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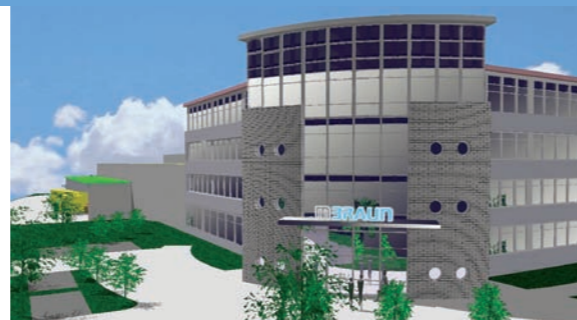


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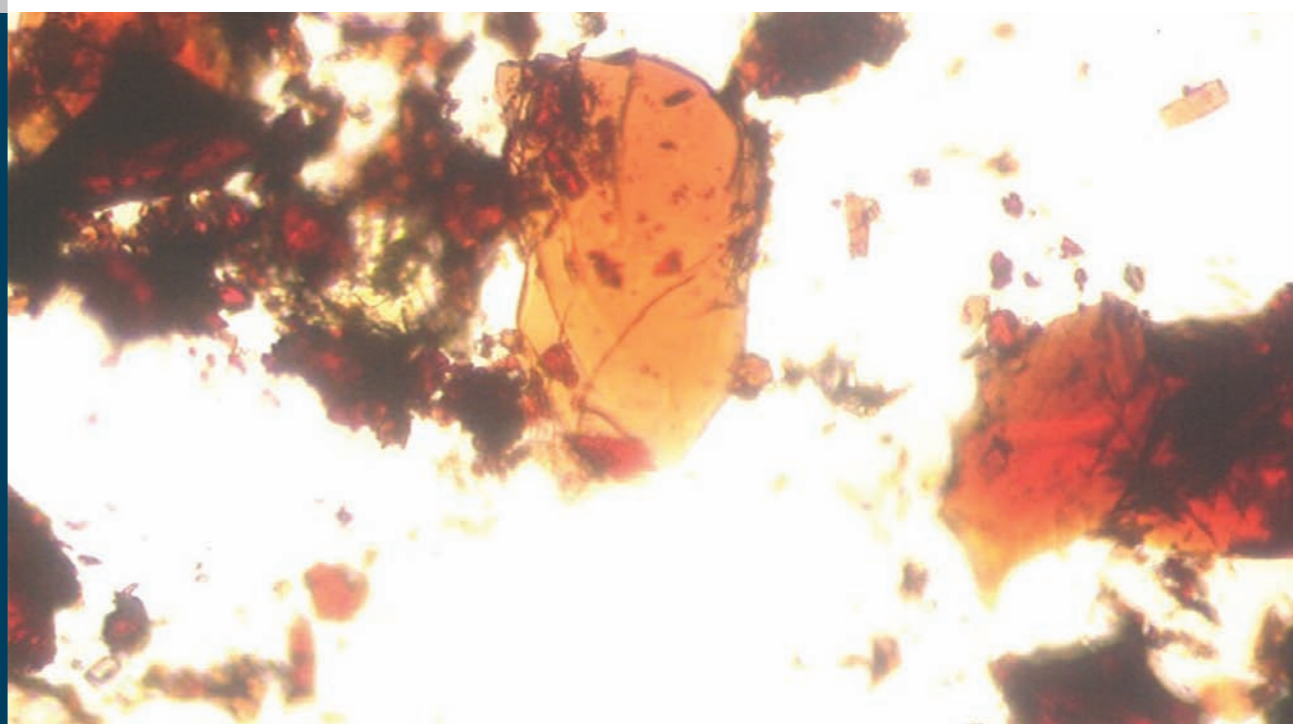
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**PICTUREBOOK**



Special edition - Achema 2003

**INERTGAS TECHNOLOGY**



## Theta / Theta-High Temperature-Diffractometer in MBRAUN Glovebox

Text: Dr. Angela Möller,  
Universität zu Köln  
Anorganic Chemistry

To investigate chemical reactions (in-situ tests), in particular of moisture-sensitive substances and phase mixtures by means of x-ray diffraction, a theta/theta diffractometer (Stoe) was integrated with a high temperature oven (Bühler, Johanna Ott) in a glovebox (MBRAUN).

