

**Determination of the
epoxide equivalent weight
epoxide resins according to
DIN 53 188**

Application

Content

Content	2
Use.....	2
Appliances	2
Electrodes	2
Reagents	2
Description.....	2
Method	4
Example	10
Hints.....	10
Literatur.....	10

Use

The epoxid equivalent weight is the mass of epoxid resin in g that contains 16g of epoxidically bound oxygen (epoxid oxygen). It is determined by titration with perchloric acid in glacial acetic acid after reaction with tetra-n-butyl Ammonium Iodide. This method is not applicable for epoxide resins containing basic groups.

Appliances

Titration tip :	TitroLine alpha plus (TL 20 plus)
Titration tip :	TZ 1509
other appliances:	printer

Electrodes

Electrode:	N 6480 EIS (LiCl/ glacial acetic acid) cable L1A or a divided pair of electrodes e.g. N 2680 + B 3920
Electrolyte:	LiCl/glacial acetic acid, L 501 4

Reagents

Solvent:	dichloromethane/glacial acetic acid
Titer determination:	with potassium hydrogenphthalat
Titration agent:	perchloric acid in glacial acetic acid (0,1 mol/L)
other reagents:	tetra-n-buthyl-ammonium iodide

Description

Preparation of 0,1 mol/L perchloric acid in glacial acetic acid:

Use commercial standard solutions.

Standardization:

0,15 to 0,2 g of dried (120°C) potassium hydrogenphthalate are weighed in exactly and solved in 60 mL of the solvent mixture while heating. After cooling to room temperature the solution is titrated with perchloric acid. Triple determination is recommended. A blank titration is carried out using the solvent mixture.

Application

Titration:

The required mass of weighed in sample is calculated according to the equivalent weight expected:

Epoxid Equivalent Weight	Sample [g]
to 300	0,5
from 300 to 750	1,0
from 750 to 1400	2,0
from 1400 to 2500	3,0
from 2500	4,0

The sample is weighed in a 100 mL beaker, 60 mL of the solvent are added and the sample is dissolved while stirring and heating. After cooling down 2,0 g of Tetra-n-butyl Ammonium Iodide are added. Now the electrode and tip are immersed into the beaker and the method is started. After the titration the electrode is rinsed with the solvent used.

Maintenance of Electrodes

If you use a combination electrode such as N 6480 with glacial acetic acid, store the electrode in the LiCl/glacial acetic acid electrolyte. You can also use a combination of the N 2680 and the B 3920 electrode. The single glass electrode is stored in 3 mol/L KCl and the reference electrode in LiCl/glacial acetic acid. The outer electrolyte of the B 3920 is also LiCl/glacial acetic acid the inner electrolyte is KCl 3 mol/L.

Method

blank value titration (page 1):

blank value

1 / 2

method number	16	no smoothing	
password	no	sample amount	:
sampler	none	no	
		statistics	:
		no	
		no. decimals	3
		std documentation	
method link	no		
method	selection mV		
edit std method	linear(EQ)		
measuring channel A			
input delay	weak / alcohol		
initial meas. value	no		
sample ID	manual		
predosing burette	no		
waiting time [s]	1		
pretitration buret	no		
reaction time [s]	30		
fill dosing unit	fill		
linear steps	0.008		
end of titration	mL		
final consumpt. ml	0.50		
drift control	fixed waiting time		
fixed waiting time	10.00 s		

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	07.03.2007
	10:08:37

blank value titration (page 2):

blank value

2 / 2

method number	16
calculation :	
formula	$\text{mL} \cdot F1 \cdot F2 / Q$
formula no.	EQ 1
value F1	1.0000
value F2	1.0000
value Q	1.0000
B = Global Memory	1
identifier	blank value
unit	mL
result:	into global memory 1

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	07.03.2007
	10:09:26

standardisation (page 1):

titre

1 / 2

method number	16	no smoothing
password	no	sample amount :
sampler	none	weight manual
		statistics :
		yes
		no. decimals 4
		std documentation

method link	no
method	selection mV
edit std method	dynamic(EQ)
measuring channel A	
input delay	weak / alcohol
initial meas. value	no

sample ID	manual
predosing burette	no

waiting time [s]	1
pretitration buret	yes
pretitration in ml:	8.00
reaction time [s]	30
fill dosing unit	fill

end of titration	mL + EQ
------------------	---------

number end points	EQ 1
final consumpt. ml	15.00
end of titration	medium jump
drift control	medium

dynamic control	medium jump
-----------------	-------------

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	07.03.2007
	10:28:04

standardisation (page 2):

titre

2 / 2

method number	16
calculation :	
formula	$Q \cdot F1 / ((mL - B) \cdot F2)$
formula no.	EQ 1
value F1	1.0000
value F2	0.2042
value Q	1.0000
B = Global Memory	1
identifier	titre
unit	mol/L
result:	into global memory 2

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	07.03.2007
	10:28:54

sample titration (page 1):

Epoxid

1 / 2

method number	16	no smoothing
password	no	sample amount :
sampler	none	weight manual
		statistics :
		no
		no. decimals 3
		std documentation
method link	no	
method	selection mV	
edit std method	dynamic(EQ)	
measuring channel A		
input delay	no / water	
initial meas. value	no	
sample ID	manual	
predosing burette	no	
waiting time [s]	1	
pretitration buret	yes	
pretitration in ml:	8.00	
reaction time [s]	45	
fill dosing unit	fill	
end of titration	mL + EQ	
number end points	EQ 1	
final consumpt. ml	25.00	
end of titration	medium jump	
drift control	medium	
dynamic control	medium jump	

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	09.03.2007
	14:54:21

sample titration (page 2):

Epoxid

2 / 2

method number	16
calculation :	
formula	$(mL-B)*F1*F2/Q$
formula no.	EQ 1
F1 = Global Memory	2
value F2	1000.0000
value Q	1.0000
B = Global Memory	1
identifier	Epoxid
unit	g

version software	14.07.04/01
device no.:	00473409
application user	
passwd.protected:	no
documented:	09.03.2007
	14:55:10

Application

Example

Hints

This application may preferably be carried out using the sample changer TW Alpha.

If you should have any questions concerning the application, you are welcome to contact us.

Literatur

DIN 53 188, Prüfung für Epoxidharze

Schott Instruments GmbH
Hattenbergstr. 10
55122 Mainz
Germany

Phone: +49 (0) 6131 / 66 – 5118
+49 (0) 6131 / 66 – 5118
Fax: +49 (0) 6131 / 66 – 5001
E-Mail: titration@schottinstruments.com
Homepage: www.schottinstruments.com