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College chemistry, 1983



The 2002 Model



After 10 years of blogging . . .

Derek Lowe, an Arkansan by birth, got his BA from Hendrix College and his PhD in organic chemistry from Duke before spending time in Germany on a Humboldt Fellowship on his post-doc. He's worked for several major pharmaceutical companies since 1989 on drug discovery projects against schizophrenia, Alzheimer's, diabetes, osteoporosis and other diseases. To contact Derek email him directly: derekb.lowe@gmail.com Twitter: [Dereklowe](https://twitter.com/Dereklowe)

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January 9, 2013

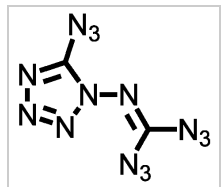
Things I Won't Work With: Azidoazide Azides, More Or Less

Posted by Derek

When we [last checked in](#) with the Klapötke lab at Munich, it was to highlight their accomplishments in the field of nitrotetrazole oxides. Never forget, the biggest accomplishment in such work is not blowing out the lab windows. We're talking high-nitrogen compounds here (a specialty of Klapötke's group), and the question is not whether such things are going to be explosive hazards. (That's been settled by their empirical formulas, which generally look like typographical errors). The question is whether you're going to be able to get a long enough look at the material before it realizes its dream of turning into an expanding cloud of hot nitrogen gas.

It's time for another dispatch from the land of spiderweb-cracked blast shields and "Oh well, I never liked that fume hood, anyway". Today we have a fine compound from this line of work, part of a series derived from N-amino azidotetrazole. The reasonable response to that statement is "Now hold it right there", because most chemists will take one look at that name and start making get-it-away-from-me gestures. I'm one of them. To me, that structure is a flashing red warning sign on a dead-end road, but then, I suffer from a lack of vision in these matters.

But remember, N-amino azidotetrazole (I can't even type that name without wincing) is the *starting material* for the work I'm talking about today. It's a base camp, familiar territory, merely a jumping-off point in the quest for still more energetic compounds. The [most alarming](#) of them has two carbons, *fourteen* nitrogens, and no hydrogens at all, a formula that even Klapötke himself, who clearly has refined sensibilities when it comes to hellishly unstable chemicals, calls "exciting". Trust me, you don't want to be around when someone who works with azidotetrazoles comes across something "exciting".



It's a beast, all right. The compound is wildly, ridiculously endothermic, with a heat of formation of 357 kcal/mole, all of which energy is ready to come right back out at the first provocation (see below). To add to the fun, the X-ray crystal structure shows some rather strange bond distances, which indicate that there's a lot of charge separation - the azides are somewhat positive, and the tetrazole ring somewhat negative, which is a further sign that the whole thing is trembling on the verge of

not existing at all.

And if you are minded to make some yourself, then *you* are on the verge of not existing at all, either. Both the initial communication and the [follow-up publication](#) go out of their way to emphasize that the compound just cannot be handled:

Due to their behavior during the process of synthesis, it was obvious that the sensitivities (of these compounds) will be not less than extreme. . .

The sensitivity of C2N14 is beyond our capabilities of measurement. The smallest possible loadings in shock and friction tests led to explosive decomposition. . .

Yep, below the detection limits of a lab that specializes in the nastiest, most energetic stuff they can think up. When you read through both papers, you find that the group was lucky to get whatever data they could - the X-ray crystal structure, for example, must have come as a huge relief, because it meant that they didn't have to ever see a crystal again. The compound exploded in solution, it exploded on any attempts to touch or move the solid, and (most interestingly) *it exploded when they were trying to get an infrared spectrum of it*. The papers mention several detonations inside the Raman spectrometer as soon as the laser source was turned on, which must have helped the time pass more quickly. This shows a really commendable level of persistence, when you think about it - I don't know about you, but one exploding spectrometer is generally enough to make recognize a motion to adjourn for the day. But these folks are a different breed. They ended up having to use a much weaker light source, and consequently got a rather ugly Raman spectrum even after a lot of scanning, but if you think you can get better data, then step right up.

No, only tiny amounts of this stuff have ever been made, or ever will be. If this is its last appearance in

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the chemical literature, I won't be surprised. There are no conceivable uses for it - well, other than blowing up Raman spectrometers, which is a small market - and the number of research groups who would even contemplate a resynthesis can probably be counted on one well-armored hand.