In this way the preparation on a metal carrier can take place under inert gas conditions.

Subsequently the reaction chamber can be evacuated and / or filled with a reactive gas (e.g.  $\text{NH}_3$ ), or have gas continuously flowing through.

After the completion of the temperature program with the plotting of the

diffractograms at the selected temperature stages, the sample can be removed and possibly prepared for further analysis techniques under inert gas.

This setup is important for the testing of very moisture-sensitive solids that can not be tested using the standard techniques in quartz capillaries at higher temperatures, as reactions with the vessel wall arise. The illustration (below left) shows the reaction process for such a system ( $\text{Na}_2\text{O} / \text{Cu}_2\text{O} / \text{Na}_2\text{CO}_3$ ).

P. Amann, A. Möller, Z. *Anorg. Allg. Chem.*, 2003, accepted.

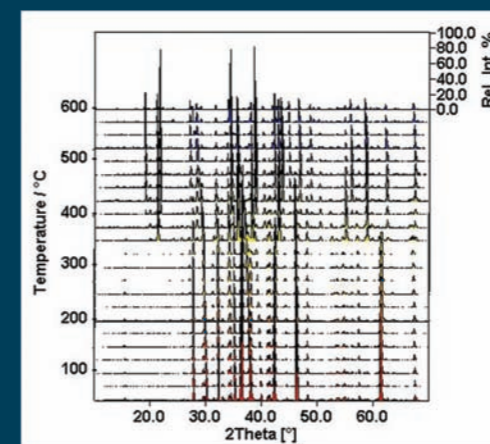
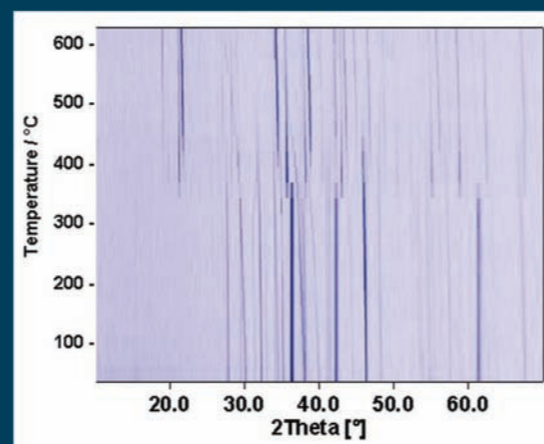
Reactivity, Synthesis and Crystal Structures of  $\text{Na}_5[\text{MO}_2]\text{X}$  with  $\text{M} = \text{Co}^+, \text{Ni}^+, \text{Cu}^+$ ;  $\text{X} = \text{CO}_3^{2-}, \text{SO}_3^{2-}, \text{S}^{2-}$  and  $\text{Na}_{25}[\text{CuO}_2]_5[\text{SO}_4]_4[\text{S}]$

