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Test and Calibration Procedures for the PFT Laboratory Equipment

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Introduction

Brookhaven National Laboratory (BNL) in the USA has developed a multiple tracer gas technique for determination of air infiltration rates in buildings. The technique is based on passive emission and passive adsorption of tracer.

The Danish Building Research Institute (SBI) and the Tracer Technology Center at BNL entered, in 1986, into a collaboration agreement. As a part of this collaboration SBI has tested the technique. On the basis of the tests and the results of the measurements, SBI decided, in 1988, to purchase the gaschromatographic equipment for the analysis of passive samplers. The equipment purchased is constructed by Princeton University, Princeton and John Booker & Co., Texas based on the work of BNL.

This paper describes briefly the outline of some of the tests and calibration procedures the Danish Building Research Institute has carried out in order to get a reliably running system.

1. Tests

Thermal Desorption

The tracer gas mixed with the room air is collected passively in the tubes by adsorption. The adsorbent material in the tubes is Ambersorp. The adsorbed tracer is taken off the adsorbent material by means of thermal desorption. In a rack holding 23 tubes the tubes are heated in two steps by heating coils for approximately 45 seconds. Numerous tests of the applied power to the heating coils, and the duration of both high power and low power time has been carried out. Figure 1 shows the results of two times thermal desorption of 23 equally exposed tubes. As can be seen, more than 99.7 pct. of the adsorbed tracer is taken off by the desorption. The present settings are maximum as the tubes are just about to be destroyed by deformation.

Rack position	Peak height 1'st desorption	Peak height 2'nd desorption	Pct. remain
1	76860	197	0.26
2	74387	140	0.19
3	75830	147	0.19
4	74567	140	0.19
5	73922	142	0.19
6	72915	150	0.21
7	69430	117	0.17
8	69197	122	0.18
9	71637	132	0.18
10	74730	132	0.18
11	71502	130	0.18
12	76397	127	0.17
13	76542	125	0.16
14	75857	145	0.19
15	69577	113	0.16
16	70077	117	0.17
17	76040	135	0.18
18	71910	117	0.16
19	78040	147	0.19
20	80625	150	0.19
21	76680	152	0.20
22	73087	152	0.21
23	81992	185	0.23
<hr/>			
Average	74426	140	0.19
SD	3415	20	0.02
Pct. SD	4.59	14.61	12.27

Figure 1. Test of thermal desorption of 23 equally exposed tubes.

Rack test

Through 8 runs 16 rack positions (position 1-16) has been tested for uniform function. In order to minimize influence from dissimilarities of the tubes, different tubes were used for each run. As seen from figure 2, analysis is performed within 5-8 pct.

Figure 3 shows results from the same test, this time rack positions versus runs. As can be seen, no significant difference regarding the rack positions is showing, but the runs are differing. Run 1 and 2 are performed the same day, run 3, 4 and 5 the following day and run 6, 7 and 8 the third day. Run 1, 3 and 6 are performed in the morning. Later in the day the temperature in the laboratory is increasing resulting in a shift in baseline and performance. This test indicates the importance of using reference tubes.

Catalyst Temperature

Figure 4 shows the chromatograms of analysis of 7 equally exposed tubes with 7 different temperatures of the catalysts. The higher the temperature of the catalyst the higher the peaks, particularly on the third isomer of PDCH, until 250 °C. Thereafter PDCH is lost. The study shows that a catalyst temperature of 250 °C seems to be most expedient, and this temperature is chosen.

Column Temperature

Figure 5 shows the chromatograms of analysis of 5 equally exposed tubes with 5 different temperatures of the column. At lower temperatures PDCH is not eluting from the column. At higher temperatures an increase in the retention times is seen, but at the same time separation of the tracers becomes difficult. A column temperature of 135 °C is chosen.

Breakthrough

Tubes are loaded with a gas, being a mixture of the tracers in use, for example when preparing for a calibration or when making reference tubes. The gas is supplied from a tank. Experience, gained from comparing the amount of tracer analysed with the amount of tracer loaded, tells that the gas flow should be from 10 to 40 ml/min. A flow less than 10 ml/min is hard to control accurately, and a flow greater than 40 ml/min may cause breakthrough, i.e. not all of the supplied tracer gas is adsorbed in the tube.

