

# ENGINEERING FRAGRANCE

## make a deodorant

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### CHEMICAL COMPOUNDS

The most common active ingredient used in deodorants is aluminium chlorohydrate. But not all deodorants contain aluminium chlorohydrate:

*In some parts of Asia people rub their armpits with ground up crystals of alum, known as tawas, which are very similar to the active ingredients in modern antiperspirants and work in exactly the same way. Elsewhere in Asia, people apply limejuice by hand or from a cloth, and in Russia apple vinegar is a traditional remedy.*

[http://www.antiperspirantsinfo.com/english/deos\\_antiperspirants.php](http://www.antiperspirantsinfo.com/english/deos_antiperspirants.php)

### WHAT YOU HAVE TO DO

Alum is potassium aluminium sulfate, an example of a *double salt*. In this activity you will make a sample of alum and use it in a formulation for a deodorant spray.

There are two methods for making alum – *from two salts* or *from aluminium*. Your teacher will tell you which method to follow.

### EQUIPMENT

making potassium aluminium sulfate (alum) from two salts

- potassium sulfate
- aluminium sulfate-18-water [HARMFUL]
- 2 x 100 cm<sup>3</sup> beakers
- evaporating dish
- filter paper and filter funnel
- sample tube
- balance to weigh to the nearest 0.01 g
- eye protection

making potassium aluminium sulfate (alum) from aluminium

- aluminium
- 1.5 mol dm<sup>-3</sup> potassium hydroxide [CORROSIVE]
- 2 mol dm<sup>-3</sup> sulfuric acid [CORROSIVE]
- 100 cm<sup>3</sup> conical flask

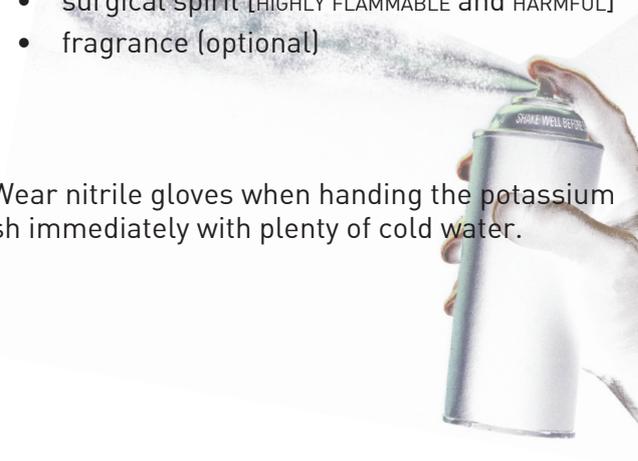
- hotplate
- 250 cm<sup>3</sup> beaker
- tripod, gauze and Bunsen burner
- thermometer (0 – 110 °C)
- ice bath
- filter paper and filter funnel
- sample tube
- balance to weigh to the nearest 0.01 g
- goggles
- access to fume cupboard

making a deodorant

- alum (this may be the sample you prepared)
- sodium hydrogencarbonate
- surgical spirit [HIGHLY FLAMMABLE and HARMFUL]
- fragrance (optional)

### SAFETY NOTES

If using the aluminium method, wear goggles. Wear nitrile gloves when handling the potassium hydroxide solution. If any gets on your skin, wash immediately with plenty of cold water.



**METHOD 1: MAKING POTASSIUM ALUMINIUM SULFATE (ALUM) FROM TWO SALTS**

compound	formula	relative formula mass	solubility at 20 °C (g / 100 cm <sup>3</sup> )
potassium sulfate	K <sub>2</sub> SO <sub>4</sub>	174	11
aluminium sulfate-18-water	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·18H <sub>2</sub> O	666	87
potassium aluminium sulfate-12-water (alum)	KAl(SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O	474	14

**Wear eye protection.**

1. Weigh out 1.74 g potassium sulfate, K<sub>2</sub>SO<sub>4</sub>. Dissolve in minimum volume of distilled water (estimate how much from the data above).
2. Weigh out 6.66 g aluminium sulfate-18-water, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O. Dissolve in minimum volume of distilled water (estimate how much from the data above).
3. Pour the two solutions into an evaporating dish and stir gently with a glass rod. Leave in a warm place to evaporate.
4. Filter the crystals that form through a fluted filter paper. Wash with a little water.
5. Carefully take the filter paper out of the funnel and lay it on a watch glass. Leave it somewhere warm to dry overnight. Put a large piece of filter paper over the top to prevent dust and dirt getting into your sample.
6. Weigh a labelled sample tube. Scrape the dry potassium aluminium sulfate-12-water into the tube and weigh again.