Comments (60) + TrackBacks (0) | Category: Things I Won't Work With

COMMENTS

1. [eugene](#) on January 9, 2013 11:01 AM writes...

"the number of research groups who would even contemplate a resynthesis can probably be counted on one well-armored hand."

Or on one arm of an old-time fluorine chemist's hand!

Seriously though, Klapoetke is a great speaker and he came to my department twice while I was doing a PhD. I heard later he was doing a sabbatical at NIST or somewhere close, and blew up a fume hood.

[Permalink to Comment](#)

2. [Rhenium](#) on January 9, 2013 11:08 AM writes...

I do quite like the prose in the first half of the post! How is the book coming along?

[Permalink to Comment](#)

3. [eugene](#) on January 9, 2013 11:08 AM writes...

Bah, I meant to say "on one old-time fluorine chemists's hand!"

The 'arm' is a bit redundant and actually bad English.

[Permalink to Comment](#)

4. [RB Woodweird](#) on January 9, 2013 11:20 AM writes...

So this is the Killer Joke of organic chemistry?

(Wenn ist das Nunstück git und Slotermeyer? Ja! Beiherhund das Oder die Flipperwaldt gersput!)

Although with two carbon atoms, it is edging off the 'organic' lawn. I bet the next graduate student in Klapoetke's lab gets the job of replacing one of the carbons with nitrogen.

[Permalink to Comment](#)

5. [Anonymous](#) on January 9, 2013 11:33 AM writes...

Great post - thanks Derek. following on from yesterday's comment, it seems that it is not just American professors that are 'totally f**king mental'

[Permalink to Comment](#)

6. [Curious Wavefunction](#) on January 9, 2013 11:39 AM writes...

In the spirit of Isaac Asimov who once wrote a fictitious paper about a compound that dissolved before it hit the solvent, these guys need to write a paper about a compound that explodes before it's even made.

[Permalink to Comment](#)

7. [Scull](#) on January 9, 2013 12:04 PM writes...

I'm so happy to see this feature back!

That's not to say that I don't enjoy all of the content that you post, but this is certainly my favorite series you've done.

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8. "Nothermaus on January 9, 2013 12:27 PM writes...

In the immortal words of a former PFE research director, "So, vee are actually making zeeks compounds on purpose?"

[Permalink to Comment](#)

9. strayxray on January 9, 2013 12:38 PM writes...

How does one actually crystallize and collect data on something that explodes *in solution*. I don't have access to the paper, but I've always wondered how they do it.

[Permalink to Comment](#)

10. Stephanie on January 9, 2013 12:39 PM writes...

I will admit to doing a little happy dance when I saw this.

And, I've never been so happy about the 4500 miles between me and this lab... although, if that stuff goes wrong, I'm sure I'll see the explosion from here.

[Permalink to Comment](#)

11. newnickname on January 9, 2013 12:42 PM writes...

@6: A Chemical Squonk, from Squonk:

"When cornered and escape seems impossible, or when surprised and frightened, [the Squonk] may even dissolve itself in tears. Squonk hunters are most successful on frosty moonlight nights [or liquid nitrogen], when tears are shed slowly and the animal dislikes moving about. ... He made a clever capture by mimicking the squonk and inducing it to hop into a sack, in which he was carrying it home, when suddenly the burden lightened and the weeping ceased. Wentling unslung the sack and looked in. There was nothing but tears and bubbles."

<http://en.wikipedia.org/wiki/Squonk>

[Permalink to Comment](#)

12. Yazeran on January 9, 2013 1:26 PM writes...

#4 adding more nitrogen...

Well I do not think you could exchange any of the carbons, however I think it is theoretically possible (from my high school fiddling with ball and stick chemical models that is) to break the second double bond and attaching a THIRD azide group to the last carbon.... (you would likely want to leave a hydrogen on the next door nitrogen though..

Personally, I would like to know if anyone tries that, so that I can safely order a trip to Tahiti or similar to be at enough distance when whomever is foolish enough to try..... :-)

Yazeran

[Permalink to Comment](#)

13. simpl re #9 on January 9, 2013 1:27 PM writes...

