



PyGC-MS of Lubricants & Persistence Studies

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Specialist Science Solutions

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protecting people and their environment through science

The Importance of Condom Lubricant Evidence

- **Apparent increase in number of sexual assault cases involving condoms**
 - Media coverage & crime shows
 - Contraction of STI's
- **Support or refute a particular account of circumstances**
- **Incapacitated complainant – help identify what happened**
- **Could change a charge of sexual assault to actual rape**

Previous research at ESR:

- **Gareth Campbell**
 - “The Analysis of Condom Lubricants for Forensic Casework”
 - PDMS method development using PyGC-MS
 - Brief investigation into persistence of lubricants *in situ*
 - 3 samples, postcoital times, 4.5hrs, 9hrs & 12hrs



Further research required:

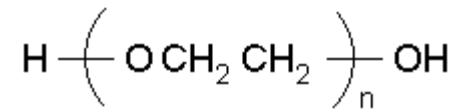
- Further persistence studies to determine how long PDMS & PEG will remain *in situ*
- Stability studies – swabs & fabric @ RT & in cool stores
- Effect of biological fluids on persistence & their interference



Condoms present on NZ market in 2004

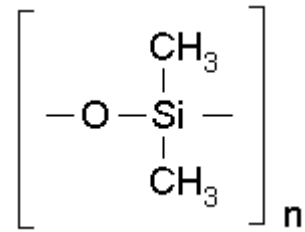
- **Durex**
 - Avanti PDMS
 - Classic PDMS
 - Close Fit PDMS
 - Comfort PDMS
 - Confidence PDMS
 - Excita PDMS
 - Extra Confidence PEG
 - Extra Safe PEG
 - Fetherlite PDMS
 - Select Banana PDMS
 - Select Orange PDMS
 - Select Spearmint PDMS
 - Select Strawberry PDMS
 - Sensation PDMS
- **Sagami**
 - Sustaining PDMS
- **Gold Knight**
 - Regular PDMS
 - Chocolate PDMS
 - Strawberry PDMS
 - Mint PDMS
- **Ansell**
 - LifeStyles Regular PDMS
 - LifeStyles Ribbed PDMS
 - LifeStyles Ultrathin PDMS
 - Mega PDMS
 - Bareback PDMS
 - Exotica PDMS

Polyethylene glycol (PEG)



- < 8 hour time interval, neg for PDMS
- Not identified in casework samples
- Further study would either reinforce <8hr time interval or provide a more confident interval

Polydimethylsiloxane (PDMS)



- < 24 hour time interval
- 22% of cases positive for PDMS
- One positive case had a reported time interval of 24 hours

In situ persistence

- **Staff participation consent form & Information sheet created**
- **50 x Sample packs prepared**
 - 2 condoms, 4 x swabs
 - Specific postcoital time period for sampling
- **Abstain from intercourse for at least 48 hours**

In situ persistence continued

- 7 kits were completed & returned
- Shimadzu GCMS-QP2010
 - PY-2020iD Double-Shot Pyrolyser (Frontier Laboratories Ltd)
 - Auto-shot Sampler AS-1020E (Frontier Laboratories Ltd)