Emission Rates

Regular weighings of the tracer sources have been made in order to establish the emission rates and dissimilarities of the sources. The sources have not been kept in a constant temperature bath, but results indicate that information on the average emission rates given by BNL, who is the manufacturer of the sources, is reliable, but with a greater deviation.

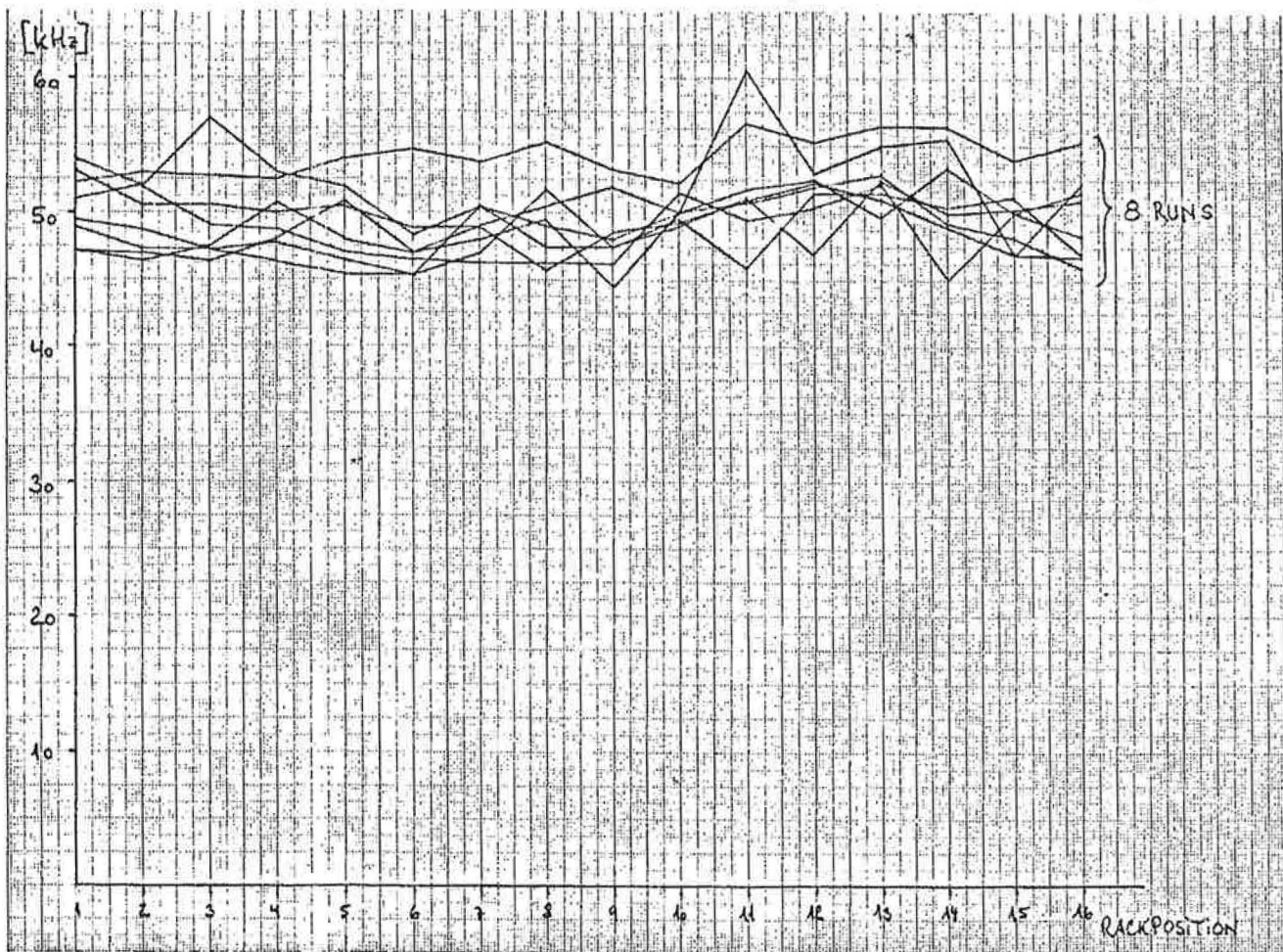


Figure 2. Test of uniform function of 16 rack positions.