**RESULTS**

weight of sample tube = \_\_\_\_\_ g

weight of sample tube + potassium aluminium sulfate-12-water = \_\_\_\_\_ g

weight of potassium aluminium sulfate-12-water = \_\_\_\_\_ g

The theoretical yield of alum from 1.74 g potassium sulfate and 6.66 g aluminium sulfate-18-water is 4.74 g. Calculate the theoretical yield from the masses of potassium sulfate and aluminium sulfate-18-water you used.

$$\text{percentage yield of alum} = \frac{\text{actual yield of alum} \times 100}{\text{theoretical yield of alum}} \%$$

Calculate the percentage yield of alum in your preparation.

**EXPLANATIONS**

Using the data from the table given earlier and these relative atomic masses:

H 1	O 16	Al 27	S 32	K 39
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explain why theoretical yield of alum from 1.74 g potassium sulfate and 6.66 g aluminium sulfate-18-water is 4.74 g.

**METHOD 2: MAKING POTASSIUM ALUMINIUM SULFATE (ALUM) FROM ALUMINIUM**

1. You will be given a piece of aluminium foil weighing about 0.5 g. Cut it into small pieces.
2. Weigh a 100 cm<sup>3</sup> conical flask to the nearest 0.01 g. Tip in the pieces of aluminium foil and weigh the flask again. Record your weighings and calculate the mass of aluminium in the flask:

weight of beaker = \_\_\_\_\_ g

weight of beaker + aluminium = \_\_\_\_\_ g

weight of aluminium = \_\_\_\_\_ g

**Carry out the next stages in a fume cupboard; wear goggles (not safety spectacles).**

3. Slowly pour 25 cm<sup>3</sup> of 1.5 mol dm<sup>-3</sup> potassium hydroxide solution into the flask. Warm the mixture gently on a hotplate. Swirl the flask occasionally until all the aluminium dissolves.
4. Allow the solution to cool. Filter through a fluted filter paper into a 250 cm<sup>3</sup> beaker. Let the filtrate cool completely.

**Your teacher may help you with this next step.**

5. Stand the beaker containing the filtrate in an ice bath. Cool the solution to about 10 °C (check with a thermometer). Carefully add 50 cm<sup>3</sup> of 2 mol dm<sup>-3</sup> sulfuric acid a little at a time. Stir well between additions. A white precipitate of aluminium hydroxide should form and then dissolve.

6. Put the beaker on a tripod and gauze. Using a Bunsen burner, bring the solution slowly to the boil. Simmer until the volume is about half the initial volume.

Note: If there is any solid that does not dissolve, filter the mixture through a fluted filter paper into another clean beaker.

7. Let the solution cool to room temperature and then place it in an ice bath. Leave for 20 minutes, stirring occasionally with a glass rod. Crystals of alum should form.
8. Filter the cold mixture through a fluted filter paper. Wash the crystals on the filter paper with a very small quantity of ice-cold distilled water.
9. Carefully take the filter paper out of the funnel and lay it on a watch glass. Leave it somewhere warm to dry overnight. Put a large piece of filter paper over the top to prevent dust and dirt getting into your sample.
10. Weigh a labelled sample tube. Scrape the dry alum into the tube and weigh again.

**RESULTS**

weight of sample tube = \_\_\_\_\_ g

weight of sample tube + alum = \_\_\_\_\_ g

weight of alum = \_\_\_\_\_ g

The theoretical yield of alum from 1.0 g aluminium is 17.56 g.

Calculate the theoretical yield from the mass of aluminium you used

$$\text{percentage yield of alum} = \frac{\text{actual yield of alum} \times 100}{\text{theoretical yield of alum}} \%$$

Calculate the percentage yield of alum in your preparation.

**EXPLANATIONS**

The equation for the overall reaction is:



Alum crystallises from solution. The crystals have the formula  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

Using these relative atomic masses:

H 1	O 16	Al 27	S 32	K 39
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explain why theoretical yield of alum from 1 g aluminium is 17.56 g.

