

OWLSTONE NANOTECH INC

Generating Calibration Gas Standards with OVG-4 and Permeation Tubes

Owlstone Whitepaper

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Permeation tubes enable the generation of NIST traceable, precise and repeatable calibration gas standards, over long periods of time, with concentrations ranging from part per trillion to high part per million. Permeation tubes are inherently safe compared to gas cylinders as they operate at low pressure and contain a small quantity of chemical. With over 500 analytes available you can generate standards even for applications that are problematic with cylinders, for example, corrosive gases and explosive / chemical warfare simulants.

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Excel Worksheet Downloads

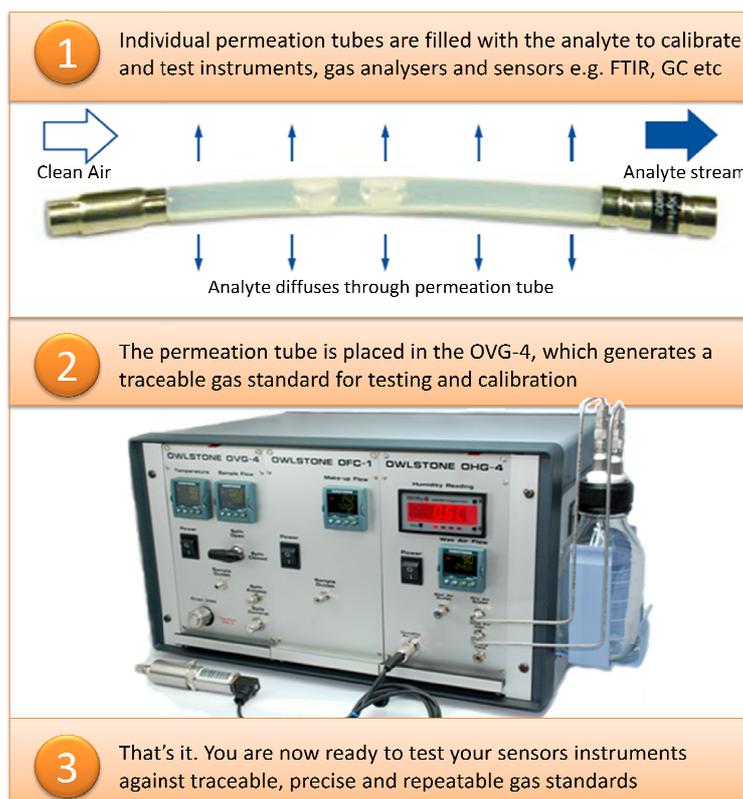
There are a range of excel spreadsheets available for download.

<http://www.box.net/ovg-excel>

Permeation Tube Fundamentals

What is a Permeation Tube?

A permeation tube is a polymer tube, typically PTFE, that contains a solid, liquid or gas analyte, sealed and crimped at both ends. The calibration chemical permeates through the walls of the tube at a constant rate for a given temperature; it then mixes with, and is carried away by a diluent, or make-up flow.



What Advantages do Permeation Tubes Provide over Gas Cylinders?

- ✓ All the Calibration Gases you need – with over 500 permeation tubes available you can create calibration gas standards for almost anything. Gas cylinders aren't suitable at all for some compounds e.g. explosive standards. They have problems with others; corrosive compounds tend to react with the internal sidewalls causing unknown changes in concentration over time.
- ✓ NIST Traceability – tubes are gravimetrically calibrated and are therefore traceable to primary standards. You'll have confidence that you are generating the concentration of calibration gas you need.
- ✓ Safety – permeation tubes contain a tiny quantity of chemical and operate at low pressure. Your Health and Safety manager will be happy that you won't need to buy high pressure, gas cylinders with high concentrations of dangerous gases to blend down to lower, useable concentrations.
- ✓ Lifetime and Stability – a tube can last up to several years in continuous operation; giving precise, stable and repeatable concentrations the whole time.

