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MONSANTO STL 8-30-45 DK

R L JENKINS

METHOD OF ANALYSIS FOR TIN TETRAPHENYL IN PYRANOL 1467..

NOTE.. THIS METHOD IS NOT APPLICABLE TO PRODUCT CONTAINING MORE THAN 0.0035 PERCENT WATER

1. WEIGH A 10 ML. -APPROX. 15 G. TO PLUS OR MINUS 0.01 G.- SAMPLE INTO A TARED 125 ML. SEPARATORY FUNNEL.
2. BUBBLE DRY HCL GAS THRU A GLASS TUBE OF SMALL BORE INTO THE SAMPLE FOR 10 MINUTES. A TUBE DRAWN TO AN ALMOST CAPILLARY TIP IS DESIRABLE. LEAVE TIP IN SAMPLE.
3. BUBBLE DRY AIR THRU THE SAMPLE FOR 30 MINUTES TO SWEEP OUT EXCESS HCL. DRY THE AIR OVER DRIERITE. CHECK FOR COMPLETE REMOVAL OF HCL BY ODOR. DO NOT REMOVE AIR LINE FROM FUNNEL.
4. PIPETTE 25 ML. OF DISTILLED WATER INTO THE FUNNEL, ALLOW TO BLOW FOR ONE TO TWO MINUTES AND THEN WITHDRAW AIR LINE.
5. STOPPER FUNNEL AND SHAKE VIGOROUSLY FOR TWO TO THREE MINUTES.
6. ALLOW SEPARATION OF LAYERS AND DECANT BULK OF WATER LAYER INTO A CLEAN DRY 100 ML. BEAKER.
7. PIPETTE INTO THE FUNNEL ANOTHER 25 ML. PORTION OF DISTILLED WATER, SHAKE VIGOROUSLY AS BEFORE, ALLOW TO SEPARATE, DISCARD THE PYRANOL LAYER AND COMBINE THE WATER EXTRACTS.
8. MIX THE WATER EXTRACTS AND PIPETTE A 20 ML. PORTION INTO A 250 ML. ERLLENMEYER FLASK.
9. ADD 50 ML. DISTILLED WATER AND THREE TO FIVE DROPS OF ALCOHOLIC PHENOLPHTHALEIN INDICATOR SOLUTION -1 G. OF INDICATOR TO 100 ML. 95 PERCENT ETHANOL- AND TITRATE WITH 0.01 N. NAOH UNTIL PINK COLOR REMAINS FOR FIVE TO TEN SECONDS.
10. DETERMINE A BLANK TITRATION WITH 0.01 N. NAOH ON 70 ML. OF DISTILLED WATER.
11. CALCULATION.
PERCENT TIN TETRAPHENYL EQUALS $\frac{(\text{NET CC. OF NAOH}) \cdot (\text{N.F. NAOH}) \cdot (40)}{(\text{SAMPLE WT.}) \cdot (1.6)}$

THIS IS THE EQUATION OF CALCULATION AS SUPPLIED TO US BY GENERAL ELECTRIC COMPANY. IT IS EMPIRICAL AND EMBODIES THE 1.6 FACTOR WHICH THEY HAVE FOUND NECESSARY IN ORDER TO OBTAIN A CORRECT RESULT.

NOTES..

AS A SOURCE OF HCL ONE MAY USE EITHER CYLINDER GAS OR IT MAY BE GENERATED. WE HAVE USED THE LATTER. WE GENERATE IT BY DROPPING CONCENTRATED -AQUEOUS- HCL FROM A SEPARATORY FUNNEL INTO 98 PERCENT H2SO4 CONTAINED IN A TWO LITER FILTER FLASK. ADD AQUEOUS HCL SLOWLY AND CAUTIOUSLY. BUBBLE THE GAS THRU 98 PERCENT H2SO4 IN A SPIRAL SCRUBBER, THEN THRU COTTON AND GLASS WOOL. INSPECT ALL RUBBER CONNECTIONS FREQUENTLY AS THEY DETERIORATE READILY AND CAUSE CLOGGING. WE HAVE REGULARLY DETERMINED TWO RESULTS ON SEPARATELY WEIGHED SAMPLES.

THIS AND ALL OTHER METHOD FOLLOWING BY MAIL TODAY

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