## MAKING A DEODORANT

Many claims have been made for various uses of baking soda (sodium hydrogencarbonate), e.g. [www.thefarm.org/charities/i4at/lib2/60soda.htm](http://www.thefarm.org/charities/i4at/lib2/60soda.htm). One is that it can remove some unpleasant odours from the air. Another, possibly related, claim is that it 'makes an excellent underarm deodorant' ([www.tipking.co.uk/tip/1609.html](http://www.tipking.co.uk/tip/1609.html)). At this website: [http://www.bakingsodabook.co.uk/Make\\_a\\_baking\\_soda\\_based\\_deodorant.shtml](http://www.bakingsodabook.co.uk/Make_a_baking_soda_based_deodorant.shtml) you can find a 'recipe' for a deodorant spray made from baking soda, alum and alcohol. The method below is based on this.

## METHOD

### **Wear eye protection.**

1. Weigh 0.5 g alum and 0.25 g sodium hydrogencarbonate into a small beaker.
2. Add 7.5 cm<sup>3</sup> of water and stir until the solids have dissolved.
3. Add 15 cm<sup>3</sup> of surgical spirit and stir well. At this stage you may want to add a little fragrance.
4. Pour the solution into a small clean spray bottle.

You may try spraying it on the back of your hand, but check with your teacher first.

## QUESTION

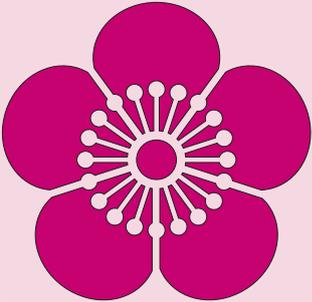
*"Everybody sweats. We have to. Perspiration is the body's biological way of cooling down. Sweat itself does not smell, but it is a wonderful culture for the bacteria that live on our skin. The bacteria breaks down sweat into aromatic fatty acids, which produce the unpleasant odour."*

<http://www.netdoctor.co.uk/hilaryjones/embarrassingprobs/bodyodour.htm>

This means body odour can be tackled by:

- reducing the amount of sweat
- treating the bacteria that produce the odour.

Explain the role of the three ingredients in the body spray you have made (four ingredients if you included scent).



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## make a deodorant

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### HEALTH AND SAFETY

A risk assessment must be made before starting any practical work. Particular care is needed with the preparation of potassium aluminium sulfate from aluminium. Goggles are essential because KOH and  $H_2SO_4$  are corrosive. Students should be supervised carefully.

### THE INVESTIGATION

Students make a sample of potassium aluminium sulfate (alum) and use it in a formulation to make a deodorant spray.

In the video students watch the production of Lynx deodorant. The active ingredient is aluminium chlorohydrate. Scientists are not entirely sure how it works, but it appears to form a plug of aluminium hydroxide that blocks the sweat duct. Aluminium chlorohydrate has largely replaced aluminium chloride-6-water in deodorants though treatments for hyperhidrosis (a medical term for excessive sweating) usually contain it.

Some deodorants, however, do not contain aluminium chlorohydrate. They are often called 'crystal deodorants' which are crystals of potassium aluminium sulfate (alum). They are known in some parts of Asia as tawas. Other deodorants contain ammonium aluminium sulfate. Some useful references:

- <http://www.crystalspringltd.co.uk/>
- <http://www.lonatural.ukcentre.com/>
- [http://www.opal-london.com/products\\_info.asp?g=%7B3CD28129-2FBE-4DF3-8DF7-0DC5C5724C36%7D](http://www.opal-london.com/products_info.asp?g=%7B3CD28129-2FBE-4DF3-8DF7-0DC5C5724C36%7D)
- <http://www.mariposa-alternative-bodycare.co.uk/index1.html>

### SUGGESTED SEQUENCE

Working individually or in pairs, students start by making a sample of potassium aluminium sulfate (alum). Two methods are available. Choose which method you would like students to follow. The first (from two salts) is a straightforward preparation. The second (from aluminium) is more complex – students must be supervised.

Students then use the alum they have made to make a deodorant.

#### **Time required: Making potassium aluminium sulfate (alum) from two salts**

About one hour to make the crystals which will be left overnight to dry. A further 15 minutes once the crystals are dry.