Calculating Concentration from Permeation Rate and Gas Flow Rate

The relationship between permeation rate of a tube and concentration is given by the following equation

$$q_d = \frac{CQ}{\left(\frac{22.4}{M}\right)} \text{ where}$$

- q_d is the permeation rate (ng/min)
- C is concentration (ppm)
- Q is flow rate (mL/min)
- M is molecular weight (g/mol)

For example to generate a 1ppm concentration of DMMP (Sarin Gas Warfare Simulant) at a sample flow rate of 50mL/min would require a permeation rate of

$$q_d = \frac{(1)(50)}{\left(\frac{22.4}{124.08}\right)} = 277 \text{ ng/min}$$

Fundamental Diffusion Equations and Calculation of Tube Permeation Rate

The permeation of gas through polymeric materials is a diffusion process due to concentration gradient between the inner and outer tube surfaces. Diffusion is defined by Fick's law; the diffusive flux goes from regions of high concentration to regions of low concentration, with a magnitude that is proportional to the concentration gradient.

$$(1) J = -D\nabla\phi = -D \frac{\delta\phi}{\delta x}$$

For a permeation tube Fick's law is expressed as

$$(2) q_d = \frac{DSA(P_1 - P_2)}{L} = \frac{P_G A(P_1 - P_2)}{L} \text{ where}$$

- q_d is the amount diffusing through polymer per unit area per unit time
- D is diffusion constant
- S is the solubility constant of the gas in the polymer
- A is the polymer area
- $P_{1,2}$ are the pressures on each side of the tube
- L is the tube thickness
- P_G is the gas permeability constant

Once equilibrated and assuming tubular geometry and negligible external pressure P_2 equation 2 reduces to

$$(3) q_d = \frac{730P_G M P_1}{\log\left(\frac{d_2}{d_1}\right)} \text{ where}$$

- q_d is the permeation rate (mg/cm tube length)
- P_G is permeation constant (cm²/sec)

- d_2 is outside diameter (mm)
- d_1 is inside diameter (mm)

The permeation constant is a function of the temperature, gas characteristics and tube properties

$$(4) P_G = P_G^o e^{-E_p/RT} \quad \text{where}$$

- P_G^o is a constant
- E_p is permeation tube activation energy
- R is molar gas constant
- T is absolute temperature

The only variable that changes in operation is the temperature. However it is usually the case that the various constants are not known a priori, which makes it difficult to calculate a desired permeation rate before building and calibrating a tube.

Permeation Rate as a Function of Temperature

The dependence of permeation rate as a function of temperature is derived from equation 4 and is given by

$$(5) \log \left(\frac{q_{d2}}{q_{d1}} \right) = \left(\frac{E_p}{2.303R} \right) \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad \text{where}$$

- $q_{d1,2}$ are the permeation rates at the two temperatures (mg/cm)
- $T_{1,2}$ are the temperatures ($^{\circ}\text{K}$)
- E_p is the activation energy
- R is the molar gas constant (1.99 cal/g-mol- $^{\circ}\text{K}$)

If the permeation rate at one temperature is determined through calibration we can calculate the permeation rate at the new temperature with the empirical relationship derived from equation 5

$$(6) \log q_{d1} = \log q_{d2} - 2950 \left(\frac{1}{T_1} - \frac{1}{T_2} \right)$$

This equation is typically accurate to +/- 5% for a 10 $^{\circ}\text{C}$ change in temperature.



For every 1 $^{\circ}\text{C}$ change in temperature the permeation rate will change by 10%. The OVG-4 Calibration Gas Generator has precision control of the incubation oven to 0.1 $^{\circ}\text{C}$ to give an accuracy of 1% on permeation rate.

Worked Example of Changing Permeation Rate with Temperature

Permeation rate 200ng/min at 25 $^{\circ}\text{C}$ or 298 $^{\circ}\text{K}$. New Temperature 40 $^{\circ}\text{C}$ or 313 $^{\circ}\text{K}$

$$\log q_{d1} = \log 200 - 2950 \left(\frac{1}{313} - \frac{1}{298} \right)$$

$$\log q_{d1} = 2.7754$$

$$q_{d1} = 596 \text{ ng/min}$$

Calculating the Lifetime of a Permeation Tube

The permeation tube lifetime of a standard tube with wall thickness of 0.062 and 0.125in is given by

$$L_s = \frac{1465\rho}{q_d} \quad \text{where}$$

- L_s is service lifetime (months)
- ρ is density (g/mL)
- q_d is permeation rate (ng/min/cm)



When permeation tubes are not in use store them in a refrigerator to significantly increase the lifetime of the tubes.

