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newscientist

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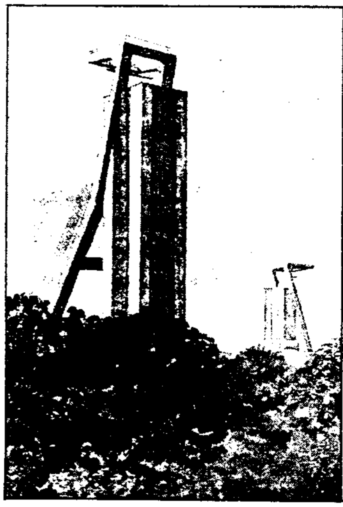

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Do-it-yourself superconductors

It is extremely easy to make high-temperature superconductors. Schools in the United States and Britain have already produced their own samples. Here is the recipe

Paul Grant

ANY moderately equipped secondary school should be able to make and test high-temperature superconductors at very reasonable cost. This is already beginning to happen in the United States. On 29 May, nine high-school students and their chemistry teacher at Gilroy High School in California became the first group of pupils to make samples of the perovskite material and to demonstrate its superconducting properties by magnetic levitation. In Britain, pupils at Helsby High School in Cheshire have also made the new material (New *Scientist,* 9 July, p 14).


IBM

"Shake 'n' bake" recipe for 1-2-3 ($YBa_2Cu_3O_{7-x}$)

Mix 1·13 grams yttrium oxide, 3.95 grams barium carbonate. 2.39 grams copper oxide
Compact
Grind in mortar and pestle
Bake in air at 950 °C (1650 °F)
Regrind in mortar and pestle
Press into pellets
Rebake pellets in flowing oxygen at 950 °C (1650 °F)
Allow to cool very slowly
Recipe by Heidi Grant

Left: Heidi Grant demonstrates superconductivity at the US National Science Foundation

Gilroy is a small Californian agricultural community of population 23 000, about 130 kilometres south of San Francisco. The town, noted locally for its excellent garlic, was founded in the early 19th century by John Cameron Gilroy, a former sailor in the Royal Navy who, according to local legend, was either put to shore in nearby Monterey because of scurvy or jumped ship after hitting an officer.

The students and teacher at Gilroy High obtained the materials they needed to make the superconductor themselves. They used the school's resources in addition to what they could borrow locally. The only help they received from us at IBM was knowledge of how to process the starting materials. Otherwise, the pupils were on their own, and they managed to duplicate one of the most dramatic scientific breakthroughs of the past 40 years and perhaps of this century. They did this without elaborate resources or the expensive funding found only at major national or international research facilities. I would like to tell you a little of their story and disclose enough details so that science teachers in other secondary schools can carry out experiments similar to the work at Gilroy.

Naturally, I am most familiar with American schools, but I will try as best I can to relate also to the British audience. Please remember that these experiments involve materials and procedures that can be hazardous if improperly used and therefore should be performed by young people only under the guidance of a qualified and experienced senior person.

I am a physicist at the IBM Almaden Research Center in California. I have worked on organic superconductors. I am now investigating the class of new high-temperature materials initially discovered by George Bednorz and Alex Müller, our colleagues at IBM's research laboratory in Zurich. The initial work of our group at Almaden has focused on understanding the structure and production of the new compounds. We were among the first to unravel the crystal structure of the new material, which remains a superconductor at the temperature of liquid nitrogen, and to reveal how best to make the materials and to optimise their superconducting properties ("The heat is on for superconductors," New *Scientist,* 7 May, p 46).

After the "Woodstock of physics" meeting last March in New York City, when several thousand scientists stayed up for an all-night session on these new materials, I was asked to brief a team of visiting British journalists on our activity in high-temperature superconductivity, and also to lecture on the same topic to the administrative staff at our laboratory. For these assignments, I wanted to put together a simple yet dramatic demonstration of superconductivity-more than just showing electrical resistance dropping to zero on a meter.

Another property of a superconductor, beyond its zero resistance, is its perfect diamagnetism. That is, a magnet placed near a superconductor will literally "see" its mirror image and, because like poles repel, superconductor and magnet will try to move away from each other. This is called the "Meissner effect".

