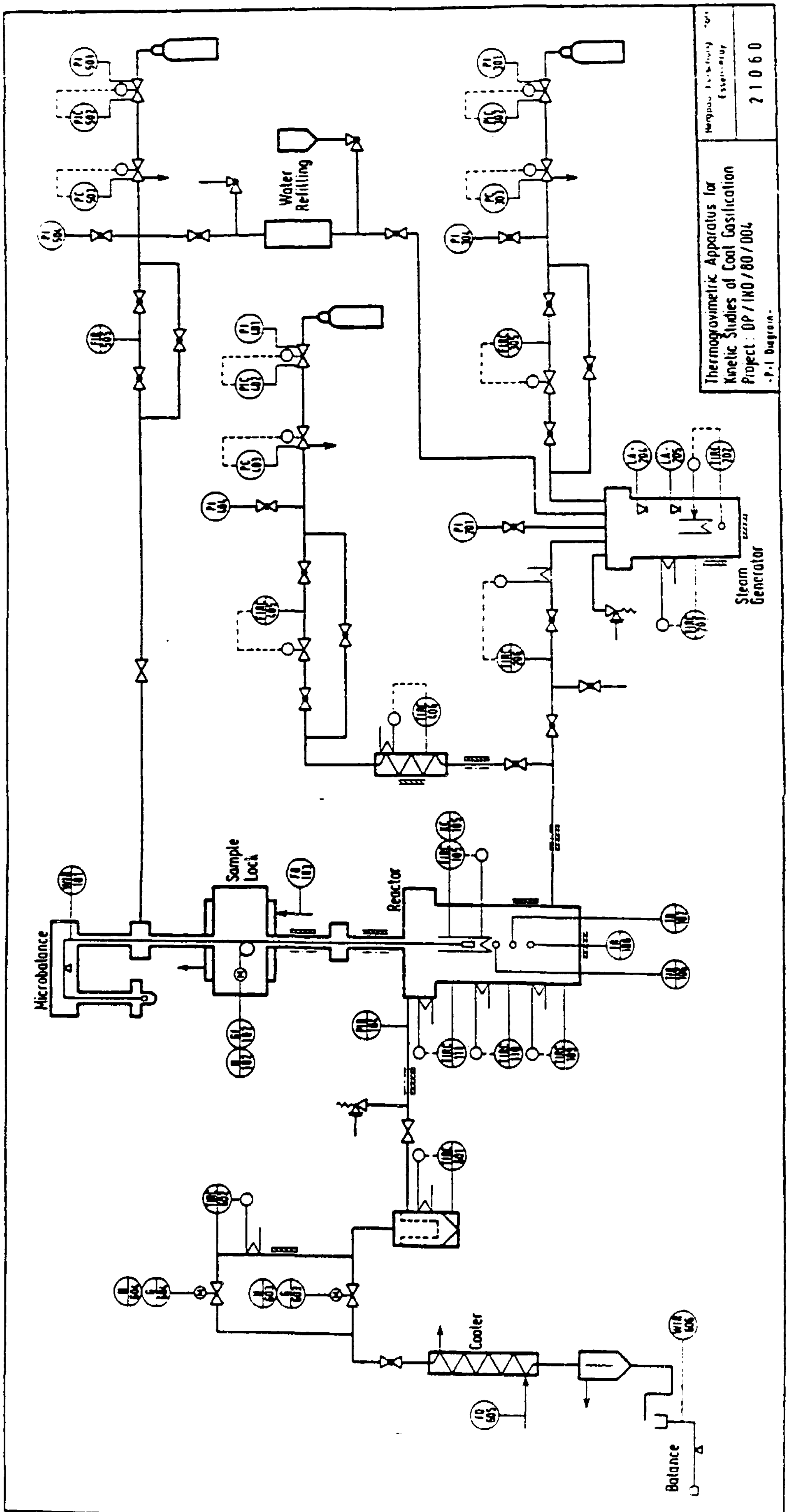


Thermogravimetric Apparatus for Kinetic Studies of Coal Gasification
Project: DP / IND / 80 / 004
- Heating Diagram -

Bergbau-Forschung ...
Essen-Kray

31062

Fig. 4


Thermogravimetric Apparatus for
Kinetic Studies of Coal Gasification
Project: DP/INO/80/004
-P-I Diagram-
21060
Microbalance
Sample Lock
Reactor
Water Refilling
Steam Generator
Cooler
Balance

3. Installation and Start-up of the TGA

After briefing at the UNDP office on 11 November 1983, work started at project site on 14 November 1983 with a meeting with Dr. Vaidyeswaran, Mr. Rao and Mr. Narasimhan, followed by an inspection of the room prepared by RRLH for housing the TGA. Two more days were needed to complete the construction of a steel frame on which the TGA was to be mounted.

The following is a detailed protocoll of the work done during installation:

15.11.1983 - Identification of additional angle steel struts for the framework according to BF piping and construction diagrams;
- Check of all parts of the TGA revealed damages of steam generator heater and some thermocouples;
- First introduction in operation of the TGA-computer to Mr. Prasad.

16.11.1983 - Welding work on the frame performed by RRL workshop;
- Marking of the holes in the frame for fixing the TGA;
- Drilling performed by RRL workshop;
- Repairing of the steam generator heater and replacement of the reactor heater thermocouple in presence of workshop and instrumentation group staff.

17.11.1983 - Continuation of Drilling

- Assembly of the steam generator and the reactor
- Testing of the computer program with evaluated and stored data using a BF-data disc and training of RRL staff in this operation
- Painting of the walls and the frame by RRL

18.11.1983 - Mounting of the TGA

- Transportation of the control rack to the TGA-room
- By evening attachments and pipe system completed up to 80%.

21.11.1983 - Continuation of mechanic work

- Installation of the voltage stabilizer and the main switch by RRL electricians
- Laying of cables for power supply and measuring circuits
- In the morning pressure test at 40 bar, during night at 60 bar
- Check of all cables and connections in the control rack, some needed to be repaired
- Testing of temperature controllers, heating tapes, pressure transducer, valve positioner, level and flow indicators
- Level indicator of the steam generator defective and one temperature controller working irregularly

- Redresses:

  a) Changing of the temperature controller from the expansion valves to the outer heater of the steam generator; this heater is of minor importance, because of the oversized internal heater

  b) Training of RRL staff how to operate the steam generator without the water level indicator

22.11.1983

- Overnight pressure test without any drop in pressure
- Preparation for pressure test at 100 bar
- During unlocking of transport safety bolts of the microbalance the two suspension bands of the counterweight side were broken. Repair by shortening the bands, balance ready for work. This repair normally has to be done by an expert of the microbalance supplier.
- Adjustment of the balance, the sample lock and the reactor in a very accurate vertical position, that the sample basket hanging with a chain on the balance comes in the middle of the heated reaction tube.

