Helium Recovery in NMR

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Project started in 2014
Funded in 2017
Installed & commissioned In October 2017
Completed (rectification & expansion) to full functionality in October 2019

Sustainable Campus Fund of The University of Edinburgh & School of Chemistry

From struggle to hassle free helium recovery

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Helium recovery - Purpose and Benefits

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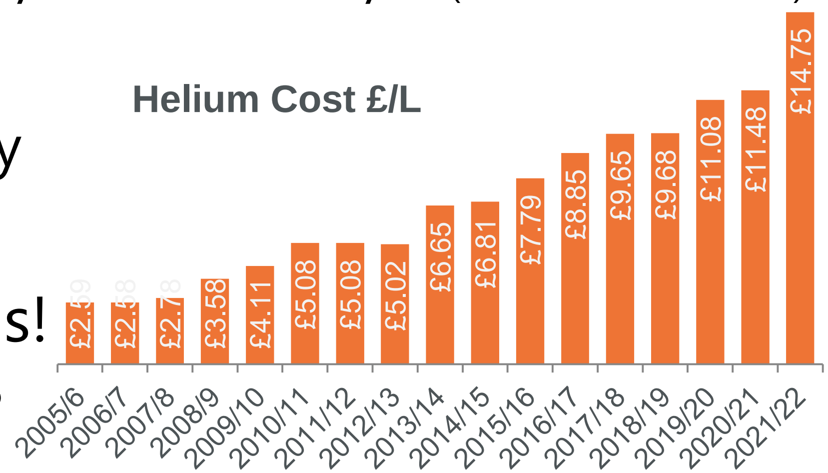
- Sustainability - Helium is non renewable resource!
- BOC liquid Helium price has rocketed to currently £18.69 (14.75+3.94) + VAT = £22.42 / Litre for 250 litres delivery.
- Contract with BOC:
  - guaranteed future deliveries 100%+ of previous year deliveries
  - payback scheme - currently £3.64 / m3 = £2.75 / L (20-25% of L He cost)
  - not affected by helium shortages
  - requested 80% of helium recovery
  - basically not buying new helium
- Could qualify for sustainably funds!
- Other companies e.g. Air Liquide?



Helium recovery – possible scenarios

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1. **Liquefier on site** – (usually Physics) – huge benefit :

- In proximity – straightforward installation either full recovery (pipes & manifolds, buffer gas bag?) or everyday boil-off only (easy - e.g. 8 mm tubing)
- Far away (depending on distance) – everyday boil-off (easy?) or full recovery system including blowers / vacuum pumps but NO need of (expensive) compressor and MCP

2. **No Liquefier on site** – buy-back contract with BOC, (AL?):

- Full recovery with complete installation to collect the helium; MCP renting from BOC - loading & uploading from truck (by forklift) – customer responsibility!

3. **Small portable liquefier** – significant purchase & running cost and still requires complete recovery installation.

Helium recovery – commercial solutions

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- Quite few companies offering either full recovery including helium liquefaction or just collecting the gas:
- https://www.cryomech.com/
- https://www.quantum-technology.com/index.html
- But only few of them are specifically design / optimised for NMR systems:
- https://qd-europe.com/at/en/products/cryogenics/helium-recovery-and-liquefaction/
- https://www.motivair.co.uk/products/special-services-products/helium-recovery

Helium recovery – Quantum Design (& Bruker?)

