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Instruction Manual

Continuous Stirred Tank Reactor (CSTR)

CEM-MKII

ISSUE 21

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1 Introduction

This manual contains instructions for the correct use and maintenance of the CEM-MKII manufactured by Armfield Limited.

The information contained in this manual is intended for the user who is required to read it carefully and to ensure that he has fully understood it before operating the machine.

The user manual must be available for ready consultation at all times.

If the manual is lost or damaged contact the manufacturer for a replacement copy.

WARNING - The manufacturer is not liable for consequences resulting from an improper use of the machine due to the user's failure to read this manual or incomplete reading of it.

The manual is an integral part of each piece of equipment and consequently must be kept throughout the entire service life of the machine and accompany it at all times, even if transferred to another user.

This manual contains instructions required for the safety, receiving, installation, storage, correct operation and maintenance of the CEM-MKII.

WARNING - Armfield Limited reserves the right to modify the specifications referred to in this manual or the characteristics of each machine. Some of the illustrations in this manual may include parts that are slightly different to those mounted on your machine.

WARNING - All practical work areas and laboratories should be covered by local regulations which must be followed at all times.

2 EC Conformity

Each machine is accompanied by an EC Declaration of Conformity signed by the representative of Armfield Limited.

The declaration of conformity states the model and serial number.

The equipment has been constructed in compliance with the essential health and safety requirements laid down in the following applicable directives:

2006/95/EC	The Low Voltage Directive
2004/108/EC	The Electromagnetic Compatibility Directive
2006/42/EC	The Machinery Directive

The following harmonised standards were also consulted for the design and construction of the equipment:

BS EN 61010-1:2010	Safety requirements for electrical equipment for measurement, control, and laboratory use.
BS EN 61000-6-1:2007	Electromagnetic compatibility (EMC). Generic standards. Immunity for residential, commercial and light-industrial environments.
BS EN 61000-6-3:2001	Electromagnetic compatibility (EMC). Generic standards. Emission standard for residential, commercial and light-industrial environments.

WARNING - This declaration is only valid if the Equipment is installed, used and maintained in compliance with the above-mentioned directives and instructions and with the instructions and equipment described in this manual.

Table of Contents

3 Disclaimer

This document and all the information contained within it are proprietary to Armfield Limited. This document must not be used for any purpose other than that for which it is supplied, and its contents must not be reproduced, modified, adapted, published, translated or disclosed to any third party, in whole or in part, without the prior written permission of Armfield Limited.

Should you have any queries or comments, please contact the Armfield Customer Support helpdesk (Monday to Thursday: 0830 - 1730 and Friday: 0830 - 1300 UK time). Contact details are as follows:

United Kingdom	International
01425 478781 (calls charged at local rate)	+44 (0) 1425 478781 (international rates apply)
Email: support@armfield.co.uk	
Fax: +44 (0) 1425 470916	

4 Copyright and trademarks

5 Symbols

 General warning indicating the potential risk of personal injury

 Danger warning

 Caution: Explosion Risk

 Electrical hazard

 Cold Burn hazard

 High Voltage hazard

 Caution: Flammable

 Rotating parts hazard

 Caution: Biohazard

 Caution: corrosive material

 Do not remove safety guards from rotating parts

 Do not repair or oil machine whilst in motion

 This symbol draws your attention to the information

 Wear protective gloves

 Wear eye protection

 Wear ear protection

 Wear safety shoes

 Wear breathing protection

6 Safety

6.1 Failure to comply with safety standards

Failure to comply with the safety standards described in this manual and those relating to common sense can cause danger to people and the environment and damage the Equipment.

Specifically, such non-observance can cause:

- inability of machine and/or plant to perform key functions;
- damage to the machine and /or plant;
- electrical, mechanical and/or chemical danger to persons;
- environmental danger due to leakage of hazardous substances.

Failure to observe and comply with these safety standards will invalidate the warranty.

Keep these instructions and all related documents together, ensure that they are legible and easily accessible to all employees.

Do not remove any safety equipment before operating the Equipment or during its operation. Make sure that there is no evident danger before powering up the Equipment. The system must be inspected regularly to check for damage and to ensure that all safety devices are in good working order.

The Equipment contains moving parts. Do not insert limbs or materials other than the processing material while the Equipment is functioning. In the event of malfunction, danger or lack of appropriate safety systems, shut down the Equipment immediately and inform the qualified personnel.

6.2 Start up, operation and maintenance

The customer is required to verify the suitability of the Equipment for his specific needs, to provide the necessary processing data for a correct selection of the Equipment type and the accessories needed to guarantee the safety of the Equipment. If the user notices that any accessories he considers useful or essential are missing in the order confirmation, it is the customer's responsibility to contact the manufacturer and request that the accessory or accessories be applied to the Equipment.

While the Equipment is being used, the safety devices provided must be present and correctly installed. Do not carry out any operation on the safety devices while the Equipment is operating.

6.3 Intended conditions of use

The Equipment is designed to give students an appreciation of the construction, design and operational characteristics of continuous stirred tank reactors.

The Equipment must always observe the operating limitations for which it was constructed and those stated in the order confirmation: observe the temperature, pressure, capacity, viscosity and speed limits. Unless otherwise stated in the order, the Equipment must not be used in environments subject to the formation of potentially explosive atmospheres.

6.4 Safety guidelines relating to maintenance, inspection and assembly work

The user must ensure that all maintenance, inspection and assembly operations related to the Equipment are carried out by qualified technicians.

Technicians must carefully read this instruction manual before acting on the Equipment. Only authorised and trained personnel are permitted to work on the Equipment.

6.5 Arbitrary production and transformation of spare parts

Changes or modifications to the machine, within the limits that do not go beyond extraordinary maintenance, are only permitted if agreed on beforehand with the manufacturer.

Only original spare parts or parts specifically declared as compatible by Armfield Limited must be used for regular maintenance operations.

These parts have been designed specifically for the system. There is no guarantee that non-original parts can withstand the loads, and function correctly and safely.

The use of non-original parts voids the warranty.

6.6 Chemical Safety

The unit is designed to use clean water (deionised or demineralised to avoid scale build up due to impurities) during normal operation, but cleaning should be carried out regularly as described in the maintenance section of this manual which may involve the use of detergents/chemicals. In addition, under certain conditions causing algal growth, it may be necessary to use disinfectants or biocides to avoid the possibility of water-borne infections as described above.

6.7 Control of Hazardous Substances

The Control of Substances Hazardous to Health Regulations

The COSHH regulations impose a duty on employers to protect employees and others from substances used at work which may be hazardous to health.

COSHH covers substances that are hazardous to health. Substances can take many forms and include:

- Chemicals
- products containing chemicals
- fumes
- dusts
- vapours
- mists
- nanotechnology
- gases and asphyxiating gases and biological agents (germs). If the packaging has any of the hazard symbols, then it is classed as a hazardous substance.
- germs that cause diseases such as leptospirosis or legionnaires disease and germs used in laboratories.

The regulations require you to make an assessment of all operations which are liable to expose any person to these hazards. You are also required to introduce suitable procedures for handling these substances and keep appropriate records.

Since the equipment supplied by Armfield Limited may involve the use of substances which can be hazardous (for example, cleaning fluids used for maintenance or chemicals used for particular demonstrations) it is essential that the responsible person in authority implements the COSHH regulations or local equivalent.

Safety data sheets

The regulations also ensure that the relevant Health and Safety Data Sheets must be available for all hazardous substances used in the laboratory.

Products you use may be 'dangerous for supply'. If so, they will have a label that has one or more hazard symbols. These products include common substances in everyday use such as paint, bleach, solvent or fillers. When a product is 'dangerous for supply', by law, the supplier must provide you with a safety data sheet.

Note: Medicines, pesticides and cosmetic products have different legislation and don't have a safety data sheet. Ask the supplier how the product can be used safely.

Any person using a hazardous substance must be informed of the following:

- Physical data about the substance.
- Any hazard from fire or explosion.
- Any hazard to health.
- Appropriate First Aid treatment.
- Any hazard from reaction with other substances.
- How to clean/dispose of spillage.
- Appropriate protective measures.
- Appropriate storage and handling.

Although these regulations may not be applicable in your country, it is strongly recommended that a similar approach is adopted for the protection of the users operating the equipment. Local regulations must also be considered.

More information can be found on http://www.hse.gov.uk/coshh/index.htm

Any such chemicals used must be stored, handled, prepared and used in accordance with the manufacturer's instructions and with all applicable local regulations. Protective clothing (e.g. gloves, eye protection) should be worn when appropriate, and users should be supplied with any relevant safety information (e.g. the correct procedure in the event of contact with skin or eyes, the correct procedure in the event of a spill, etc.).