23.11.1983

- Zero-point and output-signal of the microbalance calibrated
- After discussion with RRL staff we decided to change the cooling system of the sample lock and the condensate cooler from the proposed open circuit to a closed circuit with a circulating pump and an ice-cooled stock bucket, because RRL cannot ensure in summer a temperature of the city water below the temperature of the air-conditioned room to prevent wetting of the balance.
- Pressure test at 95 bar without any problems
- Putting all temperature controllers in operation
- Checking of all measuring signals with the computer using the program for testing the apparatus conditions.
- Adapters for the pressure reducers are provided by RRL, because of the different cylinder connectors.
- Mounting of the cylinders' holder.

24.11.1983

- Steam generator test at 10 bar
- First no-load test with steam at 10 bar and nitrogen as flushing gas has shown an excellent operation of all parts of the unit.

25.11.1983

- First test with a char made from Indian coal by RRL
- During heating-up the non-isothermal test the computer showed an increasing weight instead of a weight loss. The reason was the faulty installation of the microbalance in a sidewise inverted direction. Redress: Polarity reversal of the output-signal of the microbalance.
- Continuation of the test without any problem.

28.11.1983

- Repeating of the first test at 10 bar steam pressure and a temperature range of 350-1100°C, flushing gas nitrogen. It was not possible to store the data of this test because of a mistake : the sample basket had been lifted into the sample lock before the computer was stopped. After that, we had an intensive discussion about how to carry out a test.
- After completion of a normal test, two more tests were performed under the same conditions. The data of these runs have been stored on the run-data-disc and evaluated with the program "EVALUATE" to calculate the reactions rates from the weight loss curve.

29.11.1983

- Third test at 10 bar steam pressure and a heating rate 10 K/min up to 1100°C. The repeatibility of the first and the third test was very good. The second test has shown an unusual difference, the reason of which could not be found.

- Gasification test at 40 bar steam with nitrogen as flushing gas. In the beginning indication of some irregularities in the weight loss curve. We supposed condensation of steam on the chain, nevertheless the test was carried on with heating-up to the final temperature of 1100°C. After the test, opening the sample lock showed a lot of water inside, wet chains and wet tubes, but an intact and dry microbalance. These findings confirmed that for 40 bar steam tests helium or hydrogen as flushing gas have to be used.
  After cleaning and drying the unit was ready for the next test.

30.11.1983

- Changing of the gas lines to the cylinders and pressure regulators: a) Flushing gas for the balance to the hydrogen cylinder; b) One of the tubes for reacting gases to the nitrogen cylinder. Nitrogen is necessary to flush the unit oxygen-free before starting with hydrogen and after the test to flush hydrogen-free before opening the sample lock.

- Instructions and intensive discussion how to operate the unit with hydrogen, not only as a flushing gas but also as a reacting gas.

- Performance of two tests under following conditions:
  40 bar steam, hydrogen as a flushing gas for the balance, heating rate 10 K/min, with the final temperature 1100°C and isothermal at 1000°C. Both tests without any problems, data were evaluated and stored on the disc.

1.12.1983

- During preparations for a test with a steam/hydrogen-mixture, to show the inhibition of the product gas hydrogen on the rate of steam gasification, we localized a fault in the hydrogen flowmeter. Opening showed condensed water in the meter, but also after cleaning and drying it didn't work. We removed the flowmeter and built in a bridging tube between the connectors. The flowmeter has to be repaired by the supplier. To avoid further wetting of the flowmeter line, we changed the hydrogen tube from the steam generator inlet to the gas preheater inlet. Now a gas/steam-mixture can be made by mixing the gas streams before entering the reactor.

- For the next test the gas lines had to be changed again because of the failing flowmeter: a) reacting gas hydrogen to the second reacting gas line, provided and calibrated for carbon dioxide; b) flushing gas nitrogen to the non-metered gas line. Changing of the gas tubes was sometimes difficult, because of the different heights and the different connections of the cylinders for hydrogen, nitrogen and carbon dioxide. The adapters between the cylinders and the pressure regulators, necessary because of the different threads, were made by the RRL workshop very promptly and accurate.

- The test at 900°C and a total pressure of 41 bar, partial pressure of steam 32.5 bar and of hydrogen 8.5 bar, has been performed without any problems. The data of the test were evaluated and stored on the disc.

- In the evening we started another test to show the hydrogasification of a char. The reaction rate in hydrogen is a function of the hydrogen pressure and therefore the pressure was set to the maximum. The maximum pressure of 81 bar in this test was calculated from the flow rate of hydrogen, the intended duration of the test and the gas volume in the cylinder. The temperature was set to 900°C and the sample was lowered into the preheated reactor to achieve the maximum heating rate of > 500 K/min. The test has been performed without any problems.

2.12.1983

- Preparation of a test with carbon dioxide at 31 bar and nitrogen as flushing gas. These are very easy conditions for the unit and appropriate for the first tests to be done by RRL staff without assistance.

- During the test again a failure of the thyristor for the internal reactor heater was observed. This happened irregularly and it is unclear whether it depends on the duration or on the load. The redress was to unlock the thyristor from the base plate and lock again. It was not possible to find out which connections failed and so we couldn't repair anything. My recommendation was to work with the thyristor until the suppliers delivered a spare. In the meantime the RRL staff can make tests and achieve good results if they observe the temperature curve on the screen very carefully and do in the case of a failure in the described way. The checks and the removal of the thyristor were done in the presence of persons from the RRL instrumentation group and the laboratory staff.

- The test was accomplished without further problems and the data were evaluated and stored.

- In the afternoon a discussion took place with Dr. Vaidyeswaran, Mr. Mallikurjunan, Mr. Narasimhan, Mr. Prasad and other staff members of the RRL coal division about the progress of the TGA installation, the performed test, the observed faults with the remedial actions and the test program for the next weeks.

- Summary of the discussion: The TGA is completely tested and can be operated. Thirteen tests, most of them under different conditions, were performed successfully. Instructions on the maintenance and the repair of the most important parts of the unit were given to the staff concerned, for example:
  calibrating of the microbalance, cleaning and drying of the sample lift, replacement of the internal heaters, replacement of internal thermocouples, setting of temperature controllers and checking of input and output signals on the terminal strips of the control panel. Some parts have to be repaired or to be replaced by the supplier: thermal mass flowmeter, temperature controller 0-600°C, thyristor for the reactor heater, water level indicator for the steam generator and cooling water flow indicator. The missing discettes with the three programs, MEASURE, EVALUATE and GRAPH, and additional

data discs should be sent as soon as possible. Before doing a test without any assistance the operator of the TGA, Mr. Prasad, should make his own operation manual from the instructions and explanations received during the tests. To be on the safe side it is better that Mr. Prasad and Mr. Narasimhan, who was trained on a similar TGA at Bergbau-Forschung, are working together on this unit. It was recommended that the next test should be done with carbon dioxide under different pressures, heating rates and temperatures.