The apparatus I was able to rig up most quickly at the time was a pendulum (see photograph p 37). The traditional way of

Safety in the laboratory

AS WITH all laboratory work, it is important to adopt safe practices when making the 1-2-3 superconductor. In particular, it is important to avoid coming into contact with dangerous substances such as chemicals and liquid nitrogen. Use a dust mask and wear safety goggles at all times and handle the materials with care. Grind the ingredients in a fume cabinet.

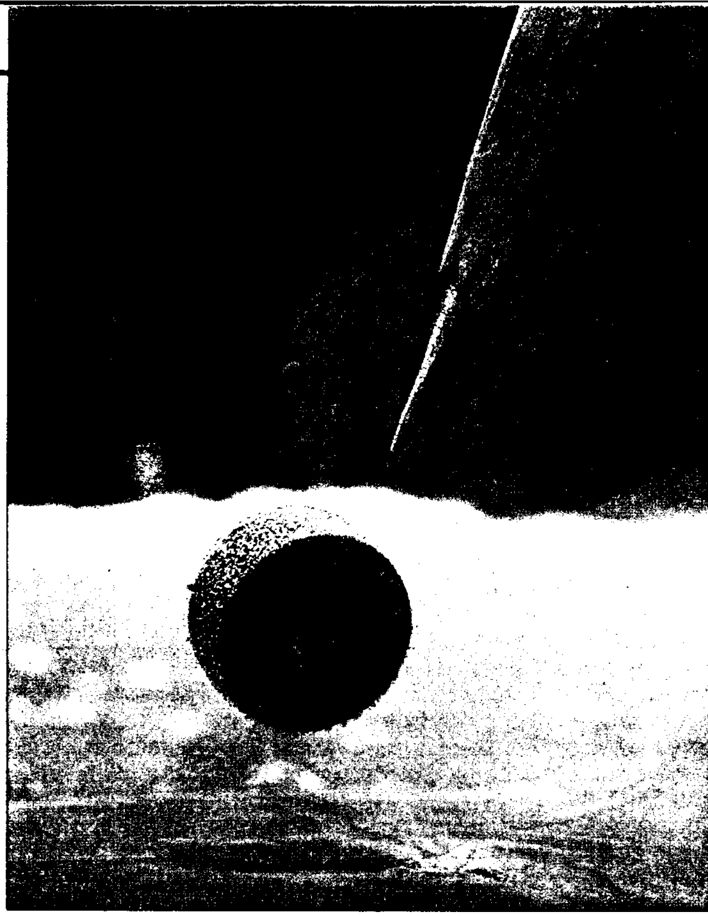
Here are some other points to remember before rushing to the laboratory to make this new material.

If you place a pellet of the 1-2-3 material in warm water, it will partially dissolve, creating a solution of barium hydroxide. This is caustic. The best way to handle the pellet is with plastic tweezers, which are also useful for retrieving magnet slivers which fall off the pellet into the liquid nitrogen! An unknown property of the superconductor is its toxicity. Preliminary studies indicate that under acidic conditions-in the stomach for example -barium can be leached from the superconductor. So do not swallow the material. And keep pellets of the material away from small children who might swallow it.

Another safety concern is liquid nitrogen, which can cause frostbite. Handle the material with care and follow any precautions suggested by the suppliers. □

performing this demonstration, however, is to levitate a small, light magnet over the superconductor. At a certain equilibrium height, the weight of the magnet is balanced by the magnetic repulsion between the magnet and its image in the superconductor.

The difficulty in the past was that you needed liquid helium to' get the temperature down to a few degrees above absolute zero to carry out the experiment. And that needs elaborate refrigeration. With the new materials, their critical temperature is so high that you can repeat this demonstration using liquid nitrogen and insulating materials made of expanded polystyrene (ordinary disposable coffee cups), a small samarium-cobalt magnet, and a pellet of the superconductor like the one tied to the string in the photograph (right)—all put together on a table top. Photographs of magnetic levitation using larger samples of the high-temperature superconductor have since appeared in at least two major American magazines, *Time* and US News *& World Report,* in numerous newspapers and on American television. IBM has made a video showing levitation and other aspects of high-temperature superconductivity. The BBC has also broadcast a demonstration of magnetic levitation.