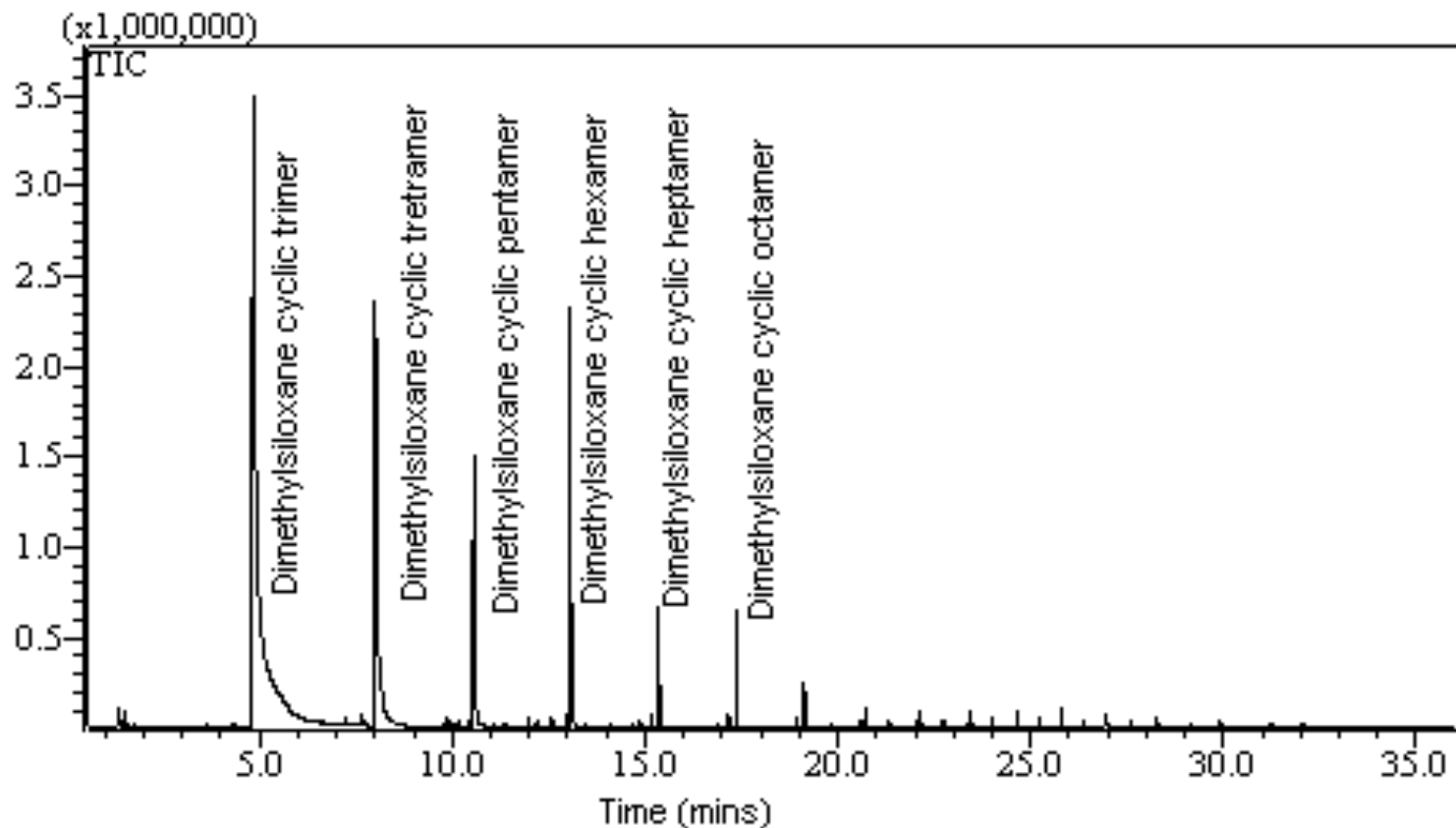
Extraction (*in situ* persistence continued

- Half of each swab extracted with ~ 400uL hexane
- Extract placed into pyrolysis cup & evaporated to dryness