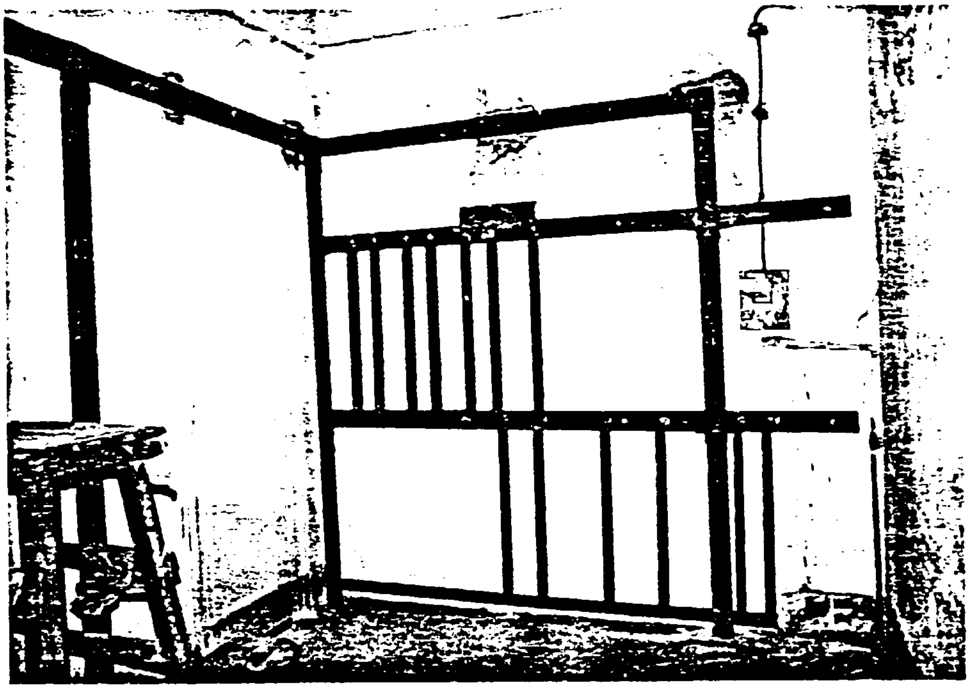
3.12.1983

- First tests performed by Mr. Prasad and Mr. Narasimhan with carbon dioxide under 31 bar. With the exception of one thyristor failure the test was carried out successfully. The recommended proceeding for the next tests was confirmed.

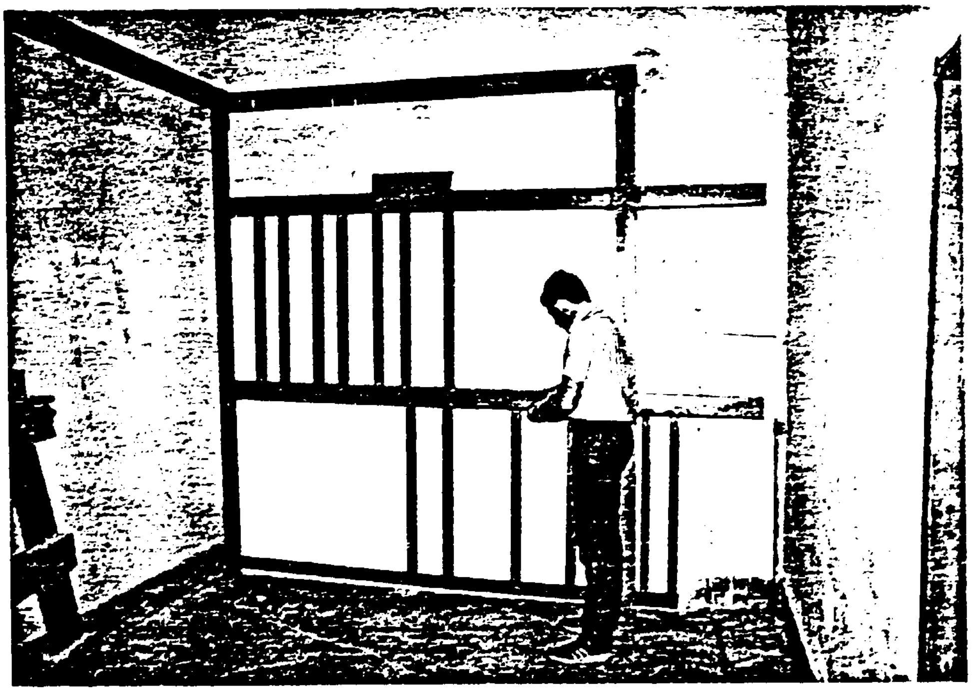
5 - 9 December 1983

- Preparation of draft final report, debriefing at UNIDO, Vienna, Austria.

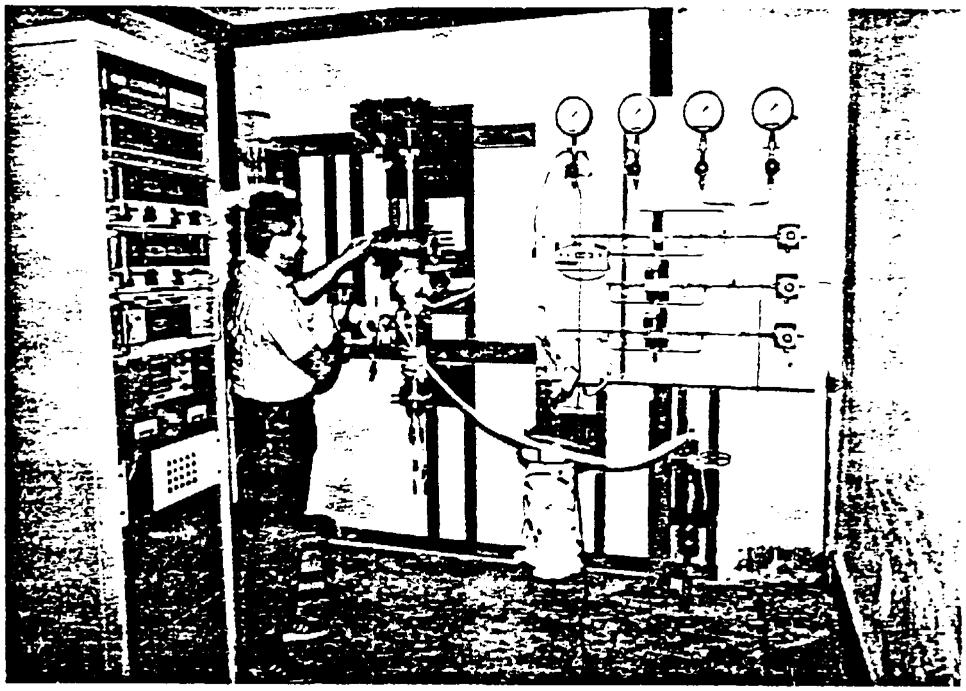
- Arrival Düsseldorf, FRG, on 9 December 1983.