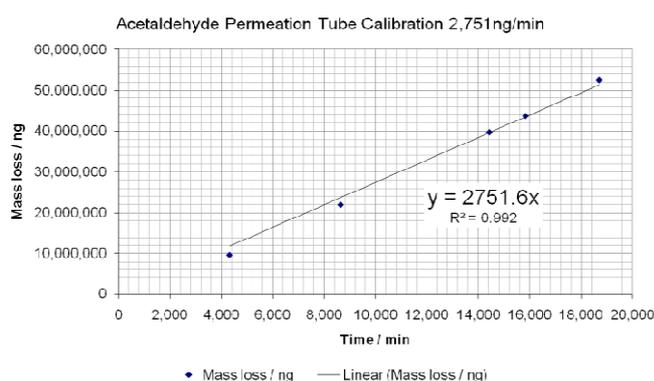
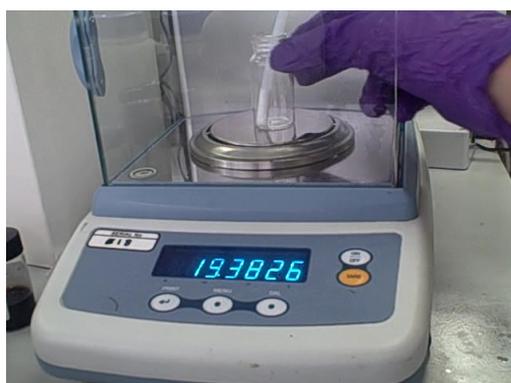
Worked Example Lifetime of Acetaldehyde Permeation Tube

For example an acetaldehyde tube with a permeation rate of 45ng/min/cm and density 0.861g/mL has a service life of

$$L_s = \frac{1465(0.861)}{45} = 28 \text{ months}$$

Calibration of Permeation Tubes

Permeation tubes are typically calibrated gravimetrically i.e. the mass loss in ng/min is directly measured over time using a mass balance. The calibrated permeation rates are certified and NIST traceable. The time it takes to perform a calibration depends on the permeation rate; with high permeation rates e.g. 1,000's ng/min there will be significant mass loss over a period of a few weeks to make an accurate calibration. For lower permeation rates e.g. 10's ng/min it can take several months to see enough measurable mass loss to make an accurate determination of permeation rate. Alternate calibration methods such a GC/MS can be used to shorten this time period at the expense of additional complexity.



Using the OVG-4 to Generate Calibration Gases with Permeation Tubes

To generate a calibration gas with a permeation tube you need to incubate the tube at a very stable and accurate temperature. A flow is then passed over the tube to generate the sample calibration gas. Owlstone has developed the OVG-4 Calibration Gas Generator to meet all your permeation tube calibration gas requirements.

BENEFITS

- High number of available analyte compounds, including solids and liquids as well as gases
- Easy generation of multi-component mixtures using combinations of tubes
- Cost savings by elimination of multiple expensive gas cylinders
- Reduced risk of exposure to dangerous chemicals due to small quantities used
- Fast and easy sample replacement
- Elimination of hazards associated with high pressure cylinders
- Quick and easy to set up and generate blended gas mixtures
- Adjustable concentration levels from ppm to ppb
- High accuracy and precision, even at the lowest concentrations
- Superior long term stability and repeatability*
- Portable, with compact footprint
- Easily integrated with the Owlstone Humidity Generator (OHG) for realistic environmental testing

**Owlstone offers an optional service for regular validation and instrument calibration*

The Owlstone OVG-4 is a system for generating trace concentration levels of chemicals and calibration gas standards. It is easy to use, cost-effective and compact and produces a very pure, accurate and repeatable output.

The very precise control of concentration levels is achieved using permeation tube technology, eliminating the need for multiple gas cylinders and thus reducing costs, saving space and removing a safety hazard. Complex gas mixtures can be accurately generated through the use of multiple tubes.