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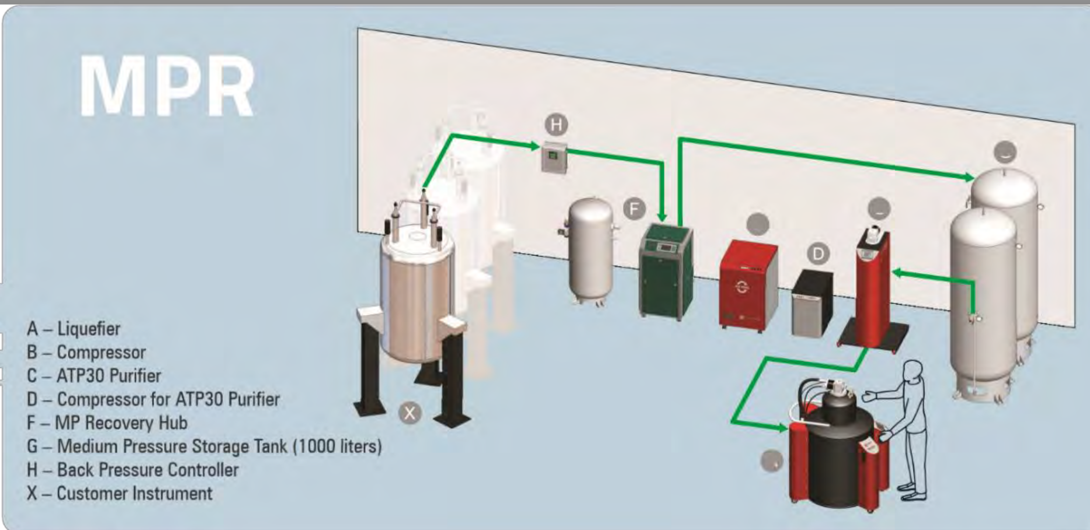
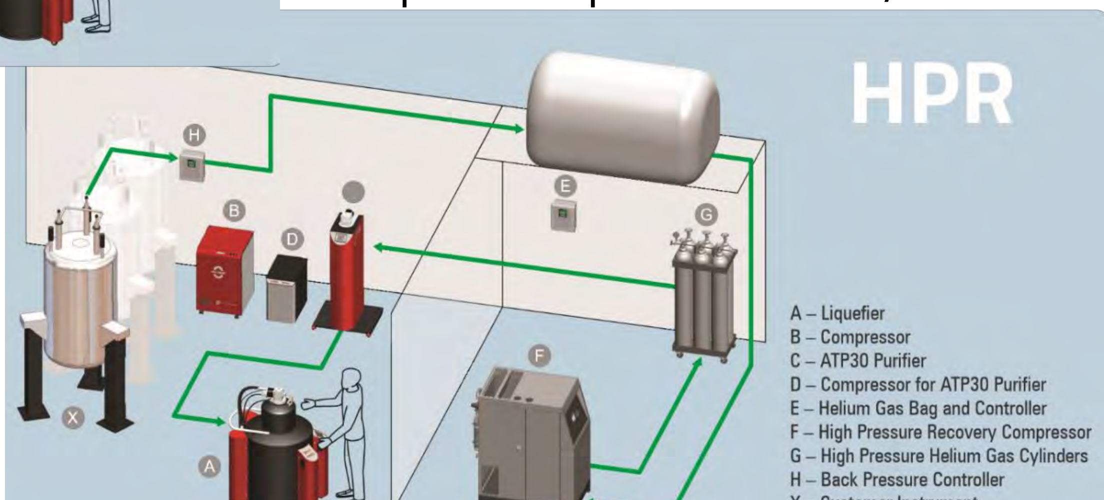


Medium Pressure Recovery
Up to 3 NMR magnets
Recommended max. 5 storage tanks @ 5 bars = 25 m3. (~100 L He refill)
High Pressure Recovery (HPR)
For large systems / laboratories
Customisable gas bag
Compressor speed ~15 m3 / hour

NexGen160, 250 & ATL160 liquefiers using (GM) cryo-cooler / cold head:
Liquefaction Rate 20+ L/day @ 50Hz = 0.83 L/hour = 0.63 m3/hour of gas
ATL160XL: 28+ litres/day @ 50Hz
Dewar Capacity 160 resp. 250 litres
250L=189 m3 requiring 21 cylinders
Water cooled indoor compressor
Requires 99.999% He purity
ATP cold head based purifier



Recovery layout calculator

Quantum Design EUROPE
Quantum Design GmbH
Im Tiefen See 58, D-64293 Darmstadt
www.qd-europe.com
Dr. Marc Kunzmann
David Appel
Dr. Tobias Adler

Helium recovery – everyday boil-off only

- Bruker Heliostat - compact, easy-to-site system that can be retrofitted to collect helium gas from installed NMR magnets. Helium gas is stored in high-pressure cylinders. Steady-state recovery rates of ~85% resulting to about 70% from helium refills.