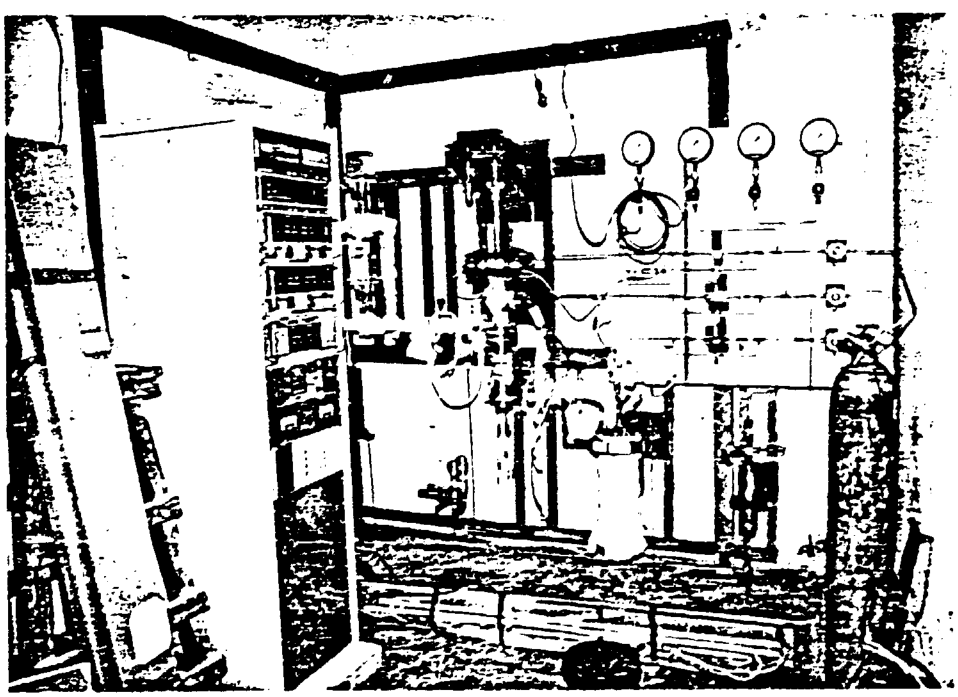
16.11.1983 Frame with the additional angle steel struts for fixing the TGA



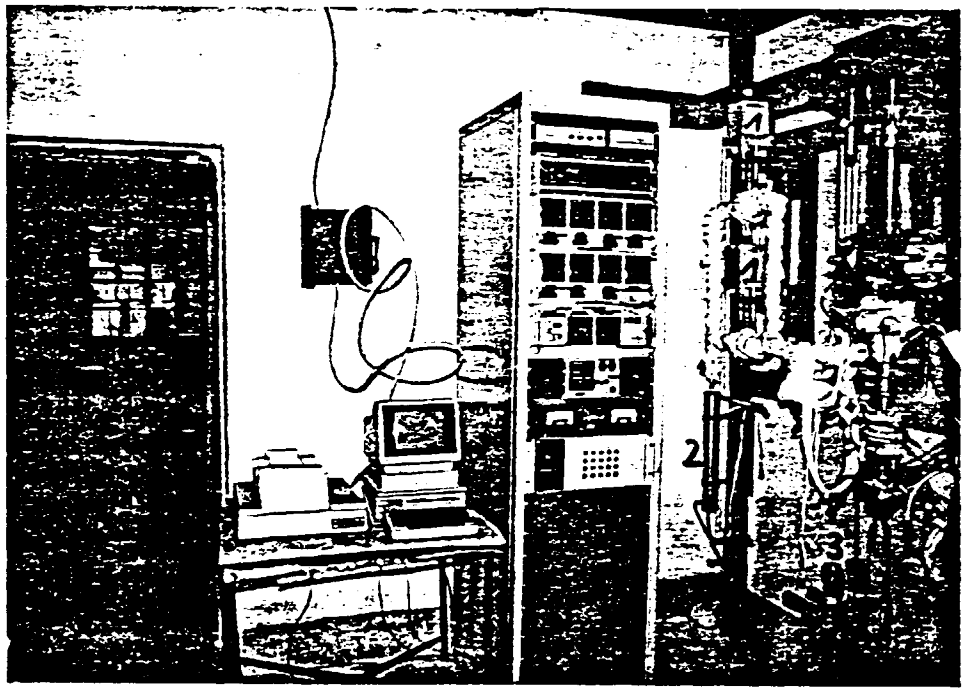
18.11.1983 Painted frame and room before mounting



19.11.1983 During installation the TGA

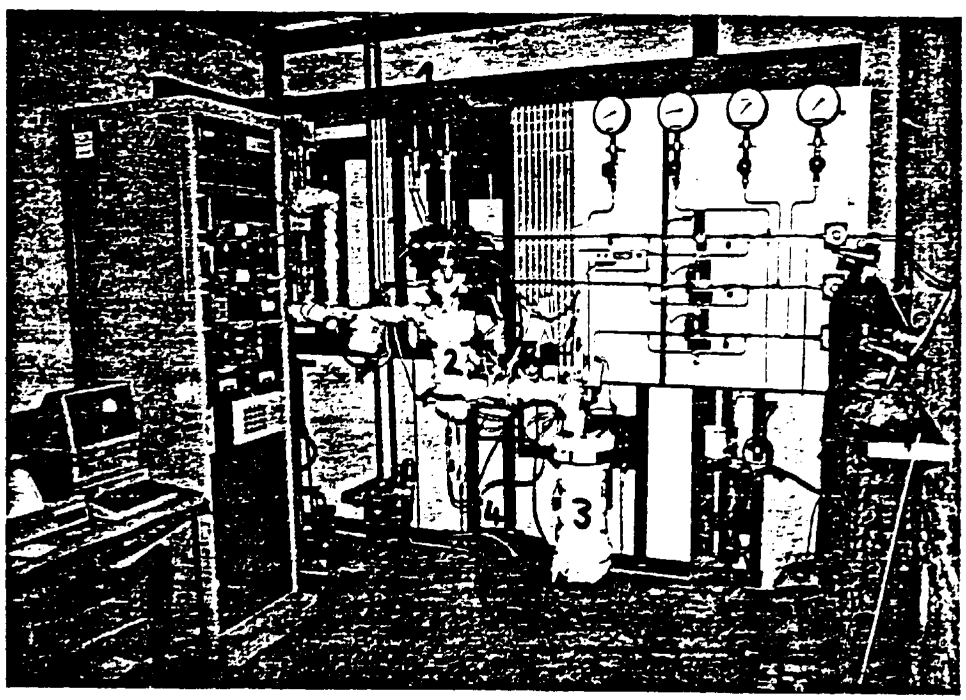


21.11.1983 During electrical installation



23.11.1983 Control rack and Computer for the TGA

Left side of the unit with expansion valves (1), condensate cooler (2), and safety valves (3)



