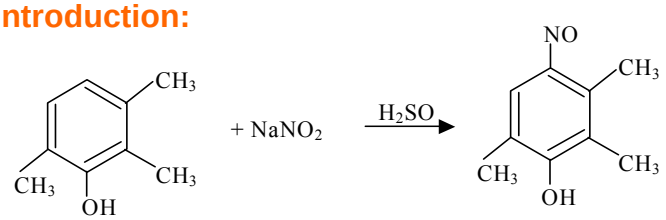
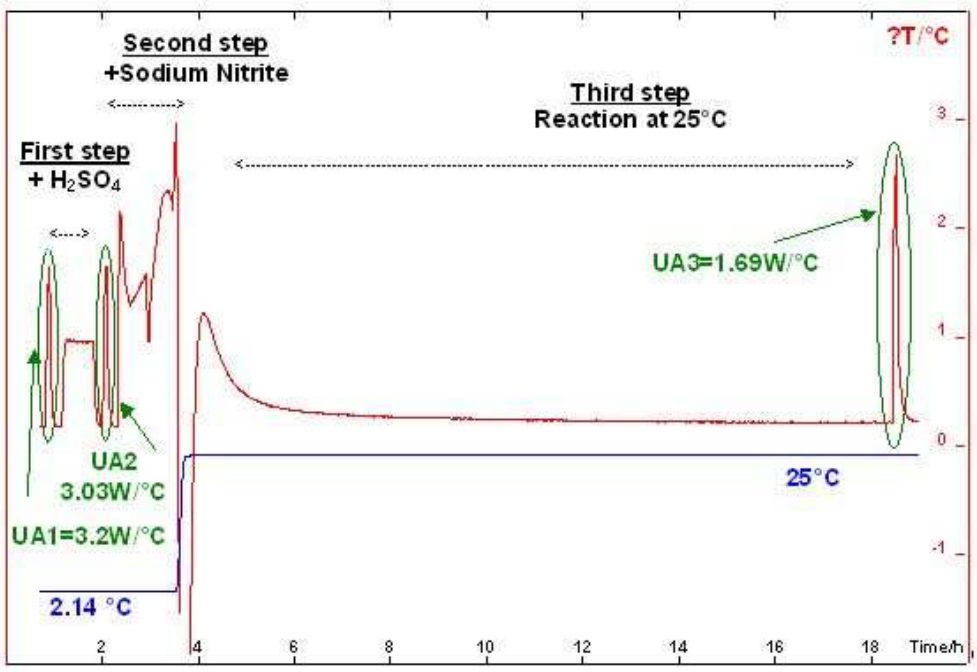
Synthesis of 2,3,6-trimethyl, 4-nitrosophenol

Introduction:



2,3,6-trimethylphenol *2,3,6-trimethyl, 4-nitrosophenol*

The synthesis of 2,3,6-trimethyl, 4-nitrosophenol is performed in three steps :
1) addition of H2SO4 to 2,3,6-trimethylphenol at 2°C
2) addition of sodium nitrite (NaNO2) still at 2°C
3) heating up to 25°C to initiate the reaction



Experimental

The DRC is used with reactors of 250 ml. A stirring at 300 rpm is produced.

The reference and measure reactors are loaded with 138 ml **(109g)** of **ethanol**.

23.03g of **2,3,6-trimethylphenol** is introduced in the measure reactor.

The solution is stirred for 30 min at 20°C and the DRC is cooled down to **2.1°C**

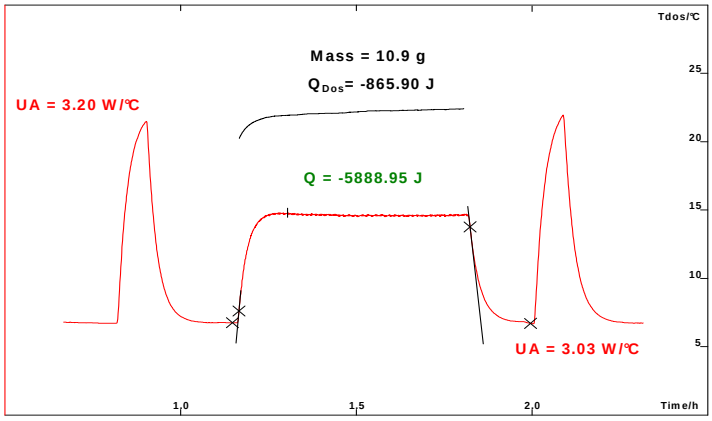
After this preparation phase, the data collection is started.

The figure hereunder presents the signals corresponding to the whole experiment.

Instrument
DRC Evolution (-80 to 150°C).

