Industrieverband
Halbzeuge und Konsumprodukte
aus Kunststoff e.V.

pro-K Fluoropolymergroup

Technical brochure 3.1

Application of PTFE-polymers in oxygen systems

Preface

Polytetrafluorethylene (PTFE) is a high performance polymer. Due to its unique properties PTFE has become an indispensable material in modern industrial society.
Among the outstanding properties of PTFE, the high chemical resistance, the broad service range, the excellent dielectric properties, the ageing resistance, the resistance to embrittlement and high purity have to be mentioned.

Due to their outstanding properties PTFE, modified PTFE and PTFE compounds are preferred materials in system engineering where complex regulations require high compatibility i.e. compliance with regulations for the contact with oxygen, food or potable water.
Apart from the selection of the appropriate and suitable materials, system and regulatory requirements in high-grade application have to be taken into account and to be considered when starting a new project. For this reason, the complex sets of rules are increasingly the focus of high-quality system engineering applications.

This brochure is addressed to all processors of PTFE and contains a lot of information about the use of PTFE in oxygen facilities.

This technical brochure is edited by the pro-K fluoropolymergroup and was elaborated by Mr. Stefan Ebmeyer, Dyneon GmbH & Co. KG in 2006. The present brochure was revised in collaboration with the "*BAM*" (Bundesanstalt für Materialforschung und- prüfung) and represents our knowledge as of October 2018.

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Industrieverband
Halbzeuge und Konsumprodukte
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1. PTFE and PTFE-compounds in oxygen systems

Due to their advantageous properties in contact with oxygen PTFE, modified PTFE and Compounds based on both are widely used as sealing materials in oxygen plants. Typical applications are sealing elements in fittings and pressure regulators as well flange connectors and screw joints.

Materials and components which are supposed to be used in oxygen plants have to be suitable from a safety point of view and are therefore subject to specific tests.

1.1 Oxygen in pressurised gas-systems

Oxygen itself is not flammable but fire promoting and may react heavily with flammable substances.
Normal air contains 21 % oxygen. An increase in oxygen concentration above 21 % significantly increases the fire hazard of flammable substances. Many materials that are not flammable under normal conditions react in an oxygen enriched atmosphere: this not only affects easily flammable oil and grease, rubber and polymers but also alumina, steel and brass may burn in an oxygen-enriched atmosphere.
This danger is present above all in facilities when compressed oxygen influxes intermittently in areas of low pressure and is adiabatically compressed. The resulting heat of compression may cause the ignition of flammable components. Flammable parts may be ignited more easily in oxygen-enriched conditions and burn faster and hotter. Fire spreads more quickly. Ignition sources that have no effect in ambient air may cause a fire in oxygen-enriched systems.
Three components are necessary to initiate a fire in oxygen containing facilities: oxygen, combustibles and an ignition source: The oxygen is enclosed in oxygen systems. Valves, pressure regulators, pipes, sealings, fittings and other components are combustible parts. The ignition power may come from the system e.g. by pressure surges or impact stress.in oxygen devices.
The fire hazard cannot be eliminated completely, but it can be reduced substantially by an appropriate risk management based on a diligent assessment of potential dangers and hazards. The entire system, the selection of materials, processing methods as well as operation and maintenance of the plant have to be developed specifically for each application.

1.2 Regulation under the regulatory framework „Technische Regeln für Gefahrstoffe TRGS 407" der" Bundesanstalt für Arbeitsschutz und Arbeitsmedizin" (BAuA)

Notes regarding oxygen are attached to the updated „BG-Merkblatt“ (leaflet) for Hazardous Substances M 034e "Oxygen” as Annex 1 " Betreiben von Sauerstoffanlagen" (Operation of oxygen systems) and as Annex 2 “Empfehlungen für die Beschaffenheit von Sauerstoffanlagen“ (Recommendations for the Quality of Oxygen Plants). These leaflets are not legally binding. They can be used as source of information and serve to compile a risk assessment.
Reaction triggering processes for fires and explosions in oxygen plants are manifold. These are, for example, heat ignition at normal or exceptional operating temperatures, for example in dry-running reciprocating compressors; friction of machine parts, for example in turbo compressors and

centrifugal pumps; friction between system parts and foreign bodies, for example in pipelines, fittings, centrifugal pumps; thermodynamic phenomena in fast gas flows, for example in fittings at throttle points; pressure surges in pipelines, for example when fittings are opened quickly; impact energy, for example in system parts for liquid oxygen.
For the operational safety of oxygen plants, the greatest safety would be achieved if the operating conditions were defined in such a way that the above mentioned processes could not take effect. But this is often not possible.
If, e.g. the oxygen is not cooled after the last compressor stage for economic reasons but is still fed hot to the reactor, all seals in downstream fittings and in the flange connections of the pipelines are also exposed to the hot oxygen.