24.11.1983 During no-load test with steam at 10 bar

Middle part of the unit with microbalance (1), reactor (2), steam generator (3) and cooling pump with stock bucket (4)

3.12.1983 After the final test at 30 bar CO_2 pressure

from the left: Dr. Vaidyeswaran, Mr. Prasad,
Mr. Sudharka Rao, Mr. Narasimhan

4. Performed test runs

In the test and training time from 24 November to 3 December 1983 we performed one no-load test and 14 tests with Indian chars (see Table 7). The first tests have been performed to check the functioning of all parts of the unit including the data acquisition and evaluation. Also detailed information were given to the RRL staff about the mode of operation of the instruments and the sequence of handling of the valves and regulators. The general handling of the computer is described in the manuals provided by the supplier. The description of handling the computer in connection with the TGA is part of the program. This information can be displayed on the screen or printed out before starting the program. The order of commands to the computer is fixed in the program as a dialog, so that the operator can see for which commands the computer is waiting.

After setting the controllers to the desired set points and inserting the sample the operator normally starts the test run with loading the program MEASURE. In this program the temperatures, flow rates and the pressure can be checked, displayed on the screen and printed out (see Tables 8 and 9). In case of a steam test the program is continued with measuring the signal of the condensate balance from which the condensate rate is calculated. The course of the condensate rate is displayed and can be printed out (see Fig. 5).

An example for a non-isothermal steam test run is seen in Fig. 6 showing the weight loss curve, the course of the condensate rate and the increasing temperature curve. This figure is a copy of the diagram shown on the screen. After the test all relevant data are stored on the disc for further evaluation using the program EVALUATE.

In this program a polynomial is fitted to the measured weight loss curve and corrections are made according to the dynamical boyouncy and the check of the theoretical and the determined amount of residue (see Fig. 7 and Table 10). At the end of the calculations time, temperature, ash-free sample weight, three different reaction rates, and burn-off, that means the carbon consumption, are printed out (Table 11). The rate r' is defined as the consumption of carbon per minute related to the input weight, the rate r" as the percentage of gasified carbon per minute related to the instantaneous carbon content of the sample and the rate r_s as the percentage of gasified carbon per minute related to the instantaneous carbon content with the power 2/3. The equation for the temperature curve and the coefficients of the polynomial are stored on a disc.

With the program GRAPH the reaction rates of one or several tests can be plotted against temperature, time or burn-off.

Date	Run-No.	Sample	Gasifying agent	Flushing gas	Pressure bar	Temperature °C	Heating rate K/min	Remarks
24.11.83	0	no load	steam	nitrogen	11			
25.11.83	1	GDK-10	steam	nitrogen	11	1100	10	Reversal of the balance signal
25.11.83	2	GDK-10	steam	nitrogen	11	1100	10	
28.11.83	3	CGP-21.11.83	steam	nitrogen	11			Not stored
28.11.83	4	CGP-21.11.83	steam	nitrogen	11	1100	10	
28.11.83	5	CGP-21.11.83	steam	nitrogen	11	1100	10	Repeatibility test of No. 4
29.11.83	6	CGP-21.11.83	steam	nitrogen	11			Computer error
29.11.83	7	CGP-21.11.83	steam	nitrogen	11	1100	10	Repeatibility Test of No. 4
29.11.83	8	CGP-21.11.83	steam	nitrogen	41	1100	10	Condensation in sample lock
30.11.83	9	CGP-21.11.83	steam	hydrogen	41	1100	10	
30.11.83	10	CGP-21.11.83	steam	hydrogen	41	1000	isothermal	
01.12.83	11	CGP-21.11.83	steam/hydrogen	hydrogen	32.5/8.6	900	isothermal	
01.12.83	12	CGP-21.11.83	hydrogen	hydrogen	81	900	isothermal	
02.12.83	13	CGP-21.11.83	carbon dioxide	nitrogen	31	1100	10	
03.12.83	14	CGP-21.11.83	carbon dioxide	nitrogen	31	1100	10	

Table 7

Table 8

Temperatures Heatings

Time m:s	Cycle	Bottom H 109 °C	Middle H 110 °C	Top H 111 °C	Gas Preh H 406 °C	Filter H 601 °C	Exp-Valv H 602 °C	St.-Valv H 206 °C
115:15	247	344.6	345.1	345.[illegible]	345.9	343.7	318.1	344.0
115:25	248	344.5	345.2	345.0	345.5	343.6	317.9	344.3
115:35	249	345.0	345.3	344.6	343.5	343.5	317.6	344.1
115:45	250	344.9	345.2	344.6	345.5	343.5	317.9	344.0
115:55	251	344.7	345.3	345.1	345.4	343.5	318.9	343.9
116:05	252	344.2	345.1	345.2	343.3	343.6	319.6	343.9
116:15	253	343.8	345.3	345.0	345.1	343.6	319.8	343.8
116:25	254	343.9	345.4	344.8	345.7	343.8	319.7	344.2
116:35	255	344.2	345.3	344.4	343.6	343.8	319.2	344.1
116:45	256	344.6	345.4	344.3	344.5	343.8	318.7	344.1
116:55	257	344.9	345.5	344.7	346.4	343.7	318.1	344.1
117:05	258	345.2	345.5	345.0	344.5	343.7	317.6	344.2
117:15	259	344.6	345.2	344.8	344.3	343.8	317.2	344.1