#### **Time required: Making potassium aluminium sulfate (alum) from aluminium**

Allow about 30 minutes for steps 1-4 and a further 30 minutes for steps 5 and 6. The next step requires time as the crystals form. It might be convenient to put the covered beaker in a refrigerator overnight. The remaining steps will take a further 30 minutes.

#### **Time required: Making a deodorant**

About 20 minutes.

## NOTES

### Making potassium aluminium sulfate from two salts

This is a straightforward preparation. It involves the preparation of a double salt by mixing solutions of two single salts (potassium sulfate and aluminium sulfate) and crystallising alum from the mixture. Students are instructed to use the minimum volume of water to dissolve each salt. They may estimate this in advance from the solubility data provided:

1.74 g potassium sulfate requires about 15.8 cm<sup>3</sup> of water; students might use, say, 20 cm<sup>3</sup>

6.66 g aluminium sulfate-18-water requires about 5.8 cm<sup>3</sup> of water; students might use, say, 8 cm<sup>3</sup>

Students measure the actual yield and calculate the percentage yield. They are asked to explain why theoretical yield of alum from 3.48 g potassium sulfate and 6.66 g aluminium sulfate-18-water is 4.74 g, given the data:

Potassium sulfate	K <sub>2</sub> SO <sub>4</sub>	relative formula mass 174
Aluminium sulfate-18-water	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·18H <sub>2</sub> O	relative formula mass 666
Potassium aluminium sulfate-12-water	KAl(SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O	relative formula mass 474

From their chemical formula it can be seen that

1 mol potassium sulfate (K<sub>2</sub>SO<sub>4</sub>) and 1 mol aluminium sulfate-18-water (Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O) give 2 mol potassium aluminium sulfate-12-water (KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O)

1.74 g potassium sulfate = 0.01 mol; 6.66 g aluminium sulfate-18-water = 0.01 mol

Theoretical yield = 0.005 mol potassium aluminium sulfate-12-water = 474 × 0.005 = 2.37 g

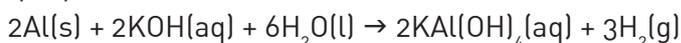
Complete crystallisation of the solution would yield 2.37 g, i.e. a 100% yield. However, following the method suggested, some alum will remain in solution and the percentage yield will be less than 100%.

### Making potassium aluminium sulfate from aluminium

This is a more complex and difficult preparation. Students must be supervised and wear goggles. The first step should be carried out in a fume cupboard. Hydrogen is released and there should be no naked flames nearby. The rate of reaction should be carefully controlled. If it is beginning to become too vigorous, take the reaction mixture from the hotplate for a short period.

The neutralisation reaction is exothermic and care must be taken that the addition of 2 mol dm<sup>-3</sup> sulfuric acid is slow enough for the heat to be dissipated between additions.

The preparation involves a number of reactions:



Followed by crystallisation to give KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O.

However, students are given the overall equation:



from which they can see that 2 mol aluminium produce 2 mol alum.

$$1 \text{ g Al} = 1/27 \text{ mol Al}$$

This can produce 1/27 mol alum.

The relative formula mass of alum is 474.

There, theoretical yield of alum = 474 × 1/27 = 17.56 g

## TECHNICIAN EQUIPMENT LIST

making potassium aluminium sulfate (alum) from two salts

- potassium sulfate
- aluminium sulfate-18-water [HARMFUL]
- 2 x 100 cm<sup>3</sup> beakers
- evaporating dish
- filter paper and filter funnel
- sample tube
- balance to weigh to the nearest 0.01 g
- eye protection

making potassium aluminium sulfate (alum) from aluminium

- aluminium
- 1.5 mol dm<sup>-3</sup> potassium hydroxide [CORROSIVE]
- 2 mol dm<sup>-3</sup> sulfuric acid [CORROSIVE]
- 100 cm<sup>3</sup> conical flask
- hot plate
- 250 cm<sup>3</sup> beaker
- tripod, gauze and Bunsen burner
- thermometer (0 – 110 °C)
- ice bath
- filter paper and filter funnel
- sample tube
- balance to weigh to the nearest 0.01 g
- goggles
- access to fume cupboard

making a deodorant

- alum (this may be the sample you prepared)
- sodium hydrogencarbonate
- surgical spirit [HIGHLY FLAMMABLE and HARMFUL]
- fragrance (optional)