# Microwave digestion in testing of metal elements in lubricating grease

## 1. Introduction

Lubricating grease plays an important part in industry engineering. It mainly used to reduce the friction, fill gaps and prevent rust. Lubricating grease is mainly composed of base oil, thickener and additives. The determination of metal elements insides lubricating grease not only helps to evaluating the quality, lifespan of the products but also place an important role in product producer's QC/QA work.

## 2. Instrument and reagents

Instrument:

The digestions were carried out with M6 microwave digestion system and HP16 high pressure digestion vessels.

M6 microwave digestion system

HP16 rotor


G-160 hot block

Reagent:

$HNO_3$ (GR)

## 3. Method

1. Weigh 0.2g lubricating grease sample into the sample cup.
2. Add $HNO_3$ into the sample cup and then place the vessel on hot block at 120$^0$C for 30 min to conduct sample pretreatment.
3. Wait until no more yellow smoke can be observed then cool the vessel to room temperature,
4. Seal the vessel and place the rotor inside cavity to conduct microwave digestion. The digestion program is listed in Table1.

Table 1. Microwave digestion method

| Step | Setting temperature(℃) | Ramp time (min) | Temperature holding (min) |
|---|---|---|---|
| 1 | 120 | 10 | 2 |
| 2 | 180 | 8 | 2 |
| 3 | 220 | 8 | 30 |

5. Take the vessels out of the cavity when the temperature falls under 60 ℃.
6. Dilute the sample with deionized water when the temperature of the sample cools to

room temperature.

## 4. Result and discussion

The final digestion solution is clear and transparent as shown in Fig. 1 below.

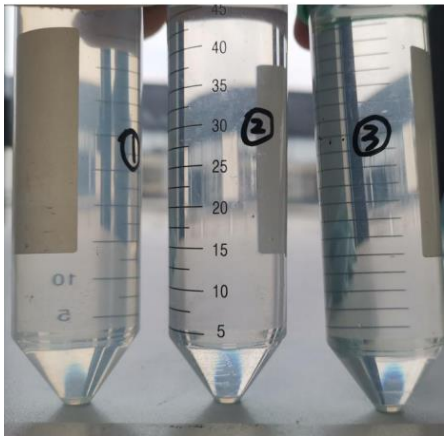


Fig.1 The digestion solution of lubricant

As shown in the digestion result, the lubricant can be digested thoroughly due to the excellent performance of HP 16 high pressure rotor. M6 coupled HP 16 rotor can maintain the reaction temperature and pressure at a relatively high level. Though HP 16 can withstand 4 MPa reaction pressure and auto-vent the pressure when the reaction pressure excesses the working limitation, pre-treatment is still conducted before the air-tight reaction. This is because that the lubricant is made of polymer and the molecular weight ranges various from one to another, so the reaction pressure should be kept in mind. To ensure there is no auto venting and element lose during the digestion process, it is a must to conduct the pre-treatment before the air-tight microwave digestion process.

## 5. Conclusion

Preekem's M6 microwave digestion system coupled with HP 16 rotor can be applied in the digestion of lubricant. Thanks to the advanced full vessel IR R-temp and precise pressure control unit, M6 can ensure the safe and precise sample digestion during the experiment.