Water Determination in Ketones

The water content in acetone (ketone) is determined by volumetric Karl Fischer titration.

Sample	Acetone 1.5-2 g
Compound	Water, H_2O M = 18.01 g/mol
Chemicals	60 mL solvent HYDRANAL® KetoSolver or apura® CombiSolvent 5 Keto
Titrant	HYDRANAL® Composite 5 K (5 mg H_2O/mL) or apura® CombiTitrant 5 Keto
Standard	Water Standard 10.0 mg/g (HYDRANAL® Water Standard 10.0)
Indication	DM143-SC Electrode
Chemistry	$CH_3OH + SO_2 + 3\ RN + I_2 + H_2O \rightarrow (RNH)\bullet SO_4CH_3 + 2\ (RNH)I$
Calculation	Water content in ppm: $\frac{(\ VEQ*CONC-(TIME*DRIFT/1000)\ ) * C}{m}$ VEQ: Titrant consumption to EP TIME: Total time since sample request C: 1000 (constant for ppm calc.)
Waste disposal	Organic solvents
Author, Version	Market Support Group Anachem Maria-José Schmid, August 2009

Preparation and Procedures

- Drawn out approximately 2-3 mL of sample into a 10 mL syringe with needle to rinse it before sample analysis.
- Discard the sample into disposal container and fill the syringe again with the sample.
- The sample is then injected into the KF titration vessel in aliquots of approx. 2-3 mL.
- The weight is determined by back-weighing technique.
- If the amount of water is very small, the titration can also be started with the parameter “Cautious” (see Control Parameters).

Remarks

- First, the concentration determination is performed by pressing “Start conc.” (Water Standard 10.0 mg/g, approx. 0.8 g).
- The concentration determination is calculated according to the formula:

  CONC = CONT*m/(VEQ-(DRIFT/CONC(alt))*TIME/1000)

  CONT = Conc. of liquid water standard (mg/g)
  CONC(alt) = CONC(alt) refers to the Setup value current at the time of calculation.
- Subsequently, the sample analysis is started by pressing “Start sample” on the display.

Instruments

- KF Compact Line Volumeters V20/V30 (V2.0)
- Titration Excellence T70/T90 (V3.1.1)
- XP205 Balance

Accessories

- LabX Titration Software
- 10 mL syringe with needle

Results

HYDRANAL® Composite 5 K / KetoSolver

No.	Comment / ID	Start time	Sample size and results		
1/6	--	20.08.2009 09:53:25	R1 = 0.209	%	Content
2/6	--	20.08.2009 11:06:25	R1 = 0.216	%	Content
3/6	--	20.08.2009 11:48:21	R1 = 0.218	%	Content
4/6	--	20.08.2009 12:02:55	R1 = 0.205	%	Content
5/6	--	20.08.2009 12:09:48	R1 = 0.209	%	Content
6/6	--	20.08.2009 12:19:20	R1 = 0.214	%	Content

Statistics

Rx	Name	n	Mean value	Unit	s	srel [%]
R1	Content	6	0.212	%	0.005	2.340

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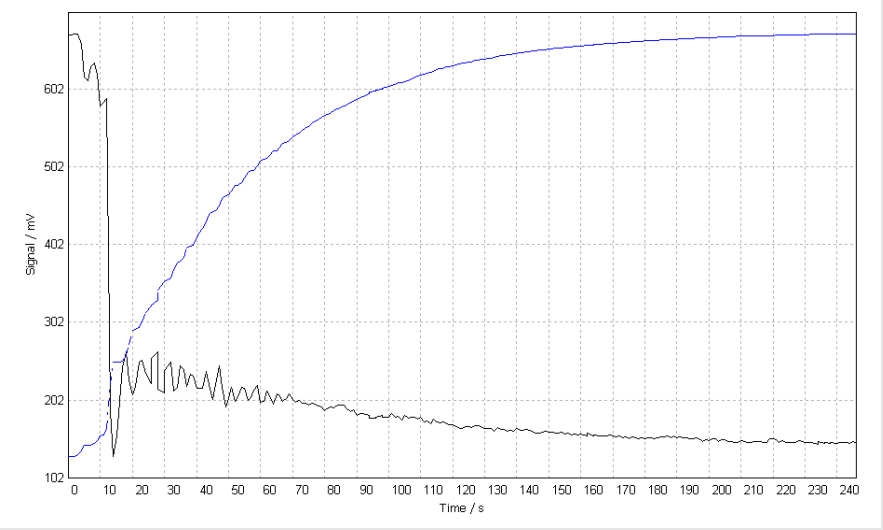
apura® CombiTitrant 5 Keto/CombiSolvent 5 Keto