In situ persistence continued

Total Ion Chromatograph of PDMS cyclic oligomers

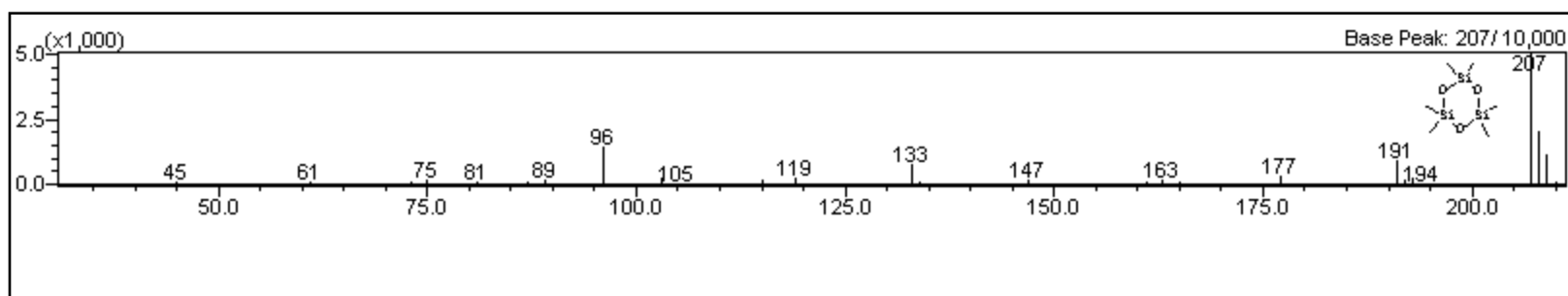


Criteria for PDMS detection

- Identification of D3 & at least two other cyclic species within D4 to D7 range
- D3 – must have isotopic cluster at 207, 208 & 209 m/z

Cyclic DMS species	M ⁺ Ion (m/z)	Additional Abundant Ions (m/z)
Cyclotrisiloxane (D3)	207	96, 133, 191
Cyclotetrasiloxane (D4)	281	73, 133, 191, 207, 249, 265
Cyclopentasiloxane (D5)	355	73, 267, 268
Cyclohexasiloxane (D6)	429	73, 147, 341
Cycloheptasiloxane (D7)	503	73, 147, 281, 327, 415

D3 Mass spectrum



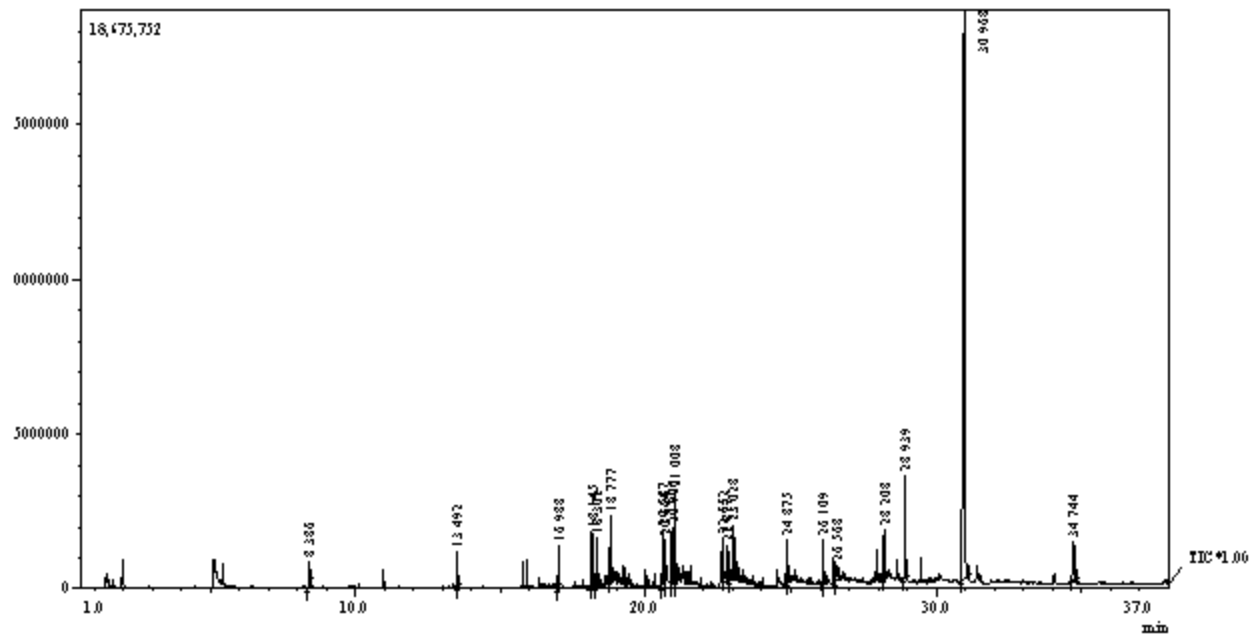
In situ results



Participant	Postcoital Time	PDMS detected
1	2 hours	D3-D7 Yes
1	35 hours	D3-D5 Yes
1	6 hours	D3-D7 Yes
1	32 hours	D3-D6 Yes
2	4.5 hours	D3-D7 Yes
2	48 hours	D3-D4 No
2	16 hours	D3-D7 Yes
2	22.5 hours	D3-D7 Yes
3	4 hours	D3-D7 Yes
3	34.5 hours	D3-D4 No
3	10.5 hours	D3, D4, D6 Yes
3	28 hours	D3-D4 No
4	12 hours	D3-D7 Yes
4	33 hours	D3-D7 Yes