How do they crystallise it? - Swiss-German for hood is Kapelle (chapel)

[Permalink to Comment](#)

14. Dave on January 9, 2013 1:48 PM writes...

One would assume that they keep the stuff cold (to reduce thermal energy levels), and keep it in the dark (to decrease the chance of photon induced decomposition)?

Dave

[Permalink to Comment](#)

15. opsomath on January 9, 2013 1:56 PM writes...

@ RB Woodweird - Holy cow, I just realized working for Klapotke must be like owning the Bottle Imp. It's fine early on, but woe is you if you get the Imp when it's been sold for only two pennies, or in this case, two carbons.

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16. gippgig on January 9, 2013 2:27 PM writes...

Note that the left half and right half are isomeric. It would be interesting to make dimers of each half. It might actually be possible to replace the azidotetrazole with pentazole; pentazole derivatives exist. I'd like to see dipentazole - 10 nitrogens and nothing else - wait a minute, how about tripentazolylamine - N16?

[Permalink to Comment](#)

17. Chemist For Life on January 9, 2013 2:40 PM writes...

I believe Edmund Hillary once said 'Nobody climbs mountains for scientific reasons. Science is used to raise money for the expeditions, but you really climb for the hell of it.'

Klapotke and others are doing it for the same reasons and the adrenaline rush of course...

In true pioneering fashion, they are pushing the boundaries of limitation. It's fantastic to see that funding is still given to pursue such crazy research! I hope it continues -without loss of limbs of course.

[Permalink to Comment](#)

18. Anonymous on January 9, 2013 3:27 PM writes...

$(-N=N-)_n$

ducks and covers

[Permalink to Comment](#)

19. zDNA on January 9, 2013 3:43 PM writes...

Soon to be available in kg quantities from Chinese suppliers.

[Permalink to Comment](#)

20. noko marie on January 9, 2013 3:45 PM writes...

No protons, so just keep this stuff away from the NMR lab; and no, you cannot have any ^{13}C or ^{15}N data!

[Permalink to Comment](#)

21. Justin Peucon on January 9, 2013 4:26 PM writes...

@9, @13: recrystallization from Et_2O and X-ray data collected at 150 K with 2 kW irradiation. From the supplementary material, I estimate the single crystal to be a 100 ng sample. Converted to standard free Gibbs energy, that means this sample was roughly 750 micro.J worth. This is an impressive figure, although I suppose the diffractometer was never in danger. I hope...

[Permalink to Comment](#)

22. Justin Peucon again on January 9, 2013 4:37 PM writes...

By the way, I now realize what substance Rambo hide in the tips of their arrows.

[Permalink to Comment](#)

23. Anonymous on January 9, 2013 4:39 PM writes...

Doesn't seem any trickier than the red phosphorus/potassium chlorate mixtures, fulminating silver or nitrogen triiodide I routinely made as a kid in my backyard lab.

[Permalink to Comment](#)

24. J. Peterson on January 9, 2013 4:54 PM writes...

Does this lab have a YouTube channel? Heck, it sounds like they could have their own show on the Discovery Channel...

[Permalink to Comment](#)

25. Yancey Ward on January 9, 2013 5:27 PM writes...

That thing has too many carbons.

[Permalink to Comment](#)

26. partial agonist on January 9, 2013 5:29 PM writes...

That lab must have more explosions than the guys at Mythbusters.

La Clair should have picked this one to dry-lab, since nobody is every really going to check you on it.

You made it? Fine.

[Permalink to Comment](#)

27. Richard L. Wells on January 9, 2013 8:33 PM writes...

Very interesting.

Lou Coury recommended this article to me.

Hope all, in general, is well with you Derek.

RLW

[Permalink to Comment](#)

28. in vivo veritas on January 10, 2013 12:44 AM writes...

Any volunteers to be the "checkers" for the Org Syn prep of this stuff?

[Permalink to Comment](#)

29. gippgig on January 10, 2013 12:48 AM writes...

A couple other "has anyone looked at this" compounds:

tetraazidoethylene - might behave similarly to tetracyanoethylene and form superconductors with TTF-like compounds

tetraazidoallene - just plain weird

[Permalink to Comment](#)

30. Stefan on January 10, 2013 9:32 AM writes...

Either München(German) or Munich(English) but not Münich ;)

[Permalink to Comment](#)

31. Derek Lowe on January 10, 2013 9:38 AM writes...

Right you are, Stefan - not sure what my forebrain was doing on that one. Fixed.

