

DASFOS C_{zr}, s.r.o. – Technology-innovation center Ostrava

Bozkova 45/914, 702 00 Ostrava 2-Privoz

Phone: + 420 59 6612092, Fax: + 420 59 6612094,

E-mail: dasfos@dasfos.com Web: <http://www.dasfos.com>



RF-33/KK

**Measuring System for doing CRI/CSR tests of coke
according to ASTM D 5341, ISO 18894 Standards**

with the possibility of

laboratory production of about 10 kg coke

Technical Information

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General

The base RF-33 system is for testing coke quality by the standard CRI/CSR tests, according to the ASTM D 5341 or ISO 18894 Standards.

The RF33/KK system is unique not only in its technical parameters but also as the scope of its possible applications is concerned. In the same equipment carbonization of the tested coal (coal mixture) can be done as well as the subsequent testing of the product quality by determining CRI/CSR values. Quenching of the produced coke is carried out directly in the retort using either so called dry quenching method by an intensive flow of gaseous nitrogen, natural cooling with selected cooling rate, or a combination of both methods. In this way, the laboratory procedure of producing coke sample from the tested coal or coal mixture approximates to the normal conditions of industrial coke production in coking batteries.

Together with other parameters of coal quality, also quite close correlation can be determined between CRI/CSR parameters from this test and those for the coke produced in an industrial coking battery from identical coal mixture. In this way, based on the laboratory tests, quality of the industrially produced coke (i.e. its CRI/CSR values) can be predicted for different coal mixtures intended as the coke oven charge. Thus, the RF33/KK system can be utilized, among others, in the coal charge preparation process (selecting and mixing of coal sorts) for the real coking process in coking batteries.

1. Technical description of individual parts of the RF-33/KK system

- a) Vertically openable test furnace type WA 1100/KK for CRI test
- b) OB-2 device with a tumbler for determining the CSR value
- c) CO₂ and N₂ gas flow control
- d) Computer control system SPW-2/KK
- e) Retort for CRI test
- f) Retort for production of coke
- g) Retort cooling box
- h) Combustion control and CO leakage detection
- i) Accessories for coal preparation for coking
- j) Accessories for preparation of samples for CRI/CSR tests

1.1 Vertically openable test furnace type WA 1100/KK

Technical parameters of vertically openable test furnace WA 1100 meet all requirements of the ISO 18894 Standard as well as requirements of ASTM D 5341 Standard, and enable production of 10 kg coke for testing. The construction of furnace allows the operators a comfortable and safe handling with test retort. Temperature in each heating zone is separately measured and regulated using special control algorithm. This ensures achievement of stable prescribed temperature in the area of test sample, at least in the 320 mm zone (four times the sample height) during entire test duration (in accordance with ISO and ASTM Standards). Placement of measuring thermocouples directly in the area between heating elements and the retort ensures quick reaction of the regulator on the change of temperature in test retort heating area due to changing external conditions, such as supply voltage drop, changes of ambient temperature, air flow changes, etc. A significant advantage of heating modules is their low thermal inertia, because heating elements do not heat working area indirectly

through heating of other carrying elements, but they act on the working area directly. Regulators are part of computer control system and are implemented into the control software by use of sophisticated algorithms. The control by software allows for comfortable tuning and making changes of heating, e.g. for experimental purposes.

The test furnace uses heating modules with resistance heating elements built in vacuum formed high-quality insulation material.

Use of KANTHAL prefabricated modules has some considerable advantages over the other furnace designs. The main features include:

- High efficiency of heat transfer to the furnace area directly from heating elements made of KANTHAL A1 material;
- Low weight of module combined with high thermal insulation capability and high specific output;
- Simple installation of individual modules, making disassembly and respective replacement easier, too;
- Favourable price, no need for stocking spare modules as they are mass produced and delivered by manufacturer in short delivery terms. User is not dependent on the supplier of RF-33-KK system.

Temperature measurements in the furnace area between heating elements and test retort are ensured by use of sheathed thermocouples. As power supplies for individual heating sections thyristor units are used, designated by the manufacturer specifically for this purpose.