(picture not in scale)

- Bauer G60-V - Helium recovery system for small volumes with integrated collection balloon. Effective delivery rate 3.6 m3 / hour, maximum operating pressure 225 bar. Duty cycle 15-20 min.

https://www.bauer-kompressoren.de/en/products/industry/air-cooled-compressors/90-350-bar-helium-g-series/60-lmin-g60-v/

High Pressure He Recovery - Motivair @ UoE - components

HR - early post installation challenges and pitfalls

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- Oscillating 40 - 60 mbar back pressure
- Efficiency dropped to 60-70% & insufficient gas bag capacity

Solution – removing / bypassing all narrow points e.g. check valves, gas and flow meters (including digital). Keep the line between magnets and gas bag as simple as possible and preferably KF40 (particularly 2K magnets).

Helium Recovery – gas bag challenges:

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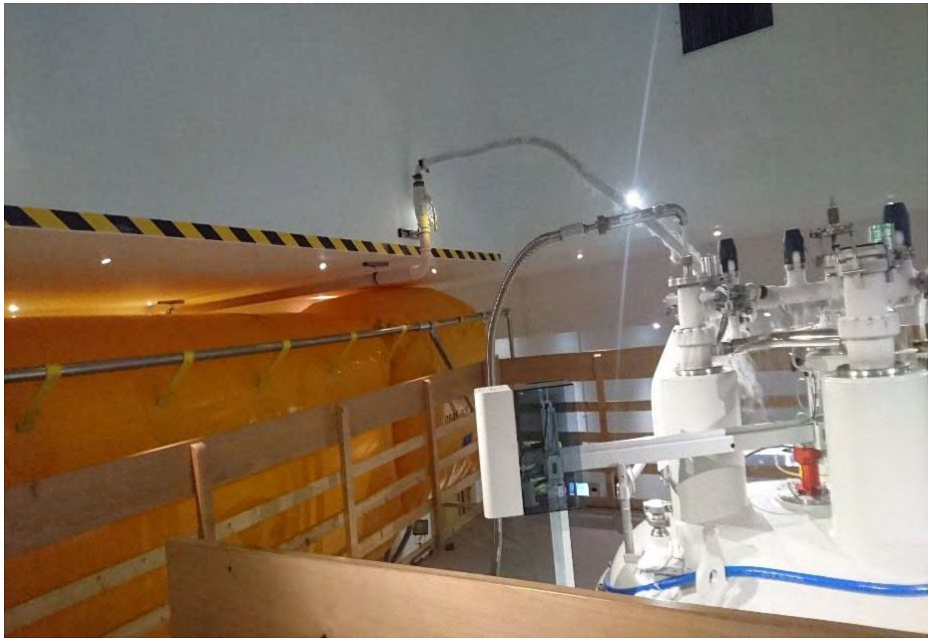
- Gas bag fixing versus available volume:
- Before – fixing straps in upper part of gas bags
- pressure in the system [mbar] / volume available [%]:
- 0.00 [mbar] / ~9 [%]
  -0.10 [mbar] / ~18 [%]
  -0.15 [mbar] / ~40 [%]
  -0.20 [mbar] /~50 [%]
  -0.30 [mbar] / ~65 [%]
  -0.40 [mbar] / ~75 [%]
  -1.00 [mbar] / ~99 [%]
  (flat pack / vacuum)



- Not good idea to expose magnets to negative pressures – it ruins refill efficiency and drags helium from magnets.

