EXPERMENTAL

EXPERIMENTAL STUDIES

Apparatus:

Borosil make glass wares were used. The burette, pipettes & volumetric flask ware calibrated in accordance with the method described by Vogal[1-2] . The digital analytical balance 'Shimadzu' make, accuracy 0.1 mg, was used for weighing. The weight used for weighting, were calibrated by IRTL, MIDC, Andheri (E), Mumbai, as per method recommended by Scott[3] .

.pH meter:

An Elico make (model LI – 120) pH meter was used for the measurement of pH. Buffer solution, prepared by pH tablets, supplied by Qualigens, Mumbai, were employed for the calibration[4] of pH meter.

Spectrophotometer:

All absorbance were measured on 'Chemito – 120' spectrophotometer using 1 cm quartz cell. The calibrationtion[5] of the spectrophotometer was checked by measuring the absorbance of potassium dichromate solution. The Observed spectra were in good agreement with the spectra reported in the litreture[6] .

Infrared:

All infrared studies carried out on 'Buck – Scientific' model – M500. The instrument was calibrated[7] before use.

Chemicals:

Most of chemicals used, were of AR grade, whenever AR quality were not available, chemically pure grade materials were used. Distilled water, prepared by distillation method or HPLC grade water supplied by SD fine chem. Ltd. Mumbai, was used for the preparation of solution & other purpose.

REAGENT PREPARATION

Preparation of thiocamphor:

Dissolved camphor (20 g) in ethyl alcohol (100ml) and colled to 0°C, added few drops of hydrochloric acid, passed hydrogen sulphide gas continuously for 5-6 hours. The colour of solution changed from yellow orange to deep red, filtered the precipitate. The precipitate was washed with dilute alcohol (!:1) and finely several time with water. Dissolved the precipitate in benzene and filtered through anhydrous sodium sulphate, benzene was evaporated to get octahedral crystal of the compound.

Preparation of Isoamylnitrite:

Dissolved 95 g sodium nitrite in 375 ml of water and cooled up to 0°C. It was treated drop wise with a mixture of 34 ml water, 34 ml of 34 N sulphuric acid and 135 ml of isoamyl alcohol, preciously cooled at 0°C. The mixture was allowed to stand at 0°C for one and half hour, it was transfered in a separatory funnel. The upper layer containing isoamyl nitrite, was removed and filtered on anhydrous sodium sulphate.

Preparation of Isonitrosothiocamphor:

A solution of thiocamphor (17g) in ether (15 ml) was mixed with isoamyl nitrite (12 g) and cooled at 0°C for one hour, sodamide (4 g) suspended in ether (7 ml) was taken in a 250 ml flask fitted with calcium chloride guard tube and was cooled to 0°C. The solution of thiocamphor in isoamyl nitrite was slowly poured into the suspension of sodamide in ether. The red solution of thiocamphor, changed to dark red. The mixture was kept at 0°C for half an hour, then mixed with ice cold water and shaked with ether to remove unchanged isoamylnitrite and thiocamphor. The aquous solution was then treated with acetic acid, whereby violet crystals of isonitrosothiocamphor separated out. The crystal were separated by filtration and washed with water.

The compound was analyzed as anal. Calcd. For C_{10} H_{15} NOS : C 60.86%; H 7.60%; N 7.10%; and S 16.22%., found C 60.80%; H 7.65%; and S 16.20%.

INFRARED STUDIES OF ISONITROSOTHIOCAMPHOR

Infrared spectrum is an important record, which may provide sufficient information about the structure of complex.[10] This technique gives spectrum containing a large number of absorption band from which a wealth of information can be derived about the structure of an organic compound. The absorption of infrared radiations caused the various band with respect to one another.

The infrared spectrum of a molecule, results due to transition between two different energy level. Polyatomic molecule may exhibits more than one fundamental vibrational absorption bands. The infrared spectrum of a substance is determined by the nature, number and relative position of its atom.

Following organic functional groups are identified with the help of Infrared spectrum:

a) **=N – OH group:**

Nitroso compound tend to dimerise, secondary and primary nitroso compound readly rearrange to oxime. In the monomeric state absorbs in the 1621 – 1488 cm^{-1} region[11], but in the solution, they exist preferably as dimers and then absorb near 1290.

The N=O bond in stable nitroso compound is characterized by a stretching vibration in the range[12] of 1621 – 1488 cm^{-1}

