Date 8·1·09 Course 2230-L

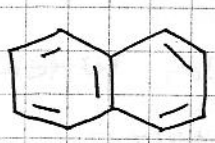
Name Dr. Stassi DiMaggio Section 4

Experiment MELTING POINT DETERMINATION

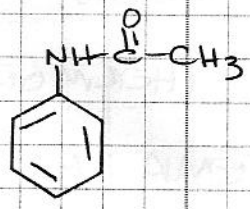
PURPOSE: TO DETERMINE THE IDENTITY OF AN UNKNOWN COMPOUND BASED ON ITS MELTING POINT

EQUATIONS: NONE

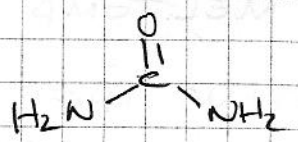
STRUCTURES:



NAPHTHALENE

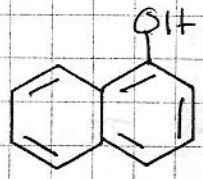


ACETANILIDE

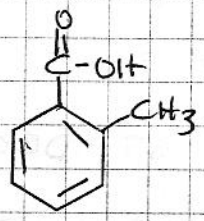


UREA

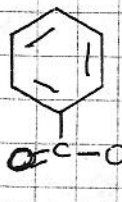
4-NITROTOLUENE



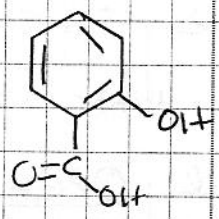
1-NAPHTHOL



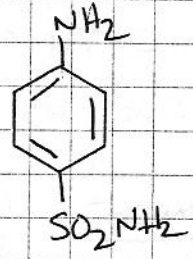
2-METHYL BENZOIC ACID



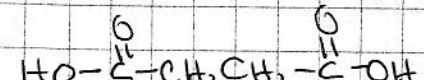
BENZOIC ACID



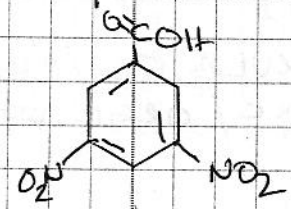
SALICYLIC ACID



SULFANILAMIDE



SUCCINIC ACID



3,5-DINITRO BENZOIC ACID

Date 8·1·09 Course 2230-L

Name DR. STASSI DIMAGGIO Section 4

Experiment MELTING POINT DETERMINATION

PROCEDURE: OBTAIN A SMALL AMOUNT OF SAMPLE IN A CAPILLARY TUBE (<2MM HIGH) AND PACK TO THE BOTTOM OF THE TUBE. REPEAT FOR EACH KNOWN. PLACE SAMPLES IN MELTEMP WITH THERMOMETER AND BEGIN HEATING THE MELTEMP AT A SETTING WHERE THE TEMPERATURE RISES ≈ 1°C/MINUTE.

RECORD MELTING TEMPERATURE RANGE WHEN THE FIRST DROPS FORM TO WHEN THE SAMPLE IS LIQUID. COOL MELTEMP AND REPEAT FOR UNKNOWN. PERFORM A MIXED MELTING POINT FOR THE 3 MOST LIKELY CHOICES FOR THE UNKNOWN.

REFERENCE: CRAGO, K. et al. ORGANIC CHEMISTRY LABORATORY MANUAL, 6TH EDITION WILEY, NEW YORK, 2005, P 32.

WWW.XULA.EDU/CHEMISTRY/DEPARTMENT/ORGANIC/ORGO.HTML

DATA: (1) A SAMPLE OF EACH OF NAPHTHALENE, ACETANILIDE AND UREA WERE OBTAINED

(2) THE SAMPLES WERE GROUND INTO A FINE POWDER

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③ THE SAMPLES WERE EACH COLLECTED IN A CAPILLARY TUBE BY TAPPING THE OPEN END OF THE TUBE ON THE COMPOUND AND TURNING THE TUBE OVER AND PACKING THE SAMPLE TO THE BOTTOM

④ A THERMOMETER WAS PLACED IN THE MELTEMP ALONG WITH THE 3 KNOWN SAMPLES AND THE DIAL WAS SET TO 3. (8:43 AM)

⑤ AFTER 5 MINUTES, THE TEMPERATURE STOPPED INCREASING AND THE DIAL WAS SET TO 4. (8:48 AM)

⑥ AT 8:52 AM THE DIAL WAS INCREASED TO 6 AND THE TEMP WAS INCREASING AT ≈ 1°C / MIN.

	OBSERVATION	1ST DROP FORMED (T °C)	COMPLETELY MELTED (T °C)
ACETANILIDE	WHITE CRYSTALS	109.1°C	112.0 °C
NAPHTHALINE	FLAT, WHITE FLAKES	79.5°C	81.5°C
UREA	OFF-WHITE POWDER	128.8°C	131.0°C
UNKNOWN #342		98.0 °C	101.5 °C
UNK + 1-NAPHTHOL		82.5°C	87.6°C
UNK + 2-METHYL BENZOIC ACID		99.8° C	102.5°C
UNK + ACETANILIDE		95.5°C	100.0°C

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(7) ONCE THE KNOWNS WERE MELTED AND THE TEMPERATURE RECORDED (SEE TABLE) THE MELTEMP WAS COOLED TO 40.0°C

(8) UNKNOWN #342 WAS PREPARED ACCORDING TO PROCEDURE (STEPS 1-3) AND THE MELTING POINT RANGE WAS OBTAINED AND RECORDED

98.0°C - ~~101.0°C~~ 101.5°C
SD

(9) SAMPLES OF PURE 1-NAPHTHOL, 2-METHYL BENZOIC ACID AND ACETANILIDE WERE OBTAINED. SMALL AMOUNTS OF UNK #342 WERE MIXED WITH EACH KNOW AND THE MIXED MELTING POINT OF EACH WAS OBTAINED AND RECORDED (SEE DATA TABLE)

Stass DiMaggio
8/1/09