[Permalink to Comment](#)

32. Curryworks on January 10, 2013 9:48 AM writes...

So I should not sue this as a click linker?

[Permalink to Comment](#)

33. David Marjanović on January 10, 2013 9:53 AM writes...

How does one actually crystallize and collect data on something that explodes *in solution*.

Apparently it doesn't explode **spontaneously** in solution.

[Permalink to Comment](#)

34. Yazeran on January 10, 2013 11:23 AM writes...

With a suitable definition of spontaneous I suppose..

Something that blows up when exposed to bit of infra red light is pretty close in my book.. :-)

Yazeran

[Permalink to Comment](#)

35. Mike Andrews on January 10, 2013 12:12 PM writes...

Very glad to see a new entry. I will read it to my wife, whose degree is in biochem, as tonight's bedtime story. If past experiences with "Things I Won't Work With" and with John Clark's Ignition are valid indicators, she will come up out of a doze, screaming "THOSE DAMNED FOOLS DID **WHAT**?"

[Permalink to Comment](#)

36. George on January 11, 2013 11:45 AM writes...

To 23. Anonymous: Wow, another basement/backyard chemist like me. Let me tell you about the time we mixed up some phosphorus and perchlorate in water and poured it on the concrete in my friend's yard to dry. While we were inside waiting his mother came home with his 2 year old brother...

Actually nothing happened. When we figured out what was happening we rushed outside. I touched the stuff with the end of a l-o-n-g bamboo pole, and the rest is history. We didn't try that again.

[Permalink to Comment](#)

37. Michael Press on January 12, 2013 3:21 AM writes...

How many nitrogens can you fit into a Volkswagon?

[Permalink to Comment](#)

38. Dan on January 12, 2013 9:47 PM writes...

Wait ... the azide groups are taking a POSITIVE charge? Given that chlorine azide is called that instead of (in imitation of its rowdier brother upstairs) triazadienyl chloride, I would guess that getting azide to take a positive charge is pretty blasted hard (even chlorine can't do it, though fluorine can).

And since azides are not known for their benevolence under the best of conditions, I can only imagine how much they want to wreak unholy vengeance on anyone that dares force them to take a positive charge.

[Permalink to Comment](#)

39. Jonadab on January 15, 2013 9:08 AM writes...

It's the two carbons that bother me. They make the whole compound impure...

- > I bet the next graduate student in Klapoetke's
- > lab gets the job of replacing one of the
- > carbons with nitrogen.

Why only one of them? Replace both while you're at it, then hang some more rings of nitrogens off the side, and call the whole thing an allotrope. Yay.

Do it in a lab in Germany, because I live in Ohio, and Germany is far away.

[Permalink to Comment](#)

40. Eric Jablow on January 15, 2013 10:18 PM writes...

Strange--in the second Klapoetke paper, the popups for compounds 2 and 3 show a C=O double bond, while the drawings further down do not. The carbamoyl radical is NH₂CO, so what's going on with the paper?

[Permalink to Comment](#)

41. Eric Pantano on January 23, 2013 2:30 PM writes...

I truly enjoy reading these posts, and often find myself laughing aloud at my desk, while dozens of bewildered students shake their heads at the "nerd in the office". I mean, come ON! These are funny posts!

And did I read correctly that there's a BOOK in the offing??? Take my money!

[Permalink to Comment](#)

42. Anonymous on January 27, 2013 7:47 PM writes...

Derek, it is always such a treat to see a new entry in your TIWWW category. You have a remarkable ability to make chemistry not only exciting for the decidedly clueless, but your phrasing is just hilarious! Keep 'em coming!

@ Eric - I hope the comments about a book coming along are not only from eager commentators like us. I would buy "How not to do it and what not to work with" the DAY it was on sale.

[Permalink to Comment](#)

43. This on February 3, 2013 1:29 PM writes...

As late as in the end of the XXth century, chemists were recognized easily: they had one hand or one eye missing ! No change...

[Permalink to Comment](#)

44. stegmüller on February 4, 2013 9:13 PM writes...

In completely unsurprising news Klapötke mentioned in an interview he did with the German newspaper Zeit that he is mostly deaf in one ear and that the other one isn't doing that well either.