As a consequence of the examples listed above the TRGS 407 states in chapter 3.2.7 "Besondere Gefährdungen durch Tätigkeiten mit Sauerstoff" (Special hazards arising from operations involving oxygen) that the use of inappropriate materials may cause ignition and fire of several parts of the plant. Materials that are not tested at the relevant operating parameters (pressure and temperature) are considered inappropriate as well. Please refer to the table of attachment 1 DGUV Information 213-073 (Merkblatt M 034 - Oxygen (DGUV Information 213-073, former BGI 617)) of the" Berufsgenossenschaft Rohstoffe und chemische Industrie". According to this brochure only sealing materials and lubricants that are appropriate from a safety point-of-view can be used. Lubricants and sealing materials are suitable if they have been tested by an approved testing institute with the result that they are suitable for use at the respective pressure level, operating temperature and installation method in terms of safety.

In addition to the BG-brochure M 034-1 "Oxygen" a list of non-metallic materials is published annually as GB brochure M 034-1 by the „Bundesanstalt für Materialforschung und -prüfung (*BAM*)“ that has been found to be suitable for the use in oxygen devices (see chapter 1.5).

1.3 *BAM* - test procedures for sealing materials

Over the last six decads the „Bundesanstalt für Materialforschung und -prüfung (*BAM*),“ a test institute recognised by the „Berufsgenossenschaft“ has developed test methods and assessment standards for the safe operation of oxygen facilities as well as tests and assessments of non-metallic sealing materials and gaskets:

1. Ignition temperature in compressed oxygen

2. Resistance to ageing in compressed oxygen

3. Impact of oxygen pressure shocks

4. Reactivity with fluid oxygen under impact stress

5. Testing of flat gaskets for flange connections

These test procedures and evaluation methods allow to determine the safety-related permissible operating conditions for temperature and oxygen pressure for the respective material.
In the following, background information and physically relevant hints on the individual test procedures are given without going into detail on procedures and methods.

1.3.1 Ignition temperature in compressed oxygen

The ignition temperature as a safety parameter for heat ignition is defined as the lowest temperature at which the reaction of the combustible material with oxygen (oxidation) from its own energy development accelerates to such an extent that it takes place under the appearance of flames. As the speed of oxidation depends on the temperature and the concentration of the reaction partners, i.e. also on the pressure of the oxygen, as with any chemical reaction, the ignition temperature is also influenced by the oxygen pressure. An increase in oxygen pressure usually results in a decrease of the ignition temperature. Thus, the ignition temperature of a substance in oxygen at normal pressure can drop by more than 200 degrees in air at normal pressure and by a further 100 degrees in highly compressed oxygen.
The experimental set up chosen by *BAM* to determine the ignition temperature of non-metallic sealings may be taken from e.g. the technical publication “Hazards by Oxygen and its Application” or from "Tests to Evaluate the Suitability of Materials for Oxygen Service" (see list of soucres, Annex).

The technical paper cited above describes that the ignition temperatures determined in compressed oxygen in the range of 200 °C are reproducible with a deviation of +/- 5 °C; at higher ignition temperatures up to 500 °C the deviations are about +/-10 °C, in some cases even more. For each sample five tests are performed. Organic binders have relatively low ignition temperatures of 130 – 200 °C at 50 – 100 bar oxygen pressure.
Very high ignition temperatures - which in most cases are higher than the operating temperatures - are found for Fluorocarbon fluids (350 – 400 °C) and pure PTFE (450 – 470 °C) each at 50 – 130 bar oxygen pressure. Pure graphite, which has an ignition temperature of 500 °C at 130 bar oxygen pressure, behaves like an intermediate between organic compounds and heavy metals and their alloys respectively.