IBM

Hovering on the brink of a revolution-the Meissner effect in action

The hard work with these new materials was to determine their structure and to define the best way to make the $YBa_2Cu_3O_{7-x}$, the superconductor that caused all the excitement. Having done that it was relatively easy to make the high-temperature superconducting compound, which we nicknamed l-2-3 after the ratio of its three cations.

It occurred to me that perhaps students at the average American high school could prepare the material and demonstrate superconductivity by magnetic levitation. I made some local inquiries and found that most high schools have enough equipment. At about the same time, I gave my daughter, Heidi, a pellet of l-2-3 and a sliver of samarium cobalt. She took these materials and some liquid nitrogen to her science class and performed the magnetic levitation experiment for her classmates using a simple Styrofoam pie plate to hold the superconductor and refrigerant.

It was a timely demonstration. Her class at Dartmouth Middle School in San Jose had just finished studying Ohm's law. The kids could appreciate what superconductivity was all about and were astounded when they saw the magnet lift off the l-2-3 pellet as it became superconducting! The experience convinced me that one of the immediate benefits of this great breakthrough would be in education.

Word got around about Heidi's demo. Paul Chu, of the University of Texas, Houston, and the man who made the first material that was a superconductor above 90 K, invited Heidi to go to Washington and repeat her experiment in front of the National Science Board, the governing agency of our National Science Foundation. She did this on 22 May, distributing to the board members kits containing pellets of l-2-3 that she had made by herself, working evenings with me in my lab at IBM. I wanted to see a high-school science class making kits with as little outside help as possibie.

My first task was to find a high-school science teacher willing to take on the challenge. I had met just such a person about a year before, through one of IBM's programmes of support for public education. David Pribyl is a chemistry teacher and head of the science department at Gilroy High, about 50 kilometres south of IBM. We invited Dave to the lab, told him what we had in mind. He enthusiastically took on the job, assigning nine of his best students to this project. We told him only the starting materials and what he had to do with them. His "recipe" is also the one Heidi followed.

The first problem is to get the starting materials, yttrium oxide (Y_2O_3), barium carbonate ($BaCO_3$) and copper oxide (CuO). The students at Gilroy scrounged their materials from the chemistry department of a local junior college.

One final comment on purity: the compounds do not have to be extremely pure (the term in the US is "spectroscopic grade"), but you would be well advised to obtain at least "reagent grade" material. The $BaCO_3$ they first used at Gilroy was "ceramic grade" and contained a lot of iron. Perovskites made from it did not exhibit superconductivity, owing to the paramagnetic iron impurity. Avoid large amounts of transition-metal impurities as they may substitute for the copper in l-2-3 and their intrinsic magnetic moments will kill the superconductivity.

Weigh out the proportions of each compound, as shown in the recipe, for mixing. The amounts given in the recipe will yield nearly 7 grams of l-2-3 (we leave it as an exercise for teachers and their students to prove that this number and those in the recipe are correct). Then mix materials with a mortar and pestle, grinding until you get a line powder, 5 to 10 minutes is usually sufficient. (A cautionary note here: $BaCO_3$ and CuO can be toxic if inhaled or ingested, I strongly advise you to use a dust mask, safety goggles and to grind the ingredients in a chemical hood. You should follow these and other normal chemical safety procedures at all times.)

You now have to react, or "tire", the lightish grey mixture of the three compounds at 900-950 °C in air for around 12 hours. How you do this depends on what equipment you have available. Most art departments in high schools in the US have kilns for work on ceramics and jewellery. I was amazed to find how widely available these kilns are. The furnace at Gilroy is typical. It consists of tirebrick surrounding a cavity of approximately one cubic foot with an insulated hinged door. Such kilns can reach the required temperatures. Unfortunately, their thermostats are often inaccurate. The first attempt by the Gilroy kids resulted in a melted mess because their thermostat on their furnace was in error by about 100 "C. Do not go much above 950 °C or the same will happen to you. It is a good idea to calibrate your furnace using a thermocouple if you are in any doubt about its temperature. (A chromel-alumel couple works fine and you can usually find one in the school physics lab.)

