Fresh

PerkinElmer
For the Better

HUMAN HEALTH | ENVIRONMENTAL HEALTH

VOLUME 44 l Summer Edition, 2014 l Environmental Health

BETTER TECHNOLOGY
BETTER KNOWLEDGE
SUPERIOR RESULTS

Fresh

WHAT'S Fresh inside...

Dear readers,

To all our readers; a "warm" welcome to this edition of Fresh. After prolonged winter in the sub-continent we have started experiencing the hot summer. The environmental and climatic changes made us think about health and food safety.

PerkinElmer's Environmental Health (EH) team is continuously working with the technologies which can be utilized for betterment of quality and safety. In this issue of Fresh we bring you some applications based on Thermal analyzers; suitable for bio-pharmaceuticals. Those who are related to food and food related quality aspects; will find the interesting experimental notes which are performed by our young scientists in the Customer Excellence Centre.

Brief introduction about new TMA4000 Thermomechanical analyser will be interesting to many of our readers. Also see the glimpses of our impressive show at "Arab Lab 2014" recently held at Dubai-UAE.

We request all our readers, users and wellwishers to send articles related to the analysis, applications performed by their team using specific technique and useful for the on-going socio economic or scientific issues related to mankind and environment. We shall try to publish these articles in Fresh or any related publications with your permission.

Have a pleasant summer and enjoy reading.
For any queries and suggestions please write to
Marketing.India@perkinelmer.com

Team Marketing
PerkinElmer (India) Pvt. Ltd.

Measurement of glass transition temperature in formulations

In designed formulations for lyophilized drugs, it is important to know the collapse temperature of the cake. If the collapse temperature is exceeded, the cake will collapse and the batch will be ruined. The collapse temperature is often associated with the Tg′ of the frozen material and measuring this transition is the best way to approximate it. In addition, it is useful to know the amount of non-frozen water, which can be estimated as the enthalpy of melting (ΔH). Both of these necessary values may be obtained with PerkinElmer's Diamond DSC (Figure 1).

Figure 1. DSC 8500

Measurement of the Tg′ is a basic analysis necessary for formulation development, as the temperature of freezedrying needs to occur below the collapse temperature. As an example a sample of protein-excipient formulation (0.4 µg plasmid DNA, 588 µg polyethylenimine (PEI) and 200 µg sucrose in 20 µL of buffered water) was loaded into a hermetically sealed aluminum pan. The sample was cooled to -50 °C in a Diamond DSC using an Intercooler II with nitrogen purge and then heated to room temperature at 20 °C/min. The run shows a clean glass transition at -29.46 °C as shown in Figure 2.

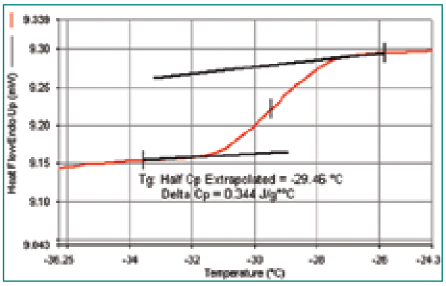


Figure 2. The Tg' of a protein-excipient formulation run as described above.

Tg’ is another measurement that can also be approached by using HyperDSC. HyperDSC is the Diamond DSC’s unique capability to heat and cool, in control, at rates up to 500 °C/min. The Diamond’s ability to heat and cool rapidly with high accuracy allows us to cool the sample at 100 °C/min, hold for 2 minutes, and reheat at 100 °C/min. While this permits substantial time savings, especially when used with an autosampler, the quality of the data is not compromised. Figure 3 shows the results of that the HyperDSC run.

The Diamond DSC or DSC8500 can provide necessary Tg’ and enthalpy data for the development of protein formulations, as well as providing the capability for both HyperDSC and StepScan techniques.