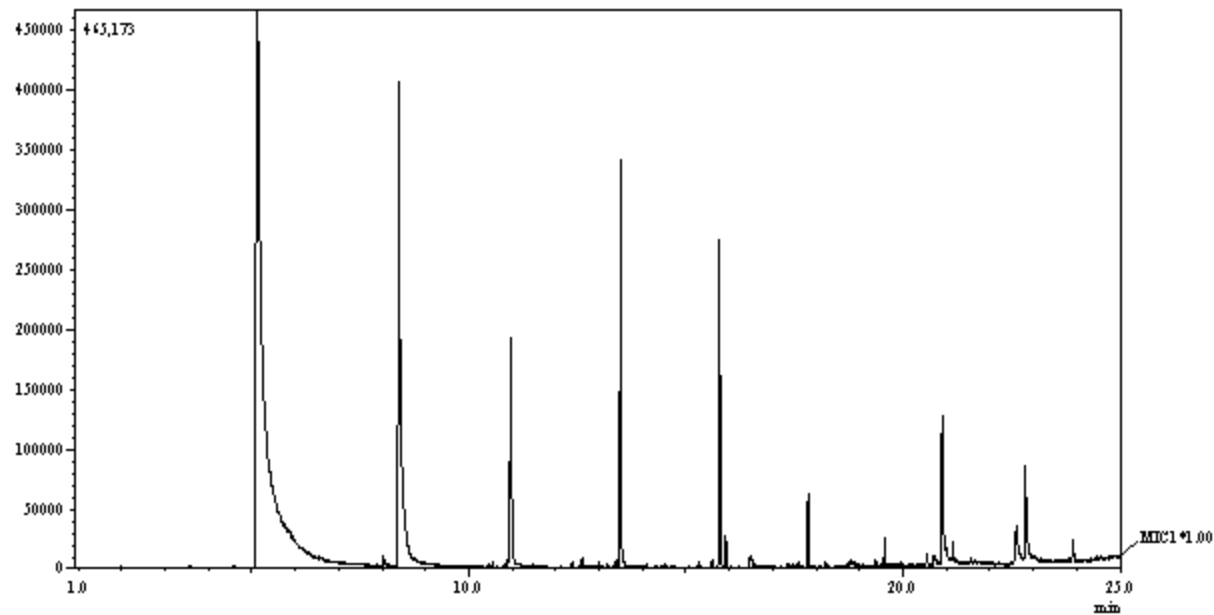
Participant 2 – 16 hour interval

Total Ion Chromatograph



Participant 2 – 16 hour interval

Multiple Ion Chromatograph m/z 73, 133, 207, 281



Uncontrolled factors present

- **Intra-individual**
 - Length of condom use
 - Natural changes in the vaginal vault
 - Degree of sampling

- **Inter-individual**
 - As above
 - Individual variations
 - Activity pre-sampling such as washing style
 - Different ability to secrete/remove lubricant

Participant	Postcoital Time	PDMS detected		
1	2 hours	D3-D7	Yes	n/c
1	35 hours	D3 only	No	D3-D5
1	6 hours	D3-D7	Yes	n/c
1	32 hours	D3-D6	Yes	n/c
2	4.5 hours	D3-D6	Yes	D7
2	48 hours	D3 only	No	D4
3	4 hours	D3-D7	Yes	n/c
3	34.5 hours	D3 only	No	D4
4	12 hours	D3-D7	Yes	n/c
4	33 hours	D3-D6	Yes	D7