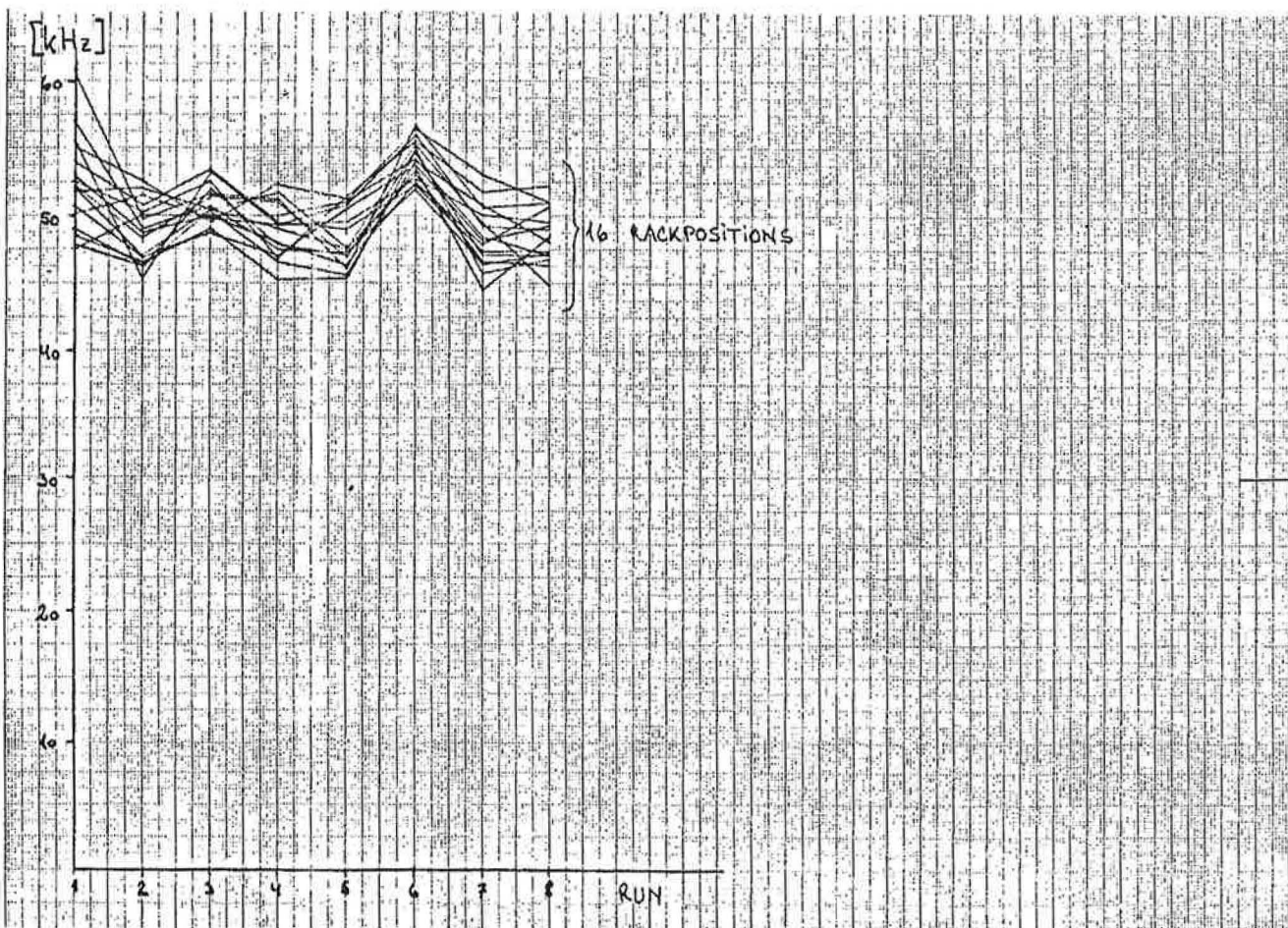


Figure 3. Test of uniform function of 16 rack positions.