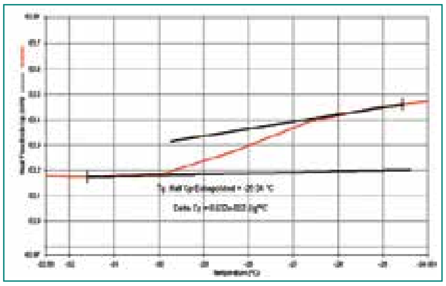


Figure 3. The same formulation as in Figure 2 but run using HyperDSC.

StepScan DSC: A New and Better MTDSC Technique

StepScan DSC is new software for the enhanced characterization of the thermal properties of materials. The technique permits the separation of DSC results into thermodynamic (reversible) and kinetic (irreversible) components for better interpretation. The method is straightforward and utilizes the traditional approach for measuring the heat capacity, Cp, for the highest possible reliability of results without interfering experimental problems. The StepScan DSC approach is only possible with the design of the power compensated DSC, with its very low mass sample and reference furnaces and rapid response time.

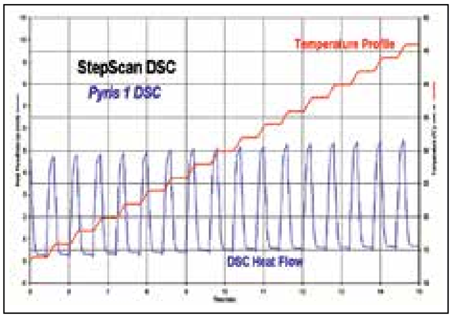


Figure 1. StepScan DSC approach showing time-temperature profile and DSC heat flow results

With the application of heating (10ºC/min) over small temperature increments (1.5 or 2ºC), and by holding for a short time interval (e.g., 30 seconds), the heat capacity that is yielded reflects the reversible aspects of the sample. Kinetic or irreversible effects (on the time scale of the experiment) are eliminated in the Thermodynamic Cp data set, which reflects ‘fast’ or reversible phenomenon, such as the sample’s heat capacity (molecular vibrations) or Tg (molecular rotations). The StepScan DSC approach also provides the kinetic or IsoK Baseline data set, which is reflective of the irreversible or ‘slow’ or irreversible processes taking place during the experiment.

Because the StepScan DSC approach requires rapid DSC response times, the technique is only feasible with the power compensated DSC, which allows for fast heating and thermal equilibration. In addition, the StepScan DSC experiments are generally faster (by a three-fold improvement) as compared to equivalent TMDSC results generated on a slower responding, heat flux DSC device.

The other advantage of the StepScan DSC approach is that it provides a direct heat capacity measurement using the traditional and time-proven means without the need for deconvolution or the extraction of sine wave amplitudes.

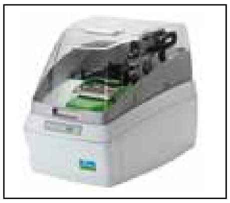
Heat Flux DSC

DSC 4000 Standard Single-Furnace

DSC 6000 Enhanced Single-Furnace

Power Compensated DSC

DSC 8000 Advanced Double-Furnace



DSC 8500 Hyper-enabled Double-Furnace

PerkinElmer®
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Use of sub ambient TGA in pharmaceutical formulations

Problem
A pharmaceutical company desires to have a method for determining the volatiles (alcohol and water) in certain formulations, such as cough syrup. Traditional thermogravimetric analysis (TGA) provides weight loss information starting from room temperature, but the cough syrup begins losing its volatiles at room temperature making it difficult to obtain an accurate assessment of the composition of the syrup. The pharmaceutical facility desires to have a better means of establishing the amount of volatiles in the cough and cold syrups and other formulations.

Solution
Most thermogravimetry experiments begin near room temperature, at approximately 23 °C. This particular choice of starting temperature is satisfactory for most TGA experiments since mass losses occur at temperatures significantly above room temperature. However, for certain weight losses occur at around 50°C. For these applications, it is desirable to start the TGA experiment well below room temperature, at temperatures approaching -20 °C. This provides a better establishment of the low temperature mass loss events.

