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Technical System Procedure for Qualitative Screening of Phosphorus, Chlorine, Bromine and Antimony in Consumer Products by X-Ray Fluorescence (XRF) Spectrometry

1. SCOPE AND APPLICABILITY

This Technical System Procedure (TSP) describes the technical steps and procedures for instrument operation and qualitative screening of phosphorus, chlorine, bromine, and antimony in solid samples using X-ray fluorescence (XRF) Spectrometry.

This TSP is based on but also represents a deviation from Environmental Protection Agency (EPA) Method 6200. EPA Method 6200 is applicable to the in situ and intrusive analysis of the 26 analytes for soil and sediment samples that are detected by Field Portable X-ray Fluorescence (FPXRF). This TSP describes the application of non-portable ARL QUANT'X EDXRF Spectrometer as a qualitative detection tool for phosphorus (P), chlorine (Cl), bromine (Br) and antimony (Sb) in consumer products. Due to the high variability of sample matrix, this method is not applicable for quantitative measurement.

2. DEFINITIONS

Below are common abbreviations/acronyms.

• +	Trace Amount Detected
• ++	Major Constituent Detected
• ARF	Authorization Request Form
• ASI	Analytical Services Incorporated
• BFR	Brominated flame retardant
• CCV	Continuing Calibration Verification
• cps/mA	Counts per second per milliamp
• ECL	Environmental Chemistry laboratory

EDXRF	Energy Dispersive X-Ray Fluorescence
EPA	Environmental Protection Agency
eV	Electron Volt
FPXRF	Field Portable X-Ray Fluorescence
High Za	High Za Scan
High Zb	High Zb Scan
ISP	Identify Spectrum Peaks
Kα	K-alpha Spectral Line
Kα/Kβ	K-alpha Spectral Line- K-beta Spectral Line Interference
Kβ	K-beta Spectral Line
keV	Kilo Electron Volt
kV	Kilo Volt
K/L	K to L Spectral Line overlap
K/M	K to M Spectral Line overlap
Lα	L-alpha Spectral Line
L/M	L to M Spectral Line overlap
MB	Method Blank
Mid Zc	Mid Zc Scan
ND	Not Detected
NH_4Br	Ammonium Bromide
OCP	Organochlorine pesticide
OFHC	Copper Thin foil, Oxygen-Free High Conductivity
OPFR	Organophosphate flame retardant
PDBE	Polybrominated diphenyl ether
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzodioxin
PCDF	Polychlorinated dibenzofuran
QSP	Quality System Procedure
QTL	Qualitative Tray List
REM	Roentgen Equivalent Man
ROI	Region of Interest

• SAR	Sample Analysis Request
• SDS	Safety Data Sheet
• TLD	Thermal Luminescent Detector
• TSP	Technical System Procedure
• XRF	X-Ray Fluorescence Spectrometry
• Z	Atomic number

3. PRINCIPLE

The XRF technology described in this method uses a rhodium x-ray tube to irradiate samples with x-rays in the 4-50 kilo volt (kV) range. When a sample is irradiated with x-rays, the source x-rays undergo either scattering or absorption by the sample. When elements within the sample absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and therefore the outer shell electrons emit energy as they fill the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom (EPA Method 6200).

4. INTERFERENCES

The total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally more significant. Some sources of interference are minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below (EPA Method 6200).

- Variations in matrix effects are due to but limited to the physical characteristics of the sample. These variations include but are not limited to such parameters as particle size, homogeneity, density, and surface condition. For heterogeneous consumer product samples, average multiple scans at different locations.
- Inconsistent positioning of the sample in front of the probe window is a potential source of error because the signal intensity decreases as the distance from the radioactive source increases. This error is minimized by maintaining a fixed distance between the x-ray window and each sample. For optimal results, ensure the sampling surface is smooth and level with the x-ray window, with the window area completely covered by the sample.
- Chemical matrix effects result from the presence of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as an x-ray absorption and enhancement and occur if heavy metals are present within the material.
- X-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing an overlapped spectrum. The degree to which a detector resolves the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks. The most common spectrum overlaps involve the K-beta spectral line (Kβ) of element Z-1 (atomic number minus one) with the K-alpha

spectral line (Kα) of element Z (atomic number). This is called the K-alpha-K-beta interference (Kα/Kβ). Since the Kα/Kβ intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, if present at large concentrations may interfere. The effects can be partially corrected mathematically through the use of deconvolution techniques. Other interferences arise from K to L, K to M and L to M spectral line overlaps (K/L, K/M, and L/M), although these overlaps are less common.

