

1976

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Gustafsson, V., "Determining the Air Content in Small Refrigeration Systems" (1976). *International Compressor Engineering Conference*. Paper 232.

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DETERMINING THE AIR CONTENT IN SMALL
REFRIGERATION SYSTEMS

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INTRODUCTION

For many reasons the presence of air in a refrigeration system is unfortunate. Not only does it reduce the capacity of the refrigeration system, but more important, it destroys the valve system of the compressor, since it promotes the formation of carbon i.e. during the oxidative break down of the refrigerator oil. In a hermetically sealed Freon refrigeration circuit using a compressor lubricated with mineral oil there exists - other things being equal - a simple correlation between air content and compressor life time.

It is therefore in the interests of both the compressor manufacturer and the appliance manufacturer to minimize the air content in a refrigeration system. To do this effectively, it is necessary to have a measuring procedure for determining the air content. Gas chromatographic analysis of refrigerants in connection with life tests on compressors and the materials used in their manufacture have been known and used for several years by compressor manufacturers, but so far this technique has not been applied to any great extent for routine control of air in finished refrigeration systems. We use gas chromatography for such measurements, and find it is a suitable method in connection with modern refrigeration system assembly line: It is quick and requires only little manpower. The equipment necessary is inexpensive to procure and easy to maintain. The technique is generally known and recognized.

This article describes how the absolute air quantity (N_2 , O_2 and Ar) in a refrigeration system is determined by first determining the vol % of NC-gas (by means of gas chromatography) in a small sample of the gas charge in the system and then, on the basis of this and knowledge of other system data, calculating the total air content. Finally, an error estimation is made for the method.

DEFINITIONS

In the following, references to refrigeration systems are taken as meaning complete units in domestic use:

Refrigerators
Deepfreezers
Air conditioning units
etc.

Described here is the way in which the NC-gas content in such complete systems is determined. The term NC-gas means N_2 nitrogen, O_2 oxygen and Ar Argon, and it is short for non condensable gas.

NC-GASSES IN REFRIGERATION SYSTEMS

As an introduction a resume of where the NC-gasses in a refrigeration system come from and what damage they produce is given. The description is a little brief but serves only to indicate the necessity of gas analysis.

In our opinion the most important source of failure is the evacuation of the assembled system before charging with Freon. In a refrigerant system the free gas volume typically amounts to 2-3 l, and the air content in this volume has to be removed. With plainly deficient or just deficient evacuation the complete system will contain air = NC-gas. But this can also be found in a well evacuated, finished refrigeration system partly that which is not evacuated from the oil partly that which is charged together with the refrigerant. As is well known oil can contain air and water which is difficult to remove (1), but these amounts are so small that they will hardly cause failure. If the refrigeration system is charged with Freon liquid direct from suppliers' tanks - via a charging board - any NC-gas which can enter the system will be very small. On the other hand it is our experience that

it is just these charging processes which are often critical, because of leaks, line-connections etc.

As a final source of failure we can consider how well the refrigeration system is sealed. A system which sooner or later develops leaks on the pressure side will lose its charge. If leaks appear on the suction side the compressor will periodically suck in air. Air diffusion due to partial pressure differences must also be borne in mind.

When the compressor is not running the air in the system will not cause damage. The oil and the other materials used are completely stable in air at room temperature.

When the refrigeration system is put into operation and the compressor begins to run the presence of air will produce two special effects:

The first will influence system capacity. The compressor and condenser are not efficiently utilized. The degree of influence will depend very much on how much of it there is in the system in relation to system dimensions and load. With a rising content of NC-gas, discharge pressure rises as well as the compression end temperature. An example of this is shown in fig. 1. If the NC-gasses are removed from the system it will operate as it was meant to. In other words the effect is reversible. The second effect may damage the valve function.

If the NC-gasses are allowed to remain in the system for long periods it will result in irreversible changes like thermal break down of the oil around the valves and oxidative break down because of oxygen in the NC-gasses.