Statistics

Rx	Name	n	Mean value	Unit	s	srel [%]
R1	Content	6	0.209	%	0.005	2.182

Titration curve



Sample 1/6 20.08.2009 09:53:25

Table of measured values

Volume mL	Increment mL	H_2O mg	Online drift µg/min	Signal mV	Change mV	Time s	
0.00000	NaN	0.0000	0.0	671.1	NaN	0	
0.00000	0.00000	0.0000	0.0	671.1	0.0	0	
0.00000	0.00000	0.0000	0.0	671.7	0.6	1	
0.00100	0.00100	0.0049	153.2	672.7	1.0	2	
0.00550	0.00450	0.0267	552.6	672.2	-0.5	3	
0.01400	0.00850	0.0680	1045.9	659.5	-12.7	4	
0.02675	0.01275	0.1299	1590.5	617.2	-42.3	5	
0.02700	0.00025	0.1311	1333.2	612.0	-5.2	6	
0.02750	0.00050	0.1335	1161.1	630.3	18.3	7	
0.03075	0.00325	0.1493	1134.0	634.6	4.3	8	
0.03800	0.00725	0.1845	1243.9	621.2	-13.4	9	
0.04950	0.01150	0.2404	1456.7	579.7	-41.5	10	
...	...	...	...	...	...	...	
0.97900	0.00025	4.7538	50.5	147.5	-0.4	232	
0.97900	0.00000	4.7538	43.3	145.6	-1.9	234	
0.97900	0.00000	4.7538	28.8	149.2	3.6	234	
0.97900	0.00000	4.7538	21.6	147.0	-2.2	236	
0.97925	0.00025	4.7550	28.8	147.5	0.5	236	
0.97950	0.00025	4.7562	36.1	146.9	-0.6	238	
0.97950	0.00000	4.7562	36.1	147.8	0.9	238	
0.97975	0.00025	4.7575	36.1	146.7	-1.1	240	
0.97975	0.00000	4.7575	36.1	147.8	1.1	241	
0.97975	0.00000	4.7575	28.8	147.9	0.1	242	
0.98000	0.00025	4.7587	28.8	147.7	-0.2	243	
0.98050	0.00050	4.7611	43.3	146.1	-1.6	244	
0.98050	0.00000	4.7611	43.3	149.1	3.0	245	
0.98050	0.00000	4.7611	43.3	146.3	-2.8	246	
0.98075	0.00025	4.7623	43.3	149.4	3.1	246	Sample 1/6

Comments

- Ketones such as acetone react with methanol forming ketals. In this reaction water is released:

  $R\text{-}CO\text{-}R + 2\ CH_3OH \rightarrow R\text{-}C(OCH_3)_2\text{-}R + H_2O$

  Therefore, methanol-free solvent and titrant are used to avoid this side-reaction. For this, special reagents (the so-called K-reagents) are commercially available.
- However, side-reactions can not be completely suppressed. The methanol-free reagents also react slowly with acetone. This leads to an increased drift value at the end of each sample titration. Thus, it is necessary to wait for a constant drift value (e.g. approx. 5 minutes) after each sample determination for the side reaction to subside.
- The drift value is also increasing with increasing number of titrated samples. Thus, the solvent must be replaced after 2-3 samples, depending on the sample size. This can be done by defining in the setup the solvent exchange after 2-3 titrated samples (Setup > Global > Solvent control > Monitoring no. of samples).
- To achieve precise results it is necessary to weigh the sample accurately, and in particular, to first clean the syringe with a few mL of sample which is then discarded.
- To ensure a more efficient cleaning, the syringe is gently shaken to allow for absorption of the moisture on the inner wall of the syringe. Subsequently, the syringe is completely filled.

Method (V2.0)

001 Title

Type	Karl Fischer titration
Compatible with	T70/T90/V20/V30
ID	M303
Title	Acetone Dry
Author	METTLER TOLEDO
Date/Time	18.08.2009 11:49:10
Modified at	18.08.2009 11:49:10
Modified by	METTLER TOLEDO
Protect	No
SOP	None

002 Sample

Number of IDs	1
ID 1	--
Entry type	Weight
Lower limit	3.0 g
Upper limit	8.0 g
Density	1.0 g/mL
Correction factor	1.0
Weight per piece	1.0 g
Temperature	25.0°C
Autostart	No
Entry	After addition
Concentration	
Titrant	KF 1-comp 5K
Nominal concentration	5 mg/mL
Standard	Water-Standard 10.0
Entry type	Weight
Lower limit	0.6 g
Upper limit	1.8 g
Temperature	25.0°C
Mix time	10 s
Autostart	Yes
Entry	After addition
Concentration limits	Yes
Lower limit	4.5 mg/mL
Upper limit	5.8 mg/mL

003 Titration stand (KF stand)

Type	KF stand
Titration stand	KF stand
Source for drift	Determination
Max. start drift	25 µg/min

004 Mix time

Duration	5 s

005 Titration (KF Vol) [1]

Titrant	
Titrant	KF 1-comp 5K
Nominal concentration	5 mg/mL
Reagent type	1-comp
Sensor	
Type	Polarized
Sensor	DM143-SC
Unit	mV
Indication	Voltametric
Ipol	10.0 µA
Stir	
Speed	35 %
Predispense	
Mode	None
Wait time	0 s
Control	
End point	150 mV
Control band	300.0 mV
Dosing rate(max)	8 mL/min
Dosing rate(min)	4 µL/min
Start	Normal
Termination	
Type	Delay time
Delay time	10
At VMax	10.0 mL
Min. time	0 s
Max. time	infinity

006 Calculation R1

Result tape	Predefined
Result	Content
Result unit	%
Formula	R1=(VEQ*CONC-TIME*DRIFT/1000)*C/m
Constant C=	0.1
Decimal places	3
Result limits	No
Record statistics	Yes
Extra statistical functions	No

007 Record

Summary	Per sample
Results	No
Raw results	No
Table of measured values	No
Sample data	No
Resource data	No
E-V curve	No
E-t curve	No
V-t curve	No
H_2O-t	No
Drift-t	No
H_2O-t & Drift-t curves	No
V-t & drift-t	No
Method	No
Series data	No

008 End of sample

Open series	Yes