The study of the drying process of materials, whether due to moisture or low temperature solvent evolution, is the most common application of subambient TGA. This would apply to aqueous or solvent formulations such as cough and cold syrups and other pharmaceutical medications, paints, coatings and hydrated inorganic substances.

The advantages of subambient TGA, for these types of applications are:
- Better definition of TGA baseline prior to water or solvent mass loss event
- Better resolution or separation of overlapping transitions occurring in the temperature region between 25 and 100 °C

PerkinElmer was the pioneer in the development of subambient TGA studies. For subambient studies with the PerkinElmer Pyris 1 TGA, the standard TGA furnace tube is replaced with a special dual walled furnace tube, which permits the circulation of a chilled fluid to permit the sample to achieve subambient conditions.

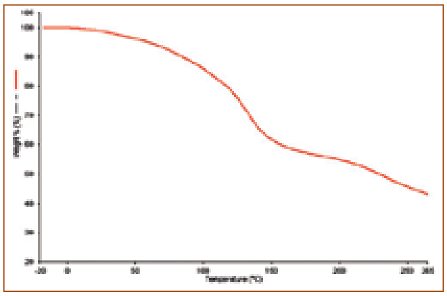

Figure 1. Low temperature TGA to suppress volatile loss.

For the analysis of the cough syrup formulation, a Neslab circulator was utilized with an ethylene glycol – water mixture. The circulator was connected to the dual walled furnace tube, and the Pyris 1 TGA was cooled to the initial starting temperature of about -20 °C.

The cough syrup specimen was obtained by chilling a portion of the syrup with dry ice to freeze the formulation. After freezing, a small specimen of the sample was chipped away and quickly placed into the pre-chilled TGA furnace and sample assembly. The mass of the sample was approximately 38 mg and the specimen was heated at a rate of 20 °C/min from the subambient starting temperature to 250 °C under a nitrogen purge. The results of the subambient TGA experiment on the cough syrup formulation are shown in Figure 1.

From these results, it may be seen that the primary benefit of the subambient TGA is to provide a better TGA baseline response going into the water and volatiles weight loss event beginning at about 20 °C. This provides a more accurate and reliable assessment of the percentage of volatiles contained in the formulation for quality assurance and other purposes.

Summary

The PerkinElmer Pyris 1 TGA may be operated under sub-ambient conditions (start temperature of -20 °C) using a special double walled furnace tube. The subambient operation provides enhanced accuracy and reliability of compositional analysis of materials and formulations containing water and other volatiles, such as alcohol-containing samples. Users who would benefit from subambient analysis include: pharmaceutical formulations such as cough and cold syrups, fruit or soft drink concentrates and hydrated inorganic materials. The use of the circulator fluid with the special dual wall furnace also provides the benefit of increasing sample throughput significantly since the circulator permits the TGA to cool back to ambient conditions much more rapidly after the completion of experiments.

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Estimation of caffeine and moisture in tea

Abstract

The NIR spectroscopy can record the spectra of solid or liquid sample without pre-treatment and is equipped with multivariate analysis for a quick and reliable substitute tool to other quantitative techniques. This experimental note demonstrates the estimation of caffeine and moisture content in tea samples using FTNIR. Caffeine is a purine derivative, which is 1, 3, 7-trimethyl xanthine, the typical concentration of caffeine in tea powders ranges from 2-5% by dry weight of the sample and moisture content ranges between 3-7%

Sample Preparation and Experimental details

All The spectra were recorded using PerkinElmer FTNIR fitted with NIRA accessory. Thirty different secondary tea powder standards for moisture and caffeine were analyzed without any sample pre treatment. The FTNIR method developed analysed moisture and caffeine content in tea samples with an error of prediction less than 0.5%.

Two separate methods were developed, one each for moisture and caffeine. Various mathematical treatments were tested and first derivative was chosen for moisture with actual SEP 0.35 and for caffeine second derivative was chosen with an actual SEP of 0.23.