- Foam and textile samples can contain high levels of target analytes that have the potential to contaminate equipment and or the laboratory. To prevent cross contamination, it is recommended that samples are prepared and analyzed one at a time. Replace laboratory gloves after handling a sample to prevent sample cross contamination. Wipe the interior surface of the XRF with a low lint laboratory grade tissue if a sample is placed directly on the surface of the XRF analysis window. Lightly wet laboratory tissue with deionized water to aid in removing small particles or fibers. Use an inert compressed gas to help remove small particles and fibers from the instrument analysis chamber. The XRF instrument is demonstrated to be free from interferences and contamination by the analysis of a Method Blank (MB) with each sample batch. If a high-level sample is observed within a batch, it is recommended to confirm the analytical setup with a MB.

5. PRESERVATION AND HOLDING TIMES

Keep samples in the packaging they arrived in and store at ≤6°C when not in use. Samples are stored in sealed glass jars or sealed plastic bags with the sample fully enclosed in aluminum foil. Store samples long term, at ≤6°C as described above.

There is no holding time limit on organophosphate flame retardants (OPFRs), polybrominated diphenyl ethers (PBDEs) and brominated flame retardants (BFRs) in foam and textile samples stored at ≤6°C in the containment vessels.

Keep samples in their containment vessels with the sample fully enclosed in aluminum foil until all analyses are complete and/or results have been accepted. Samples can be moved to room temperature for storage after results have been accepted by requestor.

Consumer product samples are subjected to a short sub-atmospheric vacuum environment when analyzed by XRF and therefore a potential loss of semi-volatile organics substances may occur. Limit repeated analysis of the same subsample and/or monitor the subsample for decrease in signal intensity.

6. EQUIPMENT AND SETUP

6.1. Equipment

- Thermo Fisher Scientific ARL QUANT'X EDXRF Spectrometer
- Edwards Vacuum Pump Model RV8
- Thermo Fisher Scientific, WinTrace Acquisition Manager, Version 7.0 (Build 53)
- Thermo Fisher Scientific WinTrace Method Explorer, Version 7.0 (Build 53)

6.2. Operating Conditions

Sample data are acquired using the Thermo Fisher Scientific Acquisition Manager (Version 7.0). Recommended XRF acquisition properties and analysis conditions are listed in Table 3. Recommended XRF Spectrum processing conditions are listed in Table 4.

7. EQUIPMENT CLEANING

7.1. Disposable Plastic and Metal

Plastic and metal consumables in this classification include XRF sample cups, Thin-Film, sample support carrier frames and scalpels. Use disposable plastics and metals as provided by the manufacture and do not reuse for subsequent sample preparation or analysis.

7.2. Reusable Metal

Properly wash reusable metals after use to prevent interference in subsequent analysis. Equipment in this classification includes reusable scissors. Recommended procedures for cleaning metals are described in Quality System Procedure (QSP): 02.0011.00, Procedure for Washing Glassware for the Analysis of PCDDs/PCDFs, PCBs, OCPs, PBDEs, BFRs, and Other Persistent Organic Pollutants in Environmental Samples.

8. CONSUMABLES

For details on the recommended consumables and supplies, see Table 1.

9. STANDARDS USED

9.1. Instrument Check

Copper Check Standard, Copper Thin foil, Oxygen-Free High Conductivity (OFHC), is used for the daily XRF Energy Adjustment.

9.2. Method Blank (MB)

Materials that are used as the MB include: (1) the Analytical Services Incorporated (ASI) Standard Number 1, see Table 2; or (2) the analyte-free foam, designated as Blank Foam A.