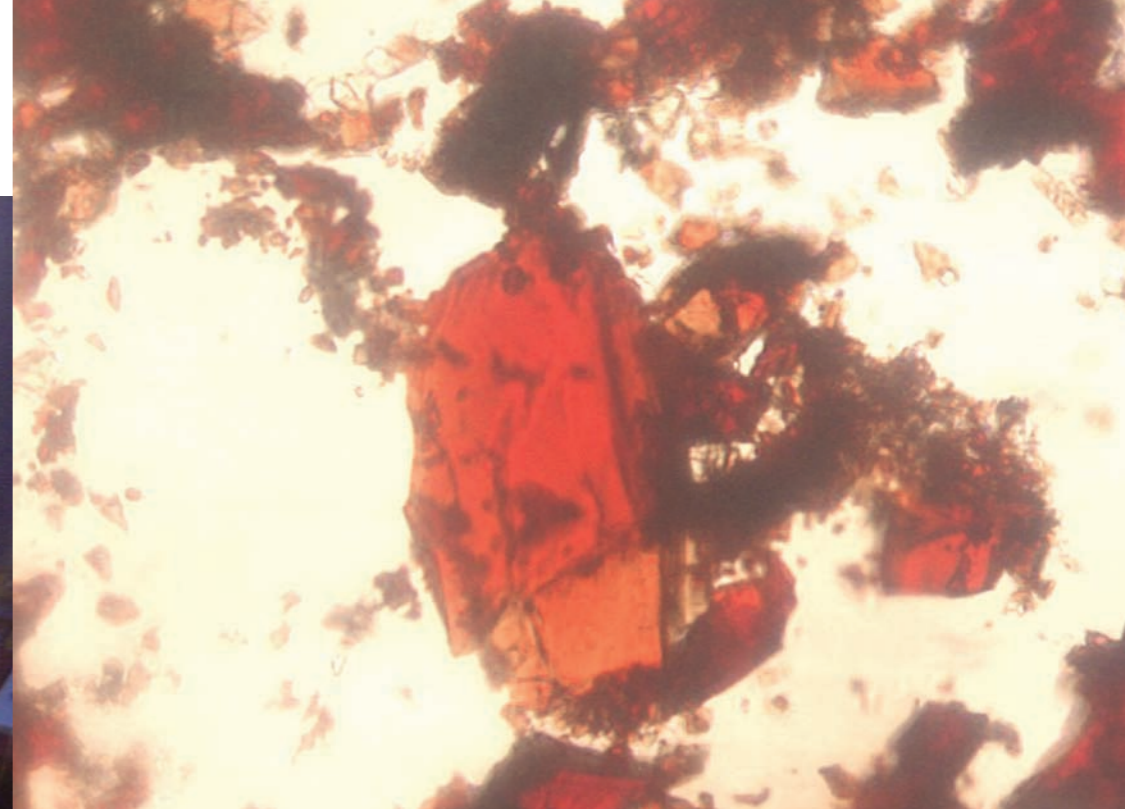
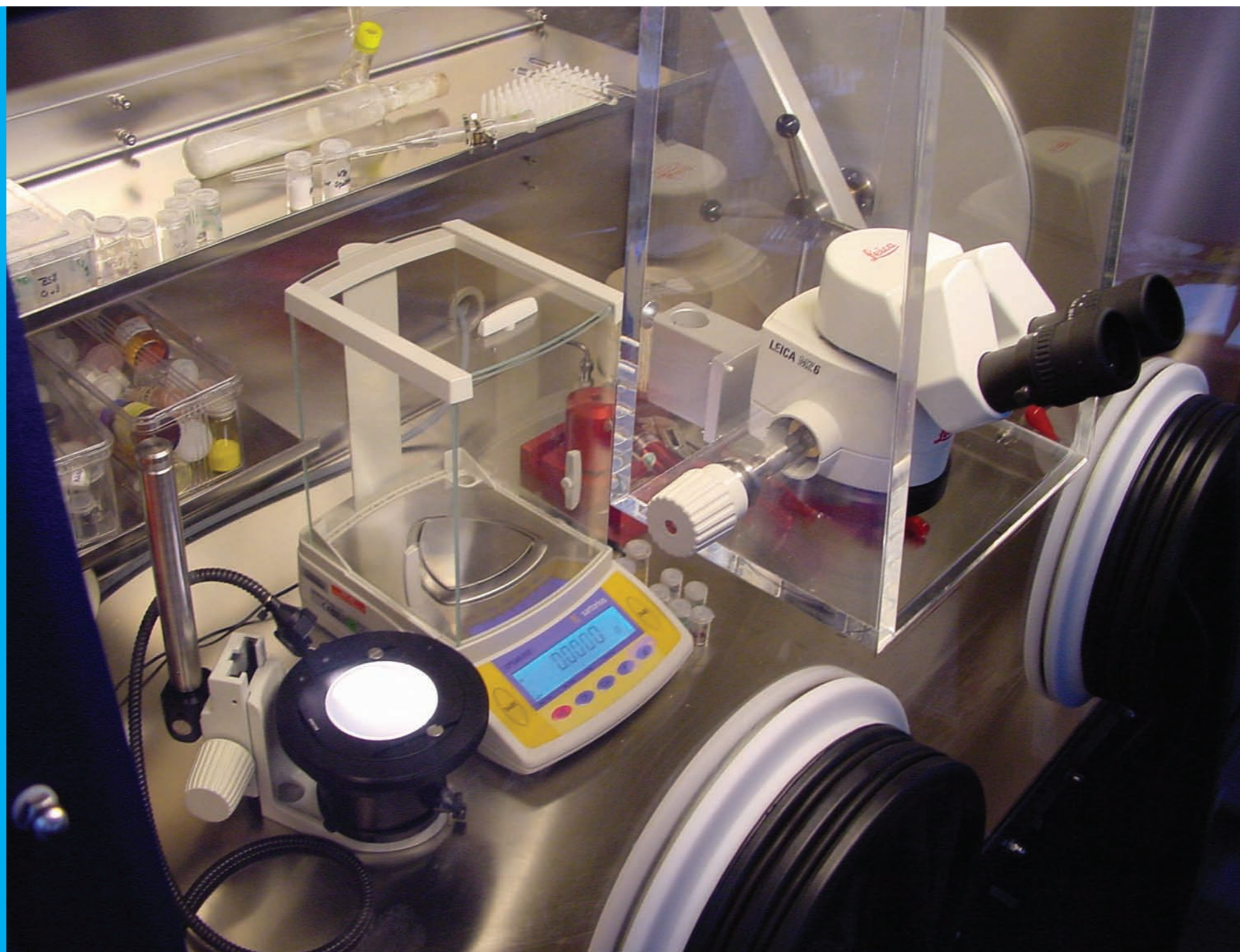