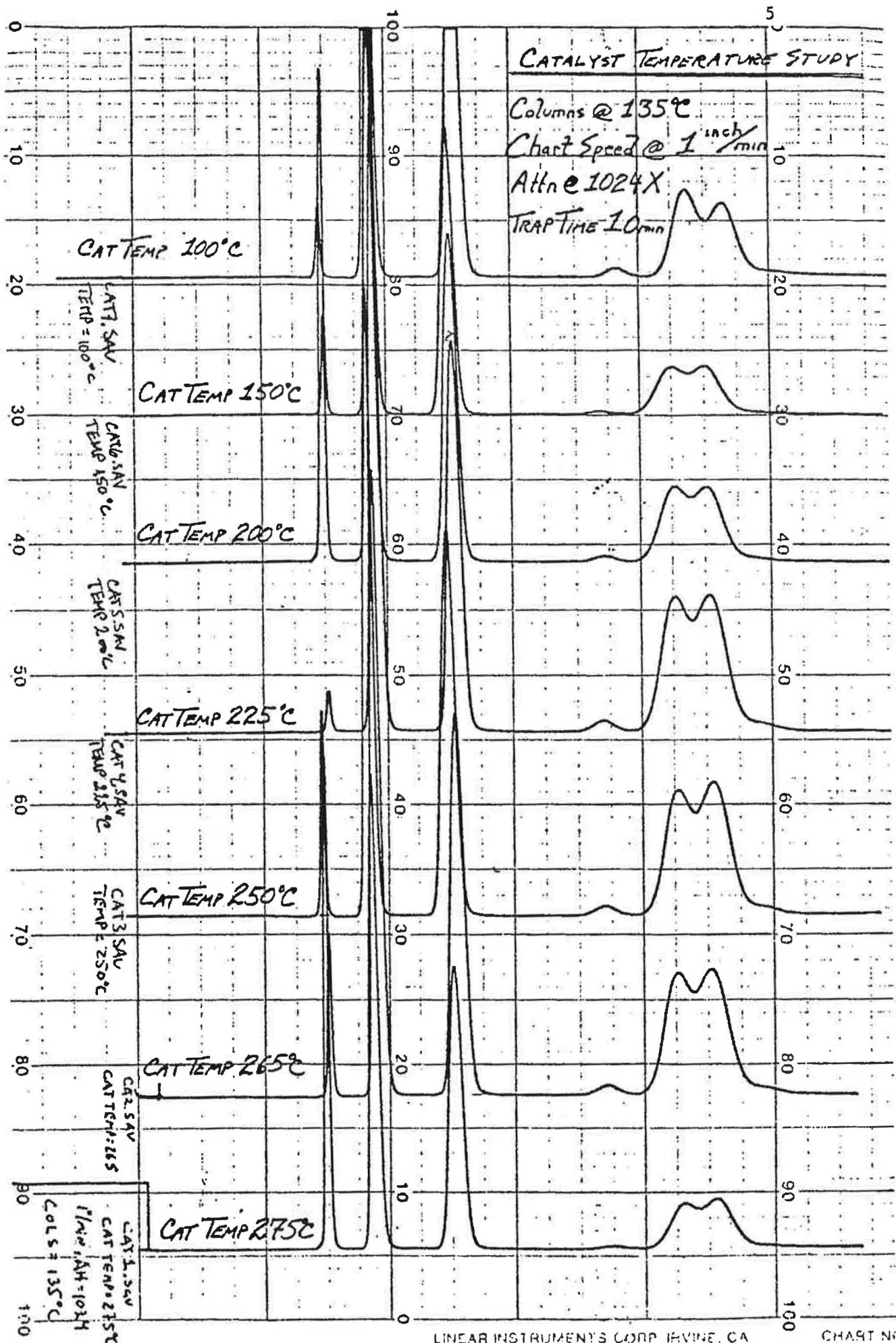


Figure 4. Catalyst Temperature Study.