A calibration is generally carried out before and after a reaction. The purpose of a calibration is to determine the exchange coefficient UA. UA is mainly a function of filling of the reactor, of the temperature and the nature of the material inside the reactor.
In this example a calibration is carried out between each step. Joule effects are applied in the measure reactor. (5 W during 300 seconds).
The experimental conditions and the calculations are explained for each of the three steps.

Step 1 - Introduction of 6ml of H2SO4 at 2℃



After the first calibration and when the signal ΔT is stable, **6 ml** (10.87g) of sulphuric acid is added with a syringe pump. The whole addition takes about 40 min.

For the calculations, the following values are used:

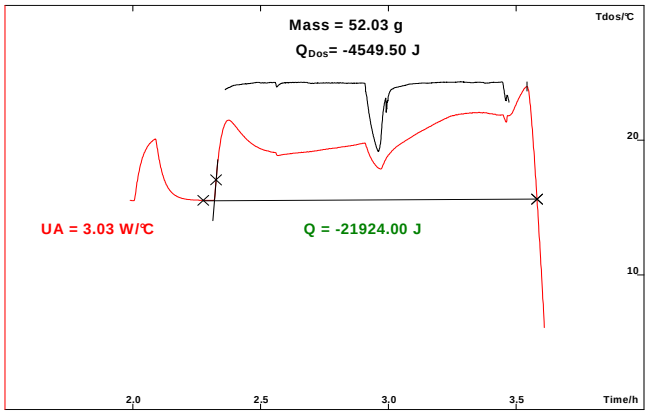
- Sulfuric acid: Cp = 4.0 J/g.K
- Temperature of the reaction: 2.14℃
- Average temperature of introduction : T_{dos} = 22℃
- mass = 109 + 23.03 + 10.87 = 142.9 g
- UA = (UA_1+ UA_2)/2 = (3.20+3.03)/2= 3.115 W/℃

(The use of the average value of UA, enables to take into account the volume variation inside the reactor and the corresponding evolution of exchange coefficient)

Results:

- **Q_{dos} = - 865.9 J**. Q_{dos} corresponds to the energy necessary to cool the added H_2SO_4 and to bring it from the introduction temperature (≈22℃) to the working temperature (2.14℃)
- **Q = 5 889.0 J.** It is the total energy of reaction of addition and it is obtained by integration of the peak of reaction and addition of Q_{dos}.
- **$\Delta T_{adiabatic}$** = 5889 / (142,9 x Cp) = **41.21 / Cp** It corresponds to the increase of temperature which would be observed in adiabatic mode during the addition of sulfuric acid.(Cp is the Cp of the mixture ethanol + 2,3,6 trimethylphenol + sulfuric acid.)

Step 2 - Introduction of Sodium Nitrite at 2℃



After the second calibration and when the signal ΔT is stable, a solution prepared with 18.03g of Sodium Nitride in 40ml of water is slowly introduced in the measure reactor. The whole addition takes about 1 h 05 min.

For the calculations, the following values are used:

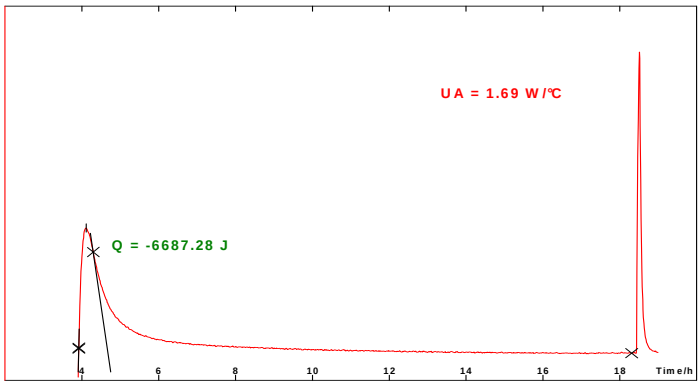
- Sodium nitrite solution: Cp = 4.0 J/g.K
- Temperature of the reaction: 2.14℃
- Average temperature of introduction : T_{dos} = 24℃
- UA = UA_2 =3.03W/℃

(In this step, it is considered that the level in the reactor does not change. Consequently, the value of UA obtained aftyer the previous step is used.)

Results:

- Q_{dos} = - 4 549.5J Qdos corresponds to the energy necessary to cool the added solution of Sodium Nitrite and to bring it from the introduction temperature (≈24℃) to the working temperature (2.14℃)
- **Q_1 = - 21 924.0 J** is the total energy of reaction of addition and it is obtained by integration of the peak of reaction and addition of Q_{dos}.

Step 3 - Reaction at 25℃



When the introduction is complete, The temperature of the bath is programmed up to 25℃.

For the calculations, the following value is used:

- UA = 1.69 W/℃

As the heating to 25℃ is rapid, the value of UA de termined at the end of the reaction is used for this step.)

Results

Q_2 = - 6 687 J is the total energy of reaction of addition and it is obtained by integration of the peak of reaction at 25℃