MBRAUN Glovebox with Theta / Theta-Diffractometer  
(Picture above)

Diagrams  
„in-situ-test“  
(Pictures right)

MBRAUN Glovebox with Theta / Theta-Diffractometer  
(Picture left)





## RUBY RED CRYSTALS

Fascination MBRAUN Glovebox

Text: Dr. Angela Möller,  
Universität zu Köln

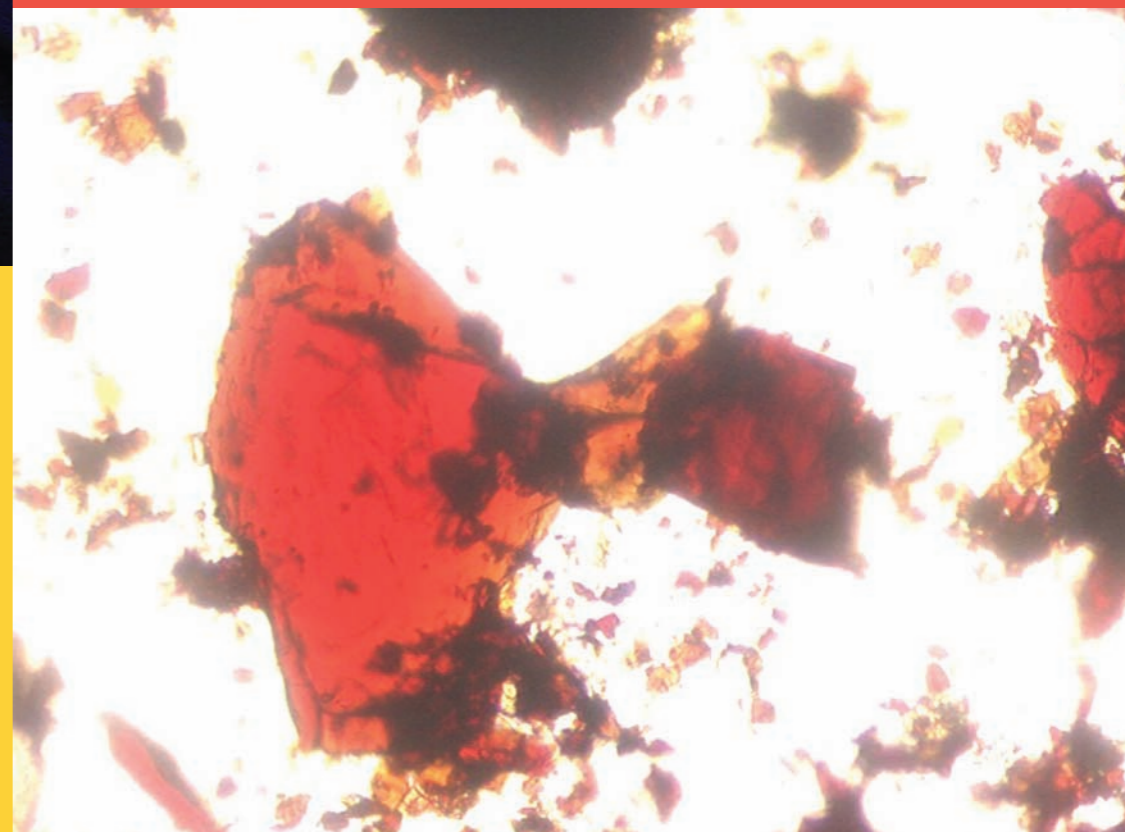
**F**or the selection of moisture-sensitive single crystals an MBRAUN glovebox equipped with a

microscope is used. By adapting a digital camera over the ocular of the microscope, the documentation of the products received can take place.