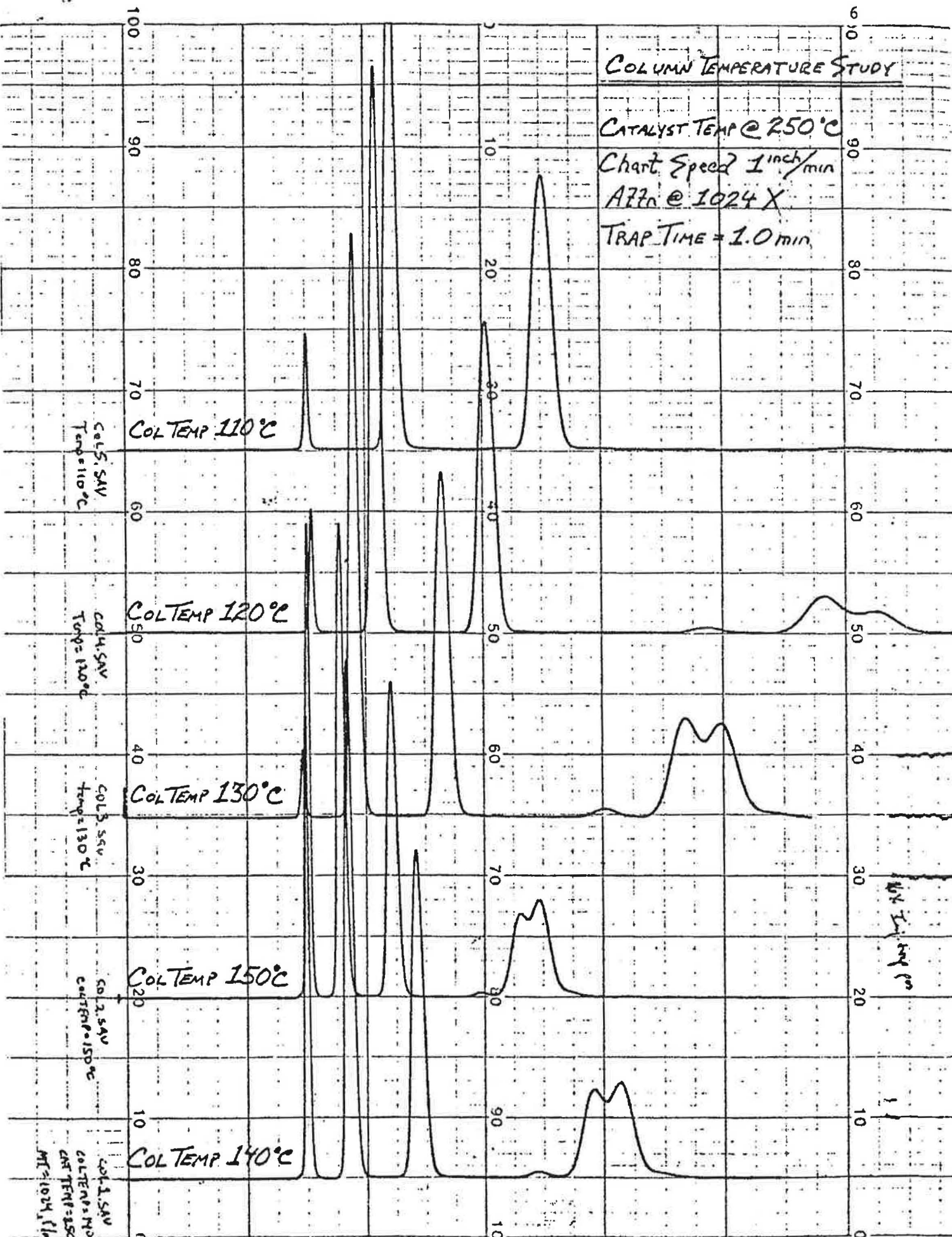


Figure 5. Column Temperature Study.

2. Calibration

Calibration curves, that is polynomial equations, giving the connection between peak height and volume of tracer have to be established for each of the tracers in use. This is done by loading a set of tubes with different but known amounts of tracer and then analyse these tubes. The loading is in practice done from a tank containing a mixture of the tracers.

$$\begin{array}{rcccc} \text{Loaded volume} & = & \text{Exposure time} & \cdot & \text{Flow} & \cdot & \text{Concentration} \\ [\text{pl}] & & [\text{min}] & & [\text{l/min}] & & [\text{pl/l}] \end{array}$$

The exposure time and the flow is measurable, but as the concentration of each tracer in the tank is unknown, and has to be measured with the instrument that is to be calibrated, the concentration of the gases in the tank has to be established first. This is done by loading tubes in another way with known amounts of tracer and comparing analysis of these tubes with analysis of tubes loaded from the tank. The Danish Building Research Institute have loaded tubes with known amounts of tracer in two ways: Injection with a syringe of samples taken from a test chamber and of samples taken from a flask.

Test Chamber

Tracer sources with a known emission rate have been placed in a stainless steel test chamber ventilated at a constant rate. The volume of the chamber, the temperature and the ventilation rate is known with great accuracy. After allowing the system to equilibrate for several weeks, samples of the air in the chamber are taken in grab sampling bags. With a syringe samples are taken from the bags and injected into a carrier gas stream flowing through a tube.

