Ethanolamines/Glycol Ethers
Tier 3 Procedure

Section Number: 651
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Title: Running A Boiling Range
References: Glossary Of Equipment Id (Text # 114) Astm D 1078-86

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651 Running A Boiling Range

1.0 Objective:

1.1 To provide a safe and accurate procedure for running boiling ranges.

2.0 Responsibility:

2.1 It is the responsibility of the operations supervisor to ensure adequate equipment, resources, and training are available to conduct this test properly.

2.2 The lab technician is responsible to the operations supervisor to ensure that the equipment involved with this test is maintained and in good working condition. The lab technician must also ensure that this test is run in accordance with astm d 1078-86.

2.3 The lab analyst or operations lab operator is responsible for fully complying with this procedure.

3.0 Safety Notations:

3.1 This test is run using a heater to boil the product. The temperature of the heater gets very hot. Care must be taken when performing this task.

3.2 Be sure not to breath the vapors that are being vented. This test must be conducted under the lab vent hood.

3.3 Check the boiling flask for cracks. If product leaks out of the flask, it may cause a fire. Be sure the bottom of the flask has been charred with tartaric acid. This promotes gentle boiling.

4.0 Equipment:

4.1 Boiling flask, graduated cylinder (100ml), teflon tape and the appropriate thermometer.

5.0 Procedure:

5.1 Clean out the condenser tube to remove any trace of liquid which might interfere with the ibp (initial boiling point). Use the cleaning swab made for this purpose.

5.2 Select a distillation flask that is dry and has been charred on the bottom.

5.0 Procedure: Cont'd.

5.3 Fill the graduated cylinder to 100 ml and transfer this product to the boiling flask.

5.4 Insert side arm of flask into condenser tube and wrap opening with 2 or 3 turns of teflon tape. Adjust the flask so that the neck of the flask is straight up and down.

5.5 Check the thermometer.

5.6 Check type against list on line 651.7 of this procedure.

5.7 Make sure that there are no gaps in the mercury.

5.8 Put the 100 ml graduated cylinder under the lower end of the condenser tube to revive distillate.

5.9 Be sure cooling water is flowing through condenser.

5.10 Turn on heat source.

5.11 Record the temperature at which the first drop falls into the graduated cylinder. This is the ibp. The ibp should occur within 15 minutes of applying the heat source.

5.12 When the distillation is 50% complete, record the midpoint temperature.

5.12 The temperature of the dry point must be recorded. The dry point is when the last drop of liquid in the very bottom of the flask boils away. Ignore any liquid which may be on the sides of the flask.

5.13 Immediately after the dry point, cut off the heat source to prevent damage to the flask.

5.14 Move the hot flask away from the heater opening so that it may cool.

5.15 Pour the sample in the cylinder into the proper waste product container. Rinse out the cylinder and put it up to dry.

5.16 After the boiling flask has cooled, rinse it and put it on the drying rack.

5.17 Subtract or add enough from the 50% point to make it equal the correct boiling range. Subtract or add the same amount from the ibp and the dry point. This is the thermometer correction factor.

6.0 Product Boiling Points (Mid Range):

6.1 mea = 170.8
db = 231.0

7.0 Thermometer Type For Different Chemicals:

7.1 mea = 103 c astm 68c
db = 105 c astm 69c

Note: These numbers are written on the back of the thermometers.

7.2 These thermometers will be stored in a rack under the vent hoods.

8.0 Calibration Check:

8.1 Clibration check of the mea and db thermometers is accomplished by comparing readings between a master thermometer (strictly for calibration checks) and a thermometer in use in the lab.

8.2 The heat source for this calibration check is the lab oven. check mea at 170 degrees plus or minus 5 degrees. check db at 210 degrees plus or minus 5 degrees.

8.3 Thermometer id:
651.8.3.1 929948 = mea master thermometer check.
929935 = mea in use thermometer.
92a3855 = db master thermometer check.
91a3772 = db in use thermometer.

9.0 Revisions

9.1	Revision #1	01-19-94	Added Section 8.0
9.2	Revision #2	12-21-94	Yearly Review
	revision 3	12-29-95	Yearly Review