6.8 Water Borne Hazards

The equipment described in this instruction manual involves the use of water/fluid, which under certain conditions can create a health hazard due to infection by harmful micro-organisms.

For example, the microscopic bacterium called Legionella pneumophila will feed on any scale, rust, algae or sludge in water and will breed rapidly if the temperature of water is between 20 and 45°C. Any water containing this bacterium which is sprayed or splashed creating air-borne droplets can produce a form of pneumonia called Legionnaires Disease which is potentially fatal.

Legionella is not the only harmful micro-organism which can infect water, but it serves as a useful example of the need for cleanliness.

Under the COSHH regulations, the following precautions must be observed:

- Any water/fluid contained within the product must not be allowed to stagnate, i.e. the water must be changed regularly.
- Any rust, sludge, scale or algae on which micro-organisms can feed must be removed regularly, i.e. the equipment must be cleaned regularly.
- Where practicable the water/fluid should be maintained at a temperature below 20°C or the water should be disinfected. In the CEB-MKIII this may not be practicable so the equipment should be drained after use and filled with fresh water for each run. Note that other hazards may exist in the handling of biocides if these are used to disinfect the water.
- After use the water system should be filled and run with water containing a mild disinfectant such as ‘Milton’ to kill any micro-organisms or algal growth then flushed with clean water and left empty.
- A scheme should be prepared for preventing or controlling the risk incorporating all of the actions listed above.

Further details on preventing infection are contained in the publication “The Control of Legionellosis including Legionnaires Disease” - Health and Safety Series booklet HS (G) 70.

6.9 Hot/Cold Surfaces

This unit contains components that that operate with a maximum temperature of 50°C and minimum temperature of 5°C.

Do not touch any surfaces close to ‘Hot Surfaces’ warning labels, any of the interconnecting tubing or components whilst the equipment is in use or returning to a safe temperature.

6.10 Hot/Cold Liquids

This unit is designed to operate with a maximum liquid temperature of 50°C and minimum liquid temperature of 5°C.

Before disconnecting any of the pipes or tubing:

- Stop the liquid pump.
- Leave time for the equipment to return to room temperature.
- Check that the temperature of the Equipment and liquid is at a safe level

Do not touch any surfaces close to 'Hot Surfaces' warning labels, any of the interconnecting tubing or components whilst the equipment is in use or returning to a safe temperature.

6.11 Leakage of hazardous fluids

If the Equipment is used to pump/operate with hazardous liquids (toxic, corrosive, flammable, etc.), the volumes of fluid that leak through the seals must be collected and disposed of without endangering human health or the environment and in accordance to local legislation.

6.12 Protective clothing

Wear appropriate protective clothing to protect body parts.

Safety gloves

Wear suitable gloves to protect your hands from various types of possible hazards: mechanical, electrical, chemical and high/low temperatures.

Clothing

Wear appropriate clothing to protect your body from chemical hazards.

Footware

Wear safety footwear to protect your feet from falling objects.

Eye Protection

Wear suitable eye protection to protect your eyes from various types of possible hazards: mechanical debris, chemicals and hot water/steam.

Ear Protection

Wear suitable ear protection to protect your ears from excessive noise.

Breathing Protection

Wear suitable breathing protection to protect your respiratory system from fumes.

6.13 Machine maintenance

Do not disassemble the Equipment before emptying the contents/fluids (if applicable). Even if the tubes are all empty, some liquid could remain in the unit. The fluid(s) can be hazardous to human health and the environment and can be very hot/cold.

All maintenance work must be carried out with the machine isolated from the power supply.

Before beginning maintenance on the Equipment remember to isolate the power supply. All the devices must be secured against automatic or accidental restart. (Where possible turn the main switch to OFF and remove the key). In particular situations where you need to run the Equipment while servicing at least 2 persons must be present so that in the event of danger one person will be able to disconnect the power supply or raise the alarm. Once maintenance has been completed remember to restore the safety devices and check that they are in good working order.

To give increased operator protection, the unit incorporates a Residual Current Device (RCD), alternatively called an Earth Leakage Circuit Breaker, as an integral part of this equipment. If through misuse or accident the equipment becomes electrically compromised, the RCD will switch off the electrical supply and reduce the severity of any electric shock received by an operator to a level which, under normal circumstances, will not cause injury to that person.

At least once each month, check that the RCD is operating correctly by pressing the TEST button. The circuit breaker MUST trip when the button is pressed. Failure to trip means that the operator is not protected, and the equipment must be checked and repaired by a competent electrician before it is used.

7 General Overview

THIS INSTRUCTION MANUAL SHOULD BE USED IN CONJUNCTION WITH THE MANUAL SUPPLIED WITH THE CEXC COMPUTER CONTROLLED CHEMICAL REACTOR SERVICE UNIT.

This Manual provides the necessary information for operating the equipment in conjunction with the CEXC Computer Controlled Chemical Reactor Service Unit, and for performing a range of Teaching Exercises designed to demonstrate the basic principles of Chemical Reactors theory and use.

The continuous stirred tank reactor in the form of either a single tank or (more often) tanks in series, is used widely and is particularly suitable for liquid phase reactions. It is particularly used in the organic chemicals industry. Advantages include consistent product quality, straightforward automatic control and low manpower requirements.

The Armfield CEM-MKII Continuous Stirred Tank Reactor is specially designed to allow detailed study of this important process. It is one of five reactor types which are interchangeable on the Reactor Service Unit (CEXC), the others being CET-MKII - Tubular Reactor, CEB-MKIII – Transparent Batch Reactor, CEY Plug flow reactor and CEZ Laminar Flow reactor.

Reactions are monitored by conductivity probe as the conductivity of the solution changes with conversion of the reactants to product and by temperature.

CEXC fitted with CEM-MKII Continuous Stirred Tank Reactor

8 Equipment Diagrams

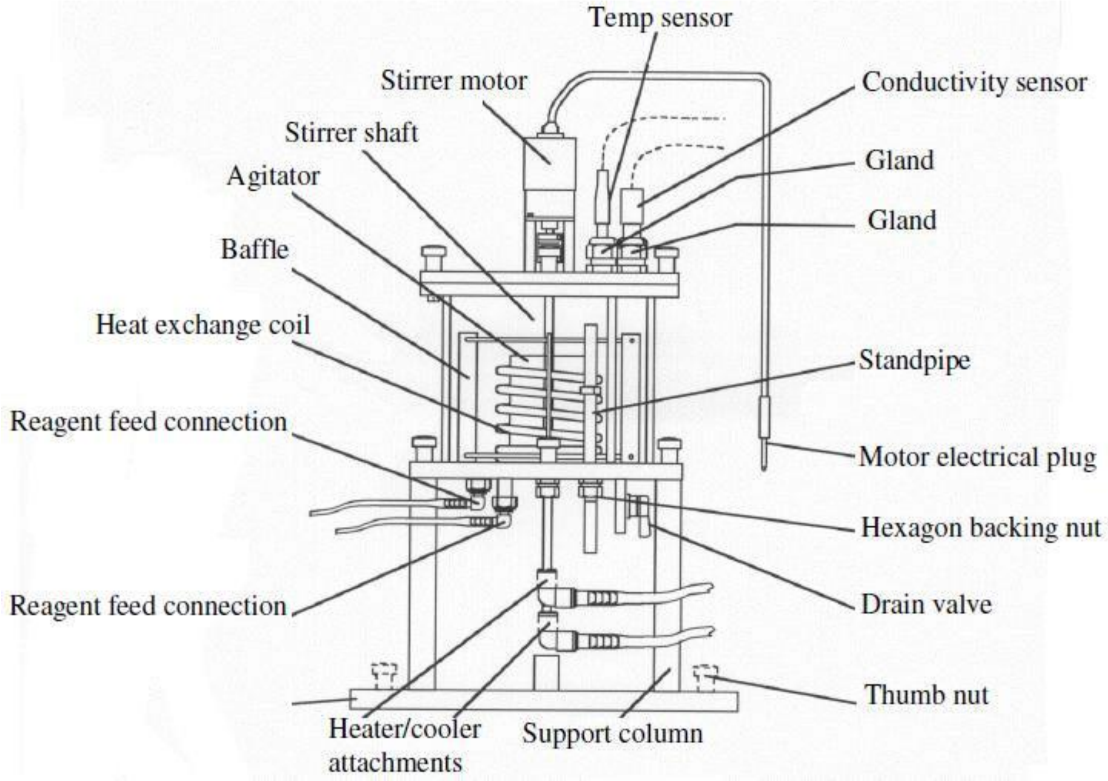


Figure 1: Reactor Unit

9 Description

Where necessary, refer to the drawings in Equipment Diagrams section 8.