From analysing the tubes and comparing the results with analysis of tubes loaded from the tank, the concentration of the tracers in the tank can be determined, and the coefficients to the polynomial equations defining the calibration curves can be calculated. Figure 6 is showing the analysis results for PMCP and PMCH. The straight lines shown are the calibration curves computed as a first order polynomial (linear).

Flasks

A few milligrams in liquid form of each tracer is weighed out on a precision balance and evaporated in a flask with a known volume. As the concentration in the flask has to be diluted, different volumes, in the order of a few milliliters, is taken out with syringes and injected into several other flasks. With a syringe samples are taken from the flasks and injected into a carrier gas stream flowing through a tube. The tubes are analysed and compared to tubes loaded from the tank. As before a basis is provided for determination of the concentrations in the tank and for calculations of the coefficients to the polynomial equations. Figure 7 is showing the analysis results.

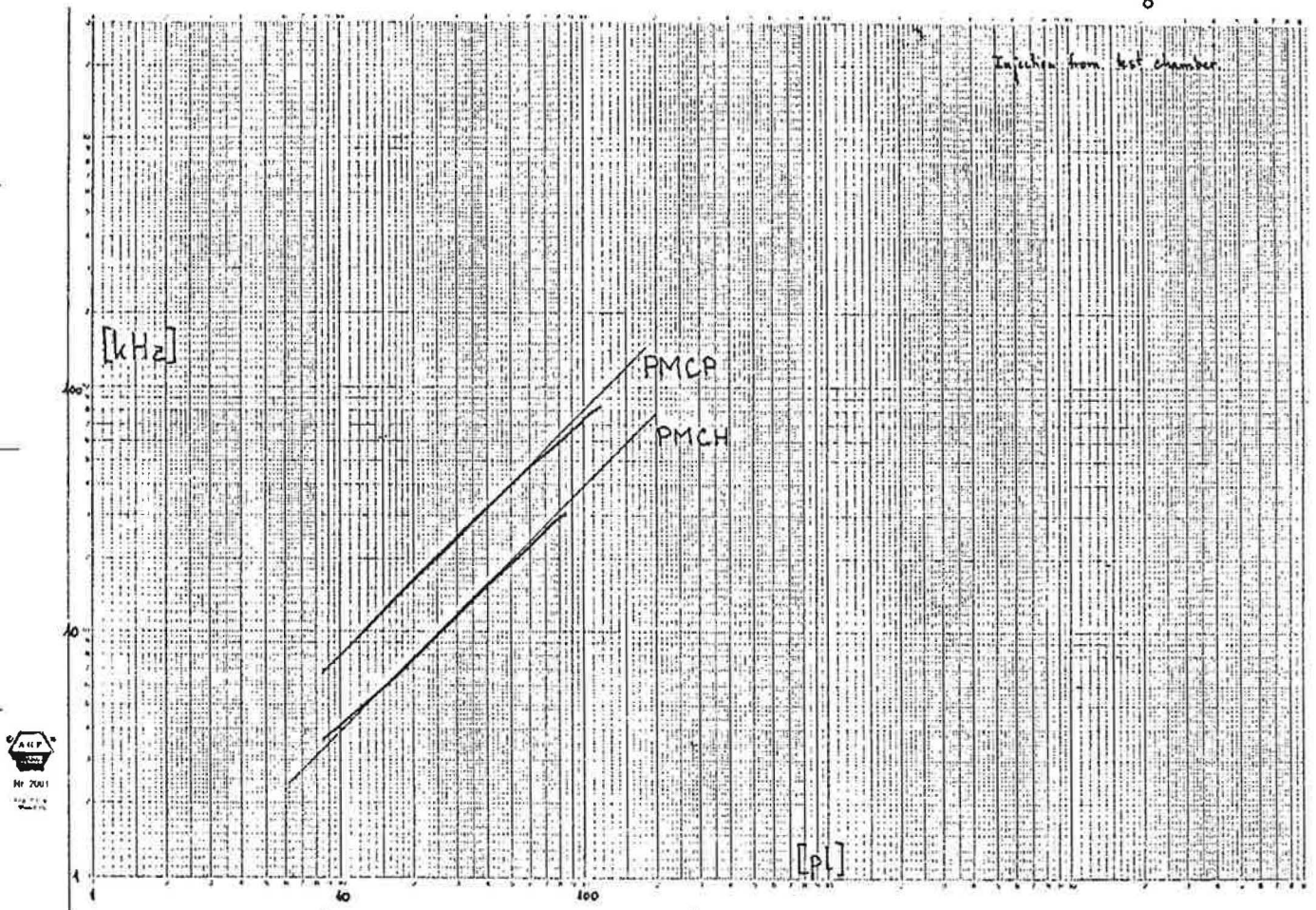


Figure 6. Injection of samples from test chamber.