9.3. Continuing Calibration Verification (CCV)

The method uses ASI Standard Number 4 as the CCV, see Table 2.

9.4. Method Creation

For details on the suggested method creation standards see Table 2.

10. PROCEDURE

10.1. Batch Quality Control (QC) Requirements

A sample batch is defined as up to 25 samples, including Quality Control (QC) samples. Multiple analyses of a sample (i.e., front, and back and/or multicomponent foam) are not included in the batch size. Analyze a MB and perform a CCV analysis prior to analyzing samples. In addition, analyze a selected Sample Duplicate for each batch of samples. Analyze a second MB followed by a CCV at the end of each batch of samples.

- MB results demonstrates instrument cleanness and absence of background and/or carryover interferences. It is recommended that a MB is analyzed after the detection of high levels of phosphorus, chlorine, bromine and/or antimony to confirm instrument cleanness.

- Ensure CCV results for ASI Standard Number 4 are within ±30% of the values listed in Table 2. Stop analysis if CCV results fall outside of the defined range, and take corrective action by following QSP: 02.0037.00, Corrective Actions.

- The Sample Duplicate shows reproducibility of the spectral results to confirm sample handling and instrument precision. Note any failures to meet this criterion due to an unavoidable lack of sample homogeneity in the case narrative of the final report.

10.2. Instrument QC requirements

The EDXRF instrument undergoes a Daily Energy Adjustment before sample analysis or method creation begins. Daily Energy Adjustment is performed with standard OFHC.

10.3. Sample Preparation

10.3.1. Analyze consumer product samples by placing the sample directly over the XRF detector window. Place samples such as plumage, polyester fibers, shredded foam and/or other mixed materials into a XRF sample cup sealed with XRF Thin-Film. Pack sample cells completely to minimize voids. To prevent cross contamination, it is recommended samples are prepared and analyzed one at a time. Subsample large foam or textile samples by removing a representative portion of the sample with a scalpel or scissors.

10.3.2. It is recommended to analyze both the exterior and interior facing surfaces of heterogenous textile samples (i.e., fabrics, batting, and barrier materials).

10.3.3. Analyze large samples of foam directly or compress a subsample of the foam into a XRF sample cup sealed with XRF Thin-Film. Separate heterogeneous foams (such as shredded foam or rebounded foam) into individual constitute foams and analyze individually when possible. For heterogeneous samples, it is recommended to average the results of the constitute components when reporting results.

10.4. Sample Analysis

10.4.1. Energy Adjustment of EDXRF Spectrometer

(1) Power on instrument at rear panel and ensure the x-ray safety key is in the “Enable” position (performed at initial instrument startup only).

(2) Access the lap top computer with the current username and password.

(3) Open sample chamber door and place energy adjustment standard (OFHC) over XRF detector window. Ensure that the window aperture is completely covered by the copper plate.

(4) Open "**Acquisition Manager**" software and navigate to **File > New > Qualitative Tray List**.

(5) On the Qualitative Tray List (QTL) window navigate to **Analyze > Energy Adjustment**.

(6) Select "**OK**" on the Start Energy Adjustment window with the preset settings.

(7) When the message "Energy Adjustment has completed." appears, select "**OK**". Then navigate to **File > Save Report…** Reports are located in the C:\Quant'x\Reports\ folder. Use "Energy Adj Month Day Year" file name format and save Energy Adjustment Report.

10.4.2. **Sample Set-up and Analysis**

(1) Before the analysis of samples, wipe the interior surface of the XRF with a lint free laboratory grade tissue, lightly wet with deionized water to remove any small fibers or other material and ensure surface is dry.

Note: Do not use excessive deionized water to avoid damage to the instrument electronics.

(2) Place sample in XRF analysis chamber and position sample over the XRF detector window. Place sample in a vented XRF cup enclosure or between two XRF Thin-Film Window Carriers when sample size is limited. It is recommended to analyze a subsample due to the potential loss of semivolatile compounds. Close XRF sample chamber door.

(3) Navigate to **File > New > Quantitative Tray List**.

Note: This method is only applicable for qualitative detection.