9.1 Overview

The reactor vessel is set on a baseplate which is designed to be located on the four studs of the CEXC service unit and then secured by thumbnuts. The reactor is supported by three pillars; position the reactor on the CEXC service unit such that a single pillar is to the front.

A stainless steel coil inside the reactor provides the heat transfer surface for either heating or cooling the chemical reactants. The coil is connected either to the hot water circulator (for exercises above ambient temperature) or the optional CW-17 chiller. The coil inlet is at the front of the reactor and the coil return is at the rear of the reactor (for exercises below ambient temperature).

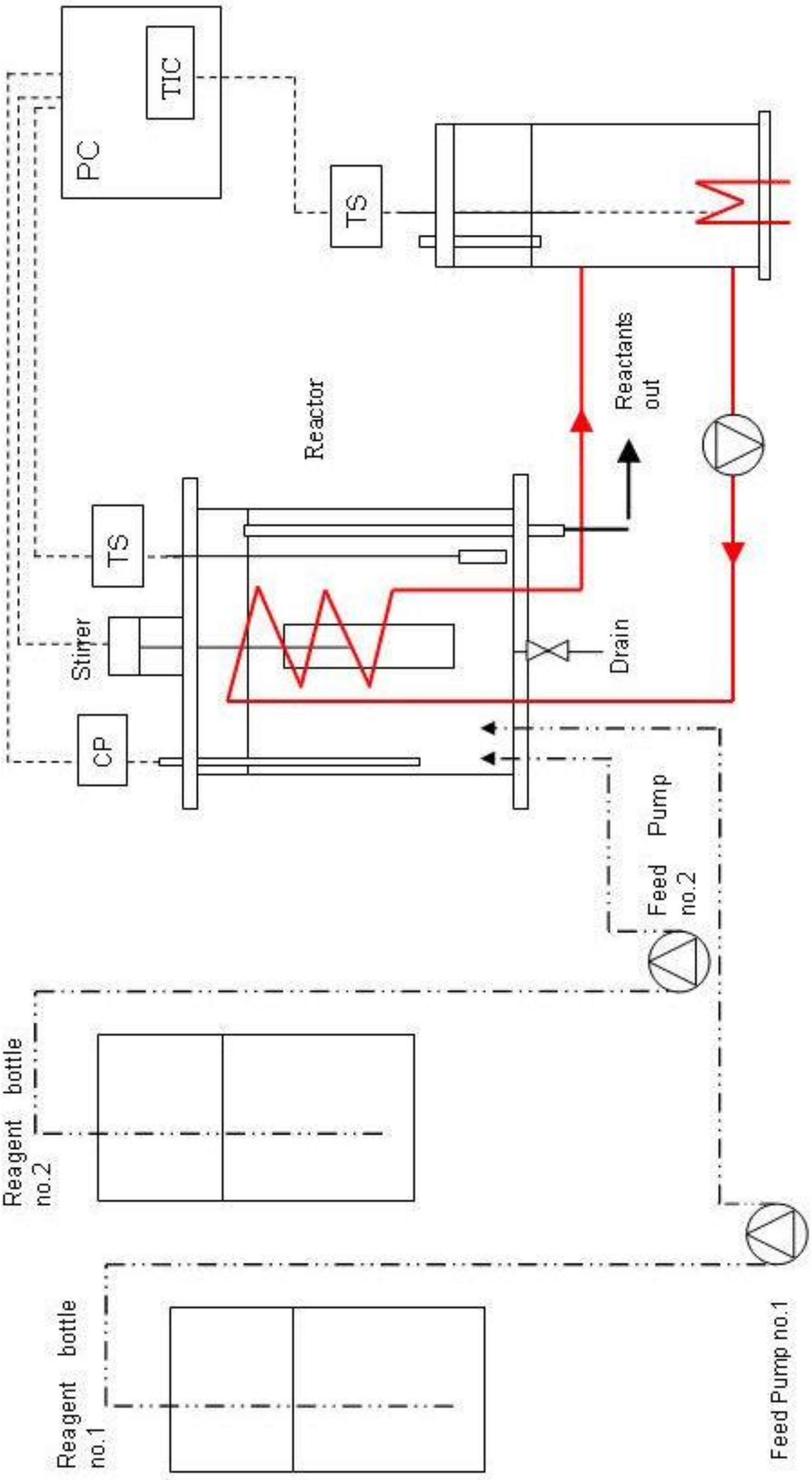
A stirrer (turbine agitator) works in conjunction with a baffle arrangement to provide efficient mixing and heat transfer. The stirrer is driven by an electric motor mounted in the lid of the reactor. The motor is controlled by the software supplied with the Service Unit. The electrical socket for the motor is located at the rear of the service unit.

Glands in the reactor lid house the conductivity and temperature sensors provided with the service unit. The larger of the two glands is for the conductivity probe. The glands are unscrewed by hand, the probes inserted completely into the reactor until they rest on the reactor base and then the glands re-tightened by hand. Electrical sockets at the rear of the service unit are provided to connect each probe. These are of different size so that the probes cannot be wrongly connected. The conductivity probe can be connected to low or high conductivity channels to suit the range of measurements expected. For normal use the probe should be connected to the high conductivity channel.

9.2 Flow of materials

The two feed pumps on the service unit pump the chemical reagents from the two feed bottles into the reactor separately through connectors in the base of the reactor. As reagents are pumped into the reactor, the level increases until it finally overflows the stand pipe and flows to drain. The stand pipe may be adjusted in height by loosening the hexagonal gland nut. A mark is etched onto the stand pipe. For maximum operating volume of the reactor, this mark should be aligned with the gland nut. A stop prevents the stand pipe from being completely removed, and this also defines the minimum working volume which is half of the maximum volume.

When the reactor is not being used, it can be drained using the valve sited on the underside of the reactor.



CEM-MKII Continuous Stirred Tank Reactor

10 Installation

10.1 Advisory

Before operating the equipment, it must be unpacked, assembled and installed as described in the steps that follow. Safe use of the equipment depends on following the correct installation procedure.

10.2 Installing the Software

Please refer to the software installation instructions supplied on the Armsoft CD ROM or data stick.

10.3 Installing the Equipment

10.3.1 Mounting the CEM-MKII onto the CEXC

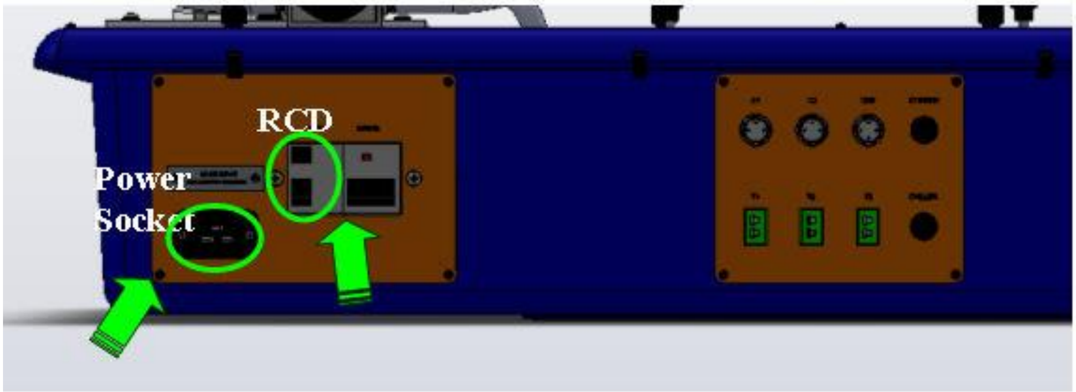
CEXC fitted with CEM-MKII Continuous Stirred Tank Reactor

- Fit the CEM-MKII assembly to the CEXC using the 4 locating studs and black thumbnuts. The reactor is supported by 3 pillars; position the reactor on the CEXC so that a single pillar support is at the front.
- Connect the two flexible pipes from the feed pumps to the connectors on the base of the reactor as shown.
- Connect the electrical plugs from the conductivity probe, temperature sensors T1 & T2 and the stirrer to the sockets located on the rear of the CEXC. For normal use the conductivity probe should be connected to the socket marked 'High Conductivity'. If measured values will not exceed 5 mS/cm then the Low Conductivity input can be used to improve accuracy.
- Fit the conductivity probe and temperature sensor to the appropriate gland on the lid of the CEM-MKII reactor ensuring that they reach the bottom of the reactor. The gland nut should be loosened prior to fitting the probe / sensor then tightened to secure them.

