

Analysis of ppb Levels of Nitrous Oxide by Gas Chromatography

- Paper # 460-7
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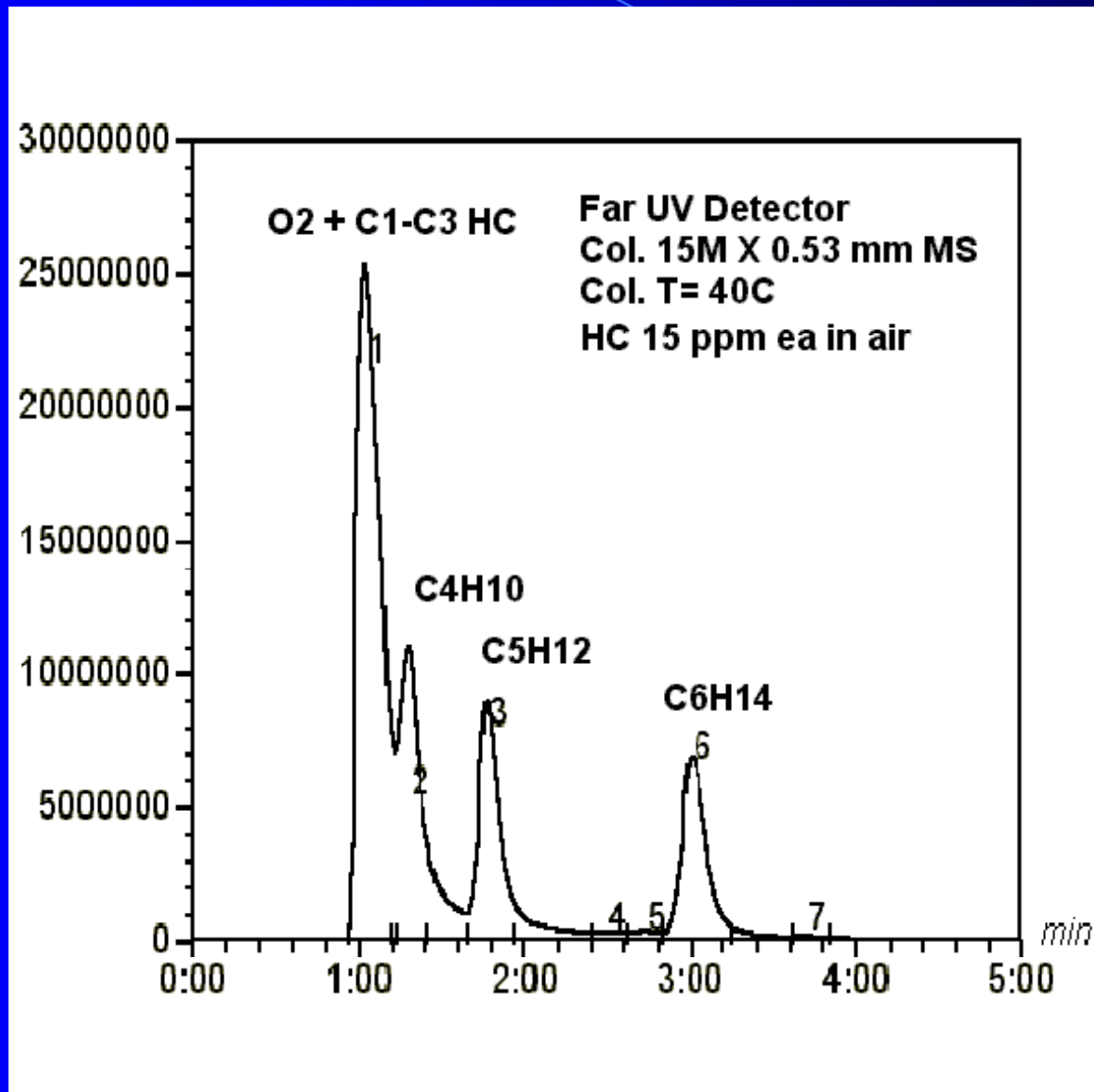
INTRODUCTION