This problem is probably by far the worst arising from NC-gasses in the system. It will show up mainly as leaky valves thereby reducing compressor capacity, increasing valve temperature etc.

Even though the system is evacuated and recharged the compressor will have been damaged to the extent where replacement is necessary.

GASANALYSIS IN GENERAL

To be able to control the evacuation and charging processes mentioned it is necessary to have a method for determining the total content of NC-gas.

The NC-gas content in refrigerants is traditionally determined by suitably freezing out or absorbing the refrigerant. Then the amount of gas residue can be determined by a pressure - volume measurement.

For taking these types of measurements there has over the years been developed special glass equipment, measuring procedures etc. (2,3,4).

Gas chromatography is an other way of making such determinations.

This method is a normal and reliable one for whole or part separations of gas mixtures in individual components. The more different these components are the easier it is to separate them one from another by gas chromatography.

In the case of NC-gas in Freon it is fairly easy to separate the NC-gasses from Freon, because the individual gas molecules are very different in size and polarity.

The principle of gas chromatography is as follows:

A small sample - one or more cm^3 - is placed on a column, which has been filled with a suitably porous material. A carrier gas flows constantly through the column carrying the sample with it. Each sort of molecules in the sample will move through the column at a different speed and separation will occur.

Each component of the sample is led by the carrier gas into a detector.

The most suitably is a hot Wire detector or similar, i.e. an electric bridge consisting of two platinum wires having the same electrical resistance, but each placed in its own chamber (= borings in a metal block). The hot wires form part of a wheatstone bridge circuit and are kept hot by the bridge current flow. When this is a constant the temperature of the wires (= the resistance) will be determined by the conductivity of the gas in the chambers i.e. carrier gas and carriergas + sample component. Since the carrier gas is chosen so that its conductivity is different to the gasses under analysis even small amounts of sample in carrier gas will change its conductivity resulting in a difference of voltage between the hot wires. This voltage difference is used as measuring reference and with the help of electronics the change is registered as a function of time on a normal recorder.

The detector signal is also sent to an integrator which convert it to digital form enabling a calculation to be made of the component concentration.

As will be seen, the sensitivity of such a detector varies partly with the material measured and partly with the amount of the material being measured. For this reason the detector must be calibrated.

This is done by analyzing a standard gas mixture, the composition of which must of course be known qualitatively and quantitatively. For example air can be used with a volume 10-100 times smaller than the volume used for the analysis itself.

For each of the components in the standard gas a factor is calculated corresponding to concentration divided by integrator output. This factor is an expression for the sensitivity of the detector to the component and concentration range concerned.

For years gas chromatography has been used

by compressor producers in connection with compressor life time tests and associated analyses of ageing processes of insulating materials, oils, etc. (5,6). However to the authors knowledge this technique has not been used within refrigerator production - perhaps because there are some difficulties in the sampling procedure.

REFRIGERATION SYSTEM CHARGE - GAS ANALYSIS

When analyzing the gas charge in a refrigeration system the task should be regarded as consisting of two operations:

1. sampling
2. gas chromatography

The results of the gas analysis will be influenced by the temperature and pressure in the refrigerator system at the time the gas sample is extracted. This is because Freon can be part gas, part liquid and partly absorbed in the oil. Conditions for NC-gasses are however a little different. They are not soluble in oil when their partial pressure are sufficiently low e.g. < 200 mm Hg and their temperature is sufficiently high e.g. > 20°F (1), and they do not condense of course.

Thus, the vol % of NC-gas found in a gas-analysis will depend on how much Freon in gas form there was at the time when the sample was taken.

To determine the total content of NC-gasses in a refrigeration system therefor requires analysis of the composition of the gasphase and determination of how large the gasphase is.

SAMPLING PROCEDURE

It is our experience that it is easiest to take samples of the gasphase when the refrigeration system is pressure and temperature equalized.

