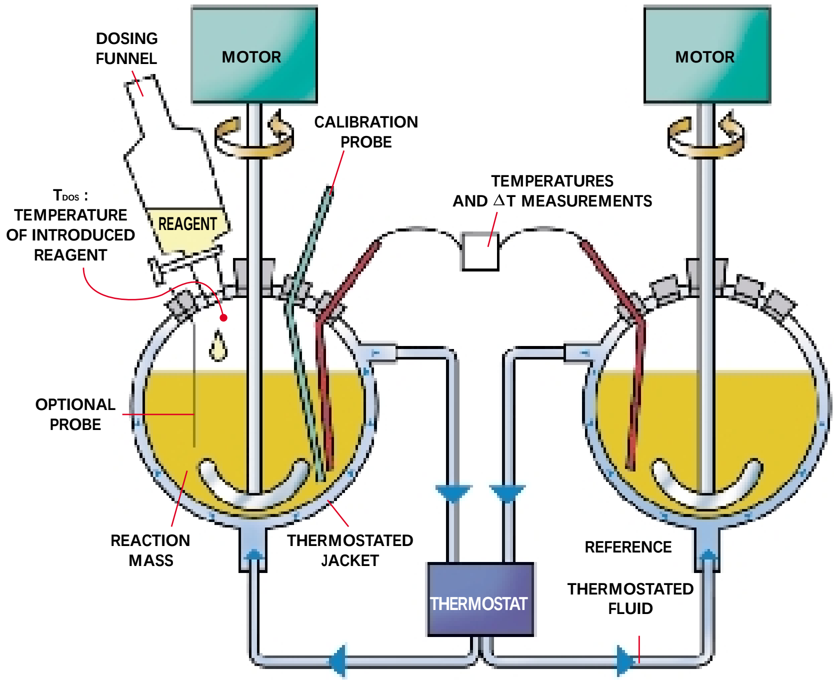
DRC Differential Reaction Calorimeter

-80°C +150°C



DRC diagram

DRC Technical characteristics

-80°C +150°C

DRC operating temperature range:
from -80°C to 150°C
Atmospheric pressure
Calibration by Joule effect

Reactor
- Double skinned flasks
- Volume: 100, 250 or 500 ml
- Material: Pyrex

Stirrer
- Anchor shape
- Material: Teflon
- Speed: 50 to 1,000 rpm
- Torque: 30 N.cm

Dosing system
- Dosing funnel
- Volume: 50 ml

Probes
- Tantalum sheathed platinum probe 100W
- Joule effect probe
- pH-meter

Cryo-thermostat
- Temperature control: ± 0.01°C

The DRC has been developed with AVENTIS, Neuville location.

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DRC, Differential Reaction Calorimeter, is used in laboratories working on the optimization of industrial processes and the safety of chemical reactions. It is a laboratory reactor capable of simulating experimental industrial conditions, permitting fast access to important thermodynamic properties linked with liquid-liquid and liquid-gas mixtures of chemical products: reaction heat, mixing heat, reaction time, maximum elevations of temperature during reaction, monitoring of reaction kinetics, heat capacities, etc. Used as a screening tool to determine reaction heats, the DRC offers numerous advantages.

• Saving of the products studied

Due to its measurement principle, the DRC allows working on very low product quantities (from 10 g). This saving of material is a considerable advantage when the reagents are only available in small quantities or are very costly.

• Saving of time

Operating time using the DRC has been reduced to the minimum. 15 minutes are needed to start and prepare the calorimeter before testing, 1 hour to measure a classical reaction and also 15 minutes to process the data. This time-saving results in an equivalent reduction of delays in industrial development.

• Ease of use

Thanks to its simple design, the DRC is extremely user-friendly and easy to use. Designed by chemists for chemists, it requires no specific training and can be used by everyone.

• Principle of differential measurement

The differential construction permits the elimination from any reaction heat measurement of parasite variations due to agitation, heat dissipation into the walls, thermostat temperature and ambient temperature variations.

• Simultaneous measurement of reaction heat and heat capacity (Cp)

Calibration of the calorimeter by Joule effect, vital for every reaction enthalpy measurement, also permits measuring the heat capacity of the chemical reaction.

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Calibration is carried out by dissipating a constant thermal power (Joule effect) into the mixture during a specified time. The temperature of the liquid therefore rises to reach an equilibrium value which will be determined by the heat flux balance and thus the heat capacity of the reaction mixture.

The major innovation provided by the DRC is the possibility of measuring the heat of a reaction and the heat capacity of the reaction mixture simultaneously. It is also possible to monitor the variation of heat capacity during the chemical reaction.

- Given the reaction heat and heat capacity, it is possible to determine the elevation of temperature ΔT in adiabatic mode.
- The capacity of measuring Qdos (heat due to the introduction of a reagent at a temperature differing from that of the environment) makes it possible to forego thermostating the liquid added. Furthermore, this cannot always be carried out.

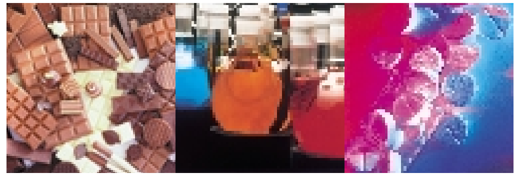
Accessories

The reagent can be introduced in the reaction flask by using a syringe. It is possible, for example, to add one or more electrochemical analysis devices (pH-meter, conductivity meter, etc.) and a gas scanning for hydrogenation study for instance.

Applications

The DRC is used daily in development laboratories to optimize industrial processes and make them safe. It has been used successfully to study a great variety of reactions. The following is a non-exhaustive list of reactions:

Oxidation reactions - Reduction reactions Hydrogenation - Epoxydation - Ozonization Bromination - Chloration - Cyanuration Diazotization - Grignard reactions (organo-magnesium) - Dehydrogenation - Wolff Kishner reaction - Reformatsky reaction - Barton reaction Mickaël reaction - Wittig reaction - Friedel and Crafts reaction (acylation) - Decarboxylation Esterification, transesterification - Oximation Methylation - Tosylation - Beckmann rearrangement - Chlorosulfonation, Sulfonation Knoevenagelc condensation - Fermentation Polymerization - Dissolution



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Principle of differential measurement

The DRC is based on the simple principle of differential thermal analysis which measures, continuously, the difference of temperature ΔT between a measurement reactor and a reference reactor. The measurement reactor is the receptacle of the reaction to be measured while the reference reactor contains a solvent having chemical and physical properties close to those of reagents introduced in the reactor studied.

The two calorimetric reactors are two double skinned flasks connected in parallel. A thermostated fluid circulates between the two skins, allowing the operator to determine the temperature desired. This experimental mode is known as isoperibolic mode (the environment is kept at constant temperature).

The difference of temperature, measured by platinum probes, between the mixture in reaction and the reference, is recorded as a function of time in order to obtain a thermogram characterized by a peak whose shape varies as a function of the reaction under study.

The heat released in the reaction environment is calculated from the area of the reaction peak on the thermogram. Simple calibration by Joule effect before and after the reaction supplies the product of the transfer coefficient by the area of exchange, UA.

As with any laboratory reactor, the operator can monitor visually the progression of the reaction in the reactor at any moment.
The technique is associated with a new data processing procedure that permits calculation of heat capacities of solvents before a chemical reaction, thus supplying the heat capacity of the mixture during and after the reaction.