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Using an On-Site Liquid Nitrogen Generator for NMR Cryogen Supply and Other Departmental Needs

Catherine F. M. Clewett* and Nick E. Flynn

Department of Mathematics, Chemistry and Physics, West Texas A&M University, Canyon, Texas 79016, United States

ABSTRACT: The purpose of this article is to elevate awareness of an on-site liquid nitrogen generator and highlight the advantages of such a purchase for an undergraduate chemistry department. Immediate access to liquid nitrogen and required instrument specifications are discussed. Economic and environmental factors include annual cost savings, convenience of on-site availability of liquid and gas forms of nitrogen, and potential for revenue generation. A conservative estimate of $850 of annual savings over a typical service contract has already been realized. Use of the on-site generator has proven very beneficial and may be an attractive option for other departments to consider.

KEYWORDS: *General Public, Laboratory Instruction, Organic Chemistry, Chemical Technicians, NMR Spectroscopy*

INTRODUCTION

The use of NMR spectroscopy is an important component of an undergraduate chemistry education.[1−5] It benefits pedagogical development in critical thinking and problem solving skills while simultaneously helping faculty teach specific concepts such as molecular structure, degree of substitution in carbons, and electronegativity.[6−12] One major consideration for using NMR instrumentation is the cost of using cryogenic fluids to maintain the superconductivity of the magnet.[13] Liquid nitrogen is used to help conserve the liquid helium, which is used to cool the superconductive materials used in the magnet.[14] Costs and logistics associated with using these fluids can be problematic primarily for smaller institutions either in remote locations or with limited research budgets. In considering whether or not to bring a high-field NMR magnet online, West Texas A&M University (WTAMU) considered the cost difference in on-site generation compared to a traditional cryogen delivery contract. A liquid nitrogen generator was acquired for on-site generation of liquid nitrogen at WTAMU. There are a number of considerations in using an on-site generator including generator specifications, environmental, and economic considerations.

BENEFITS

There are several direct benefits to using an on-site generator as opposed to purchasing liquid nitrogen from an external supplier. One is immediate access to liquid nitrogen without having to conserve dewar levels. This allows fills of the NMR spectrometer irrespective of delivery schedules. There is the potential for generating revenue to cover the small maintenance costs and provide additional funds for the department (see section on economic considerations). Liquid nitrogen is readily available for in-class demonstrations, outreach demonstrations, and laboratory experiments.[15] With access to pure liquid nitrogen on campus, production of nitrogen gas for other applications is also possible. These applications include the generation of an inert atmosphere to store chemicals and samples, the ability to propel liquid nitrogen for cryogenic fills of the NMR spectrometer, and a carrier gas in gas chromatography applications.

SPECIFICATIONS/OPERATING CONDITIONS

Important considerations when purchasing a liquid nitrogen generator are the instrument specifications as well as annual need for liquid nitrogen to fill the NMR magnet. These details include purity of liquid nitrogen and purity of nitrogen gas, production capacity of the generator at altitude, and storage capacity. Typical 300−400 MHz magnets use 1600−1800 L/year of liquid nitrogen, and manufacturers specify that it must be 99.99% pure whether in liquid or gaseous form. It is common practice in the NMR community to refill the liquid nitrogen on a weekly basis for convenience, although the manufacturer's hold times range from 11 to 16 days for high field magnets (7.1−9.4 T or 300−400 MHz).[16−18] Therefore, absolute minimum production capacity must be 40 L/week (6 L/day) with 40 L of storage, allowing for cryogen evaporation during storage.

The Cryogenic Society of America provides a very useful buyer's guide for everything cryogenic from air separation/liquefaction equipment to transport cylinders.[19] This buyer's guide makes it easy to compare different suppliers of generators. There are a few companies, such as MMR Technologies (Mountain View, CA) and Cryomech Inc. (Syracuse, NY), which provide small capacity liquid nitrogen generators with liquid nitrogen at 98% purity. These units do not produce laboratory quality liquid nitrogen, especially the

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purity required for NMR magnets, but are an economical option for laboratory demonstrations or tissue storage. Bruker (Billerica, MA) has introduced new magnets that eliminate cryogen fills and would be an option if a campus only wanted NMR capabilities. These units are expensive, adding an additional $77,000 to the purchase price of the spectrometer system as well as requiring yearly maintenance contracts. They do not make nitrogen available for other purposes.