10.3.2 Connection to the Hot Water Circulator

- Connect return pipe of the HWC (1) to the connection for the top of the coil (rear).
- Connect the supply pipe of the HWC (2) to the connection for the bottom of the coil (front).

10.3.3 Connection to Electricity Supply



- Check that the voltage specified on the equipment matches the supply voltage.

  Note: this unit **must** be earthed.

- Connect the power socket at the rear of the plinth to a suitable mains electricity supply.
- Ensure that circuit breakers and RCD are ON (up).
- The on/off switch for the apparatus is located on the orange panel on the front of the plinth. Switch on the apparatus.

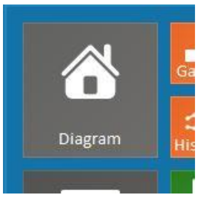
10.3.4 Connection to a PC for Control and Data Logging

Ensure that the software has been installed as described in the section 'Installing the PC software'.

Switch on the PC and connect the USB lead from the CEXC to the PC.

Load the CEM software then choose the appropriate experiment from the menu. For detailed information about operating the software refer to the section 'Operating the Software'.

Display the mimic diagram by choosing Diagram:



On the 'Diagram' screen check that the message in the bottom right corner of this screen displays 'OK: IFD5 on COMx', where x is the number of the USB port.

If the message continually displays 'Scanning….', then the PC has not located the USB interface on the CEM-MKII, and it will be necessary to check the connection of the USB and the installation of the USB driver.

If an error message occurs, then check the USB connection and if necessary, re-install the software.

Refer to the section 'Operating the Software' if necessary.

Connect the mains supply to the CEXC then switch on the CEXC. Click the 'Power On' button on the mimic diagram:

Confirm that the appropriate measured variables are displayed on the mimic diagram.

The software is ready for use.

11 Operation

Where necessary, refer to the drawings in Equipment Diagrams section 8.

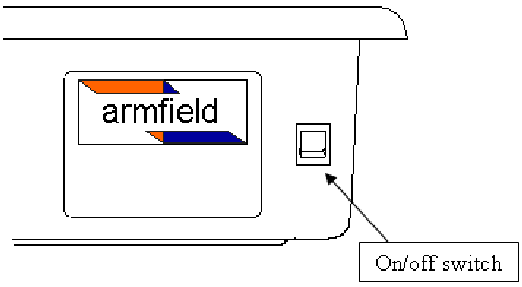
11.1 Operating the Software

Please refer to the software operating instructions supplied on the Armsoft CD ROM or data stick.

11.2 Operating the Equipment

11.2.1 Switching on the unit

The unit is switched on using the switch on the front of the unit. The circuit breakers and RCD device located at the rear of the unit should be turned on beforehand. Both the temperature controller and conductivity display should illuminate.



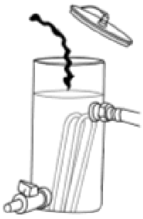
11.2.2 Filling the feed bottles

Unscrew the feed bottle caps and pour appropriate reagent solutions in using a suitable funnel if necessary.

11.2.3 Operating the Hot Water Circulator

The hot water circulator vessel should be filled with clean water before use, and drained after use if the equipment is not going to be used for some time. When in use fill the vessel by pouring clean (preferably demineralised) water into the vessel until the level is approximately 30 mm from the top. Click the ‘Hot Water Circulator’ button to operate the circulating pump then continue topping up the vessel until the level remains constant approximately 30 mm from the top. Replace the lid on the circulator.

The heater will not operate if the lid is removed or if the water level is below the level electrode. Top up the level of this vessel as necessary to maintain the level above the tip of the level electrode located in the lid.



Operation of the heater is controlled by a PID controller in the CEM software. The PID controller maintains the required Hot Water Temperature T2 to obtain a steady Reactor Temperature T1. Any offset in the required Reactor temperature T1 can be corrected by adjusting the set point of the Hot Water Temperature T2 in the PID controller. The Set Point temperature, Proportional Band, Integral, Derivative and Cycle times are all set to default values when the software is loaded but all of these may be adjusted by the user if required. Default settings for the PID controller are listed below.

11.2.4 Operation of Data Logger and Software

The Continuous Stirred Tank Reactor is controlled using the CEM software supplied, which allows real-time monitoring and data logging of all sensor outputs and control of the heater, stirrer and pumps. Recorded results can be displayed in tabular and graph format. The software runs on a Windows™ PC which connects to the CEXC using a USB interface.

Installation of the software is described in the Installation Guide, and the software must be installed before connecting the PC to the CEXC. The software may then be run from the Start menu (Start > Programs > Armfield Chemical Reactor Software > CEM).

Operation of the software is described in a walkthrough presentation within the software, and also in the online Help Text accessible via the software Help menu. Operation and setting of specific controls is also provided within the experiments described in this manual.

11.2.5 Mimic Diagram and software

The equipment is controlled from the Mimic Diagram screen in the software. This shows all the sensor outputs, and includes controls for the pumps, stirrer and hot water Circulator.

Before operating the equipment click ‘Power On’ to enable the pumps, stirrer and hot water circulator.

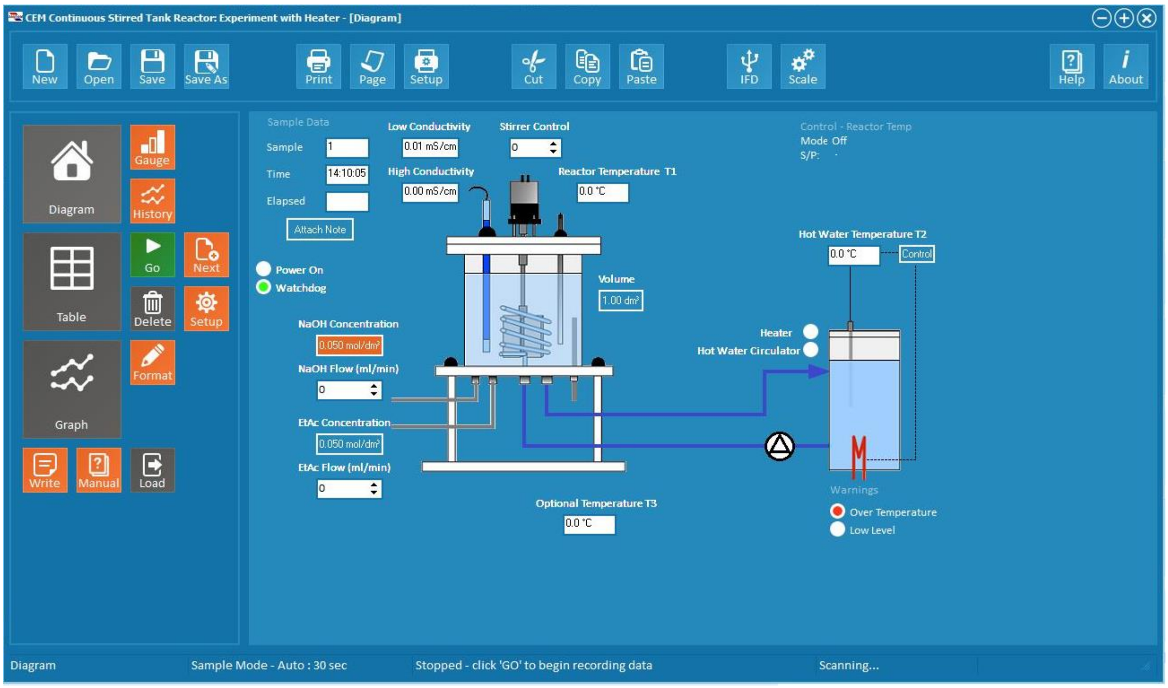
The software includes a readout for an optional temperature sensor T3 (not supplied).

Feed pump speeds are controlled using up/down arrows or typing the flow rate in a value between 0 and the maximum of 150 ml/min.

Concentration values must be typed in on each experiment if is required for the software to carry out the subsequent calculations.