(4) Enter Environmental Chemistry Laboratory (ECL) number or other identifying sample name in the sample field (i.e., ARF No. followed by ECL No.) and load current analysis method located at C:\Quant'x\Methods\ by selecting the page icon "🗋" in the method field. All scans and sample analysis reports are stored in the method file.

(5) To start the analysis, move the cursor to the next tray list field and wait for method to load. Start analysis by selecting "**Go**".

(6) Once the sample analysis is complete, check the position of any small fabric or foam samples to ensure the sample did not move off the XRF detector window during evacuation or pressurization of the analysis chamber.

(7) Print EDXRF Analysis Report page after all 3 scans are complete. Locate the analysis report by date/time and sample name and open the file from the "Method Explorer" window. Navigate to **View > Method Tree > Analysis Reports**. Sample analysis results are saved for each sample by saving the method file after each sample analysis is complete.

(8) To view analysis spectra, navigate to the "**Spectra**" subfolder under Method Contents and expand.

(9) Locate the analyzed samples and open each XRF scan one at a time: High Zb scan (High Zb), Low Zb scan (Low Zb), and Mid Zc scan (Mid Zc). Select the

"**Identify Spectrum Peaks**" (ISP, red fingerprint icon) to label the spectrum peaks by element name.

(10) Select "**Toggle KLM Markers**" (KLM icon) and press **Ctrl + Left** or **Ctrl + Right arrow key** to move the KLM marker position in alignment with the peaks in the acquired spectrum to identify and confirm each element of interest (P, Cl, Br and Sb).

(11) For subsequent sample analysis, return to the "Acquisition Manager-MTL" window and select the "**View Sample List**" icon. Enter identifying sample name and proceed with analysis as described above. Use unique sample analysis names ending in _01, _02 and/or other identifying sample characteristics if multiple scans are performed on a sample.

10.5. Data Analysis

Data analysis is performed using Thermo Fisher Scientific's WinTrace Method Explorer, Version 7.0.

10.5.1. Review the estimated P, Cl, Br and Sb levels reported on each sample EDXRF Analysis Report page.

10.5.2. Review each spectrum acquired for P, Cl, Br and Sb for element identification. It is recommended analyst use the KLM markers function to identify the spectral peak(s) in each Region of Interest (ROI) corresponding to P, Cl, Br and Sb.

10.5.3. Assign a result of Not Detected (ND), Trace Amount Detected (+) or Major Constituent Detected (++) on the Laboratory Report for Elemental Screening by XRF.

10.5.4. Follow pertinent instructions in QSP: 02.0053.00, Data Upload, Report Generation and Editing Using ECL LIMS to upload, process the data, and to print out a draft report packet.

10.5.5. Use FRM 07.0322.00, XRF Sample Analysis Data Package Review Checklist to review and confirm the data in the report packet and submit the packet to an approved staff member, and the supervisor for review.

10.5.6. A final report is regenerated in LIMS when corrections are required. The corrected final report is then, reviewed and confirmed by the analyst and approved staff and approved for release by the supervisor. The final report is signed by the analysts, the reviewer, and the supervisor.

10.6. Data Reporting

Record data relevant to sample preparation and analysis on form (FRM): 07.0034.01, Sample Analysis by X-Ray Fluorescence Spectrometry (XRF) and/or laboratory notebook.

A complete report packet includes the following printouts and/or copies:

10.6.1. Laboratory Report - Elemental Screening by XRF

10.6.2. FRM: 07.0145.00, Authorization Request Form (ARF)

10.6.3. FRM: 07.0146.00, Sample Analysis Request (SAR) form

10.6.4. FRM: 07.0071.00, Sample Receipt Checklist documentation

10.6.5. Requestor’s version of chain-of-custody/sample identification documents when applicable

10.6.6. Daily Energy Adjustment Report

10.6.7. EDXRF Analysis Report and four ROIs (XRF spectra) for each QC sample and customer sample analysis

10.6.8. Other pertinent sample and/or laboratory documentation as deemed necessary

10.7. Method Creation

Initial Method Creation uses 5 to 6 concentration points for each element (P, Cl, Br and Sb) to construct calibration curves using the recommended method creation standards, analysis condition and acquisition properties listed in Table 2, 3 and 4. Create or update the method every two years or when deemed necessary by analyst. It is recommended to reference WinTrace User’s Guide Version 4.0: Setting up a Method (Chapter 3) and Create a Standards Library (Chapter 4) for method creation guidence in addition to the steps listed below.