BAM limits the permissible operating temperature for the material to a value which is 100 °C lower than the ignition temperature at the respective oxygen pressure. This safety margin must be observed without fail, as it not only covers the risk of unforeseen accidental increases in operating temperature, but also takes into account the fact that the ignition temperature value is not a material-specific constant at a given oxygen pressure. This means that the value of the ignition temperature depends on the test method used which may differ from the conditions that prevail in the plant.
According to the technical document “Hazards by Oxygen and its Application” a safety margin of 100°C between the ignition temperature and the operating temperature has proven effective. Due to the specific test procedure a safety margin of only 50 °C is considered sufficient for flat gaskets in flange connections.

1.3.2 Resistance to ageing in compressed oxygen

If, when including the safety margin explained in the previous paragraph, a temperature should result at which the material is already subject to thermal decomposition or is not resistant to ageing in compressed oxygen (resistant to oxidation), the maximum permissible operating temperature must be set at an even lower value. For this purpose, the material is tested for resistance to ageing.
This test is performed in a gastight container, in which a weighed sample is exposed to compressed oxygen for 100 hours at a temperature which usually is 100° C lower than the determined ignition temperature. The filling pressure of oxygen at 20 °C depends on the intended operating pressure. During this artificial ageing process it is examined whether the sample reacts with oxygen or changes otherwise.

The criteria for resistance to ageing under the chosen experimental conditions are the following: maintaining of the appearance/external condition, of the mass of the sample and of the value of the ignition temperature after ageing. If the results of the test are not satisfactory, the tests are repeated at temperatures 25 °C lower iteratively until the results correspond to the values of the initial sample. In this way, the maximum acceptable operating temperature for the material is defined.

1.3.3 Impact of oxygen pressure shocks

A further criterion for assessing the reactivity of organic substances with oxygen is their behaviour under the effect of oxygen pressure shocks i.e. when low pressure oxygen is suddenly compressed to higher pressure. If such pressure surge occur so quickly that the process is adiabatic[1], considerable temperature increases result. According to Poisson's equation, for example, a temperature increase to 462 °C is calculated if oxygen of 20 °C is compressed from 1 bar to 25 bar. This temperature is higher than the ignition temperature of most organic materials. This means that sealings (e.g. gaskets) lubricants, hydraulic liquids etc. are ignited and can react explosively if they are exposed to such oxygen pressure shocks.
Pressure shocks happen in pipes for example when pressurized armatures are opened very quickly as well as in piston compressors. However, such compression processes are generally not adiabatic, which means that the resulting peak temperature lies between the initial temperature and the theoretically calculated maximum temperature.
The test set up designed by *BAM* for the determination of the maximum acceptable oxygen pressure at specific temperatures for non-metallic sealing materials can be taken from the technical paper “Hazards of oxygen and its application” or „Tests to Evaluate the Suitability of Materials for Oxygen Service“.

[1] Adiabatic is the term used to describe a process when a change in state of a gas prevents any heat absorption or release from the environment, i.e. the gas forms a closed system in which the amount of heat does not change (dQ = 0).

1.3.4 Reactivity with fluid oxygen under impact stress

The speed of heterogeneous combustion reactions is significantly influenced by the size of the reactive surface of the combustible substance. This means that a shredded or porous substance can burn much faster than the same substance in a compact form. If at the same time oxygen is present in a high concentration, e.g. by absorbing liquid oxygen in a crushed or porous material, the reaction may occur very fast and in case of an external ignition e.g. by a blasting cap even as a detonation.

Fires and explosions of organic substances in contact with liquid oxygen, can often be triggered by impact stress alone. In such cases impact loads of only a few Nm can be sufficient to trigger explosion-like reactions.
In the presence of liquid oxygen powders, chips and swarf of alumina, magnesium, silica, tin, titan and other alloys may be ignited by impact stress and react explosively.
PTFE, which is frequently used as sealing material, is rather insensitive against impact stress in liquid oxygen, as are several metals and alloys such as copper, tin bronze, nickel and nickel-chromium steels, which, even in finely powdered form, prove to be impact resistant after soaking with liquid oxygen.