There is only one source in the USA for a small capacity liquid nitrogen generator that has sufficient purity for research laboratory work: the Kelvin International Corporation NL84a nitrogen generator (Newport News, VA). This company also has experience with systems for high altitude (greater than 1 km) locations, a requirement for the WTAMU site. The NL84a system includes an attached 40 L storage tank, 10−15 L/day (70−100 L/week) production capacity, and fully automatic liquid nitrogen level sensing with programmable control. The unit includes an air compressor and an optional closed loop water cooling system. This water saving option is important in a water restricted area such as WTAMU's location in the desert southwest. Installation is simple requiring two electrical connections of bayonet fittings, two standard Teflon plumbing connections, and wiring the instrument to 220 VAC power. The particular unit provided meets and exceeds the 6 L/day (40 L/week) required for the NMR spectrometer, producing an average of 15 L/day (105 L/week). Due to noise levels of the air compressor (∼85 dbA), it is best to site the instrument in a loading dock or service porch.

The liquid nitrogen generator is a low maintenance device that is easy to learn and use. Other than standard safety training in regard to liquid cryogens and waiting the 3−4 days for the generator to produce 40 L of nitrogen, there really is nothing the operator needs to do. Filling a container is a push-button operation. The generator requires an air compressor and water cooling for the cold head. Maintenance is minimal. A picture of the complete system is provided in the abstract graphic showing the ease of use by an undergraduate student operator. The oil-free air compressor tank needs to be checked weekly for moisture accumulation. The coolant in the closed circuit water system needs to be cleaned and refilled with a 50/50 mixture of water and ethylene glycol yearly. The only scheduled maintenance for the generator occurs at 10,000−12,000 operating hours, which can be 2−3 years depending on use. Properly trained personnel monitor instrument levels, operate the generator, and perform routine maintenance. These can be additional tasks for a student worker who is also employed to fill the NMR magnet. The duties generally take less than an hour/week to perform, adding little to the expenses of the department.

Filling the NMR magnet can be accomplished in two different ways. If the installation of the liquid nitrogen generator is close enough to the magnet (but far enough away not to disturb the magnetic fields), the magnet can be filled directly from the 40 L holding tank via an insulated Teflon hose. The holding tank is pressurized at 10 psi, which is plenty of pressure to move the liquid and not so much pressure as to cause difficulties with filling. If the generator cannot be installed within view of the NMR magnet, a pressurizing transport dewar with a liquid withdrawal device can be used. This accessory is available from Kelvin International, or a Taylor Wharton (Theodore, AL) 50LD dewar can be purchased from most scientific supply houses.

■ ECONOMIC AND ENVIRONMENTAL FACTORS

Economic and environmental factors also suggest that on-site generation of liquid nitrogen is more advantageous than offsite delivery. Annual costs for liquid nitrogen delivery are usually negotiated with suppliers. For small schools with an NMR spectrometer, negotiated costs for liquid nitrogen are on the order of $1.79−$3/L,[20] including delivery, fuel, and Hazmat charges, see Table 1. Most specialty gas providers also charge a daily tank rental fee, adding another $270/year to the costs. In rough figures, which are very dependent on negotiated rates, the annual cost to provide liquid nitrogen for a small NMR magnet is $3228−$5400 per year (assuming 1800 L/year use). These costs do not include supplying cryogens for demonstrations or other laboratory uses. For smaller schools without excess liquid nitrogen available, delivery schedules make it inconvenient to obtain cryogens on short notice for demonstrations or new experiments. Although fuel charges are part of the contract, the environmental effects of gasoline or diesel consumption by delivery trucks is generally not considered in an economic analysis.

