

### Distillation Setup for ASBC Method Beer 25A

These notes contain the suggested ways to process beer samples for diacetyl analysis. The basic Apparatus setup is shown in Figure 0.1



Figure 0.1 Apparatus configuration for distillation of beer samples for diacetyl analysis. Note receiver (in beaker on jack stand) is surrounded by ice. Also note carborundum boiling stones, Antifoam A and carbon dioxide flow meter. (28Jun00 - DSCN0222.pct)

1. Place 500 mL mantle on counter
2. Place 500 mL distiling flask with gas introduction neck in mantle
3. Connect gas neck to output of flow meter, input to  $CO_2$  bottle
4. Open stopcock. No gas flow at this point

5. Weigh out 1 gram of monobasic potassium phosphate  $KH_2PO_4$  and place in bottom of flask
6. Add 25 mL of 0.1N NaOH. Swirl to mix
7. Measure out 250 mL of cold, undecarbonated beer into volumetric flask. Suck foam out with pipet.
8. Add a few carborundum boiling stones to flask.
9. Pour beer in gently. Swirl to mix with buffer.
10. Add about 0.1 mL Antifoam A. The "slick" should be 1 cm in diameter or a little larger.
11. Place distillation column in neck of flask. Secure neck of flask and column with clamps on vertical rod of mantle.
12. Place Claisen adapter at top of column. Stopper top output.
13. Place a ring stand to right of mantle.
14. Attach condenser with cooling connections already made to sloping port on Claisen adapter. Stabilize with clamp on second stand. Orient with hose connections down so that drip tip will be down.
15. Place jack stand under outlet end of condenser.
16. Place 500 mL beaker on jack stand.
17. Place 0.8 mL of 6% hydroxylamine hydrochloride ( 0.6 gram in 9.4 mL water) in the bottom of 30 mL bubbler chamber. Add about 2 mL DI water.
18. Connect bubbler to vacuum adapter, connect vacuum adapter to condenser and raise jack stand so that bubbler is supported by bottom of beaker on jack stand.
19. Check ground glass connections for tightness and turn on  $CO_2$ . Establish a flow reading of 60 on flow meter (144 ml/Min).
20. Wait 5 minutes for air to be swept out of apparatus. Reduce flow to 10 (12 mL/min).
21. Set mantle to 10.

22. After 13 minutes move heat back to 6. There should be foam on surface of beer and some reflux from top of flask but none from column yet. Watch foam situation like a hawk and be prepared to back off heat to 5 or less if there is danger of foam entering the column.
23. After a few minutes, reflux should begin from column. Alcohol in reflux should be enough to collapse the foam. After 18 minutes or so foam should be under control. Advance heat top 7 when foam is under control.
24. By 22 minutes or so drops should be appearing at condenser drip tip at the rate of a bit less than 1 per sec.
25. Collect until 30 mL have accumulated. Note that vacuum adapter tube displaces a fair amount of liquid so that liquid level will have to be well above 30 mL mark on bubbler - about up to bottom of ground glass of adapter. This will take about 50 minutes.
26. Have 100 mL beaker ready. Disconnect bubbler from adapter. Lower jack stand. When stand is low enough that bubbler can be removed do so immediately placing beaker under dip tube so as to collect any additional drops which fall into beaker.
27. Empty bubbler tube into beaker and replace on vacuum adapter. Raise jack stand and large beaker to support.
28. Grab small beaker with ringstand tube clamp (it will just do it) and lower into 80C water bath. Be sure bath is full enough to cover liquid in bottom of beaker.
29. Collect another 5 mL or so of distillate to rinse bubbler. Swirl to collect any dimethylglyoxime left on walls of tube and add to beaker. Total time to this point should be just under an hour from when heat was turned on.
30. Leave in water bath at 80C for 15 minutes.
31. Transfer to boiling water bath until reduced to 15 mL. This should be about another 15 minutes for total of 1:30 to this point. Cool to room temp.
32. Transfer quantitatively to 25 mL volumetric flask using thistle funnel. Leave enough space for addition of 3.3 mL additional reagents.
33. Put 25 mL DI water into a volumetric flask.

34. To each flask add 1.0 mL  $K_2HPO_4$  solution (Reagent c). Allow to stand 5 minutes.
35. Successively add to each flask 0.6 ml  $NH_4OH$  (Reagent d), 2.5 mL saturated tartrate solution (Reagent c) and 0.2mL  $FeSO_4$  (Reagent f).
36. Read diacetyl concentration against blank in 1" cell using DR/4000U User Method 9.

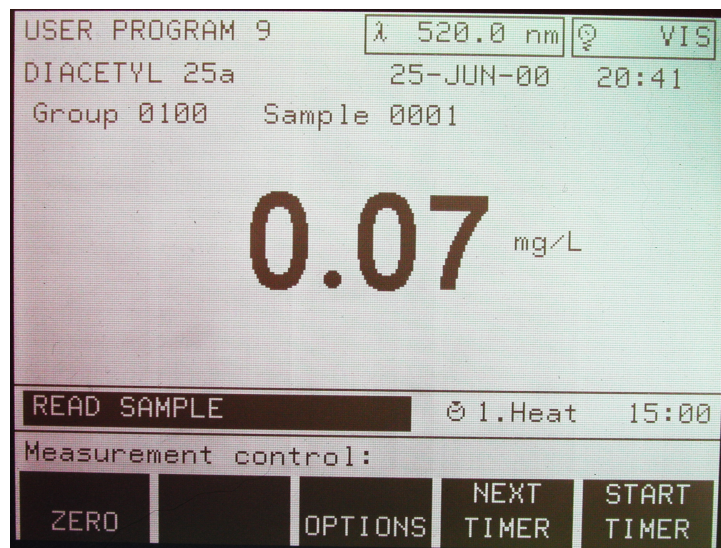


Figure 0.2 Sample screen from spectrophotometer during diacetyl assay of a beer. (28jun00 DSCN0223.PICT)