(1) Open “**Method Explorer**” and navigate to “**Method Contents**”.

(2) Navigate to and select “**Elemental Peak Profiles**” in the method tree on the left side of the screen.

(3) Open tab “**Spectrum**” and choose “**Import…**”

(4) Select the file for the element being added to the calibration for the correct filter, voltage, and count rate conditions (e.g., Cl K Series 8 kV Cellulose Reference low rate).

(5) Repeat above steps for the elements and all conditions being added.

(6) Navigate to “**Analytes and Conditions**” on the left side of the screen. Remove **Mid Zb** row from the bottom with the right click and “**Delete**”.

(7) Open tab “**Conditions**” and choose “**Add**”.

(8) Choose the following conditions: Low Zb, Mid Zc and High Zb.

(9) Select Cl and P for Low Zb, Br for Mid Zc and Sb for High Zb respectfully.

(10) Navigate to “**Calibration**” field on the left and enter standards information as is provided in Table 2. Files from C:\Quant’x\Standards\ are opened by choosing “**Calibration**” and then select “**Add Standard**”.

(11) Save the method by clicking “**File**” and then select “**Save as**”.

(12) Then select “**Calibration**” tab on top and choose “**Calibrate**”, place the standard on the instrument window, one after another when requested by “**Method Explorer**” program.

(13) Save method again once all the scans are complete.

10.8. Method Performance

Method performance data are recorded and tracked on control charts by monitoring CCV results. In addition, data for method creation standards are verified and tracked (i.e., analyte signal (cps/mA) and/or contamination) on control charts according to established procedures.

11. MAINTENANCE AND TROUBLE SHOOTING

Maintenance required on an as-needed basis, such as changing mechanical pump oil, cleaning XRF air intake screens and testing the interlock circuit. Record maintenance performed in the XRF maintenance logbook.

See Chapter 8 “Periodic Maintenance” of Quant’x Technical Manual.

12. REFERENCES

12.1. United States Environmental Protection Agency, “Method 6200 – Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment”, SW-846, Revision 0, February 2007. https://www.epa.gov/hw-sw846 (Accessed on September 7, 2021).

12.2. United States Environmental Protection Agency, “Chapter One of the SW-846 Compendium: Project Quality Assurance and Quality Control”, Hazardous Waste Test methods / SW-846, Revision 2, July 2014. https://www.epa.gov/hw-sw846 (Accessed on September 7, 2021).

12.3. United States Environmental Protection Agency, “Chapter Three of the SW-846 Compendium: Inorganic Analytes”, Hazardous Waste Test Methods / SW-846, Revision 6, December 2018. https://www.epa.gov/hw-sw846 (Accessed on September 7, 2021).

12.4. Petreas, M., Gill, R., Takaku-Pugh, S., Lytle, E., Parry, E., Wang, M., et al., 2016. Rapid methodology to screen flame retardants in upholstered furniture for compliance with new California labeling law (SB 1019). Chemosphere 152, 353–359.

12.5. QSP: 02.0011.00, Procedure for Washing Glassware for the Analysis of PCDDs/PCDFs, PCBs, OCPs, PDBEs, BFRs, and Other Persistent Organic Pollutants in Environmental Samples.