Helium Recovery – rectifications & expansion

- fixing straps in lower part or in the middle of gas bags
- Back pressure less than ~0.2 mbar when inflated
- gas bags will empty just by their weight when collapsing

Original gas bag size 4 + 12 (effectively ~3+8) m3
Negative pressure (~ -0.4 mbar) needed to achieve that volume

Additional 20 m3 gas bag totalling to 36 m3 volume

Helium Recovery – 4.2 K magnet protection:

- Protecting 4.2K magnets and recovery system:
- LewVac LF NR adjustable pressure relief valves ~50 mbar
- Bruker HF NR relief valves ~5-15mbar - directional





Helium Recovery system & 2K magnet protection

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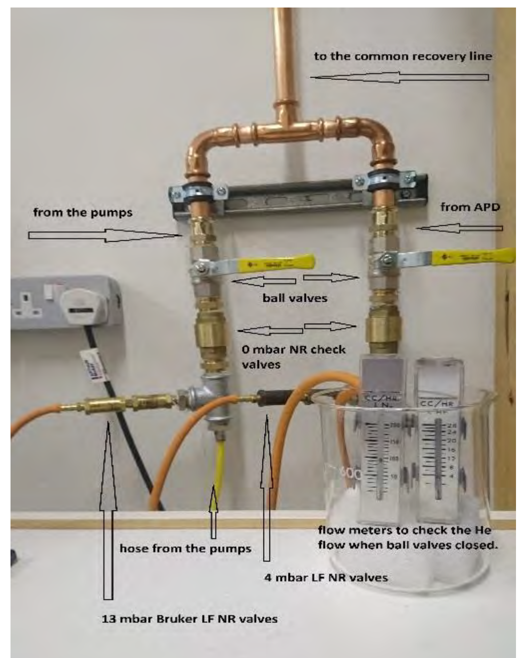
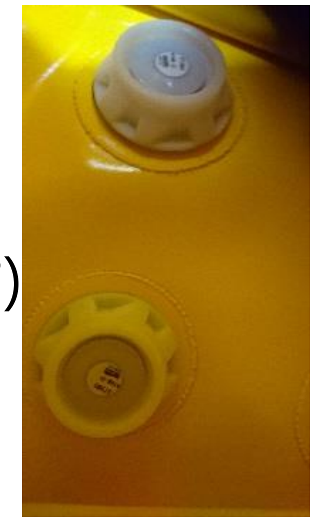
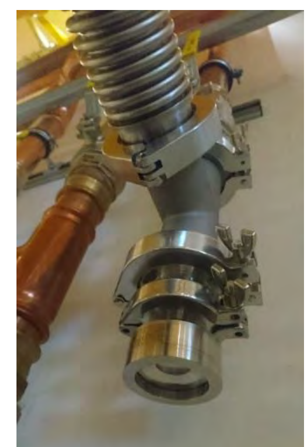
- Protecting 2K magnets and recovery system:
- Bruker HF NR pressure relief valve 70 mbar
- Bruker/Oxford LF NR relief valves ~10mbar
- Burst disk – ultimate protection (exaggerated?)
- Bag relief valves~70 mbar







Helium Recovery – Helium refills

- 2K (800 MHz) magnet has reliable helium probe – easy.
- 4.2K magnet helium probe tends to “freeze” at magical number. Some magnets don’t read until the end of refill.
- With HR in place you loose visual “plume” control. ☹
- Solutions: ping-pong ball valve, **pressure gauge**, Bruker service HF NR valve (noise) – or all of them – paranoia ☺

Ping pong ball valve does NOT create any significant back pressure << 1 mbar

Helium Recovery – Control Panel & operation

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- Control panel monitors pressure in the gas bags (either all or selected) and switches HP compressor accordingly:
- everyday boil off collection: 0.8 mbar ON / -0.2 mbar OFF (once a week for ~ 2.5 hours when all gas bags are full).
- He refills: -0.1 mbar ON (very beginning of refill) & -0.2 OFF after emptying all gas bags.
- Compressor cat off @ -1(?) mbar to protect magnets from negative pressures and @ +210 bar when MCP is full or isolated.
- Compressor capacity 13 m3 / hour
- Pressure during He refills ~0.1 mbar
- Gas meter upstream to compressor to monitor overall gas collected.