Table 1. Comparison of Costs of a Liquid N_2 Generator vs "Traditional" Cryogen Delivery

Parameter or Consideration	On-Site Generator	Traditional Delivery
Initial cost	$42,000	$3228−$5400[a]
Set-up time (plumbing connections for cooling H_2O), hours	2	None
Operational learning curve, time/difficulty, hours	2, easy	2[b]
Cost cylinder rental/demurrage/year	$0	$270
Cost of staff-hours to maintain N_2 levels/year, estimated	$520[c]	$780[c]
Cost of air pollution, delivery truck exhaust	Unknown, but ∼0	Unknown, but >0
Revenue income from intramural sale of excess $N_{2(l)}$ $/year	$700	$0
Total annual cost/year, estimated[d,e] [$\$_{costs} - \$_{revenue}$]	$4020	$4872−$6457
Total annual savings from on-site generation [$cost/year_{(total traditional)} - cost/year_{(total generator)}$]	$852−$2437	$0

[a]Range given from survey[20] at $1.79−$3/L. Expenses include delivery, Hazmat, and fuel surcharges. [b]Estimated time to arrange contract. [c]Staff hours calculated at a student wage of $10/h. [d]Calculated by amortizing the initial cost of the generator over 10 years with an interest rate of 0%. [e]Assuming 1800 L/year.

The only costs involved for on-site generation of the same amount of liquid nitrogen are for the power to run the generator and the initial purchase price. Most universities would consider power costs overhead charges, and a department would never see them. For the NL84a generator, running at 100% service factor and producing 15 L/day, the liquid nitrogen costs less than $1/L.[21] Initial outlay for the NL84a was $42,000, paid for with state higher education assistance funds. The ability to readily generate nitrogen gas on-site will lead to the need for fewer nitrogen canisters and thus lower or even eliminate tank rental and demurrage charges per month. Fuel consumption by delivery trucks is eliminated, thus presenting an added environmental advantage.

In addition to saving the department's money on liquid and gaseous nitrogen, there are some potential sources of revenue generation for maintenance of the equipment. These include

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selling liquid nitrogen to student medical service facilities on campus for wart removal, selling to local doctors' offices, and charging fees for use outside the department.[22] At WTAMU, we have contracted to sell liquid nitrogen to campus medical services for $50/(25 L) on a monthly basis and the engineering department at a rate of $20/month. We estimate that we will be providing these services for 10 months/year. Liquid nitrogen produced for a research laboratory is of sufficient purity to be sold to area medical clinics. Researchers, both inside and outside the department, could be charged liquid nitrogen fees that are well below the cost of delivered nitrogen. Academic research applications of liquid nitrogen include the storage of biological samples, low temperature reactions, materials science, and physical chemistry experiments.[23,24]

WTAMU has had the generator for just over a year. Table 1 compares the costs and revenue of the on-site generator and compares them to traditional cryogen delivery. The range of liquid nitrogen costs are derived from a survey of small school users who are members of the Association of Managers of Magnetic Resonance Laboratories.[20] Yearly costs are calculated by amortizing the purchase price over ten years at 0% interest. The staff hours were estimated at 1 h/week for a student earning $10/h. Revenue was calculated assuming that the department was selling nitrogen to student medical services on a monthly basis and actual charges to the School of Engineering for research and demonstrations. Overall, WTAMU is saving $850 per year, which gives a break-even point of just over 8.5 years, based on purely economic considerations. It is difficult to quantify other cost-independent advantages such as the ability to do in-class demonstrations and outreach activities like liquid nitrogen ice cream on open house days.

■ CONCLUDING REMARKS

The purpose of this paper is to elevate awareness of on-site liquid nitrogen generators and highlight the advantages provided by this equipment including cheap and immediate access to liquid and gaseous nitrogen. An important item of note is the purity of nitrogen that is generated. Not all liquid nitrogen generators produce liquid nitrogen of the purity needed for medical, research, or NMR applications. Finally, economic and environmental factors make the use of an on-site liquid nitrogen generator advantageous.

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: cclewett@wtamu.edu.

Notes

The authors declare no competing financial interest.

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