- Nitrous oxide (N_2O) is produced in the soil by the decomposition of nitrogen compounds. This results in an average atmospheric concentration of approximately 0.25 ppm. It is also used in dental offices and in hospital operating rooms during surgery.
- Methods used for determination of N_2O include infrared (IR) spectroscopy and gas chromatography. The former method is useful to just below 1 ppm although photoacoustic IR has a detection limit of 30 ppb. The GC detectors used for N_2O include the thermal conductivity detector (TCD) for high levels (> 100 ppm) and the electron capture detector (ECD) (100 ppm to app. 0.1 ppm).
- We will be evaluating a Far UV absorbance detector coupled to a concentrator to detect low ppb levels of N_2O

FUV Detector

- Consists of a uv lamp (120 nm), a 1 cm pathlength cell (50 uL) and a UV photodiode that measures lamp intensity
- As a sample enters the cell, the signal will decrease in proportion to the absorption coefficient of the species
- Response- organic HC, fixed gases & inorganic gases as shown in next slide

Chromatogram of HC with FUV Detector



Far UV Detector

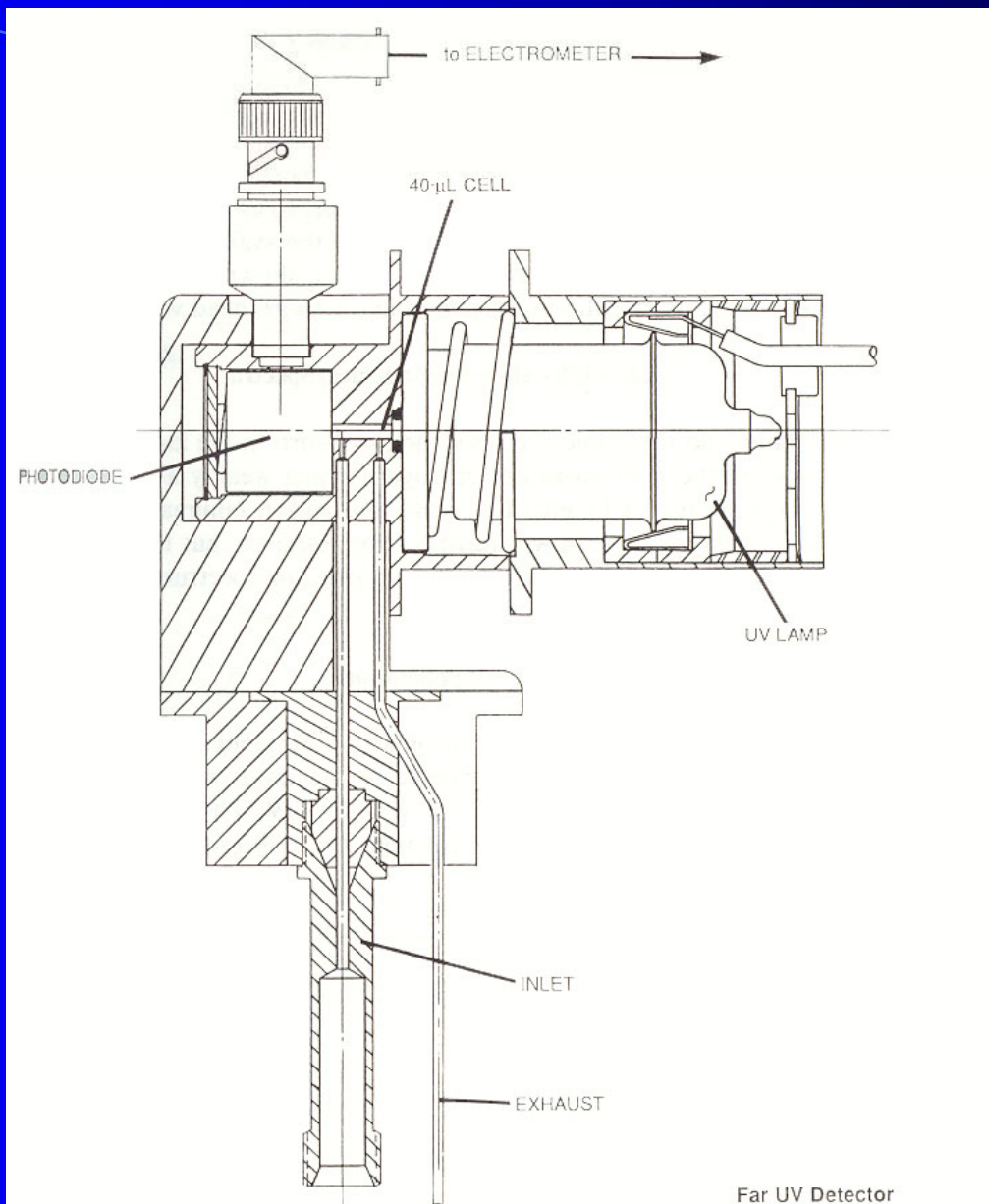


Figure 4.20. Schematic drawing of far-UV absorbance detector.

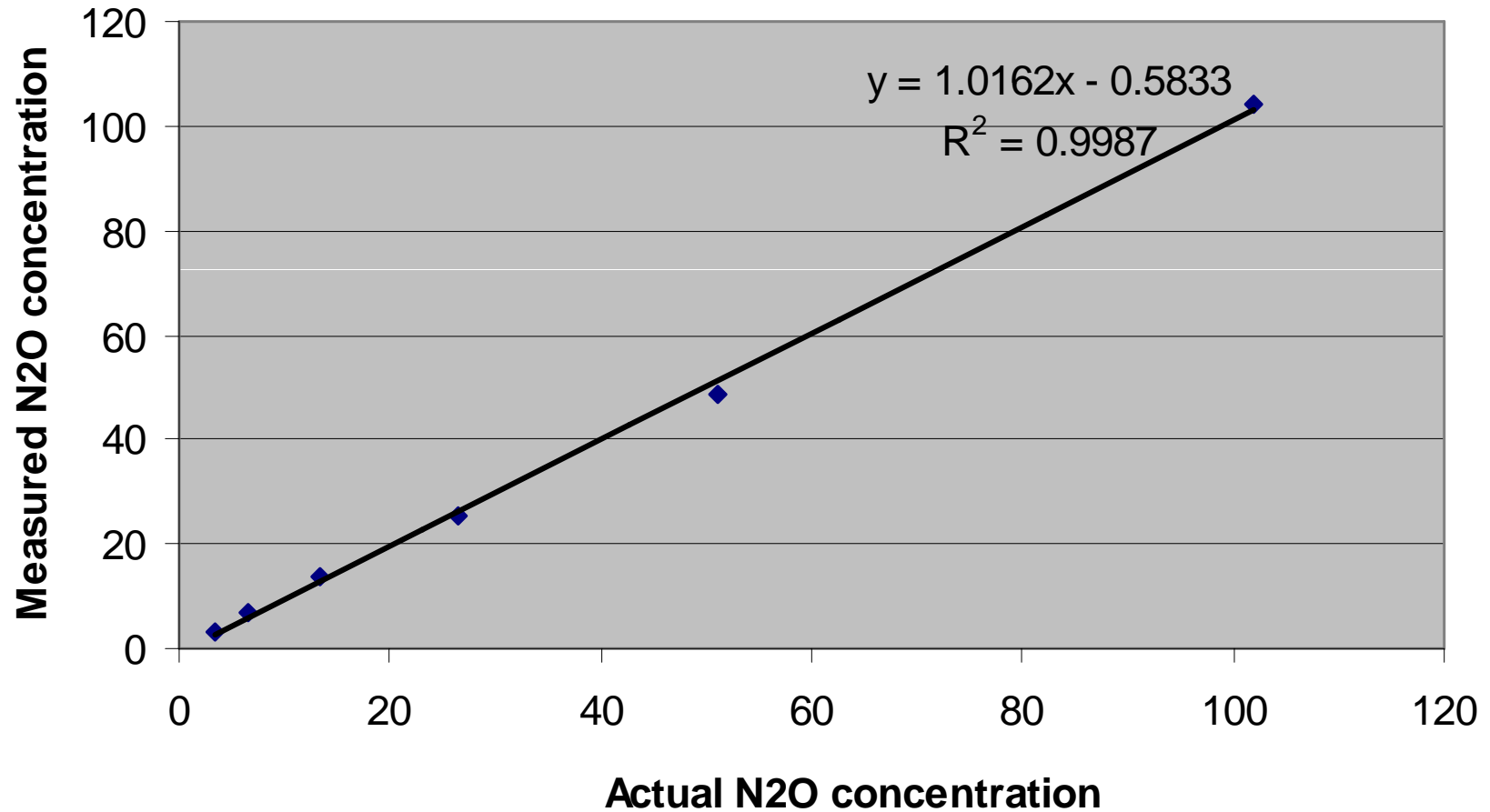
Photo of Model 301-B



Equipment used

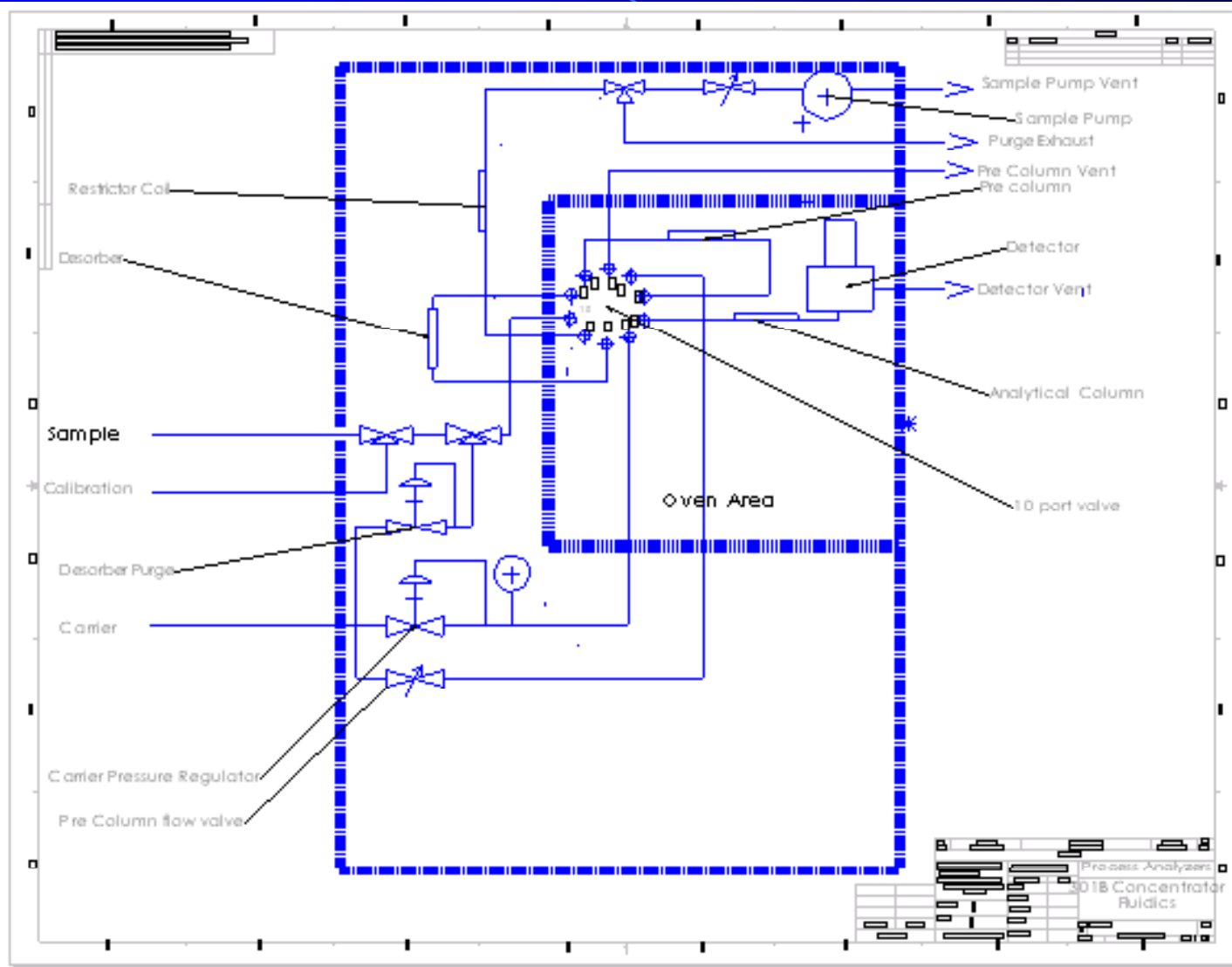
- PA Model 301-B GC
- Modified by adding a 6 port manual valve with sample loops from 0.1 to 50 cc
- Low ppm concentrations prepared by dilution of gas standard (1000 ppm N₂O) in zero air
- 10 M x 0.53mm PLOT Molesieve
- Far UV detector (50 uL dead volume)

Actual vs Measured N2O by GC-FUV (ppm)