The OVG-4 is an ideal tool for numerous applications, ranging from calibration of explosive detectors in military and homeland defense to validation of personal monitors in industrial health and safety.

Current customers include – SELEX GALILEO, US Army, US Air Force, US Defense Threat Reduction Agency, Home Office Scientific Development Branch, DSTL, Commissariat à l'Énergie Atomique.



Adding Precision Humidity with the Owlstone OHG-4 Humidity Generator

The OVG-4 can be integrated with the OHG-4 Humidity Generator to create realistic test atmospheres to meet all your calibration and environmental testing requirements.



OVG-4 Calibration Gas Generator Frequently Asked Questions

What is the OVG-4 Calibration Gas Generator? The OVG-4 is an instrument that can generate accurate, precise and repeatable calibration gases for applications ranging from sensor / instrument test and calibration to generation of test atmospheres in corrosion chambers.

What is a permeation tube? A permeation tube is a small PTFE tube filled with the calibration analyte. When precisely heated the analyte diffuses through the tube at a controlled rate to generate an accurate calibration gas.

Does the OVG-4 generate traceable calibration gases? Yes. The permeation tubes are gravimetrically calibrated to NIST/UKAS traceable primary standards. Each tube comes with its own calibration certificate.

What and how many calibration gases can the OVG-4 generate? Generating new calibration gas standards is as easy as inserting a permeation tube into the OVG-4. You can view a [full list of permeation sources](#) and request pricing and availability. You can also [watch a video](#) of the OVG-4 in operation and see how easy it is to generate new calibration gas standards.

Can the OVG-4 provide mixtures of multiple calibration gases simultaneously? You can put multiple tubes into one oven to generate a gas mixture. If you want to control individual concentrations you can attach the outputs of multiple OVG units together to generate arbitrary calibration gas mixtures.

How do I calculate the calibration gas concentration generated by the OVG-4? Each permeation tube comes with a calibration certificate; these values are entered into the Owlstone OVG-4 concentration calculator software along with the instrument settings to obtain the concentration of gas. You can [launch the Calibration Gas Calculator](#) to see how you can easily change the calibration gas concentrations by adjusting temperature and flow on the OVG-4.

What is the lowest and highest calibration gas concentrations I can generate with the OVG-4? This depends on the permeation rate of the tube, the flow and the temperature. The OVG-4 can typically generate calibration gas concentrations from tens of part per trillion (ppt) levels up to tens of part per million (ppm). You can [launch the Calibration Gas Calculator](#) to see how to increase and decrease concentrations.

What sample flow rates are attainable with the OVG-4? The sample flow from the OVG-4 is controlled by a mass flow controller and can be adjusted from 50mL/min up to 3L/min. This can be further diluted with an arbitrary makeup flow to generate any total flow required for your application.

Can I further dilute the output of OVG-4 with a make-up flow? Yes, the output of the OVG can be blended with a make-up flow. The Owlstone OFC Flow Controller allows you to blend additional flows and the OHG Humidity Generator allows you to blend with humidified air for complete environmental testing.

Can I change the calibration concentration but keep the total flow the same? Yes. Owlstone has developed a unique split flow capability that allows you to change concentration while keeping total flow the same. This is very useful if you are testing / calibrating flow sensitive sensors or instruments.

How accurate and repeatable is the calibration gas generated by the OVG-4? You can achieve target calibration gas concentrations with a +/-5% error. The unit is extremely stable and repeatable over time; the sample flow is controlled with a mass flow controller and a large thermal mass incubation oven is adjusted to temperatures with an accuracy of +/- 0.1°C.

Can I control the OVG-4 through software? Yes, the temperature and flow can be controlled through software provided with the OVG-4. In addition there is an RS-485 port that allows you to integrate the OVG-4 into your custom test set-up (e.g. Labview) for fully automated control and integration.

Is the OVG-4 system modular? Can I add more units later? The system is fully modular, the OVG-4, OHG Humidity Generator and OFC Flow controller can easily be added or removed from the 19" GENSYS rack. You can get a single OVG-4 and the other two slots are blanked off. If you want to extend your calibration gas capability down the line additional units can easily be added to the empty slot.