Considerable advantage of this furnace unit is its small headroom required, as loading and unloading of retort does not require the use of a crane crab.

Opening and closing of furnace, as well as loading and unloading of the CRI or coking retorts from the furnace, are carried out automatically by servo drives controlled by the control system.

1.2 OB-2 device with a tumbler for determining the CSR value

Handling with sample after CRI test and the detailed description of CSR test are given in ISO 18894 or ASTM 5341 Standards, so there is no need here to repeat a description of procedure of sample preparation for this test after CRI reaction. The design of this equipment meets all requirements of ISO 18894 or ASTM 5341 Standards.

The OB-2 device for CSR tests is equipped with an electric motor and regulation of actual speed with an accuracy of +/- 0.03 rpm, using a feedback controlled frequency converter and a power regulator. Rotational speed, number of revolutions and operational time are measured electronically by the control system. This control system also monitors and records the actual rotational speed of the tumbler. Actual values are shown on a display.

The revolving tumbler is furnished with a protective Plexiglas cover.

1.3 CO₂ and N₂ gas flow control

The gas flow control equipment here stands for the manifold of CO₂ and N₂ gases inlet into test furnace, quick couplings, feed-check, and regulation and closing valves. Measurement of actual flow rate is done using mass flow meter with internal electromagnetic proportional valve. Gas flow rates of both N₂ and CO₂ gases are regulated with accuracy better than 1 %, for the actual volumes and for the time sequence, too. This is in accordance with the explicit prescription of ISO 18894 or ASTM D 534 Standards.

Differential pressure between gas pressure on the retort inlet and gas pressure on the retort outlet is measured for monitoring the test course and for detection of possible backpressure on the outlet. After hanging the retort onto the boom, on which retort automatically loads into test furnace, and after connecting inlet and outlet gas pipes by quick couplings, gas tightness inspection of retort shall be done.

It is assumed that gases connected to the furnace have the purity and further physical parameters as defined in ISO 18894 or ASTM D 5341 Standards.

1.4 Computer control system (SPW-2/KK type control)

Complete CRI testing, as well as CSR test and procedure of production 10 kg coke are controlled automatically by control computer. Individual steps and test procedures are automatically and continually backwards monitored during entire test. All measurements are computer processed through interface measurement cards. By use of power actuators, the computer system controls the course and stability of temperatures in individual sections of test furnace, in accordance with prescriptions of ISO 18894 or ASTM D 5341 Standards for CRI testing, and also for the 10 kg coke making procedure.

Based on measurement of actual temperatures in test retort, individual heating sections are regulated in order to assure stable temperature of test sample in the retort and to keep temperature on the value prescribed by the Standard. The control system allows for checking the distribution of temperature field across the height and through the cross-section of test sample. For this, four sheathed thermocouples are installed in each quadrant for analysis of the temperature field.

Temperature regulation is solved in a comprehensive manner using digital regulation algorithms such as multiparameter regulation with additional variables. This are, for example, supply voltage value, ambient temperature, actual gas flow rate, and temperature values from temperature field measurement are. Algorithms used are adjustable, using the recorded parameters and response histories from previous tests for that.

The control computer also directly controls carrying out of CSR test, i.e. it does the control of actual rotational speed of test tumbler, measurement of time and number of revolutions.

The testing workplace is equipped with electronic weighing scale for weighing individual components of the tested sample before and after CRI and CSR tests. For automatic recording of data, the scale output is connected to the control system. This solution allows for effective processing of all measured values, its long-term archiving, and compilation of final test protocols.

A special safety sensor for monitoring leakage of dangerous CO gas is installed in the area of furnace unit. The monitoring is important especially during the testing process itself. In case of increased CO concentration in air, i.e. upon leakage of this dangerous gas from retort or due to other non-tightnesses, alarm signal is turned on.

The system software allows for remote monitoring and control of the testing operations from a distant PC computer (if having necessary access rights, of course) through ETHERNET network connection.