The illustrations show red  $\text{Na}_5[\text{CoO}_2][\text{CO}_3]$  crystals. Here cobalt is in the unusual oxidation stage +1!

**MBRAUN Glovebox with microscope**  
(Picture above)

**Red crystals**  
(Pictures right)





# Tripledecker-Sandwich-Complexes

## of Calcium, Strontium and Barium

Text: Prof. Dr. Helmut Sitzmann, Universität Kaiserslautern

The first Tripledecker-Sandwich-Complexes of

the heavy alkaline earth metals calcium, strontium and barium are thermally stable to over 250 °C as well as being without decomposition fusibly and sublimatable despite



the high molecule mass,  $[(^4\text{Cp})\text{M}(\text{C}_8\text{H}_8)\text{M}(^4\text{Cp})]$   
 $[^4\text{Cp} = \text{C}_5\text{H}(\text{CHMe}_2)_4;$   
 $\text{M} = \text{Ca}, \text{Sr}, \text{Ba}).$

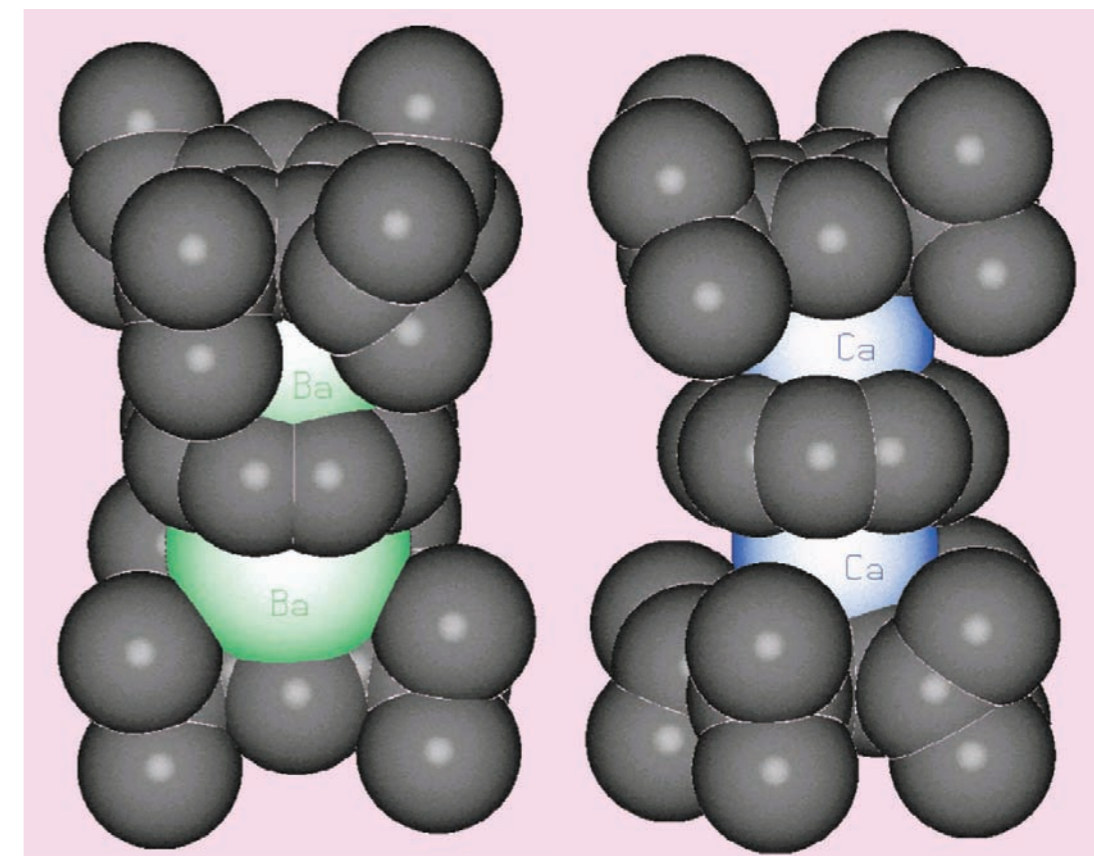
The potassium cyclo-octatetraenid used for the synthesis of the first neutral Tripledecker-Sandwich-complex with main group element central atoms,  $\text{K}_2\text{C}_8\text{H}_8$ , explodes on contact with air. The colourless, crystalline alkaline earth metal bonds also decomposed immediately in air.