The test arrangement and the procedure for testing the reactivity with liquid oxygen under impact stress can be found in the technical papers "Hazards due to oxygen and their prevention" or "Tests to Evaluate the Suitability of Materials for Oxygen Service" (see list of sources, annex). The test device essentially consists of a cup-shaped sample container made of copper sheet metal into which about 0.5 g of the comminuted test material is placed. The sample is poured with liquid oxygen and exposed to the impact stress of a drop hammer (mass 76.5 kg). The drop height of the hammer is variable.

A reaction of the sample with liquid oxygen is usually recognisable by the formation of a flame and a more or less violent explosion bang. By variation of the drop height the impact energy at which no reaction occurs is determined. This result has to be confirmed ten times under the same conditions. The tests are stopped if reactions are observed at an impact energy of 125 Nm or less (corresponding to a hammer drop height of 0.17 m or less). In this case, the material is considered unsuitable for liquid oxygen systems from a safety point of view.

1.3.5 Testing of flat gaskets for flange connections

Inner sealings of armatures, piston rings of compressors, friction bearings of test devices with rotating components, membranes of measuring instruments and pressure regulators have a more or less big contact surface caused by their construction and installation respectively. Under special circumstances this may become even worse when abrasion particles are deposed at places of low gas current. The situation is much better with flange sealing as they are not stressed dynamically. In first place they do not produce abrasion particles and in second place only a small area is exposed to oxygen. Furthermore the relative big mass of the metallic flanges serve as a heat conductor, thus preventing an ignition.

Therefore a special test procedure has been developed for this kind of sealing, which takes into account the favourable installation conditions described above.
Basically, the test device consists of two steel pipes, each 1m long with a nominal size of 65 mm and a nominal diameterof160mm respectively which are welded to standard flanges. The pipe sections including the test specimen are welded "gas-tight" to the flange. The gasket is big enough to protrude into the pipe.
The test device is heated from outside to a temperature which is at least 50 °C lower than the ignition temperature of the sealing. After closing the device it is filled up to the intended oxygen pressure and the part of the sealing that protrudes into the pipe is ignited by an electric glow wire.
The relevant criterion for the assessment of the sealing is its behaviour after ignition. If the gasket burns between the sealing surfaces of the flanged joint or if the fire is transferred to the steel (usually part of the test equipment is destroyed in the process), the gasket is considered unsuitable. If only those parts of the gasket protruding into the pipe burn but the fire is not transferred pipeline or the flanges and the gasket does not continue to burn between the flanges there are no safety concerns about using the gaskets up to the applied test pressure and specified temperature.

Oxygen pressure and test temperature are adjusted to each other until in five tests:

a) the flange connection does not become leaky during the tests due to softening or burning of the gasket between the flanges
b) the sealing does not keep burning between the flanges

Only if oxygen pressure and test temperature meet the requirements according to a) and b), the sealing is considered appropriate for these conditions.
The special feature of this test method, which consists of exposing the sealing under high oxygen pressure to a very hot flame, is that it allows to recognize that ignition hazards can be reduced to a great extent if the flammable sealing is mounted in an appropriate way.
The safety margin between the ignition temperature of the sealing material in compressed oxygen and the permissible operating temperature is reduced by *BAM* to 50 °C for this type of sealings.
Suitable materials with maximum permissible values for oxygen pressure and temperature can be found in leaflet M 034-1 in the "List of non-metallic materials" (chapter 2. "Dichtungsmaterialien/Gaskets").

1.3.6 Assessment of materials

The test methods described help to perform a safety related assessment of non-metallic sealing materials and gaskets for the use in oxygen devices, both for gaseous oxygen at normal and especially elevated pressure and for liquid oxygen.
It may happen that materials which according to their designation are regarded to be non-flammable must be tested as they may contain flammable components due to their processing. On the other hand it is not necessary to perform each individual test if the application of the material in question is limited to certain operating conditions. If, for example, the use is limited to oxygen at normal temperature (the temperature range up to 60 °C applies as such), the ignition temperature and ageing resistance are usually not determined.

The assessment of the materials to be derived from the sum of the test results has exclusively safety-related character. For example, information about the elasticity of the materials, their behaviour at very low temperatures, their abrasion resistance, hardness and other physical properties are not part of the test procedures described. These details are to be obtained from the manufacturer.