Temperatures Reactor

Time m:s	Cycle	Sample T 265 °C	Gas I T 215 °C	Gas II T 165 °C	Tube TIRC 105 °C	Bottom H 109 °C	Middle H 110 °C	Top H 111 °C
114:05	240	337.9	336.5	333.9	349.1	346.9	345.2	344.6
114:15	241	338.6	337.1	334.4	351.8	347.1	345.3	345.0
114:25	242	339.8	338.0	335.1	351.6	346.7	345.3	345.1
114:35	243	339.9	338.2	335.3	350.0	346.2	345.3	344.6
114:45	244	339.2	337.7	335.0	348.3	345.6	345.2	344.4
114:55	245	338.2	337.0	334.4	346.9	345.0	345.4	344.7
115:05	246	337.3	336.2	333.7	348.4	344.7	345.2	345.0
115:15	247	338.5	337.1	334.4	351.6	344.6	345.1	345.1
115:25	248	340.1	338.5	335.4	352.1	344.5	345.2	345.0
115:35	249	340.5	338.7	335.8	350.7	345.0	345.3	344.6
115:45	250	339.8	338.3	335.4	349.0	344.9	345.2	344.6
115:55	251	339.3	337.9	335.3	347.3	344.7	345.3	345.1
116:05	252	338.4	337.1	334.6	347.4	344.2	345.1	345.2

Gas flows

Time m:s	Cycle No	Flushing Gas FIR 505 % of max.	Gas 1 FIR 405 %	Gas 2 FIR 305 %
116:35	255	51.8	1.1	-.1
116:45	256	51.7	1.1	-.1
116:55	257	51.7	1.1	-.1
117:05	258	51.8	1.1	-.1
117:15	259	52.0	1.1	-.1
117:25	260	51.9	1.1	-.1
117:35	261	51.8	1.0	-.1
117:45	262	51.8	1.1	-.1
117:55	263	51.8	1.1	-.1
118:05	264	51.9	1.1	-.1
118:15	265	51.8	1.0	-.1
118:25	266	51.7	1.1	-.1
118:35	267	51.6	1.1	-.1

Temperatures Steam-Generator

Time m:s	Cycle No	inside TIRC 202 °C	outside H 203 °C
114:55	245	282.4	268.7
115:05	246	282.5	268.7
115:15	247	282.6	268.6
115:25	248	282.6	268.6
115:35	249	282.5	268.6
115:45	250	282.6	268.6
115:55	251	282.5	268.6
116:05	252	282.5	268.6
116:15	253	282.4	268.6
116:25	254	282.5	268.6
116:35	255	282.4	268.6
116:45	256	282.4	268.6
116:55	257	282.6	268.6

Pressure

Time m:s	Cycle No	Pressure P 101 bar abs.
116:15	253	61.4
116:25	254	61.4
116:35	255	61.5
116:45	256	61.5
116:55	257	61.5
117:05	258	61.4
117:15	259	61.4
117:25	260	61.4
117:35	261	61.4
117:45	262	61.4
117:55	263	61.4
118:05	264	61.4
118:15	265	61.4