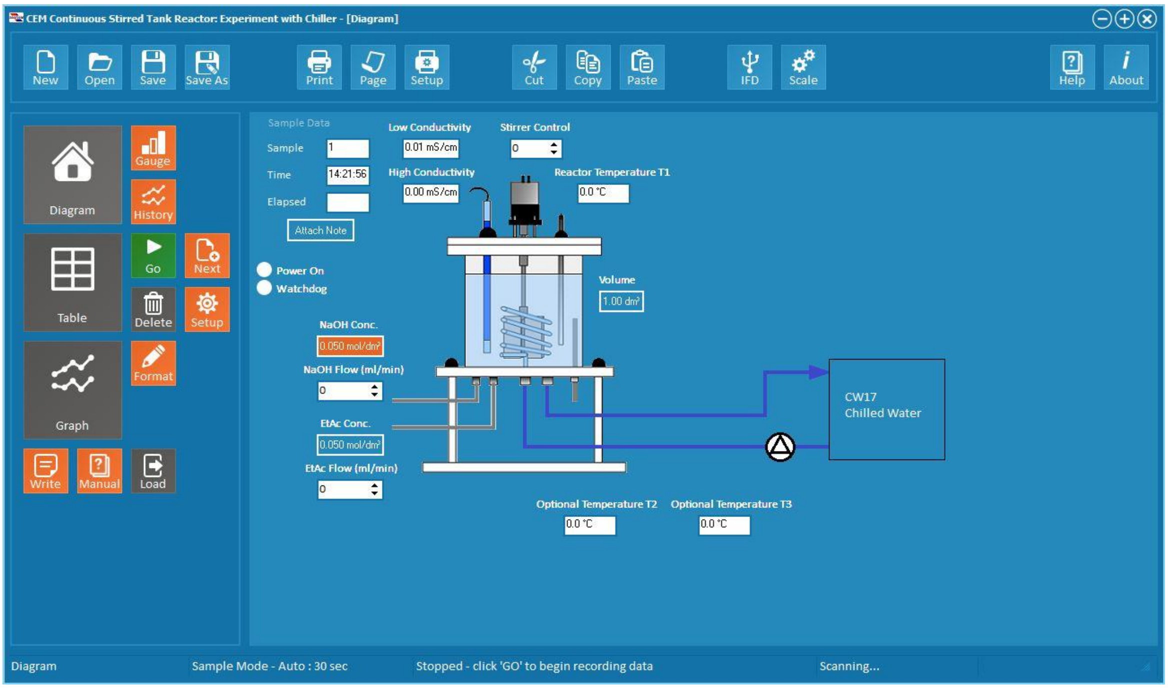
Conductivity and temperature values will be data logged when ‘GO’ is clicked.

The CEM-MKII Continuous Stirred Tank Reactor can be used with the integral hot water circulator for operating temperatures above ambient.



CEM-MKII Continuous Stirred Tank Reactor with Heater

Alternatively, it can be used with an optional Chiller (CW-17) for operating temperatures below ambient. The use of the heater or chiller is chosen in the initial menu when loading the CEM software. When using the hot water circulator the required temperature is set using the PID controller on the mimic diagram. When using the optional CW-17 chiller the required operating temperature is set using the thermostat located on the CW-17.



CEM-MKII Continuous Stirred Tank Reactor with Chiller

The software automatically generates a series of 'Watchdog' pulses that are required by the CEXC, ensuring that the hardware shuts down safely in case of a software or communications failure.

11.2.6 Controlling the Hot Water Circulator (HWC)

The heater is controlled by a PID controller in the software. Click 'Hot Water Circulator' to start the circulator pump then click 'Control' to open the controller window.

The PID controller initially defaults to Off with the heater turned off. The Mode of Operation must be changed to Manual or Automatic as required to turn on the heater. In Manual operation the required Manual Output (heater power) is adjusted by the operator as necessary to obtain the required hot water temperature T2. In Automatic operation the PID controller varies the heater power continuously to correct for any error.

The hot water temperature T2 is chosen as the default process variable to provide a stable temperature T1 inside the reactor. Control of T1 directly would be difficult because of the lag in the process and significant overshoots in batch or low flow conditions. As a consequence, Reactor temperature T1 will always be stable but slightly lower than T2. If it is required to maintain Reactor Temperature T1 at a particular temperature, then an appropriate offset can be applied to the setpoint of the controller so that T2 is elevated slightly to compensate.

If required temperatures T1 or T3 (type K thermocouple sensor not supplied) can be selected as the alternative process variable but appropriate P, I, D and CT settings will need to be entered for the system to operate.

Default PID Settings for the Hot Water Circulator:

Process variable = Hot Water Temperature (T2)

Control Variable = Heater

Control Action = Reverse

SP = Chosen by the user IT = 0 Cycle time = 10

PB = 1% DT = 0

11.2.7 Operating the CEM-MKII

Temperature T1 in the reactor is monitored on the software and is controlled by circulating heating or cooling liquid through the internal coil.

There are two modes of operation with the CEM: Experiment with HEATER and with CHILLER.

When using the Chiller CW-17 PID settings are not required. See the Installation section for appropriate controller settings when using the CW-17.

When using the Heater the temperature sensor T1 supplied with CEXC, which is Reactor Temperature on the software, should be set as the Process Variable to be controlled in the PID loop. This sensor must be immersed in the reactor vessel and will be data logged.

The volumetric ratio in which the reactants are mixed is defined by the relative flow rates of the two pumps. If the pumps are operated at the same flow rate then the reactants are mixed in equal volumes.

The degree of mixing may be adjusted using the agitator speed control box on the main screen of the software.

The extent of conversion of the reactants is determined from the conductivity, which is measured by the conductivity probe.

12 Equipment Specifications

12.1 Overall Dimensions

Vessel diameter: 0.153 m

Maximum vessel depth: 0.108 m

Maximum volume: 2.0L

Minimum vessel depth: 0.054 m

Minimum operating volume: 1.0 L

12.2 Environmental Conditions

This equipment has been designed for operation in the following environmental conditions. Operation outside of these conditions may result reduced performance, damage to the equipment or hazard to the operator.

a) Indoor use;
b) Altitude up to 2000 m;
c) Temperature 5 °C to 40 °C;
d) Maximum relative humidity 80 % for temperatures up to 31 °C, decreasing linearly to 50 % relative humidity at 40 °C;
e) Mains supply voltage fluctuations up to ±10% of the nominal voltage;
f) Transient over-voltages typically present on the MAINS supply;

   Note: The normal level of transient over-voltages is impulse withstand (over-voltage) category II of IEC 60364-4-443;

g) Pollution degree 2.

   Normally only nonconductive pollution occurs.

   Temporary conductivity caused by condensation is to be expected.

   Typical of an office or laboratory environment.

13 Routine Maintenance

13.1 Responsibility

To preserve the life and efficient operation of the equipment it is important that the equipment is properly maintained. Regular maintenance of the equipment is the responsibility of the end user and must be performed by qualified personnel who understand the operation of the equipment.

13.2 General

The equipment should be disconnected from the electrical supply when not in use. After use the feed bottles, reactor vessel, sump tray and pipework should be washed through with water to remove chemical residues and then drained.

13.3 RCD Test

Test the RCD by pressing the TEST button at least once a month. If the RCD button does not trip when the Test button is pressed, then the equipment must not be used and should be checked by a competent electrician.

13.4 Temperature sensor calibration

The temperature sensors are calibrated before delivery and should not require re-calibration. However should calibration become necessary follow the below procedure. This should only be done once the unit has fully warmed up.

The temperature conditioning circuit (which provides the reading from the conductivity probe supplied with the CEXC service unit) is located on a printed circuit board inside the plinth on the right-hand side. This circuit is calibrated before despatch and should not require re-calibration. However, should re-calibration become necessary the appropriate calibration potentiometers can be located using the diagram given in the CEXC manual (Routine Maintenance).

Connect CEXC service unit to a PC and start up the Armfield software. Open mimic diagram screen where T1, T2 and T3 windows are displayed.

If a thermocouple calibrator is available:

Connect Thermocouple calibrator simulator to T1 input socket. Set to 25 ºC and adjust VR1 (T1 ZERO) and VR2 (T1 SPAN) on the PCB to give 25 ºC displayed on PC. Check accuracy at 15 ºC and 40 ºC.

Repeat the same procedure for T2 by adjusting VR3 (T2 ZERO) and VR4 (T2 SPAN) on the PCB to give 25 ºC displayed on PC, and VR5 (T3 ZERO) and VR6 (T3 SPAN) for T3.

If a thermocouple calibrator is not available:

Temperature sensor T1, T2 and T3 should be dipped into crushed ice, and then adjust the ZEROS to give 0 ºC, then sensors should be dipped into boiling water and then adjust the SPANS to 100 ºC.