Helium Recovery – helium: some useful numbers

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- Helium Densities [kg/m^3] :
- liquid @ 4.2 K = 125.01 kg/m^3
- gas @ 4.2 K = 16.533 kg/m^3
- gas @ 300 K = 0.160 kg/m^3
- Expansion ratios / factors (density ratios):
- Liquid to gas @ 4.2K - 7.56 & @ 300K – 781.31 (757 common)
- 4.2K (cold) gas to 300K (warm) gas - 103.33
- magnet boil off (B) factor = [(B*7.56 –B)*103.33]/B = 781.31 - 103.33 = 677.98 (652 when derived from 757)
- Total gas generated/collected during helium refill [m3] = (Loses[L])*0.757 + (Cold gas out of magnet[L])*0.103
- Isobaric Properties of Helium:

https://cds.cern.ch/record/1444601/files/978-1-4419-9979-5_BookBackMatter.pdf

https://webbook.nist.gov/cgi/fluid.cgi?Action=Load&ID=C7440597&Type=IsoBar&Digits=5&P=1&THigh=300&TLow=0&TInc=0.2&RefState=DEF&TUnit=K&PUnit=atm&DUnit=kg%2Fm3&HUnit=kJ%2Fmol&WUnit=m%2Fs&VisUnit=uPa*s&STUnit=N%2Fm#Info

Helium Recovery – gas bag size calculations

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- Loses = OUT (of transport dewar) – IN (to the magnet)
- Efficiency (Eff.) = IN / OUT (*100 [%])
- Cold gas out of magnet [Litres] replaced by liquid = IN [Litres]
- Compressor capacity (speed) [m^3/hour] = Comp. (e.g. 16 m^3/h)
- Gas bag volume = total gas generated – Comp. speed [m^3/h]*T

  = Loses + Cold gas out - Comp. speed [m^3/h]*Time of refill

  Loses[L]*0.757 + IN[L]*0.103 – Comp. [m^3/h]/60*T(refill)[min]
- Typical 205 L refill (Eff. 85%) in 1 hour = 27.3+20.7-16=32 [m^3]
- Everyday boil off collection only = IN[L]*0.652 [m^3]

  liquid helium equivalent = IN*0.652/0.757*Eff. = ~ IN*0.7[L]

  about 70+ % collection out of liquid helium used for refills.

Helium Recovery – Helium refill examples:

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	Prerequisites (UoN 2014)	Before HR (UoE 2017)	with HR (09/06/2021)	with HR (27/07/2021)	with HR (13/10/2021)
MHz	800	**800**	**800**	**600**	**300-600**
Delivery / transport dewar		235 – 240 litres	248 litres	95 litres	228 litres
Refill / everyday boil off	180 l He 50 days 2.1 m^3 / day	200 l 56 days 2.16 m^3 / day	218 l 64 days 2.22 m^3 / day	83 l 100 days 0.69 m^3 / day	196 l He 91 days 1.48 m^3 / day
Refill time [minutes]	ca 40 min	ca 80 min	ca 55 min	38 min	ca 150 min
Refill speed [litres / min]	4.5 [l/min]	2.5 [l/min]	3.4 [l/min]	2.2 [l/min]	N/A
Refill loss (efficiency)	20% = 27 m^3	20% = 27 m^3	12% = 23.5 m^3	12% = 8.6 m^3	14% = 24 m^3
Cold gas replaced by liquid			22 m^3	8.4 m^3	19.8m^3
Total gas expected	27 m^3 (40 m^3/h)	27 m^3 (20 m^3/h)	45.2 m^3 (49.5 m^3 / h)	17.0 m^3 (26.9 m^3 / h)	43.9 m^3 (17.6 m^3 /h)
Measured by gas meter			45.2 m^3	17.0 m^3	43.9 m^3
Back Pressure	100 mbar		~0 mbar	~0 mbar	~0 mbar
Everyday boil off measured			2.22 m^3 / day	0.34 m^3 / day	1.0 m^3 / day