The substances were manufactured in the accompanying illustrated glovebox, in which the samples for spectroscopic and analytic investigations were also gathered.

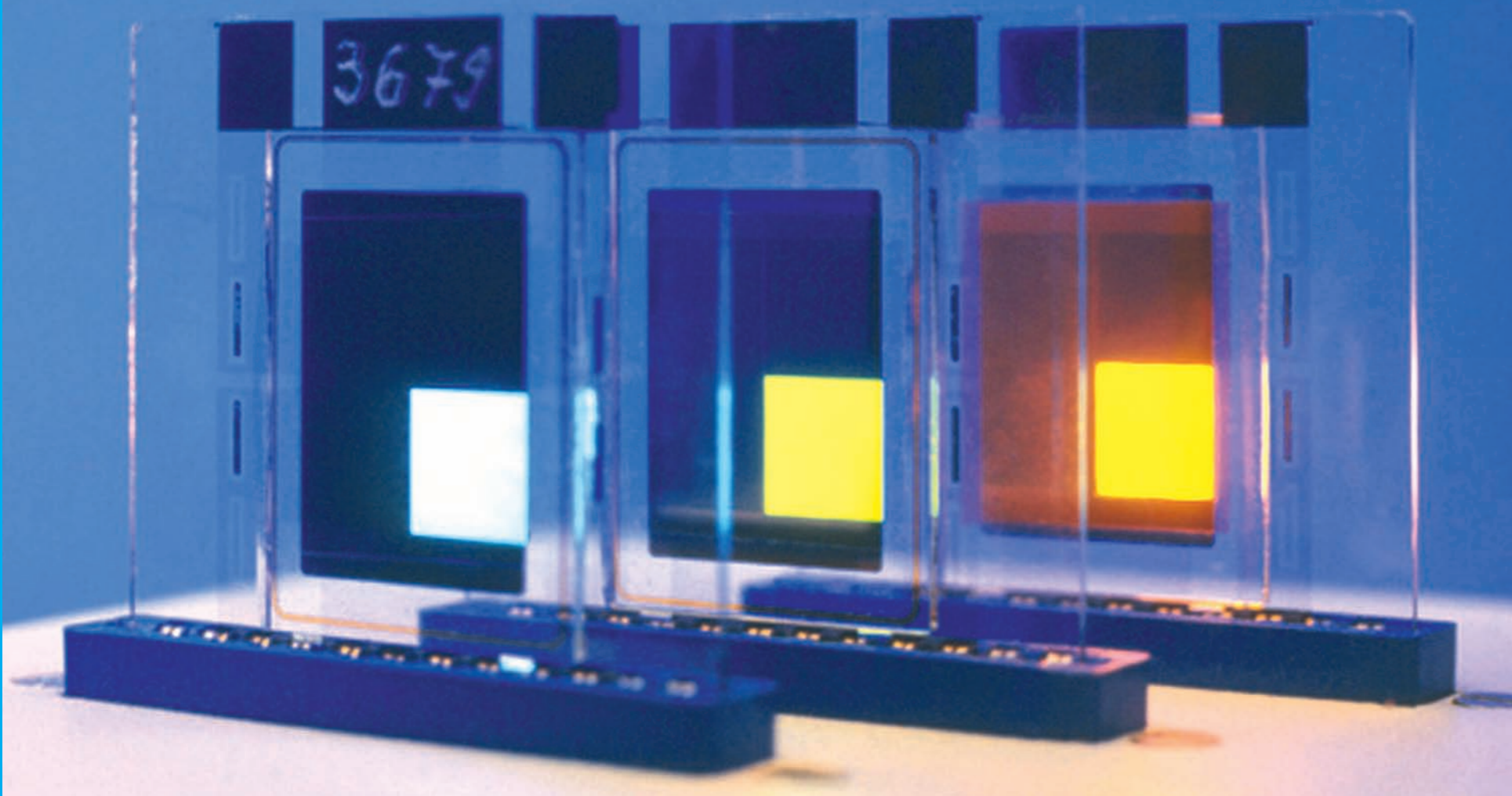
The illustration right shows the crystal structures of the barium and calcium bonding (carbon atoms dark grey, to provide a better overview hydrogen atoms are not shown).