[Permalink to Comment](#)

45. Anonymous on February 11, 2013 10:35 PM writes...

I'm seeing a pattern here... explosive polyazides, pyrophoric alkyl organometallics (though not all of them or you wouldn't be a pharmaceutical chemist), and chalcogen analogs that make a skunk wrapped in a burning tire smell like Chanel No 5... Yeah I wouldn't work with them either. Good thing I'm a software developer, and my dad is the inorganic chemist.

[Permalink to Comment](#)

46. Anonymous on March 20, 2013 1:36 AM writes...

Yay! Another one! I'd almost given up hope. Thank you Sir. Be safe!

[Permalink to Comment](#)

47. Peter on April 2, 2013 12:29 PM writes...

These articles are better than the BOFH articles in The Register!

I'm an electrical engineer, my lab accidents are usually accompanied by a blue flash and acrid smoke. Since I work with microprocessors, the scale is manageable.

My hat is off to those chemists...glad my lab isn't anywhere near them.

//I'll buy your book the day it comes out, too!

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48. Randy Owens on April 6, 2013 7:29 PM writes...

@ Peter #47: That's not acrid smoke; it's *magic smoke*. And you're not supposed to let it out; it's what makes the widgets work.

[Permalink to Comment](#)

49. Christian on April 11, 2013 12:55 PM writes...

For those who read German, here is an interview with Klapötke. He does this stuff to find new materials for ecological bombs (!).

[Permalink to Comment](#)

50. David Edwards on May 14, 2013 10:07 PM writes...

I've just downloaded the paper. There's a compound listed therein that contains no less than TWENTY nitrogen atoms, with six carbon atoms apparently included just for the fun of it. It rejoices in the wonderful name of tetraazidoazotriazine (TAAT for short).

Full paper available here: <http://www.chtf.stuba.sk/~szolcsanyi/education/files/Chemia%20heterocyklickych%20zlucenin/Prednaska%203/Doplukove%20studijne%20materialy/Tetra-%20and%20Pentazoles/C2N14%20-%20An%20Energetic%20and%20Highly%20Sensitive%20Binary%20Azidotetrazole.pdf>

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51. rrr on June 28, 2013 4:58 AM writes...

I wonder how it compares to N13, which is extreme in itself as it is able to be detonated by alpha particles.

[Permalink to Comment](#)

52. anders on August 21, 2013 12:58 AM writes...

I do not see how this is any more extreme than tetraazidomethane.

Obviously the instability of the type of compounds are not determined by merely the number of nitrogen atoms in the molecule, but rather by the ratio of nitrogen to other atoms.

I remember reading about another bicyclic compound, formula CN₇H, that was formed by treating 1,5-diamino tetrazole with nitrous acid. (allowed to react for 30 minutes at zero degrees, 55% yield, in case anyone is interested)

[Permalink to Comment](#)

53. Anonymous on October 22, 2013 8:38 PM writes...

I discovered two important things today. One, pentazole exists (an aromatic ring with a single hydrogen attached). Two, pentazenium also exists (a nitrogen-only linear cation). Sure, they're both sensitive explosives, but that's to be expected, with five nitrogens and no carbons to hold them together. I wonder if there might be any compounds of these (and perhaps the more mundane amines and azides), possibly with some carbon glue to keep things from spontaneously exploding at LN₂ temperatures.

[Permalink to Comment](#)

54. Kenny Strawn on December 1, 2013 9:06 PM writes...

Some Livermore chemists (Riad Manaa in particular; see [here](#)) have actually created successful computer simulations of what would happen if six N₁₀ (aka dipentazole) molecules were tightly packed... Sure enough, the results of the calculations show that a perazabuckminsterfullerene (N₆₀) molecule might be possible.

Man, that's some crazy stuff...

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55. John Cowan on January 15, 2014 11:28 AM writes...

Thiotimoline, the compound that dissolves before it hits the water, is basically an only slightly exaggerated version of catecholamine, the subject of Asimov's thesis. Asimov knew that the thiotimoline article would come out in *Astounding* just before his orals, so he asked Campbell to publish it under a pseudonym. But Campbell forgot, and you can guess what the last question on those same orals was

[Permalink to Comment](#)

56. Kenny Strawn on February 25, 2014 5:46 PM writes...

@anders CN₇H -- could that be azidotetrazolic acid?! Seems likely to me...