12.6. Quant’x Technical Manual, Thermo Fisher Scientific, PN 269-173800, Rev A, thermofisher.com.

12.7. WinTrace User’s Guide version 4.0. Thermo Fisher Scientific, PN 269-173900, Rev A, thermofisher.com.

12.8. QSP: 02.0037.00, Corrective Actions.

12.9. QSP: 02.0053.00, Data Upload, Report Generation and Editing Using ECL LIMS.

12.10. FRM: 07.0034.01, Sample Analysis by X-Ray Fluorescence Spectrometry (XRF).

12.11. FRM: 07.0071.00, Sample Receipt Checklist**.**

12.12. FRM: 07.0145.00, Authorization Request Form (ARF).

12.13. FRM: 07.0146.00, Sample Analysis Request (SAR) form.

12.14. FRM 07.0322.00, XRF Sample Analysis Data Package Review Checklist

13. TABLES

Table 1. Recommended Consumables and Supplies

Name	Description	Supplier/No.	VWR[1] Cat. No.
XRF Sample Cups	Double Open-Ended, Caps, Vented, 30.7 mm O.D. x 22.9 mm High	Chemplex Industries[2] [No. 1330]	15566-158
XRF Thin-Film	Prolene 4.0 µm, Thin-Film Continuous Roll	Chemplex Industries [No. 416]	15566-030
XRF SpectroMembrane	Prolene 4.0 µm, XRF Thin-Film Sample Support Carrier Frames	Chemplex Industries [No. 3018]	89079-840
Vacuum Pump Oil	Mechanical Vacuum Pump Oil, Ultra Grade 19 Oil	Edwards [No. H11025015]	EVH110-25-015
Oil Mist Filter	EMF 10 Mist Filter (RV8 Pump)	Edwards [No. A462-26-000]	EVA462-26-000
Kimwipes	Kimberley-Clark Professional KIMTECH Science, Delicate Task Wipers	Kimberly-Clark [No. 34256]	21905-049
Aluminum Foil	Boardwalk, Extra Heavy Duty Aluminum Foil, 18 in x1000 ft	Boardwalk [No. BWK7136]	89125-940
Reclosable Poly Bags	12 in x 15in x 4 mil RedLine Reclosable Polyethylene Bags	MiniGrip [No. MGRL4P1215]	46300-562
Disposable Scalpels	Swann-Morton Disposable Scalpel, Stainless Steel, Blade No. 21	Cincinnati Surgical [No. SM0507]	75870-500
Deionized Water	Deionized water or Baker Analyzed, JT Baker LC-MS Grade	Avantor [No. BAKR9831-03]	JT9831-3

[1] vwr.com, [2] chemplex.com

Table 2. Suggested XRF Method Standards

ASI[3] Standard Number	Br (mg/kg)	Cl (mg/kg)	P (mg/kg)	Sb (mg/kg)
1 (071320JP-1)	0	0	0	0
2 (071320JP-2)	50	51	50*	51
3 (071320JP-3)	100	100	100	100
4 (071320JP-4)	500	500	502	498
5 (071320JP-5)	1000	1000	1002	1007
6 (071320JP-6)	2000	2057	2001	2004
7 (071320JP-7)	5000	5044	5004	5053

[3] ASI Standards, Bromine, Chlorine, Phosphorus and Antimony in Polyethylene, 31 mm discs, product code: PLPE8-4ED-31, asistandards.com

*50 mg/kg Phosphorus standard is not used in method creation and/or calibration.

Table 3. Recommended XRF Analysis Conditions and Acquisition Properties

Analyte	Condition	Filter	Voltage	Atmosphere	Count Rate	Live Time	Energy Range
Sb	High Zb	Cu Thick	50 kV	Air	High	100 Sec	0-40 keV
Br	Mid Zc	Pd Thick	28 kV	Air	Medium	100 Sec	0-40 keV
P, Cl	Low Zb	Cellulose	8 kV	Vacuum	Low	100 Sec	0-40 keV

Table 4. Recommended XRF Spectrum Processing Conditions

Analyte	Condition	Line	Processing	Filter Escape Peak	Quant	Lower ROI	Upper ROI	Peak Profiles
Sb	High Zb	Ka	XML	Yes	Yes	25280	31020	Sb wax (Sb K Series)
Br	Mid Zc	Ka	XML	Yes	Yes	11180	13980	NH_4Br (Br K Series)
Cl	Low Zb	Ka	XML	Yes	Yes	1980	3240	Cl (Cl K Series)
P	Low Zb	Ka	XML	Yes	Yes	1380	2560	P (P K Series)

14. REAPPROVAL

Signatures	Date