Printing of test protocols and evaluation and compilation of files from the tests can be done on these distant computers, too.

1.5 Retort for CRI test

This retort, 900 mm long, is made of stainless Cr-Ni steel. It is a thick wall tube of 78 mm

diameter and 3 (5) mm wall thickness.

Its upper flange cover for gas-tight closing is equipped with a special opening which is independently closable. Through this opening the test sample is filled into retort. The hole diameter is such that it is not possible to insert into retort any coke pieces of larger dimensions than the Standard permits. The upper cover also includes outlets for the outgoing technological gases. Outlets are fitted with quick couplings with dividing valves.

The lower cover is equipped with a special outlet used for adjustment and holding of grates (241 holes D 2.5 mm) in prescribed distance in retort, both for supply of technological gases, and for connecting cables of individual thermocouples. Such design features some advantages. Firstly, material of retort is not corrupted in the area of grid location, where the coke sample lies during test. Otherwise, this area is critical for retort wear and tear. Other advantages include good wire conduct to thermocouples, which is not influenced by hot exhaust gases, thus allows more stable measurement of temperature in the coke sample.

Lower part of retort is filled with ceramic Al_2O_3 balls for preheating the CO_2 and N_2 gases. Ceramic balls lie on the lower grate and fill the space between both grates.

Temperature sensors used are sheathed thermocouples. One of them is of 3 mm and four of 2 mm diameter, all ended with an OMEGA connector.

The central thermocouple is located in retort axis 55 mm above the grate which roughly corresponds with the center of coke sample. Thermocouples for measuring the temperature field in coke sample are located in various heights and at various distances from the axis, always 90° from each other on the circumference. First thermocouple is 25 mm above the plate and 12.5 mm from the axis, the second one then 40 mm above the grate and 20 mm from the axis. The third one is 70 mm above the grate and 27.5 mm from the axis, and fourth one 85 mm above the grate and 35 mm from the axis. If needed, different height levels can be easily adjusted.

1.6 Retort for production of coke

This retort is 1100 mm long and is made of a thick-wall, Cr-Ni stainless heat-resisting steel with 130 mm diameter and 5.5 mm wall thickness. The retort has upper and lower covers. The partition screen, on which the coal charge is placed, is situated at such position that ensures the charge to be in the zone of stabilized temperature.

The upper cover can be gas-tightly closed. Through this cover coal charge is inserted into the retort. There is also an outlet for escaping gaseous products of coking.

The lower cover has a special outlet which serves for adjusting and holding the coal charge at prescribed position in the retort, and also for outlet of measuring thermocouple. This design is the original one and has some advantages. First, the material of retort is not influenced at the position where coal sample is placed. Further on, the wires of measuring thermocouple are not influenced by hot escaping gases so that more stable measurement of temperature inside of coal sample being coked is ensured.

Combustion of gaseous products from the coking process is done directly at the retort outlet by a special burner. Measurements were made concerning concentration of selected gaseous constituents (CO , CO_2 , HC , and O_2) in the already burnt waste gases during coking. The concentrations of them were minimal, deeply under allowed values.

1.7 Retort cooling box

The retort cooling box is made of steel, is movable and vertically openable. Fast cooling of retort is assured using a radial blower. Thanks to movability of the box, it is easy to place the retort in the opened box. The retort, automatically unloaded from opened furnace and further transported by a rail, is easily placed into prepared cooling box for quick cooling down. During cooling down, retort stays connected to N₂ supply for preventing from all reactions. Also temperature measurement in the sample stays connected. This way of handling with retort during cooling down is very safe and comfortable for the operator.

During cooling down, temperature in the retort area is measured, and upon cooling down to preset temperature (60°C), acoustic and optical alarms are turned on, signaling to the operator that the retort is ready for further handling.

1.8 Combustion control and CO leakage detection

The CO concentration sensor is located above the furnace. It can work either standalone with acoustic and optical signalization, or it can be connected to the computer control system which, in case of hazardous leakage of gases, makes adequate action and terminates the test. Up to four sensors can be connected to the control panel. Sensors can be located as required.

