TOTAL METALS DIGESTION PREP CHECK LIST
(MILESTONE MICROWAVE)

BATCH ID: _____ PREP ANALYST: _____

START TIME: _____ END TIME: _____

1. ___ CHECK BACKLOG FOR SAMPLES
2. ___ CHOOSE UP TO 20 SAMPLES THAT HAVE BEEN CONDITIONED (DRIED & GROUND).
3. ___ PREPARE A PREP SHEET IN THE LIMS PREP WORKSHEET MANAGER & AN EXCEL SPREAD SHEET FROM THE METALS TOTAL DIGESTION FOLDER (FORM MT-TD).
4. REVIEW ALL PERSONAL PROTECTION EQUIPMENT INCLUDING:
    a. ____Ca GLUCONATE _____ EXPIRATION DATE
    b. ____HF TEST STRIPS _____ LOT & EXPIRATION DATE
    c. ____ LAB COAT
    d. ____RUBBER APRON
    e. ____ SILVER SHIELD SLEEVES
    f. GLOVES, 18MM
        i. ___ NON-LATEX
        ii. ___ BUTYL, NEOPRENE OR HEAVY NITRILE
    g. ___ FACE SHIELD IN CONJUNCTION WITH FUME HOOD SASH
    h. ___ CLOSED-TOED SHOES
    i. ___ LONG PANTS, LONG SLEEVE SHIRT, HIGH NECK

5. ___ WEIGH APPROXIMATELY 0.250 g OF SAMPLE DRIED @ 60 °C INTO ITS CORRESPONDING MICROWAVE VESSEL.
6. ___ ADD SPIKES TO THE CORRESPONDING LABELED MICROWAVE VESSEL.

    ____ 1000 uL SED SPK 4A

    ____ 1000 uL SED SPK 4B

    ____ 2000 uL ICPMINA

7. ___ ADD 2 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

    ___ ADD 2 mL OF NITRIC ACID (HNO_3).

8. ___ WAIT 1 HOUR UNTIL EFFERVESCENCE HAS SUBSIDED OR FINISHED.
9. ___ ADD 1 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

    ___ ADD 6 mL OF NITRIC ACID (HNO_3).

10. ___ CAP VESSELS AND PLACE SAMPLES IN VENTED OVEN FOR 1 HR. AT 60 °C.
11. ___ WEIGH MICROWAVE VESSELS AND LOAD VESSELS INTO CAROUSELS.
12. ___ LOAD “FIRST SESSION” PROGRAM & MICROWAVE.
13. ___ WEIGH (COOLED) MICROWAVE VESSELS.
14. ___ ADD 1 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

    ___ ADD 1 mL OF NITRIC ACID (HNO_3).

15. ___ WAIT UNTIL EFFERVESCENCE HAS SUBSIDED OR FINISHED. IF REACTION IS EXCESSIVE, CAP & PLACE IN CAROUSEL.

16. ___ WEIGH MICROWAVE VESSELS AND LOAD VESSELS INTO CAROUSELS.
17. ___ LOAD “SECOND SESSION” PROGRAM & MICROWAVE.
18. ___ WEIGH (COOLED) MICROWAVE VESSELS.
19. ___ DON HF PPE AND ADD 3 mL OF HYDROFLUORIC ACID (HF).
20. ___ WEIGH MICROWAVE VESSELS.
21. ___ LOAD “THIRD SESSION" PROGRAM & MICROWAVE.
22. ___ WEIGH (COOLED) MICROWAVE VESSELS.
23. ___ DON HF PPE AND ADD 3 mL HCl. SECURE VESSELS BACK INTO WEDGES AND PLACE WEDGES IN VENTED 60 °C OVEN FOR 2 HRS.
24. ___ ALLOW VESSELS TO COOL.
25. ___ DON HF PPE AND TRANSFER SAMPLES TO CORRESPONDING LABELED HOTBLOCK VESSEL.
26. ___ DON HF PPE AND ADD 4 mL of 5 % BORIC ACID SOLUTION TO EACH TUBE.
27. ___ FILL TO 50 mL VOLUME WITH CCB (2 % HNO_3 / 1 % HCl).
28. ___ HEAT VESSELS FOR 4 HRS. AT 95 °C / (< 15 mL). **DOCUMENT BLOCK TEMPERATURE (1)**.
29. ___ SLOWLY ADD 1 mL H_2O_2 AND 1 mL HNO_3
30. ___ HEAT VESSELS FOR 1 HR. AT 95 ˚C. **DOCUMENT BLOCK TEMPERATURE (2)**.
31. ___ BRING TO 50 mL VOLUME WITH CCB (2 % HNO_3 / 1 % HCl).

32. ___ MAKE A 50 mL [TOTAL VOLUME] DILUTION OF 2 (ALL BATCH QC & SAMPLES) WITH CCB (2 % HNO_3 / 1 % HCl). HEAT DILUTED VESSELS FOR 4 HRS. AT 95 °C. **DOCUMENT BLOCK TEMPERATURE (3)**.

33. ___ FILL TO 50 mL VOLUME WITH CCB (2 % HNO_3 / 1 % HCl).

34. ___ COMPLETE LIMS PREP SHEET.

NOTES: