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Chemistry Discourse

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Subject: **P2P tube furnace write up attempt**

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Down

Mastermind
(Hive Bee)
07-23-00
01:44
No 31892

P2P tube furnace write up attempt

OK, Osmium. This is what I came up with for a write up (P2P from PAA and acetic acid; tube furnace method). Since the apparatus isn't too common it might be confusing to some people.

Description of the apparatus

The apparatus utilizes a 98% silica glass (pyrex also works according to Organic Synthesis) combustion tube (referred to as catalyst tube here) about 1.1 meters long and about 2.5 cm in diameter with a ground glass 29/42 female to 24/40 male straight adapter glued onto the outlet end of the catalyst tube using epoxy glue. The thermocouple used to monitor the temperature includes a number of cylindrical ceramic beads (arranged in a line), each having two holes, with each hole adapted to receive a thermocouple wire. The cylindrical beads hold the two thermocouple wires parallel to each other while providing electrical insulation. The thermocouple wires are twisted at one end and bent upward slightly so that they contact the catalyst tube when held against it. The thermocouple is placed about 0.3 meters from the inlet end and on the outside of the catalyst tube (pointing away from the inlet end of the catalyst tube) and the catalyst tube and thermocouple are wrapped with a heating tape rated at about 800 degrees C maximum along about 0.5 meters length at the center of the catalyst tube leaving about 0.3 meters uncovered at both ends of the catalyst tube. Glass ribbons or strings provided at each end of the heating tape are tied around the tube to hold the heating tape and thermocouple in place. The portion of the catalyst tube covered with the heating tape is then wrapped with glass wool for insulation. A glass wool plug is placed through the outlet end of the glass tube and pushed using a wooden or glass rod to the point where the heating tape ends to support the catalyst.

The part of the catalyst tube covered with the heating tape is filled with pea sized pumice supporting the catalyst (described later) and supported using standard ring stands and clamps at an angle of about 10-15 degrees from the horizontal. An angled (110 degree) adapter adapted to receive a #4 stopper at one end and narrows to about 3/8 inches in diameter at the other end is connected to the inlet of the catalyst tube using a one hole stopper. A two hole #4 stopper supporting a separatory or dropping funnel and a short piece of glass tubing having a 90 degree bend is connected to the inlet of the angled adapter. A standard angled (110 degree) adapter having 24/40 ground glass joints at both ends is connected to the straight adapter at the outlet or lower end of the catalyst tube and a round bottom flask is connected to the outlet end of the vacuum adapter to receive the products from the catalyst tube. A length of rubber tubing is connected to the side arm of the vacuum adapter. A stiff piece of tubing, such as, metal or glass can be connected to the other end of the rubber tubing and can be lead outside through a window, to a window fan or, preferably, to a container of water to allow observation of the rate of gases passing through the catalyst tube by observing bubbles produced in the container of water.

The separatory funnel at the inlet end is used to control the addition of a mixture of PAA and acetic acid in the catalyst tube and the 90 degree piece of glass tubing at the inlet end (supported by the two hole #4 stopper) is used to introduce air to process or regenerate the catalyst, or to introduce an inert gas, such as CO₂ or N₂, into the catalyst tube when making the ketone.

Here's a picture of the tube furnace:



Mastermind
(Hive Bee)
07-23-00
01:47
No 31893

Re: P2P tube furnace write up attempt

This is the catalyst I described in the newbie section.

The catalyst.

The magnesia catalyst was made using pea sized pumice (some patents were seen supporting similar catalytic materials using perlite for cracking petroleum hydrocarbons; other art). Enough pea sized pumice to fill FOAF's combustion (now catalyst) tube was

covered with enough Milk of Magnesia so that the pumice 'had looked like it had been white washed' (good description) and at the same time the pores were not clogged (for maximum catalytic surface area). A 98% silica combustion tube was used which was wrapped with a heating tape (max temp 800 degrees C) and was covered with glass wool as insulation. 2-3 teaspoons of calamine lotion (ZnO and Fe oxide; see US patent 5750795, for example, especially tables 1 and 2) was added to the catalyst and it was heated, alternately, on a gas stove and in a microwave oven to dry it and convert it to MgO. It was found that when the catalyst was placed in the tube and heated to about 500+ degrees C (with air pumped through it) that the $Mg(OH)_2$ could more easily be dehydrated (H_2O given off and condensed) and converted to MgO.

*Note: add calamine lotion before putting in combustion tube

Mastermind
(Hive Bee)
07-23-00
01:49
No 31894

Re: P2P tube furnace write up attempt

P2P tube furnace process.

The tube furnace is heated to 430-450 degrees C, and simultaneously the tube is swept out thoroughly with an inert gas, such as N_2 or CO_2 , introduced through the 90 degree glass tubing bend (at the inlet end). The inert gas can be passed first through a wash bottle to estimate the rate of flow or the flow can be observed by placing the rubber tubing from the vacuum adapter in a container of water (for observing the rate in bubbles per second). A solution of 136 g. (1 mole) of phenyl acetic acid (PAA) in 120 cc. of glacial acetic acid (120 g., 2 moles) is placed in the separatory funnel and introduced into the tube furnace at a rate of twelve to fifteen drops per minute. Meanwhile, a very slow stream of the inert gas (one bubble per second) is passed through the tube to keep the gases in motion. After all of the solution has been added, the separatory funnel is rinsed with 10 cc. of glacial acetic acid, and this is passed through the catalyst tube to facilitate removal of the product. The distillate consists of a red or brown red oily layer and an aqueous layer. Both layers are treated with about 300 cc of water and $NaHCO_3$ is added until the effervescence stops and the mixture is alkaline to litmus or pH paper ($NaHCO_3$ turns pH paper green). The P2P layer is separated (the emulsion can be easily separated with a centrifuge if available) and the aqueous layer is extracted with an organic solvent (ie. 3X 30 ml of toluene). The solvent is dried and evaporated to get more the P2P. The P2P can be distilled under a vacuum or at atmospheric pressure (fraction collected at 210-230 degrees C). The yield is typically about 70 %.

References:

Vogels

Organic Synthesis Col. Vol. II pg. 389-391 (methyl benzyl ketone)

US patents:

2108156

2612524

2697729

2811559

3075016

3660491

4172097

4754074

5750795

Mastermind
(Hive Bee)
07-23-00
02:08
No 31901

Re: P2P tube furnace write up attempt

Maybe the description of the apparatus is too detailed. A generalized description might be better if you wanted to put it on Rhodiums page. Various ways to set it up are possible. The tube itself can be glass, ceramic or metal. One problem with metal is that it conducts electricity and might be a problem if a heating tape becomes frayed (starts to lose insulation). Also, you don't want to short out the thermocouple either. Glass has the advantage that you can see through it and both glass and ceramic have been used in combustion tubes. Ceramic also might be a good choice since its tougher than glass and doesn't conduct electricity.

WizardX
(Wizard Master)
07-23-00
02:53
No 31910



Re: P2P tube furnace write up attempt

Mastermind: Have the combustion tube vertical, as gravity will help move the GAA/PAA solution through the combustion tube with the N_2 gas flow. Al_2O_3 works as well as a catalyst.

Mastermind
(Hive Bee)
07-23-00
04:14
No 31936

Re: P2P tube furnace write up attempt

WizardX, The apparatus was modeled as closely as possible to the apparatus from the Organic Synthesis article which shows a picture of the tube furnace tilted slightly from the horizontal. I've seen other similar things where the reaction tube is horizontal or vertical. For example, US patent 2,811,559 shows a vertical tube furnace which is used for the same type of reaction (ie. cross decarboxylation of carboxylic acids). The catalyst is MgO and the products in examples I-IV are methyl undecyl ketone and stearaldehyde. The problem with having the tube furnace vertical is that maybe the reactants would come through too fast and wouldn't have time to react, depending on the porosity of the catalyst. It was felt that if pea sized pumice was used as in the Organic Synthesis article that the catalyst tube should be set at the same angle as shown in the article.