The methods which showed maximum variance in the first two Latent Variables (LV) were chosen among the various trial methods. The methods were validated using full cross validation method wherein, the specific sample will be left out and the calibration is performed using the rest of the data and is validated against that calibration.

Instrumental conditions

Frontier NIR with NIRA accessory	
Range	10000cm^{-1} to 4000cm^{-1}
Number of scans	32
Resolution	4 cm^{-1}
Baseline correction	Derivative
Normalization	SNV (without detrending)
Smoothening	Yes
Algorithm used	PLS 1

Analytical results & conclusions

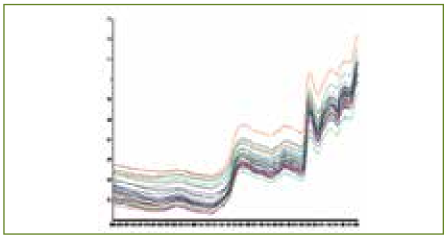

Fig 1. Raw data

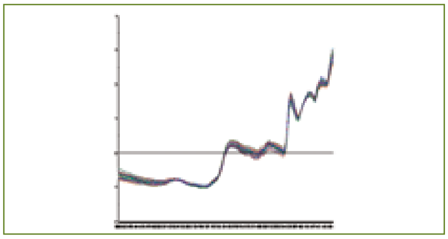

Fig 2.SNV corrected

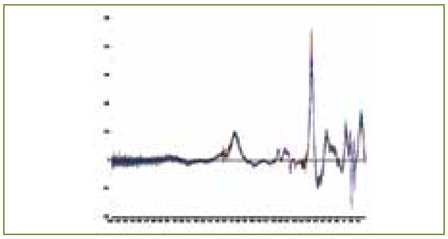

Fig 3. First derivative spectra

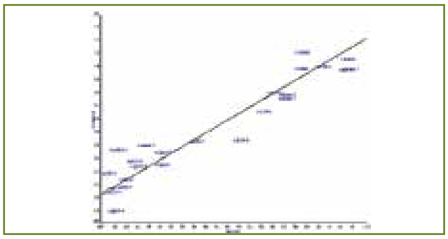

Fig 4.Estimated Vs Specified plot for caffeine / Full Cross Validation

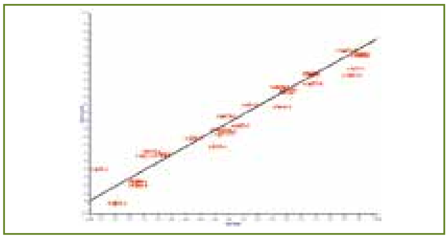

Fig 5.Estimated Vs Specified plot for moisture / Full Cross validation

Calibration report

1.Caffeine	**Number of LVs used: 6**				
LV Number	**Correl.of LV with property**	**Regression Coefficient**	**Std. error of R.C**	**t-value Lev.%**	**Sig.**
1	0.9216	3.706	0.0121	305.15	0.00
2	0.2746	1.104	0.0121	90.93	0.00
3	0.2303	0.9262	0.0121	76.27	0.00
4	0.1359	0.5466	0.0121	45.01	0.00
5	0.0547	0.219	0.0121	18.11	0.00
6	0.0247	0.09913	0.0121	8.16	0.00
Std Error of Prediction:		Estimate = 0.01429		Actual = 0.2324	
Multiple Correlation		Estimate = 0.9999			
Mean Property Value		Estimate = 3.044			
% Variance (R squared)		Estimate = 99.9836			
Std Error of Estimate (SEE)		Estimate = 0.01214			
F-value		Estimate = 1.827e+004			