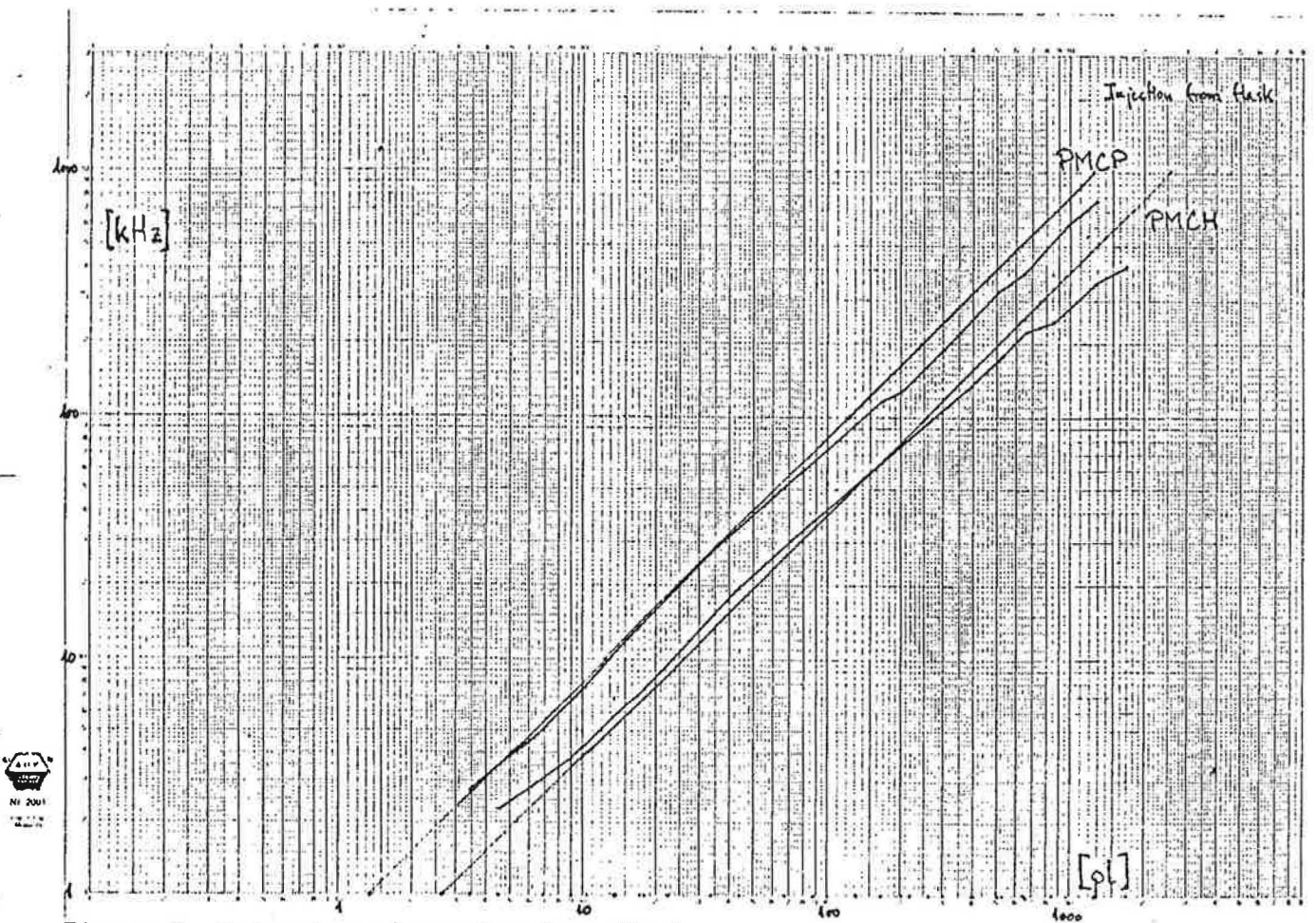


Figure 7. Injection of samples from flasks.