What else do I need before I can use the OVG-4 Calibration Gas Generator? You can download the pre-installation guide to see what else you need to get started.

Do I need to heat the transfer line from the OVG-4 to the sensor / instrument under test? For low volatility compounds we would recommend using a simple wrap heater on the transfer line to avoid analyte condensation and memory effects.

How much training is required to use the OVG-4 Calibration Gas Generator? In short the OVG-4 is very straightforward to set-up and use. You can [download a copy of the user guide](#) to see for yourself.

What warranty is offered on the unit? One year limited warranty. We also offer extended warranty options. You can download our terms and conditions.

What servicing is required and when? We recommend that the mass flow controllers are recalibrated once a year.

Is the OVG-4 CE certified? Yes, the instrument is fully CE marked and certified to ISTA 3A.

Where can I view the full OVG-4 technical specification? You can view a [html version](#) and [download a pdf](#) version. You might also want to take a look at the [user guide](#).

Appendix A: List of Permeation Sources

List of Permeation Tubes				
1 - Propanol	2-(Ethylamino)ethanol	a - Pinene	Butyl cellosolve	Diethyl phosphite
1,1,3-Trimethoxypropane	2,3,5-Trimethylnaphthalene	Acenaphthalene	Butyl chloride	Diethyl sulfate
1,2-Propyleneimine	2,3,5-Trimethylphenol	Acetaldehyde	Butyl isocyanate	Diethylamine
1,2,4-Trichlorobenzene	2,2-Dimethylbutane	Acetamide	Butylamine	Diethyldimethylphosphoramidate
1,2,3-Trimethylbenzene	2,3-Dimethyl-2-butene	Acetic acid	B-utylbenzene	Diethylenetriamine
1,2,4-Trimethylbenzene	2,3-Dimethylphenol	Acetone	Butyraldehyde	Diethylethanolamine
1,1,1 tetrafluoroethane	2,4_Dinitrotoluene	Acetonitrile	Butyric acid	Diethylmethylphosphonothioate
1,1,1-Chlorodifluoroethane	2,4-Diaminotoluene	Acetophenone	Butyronitrile	Diisopropyl methylphosphonate
1,1,1-Dichlorofluoroethane	2,4-Lutidine	Acetylene	Camphene	Diisopropylamine
1,1,2,2-Tetrachloroethane	2,6-Diisopropylphenol	Acrolein dimethyl acetal	Carbon dioxide	Dimethoxymethane
1,1,2-Trichloroethane	2,6-Disopropylphenyl isocyanate	Acrolein	Carbon disulfide	Dimethyl disulfide
1,1,3,3-Tetramethyldisiloxane	2,6-Toluene diisocyanate	Acrylamide	Carbon monoxide	Dimethyl ether
1,1-Dichloroethane	2-Aminoacetophenone	Acrylic acid	Carbon tetrachloride	Dimethyl ethoxysilane
1,1-Dimethylhydrazine	2-Bromopropane	Acrylonitrile	Carbonyl fluoride	Dimethyl formamide
1,2 - Propanediol	2-Butanone	Acrylonitrile-d3	Carbonyl sulfide	Dimethyl mercury
1,2 Epoxybutane	2-Butyl mercaptan	Allene	Catechol	Dimethyl methylphosphonate
1,2,3-Trichloropropane	2-Chlorethyl methyl sulfide	Allyl chloride	Cellosolve acetate	Dimethyl phosphite