[Permalink to Comment](#)

57. Jerry on April 3, 2014 8:11 PM writes...

I think I once suggested a compound that would be fairly insane to synthesize, azopicrin, (N₃)₃CNO₂, triazonitromethane. Notice it is a stoichiometrically balanced explosive, decomposing to five N₂ and one CO₂ molecules. I am sure that it is as sensitive as it is powerful. As far as I know it has never been made though this would be quite easy to accomplish. Just reflux chloropicrin with sodium azide in some suitable unreactive solvent, say dimethylformamide? Judging by every other compound of this sort I have heard described, it should be an oily liquid which needs very little excuse to violently detonate, say just looking at it too hard. It would be a good idea to use a solvent

that azopicrin might reasonably be expected to be highly miscible with to avoid that dread sight of observing an oily layer settling out on the bottom of the reaction flask.... In that case I hope you have your running shoes on! I originally thought this compound was emphatically useless and would hopefully never be synthesized but now I am not so sure. Notice that if you photochemically disassociate the 3 azide groups, you will get the CNO₂ radical, basically a carbon atom attached to a nitro group. Now, to do this without committing hara-kiri, you should have azopicrin dissolved in a suitable and nonreactive solvent to a safe, non-explosive concentration. Maybe using it as a dilute vapor would work too. Now, I have to wonder what makes good solvents for organo-azide compounds? Does anybody really know? I wonder if something fluorocarbon based might be the thing? Anyway, once you have azopicrin as a vapor or solution in a safe concentration, you could make some very interesting things, things very difficult and expensive to otherwise make assuming they have been successfully synthesized at all. One such is dinitroacetylene which apparently has not been synthesized yet though one hopeful monograph was titled, "On the Path to Dicyanoacetylene". Obviously this compound would be expected to result if you combined two CNO₂ radicals together. Photolytic dissociation of azopicrin should be a plentiful source of NCO₂ radicals. Maybe you could generate dicyanoacetylene by freezing azopicrin in some kind of cold matrix and zapping it with ultraviolet light of the appropriate frequency? Anyway, there is every reason to suspect this compound would be highly unstable but perhaps it could be isolated under cryogenic conditions? Higher polymers of CNO₂ radical would also be interesting. I rather doubt you would get tetranitrotetrahydrofuran or its isomer tetranitrocyclobutadiene but who knows? Maybe under cryogenic conditions something might show up or maybe if you warm up the dicyanoacetalene just a bit..... Much more practically important would be explosive polymers. The well known hexanitrobenzene would be a very likely result, but this compound is sensitive to light and perhaps under illumination its formation could be suppressed in favor of its much more interesting isomer, hexanitroprismane and (let us hope!) octanitrocubane. These substances are both reasonably stable and exceptionally powerful. Octanitrocubane if it can be isolated in its high density crystalline form would be expected to be the most powerful known conventional explosive. Unfortunately, making it from already scarce and expensive cubane is so difficult that only minute amounts have been synthesized, too small to test its explosive properties. The most hopeful projection suggests that it would be as costly as gold to make. I suspect that an easy and inexpensive method of making hexanitroprismane and octanitrocubane would be of great practical and military interest. I am sure it's kind of a long shot but maybe somebody might be fooling around with azopicrin in the near future after all? Just be damn careful!

Cheers,

Jerry ^,^,^

[Permalink to Comment](#)

58. [Kenny Strawn](#) on June 5, 2014 9:07 PM writes...

Out of curiosity: I wonder if anyone has had any success reacting tetrachlorohydrazine (which has in turn [supposedly] been synthesized by reacting hydrazine with hypochlorous acid) with sodium azide to produce tetraazidohydrazine...

[Permalink to Comment](#)

59. [athE1st](#) on October 10, 2014 4:18 PM writes...

I bet the next graduate student in Klapoetke's lab gets the job of replacing one of the carbons with nitrogen.

Why replace one, there are two. That would add two and free up two nitrogens with double bonds to each other. Seems like N₂ is possible, if only briefly.

[Permalink to Comment](#)

60. [joeylawn](#) on November 17, 2014 1:53 PM writes...

How about N(N₃)₃, or Octaazacubane?

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