H. Sitzmann, M. D. Walter, G. Wolmershäuser, *Angew. Chem.* **114** (2002) 2315-2316;

*Angew. Chem. Int. Ed. Engl.* **41** (2002) 2421-2422; M. D. Walter, G. Wolmershäuser, H. Sitzmann, *J. Am. Chem. Soc.*, submitted.



## Organische Leuchtdiode **OLED** (Organic Light Emitting Diode)



Text: Dr. Wedel,  
Fraunhofer Institute for  
Applied Polymer Research

The simplest form  
of an Organic  
Light Emitting  
Diode, (OLED) consists  
of three layers. A layer of

glass (or foil) coated with  
indium-tin-oxide serves  
as the transparent carrier  
and first electrode. A thin  
organic layer (only about  
100 nm) is put on this and  
this is then covered with a  
second electrode (metal  
electrode).

At the Fraunhofer IAP  
the technologies for the  
production of indicators  
and smaller displays on  
the basis of polymers are  
currently being developed  
with the help of MBRAUN  
Inert Gas Systems and  
BOC Edwards.

**Organic Light Emitting Diode**  
(Picture above)

**Glovebox with Laminar Flow**  
(Picture above right)

**OLED-Display**  
(Picture right)



Each step in production is  
carried out under special  
conditions in a purified  
chamber, as the „glowing“  
layers are extremely thin  
and every particle of dust  
can cause disturbance  
and lead to failure. Such  
displays are available  
in various colours (red,  
green, blue) and besides  
being sufficiently bright  
give excellent radiance.





Text: Oliver Minge,  
Norbert W. Mitzel and  
Hubert Schmidbaur

Anorganic-chemical  
Institute, Technische  
Universität München

The use of tri(alkoxy)silanes  $(RO)_3SiH$ , which have recently become commercially available in greater than research

scale, has been probed for the preparation of hydrogen-rich arylsilanes  $ArSiH_3$ . It was found that the silylation of aryl-lithium or (in situ) aryl-Grignard reagents is followed by RO/H ligand redistribution and can lead to fully hydrogenated products in a one-pot reaction without employment of any additional metal hydride.

**MBRAUN  
Inert Gas System  
Labstar®**  
(Picture above)

Anorganic hydrocarbons with silyl substituents  $-SiH_3$  are important precursors for the generation of di-, oligo-, and polysilanes with aryl substituents. These aryl-polysilanes have great potential for applications in a series

oligo-, and polysilanes with aryl substituents. These aryl-polysilanes have great potential for applications in a series of optoelectronic devices owing to their unique electronic structure with conjugated arene and

commercially available. They appeared to be very convenient reagents since they combine the experimental advantages of the tetra(alkoxy)silanes and the hydride functionality.

## Synthetic pathways

### to hydrogen-rich polysilylated arenes from trialkoxysilanes and other precursors\*

(\*Organometallics 2002, 21, 680-684)

of optoelectronic devices owing to their unique electronic structure with conjugated arene and polysilane systems.

Several efficient methods have been developed for dehydrogenative or desilanative coupling RO/H ligand redistribution and can lead to fully hydrogenated products in a one-pot reaction without employment of any additional metal hydride.

Anorganic hydrocarbons with silyl substituents  $-SiH_3$  are important precursors for the generation of di-,

polysilane systems.

Several efficient methods have been developed for dehydrogenative or desilanative coupling reactions of simple silyl-arenes to generate silyl or disilyl bridges between the hydrocarbon units.

We tried to open new or modified synthetic pathways to hydrogen-rich aryl-silanes using inter alia tri(alkoxy)silanes as starting materials.

These mixed alkoxide hydrides of silicon have recently become

All reactions were carried out in MRAUN gloveboxes under a controlled atmosphere of purified nitrogen.