Combusted gases may be brought into the open space or to an exhaust hood.

2. Technical parameters of the RF-33/KK system

2.1 Vertically openable furnace WA 1100 for CRI test

- working temperature in the furnace	1 100°C +/- 1°C
- zone of stabilized temperature	> 400 mm
- electric resistance heating blocks	exchangeable
- material of heating elements	Kanthal A1
- time to reach 1 100°C temperature	max. 40 min.
- maximum power input	approx. 10 kVA
- power supply	3 × 400V/ AC
- control of furnace heating	digital, by power thyristors
- position of retorts	vertical
- opening of furnace	automatic
- temperature alarm	automatic
- insertion of retort into the furnace	automatic, by electric drive
- measurement of temperature in the sample	roughly in sample center
- measurement of temperatures in furnace	in each zone independently
- measurement of temperature field in the sample	by four thermocouples
- measurement of actual heating current (voltage)	for each block independently

2.2 OB-2 device with revolving tumbler for CSR test

- number of test tumblers	1
- inner length	700 mm, +/- 1 mm
- inner diameter	130 mm, +/- 1 mm
- tumbler wall thickness	7 mm
- tumbler bottom thickness	10 mm
- driven by	AC motor /3 x 400 V
- rotational speed	20 rpm, +/- 0.1 rpm
- revolution counter	contactless
- indication of revolutions number and time	by digital display
- rotational speed control	by frequency converter
- automatic shutdown	by the control system, preset time, or preset number of revolutions
- safety protection	Plexiglas cover

2.3 Computer control system SPW-2/KK

- control computer used	PC system
- CPU	2.2 GHz
- RAM memory	512 MB
- HD memory	160 GB HDD
- other memory drives	FDD 3.5“, USB
- flat screen display	19“ TFT LCD
- keyboard	wireless
- mouse	wireless
- color printer	HP
- remote access	by Internet
- computer network	Ethernet 10/100
- measurement and control interface units	IDAM, TEDIA

2.4 Control of temperatures in the furnace and in CRI retort

- | | |
|--|------------------------------|
| - temperatures in furnace and in retort | controlled by control system |
| - rate of retort temperature increase to 1 100°C | controlled by software |
| - accuracy of temperature control | better than +/- 1°C |
| - temperature in retort during the test itself | 1 100°C +/- 1°C |
| - length of uniform temperature zone | > 400 mm |

2.5 Control of the CSR tumbler rotational speed

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|---|------------------------|
| - speed controller | frequency converter |
| - requested speed (according to the Standard) | 20 rpm +/- 0.1 rpm |
| - number of tumbler revolutions during test | 600 revolutions/30 min |

2.6 Control of gas flow

Control of N₂ flow

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|--------------------------------|-------------------------------|
| - measurement method | mass flow meter (OMEGA / USA) |
| - measurement range | max. 10 l/min |
| - accuracy of gas flow control | +/- 1 % |

Control of CO₂ flow

- | | |
|--------------------------------|-------------------------------|
| - measurement method | mass flow meter (OMEGA / USA) |
| - measurement range | max. 10 l/min |
| - accuracy of gas flow control | +/- 1 % |

2.7 Retort for CRI test

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|--|---------------------------------|
| - retort length | 900 mm |
| - retort material | stainless steel |
| - retort ending | screw cap |
| - inner diameter | 78 mm |
| - tube wall thickness | 3 (5) mm |
| - bottom thickness | 10 mm |
| - gas preheating | ceramic balls |
| - measurement of temperature in retort | 1 sheathed thermocouple D 3 mm |
| - measurement of temperature profile | 4 sheathed thermocouples D 2 mm |
| - grate for placement of sample | stainless steel |
| - checking the gas tightness of retort | automatic |