2. Moisture	**Number of LVs used: 6**				
LV Number	**Correl.of LV with property**	**Regression Coefficient**	**Std. error of R.C**	**t-value Lev.%**	**Sig.**
1	0.9074	5.356	0.1012	52.91	0.00
2	0.3308	1.953	0.1012	19.29	0.00
3	0.0724	0.4276	0.1012	4.22	0.03
4	0.1228	0.7247	0.1012	7.16	0.00
5	0.1813	1.07	0.1012	10.57	0.00
6	0.0850	0.5017	0.1012	4.96	0.01
Std Error of Prediction:		Estimate = 0.1172		Actual = .3564	
Multiple Correlation		Estimate = 0.9966			
Mean Property Value		Estimate = 5.868			
% Variance (R squared)		Estimate = 99.3236			
Std Error of Estimate (SEE)		Estimate = 0.1012			
F-value		Estimate = 562.9			

Figures 4 & 5 demonstrates the calibration plots for the estimation of caffeine and moisture for the validation data. The Table 1 gives the prediction report for the component caffeine and moisture

Table 1: Prediction Report

Component	Caffeine				Moisture			
Sample	1	2	3	4	1	2	3	4
Normalization	0.189	0.165	0.148	0.181	0.195	0.173	0.155	0.186
RMS error	5.141e-005A	4.373e-005A	3.338e-005A	3.533e-005A	0.0002A	0.0002A	0.0002A	0.0002A
Peak to peak error	0.0004A	0.0004A	0.0004A	0.00035	0.0018	0.0025	0.0021	0.0023
Total M distance	0.264	0.543	1.113	0.629	0.455	0.306	0.620	0.252
Residual Ratio	1.254	1.187	0.863	0.645	0.576	0.869	1.505	0.858
Actual value	2.400	4.210	3.800	2.240	7.490	6.500	6.830	4.780
Calculated value	2.577	4.207	3.799	2.231	7.457	6.469	6.852	4.904

Conclusions

FTNIR coupled with multivariate data analysis is an extremely versatile technique and is many ways unique. Unlike traditional analysis it does not isolate a specific component in the sample to be tested instead the entire sample is characterized by its absorption properties in the near infrared. The method demonstrated FTNIR as an alternative quick analytical technique for the estimation of moisture and caffeine content in tea samples.

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Identification of components of mint oil in flavored tea

Abstract

Peppermint oil is one of the most extensively used essential oils, both medicinally and commercially. It is an important ingredient in a variety of consumer products ranging from candy to nausea remedies. Minor variations in peppermint oils from different sources can significantly alter the flavour and ultimately the quality of a product. Consequently, careful analysis of peppermint oil components is important to ensure the manufacture of uniform products over time. Gas chromatography (GC) with flame ionization detection (FID) or Mass spectrometric detection is widely used to characterize essential oils. However, the analysis of complex matrices often involves lengthy and time consuming sample preparation steps. The present work describes a technique that requires no sample preparation for the identification of peppermint oil components in flavored tea by GC-MS. An Automated Thermal Desorption (ATD) sampler was coupled to a gas chromatograph equipped with an electron impact ionization (EI) mass spectrometer (MS) and employed to identify the components of peppermint oil.

Sample introduction

0.5 ml of neat peppermint oil was injected into a Tenax GR tube and was loaded into the ATD carousel. The tube was automatically analysed as per the instrumental conditions listed below.

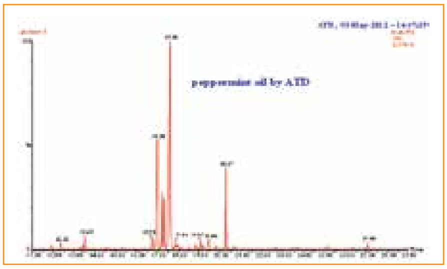
Peppermint tea was directly filled in an empty glass tube to about 0.5 cm length and held in place with glass wool plugs. The same conditions were adopted for the sample.