3. Final remarks

Calibration by routine of the system is based on loading a number of tubes with gas from a tank, containing a mixture of the tracers in use, and subsequent analysis of the tubes. The loaded volume is equal to the exposure time multiplied by the gas flow multiplied by the concentration of the particular tracer in the tank. The determination of the concentration of each of the tracers in the tank is done through the above described methods. Therefore, calibration of the system is not only influenced by uncertainties inherent in the principals of gaschromatographic analysis, but also by the uncertainties inherent in the methods used for determination of the concentrations in the tank.

If calibrating the system on the basis of concentrations in the tank, determined by injections of samples from a test chamber, uncertainty of emission rates of the sources, uncertainty of the temperature in the chamber, i.e. temperature influence of the emission rates, and uncertainty of the ventilation rate of the chamber have to be taken into account.

If the system is to be calibrated on the basis of concentrations in the tank, determined by injections from flasks, uncertainty of the weighing of the tracers in liquid form and uncertainty of the volumes of the flasks will have to be considered.

Generally, the uncertainty of the injected volume will have to be taken into account.

On the basis of the tests and the calibration procedures, The Danish Building Research Institute have established a reliable system with a good reproducibility.

10.5.1989

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den integrerande PFT-metoden****Listan om deltagare****Danmark**

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Hans Stymne	SIB, Gävle

SBI STATENS BYGGEFORSKNINGSINSTITUT
 Danish Building Research Institute

PFT-measurement

Building : Fictive building Date: 13.06.89
 Project : Martin Liddament
 Emitter file: FICTION.EMT Start: 14.02.89 at 21:00 GC analysis: 16.03.89
 Input file : FICTION.CAT End: 16.02.89 at 09:30 # Hours: 36.5

Results

Building Infiltration Rate: 85.6 m³/h, (5.5) [6%]
 Building Exchange Rate : 0.54 ACH (0.03)

Zone	Infiltration			Exfiltration			Total Flow		
	(m ³ /h)	SD	%SD	(m ³ /h)	SD	%SD	(m ³ /h)	SD	%SD
1	47.1	4.7	[10]	54.2	6.2	[11]	61.4	6.1	[10]
2	25.1	2.5	[10]	24.7	3.4	[14]	33.6	3.4	[10]
3	13.5	1.4	[10]	6.8	2.0	[29]	17.9	1.8	[10]

Interzone flow (m³/h)

From-To	Rate	SD	%SD
1 to 2	4.7	0.5	[10]
2 to 3	1.8	0.2	[10]
1 to 3	2.5	0.3	[10]

Interzone flow (m³/h)

From-To	Rate	SD	%SD
2 to 1	7.1	0.7	[10]
3 to 2	3.9	0.4	[10]
3 to 1	7.2	0.8	[10]

GC-analysis

Zone	Tube	Measured Volume (pl)		
		PMCP	PMCH	PDCH
1	1234	9.5	1.5	1.4
2	5678	1.5	11.5	1.4
3	9012	1.5	1.4	10.5
blk	3456	0.1	0.1	0.1

Average Zone Concentration (pl/l)

Zone	Type	PMCP		PMCH		PDCH	
		%SD	[]	%SD	[]	%SD	[]
1	PMCP	29.2	[0]	4.9	[0]	4.9	[0]
2	PMCH	4.6	[0]	37.6	[0]	4.9	[0]
3	PDCH	4.6	[0]	4.6	[0]	36.7	[0]

Zone and emitter data

Zone	ID	Volume(m ³)	Type	Number
1	Stue m.v.	80	PMCP	1
2	Soverum 1	50	PMCH	1
3	Soverum 2	30	PDCH	1

Emission Rate (nl/h). Ref temp: 25.0

Ref rate	Purity	Temp(C)	Est rate
1980	1.00	22.0	1727
1470	1.00	21.0	1225
798	1.00	20.0	635

Notes:

Uncertainty of GC = 10 % Uncertainty of mixing = 5 %
 Uncertainty of adsorbers = 2 % Uncertainty of emission rate = 10 %
 Rack factors: PMCP: 1.00 PMCH: 1.00 PDCH: 1.00
 Rack factors are not included in measured volumes.
 Uncertainty of conc. matrix = 0.006 Uncertainty of flow matrix = 0.093
 Condition nr. of conc. matrix = 1.07