1,2-Butadiene	2-Chloroethanol	Allyl isothiocyanate	Cellosolve	Dimethyl phthalate
1,2-Dichlorobenzene	2-Chloroethyl ethylsulfide	Ammonia	Chlorine	Dimethyl sulfate
1,2-Dichloroethane	2-Chloroethyl phenyl sulfide	Amyl acetate	Chlorobenzene	Dimethyl sulfide
1,2-Dichloropropane	2-Chlorophenol	Amyl alcohol	Chlorobenzene-d5	Dimethyl sulfone
1,2-Diethybenzene	2-Chloropropane	Aniline	Chlorodibromomethane	Dimethyl sulfoxide
1,2-Difluorobenzene	2-Ethylhexanol	Anthracene	Chloroform	Dimethyl trisulfide
1,2-Dimethoxyethane	2-Ethylhexyl acrylate	Argon	Chloriodomethane	Dimethylacetamide
1,2-Dimethylnaphthalene	2-Ethylimidazole	Arsenic trichloride	Chloromethyl methyl ether	Dimethylamine
1,2-Ethanedithiol	2-Ethyltoluene	Arsine	Chloropicrin	Dimethylethanolamine
1,3-Butadiene	2-Heptanone	Benzaldehyde	cis-2-Butene	Dimethylsilane
1,3-Dichlorobenzene	2-Hexanone	Benzene	Crotonaldehyde	Dioxane
1,4 Thioxane	2-Methoxybutane	Benzene-d6	cs-1,2-Dichloroethylene	Dipropylene glycol methyl ether
1,4-Diaminocyclohexane	2-Methoxyethyl ether	Benzofuran	cs-1,3-Dichloropropene	Disilane
1,4-Dichloro-2-butene	2-Methyl pentane	Benzoic acid	Cumene	Disopropyl fluorophosphate
1,4-Dichlorobenzene	2-Methyl pyrazine	Benzyl alcohol	Cyclohexane	d-Limonene
1,4-Difluorobenzene	2-Methyl-1-butanol	Benzyl chloride	Cyclohexanol	Dodecafluorocyclohexane
1,6-Dichlorohexane	2-Methylbutanal	Benzyl mercaptan	Cyclohexanone	Dodecane
1-Bromo-4-fluorobenzene	2-Methylnaphthalene	Bibenzyl	Cyclopentane	Dowtherm
1-Bromobutane	2-Nitrotoluene	Bicyclo (2.2.1) hepta-2.5-diene	Cyclopropanecarboxaldehyde	Epichlorohydrin
1-Bromopropane	2-Pentanone	Biphenyl	Decane	Ethane
1-Butene	3-(Methylthio)propionaldehyde	bis-2-Chloroethylether	Deuterium oxide	Ethanol
1-Chloro-2-methylpropane	3,5-Difluoroaniline	Boron trichloride	Diacetone alcohol	Ethyl acetate
1-Methoxy propanol acetate	3-Buten-2-ol	Boron trifluoride	Dibutyl disulfide	Ethyl acetylene
1-Methoxy-2-propanol	3-Carene	B-Pinene	Dibutyl sulfide	Ethyl acrylate
1-Methoxy-2-propyl acetate	3-Cyanopyridine	Bromine	Dichlorosilane	Ethyl benzene
1-Methyl-2-propanol acetate	3-Ethyl pyridine	Bromochloromethane	Dichlorvos	Ethyl carbamate
1-Methylbutadiene	3-Methyl pyridine	Bromodichloromethane	Dicyclohexylmethylphosphonate	Ethyl chloride
1-Methylnaphthalene	3-Methyl valeric acid	Bromoform	Dicyclopentadiene	Ethyl disulfide
1-Pentene	3-Methyl-1-butanol	Bromonitromethane	Diethyl ether	Ethyl lactate
2-(2-Methoxyethoxy)ethanol	4 -Vinylpyridine	Butane	Diethyl ethylphosphonate	Ethyl mercaptan
2-(Diethylamino)ethanethiol	4-Aminobiphenyl	Butanol	Diethyl malonate	Ethyl methyl ether
2-(Disopropylaminc)ethanol	4-tert-Butyltoluene	Butyl acrylate	Diethyl methylphosphonate	Ethyl methyl sulfide
	4-Vinyl cyclohexane		Diethyl methylphosphonite	