Detection limit- 500 ppb

Concentrator for N₂O

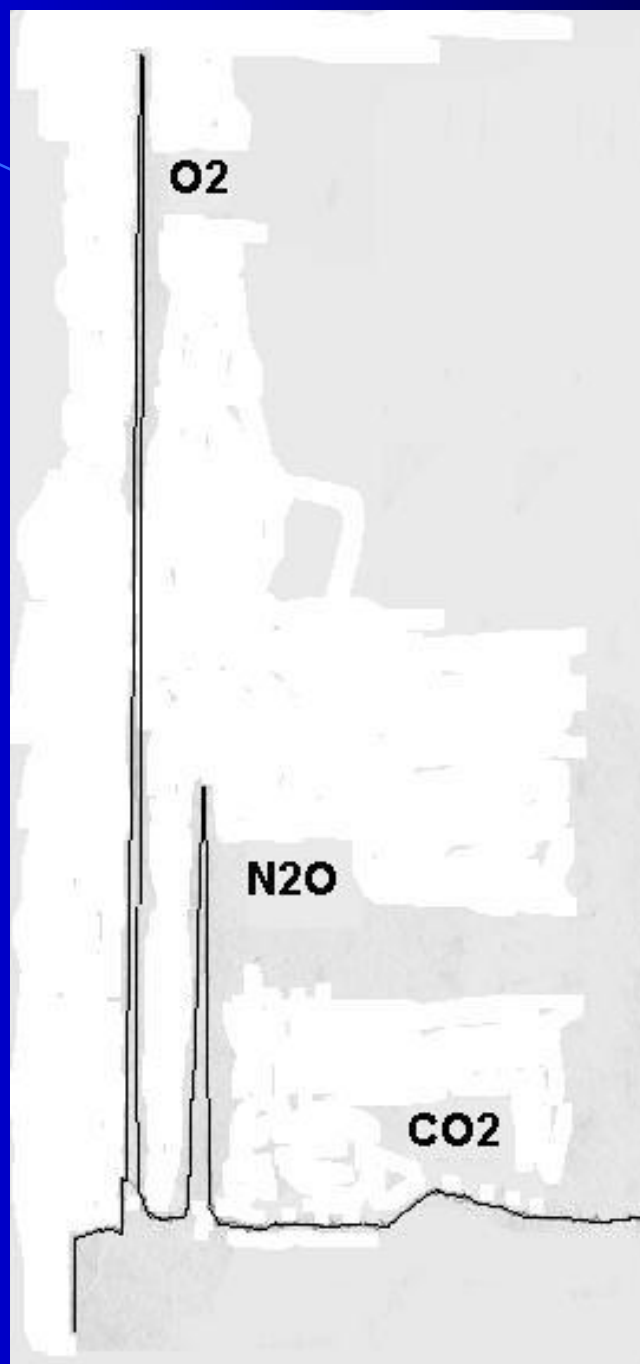


Concentrator Parameters

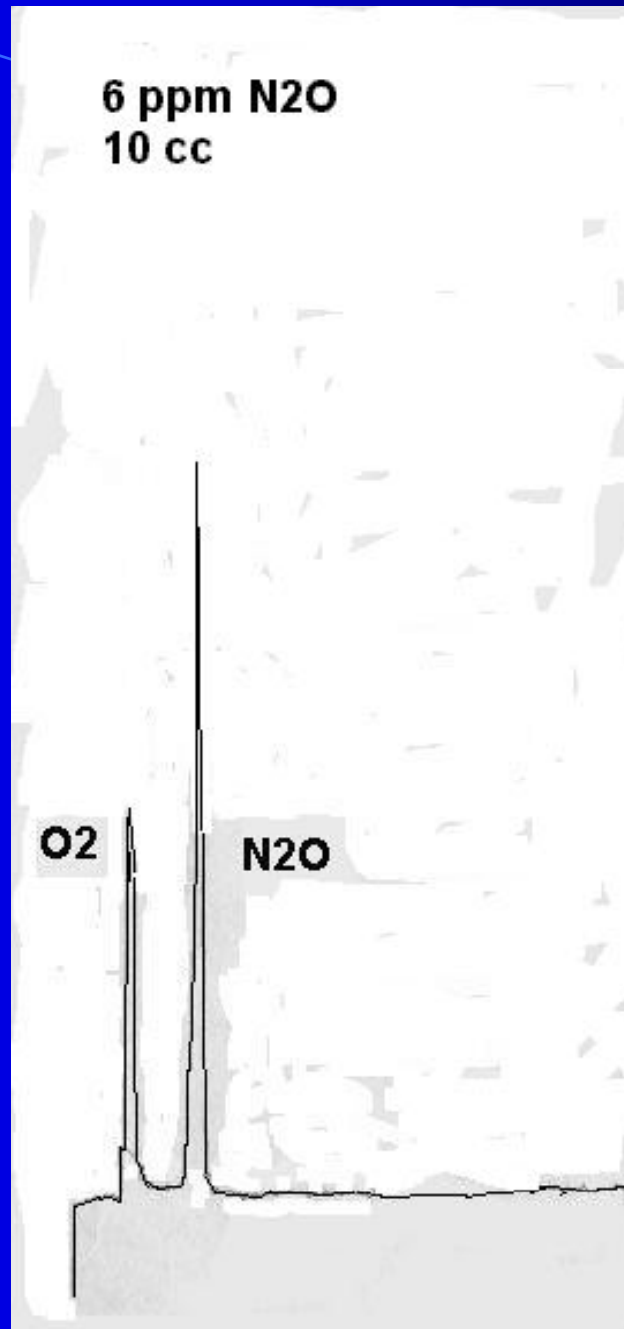
- Material : molecular sieve- 80/100 mesh
- Desorb volume- 10-50 cc
- Desorb temperature: 150°C
- Time to max.T – 4 seconds
- Can vary T_{\max} from 1 second to 4 min.
- Cycled between runs to clean column
- All parameters are under PC control