1.4 PTFE and PTFE-compounds for use in contact with oxygen

As described in chapter 1.3 components made of PTFE and PTFE compounds have proven as sealing materials and gaskets in oxygen devices for decades.
Non-metallic materials made of PTFE and PTFE compounds are tested for specific applications in oxygen devices by *BAM* according to the test methods described in chapter 1.3. Based on these results *BAM* derives a safety assessment of the tested materials and summarizes the results in a test report.
The table "PTFE and PTFE-compounds for the application in contact with oxygen" on page 10 provides an abstract about materials tested by *BAM* including the applied test methods which is not complete but gives a good overview. Further information on specific non-metallic materials made of PTFE and PTFE compounds can be obtained from PTFE respective raw material suppliers or processors. The tests commissioned by the raw material manufacturers to *BAM* are generally intended to provide purely orienting test results.It is important that the tests at the *BAM* always refer to a certain submitted test sample. Accordingly, the test results are only valid until the ingredients and formulation of the tested material change. If there are changes new test samples have to be submitted. In order to prevent damage caused by changed materials *BAM* recommendeds to repeat testing every 10 years even if the ingredients and formulations remain unchanged.

Therefore the list of non-metallic substances is published only for 10 years.
BAM tests are often test which have to be performed on a specific workpiece (article) and are usually initiated by the distributor.

PTFE and PTFE-Compounds for the application in contact with oxygen

The following products have been technically assessed by *BAM* as reference samples or semi-finished products (see also chapter 1.6 safety related assessment of semi-finished products and finished articles for the application in oxygen devices).

produkt - description	***BAM* – test method**					***BAM* – listing**
	1	**2**	**3**	**4**	**5**	
virgin PTFE (S-PTFE)	X	X	X	X		✓
virgin PTFE, modified (S-PTFE, modified)	X	X	X	X		✓
glass fibre-PTFE-compounds	X	X	X	X	X	✓
carbon-PTFE-compounds	X	X	X	X		✓
carbon-graphite-PTFE-Compounds	X	X	X	X		✓
glass fibre-graphite-compounds	X		X			✓
conductivity compound-PTFE	X	X	X	X		✓
graphite-PTFE-compound	X	X	X			✓
bronze-PTFE-compound	X	X	X	X		✓
carbon-bronze-PTFE-compound	X	X	X			✓

Comments:

BAM-test methods:

1) Determination of the ignition temperature in compressed oxygen
2) Test of ageing resistance in compressed oxygen dependant on the temperature
3) Behaviour after exposure to oxygen pressure shocks depending on pressure and temperature
4) Reactivity with liquid oxygen under impact stress
5) Testing of flat gaskets for flange connections in gaseous oxygen

***BAM*-Listing:** see chapter 1.5

1.5 List of non-metallic materials [2]

The materials which have been found suitable by the Federal Institute for Materials Research and Testing (*BAM*) for use in plant components for oxygen are listed in the "List of non-metallic materials", (BG-Merkblatt (leaflet) M 034-1) for 10 years with corresponding release limits.
This list can be purchased as “BG Merkblatt M 034-1” from “Jedermann-Verlag, Heidelberg”. The leaflet is updated and reprinted annually. A pdf-document can also be downloaded from their website.
The annex of this list contains the addresses of the manufacturer and processors of the non-metallic materials and the test methods the *BAM* used.

It is recommended that processors use the current list of “BG-Merkblatt M 034-1” as a working basis.

PTFE and PTFE-compounds are listed in the following applications with contact to oxygen:

Section 2: Sealing materials for flanges of any type
Section 2.2: Sealing materials for flanges only with tongue and groove
Section 4: Seals for valves and fittings
Section 5: Materials for piston rings in compressors

1.6 Safety related assessment of finished articles for use in contact with oxygen

Non-metallic materials for the use in contact with oxygen are tested by *BAM* with respect to their safety in contact with oxygen.
If the test is successful, the upper limits for pressure and temperature of the materials are listed in the “list of non-metallic materials”.

Non-metallic materials in PTFE-processing may be the following:

- P_0: virgin powder of the manufacturer
- P_1: processed powder as semi-finished product by the manufacturer (reference sample)
- P_2: processed powder as semi-finished product by the professional user
- P_3: mechanically treated semi-finished products of the professional user
- P_4: Finished products of PTFE (of the final customer) for installation in oxygen devices

A final customer is a professional user who takes over the processed semi-finished article (P_3) and prepares it (P_4) for the installation into an oxygen device (possibly after a final processing step).
It is highly recommended that the final customer has the finished article (P_4) tested and assessed by *BAM*.