You should put the mixed material in an alumina boat or crucible and insert this into the furnace. If you do have a thermocouple, put its tip near, not in, the mixed compound in the boat. Choose your crucibles with care. We have found that the mixture reacts with many varieties of alumina and quartz. One brand of crucible that works well is the high-alumina crucibles made by Coors Porcelain Company (that's right, related to the famous American beer makers). A convenient size is a boat around 12 millimetres high, 17 millimetres wide and about 90 millimetres long. You can get by with lower grade boats and crucibles, -but they won't last and you have to reject any material that was in direct contact with the crucible. As with other items in this project, teachers may be able to "liberate" suitable vessels from local firms and colleges.

Another important factor in the production of high-temperature semiconductors is the cleanliness of the kiln you are using. If the kiln has been used primarily for work with ceramics and metal, contamination should not be a problem. Any carbon and organic substances left in the kiln will be completely pyrolised at the temperatures used. For example, the carbonate (CO,) anion in $BaCO_3$ dissociates into carbon dioxide and oxygen.

After about 12 hours, you should turn off the kiln and leave it to cool. Exactly how long this cooling takes depends on the thermal insulation of the unit-on average it takes about 5 or 6 hours to reach 100 "C. The reacted material should look black. Any green tint means something went wrong, most likely you made a mistake in calculating or measuring the starting proportions. If you get it right, the product will be a weakly fused mass, easily broken apart with a metal spatula.

Now press on

You now have the l-2-3 superconductor, but not in a usable shape. You must now grind it up again with mortar and pestle in preparation for pressing it into pellets. Obtaining or making the pellet press probably presents the greatest challenge in the project. High schools in the US generally have well-equipped machine shops with hydraulic presses and the ability to machine an appropriate pellet die and anvil. However, you would save time by borrowing the die and anvil assembly from a nearby firm or college. At IBM we use a pellet press assembly one-half inch in diameter designed to make samples for infrared spectroscopy. IR spectroscopy is a common analytical tool in many industries and it should not be too difficult to find such a unit. We lent the students at Gilroy one of ours and they pressed their own pellets using the hydraulic jack in their own machine shop. If the school's machine shop does not have a hydraulic press, you should be able to find one in a car-body repair shop. Try your local pharmacist, where they use such presses to make pills.

A pressure of 15 000 to 18 000 pounds for a half-inch pellet will more than do the job. You should put enough of the ground-up l-2-3 into the pellet die to make a pellet about 1/32 inch (0·8 millimetres) thick. The amounts detailed in the recipe should produce enough material to make about half-a-dozen pellets. Handle the pellets carefully at this stage: they are fragile and break easily. If this happens, simply regrind and repress.

As the recipe shows, you now have to bake, or anneal, the pellets again. There are two reasons for this second stage: first, to sinter or fuse the grains in the pellet to improve the mechanical and electrical properties, and secondly, and very importantly, to equilibrate thoroughly the oxidation of the l-2-3, an essential process to make a good superconductor. It is best, but not essential, to anneal your material for the second time in flowing oxygen. The kids at Gilroy obtained a tank of oxygen from their machine shop, where pupils use it for welding. They fed the oxygen into their kiln through a flexible hose, which they attached to a stainless-steel tube a half a centimetre in diameter through a "breather hole" at the top of the furnace. You don't need much oxygen; if you can just feel the gas flow with your lips, that's enough.

