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HEXAVALENT CHROMIUM DETERMINATION IN ELECTRONIC DEVICE COMPONENTS WITH ETHOS X FOLLOWING IEC METHOD 62321-7-2: 2017

Hexavalent chromium, despite its risk for human health, has been used in industries for a widespread application. The correct disposal of Cr (VI) added materials is crucial and fundamental to protect water and soil from dangerous contamination. Cr (VI) is reported by the Restriction Hazardous Substances (RoHS) and the International Electrotechnical Commission (IEC) has established a specific testing method (IEC 62321-7-2: 2017) to determine Cr (VI) in electronics and polymers. Milestone ETHOS X with MAXI 24 HP rotor was applied in this study to extract Cr(VI) in fully compliance with the official method.

INTRODUCTION

Hexavalent chromium is highly oxidising and the form that is most hazardous to the environment and to health. Despite its risk, it has been used in manufacturing electronics and polymer for its anticorrosive properties as coating of metal, as pigment in plastics/paints and in emerald glass just to mention some of its uses.

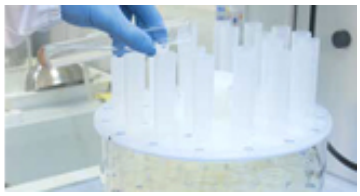
The widespread use of these products led to an increased attention to their impact on the environment. The correct disposal of the Cr (VI) added materials is crucial and fundamental to protect water and soil from dangerous contamination.

For the above reasons Cr (VI) is reported by the Restriction Hazardous Substances (RoHS) and the International Electrotechnical Commission (IEC) has established a specific testing method (IEC 62321-7-2: 2017) to determine Cr (VI) in electronics and polymers.

The method describes procedures to first extract Cr (VI) from polymer and electronic, then to analyse it via colorimetric method reacting Cr(VI) with diphenylcarbazide and measuring at 540 nm.

Sample preparation is the pivotal step of the method, the use of microwave heating has been described in the IEC 62321-7-2: 2017 to enhance the efficiency of the extraction from difficult insoluble/unknown polymers or electronic materials.

The application report will describe the use of Milestone ETHOS X and its capability to overcome specific challenge in the application of the IEC 62321-7-2: 2017 method.



EXPERIMENTAL

INSTRUMENT

The IEC 62321-7-2: 2017 method requires a microwave device capable of maintaining the solution at temperature between 150-160 °C and microwave digestion vessels with pressure capability over 1.0 MPa.
Milestone ETHOS X equipped with MAXI-24 HP is in full compliance with all the requirements and it allows the extraction of 24 samples simultaneously. Despite the study has been performed with MAXI-24 HP, Milestone SK-15 rotor can be used as well for this application. Moreover, Milestone Simultaneous Filtration System (SFS-24) further speeds up the work up procedure described in the method.

Figure 1 – Milestone MAXI 24 HP (left) and SFS24 filtration system (right).

The easyTEMP contactless sensor directly controls the temperature of all samples and solutions, providing accurate temperature feedback and high safety.

PROCEDURE

The procedure is available on Milestone Method (for further information please, refer to the IEC 62321-7-2: 2017 method).

RESULTS AND DISCUSSION

As reported in the method, a spike recovery study is described to evaluate the efficiency of the digestion/extraction system. In this study, several polymer and electric samples were spiked with potassium dichromate solution in order to test the capability of Milestone ETHOS X to achieve good recovery. In table 1 was reported the microwave run used according to the method requirements.

Table 1 – ETHOS X microwave digestion program

STEP	TIME	T	POWER
1	00:15:00	155 °C	1800 W
2	01:30:00	155 °C	1800 W

ETHOS X was able to keep the set temperature of 155°C for 90 min. as reported by the method. easyTEMP sensor ensure perfect temperature control in all the vessel reading the internal temperature and providing simultaneous feedback for the power emission.

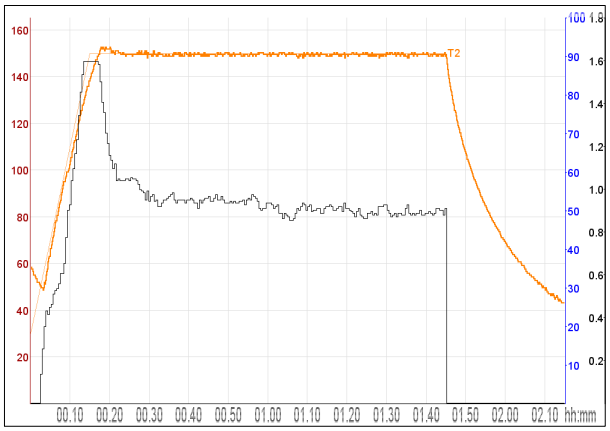


Figure 2 – ETHOS X microwave run result.

The spike recoveries were calculated according to the formula described in the IEC method and reported below.

$$SR = \frac{SS-US}{SA} \times 100$$

- SR: spike recovery in %
- SS: Cr (VI) concentration in the spiked sample in µg/g
- US: Cr (VI) concentration in the unspiked sample in µg/g
- SA: Cr (VI) concentration used in the spike solution in µg/g

Table 2 shows the recovery results obtained working with different sample at variable spike concentration ranging from 50 to 100 (µg/g)

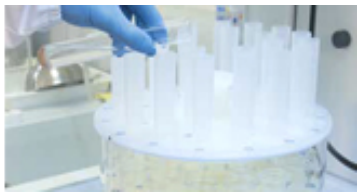


Table 2 – Milestone ETHOS X recovery study with MAXI 24 HP

Sample	Sample Amount (g)	Spike (µg/g)	Average Spike Recovery (%)* n=3	RSD (%) n=3
PP-1	0.1454	100	82	2.3
PP-2	0.1478	50	80	2.6
ABS-1	0.1479	100	103	1.8
ABS-2	0.1498	50	89	2.1
PVC-1	0.1614	100	94	2.4
PVC-2	0.1509	50	93	1.8
Electr. board-1	0.1548	100	88	2.4
Electr. board-2	0.1665	50	79	2.9

**The recovery has been calculated according to the formula reported in the IEC 62321-7-2: 2017 method*

The recoveries were good for all the samples tested at different spike level.

CONCLUSIONS

ETHOS X enables simultaneous digestion and extraction of up to 24 samples. The data shown in this work demonstrate that ETHOS X equipped with MAXI 24 HP is the right combination to provide high quality data with an ultimate ease of use. Thanks to ETHOS X, the applicability of the IEC 62321-7-2: 2017 is easy, cost effective and enhances the throughput on this specific analysis.

ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.