This is done, after the system has been in operation for the required time, by stopping the unit and opening the cabinet door. In our standard procedure a system is thus pressure- and temperature equalized in a minimum of 12 hrs.

The gassample is extracted and at the same time measurements are taken of equalized pressure and temperature. While this is being done a check must be made to see that the equalizing conditions are less than the saturation conditions of the Freon concerned. If this is the case the Freon will be found only as gas or absorbed in the oil. The NC-gasses will be equally distributed throughout the gas phase.

The amount of gassample taken must on the one hand be large enough to enable the gas chromatographic analysis to be made, and on the other hand be small enough not to di-

sturb the system.

If the gassample is transferred direct and a suitably thin, diffusion tight, clean tube is used the necessary amount of sample will be so small that under no circumstances will conditions in the system be influenced.

Therefore it is not necessary to re-evacuate and charge the system.

If the above procedure cannot be carried out the sample must be taken in a gasbomb. The size of the gasbomb and the procedure involved is however of some importance.

In our experience the most appropriate method is to connect the bomb to the system (e.g. to the compressor process connection) so that it becomes part of the system volume. In this way it is evacuated and charged in the same way as the rest of the system. When the sample is due to be taken the bomb is merely removed from the system and completely sealed. A suitably volume for the bomb will be 100-200 Ncm³.

There will always be a certain error when sampling by the bomb method. The magnitude of the error must be established by comparing the direct gasanalysis with analysis taken by bomb (both analyses on the same system).

SAMPLE CHROMATOGRAPHY

Chromatography is carried out by taking from the bomb or refrigeration system itself the amount of gas which is necessary to rinse and charge the supply-lines and the analysis loop in the chromatograph. From the analysis loop - which is calibrated accurately to contain one cm³ at 760 mm Hg and 20°C for example - the gassample is lead to the column by the carrier gas. In the column the separation occur as previously described.

By using the factor arrived at earlier the concentration of the components found can be calculated and recorded.

The special conditions we work with for such analysis can be seen in fig. 2.

As a result of the gasanalysis the content of NC-gas = PCNC is found from the sample taken.

As NC-gasses are only found in the gasphase (provided PCNC < ~5 vol %) the next stage is to determine the size of the gasphase in the system concerned.

DETERMINATION OF THE SIZE OF THE GASPHASE

The amount of Freon in the gasphase can be calculated as the difference between the total charge of Freon and the amount absorbed in the oil:

$$GR_{12}(\text{gas}) = GR_{12}(\text{total}) - GR_{12}(\text{oil}) \quad (1)$$

The amount of Freon absorbed in the oil at

the time of sampling can be calculated from an absorption diagram as shown on fig. 3. From this diagram the wgt % oil in the Freon + oil mixture under pressure and temperature equilibrium can be determined. The amount of Freon absorbed is calculated thus:

$$GR_{12}(\text{oil}) = \frac{100-W}{W} \cdot G_{oil}$$

and then the amount of Freon remaining in the gasphase is calculated:

$$GR_{12}(\text{gas}) = GR_{12}(\text{total}) - \frac{100-W}{W} \cdot G_{oil} \quad (2)$$

CALCULATION OF THE TOTAL AMOUNT OF NC-GAS IN A REFRIGERATION SYSTEM

How much vol % NC-gas there was in the gas-phase of the system was determined by the gaschromatographic analysis of a sample of the charge, i.e.:

$$PCNC = \frac{NC}{GR_{12}(\text{gas}) \cdot VR_{12} + NC} \cdot 100 \quad (3)$$

This equation expresses that the measured vol % NC-gas corresponds to the proportion between the total amount of NC-gas and the amount of Freon in the gasphase + the amount of NC-gas, under standard conditions. From this we obtain the total amount of NC-gas as:

$$NC = \frac{GR_{12}(\text{gas}) \cdot VR_{12} \cdot PCNC}{100 - PCNC} \quad (3a)$$

By using the obtained expression for $GR_{12}(\text{gas})$ eq. 3a becomes:

$$NC = \frac{[GR_{12} \div (\frac{100-W}{W}) \cdot G_{oil}] \cdot VR_{12} \cdot PCNC}{100 \div PCNC} \quad (3b)$$

and the total amount of NC-gas is determined.