Liquid helium expansion factor = 757; magnet boil off factor = 652; 4.2 K cold to 300K gas expansion factor =103
Density [kg/m^3] - liquid helium @ 4.2 K = 124.73 kg/m^3; gas @ 4.2 K = 16.757 kg/m^3 ; gas @ 300 K = 0.166 kg/m^3

Helium Recovery – Helium refill summaries:

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1	800 spectrometer / year	unit	18/19	19/20	20/21
2	Transfer dewar helium OUT - calculated from total gas registered (7) & He IN	[L]	1660	1407	1448
3	Magnet helium refills IN (by magnet helium probe)	[L]	1379	1236	1260
4	Everyday magnet boil off gas collected	[m3]	~800	794.2	821.0
5	Liquid He equivalent of boil off (boil off gas (4) / 0.652) (compare to refill - 3)	[L]	~1226	1218	1259
6	Liquid He equivalent of everyday boil off collected (boil off gas (4) / 0.757)	[L]	~1057	1049	1084
7	Gas helium collected during helium refills (tr. efficiency & magnet cold gas)	[m3]	352	256.6	269.4
8	Liquid helium equivalent of gas collected during refills (gas (7) / 0.757)	[L]	465	339	356
9	Helium collected from everyday boil off + refills (6+8) (compare to 2)	[L]	1522	1388	1440
10	Helium refill/transfer efficiency - IN / OUT (3/2)	[%]	83	88	87
11	Helium recovery efficiency - Total helium collected / helium OUT (9/2)	[%]	92	98.6	99.4
12	boil off ONLY recovery efficiency – liquid equivalent / helium OUT (6/2)	[%]	~64	74.5	74.9
13	Total gas He collected from all **NMR & MS spectrometers** – boil off + refills	[m3]	2215	2302	2397
14	Gas Helium compressed to MCP (135 m3) by HP compressor @ 200 bar	[m3]	2008	1917	2239
15	High Pressure Helium Recovery efficiency - MCP volume / total gas (14/13)	[%]	91	83	93

Helium Recovery NMR 2018/19 & overall summary

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- 6 NMR magnets 300 - 800 MHz:
- NMR liquid helium purchases ~2,400 litres = £22,500 + VAT
- Liquid helium transferred to NMR magnets - 2,030 litres with efficiency of about 85%
- Annual everyday boil off of all NMR magnets - 1,270 m^3
- Gaseous helium collected during helium refills ~ 500 m^3
- Estimated total gaseous helium collected from all NMR magnets ~ 1,800 m^3 equivalent to 2,380 litres of liquid helium.
- Total NMR BOC payback / savings of about £5,040 + VAT representing ~22% saving of annual NMR liquid helium cost
- Total volume of gas He collected from all (NMR +MS) magnets (Oct. 2017 to Jan. 2022 -1560 days) 9640 m^3
- Estimated Total BOC payback assuming 88% HP efficiency & £3/m^3 would be ~ £25,500 representing ~40% of the initial cost + service.

Helium Recovery Sustainability - thoughts

- There is NO doubt about sustainability!
- It's all about how much helium you are using and your budget or chances to get some (sustainable?) funding.
- HPR & BOC contract (initial cost ~£70k); current price £14.75 / £3.53(?) per L + 7% inflation. Liquid helium usage & payback:

1000 L ~11+ years; 2000 L ~7+ years; 3000 L ~ 5+ years

- HPR + liquefier (initial cost ~£300k + VAT). Usage & payback:

3000 L ~14+ years; 5000 L ~ 10+ years; 7000 L ~ 7+ years

- QD NexGen liquefier (250 L in 12.5 days) can produce up to 7000 litres / year in continuous use.
- What is more sustainable:

1. transport helium by lorry BOC – customer?
2. Liquefy your "own" helium for extra cost?

Helium recovery – summary & acknowledgments

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- Project funded (50%) by Sustainable Campus Fund of The University of Edinburgh
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- Dr. Logan Mackay – head of MS Facility University of Ed.
- Dr. Huw Williams – University of Nottingham

- Google photos - https://photos.app.goo.gl/wbBbAVSzeeBnrYY97