**Chromatogram of 50 ppm
 N_2O with FUV Detector**

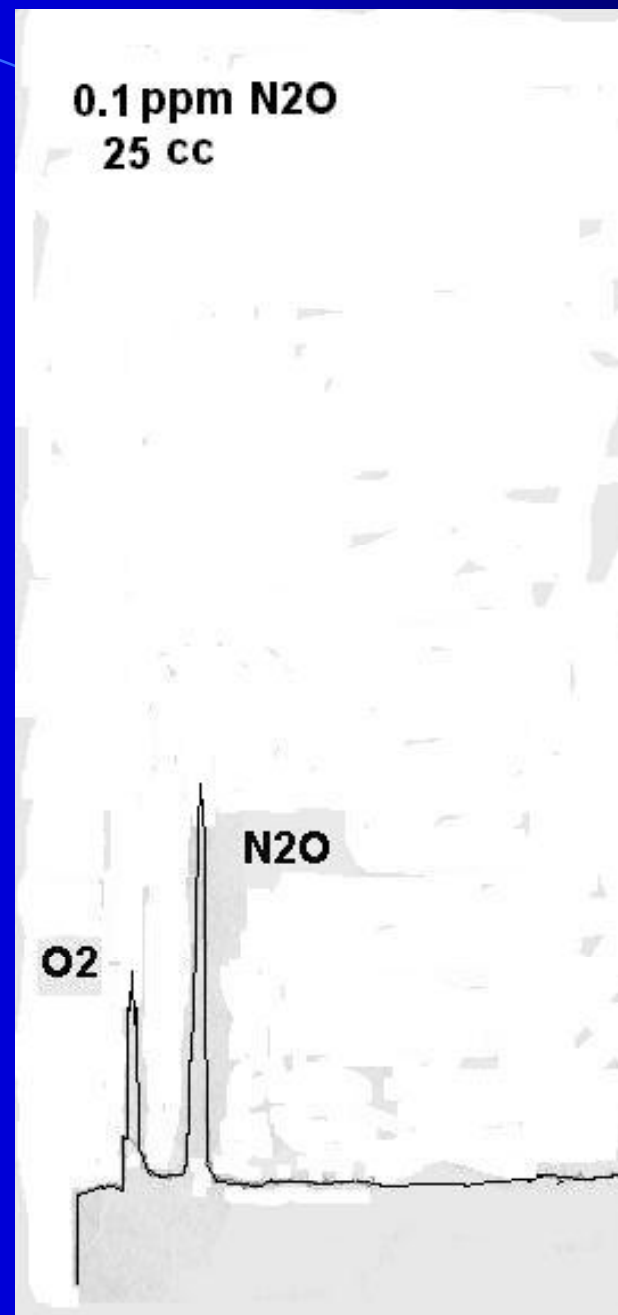
10 M x 0.53 mm PLOT
Molesieve, 15 cc/min.
80C; 50 ppm N_2O in
ambient air