ESTIMATED ERROR ON NC-GAS DETERMINATION

As can be seen from eq. 3b, the absolute NC-gas content is a function of the following 5 parameters:

$$NC = f(GR_{12}(\text{total}), W, G_{oil}, VR_{12}, PCNC)$$

If the root of least squares principle is used to determine the absolute error of NC it becomes this:

$$\sigma_{NC} = \sqrt{\left(\frac{\partial NC}{\partial GR_{12}} \cdot S_{GR_{12}}\right)^2 + \left(\frac{\partial NC}{\partial W} \cdot S_W\right)^2 + \left(\frac{\partial NC}{\partial G_{oil}} \cdot S_{G_{oil}}\right)^2 + \left(\frac{\partial NC}{\partial VR_{12}} \cdot S_{VR_{12}}\right)^2 + \left(\frac{\partial NC}{\partial PCNC} \cdot S_{PCNC}\right)^2} \quad (4)$$

Here, σ_{NC} is the absolute error of NC, while $S(GR_{12})$, etc. are the absolute errors

of the individual measurements. The partial differentials are obtained by the differentiation of eq. 3b:

$$\frac{\partial NC}{\partial GR_{12}} = \frac{VR_{12} \cdot PCNC}{100 - PCNC}$$

$$\frac{\partial NC}{\partial W} = \frac{100 \cdot G_{oil} \cdot VR_{12} \cdot PCNC}{(100 - PCNC) \cdot W^2}$$

$$\frac{\partial NC}{\partial G_{oil}} = \frac{VR_{12} \cdot PCNC \cdot (100 - W)}{(100 \div PCNC) \cdot W}$$

$$\frac{\partial NC}{\partial VR_{12}} = \frac{[GR_{12} \div \frac{100-W}{W} \cdot G_{oil}] \cdot PCNC}{100 - PCNC}$$

$$\frac{\partial NC}{\partial PCNC} = \frac{[GR_{12} \div \frac{100-W}{W} \cdot G_{oil}] \cdot VR_{12} \cdot 100}{(100 \div PCNC)^2}$$

EXAMPLE OF CALCULATION

A refrigeration system has the following charges:

185 g Freon 12
365 g oil

Equalizing pressure and temperature are measured as 3.7 kp/cm² and 20°C.

From fig.3., the oil-Freon phase contains 70 wgt %, i.e. $W = 70$.

A gassample is taken containing 2.0 vol % NC-gas at 1 atm. and 20°C, i.e. $PCNC = 2.0$. The specific volume of Freon 12 is 0.20 l/g (see ref. 7) at 1 atm. and 20°C. i.e. $VR_{12} = 0.201$.

Then the absolut amount of NC-gas can be determined by eq. 3b:

$$NC = 0.126 \text{ l}$$

The absolute error on this value can also be determined, using the following factors:

$GR_{12} = 185 \text{ g}$ $S(GR_{12}) = \pm 5 \text{ g}$
 $W = 70 \text{ wgt \%}$ $S(W) = \pm 2 \text{ wgt \%}$
 $G_{oil} = 365 \text{ g}$ $S(G_{oil}) = \pm 5 \text{ g}$
 $VR_{12} = 0.201 \text{ l/g}$ $S(VR_{12}) = \pm 0.001 \text{ l/g}$
 $PCNC = 2.0 \text{ vol \%}$ $S(PCNC) = \pm 0.05 \text{ vol \%}$

$P_{\text{equa.}} = 3.7 \text{ kp/cm}^2 \pm 0.1 \text{ kp/cm}^2$
 $T_{\text{equa.}} = 20^\circ\text{C} \pm 0.5^\circ\text{C}$

The partial differentials are calculated to:

$$\frac{\partial NC}{\partial GR_{12}} \cdot S_{GR_{12}} = 0.0204 \text{ l}$$

$$\frac{\partial NC}{\partial W} \cdot S_W = 0.0608 \text{ l}$$

$$\frac{\partial NC}{\partial G_{oil}} \cdot S_{G_{oil}} = 0.0009 \text{ l}$$

$$\frac{\partial NC}{\partial VR_{12}} \cdot S_{VR_{12}} = 0.0006 \text{ l}$$

$$\frac{\partial NC}{\partial PCNC} \cdot S_{PCNC} = 0.0030 \text{ l}$$

The absolute error is calculated according to eq. 4:

$$\sigma_{NC} = 0.065 \text{ l}$$

The relative error is determined:

$$\sigma_{NC}(\text{relativ}) = \frac{\sigma_{NC}}{NC} \cdot 100 = \sim 50 \%$$

Apart from the estimated error determination showing how large the error really is it also points out which of the individual measurements produce the largest effect. In this case it turns out that the weakest point is the determination of the amount of Freon absorbed in the oil, and this result must be considered as quite reasonably.

CONCLUSION

Overall, the following operations are necessary (after the routine has been established) to determine the total content of NC-gas in a refrigerator system with the use of gas chromatography:

1. Run the system in
2. Allow pressure and temperature to equalize 12 hrs. min. with a open cabinet door
3. take a sample of the gasphase
4. At the same time measure equalizing pressure and temperature
5. Determine the content of NC-gas in the sample with a gas chromatograph
6. Calculate size of gasphase
7. Calculate total amount of NC-gas in the system

We find that this procedure offers a series of advantages which make it attractive to producers of units:

1. The analyses are reproducible - although it should be pointed out that each time a gassample is taken the total amount of NC-gas in the system becomes reduced.
2. The estimated error on the analyses can be easily determined. Calculations have shown that it is of a reasonably magnitude.
3. Analyzing time is very short 5-15 min. At the same time need for manual efforts is very small.
4. The refrigeration system under analysis need not be removed from the production line.

There is a disadvantage. Knowledge of gas chromatography is a necessity and gas chromatographic equipment must be made available.

This disadvantage must not however be exaggerated, gas chromatography is a very widespread analyzing tool and the equipment in the simple form necessary to carry out the measurements we have been talking about is

not frighteningly expensive.

NOMENCLATURE

NC (l)	= Total volume of NC-gas in a refrigerator system
PCNC (vol %)	= The vol % NC-gas in a sample of the gasphase at standard conditions
GR12(total)	= Total Freon charge
GR12(gas) (g)	= Amount of Freon in the gasphase
Goil (g)	= Amount of oil charged the compressor
W (wgt %)	= Wgt % oil in the Freon - oil phase
Pequa. (kp/cm)	= Equalized pressure
Tequa. (°C)	= Equalized temperature
VR12 (l/g)	= Specific volume of Freon at gas analysis conditions
σ_{NC} (l)	= Estimated error for NC

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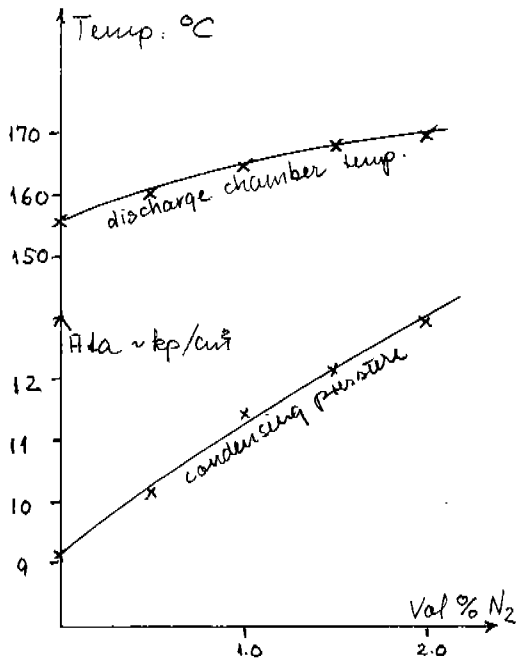


fig 1.

