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. ___ WEIGH APPROXIMATELY 0.250 g OF SAMPLE DRIED @ 60 °C INTO ITS CORRESPONDING MICROWAVE VESSEL.
5. ___ ADD SPIKES TO THE CORRESPONDING LABELED MICROWAVE VESSEL.
    - ____ 1000 uL SED SPK 4A
    - ____ 1000 uL SED SPK 4B
    - ____ 2000 uL ICPMINA
6. ___ ADD 2 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

   ___ ADD 2 mL OF NITRIC ACID (HNO_3).
7. ___ WAIT UNTIL EFFERVESCENCE HAS SUBSIDED OR FINISHED. IF REACTION IS EXCESSIVE, CAP & PLACE IN CAROUSEL. PLACE SAMPLES IN VENTED OVEN FOR 1 HR. AT 60 °C.

8. ___ ADD 1 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

   ___ ADD 6 mL OF NITRIC ACID (HNO_3).

9. ___ WAIT UNTIL EFFERVESCENCE HAS SUBSIDED OR FINISHED. IF REACTION IS EXCESSIVE, CAP & PLACE IN CAROUSEL. PLACE SAMPLES IN VENTED OVEN FOR 1 HR. AT 60 °C.

10. ___ WEIGH MICROWAVE VESSELS AND LOAD VESSELS INTO CAROUSELS.

11. ___ LOAD “FIRST SESSION” PROGRAM & MICROWAVE.

12. ___ WEIGH (COOLED) MICROWAVE VESSELS.

13. ___ ADD 1 mL OF 30 % HYDROGEN PEROXIDE (H_2O_2).

    ___ ADD 1 mL OF NITRIC ACID (HNO_3).

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

15. ___ WEIGH MICROWAVE VESSELS AND LOAD VESSELS INTO CAROUSELS.

16. ___ LOAD “SECOND SESSION” PROGRAM & MICROWAVE.

17. ___ WEIGH (COOLED) MICROWAVE VESSELS.

18. ___ ADD 3 mL OF HYDROFLUORIC ACID (HF).

19. ___ WEIGH MICROWAVE VESSELS.

20. ___ LOAD “THIRD SESSION" PROGRAM & MICROWAVE.

21. ___ WEIGH (COOLED) MICROWAVE VESSELS.

22. ___ ADD 3 mL HCl. SECURE VESSELS BACK INTO WEDGES AND PLACE WEDGES IN VENTED 60 °C OVEN FOR 2 HRS.

23. ___ ALLOW VESSELS TO COOL.

24. ___ TRANSFER SAMPLES TO CORRESPONDING LABELED HOTBLOCK VESSEL.

25. ___ ADD 4 mL of 5 % BORIC ACID SOLUTION TO EACH TUBE.

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

27. ___ HEAT VESSELS FOR 4 HRS. AT 95 °C / (< 15 mL). **DOCUMENT BLOCK TEMPERATURE (1)**.

28. ___ SLOWLY ADD 1 mL H_2O_2 AND 1 mL HNO_3

29. ___ HEAT VESSELS FOR 1 HR. AT 95 °C. **DOCUMENT BLOCK TEMPERATURE (2)**.

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

31. ___ MAKE A 50 mL [TOTAL VOLUME] DILUTION OF 2 (ALL BATCH QC & SAMPLES) WITH CCB (2 % HNO_3 / 1 % HCl) AND A HOT BLOCK DIGESTION BLANK (CCB). HEAT VESSELS FOR 4 HRS. AT 95 °C. **DOCUMENT BLOCK TEMPERATURE (3)**. A HOTBLOCK D-BLK (CCB SOLUTION ONLY) MUST BE PREPARED AS WELL.

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

33. ___ COMPLETE LIMS PREP SHEET.

NOTES: