-Federal Institute for Materials Research and Testing-

BUNDESANSTALT FÜR MATERIALFORSCHUNG UND -PRÜFUNG
(BAM)

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# R e p o r t

about the investigation of the chemical stability of ethylene oxide and 1,2-propylene oxide in the presence of the sealing material "SIGRAFLEX-UNIVERSAL V(100-400) 10 C2I"

## 1. Application

The firm SIGRI GmbH, has applied for investigations of the chemical stability of ethylene oxide and 1,2-propylene oxide in contact with the composite material "SIGRAFLEX-UNIVERSAL V(100-400) 10 C2I"(in following called "Sigraflex") at the Federal Institute for Materials Research and Testing (BAM) with the letter HLE5/mec/bös from November 27, 1989.

## 2. Investigation material

### 2.1 Composite material

Sheets of dimensions 120 mm x 20 mm and rings of outside diameter 190 mm, inside diameter 140 mm, consisting of the material "Sigraflex", were made available as investigation material to the BAM. According to the specification by the SIGRI GmbH the material is a laminated graphite rolled with a filling material consisting of stainless steel pike sheet.

### 2.2 Ethylene oxide; 1,2-Propylene oxide

The ethylene oxide was obtained from the firm Linde AG whith the following specification.

purity: 2.7 = 99,7%
manufacturing date: May 30, 1989
evaporation residue: 0.03 %

The 1,2-propylene oxide was obtained from the firm E. Merck Darmstadt whith the following specification.

quality: 1,2-propylene oxide for synthesis
content: more than 99%
evaporation residue: 0.004 %

## 3. Investigation programme

### 3.1 Warm storage tests

One litre of ethylene oxide and one litre of 1,2-propylene oxide, respectively, were stored in contact with 6 sheets "Sigraflex" each in special autoclaves of stainless steel in warming cupboards. The autoclaves were sealed with rings of "Sigraflex". The storage temperature was 60°C. The duration of the storage was 6 weeks.

The analogous storage tests were carried out without the addition of "Sigraflex". In these tests the autoclaves were sealed with rings of PTFE.

The pressure within the autoclaves and the temperature of the autoclaves were continuously measured during the storage time and automatically recorded. The temperature in the warming cupboards was also measured and recorded for the, if necessary, uneqivocal determination of temperature and pressure changes due to chemical reactions and not to the change of environmental temperature.

During the six weeks-storage no pressure and temperature changes caused by any reaction were observed.

## 3.2 Analytical investigations

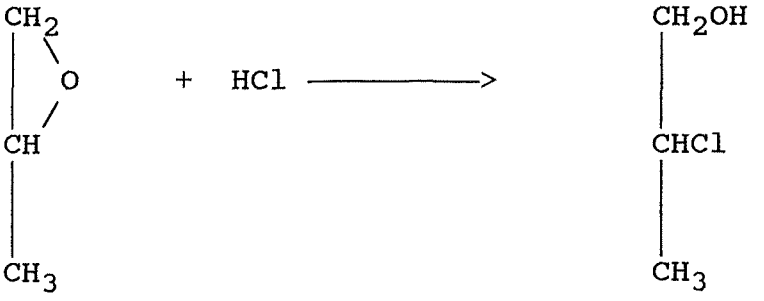
### 3.2.1 Determination of the contents of ethylene oxide and 1,2-propylene oxide according to a method by Deckert[1] [2] and Kerkow[3].

Principle:

Ethylene oxide and 1,2-Propylene oxide, respectively, react with hydrochloric acid saturated with metal chlorides to 2-chloro-ethanol and to 2-chloropropanol, respectively.

a.


b.




The consumption of hydrochloric acid is the measure for the content of ethylene oxide and 1,2-propylene oxide, respectively.

There were determined

a) non-stored
b) stored for 6 weeks 60°C, resp.
c) stored for 6 weeks 60°C, resp., in contact with "Sigraflex"

samples of ethylene oxide and 1,2-propylene oxide, resp., each time.

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[1] Deckert W. Fr. Z. f. Anal. Ch. **82** 297 (1930)
[2] Deckert w. Ang. Chem. **45** 785 (1932)
[3] Kerkow F.W. Fr. Z. f. Anal. Ch. **108** 249 (1937)

The results for the samples stored with and without "Sigraflex" agree with the results for the unstored samples within the limits of the relative errors of the analytical method. A change caused by the influence of "V(050-400) 10 C3I" on ethylene oxide and 1,2-propylene oxide, resp., was not established.

### 3.2.2 Determination of the polymerizate fractions

Samples of ethylene oxide and 1,2-propylene oxide - unstored, stored for 6 weeks, at 60°C and stored for 6 weeks at 60°C in contact with "Sigraflex" - were evaporated and the masses of the residues (polymerizates) were determined.

Results

| | | |
|---|---|---|
| Ethylene oxide | unstored | 0,03 % residue |
| Ethylene oxide | stored without "Sigraflex" | 0,36 % residue |
| Ethylene oxide | stored with "Sigraflex" | 0,51 % residue |

| | | |
|---|---|---|
| 1,2-Propylene oxide | unstored | 0,004 % residue |
| 1,2-Propylene oxide | stored without "Sigaflex" | 0,010 % residue |
| 1,2-Propylene oxide | stored whith "Sigraflex" | 0,025 % residue |

Under the given experimental conditions there were found only slightly higher amounts of residues in the samples of ethylene oxide and 1,2-propylene oxide, resp., stored in contact with "Sigraflex" than in the samples of ethylene oxide and 1,2-propylene oxide, resp., stored without that addition.

### 3.2.3. Investigation of the evaporation residue for the content of polyethylene oxide

The evaporation residue of the ethylene oxide sample stored in contact with "Sigraflex" was investigated according to the method published by Seher [4] which principle is described in the following.

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[4] Seher A.; Fette, Seifen, Anstrichmittel 63 617 (1961)

After oxonation of the polymerizate with barium chloride in acid medium and precipitation of the formed oxonium cation with sodium tetraphenylborate solution the complex precipitate is sucked off. After being dissolved in dimethylformiate the complex is decomposed by mercury(II) nitrate, the excess of mercury(II) nitrate is back-titrated according to the method of Vollhard. The consumption of mercury(II) nitrate is the measure for content of polyalkene oxide.

A mass fraction of 82 % was found for polyalkene oxide.

## 4. Summary and review

On application of the firm SIGRI GmbH, Meitingen, ethylene oxide and 1,2-propylene oxide were investigated for their chemical stability in contact with the composite material "SIGRAFLEX-UNIVERSAL V(100-400) 10 C2I". By warm storage tests for 6 weeks at 60°C it was confirmed that ethylene oxide as well as 1,2-propylene oxide remain stable in the presence of the composite material "SIGRAFLEX-UNIVERSAL V(100-400) 10 C2I". Under the given experimental conditions no change owing to the influence of "SIGRAFLEX" on ethylene oxide and 1,2-propylene oxide could be detected.Insignificantly higher amounts of evaporation resuide were found merely on the ethylene oxide stored with "Sigraflex" in comparison with ethylene oxide stored oxide without any additive.

On the evaporation residue a mass fraction of about 80% polyalkene oxide was ascertained. These findings are non-critical whith repect to the relevant safety technological aspects, they are, at most, of influence on the product quality.

On the part of the BAM there are no objections to using "SIGRAFLEX-UNIVERSAL V(100-400) 10 C2I" in contact as well with ethylene oxide as with 1,2-propylene oxide, also to a temperature of 60°C for a short time.

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