Table 9

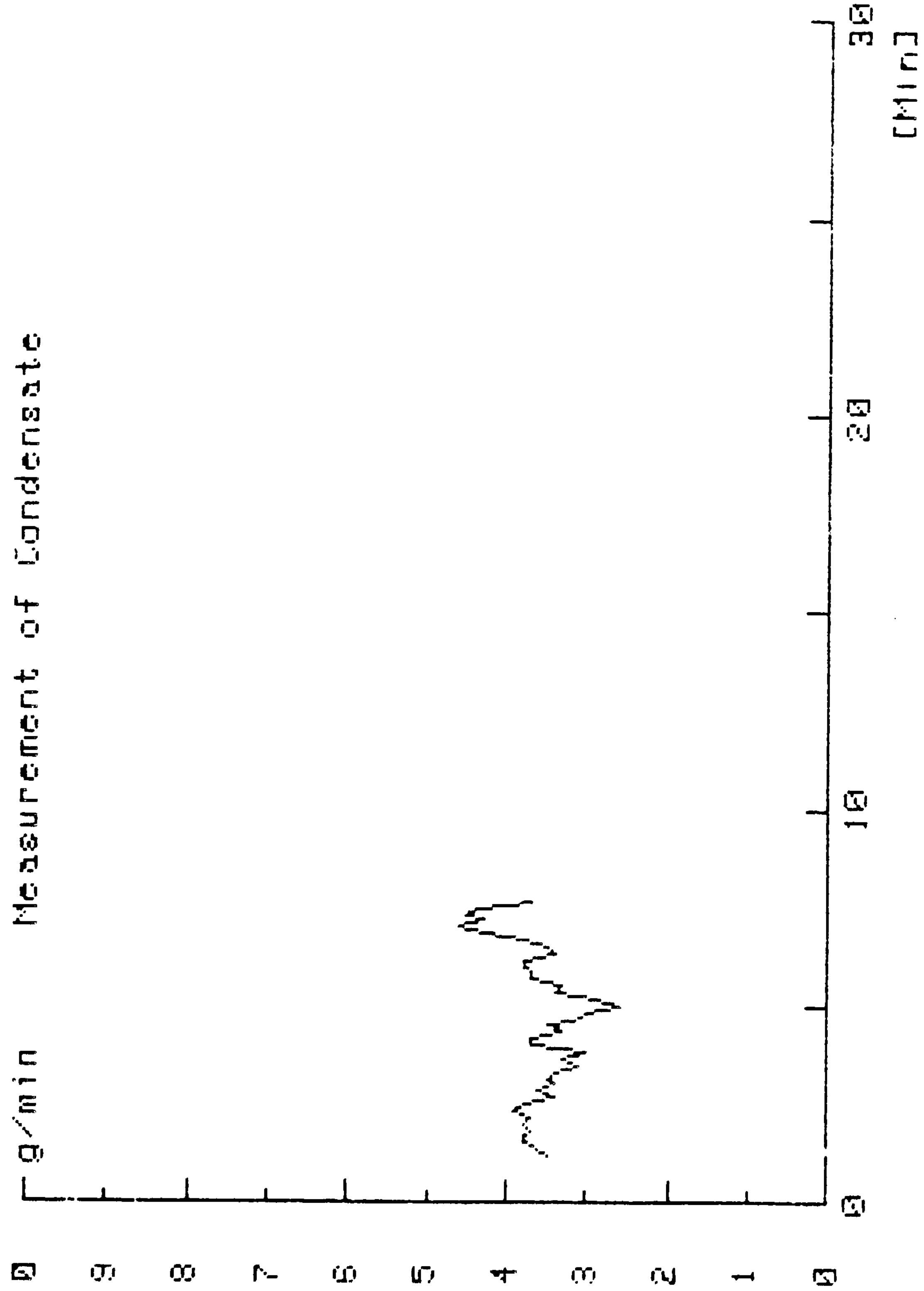


Fig. 5

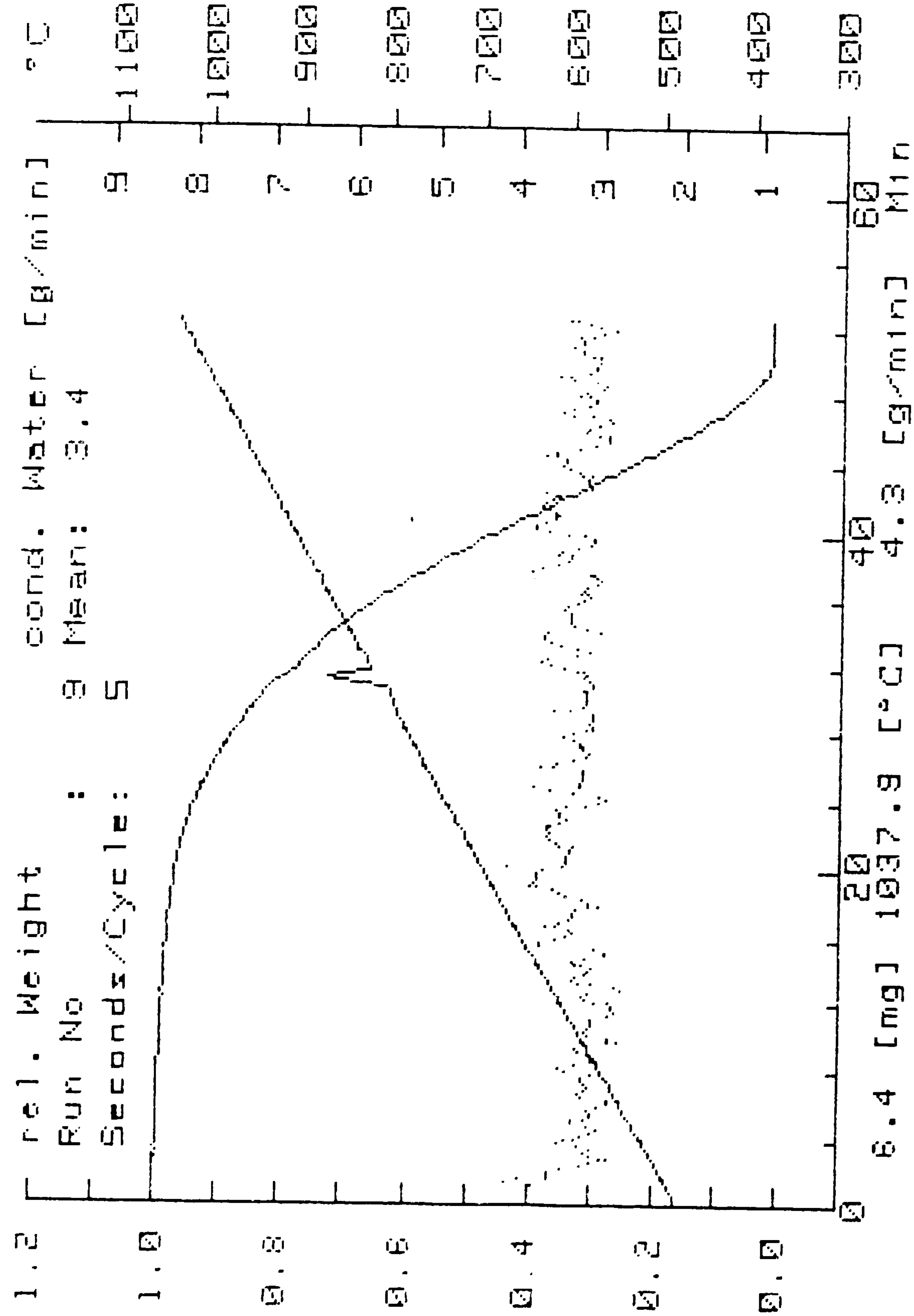

rel. Weight
Run No : 9
Seconds/Cycle: 5
Cond. Water [g/min]
Mean: 3.4
6.4 [mg] 1037.9 [°C] 4.3 [g/min]
Min
°C
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0
20
40
60
1
2
3
4
5
6
7
8
9
300
400
500
600
700
800
900
1000
1100