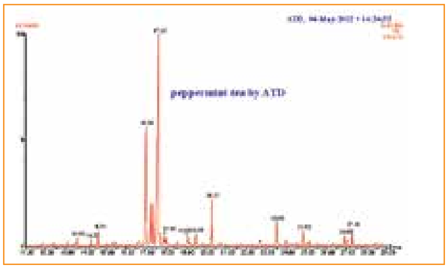
Clrus SQ 8 GCMS with TurboMatrix ATD

Instrumental conditions

Instrument	Clarus™ 680 Gas Chromatograph
Analytical Column	PerkinElmer Elite -5 MS (30 meter, 0.25 mm i.d., 0.25 µm df)
Oven temperature programme	70 °C hold for 2.0 min, 3 °C/min to 250 °C and hold for 2.0 min, runtime is 40 minutes.
Thermal desorber	ATD-650
Trap adsorbent	Tenax TA
Carrier gas	Helium
Tube desorb flow	50.0 ml/min
Tube desorb time	5.0min
Trap low	-30 °C
Trap high	300 °C at 40 °C/min
Inlet split	20 ml/min
Outlet split	30 ml/min
Valve	200 °C
Transfer line	200 °C
Tube temperature	250 °C
MS parameters	SQ-8 mass spectrometer
Source temperature	200 °C
Interface temperature	180 °C
Scan range	m/z 40-400
Scan time	2.01-40 min
Multiplier	1800 V

Analytical results & conclusions





The major components identified in peppermint oil were limonene, cineole, menthone, menthofuran, isomenthone, menthyl acetate, isopulegol, menthol, pulegone and carvone. The major components in the chromatogram for peppermint tea matched those found in neat peppermint oil.

Conclusion

This note presents a simple method for the identification of peppermint oil in various sample matrices. There is no sample preparation step involved, which makes the method time efficient. The sample can be placed directly into a diffusion tube or in an empty tube and the components characterized by ATD-GC-MS. This experimental note demonstrates the ability of the PerkinElmer ATD 650 with Clarus™ 680 GC and SQ8 mass spectrometer to characterize peppermint oil in peppermint tea.

Author
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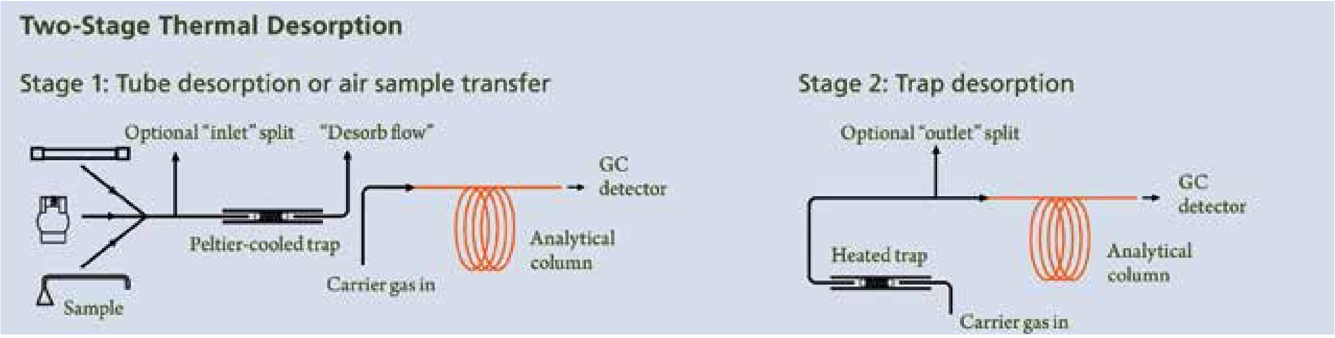


For superior resolution and sensitivity, PerkinElmer instruments feature a two-stage thermal desorption process that concentrates analytes before they are introduced into the gas chromatograph.