b) **>C – CH3 group:**

The mode of >C – CH3 group[13] gives to an absorption at about 2872 cm^{-1}

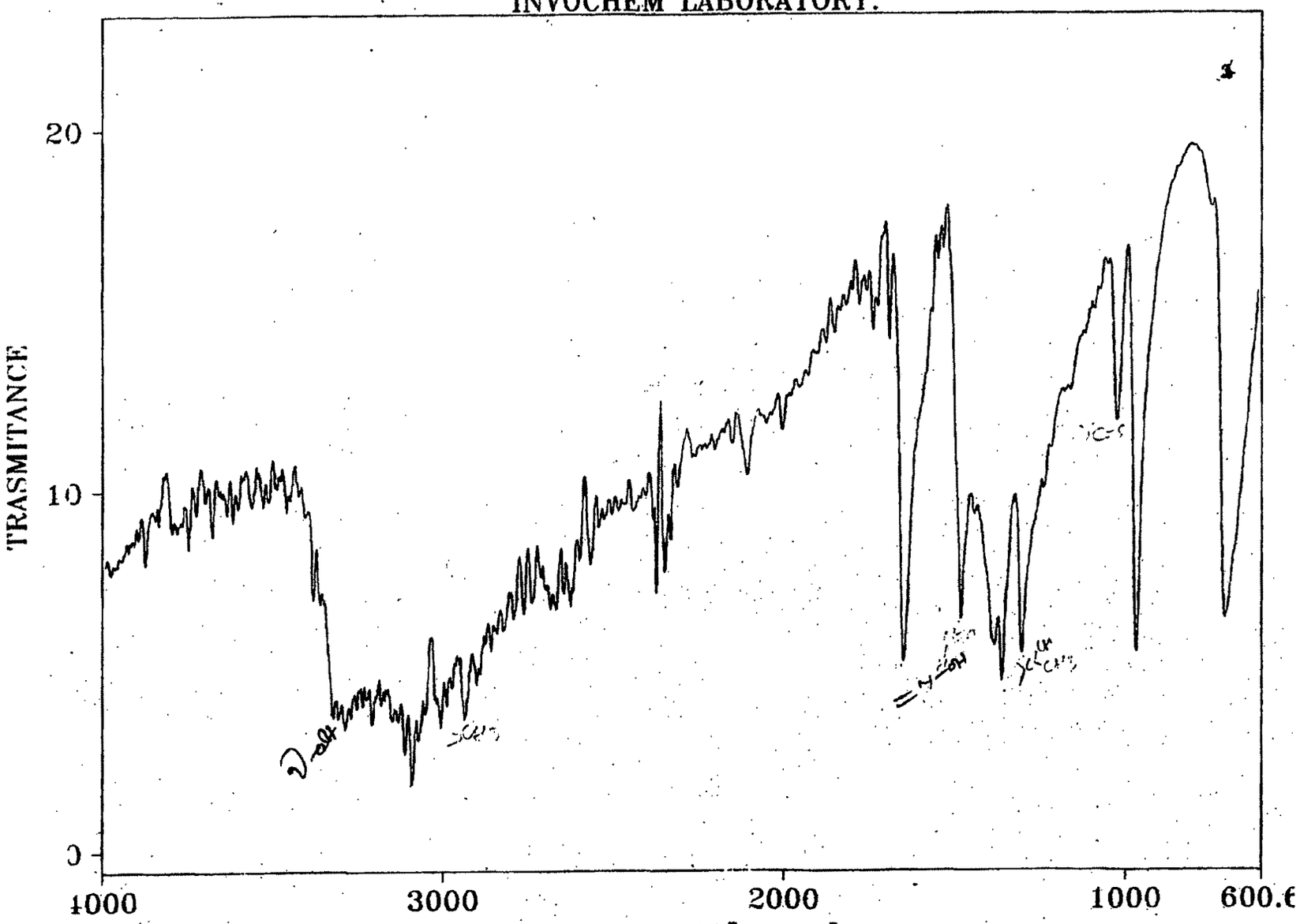
c) > C=S group:

The range of >C=S group[14] occurs in the range of 1400 - 1000 cm^{-1}

d) >C<$^{CH3}_{CH3}$ group :

The frequency of **>C<$^{CH3}_{CH3}$** group is at about[15] 1340 -1460 cm^{-1}

INVOCHEM LABORATORY.



PARTY : SANJAY KUMAR SINGH

SAMPLE : 1

B.NO. :

R.NO. :

DATE : 03/10/03

REFERANCES

1. A. I. Vogal text book of "quantitative inorganic analysis", Longmans Green & co. ltd. London. 1961.
2. Indian Pharmacopoeia 1996, pg. A-8
3. W.W. Schott " Standard methods of Chemical Analysis" Vol. I, Von Nostrend company Inc. 1939.
4. Indian Pharmacopoeia 1996, pg. A-95
5. Indian Pharmacopoeia 1996, pg. A-78
6. S. D. Rose and D. W. Wilson "Specctrovision" 4,10. (1961)
7. Indian Pharmacopoeia 1996, pg. A-74
8. D. C. Sen J. Ind Chem. Soc. 12, 647 (1935)
9. D. C. Sen J. Ind Chem. Soc. 12, 751 (1955)
10. Y. R. Sharma, "Elementary Organic Spectroscopy" pg. 65 (1999)
11. Margareta Avarm & Gh. D. Mateescu. Pg. 312, (1978)
12. Margareta Avarm & Gh. D. Mateescu. Pg. 313, (1978)
13. Y. R. Sharma, "Elementary Organic Spectroscopy" pg. 91 (1999)
14. Margareta Avarm & Gh. D. Mateescu. Pg. 219, (1978)
15. Margareta Avarm & Gh. D. Mateescu. Pg. 124, (1978)