Reasons for the loss of cyclic DMS oligomers

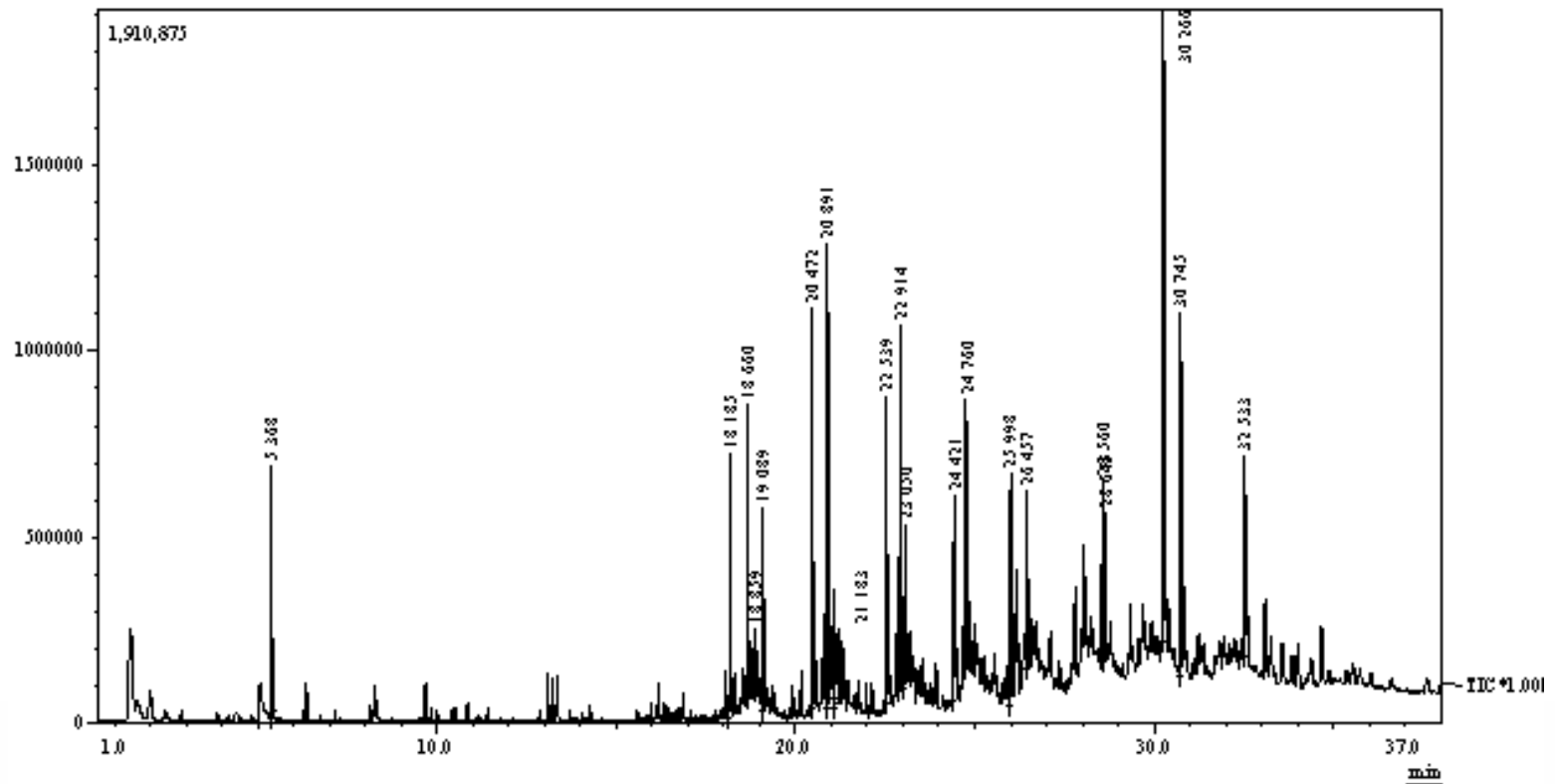
- **Instability of PDMS**
- **Decrease in sensitivity of the instrument**
- **Less PDMS present**

Persistence in presence of biological fluids

- Urine
- Blood (heparin)
- Faeces
- Semen
 - Swabs (sterile) stored in fridge
 - Fabric (cotton) stored at room temperature