Fig. 6

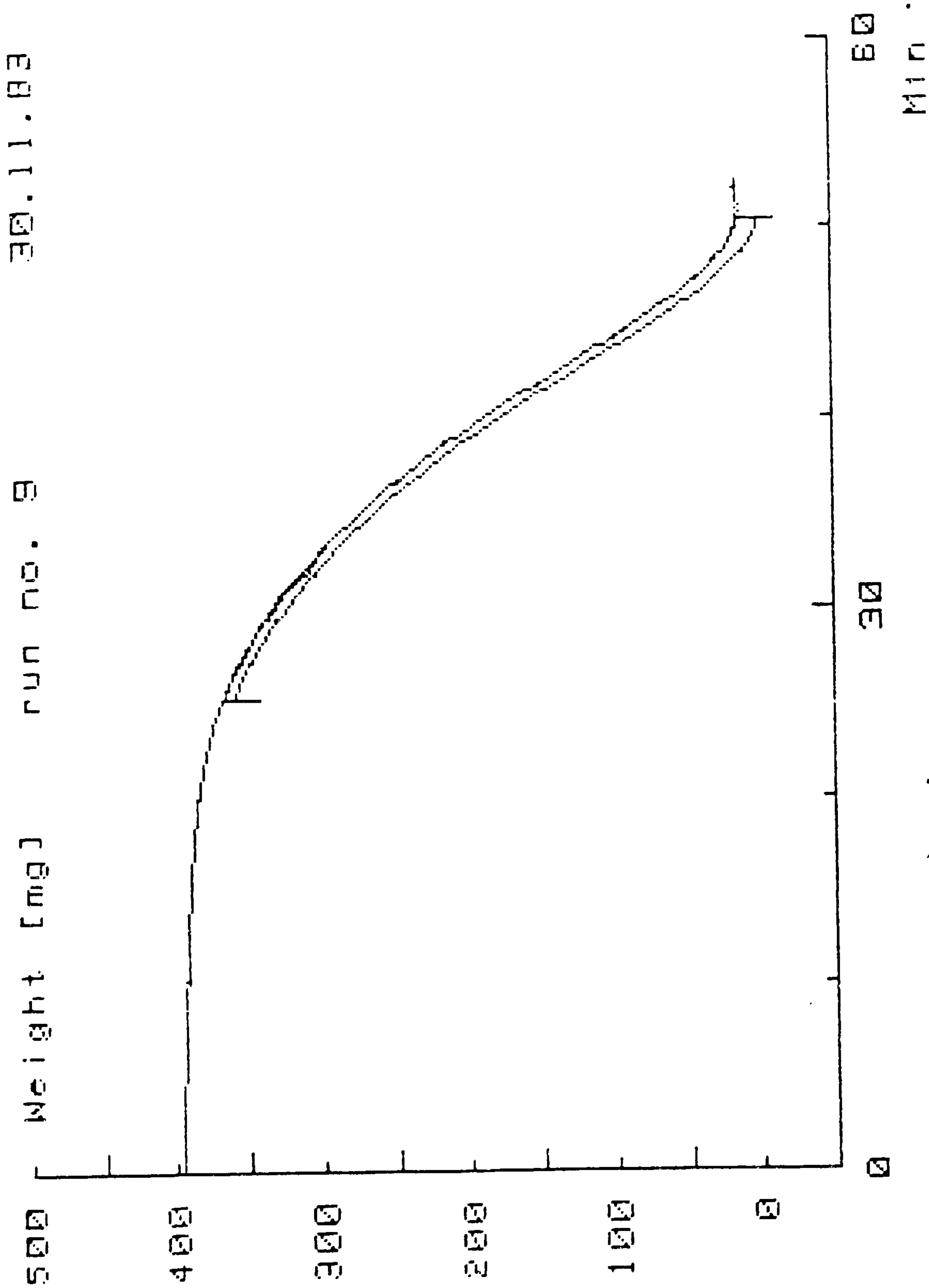


Fig. 7

Run No. 9

Evaluation
Experimental Conditions

Date of evaluation: 30.11.83	Date of run:	30.11.83
CGP-21.11.83		
nonisothermal mode	10.0	[K/min]
Weight of char inserted	513.1	[mg]
Weight of sample holder	3724.7	[mg]
Corrected content of ash in char (raw)	21.6	[%]
Content of moisture in char	1.1	[%]
Total pressure	41.0	[bar]
Composition of the gasifying agent		
Partial pressure of H2O	41.0	[bar]
Partial pressure of CO2	0.0	[bar]
Partial pressure of CO	0.0	[bar]
Partial pressure of H2	0.0	[bar]
Partial pressure of CH4	0.0	[bar]
Residiue	111.0	[mg]
Carbon in residiue	0.0	[mg]
Fitting of the polynominal		
Coefficients:		
A(0) = 3.64878559640E+02		
A(1) = -3.21520558051E+00		
A(2) = -1.29620588331E+00		
A(3) = 1.51972492935E-01		
A(4) = -1.19475466565E-02		
A(5) = 3.91601579512E-04		
A(6) = -4.06303371947E-06		
Standard deviation	.78	[mg]
Shift of time axis	-25.2	[min]
Fault of the dynamical boyouncy	14.4	[mg]
Validity of the polynominal:		
Lower bound	25.2	[min]
Upper bound	50.3	[min]
Average rising of temperature	10.7	[grd/min]
Corrected coefficients:		
A(0) = 3.57867086971E+02		
A(1) = -3.49477535543E+00		

Table 10

Run No. 9

Time min:sec	Temp. grd C	Weight mg	r´ -mg/min	r´´ %C/min	rs %C/min	burn off %
25:00	750.0	358.4	3.050	.851	.823	9.596
25:30	755.1	356.6	4.310	1.209	1.167	10.062
26:00	761.5	354.1	5.365	1.515	1.459	10.674
26:30	766.5	351.2	6.248	1.779	1.709	11.408
27:00	772.2	347.9	6.988	2.009	1.923	12.244
27:30	777.3	344.3	7.612	2.211	2.110	13.166
28:00	781.9	340.3	8.145	2.393	2.275	14.160
28:30	786.9	336.1	8.609	2.561	2.424	15.217
29:00	792.3	331.7	9.022	2.720	2.563	16.330
29:30	794.7	327.1	9.403	2.875	2.696	17.492
30:00	798.4	322.3	9.766	3.030	2.828	18.701
30:30	801.4	317.3	10.124	3.190	2.962	19.955
31:00	832.7	312.2	10.487	3.359	3.102	21.254
31:30	851.4	306.9	10.865	3.541	3.251	22.601
32:00	821.9	301.3	11.264	3.738	3.411	23.996
32:30	826.6	295.6	11.689	3.955	3.586	25.443
33:00	835.9	289.6	12.144	4.193	3.776	26.945
33:30	839.8	283.4	12.631	4.456	3.985	28.507
34:00	846.0	277.0	13.149	4.747	4.212	30.133
34:30	851.0	270.3	13.699	5.068	4.461	31.825
35:00	855.8	263.3	14.277	5.422	4.731	33.589
35:30	862.1	256.0	14.879	5.812	5.024	35.427
36:00	865.6	248.4	15.502	6.241	5.340	37.343
36:30	873.0	240.5	16.139	6.711	5.681	39.338
37:00	876.7	232.3	16.783	7.226	6.046	41.414
37:30	882.2	223.7	17.427	7.790	6.437	43.571
38:00	887.6	214.8	18.062	8.407	6.854	45.809
38:30	893.7	205.7	18.679	9.083	7.298	48.126
39:00	897.2	196.2	19.269	9.823	7.769	50.520
39:30	903.0	186.4	19.821	10.634	8.269	52.985
40:00	910.0	176.4	20.324	11.525	8.797	55.517
40:30	913.4	166.1	20.769	12.505	9.357	58.109
41:00	918.4	155.5	21.142	13.588	9.948	60.753
41:30	923.3	145.0	21.435	14.788	10.574	63.439
42:00	929.4	134.2	21.634	16.123	11.236	66.155
42:30	934.4	123.3	21.729	17.618	11.938	68.891
43:00	937.1	112.5	21.708	19.301	12.682	71.631
43:30	945.6	101.6	21.561	21.212	13.476	74.361
44:00	949.4	90.9	21.277	23.399	14.323	77.064
44:30	955.7	80.4	20.846	25.930	15.234	79.722
45:00	960.3	70.1	20.259	28.895	16.219	82.316
45:30	966.0	60.2	19.505	32.421	17.293	84.825
46:00	970.3	50.6	18.578	36.690	18.477	87.228
46:30	976.0	41.6	17.468	41.976	19.801	89.503
47:00	980.7	33.2	16.170	48.710	21.310	91.626
47:30	985.4	25.5	14.678	57.613	23.077	93.574
48:00	991.1	18.6	12.987	70.003	25.226	95.320
48:30	996.9	12.5	11.094	88.581	28.002	96.841
49:00	1000.1	7.5	8.995	120.051	31.978	98.110
49:30	1005.9	3.6	6.691	187.811	39.047	99.101
50:00	1011.9	.8	4.192	500.218	64.144	99.789

Table 11