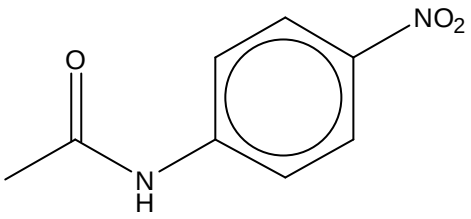
Sulfuric acid as a dehydrating agent – the dehydration of N-(4-nitrophenyl)ethanamide

Description

Concentrated sulfuric acid is added to N-(4-nitrophenyl)ethanamide (4-nitroacetanilide) and gently heated. After a lag time, a solid sponge of carbon forms and is ejected from the beaker in the shape of 'serpents' up to a metre high.



N-(4-nitrophenyl)ethanamide

Topic

Sulfuric acid as a dehydrating agent.

Timing

Five to ten minutes.

Level

The demonstration could be carried out at almost any level simply as a visually exciting piece of chemistry.

Apparatus

- Bunsen burner, tripod, gauze and heatproof mat
- 100 cm^3 beaker

Chemicals

- About 10 g of N-(4-nitrophenyl)ethanamide (4-nitroacetanilide)
- About 2 cm^3 of concentrated sulfuric acid (very corrosive)

Note. The purity of the N-(4-nitrophenyl)ethanamide appears to be critical to the success of this demonstration. Suitable material may be obtained from Fluorochem, Wesley Street, Old Glossop, Derbyshire SK13 9RY Tel: 01457 868921. The compound is expensive - £42 for 25 g at 2003 prices. Current prices and delivery information can be found in Fluorochem's on-line catalogue at www.fluorochem.co.uk (accessed September 2003).

Method

Work in a fume cupboard. Weigh 10 g of N-(4-nitrophenyl)ethanamide into a 100 cm^3 beaker and stand the beaker on a tripod and gauze. Add 2 cm^3 concentrated sulfuric acid and heat *gently* from below with a Bunsen burner. After a lag time of up to a couple of minutes the following changes occur:

1. The mixture blackens.
2. The black liquid starts to froth and bubble.
3. The liquid starts to smoke.

4. A solid sponge of carbon forms suddenly in the shape of ‘serpents’ up to a metre high.

After the experiment dispose of the carbon sponge as follows. Wearing gloves collect the foam and place it in a large beaker of water and leave it for some minutes to dilute any remaining acid. Small quantities can be broken up with a gloved hand and flushed down the sink with copious amounts of water. Larger amounts can be placed inside several sealed plastic bags (such as bin bags) and placed in the dustbin.

Visual tips

The demonstration is carried out in a fume cupboard, so visibility can be a problem. The use of a portable fume cupboard is recommended where this is available.

Teaching tips

This demonstration is similar to the dehydration of sucrose by concentrated sulfuric acid but is much more spectacular. Details of the dehydration of sucrose can be found in T. Lister, *Classic Chemistry Demonstrations*, London: Royal Society of Chemistry, 1995, pp 139-140. Teachers may wish to carry out this demonstration before the more spectacular one with N-(4-nitrophenyl)ethanamide described here.

Theory

It is probably an oversimplification to regard the reaction as simply a dehydration. It seems likely that, as in the case of the dehydration of sucrose, a mixture of products is formed including carbon oxides, sulfur oxides nitrogen oxides and steam, as well as carbon.

Further details

There is an article on the dehydration of sucrose in School Science Review, E G Meeks, *Sch. Sci. Rev*., 1979, 61 (215), 281.

Safety

- Wear eye protection.
- Your employer’s risk assessment should be consulted before carrying out this activity. This activity is *unlikely to be* covered by model (general) risk assessments used in UK schools. CLEAPSS has prepared a special risk assessment (below) that is likely to be acceptable to most employers. It is, however, the responsibility of the teacher carrying out the activity to check that this risk assessment is in fact acceptable to their employer. Bear in mind also that this may need some modification to suit local conditions.

Acknowledgement

This demonstration was developed by Dr Colin Chambers, formerly of Bolton Grammar School.

Model risk assessment

The model risk assessment below has been provided by CLEAPSS.

CLEAPSS SCHOOL SCIENCE SERVICE

Risk Assessment (to meet the COSHH and/or the Management of Health and Safety at Work Regulations)

Applicant: Royal Society of Chemistry

School/LEA: This risk assessment prepared by CLEAPSS is likely to be acceptable to most education employers in England, Wales and Northern Ireland.

Operation: **The dehydration of N-(4-nitrophenyl)ethanamide**

This risk assessment applies to the procedure as described in the accompanying RSC document as outlined below. No attempt should be made to deviate from the quantities, reagents, etc.