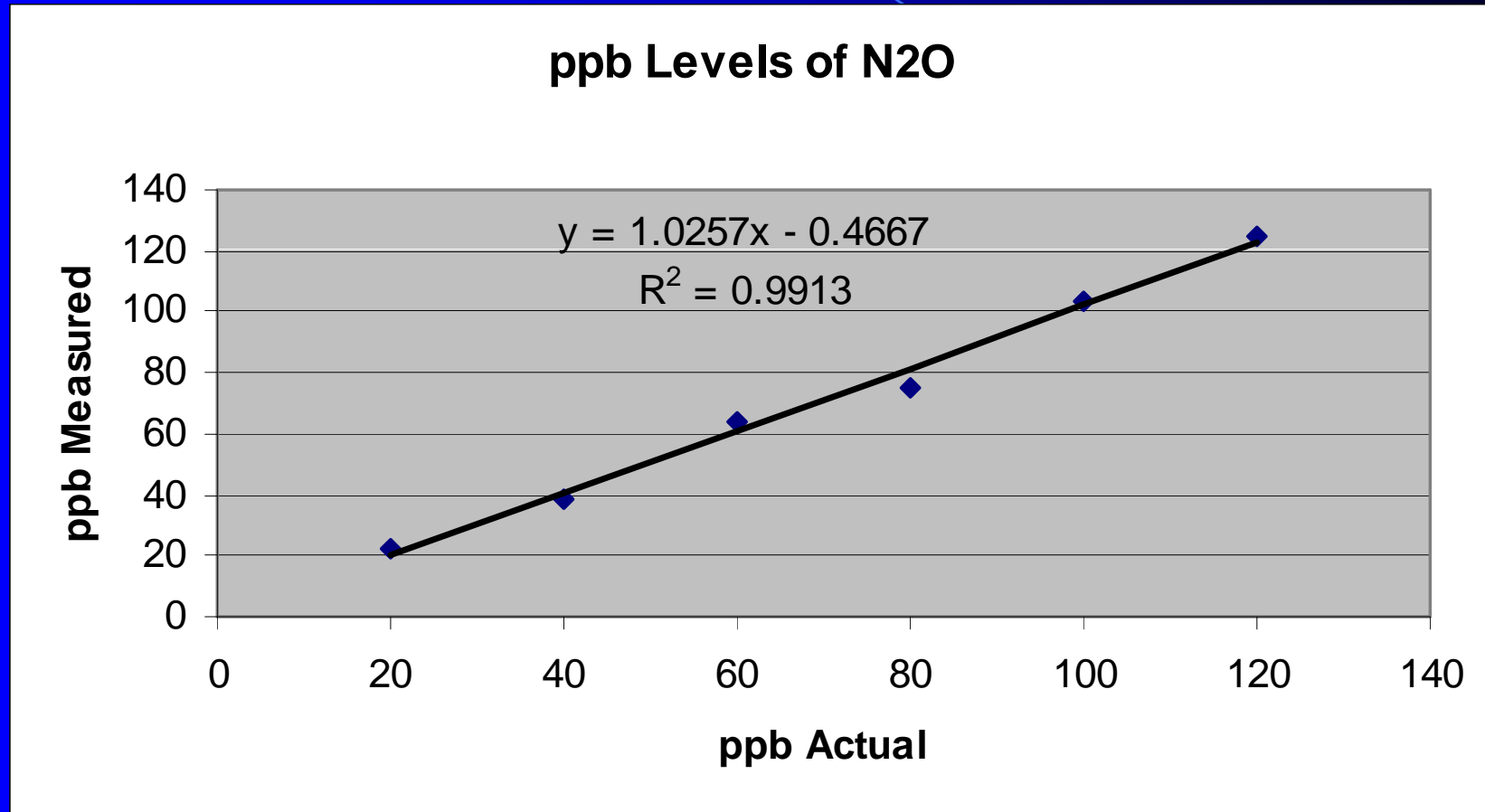
**N₂O 10 fold
concentration**



100 ppb N₂O
25 fold
concentration



Calibration Curve for Concentrator



SUMMARY

- By coupling a FUV detector to a PLOT column, we have been able to detect from 500 ppm down to 250 ppb of N₂O.
- This covers approximately the same range as the ECD
- We have evaluated a concentrator/ thermal desorber
- That improves the detection of N₂O to 5 ppb.
- The FUV (without a concentrator) is will provide a range that would be adequate for workplace monitoring of N₂O.
- The FUV with a concentrator is ideal for monitoring N₂O in ambient air