13.5 Calibration of the conductivity sensor

The Low and High conductivity conditioning circuits (which provide the reading from the conductivity probe supplied with the CEXC service unit) are located on the printed circuit board shown above inside plinth. These circuits are calibrated before despatch and should not require re-calibration. However, should re-calibration become necessary the appropriate calibration potentiometers can be located using the diagram below.

Ensure the equipment has been connected to the electrical supply and switched on for at least 20 minutes. To access the PCB remove the cover plate on the right hand side of the plinth by unscrewing the four fixing screws. It is not necessary to detach the PCB from the plinth.

Disconnect the conductivity probe from either socket at the rear of the unit. Connect an AC Voltmeter (Range AC mV) to pins 1 and 2 of the High conductivity socket and adjust potentiometer VR10 on the PCB to give a reading of 50mV (RMS) on the Voltmeter (probe excitation voltage).

Disconnect the Voltmeter then reconnect the probe to the High conductivity socket having removed the probe from the reactor fitted to the CEXC.

High conductivity Calibration (Range 0 – 20 mS/cm)

Fill a small beaker with a Conductivity standard solution (e.g. 0.1M KCl giving a conductivity of 12.88mS at 25°C) and measure the temperature of the standard solution using a suitable thermometer. From the table supplied determine the actual conductivity of the solution at the measured temperature.

Immerse the probe into the Conductivity standard solution in the beaker then adjust potentiometer VR7 to give a reading of the standard solution in the ‘High conductivity’ window on the software to match the conductivity.

Remove the probe from the conductivity solution and rinse the probe in de-mineralised water.

12.88 mS/cm at 25ºC 0.1 M KCl

ºC	mS/cm	ºC	mS/cm
5	8.22	20	11.67
10	9.33	21	11.91
15	10.48	22	12.15
16	10.72	23	12.39
17	10.95	24	12.64
18	11.19	25	12.88
19	11.43	26	13.13

Low conductivity Calibration (Range 0 – 5 mS/cm)

Disconnect the conductivity probe from the High conductivity socket then reconnect it to the Low conductivity socket.

Fill a small beaker with a Conductivity standard solution (e.g. 0.01M KCI giving a conductivity of 1.41mS at 25°C) and measure the temperature of the standard solution using a suitable thermometer. From the table supplied determine the actual conductivity of the solution at the measured temperature.

Immerse the probe into the Conductivity standard solution in the beaker then adjust potentiometer VR8 to give a reading of the Standard solution in the 'Low conductivity' window on the software.

Remove the probe from the conductivity solution and rinse the probe in de-mineralised water.

When the conditioning circuit has been re-calibrated replace the panel and re-install the probe in the reactor on the CEXC service unit.

1.413 mS/cm at 25ºC 0.01 M KCl

ºC	mS/cm	ºC	mS/cm
5	0.896	20	1.278
10	1.02	21	1.305
15	1.147	22	1.332
16	1.173	23	1.359
17	1.199	24	1.386
18	1.225	25	1.413
19	1.251	26	1.441

14 Laboratory Teaching Exercises

14.1 Index to Exercises

14.2 Nomenclature

Symbol	Name	Unit
a_{μ}	sodium hydroxide conc. in feed vessel	(mol/dm^3)
a_o	sodium hydroxide conc. in mixed feed	(mol/dm^3)
a_1	sodium hydroxide conc. in reactor at time t	(mol/dm^3)
a_{∞}	sodium hydroxide conc. in reactor after ∞ time	(mol/dm^3)
b	ethyl acetate conc.	(mol/dm^3)
	(same subscripts as above for a)	
c	sodium acetate conc.	(mol/dm^3)
	(same subscripts as above for a)	
F	total volume feed rate	(dm^3/s)
F_a	volumetric feed rate of sodium hydroxide	(dm^3/s)
F_b	volumetric feed rate of ethyl acetate	(dm^3/s)
k	specific rate constant	
r	reaction rate	
t_R	residence time (s)	
t	elapsed time (s)	
T	reactor temperature	(K)
V	volume of reactor	(dm^3)
X_a	conversion of sodium hydroxide = $\frac{a_0 - a_1}{a_0}$	
X_c	conversion to sodium acetate = $\frac{c_1 - c_0}{c_{\infty}}$	
Λ	conductivity (Siemens/cm)	
Λ_o	initial conductivity	
Λ_1	conductivity at time t	
Λ_{∞}	conductivity after ∞ time	
Λ_a	sodium hydroxide conductivity	
Λ_c	sodium acetate conductivity	

C	concentration in reactor at time *t* after input step change	
C_0	concentration of the input	
t_c	time constant	

14.3 Common Theory

The Armfield continuous stirred tank reactor is designed to demonstrate the mechanism of a chemical reaction in this type of reactor as well as the effects of varying the process conditions such as reaction temperature, reactor volume, stirring rate, feed rate etc.

The reactor volume can be varied by adjusting the height of the internal standpipe. The actual volume must be checked by filling the reactor with water to the overflow then draining the reactor contents into a measuring cylinder.

The conductivity of the reacting solution in the reactor changes with the degree of conversion and this provides a convenient method for monitoring the progress of the reaction either manually or by computer.

The reaction chosen is the saponification of ethyl acetate by sodium hydroxide as it can be carried out under safe conditions of temperature and pressure and is well documented.

The experiments involve the collection and storage of conductivity data. The USB port located at the front of the Service Unit must be connected to the computer. This will enable data logging of the conductivity and temperature sensor at selected time intervals over a selected period of time.

Although it may be possible to carry out demonstrations using other chemicals, it is not advisable as the materials of construction of the reactor may not be compatible.

As the standard reaction is exothermic the heat generated by the reaction will result in a rise in temperature of the vessel contents that is unavoidable. If it is required to perform the trial at a temperature below the ambient temperature, the optional chilled water circulator CW-17 (not supplied) must be connected to the coil in the reactor vessel.

Before carrying out reactions involving any other reagents please refer to Armfield Ltd. for advice.

Dilution of Ethyl Acetate

Armfield recommends the use of a 0.1 M solution of Ethyl Acetate in the CEM-MKII reactor. This should be made by diluting concentrated Ethyl Acetate as follows:

$$Volume\ of\ concentrate = \frac{Mol\ Wt}{10}\ x\ \frac{1}{Density} = \frac{88.11}{10\ x\ 0.90} = 9.79\ ml\ per\ litre\ of\ solution$$

Therefore add 9.79 ml of concentrated Ethyl Acetate to 900 ml of deionised or distilled water.

Shake the mixture vigorously until the two liquids have mixed. Add further water to make up the final volume to 1000 ml.

Note: The practice of making a strong solution (e.g. 1M) then further diluting this to the required concentration (e.g. 0.1M) cannot be applied when using Ethyl Acetate. The required dilution should be made directly as stated above.

Dilution of Sodium Hydroxide

Armfield recommends the use of a 0.1 M solution of Sodium Hydroxide in the CEM-MKII reactor. This may be made by adding 4.0 g of NaOH to 960 ml of deionised water then making up the solution to 1000 ml.

Website: **www.armfield.co.uk** Email: **support@armfield.co.uk** Customer Support: **01425 478781**

15 Exercise A - To find the reaction rate constant in a Continuous Stirred Tank Reactor

Theory

The reaction:

$NaOH + H_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

Sodium Hydroxide + Ethyl Acetate → Sodium Acetate + Ethyl Alcohol

can be considered equi-molar and first order with respect to both sodium hydroxide and ethyl acetate, i.e. second order overall, within the limits of concentration (0 - 0.1M) and temperature (20 - 40°C) studied.

The reaction carried out in a Continuous Stirred Tank Reactor or Tubular Reactor eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place.

The steady state conditions will vary depending on concentration of reagents, flowrate, volume of reactor and temperature of reaction.

Method

Make up 2.5 litre batches of 0.1M sodium hydroxide and 2.5 litres of 0.1M ethyl acetate.

Important: It is essential when handling these chemicals to wear protective clothing, gloves and safety spectacles.

Remove the caps of the reagent bottles and carefully fill with the reagents to a level approximately 50 mm from the top using a funnel if necessary. Refit the caps.

Start the software using the option of the experiment with heater.

Click the 'Power on' button on the mimic diagram, click the 'Hot Water Circulator' button and water will start to recirculate through the hot water circulator and coil in the reactor.

Adjust the set point of the PID controller to 30°C.

Change the PID 'mode of operation' to 'Automatic'.

Collection of conductivity data should continue until a steady state condition is reached in the reactor and this takes approximately 30 minutes. It is advisable to set the data collection period to, say, 45 minutes.

Set the pump speed controls to 40 ml/min flowrate in the software.

Fill the concentration of both solutions in their respective boxes in the software.