Determination of sulphur in insulation oil

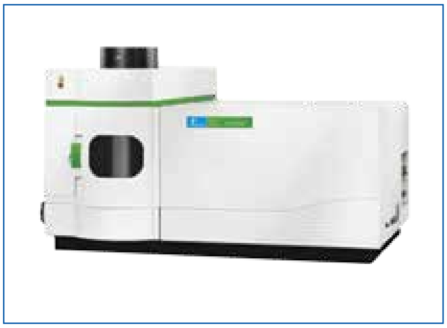
Abstract

The standard test method ASTM D7151-05 for the determination of elements in insulating oils by Inductively Coupled plasma Atomic Emission Spectroscopy does not describe the determination of sulfur. In this experimental note the work was carried out for the determination of Sulfur in insulating Oil using that methodology.

Sample preparation

Diluent: Reagent grade kerosene was used as a diluent solution for all standard and sample solutions.

Base oil: White oil (75cST) was used as base oil since it has a similar room temperature viscosity as the sample and it is free from the analyte of interest.



Optima 8x00 ICP-OES Spectrometer

Internal standard (100µg/g): Oil soluble Yttrium was selected as an internal standard in order to compensate for the viscosity difference between the standards and the samples. 25g of 1000 ppm Yttrium standard was weighted in a 500 mL volumetric flask and 250g kerosene was added as diluent. The solution was mixed thoroughly and transferred into a disperser bottle. The concentration of the internal standard in the prepared solution is 100µg/g.

Preparation of calibration standards and sample:

Blank: Blank was prepared by adding 1 g of base oil, 1g of internal standard and diluted to 25 g with kerosene. Three calibration Standards were prepared by taking 0.25g, 0.5g and 1g of 1000 µg/g stock Organometallic standard. 1 g of base oil and 1 g of internal standard were added and diluted to 25 g with the diluent.

Sample solution was prepared by taking 1g of sample, 1g of internal standard and the solution was diluted to 25g using kerosene as diluent.

Instrumental conditions

Optima™ 8000 Optimized Experimental Conditions	
Plasma gas flow	10 L/min
Auxiliary gas flow	1.4 L/min
Nebulizer gas flow	0.5 L/min
RF power	1500 Watts
Purge Flow	High
Plasma view	Radial
Read parameters	Auto (1 to 5 (Min-Max))
Peristaltic pump flow rate	1 mL/min
Processing Peak	Area
Calibration	Linear Calculated Intercept
Spray Chamber	Cyclonic Baffled
Nebulizer	Low flow GemCone
Injector	Alumina 1.2 mm i.d.
Quartz torch	3-slot

The sample was diluted to fit within the concentration range in the calibration curve.

Analytical results & conclusions

The prepared sample solutions were analyzed on Optima™ 8000 ICP-OES (PerkinElmer, Inc., Shelton, CT, USA) equipped with WinLab32™ for ICP, Version 7.0 software. The three point calibration curve gave a correlation coefficient better than 0.999. After verifying the calibration curve by using a quality control check standard at the midpoint of calibration, the prepared sample solutions were aspirated. As the concentrations were in the high ppm level, radial mode was used for the said analysis. The wavelengths used for the analysis and results are given in table 2. For further confirmation, spiked recoveries were carried out at a concentration of 40 µg/g in sample. The experimental parameters are shown in Table 1 and results of analysis are in Table 2.

Table 2. Results of Analysis

Elements and wavelength	Concentration (µg/g)			
	In sample solution	In spiked solution	% recovery	Concentration in sample
S, 180.669	42.70	82.89	100.4	1067
S, 181.975	42.93	83.15	100.5	1073

Conclusion

These experimental results confirm the effectiveness of the analysis procedure and also demonstrate the capability of the Optima™ 8000 ICP-OES to determine the concentration of Sulfur in difficult matrices such as insulating oil. The excellent spike recoveries showed that the sample preparation protocol was excellent and that the matrix is not interfering with the final results. This experimental note proves that ASTM test method D7151-05 can be followed for the determination of Sulfur in lubricating oils.