ANALYSIS DETAILS:

Gas chromatograph : 5830A Hewlett Packard
 Column : 2 m 1/8" steel tube
 filled with Porepak Q
 Oven temperature : 120°C
 Detector : Hot wire 200°C
 Injection temp. : 200°C
 Carrier gas : He
 flow 30 ml/min
 pressure 4 kp/cm²
 Analysis loop : 0.5 cm³

fig. 2.

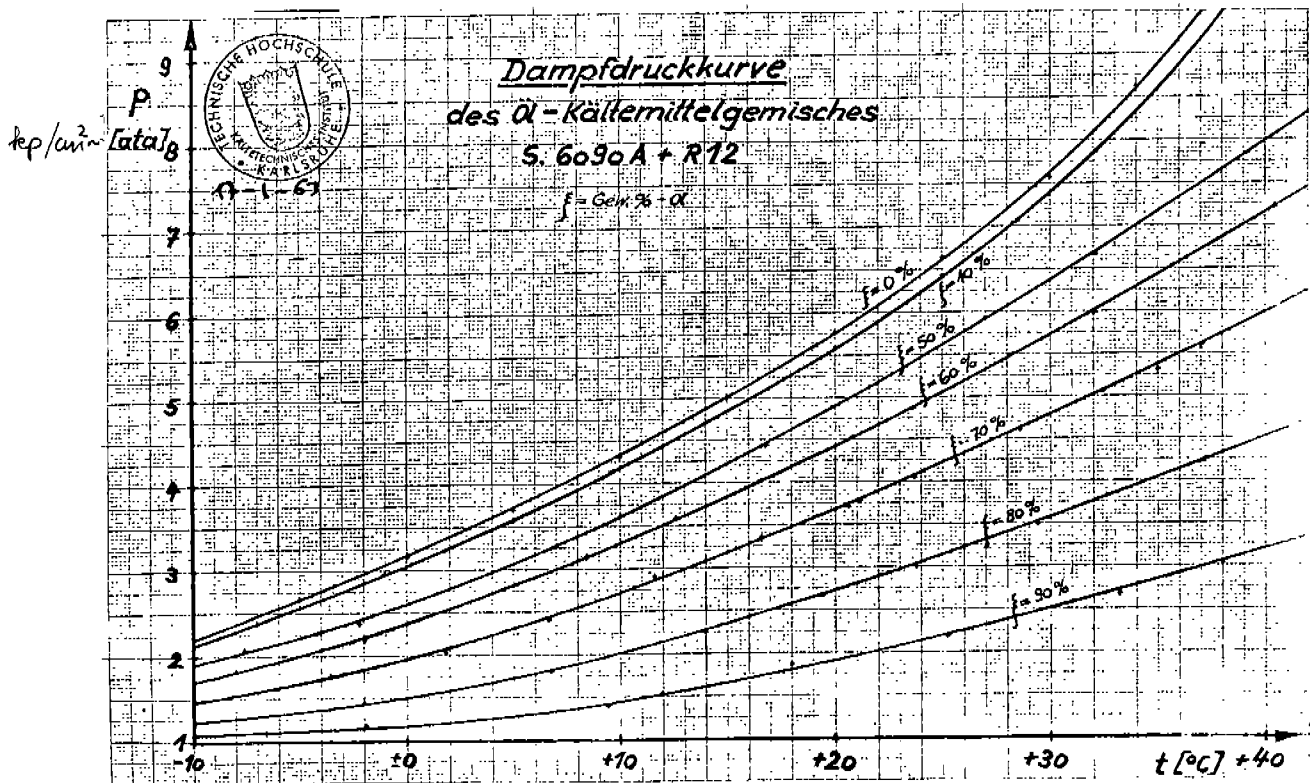


fig. 3