Fill the volume of the reactor in the box on the software. Set the stirrer speed to 50% in the software.

It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.

Interpretation of Results

Having recorded the conductivity of the contents of the reactor over the period of the reaction, the conductivity measurements must now be translated into degree of conversion of the constituents.

Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst ethyl acetate and ethyl alcohol do not. The conductivity of a sodium hydroxide solution at a given concentration and temperature, however, is not the same as that of a sodium acetate solution at the same molarity and temperature and a relationship has been established allowing conversion to be inferred from conductivity.

The calculations are best carried out using a spreadsheet such as EXCEL so that the results can be displayed in tabular and graphical form.

Note that the software performs all the calculations needed during experiment. However it is recommended to go through all the procedure and calculations for better understanding.

On conclusion of the experiment using the Armfield data logger, a set of readings of conductivity with time will be stored in the computer.

At this point, this data can be transferred onto the spreadsheet.

Start the spreadsheet program.

Now enter the following known constants from the experiment using the Nomenclature. Ensure use of correct units.

F_a =

F_b =

a_μ =

b_μ =

c_μ =

T =

V =

Using the spreadsheet, calculate the values of $a_o, b_o, c_\infty, a_\infty, \Lambda_{c\infty}, \Lambda_{ao}, \Lambda_{a\infty}, \Lambda_o$ and Λ_∞

from the following formulae:

$$a_0 = \frac{F_a}{F_a + F_b} \cdot a_\mu$$

$$b_0 = \frac{F_b}{F_a + F_b} \cdot b_\mu$$

c_∞	=	b_o	for $b_o < a_o$
c_∞	=	a_o	for $b_o \geq a_o$
$\Lambda_{c\infty}$	=	$0.070[1+ 0.0284(T-294)]\ c_\infty$	for $T \geq 294$
Λ_{ao}	=	$0.195[1+ 0.0184(T-294)]\ a_o$	for $T \geq 294$
Λ_o	=	Λ_{ao}	assumes $c_o = 0$
a_∞	=	0	for $a_o < b_o$
a_∞	=	$(a_o - b_o)$	for $a_o \geq b_o$
$\Lambda_{a\infty}$	=	$0.195[1+ 0.0184(T-294)]\ a_\infty$	if $a_\infty \neq 0$
Λ_∞	=	$\Lambda_{c\infty} + \Lambda_{a\infty}$	

For the values of each of the above, the spreadsheet can be used to calculate values of sodium hydroxide concentration (a_1) and sodium acetate concentration (c_1) and the degree of conversion (X_a) and (X_c) for each of the samples of conductivity taken over the period of the experiment.

These can be calculated and listed in columns (use spreadsheet COPY facility) alongside the readings of conductivity using the following equations:

$$a_1 = (a_\infty - a_0)\left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right] + a_0$$

$$c_1 = c_\infty\left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right]$$ for $c_0 = 0$

$$X_a = \frac{a_0 - a_1}{a_0}$$

$$X_c = \frac{c_1}{c_\infty}$$ for $c_0 = 0$

To calculate the specific rate constant, k:

The overall mass balance at steady-state condition may be written as:

Input – Output ± Reaction = 0

i.e. for a reactant a in a reactor of volume V

$$\frac{d(Va_1)}{dt} = F \cdot a_0 - F \cdot a_1 - V \cdot k \cdot a_1^2$$

For the continuous reactor operating at steady state the volume may be assumed constant and

$$k = \frac{F}{V} \cdot \frac{a_0 - a_1}{a_1^2} = \frac{(F_a + F_b)}{V} \cdot \frac{(a_0 - a_1)}{a_1^2}$$ mol/dm³ sec

The steady state concentration of NaOH in the reactor (a_1) may be used to calculate the specific rate constant (k).

Comment upon the results obtained. How did temperature affect the reaction rate and the conversion?

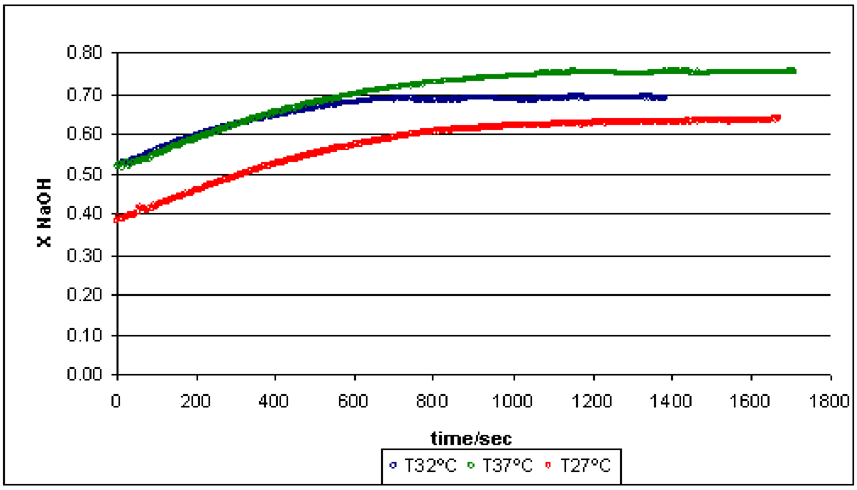
Notes:

It is recommended that this experiment should be repeated at various other temperatures to investigate the relationship between the specific rate constant (k) and the temperature of reaction. If the reactor temperature is below ambient Chiller should be required and ‘Experiment with Chiller’ option should be chosen from the software. See below for examples of experimental results.

It is further recommended that the experiment be repeated using dissimilar flow rates for the caustic soda and ethyl acetate solutions to investigate the effect that this will have upon the saponification process.

Treatment Results

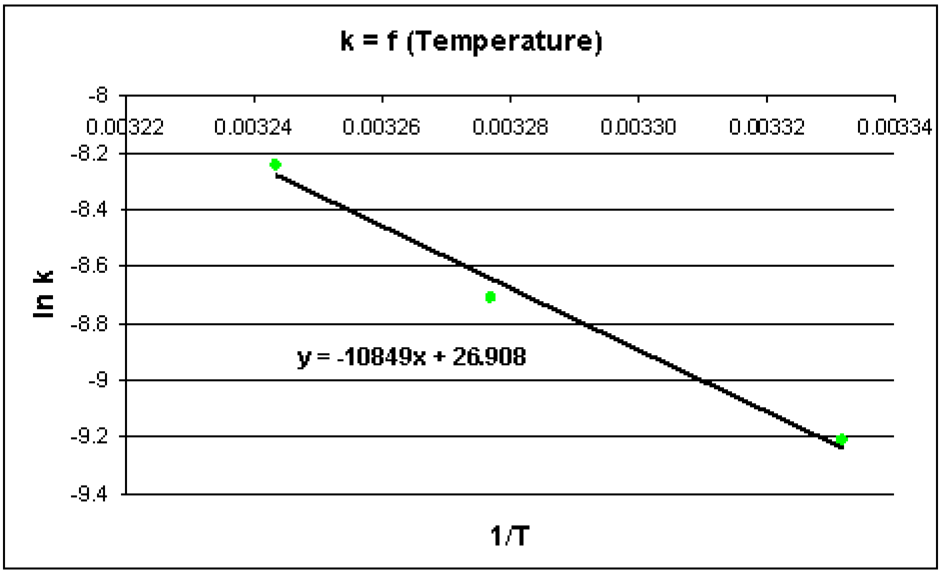
Conversion of NaOH at different temperatures:



Once the kinetic constant at three different temperatures is known is straightforward to apply the Arrhenius law and calculate the frequency factor and the activation energy values for:

$$\ln k = \ln A - \frac{E}{R}\frac{1}{T}$$

Plotting Rate constant vs Temperature



$$\ln \grave{A} = 26.908 \qquad \grave{A} = 4.85 \cdot 10^{11}\ m^3/mol\,\sec$$

$$\frac{E}{R} = 10849 \qquad E = 90.19\ KJ/mol$$

Obtention of the reaction rate constant in function of the temperature:

$$k = 4.85 \cdot 10^{11} \cdot e^{\left(-\frac{90.19}{RT}\right)} \qquad T\left(^{\circ}K\right)$$

16 Exercise B - To determine the effect of inadequate mixing on the reaction rate

Theory

The rate of reaction is measured by the number of reactants converted to products in a unit of time. In order for reaction to occur, particles must come into contact and this contact must result in interaction. The rate of reaction depends on the collision frequency and collision efficiency of particles of the reacting substances. These factors are optimised by thorough mixing of the reactants using stirrers and baffles within the reactor. Inefficient mixing will result in reduced reaction rates.