Details of operation

Concentrated sulfuric acid (2 ml) is added to 10 g of N-(4-nitrophenyl)ethanamide (4-nitroacetanilide) in a 100-ml beaker and gently heated. After a delay, a solid sponge of carbon forms and is ejected from the beaker in the shape of 'serpents' up to a metre high.

Substance(s) possibly hazardous to health:	(a) Concentrated sulfuric acid (b) N-(4-nitrophenyl)ethanamide (4-nitroacetanilide) (c) Various gaseous products.
Classification under CHIP3 Regulations 2002	(a) Concentrated sulfuric acid is very corrosive. (b) N-(4-nitrophenyl)ethanamide (4-nitroacetanilide) is not specified. Hazards will be sent to you by the supplier. One supplier (Aldrich) classifies this as irritant. (c) Nitrogen oxides are very toxic and corrosive. Sulfur dioxide is toxic and corrosive. Carbon monoxide toxic and extremely flammable.
Particular risks:	(a) Sulfuric acid reacts with water violently. See *Hazcard* 98. All equipment must be dry before the demonstration begins. Unreacted acid may contaminate the carbon sponge. (b) Various gaseous products will be produced from the reaction. See *Hazcards* 21 (carbon monoxide), 68 (nitrogen oxides) and 97 (sulfur dioxide).
Maximum exposure limits:	-
Occupational exposure standards: (mg m^{-3})	(a) Sulfur dioxide: 5.3 (LTEL), 13 (STEL) (b) Nitrogen dioxide: 5.7 (LTEL), 9.6 (STEL) (c) Carbon monoxide: 35 (LTEL), 232 (STEL)

Risk assessment

- The equation for the redox reaction is complex. The amounts of N-(4-nitrophenyl)ethanamide and sulfuric acid are 0.056 mol and 0.038 mol respectively. The full oxidation of the organic compound would produce a maximum of 0.112 mol of nitrogen dioxide but less than this is likely as the only oxidiser is the sulfuric acid which is mainly acting as a dehydrating agent. Carbon and carbon monoxide and will be produced in varying amounts. Reduction of sulfuric acid could produce 0.038 mol of sulfur dioxide but much less than this is likely as most of the acid acts as a dehydrating agent.

- Assuming a room of 240 m^3 and using the amounts of starting materials, the maximum atmospheric concentrations of the gases are 21 mg m^{-3} (nitrogen dioxide), 10 mg m^{-3} (sulfur dioxide) and 52 mg m^{-3} (carbon monoxide)[1]. The OES values (STEL) for nitrogen dioxide and sulphur dioxide would be exceeded and localised levels would be even higher although in practice, far less gas will be produced. Even so, because of the uncertainty, the demonstrator should work in a fume cupboard
- The demonstrator should stick precisely to the instructions, including quantities and reagents.
- The demonstrator should wear eye protection to BS EN166 3 standard or a face shield. The audience should wear eye protection
- The demonstrator should wear gloves that will protect from chemical splash.
- The pupils should be arranged about the fume cupboard so that all can see the demonstration. They should be no closer than 3 m.
- The safety of the person cleaning up the experiments should not be put at risk. It is important to dispose of any carbon sponge as directed in the notes. That person should wear eye protection and gloves.

Assessors:

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Mr R Worley, MSc, BSc *Advisor*

If further clarification is required, contact:

The School Science Service, Brunel University, Uxbridge UB8 3PH

Tel: 01895 251496

Notes

COSHH stands for Control of Substances Hazardous to Health. The regulations require that an assessment of risk should be made before substances hazardous to health are handled.

The substances covered are the reactants, the products and any intermediate or side products that are very toxic, toxic, harmful corrosive or irritant. Just because a substance carries no hazard label does not mean that it is completely safe.

The Management of Health and Safety at Work Regulations require a similar risk assessment for substances with other hazard classifications or activities involving hazardous procedures.

MEL stands for the Maximum Exposure Limit. On no account should the level of vapour exceed this value and as far as reasonably practicable the employer should see that exposure is kept as low as possible.

OES stands for Occupation Exposure Standard. Exposure should be either at the standard or preferably below. These values represent good practice. There are 2 limits. LTEL stands for long term exposure limit and is averaged over an 8 hour time weighted average (TWA) period. STEL is the short term exposure value and is averaged over a 15 minute TWA period. It is the value more relevant to schools. If a STEL is not specifically prescribed then the STEL for that substance is 3 times the LTEL value.

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[1] The concentration of carbon monoxide will be significantly less than this as much of the carbon in the organic compound forms the carbon sponge.