2.8 Retort for making coke

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|--|------------------------|
| - retort length | 1 100 mm |
| - retort material | stainless steel |
| - retort ending | flange |
| - inner diameter | 130 mm |
| - tube wall thickness | 5 mm |
| - bottom thickness | 10 mm |
| - measurement of temperature in the retort | by K-type thermocouple |
| - checking the gas tightness of retort | automatic |

2.9 Retort cooling box

- design movable
- forced cooling in the box by air fan
- temperature measurement by thermocouple

2.10 CO leakage detection system

- CO concentration sensor fixed above furnace
- connection for automatic termination of test possible
- system diagnostics automatic during entire test

3. Additional information

3.1 Test furnace operation

For the CRI test, the operator hangs the prepared retort with test sample onto the hanger on one side of test furnace and connects the connectors (measuring cable, feeding of gases, and exhausts). Then the operator starts the measuring process, and after tempering the furnace to requested temperature, the retort moving on boom on which it is hanged automatically loads into the furnace that meanwhile got opened. Tempering of furnace is done very quickly thanks to high thermal insulating properties of the material of heating modules. After test completion, in the time given by ISO/DIS 18894 or ASTM D 5341 Standards, the retort automatically unloads from the test furnace and stays hanged on the boom until cooled down to room temperature. Cooling down is done using the mobile cooling box with a fan. Ending of the cooling down is signaled to the operator who can now safely handle the retort.

3.2 Retort handling trolley

This device is used for safe and easy handling with retort. Its hanger is designed for easy tilting of retort (emptying it after the test) and for easy hanging of retort (using lever movement) onto a travel boom to the furnace, and for removing retort from the furnace after ending the test. The trolley is also used for transporting of retort between individual locations.

3.3 Visualizations

3.3.1 Entering of input parameters

Mandatory data, i.e. certificate number, dimension of grate used for CSR determining (10 or 9.5 mm), initial weight of sample taken (if less than 50 kg), special notes, operations performed considered as non-obligatory according to the ISO Standard, date of test sample preparation, name of laboratory technician. It is possible to complement with other suitable data.

3.3.2 Results – test protocol

The protocol shall include the following:

Certificate number and name of the Standard used for carrying out the test, dimension of grate used for CRI determining (10 or 9.5 mm), original weight of sample taken (if less than 50 kg), parameters of coke sample drying. Further on: Automatically processed results of performed tests – initial weight of coke test sample for CRI reaction, weight of coke test sample after CRI reaction, total time of reaction, total CO₂ flow volume, total N₂ flow volume, maximum deviation of temperature during reaction, weight of coke charge after CSR

test, maximum deviation from preset rotational speed for each test separately – resulting CRI reactivity, resulting strength after CSR test, and comparison with obligatory criteria given by the Standard i.e. decision whether third or fourth testing is needed as regards both CRI and CSR tests. Further, special notes, operations performed considered as non-obligatory from the point of view of the ISO Standard, date of test sample preparation and date of test, name of laboratory technician. Also other parameters can be added as per user needs.

3.3.3 Additional graphical records (optional for printing)

- time chart of central thermocouple temperature during CRI test
- time chart of temperatures characterizing the temperature profile
- actual gas flow rates of during CRI test
- time chart of temperatures in furnace heating sections during CRI test
- actual values of tumbler's rotational speed during CSR test

3.3.4 Archived data

- all measured and regulated parameters of all tests are archived and can be reproduced on the display or printed, being sorted either by date of test or by automatically generated certificate number
- a time archive of all manipulations with system is saved in form of special files, including records of detected failures during automatic checking operations
- test results are also processed and archived in *.xls files intended for further use.

3.4 **Computer software**

3.4.1 Operating systems

Control computer	WIN 7 (Win 10)
Visualizations	“DELPHI” environment
Remote access	Team Viewer

3.4.2 Main application software for

- entering data into test protocol
- digital temperature regulation
- controlling CRI and CSR tests pursuant to ISO/DIS 18894 or ASTM 5341 Standards
- controlling the coking process
- archiving of individual certificates and its displaying and print of final protocol
- automatic control of observation of measuring procedures during certifications.