PVnRT_NC8
(Newbee)

Re: P2P tube furnace write up attempt

07-23-00
05:58
No 31964

wow very nice looking appratus, as far as heating goes this thing it is wrapped with nicrome wire heating element right??

A010

Mastermind
(Hive Bee)
07-23-00
06:12
No 31971

Re: P2P tube furnace write up attempt

For your entertainment and mine and since I like posting pictures, here's a picture of the thorium nitrate bottle used to make the thoria catalyst in the early 80's (farther back than the statute of limitations (7years)) according to the Organic Synthesis article. The thorium nitrate was used a number of times to make the thoria catalyst. It was found that when the thoria catalyst was fresh the yield of P2P wasn't too bad, but the catalyst tended to lose activity after a couple of runs. Masking tape was put on the bottle to conceal the source. Also, as many of you know, thorium is radioactive and not the best thing to play around with.

□

Mastermind
(Hive Bee)
07-23-00
06:27
No 31978

Re: P2P tube furnace write up attempt

PVnRT_NC8, I'm familiar with the $PV=nRT$ from the repetitive word problems from all the chemistry courses, especially PChem a long time ago, but what is the NC8 for?

To answer your question, PV, the combustion (catalyst) tube is wrapped with a heating tape which is coated with a woven fiber glass covering. The heating tape was supposed to be rated at 800 degrees C. Over time the fiber glass covering tended to fray (come apart) and expose the wire heating element. After it was used for some ketene experiments at 800 degrees C that was when the fiber glass covering started to come apart. It might be best if you copied and pasted all of my posts to notepad and store it on a floppy disc or your hard drive, etc., or even print it all out if you're interested in this. It might take time to study it all. You can download these threads directly, but then they're stored as HTML and you need a browser to read it offline.

PVnRT_NC8
(Newbee)
07-23-00
21:17
No 32193

Re: P2P tube furnace write up attempt

Mastermind,

The NC8 was actually supposed to be ZC8, I suppose it was a typo...It was supposed to stand for $N<\infty$...I have come to believe that in crystallizations the classical literature reports that, (though not specifically.) Yields were surprisingly higher than expected! See the classic on Rhod's page dealing with isomeric differences and yields concerning the catlike-ephedrine. The documents make me tend to believe these high yields were not expected [at this time] and this was a rather historic and revolutionary discovery; more specifically... the procedure where the one isomer is isolated put into etoh-i-proh tartaric acid mixture, left for "X" days and re-separated and components weighed, I have not repeated the experiments but I am hopeful that in fact there really is something more there between the lines than most people interpret upon the first few perusals.

Thorium is still available in the form of black looking needle like crystals if ya know where to dig them using a giger counter would make rocking them up real simple, I seem to recall they are a alloy of lead and radioactive thorium, the Beartooths/ Bitterroots?? Ranges pop into recent memory, we had a few around the house apparently they are at the university and in a private collection now.

A010

wayupnorth
(Stranger)
07-26-00
04:26
No 33194

Re: P2P tube furnace write up attempt

Fresh stuff at universities where they have petroleum engineering departments. So much college stuff with so little care when it disappears, if your not greedy and leave them some to work with till they get more. You should see the Journalism photo department storage rooms. Students love not being broke and when their just getting some for a friends development use, they or no one else really cares. The rooms at most universities are unlock more often than locked. And photo classes have so many new non full time students taking classes in the evening.

Mastermind
(Hive Bee)
07-28-00
00:46
No 34007

Re: P2P tube furnace write up attempt

The point of using the magnesia catalyst is as an alternative to the thoria catalyst in the Organic Synthesis article. Thorium is radioactive and isn't OTC anyway. It was found, surprising, that the magnesia catalyst gave high yields of P2P.

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