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New arrivals

Thermo Mechanical Analyzer TMA 4000

The PerkinElmer TMA 4000 is a uniquely designed thermo mechanical analyzer (TMA) designed for optimal performance in measuring the coefficient of thermal expansion (CTE) in materials. This rugged and easy-to-use design uses both electronics and a unique Archimedean float system to control the probe, allowing accurate determination of CTE and of transitions. In these times of budgetary constraints and changes in material requirements driven by regulations like RoHS, understanding how a material changes with temperature is more important than ever. Test methods for TMA are now required for many industries and the TMA 4000 gives you a tool designed to collect this data. Data can be collected from samples in many forms with the TMA 4000's range of probes including expansion, compression, flexure, extension, and volumetric (dilatometry).

Who will be requiring TMA 4000
Because expansion and glass transition (softening) are fundamental properties of materials, TMA is critical to a wide range of industries and products.

Electronics industry
Mismatched thermal expansion can cause failures of laminated board, encapsulated chips, packing, and soldered parts. Too low a softening point can cause material failures when operating temperatures get high.

Food and food packaging
Thermal size changes with temperature affect laminated films, seals, and material volumes. Mouth feel is strongly related to softening points occurring at certain temperatures. Changes in temperature also mean changes in the volume of products enclosed.

Polymers, automotive, and pipelines
expansion and contraction due to heating or cooling can affect whether motors bind, s eals leak, or gaskets fail. Welds in materials like Invar$^{®}$ must be checked to see if welding alters the expansion of the metal.

Events & Updates

PerkinElmer’s good show at ArabLab 2014

PerkinElmer GULF Team successfully participated in ARABLAB 2014 with regional partner Emphor FZCO. The event took place from 17th till 20th March 2014 at Dubai Exhibition Center in UAE.

PerkinElmer participate at this Mecca of trade shows biennially. ArabLab is the largest technology show serving the Middle East & Africa and showcases the very latest laboratory and instrumentation equipment from all the leading manufacturers in the world. The tradeshow saw more than 10000 visitors visiting more than 800 exhibitors.

The event showcased PerkinElmer’s capabilities in Environmental Health protection and detecting the environmental threats. PerkinElmer’s stall saw a large footfall of visitors all four days - thanks to the attractive booth design and the product showcase. PerkinElmer Booth was a Mezzanine floored booth with large attractive posters addressing the segments we cater to. Among product displays the main attraction was the New Generation of GCMS Analysis - AxION iQT™ GC/MS/MS system. The other technologies displayed included ICP OES, Atomic Absorption Spectrometer, DSC, FTIR and Gas Chromatography.

The booth was efficiently manned by the PerkinElmer and Emphor teams; keeping the visitors engaged in value added discussions and product demos. The trade show also brought the channel partners from GULF region together. The close interactions and business growth in the region were the major topics for discussions. These meetings were addressed by Andy Attwood, Barry Hynes, Simon Evans and Sameer Verma.

To summarize, it was a superb display of knowledge and co-ordination within the teams. Sure this trade show will generate quality leads and enhanced business from the region in coming days.

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Fresh

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HUMAN HEALTH

ENVIRONMENTAL HEALTH

COMPREHENSIVE
SERVICE SOLUTIONS
TO MAXIMIZE YOUR LAB
PRODUCTIVITY

PerkinElmer OneSource Service Contracts provide dependable coverage to enhance your productivity.

Avoid unexpected service costs
Unexpected instrument failure not only disrupts laboratory productivity, but can be expensive to fix. Choosing a service contract that provides breakdown cover insures against unexpected costs, saving your laboratory money.

Minimize instrument downtime
Having instruments under a service contract will guarantee a response time from our dedicated engineering team. Highly skilled, they deliver >90% first time fix rate ensuring your laboratory gets up and running as soon as possible.

Extend instrument life
Regular preventive maintenance will ensure your instrumentation is fully optimized, increasing the efficiency of your laboratory.

Choose the plan that's right for your laboratory

Labor
Enhance laboratory productivity by maintaining instrument efficiency; with a priority breakdown response and technical support.

Comprehensive
For laboratories where maximizing instrument uptime is critical to performance

OneSource®
Laboratory Service

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