Considering the reaction between sodium hydroxide and ethyl acetate, if the initial concentrations are equal (both a_0) and the conversion (X_a) then the concentrations are as follows:

$$NaOH + CH_3COOC_2H_5 \rightarrow C_2H_5OH + CH_3COONa$$

$$(a_0-X_a) \quad (a_0-X_a) \qquad (X_a) \quad (X_a)$$

Method

Repeat Exercise A after removing the baffles from the reactor. This is achieved by removing the conductivity and temperature probes then removing the lid of the reactor. The baffle arrangement simply lifts out.

Repeat the experiment with baffles removed and no stirring action.

Three sets of data will be obtained:

a) Stirred reactor with baffle (see Exercise A)
b) Stirred reactor, no baffle
c) Un-stirred reactor, no baffle

Graphs of the reaction conversion with time can be plotted using the data logger (or using the manual readings obtained if not using the logger).

Comment on the results obtained. How did removal of the baffle affect the reaction rate? What effect does stirring have on the reaction rate?

17 Exercise C - Determination of the Residence Time using tracer techniques

Theory

Effect of a step input change, calculation of the average residence time.

If C = concentration in reactor at time *t* after input step change

C_0 = concentration of the input

Then $C = C_0\left(1 - e^{-\frac{t}{tr}}\right)$ where tr = time constant

$$\frac{C}{C_0} = \left(1 - e^{-\frac{t}{t_c}}\right) \longrightarrow 1 - \frac{C}{C_0} = e^{-\frac{t}{tr}}$$

$$\ln\left(1 - \frac{C}{C_0}\right) = -\frac{1}{tr} \cdot t$$

Hence t_c may be found graphically.

Method

Make up 2.5 litres of a solution of 0.1M KCL and fill one of the feed bottles. Fill the other feed bottle with demineralised water.

Using the Armfield data logger, initiate the program.

Set the reactor stirrer to a speed of '50%' and press ‘Power on’ button to start it up. The experiment can be carried out at room temperature initially. If other reactor temperatures are required this is achieved using the hot water circulator and setting the PID temperature controller in the software as detailed in previous experiments.

Start the water feed pump by setting the pump speed control to maximum in order to fill the reactor to the overflow as quickly as possible. When the reactor is full, stop the feed pump.

Start the KCL solution feed pump by setting the pump speed to 100 ml/min.

The conductivity of the reactor contents will begin to increase and, after a period of approximately 45 minutes to 1 hour, will approach the conductivity of the feed solution and will reach the steady state.

On conclusion of the experiment using the Armfield data logger, a set of readings of conductivity with time among other calculations will be stored in the computer.

At this point, only conductivity with time data can be transferred onto the spreadsheet.

Start the spreadsheet program.

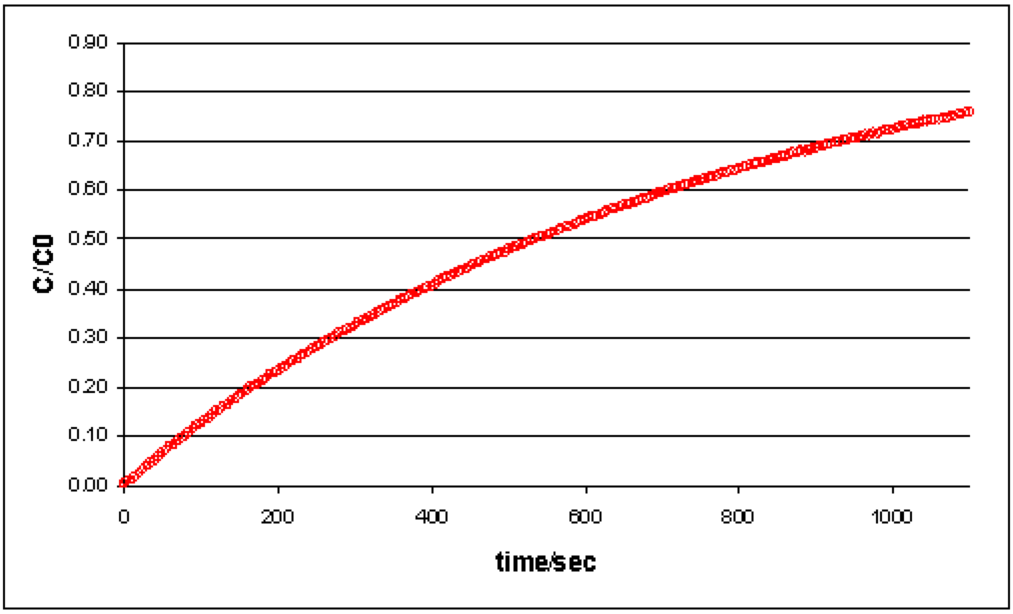
Calculate Λ/Λ_0 which is the equivalent to C/C_0 where Λ is the conductivity at time *t*, and Λ_0 is the conductivity of KCL at the steady state (maximum conductivity reached), for readings of *t* throughout the experiment.

Plot this value against *t* and calculate the Napierian logarithm of $(1-\frac{C}{C_0})$

Plot $\ln\left(1-\frac{C}{C_0}\right)$ vs time and calculate the slope (straight line graph passing through the origin). The slope is the average residence time t_R which should be equal to $\frac{V}{F}$ where V is the reactor volume and F is the total flow rate into the reactor.

Treatment Results

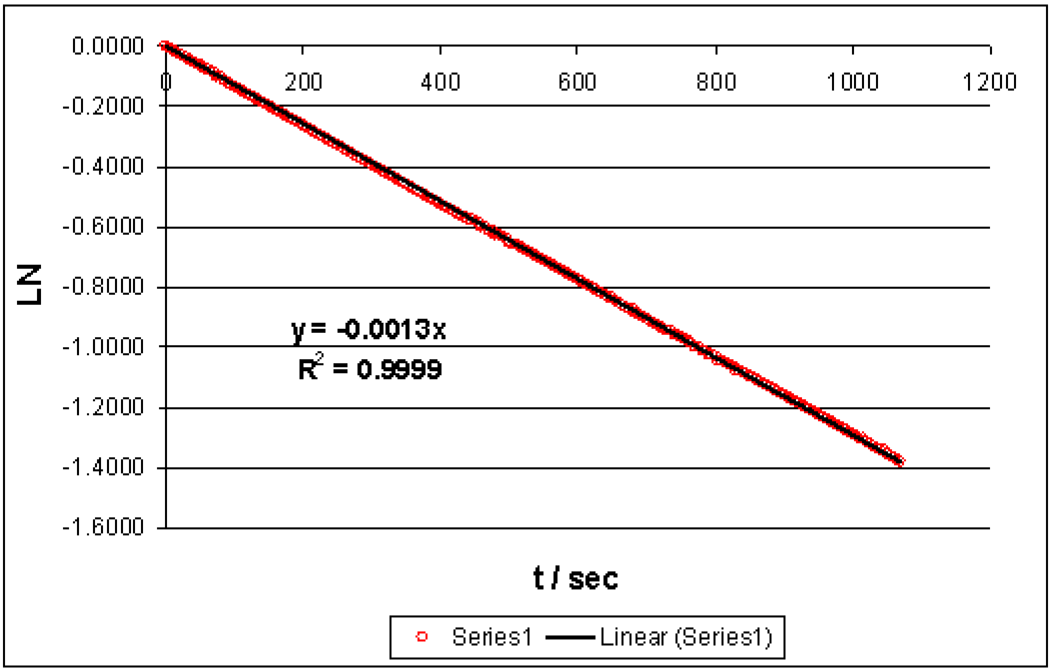
The normalisation of the concentration $\frac{C}{C_0}=\frac{\Lambda}{\Lambda_0}$ can be plotted against time:



To calculate the experimental residence time:

Calculate the Napierian Logarithm of $(1-\frac{C}{C_0})$

Plot $\ln\left(1-\frac{C}{C_0}\right)$ vs time and calculate the slope



$$Ln\left(1-\frac{C}{C_0}\right) = -0.0013 \cdot t$$

$$-0.0013 = -\frac{1}{tr} \longrightarrow tr = 769.23\ \text{sec}$$

The Average Residence Time for a working volume of 1369ml and a working flow rate of 106.5ml/min:

$$\tau = \frac{Vreactor}{Flowrate} = \frac{1369}{106.5} = 771\ \text{sec}$$

The difference between the theoretical and the experimental value may be caused by experimental errors, such inaccuracies on the measurement of the flowrate or reactor working volume, etc.

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