



# Quality control of BioDME produced via black liquor gasification

Olov Öhrman, Esbjörn Pettersson, Marcus Gullberg, Urban Westbom  
Energy Technology Centre in Piteå, Sweden

[olov.ohrman@etcpitea.se](mailto:olov.ohrman@etcpitea.se)

[www.etcpitea.se](http://www.etcpitea.se)

# Agenda

- Introduction to the work
- Analysis methods
- Results
- Conclusions

# BioDME



- Need to be able to analyze BioDME samples
- Feedback to the Plant operation on product quality
- Secure that the fuel used in the field test reach specification
- Comparison with fossil DME and BioDME – are there different impurities?
- Log of analysis – stability, conversion etc.

# Analysis methods

- DME test sample
- Gas chromatography
- Karl Fischer titration
- Total sulfur
- Residue

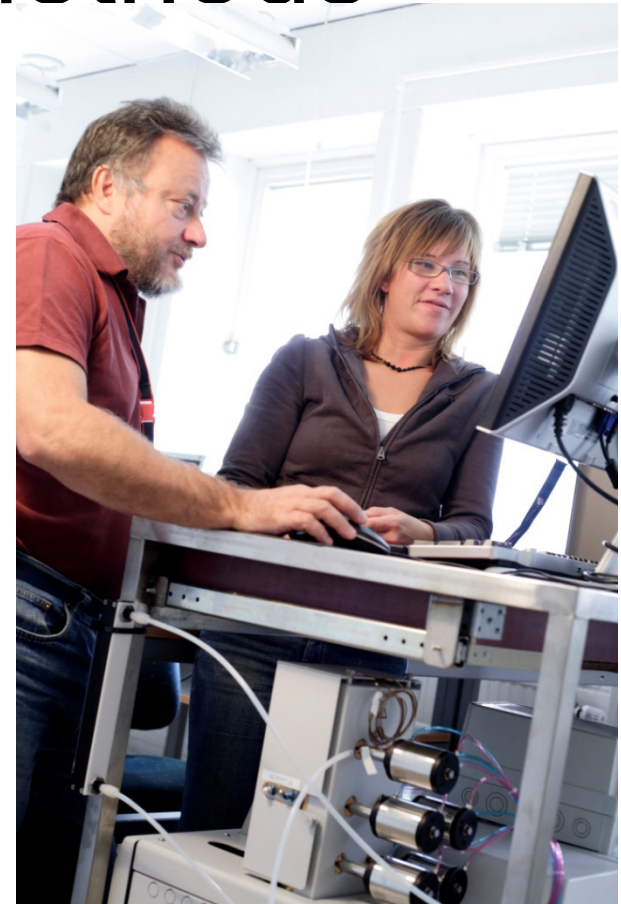
# DME test sample

- Filling of DME to a 1 gallon teflon lined swagelock double valve cylinder from the Piteå filling station



# Analysis methods

- **Gas chromatography**
  - Transfer of DME slowly to gas bag
  - Injection to GC from gas bag
  - 10 measurements
  - TCD detectors for H<sub>2</sub>, CO, CO<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>
  - PFPD detector for sulfur compounds
  - FID detector for Hydrocarbons and oxygenates



# Analysis methods

## Karl Fischer titration

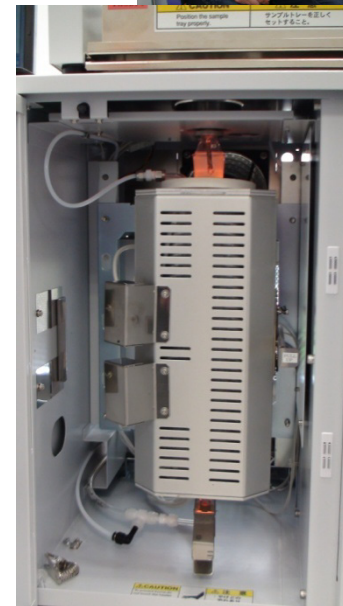
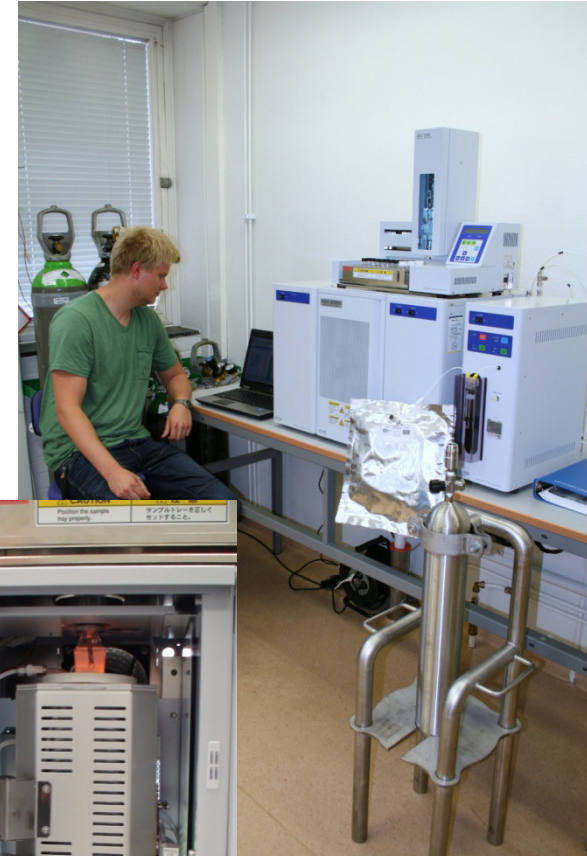
- Coulometric method
- Pressure vessel with DME
- Needle valve to feed DME to titrator through a syringe
- Weigh pressure vessel before and after
- Titration → water content
- 10 measurements from the same sample



# Analysis methods

## Total sulfur by UV-F

- Transfer of DME from 1 gallon pressure vessel to gas bag
- Automatic injection of DME gas to analysis instrument
- 10 measurements on each gas bag



# Analysis methods

## Residue by evaporation

- Transfer of DME from 1 gallon pressure vessel to 500 cm<sup>3</sup> cylinder
- Cooled stainless steel coil (-50 °C)
- 50-100 g liquid DME collected in stainless steel cup
- Evaporation, and drying at 105 °C
- Masses of cylinder and cup → residue content
- 10 measurements



# Results

## Gas chromatography

	ppm (wt/wt)	RSD %
C1	2	9.9
C2	45	2.4
C3	6511	2.9
C4	653	3.8
CO2	18	14.6
Methanol	1328	3.4
Ethyl mercaptan	8.4	7.5

# Results

## Total Sulfur

	ppm (wt/wt)	RSD %
Rinse 1 time with DME	3.044	0.61
Rinse 3 times with DME	3.653	0.46
Filling very fast	4.227	1.16

**3.653 ppm Total Sulfur corresponds to 7.1 ppm Ethylmercaptan  
8.4 ppm Ethylmercaptan was measured by GC**

# Results

- Karl Fischer:
  - 148.6 ppm H<sub>2</sub>O with RSD = 3.08 %
- Residue:
  - 580 ppm with RSD = 3.4 %

# Conclusions

- DME samples were analyzed for impurities with satisfactory reproducibility
- Filling of gas sample bags were important handling
- The methods are now in use for analysis of the BioDME produced in Piteå
- Further optimization may be needed depending on impurities and stability

# Acknowledgements

- Kempe Foundation for financial support of Total sulfur and Karl Fischer instrument