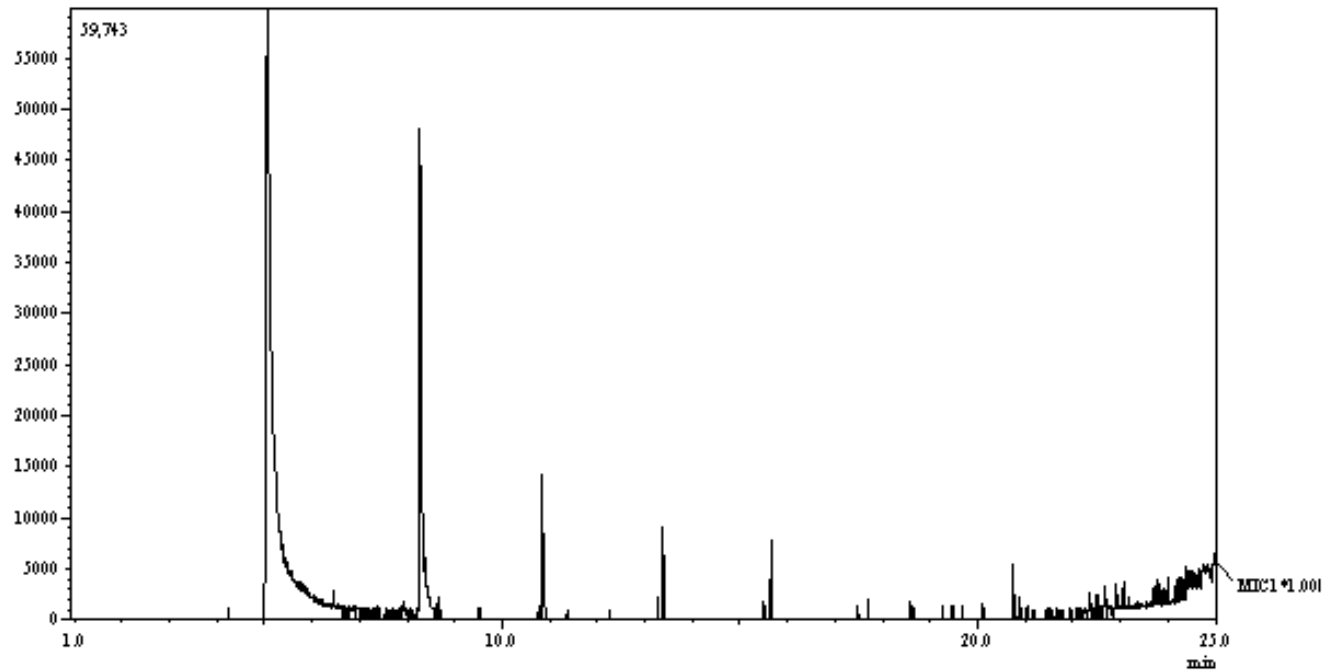
Swab extract in presence of faeces

Total Ion Chromatograph



Swab extract in presence of faeces

Multiple Ion Chromatograph m/z 73, 133, 207, 281



Conclusions

- PEG no longer used in NZ condoms
- PDMS detected at 35 hours
- Persistence varied between & within individuals
- No affect due to storage temperature
- Biological fluids appear to have no effect
 - Persistence or interference
- PyGC-MS is a sensitive technique for PDMS analysis
 - But must make sure of clean system

Further work

- **Extension of study is desirable**
 - **More couples & samples required**
 - **Couples providing anal samples**

References

1. Gareth Campbell. **The Analysis of Condom Lubricants for Forensic Casework. Masters of Science Thesis, University of Auckland, 2004.**
2. Fujimoto, S., H. Ohtani and S. Tsuge. **Characterization of polysiloxanes by high-resolution pyrolysis-gas chromatography-mass spectrometry. Fresenius Z Anal Chem (1988) 331:342-350.**
3. Camino, G., S. Lomakin and M. Lazzari, 2001, **Polydimethylsiloxane thermal degradation. Part 1. Kinetic aspects, Polymer Vol. 42, 2395-2402.**