List of Permeation Tubes			
Ethyl morpholine	Hydrogen sulfide	Methyl iodide	tert-Butanol
Ethyl pyrazine	Hydrogen	Methyl isobutyl ketone	tert-Butyl chloride
Ethyl sulfide	Indan	Methyl isocyanate	tert-Butyl ethyl ether
Ethylamine	Indene	Methyl isopropyl ketone	tert-Butyl mercaptan
Ethylbenzene-d10	Iodine	Methyl isothiocyanate	tert-Butyl methyl ether
Ethylbenzene-d10	Iodoethane	Methyl mercaptan	Tetrachloroethylene
Ethyl-diethanolamine	Isoamyl acetate	Methyl methacrylate	Tetradecane
Ethylene	Isobutane	Methyl phosphonic dichloride	Tetraethyl orthosilicate
Ethylene dibromide	Isobutanol	Methyl phosphonic difluoride	Tetrahydrobenzaldehyde
Ethylene glycol	Isobutyl acetate	Methyl salicylate	Tetrahydrofuran
Ethylene oxide	Isobutyl acrylate	Methyl vinyl ketone	Tetrahydrofurfuryl alcohol
Ethylenediamine	Isobutyl chloroformate	Methylamine	Tetrahydrothiophene
Ethyleneimine	Isobutyl mercaptan	Methylaminoethanol	Tetramethylsilane
Fluorobenzene	Isobutylbenzene	Methyldiethanolamine	Thiodiglycol
Formaldehyde *	Isobutylene	Methylene bromide	Thionyl chloride
Formamide	Isobutyraldehyde	Methylene chloride	Thiophene
Formic acid	Isooctane	Monoethanolamine	Thiophosphoryl chloride
Freon 11	Isopentane	Morpholine	Toluene -2,4-diisocyanate
Freon 113	Isoprene	m-Tolualdehyde	Toluene
Freon 116	Isopropanol	n - Propyl acetate	Toluene-dp
Freon 12	Isopropyl acetate	n - Propyl mercaptan	tr-1,3-Dichloropropene
Freon 13	Isopropyl ether	N,N-Dimethylaniline	trans-Butene
Freon 21	Isopropyl mercaptan	n-Amyl acetate	Tributylamine
Freon 22	Isovaleraldehyde	Naphthalene	Trichloroethylene
Freon 114	Krypton	n-Butanol	Trichlorosilane
Furan	m - Xylene	n-Butyl acetate	Tridecane
Furfural	Malathion	n-Butyl mercaptan	Triethyl phosphite
g-Butyrolactone	Maleic anhydride	Neon	Triethylamine
Germane	m-Cresol	n-Hexene	Triethylenetetramine
Glycerol	Menthol	Nicotine	Triethylphosphate
Helium	Mesitylene	Nitric acid	Trifluoroacetic acid
Heptane	Methacrolein	Nitric acid-d	Trimethyl phosphite
Heptanenitrile	Methacrylic acid	Nitric oxide	Trimethylamine
Heptyl cyanide	Methane	Nitric-15N-acid	Trimethylsiane
Hexachloro-1,3-butadiene	Methanesulfonylfluoride	Nitrobenzene	Undecane
Hexachlorobenzene	Methanol	Nitrogen	Vanillin
Hexadecane	Methyl acetate	Nitrogen dioxide	Vinyl acetate
Hexaldehyde	Methyl acetylene	Nitrogen trifluoride	Vinyl acetylene
Hexamethylcyclotrisiloxane	Methyl acrylate	Nitrous oxide	Vinyl bromide
Hexamethyldisilazane	Methyl amine	n-Methyl pyrrolidinone	Vinyl chloride
Hexamethyldisiloxane	Methyl benzoate	N-Nitrosodimethylamine	Vinyl fluoride
Hexamethylene-1,6-disocyanate	Methyl bromide	N-Nitrosomorpholine	Vinylcyclohexene monoxide
Hexane	Methyl cellosolve	n-Nonane	Vinylidene chloride
Hexanenitrile	Methyl cellosolve acetate	n-Octane	Water
Hexanoic acid	Methyl chloride	n-Propyl benzene	Xenon
Hexanol	Methyl chloroform	o-Toluidine	α-Methylstyrene
Hydrogen bromide	Methyl Chloroformate	o-Anisidine	α-Terpinene
Hydrogen chloride	Methyl cyclopentane	o-Cresol	ε-Caprolactam
Hydrogen cyanide	Methyl hydrazine	Octamethylcyclotetrasiloxane	
Hydrogen fluoride		Octyl cyanide	