It is in the responsibility of the professional user to decide whether their specific formulation and processing is appropriate for the intended application. Professional users have to check whether the

[2] BG-Merkblatt M 034-1 in addition to BG-Merkblatt M 034 “Sauerstoff” BG RCI issue July 31, 2017

non-metallic made for the particular application is in compliance with the list of non-metallic materials in terms of raw material and processing. The test values determined by *BAM* can only be used as a basis if the test sample submitted to *BAM* and the later series sample for oxygen applications are identical.
Generally speaking, the professional user has to assess whether the non-metallic materials listed in the "List of non-metallic materials" finally represent the processed semi-finished articles for the direct application in oxygen devices (1) (e.g. sealing materials of sealing manufacturers) or if they are raw materials or semi-finished goods which have been provided as reference samples to *BAM* for orienting oxygen tests.
Raw material tests by *BAM* can only provide orienting results. The investigations are performed on semi-finished goods (reference sample P_1 that have been processed by raw material manufacturers under laboratory conditions e.g. in application labs or quality labs based on recommended standard processing conditions. These processing conditions do not represent standard industrial conditions.
Professional users of raw materials should perform a safety related assessment of their semi-finished goods and finished articles which are designed for the sealing uses in oxygen devices, since the behaviour of sealing materials in fittings (valves and pressure stats), flange connexions in pipes and tube fittings for PTFE can depend on the processing parameters for moulding, sintering and machining.
The "ignition temperature in compressed oxygen" of a material also depends, for example, on the manufacturing process, the cleanliness of the process and the use of flammable components (processing aids such as machine oils, release agents and contaminants).
These parameters can influence the permissible upper limits for pressure and temperature in the test procedure "ignition temperature in compressed oxygen".

1.7 Bundesanstalt für Materialprüfung (*BAM*)

For information and tests aimed at the use of PTFE and PTFE compounds in oxygen devices only DIN EN ISO certified labs like *BAM* are qualified:

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Annex: List of references

"Gefahren durch Sauerstoff und ihre Abwendung", MODERNE UNFALLVERHÜTUNG, Issue 20, pp. 127 - 131, Vulkan Verlag, Essen
("Hazards of oxygen and how to avoid them", MODERN ACCIDENT PREVENTION)

"Tests to Evaluate the Suitability of Materials for Oxygen Service", Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986, D. W. Schroll, Ed., American Society for Testing and Materials, Philadelphia, 1988, pp. 268 - 278

BG-Regelwerk BGR 500 "Betreiben von Arbeitsmitteln", Teil 2, Kapitel 2.32, "Betreiben von Sauerstoffanlagen" (siehe www.hvbg.de, Register "Prävention").
("BG" rules and regulations BGR 500 "Operation of work equipment", part 2, chapter 2.32, "Operation of oxygen systems" (see www.hvbg.de, "Prevention" tab).)

BG-Merkblatt M 034 "Sauerstoff" (Bezugsquelle: Jedermann-Verlag)
(BG leaflet M 034 "Oxygen" (source of supply: Jedermann-Verlag))

BG-Merkblatt M 034-1, "Liste der nichtmetallischen Materialien", in Ergänzung zum BG-Merkblatt M 034 "Sauerstoff" (Bezugsquelle: Jedermann-Verlag)
(BG leaflet M 034-1, "List of non-metallic materials", as a supplement to BG leaflet M 034 "Oxygen" (source of supply: Jedermann-Verlag))

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www.bam.de

Arbeitsgruppe "Sicherer Umgang mit Sauerstoff" (*Working group "Safe use of oxygen"):*
www.bam.de/kompetenzen/arbeitsgebiete/abteilung_2/fachgruppe_21/fg_21_ag3.htm

Industriegaseverband e. V.:
www.industriegaseverband.de

Hauptverband der gewerblichen Berufsgenossenschaften (*Main Association of the Industrial Employer's Liability Insurance Associations*):
www.hvbg.de

Jedermann Verlag:
www.jedermann.de