Where to buy your bits

A number of companies can supply the starting materials to make l-2-3 superconductor, including two American companies with British subsidiaries. The compounds do not have to be terribly pure (99.9 per cent is more than adequate). They are quite cheap. However, teachers should tap local companies and colleges for the materials. Ten grams of each goes a long way. Some laboratories may already hold supplies of copper oxide and barium carbonate. Yttrium oxide is harder to come by and you may have to buy some. Unfortunately, it is in short supply at the moment

Where to obtain materials in Britain

Starting compounds: Ventron-Alpha Products, Station Tower, Station Square. Coventry CV1 2GH; Aldrich Chemicals, The Old Brickyard, New Road, Gillingham, Dorset SP8 4JL.

Ventron-Alpha quoted the following prices: f20 for 50 grams of yttrium oxide; f4.60 for 100 grams of barium carbonate; and f 16.50 for 25 grams of "ultrapure" copper oxide.

Suppliers of samarium cobalt magnets: Edmund Scientific, 101 E Gloucester Pike, Barrington, New Jersey, NJ 08007, US.

Liquid nitrogen costs about f 1 per litre in small quantities from Air Products, Hershan Place, Molesey Road, Walton-on-Thames; or from Customer Relations Department, BOC Ltd, The Priestley Centre, The Surrey Research Park, Guildford, Surrey GU2 5XY.

As in the first reaction, heat the material at 950 °C for 12 hours. Use the same boats or crucibles to anneal the pellets as you used for the initial mixture. You now have to be very careful about how you cool your material. At this stage this is far more important than it was in the first cycle. It is best to reduce the temperature no faster than 100 °C per hour, with oxygen flowing if you can manage it. The crucial temperature region is from 700 to 400 "C. Between these temperatures, the l-2-3 undergoes a transition from the tetragonal to orthorhombic phase and the material takes up most of the oxygen that it needs to become a good superconductor. You now see the origin of the "x" in the formula for l-2-3, $YBa_2Cu_3O_{7-x}$. It is a measure of the amount of oxygen that is incoporated into the material. As far as we can tell, x should be as close to zero as possible to get the best superconductivity in 1-2-3.

You must cool the material slowly to get x near to zero. This raised a problem for the Gilroy team. The furnace had to be on for about 20 hours to anneal and cool the material. At IBM this is no problem, we run our kilns all night unattended. However, insurance requirements and safety regulations at the high school meant that someone had to monitor the electrical equipment at all times. Moreover, the school's rules do not allow students to be in school buildings overnight unless there is a teacher to supervise them. So Dave Pribyl and the students had to spend several long nights tending cool-down cycles, a real test of their dedication. Actually, at IBM we react and anneal our materials for 12 hours primarily because it's most convenient for us to start up runs in early evening and let them go overnight. We have not tried to see if you can curtail the second stage. This might be a good project for some high school to attempt!

Once you have annealed the pellet, you can try the levitation test. You need four more items for this final step: a magnet made of samarium cobalt; liquid nitrogen; something to hold the pellet, magnet and liquid nitrogen; and a small loop of wire that you can pass around the levitated magnet to prove to sceptics that "no strings are attached".

You need samarium-cobalt, $SmCo_5$, for the magnet, because you want a large dipole moment-to-weight ratio so that your magnet will hover as high as possible above the surface of the pellet. You want a piece of magnet no more than one-third the pellet's diameter. You can easily "make" such small slivers from a standard piece of magnet by wrapping it in a cloth and hitting it with a hammer. Samarium cobalt is brittle, and a hammer blow should give you a number of slivers suitable for the levitation experiment.

Of course, what makes the new superconductors exciting is that liquid nitrogen can now be the refrigerant. This is cheap, plentiful, easy-to-store and nonpolluting (it's 80 per cent of our atmosphere). Before last year, the Stone Age of superconductivity, liquid helium was the only practical refrigerant for superconductors. This is difficult to obtain in some countries (the US and the Soviet Union practically have a monopoly on the supply), expensive (about £5 per litre if you buy large amounts) and difficult to store.

I have worked with liquid helium most of my career. Only on a dozen or so occasions have I actually seen it! Almost always, liquid helium hides in closed steel dewars, doubly insulated and entirely invisible. Liquid nitrogen, on the other hand, has widespread uses. Your "neighbourhood dermatologist" will use it to freeze and remove warts! The students at Gilroy obtained their liqud nitrogen from just such a source-a helpful local physician. Hospitals these days also use liquid nitrogen, so don't overlook them as possible donors.

The "cryogen" is used by many plastics-forming firms. If there is one in your town, you should be able to pry away a few litres with no difficulty. Any local high-tech company or university is an obvious possibility. Finally, if you have to pay for it, you can buy small amounts from welding shops, at least here in California, but it's expensive that way. They charge about £ 10 to till a 3-litre Dewar, but the price goes down rapidly the more you buy.

Speaking of Dewars, you need one to store the liquid nitrogen if you don't already have a proper container. Perhaps you can borrow one. If not, a large Thermos flask will serve, preferably stainless steel rather than glass or hard plastic as these may crack when you fill it. Remember not to seal the Thermos-the liquid nitrogen is always boiling and will expand and probably explode your bottle! You can put small amounts of liquid nitrogen into polystyrene coffee cups; these are especially convenient to use during the levitation experiment. Do remember, though, that liquid nitrogen is very cold. If you spill even a small amount onto yourself you can get serious frostbite. You should use safety glasses and wear gloves while pouring liquid nitrogen-be especially wary of spillage that runs along the table onto your clothes.

Finally, you need something to hold the pellet, magnet and liquid nitrogen. You need a good insulator so that the liquid nitrogen will not boil away too quickly. Polystyrene is a good material-you can cut off the bottom of a coffee cup, for example. We have found that a pie plate from the school cafeteria works very well. Simply place the pellet in the middle of the pie plate, lay the magnet on top, and carefully pour in a little liquid nitrogen. The nitrogen will fizz rather violently for a while as it cools down both the pellet and the surface of the pie plate.

Try not to submerge the pellet-just surround it with liquid nitrogen until you see air beginning to condense on its top as a dark film. When this happens, you are very close to some excitement. The magnet should lift off in a few seconds and levitate. Watch closely with the surface of the pellet at eye level. (Don't forget the safety goggles.) The magnet may twitch slightly just before taking off. The feeling you now have as the magnet levitates and you pass the loop of wire around it is indescribable if you have the slighest sense of wonder. The best I can do is quote one of the students at Gilroy as reported. in a local newspaper. "Oh, it was totally terrific," said Jessica Roney, 16. "I Jumped four feet in the air. It was overwhelming."

The week after they "got it", I invited Pribyl and his students up to our lab for a celebration lunch and a tour. When he had first called to tell me they had succeeded, I was just about as excited as I was a few months earlier in March when our group had cracked the structure of l-2-3. A group of high-school chemistry students had repeated one of the monumental discoveries of the physics of condensed matter. No longer would superconductivity remain accessible only to those few laboratories with specialised and expensive refrigeration facilities. Superconductivity, an esoteric subject even for physicists, had been put in the hands of the people for their own investigation and education. There has been much speculation in technical circles and the popular press as to the first practical application of these high temperature superconductors. In my mind, the first application has already happened-it is to science education.

In addition to their experimental success and further understanding of superconductivity, the students learnt another key lesson-the importance of perseverance in scientific endeavour. At lunch I asked the pupils how they felt the two times they failed to make a pellet that worked. Of course, they had all been depressed. I then asked them to reflect on Müller and Bednorz and how they had tried and failed for almost three years, making and measuring dozens and dozens of pellets of differing materials and compositions in pursuit of an idea they had which they felt would lead to a new class of superconductors. The students immediately got the message. They recognised that although they had failed on two occasions, they were secure in the knowledge that because l-2-3 did actually exist, success would eventually follow. Previous work had placed no such stake in the ground for the two Swiss researchers. They had only their faith in their unproven model to keep them going failure after failure until they achieved their spectacular breakthrough barely a year ago. Yet they kept at it. Maybe this was the most important lesson for the students. □


The Dispatch Gilroy

Pupils at Gilroy High School in California make their own high-temperature superconductor

Paul Grant is a research scientist at IBM's research centre at Almaden in California. Before switching to the new high-temperature materials, he worked with organic superconductors.