

J. L. Co. (Canada) Ltd

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Visit to Iannesse Eastman Co. by Messrs. Laporte, de Souza & Wade on March 7 & 8
Tar Investigation at Iannesse Eastman Company

150 cigarettes are smoked at a time and the particulate phase collected in a wet ice bath. Temperatures in the burning zone have been found to be 900°C. on puffing, 700°C. on free burning. This appears high compared to other observers. No attention is paid to length of cigarette smoked, nor amount of cc's. of air per puff. The cigarettes are puffed three times per minute. The tars collected are dissolved in methylene chloride. This solvent has been found to not interfere with activity of various polycyclic aromatics such as benzopyrene. $CHCl_3$ and CCl_4 will both react with benzopyrene, usually with aid of light, although $CHCl_3$ will even react, under extreme conditions, in the dark. Ultraviolet light or even ordinary light itself will deactivate benzopyrene and hence all reactions and procedures are carried out under a yellow filter.

The soluble fraction is extracted with 2% sulphuric acid and 2% sodium carbonate solutions (see chart for detailed procedure). From 100 gm. of whole tar 50 gm. of neutral tars are obtained. These neutral tars are subjected to elution chromatography. 20 liters each of hexane benzene, ethylacetate, methanol and pyridine-methanol (90 - 10) are passed through 10 lb. of silica gel under nitrogen which helps to give a positive pressure besides reducing oxidation.

The silica gel is purified by washing with methanol until ultraviolet indicates no further impurities. The gel is heated at 100°C. until it reaches the required activity according to the manufacturer's instructions. Silica gel was chosen because it was most inert of substances. For alumina to be used the acidic substances must be eliminated.

On loading of column vacuum is used for better settling. Then the tars, dissolved in 200 ml. of benzene (methylene chloride is too powerful a solvent) are added to column and the 20 liters of solvent added.

Each elution is taken off and distilled under 2 inches of pressure. All connections are stainless steel and polyethylene. No lubricants nor silicones are used in any stopcocks or connections. The residues are taken up in methylene chloride and filtered to eliminate silica gel particles. The solvent is evaporated off until constant weight is obtained (about 4 hours). 95 - 97% of the tar is recovered by addition of the 5 fractions. The column is finally washed with methanol-water which takes out everything. Finally the pyridine-methanol elution could be replaced by the methanol-water elution. The biggest residue comes down with ethylacetate.

All solvents are purified to 1 ppm impurity.

The respective residues are chromatographed again with the same solvents in the same order and the eluates (about 200 ml. each) checked in a fluorescence analyzer. As the fluorescence falls to a low with the hexane benzene is added. The fluorescence immediately increases. As that falls the next solvent is added and so forth.

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The following compounds have been identified in the hexane fraction:

anthracene
fluoranthrene
pyrene
chrysene
hentriacontane
high mol. wt. ester
high mol. wt. alcohol (sterol)
Coumarin
guaiacol
o. m, p-cresols
2,4-xyleneol
Aliphatic acids
Aromatic acids

No benzpyrene has been found. A curve that looked like benzpyrene was obtained but on rechromatography it was broken up into 4 unidentified polycyclics. This suggests that reports by previous investigators on identification of benzpyrene were possibly erroneous.

It is interesting to note that when 10 mg. of benzpyrene were spiked in the whole tar, 40% showed up in the acidic fraction (identified by infrared) 50% in the hexane and 10% in the benzene fraction. No explanation for its appearance in the acidic fraction has been forwarded.

The gas phase of cigarette smoke was previously investigated. No polycyclics were found. (Hobbs at Duke also has not found polycyclics in the gaseous phase). All the C₁ to C₄ hydrocarbons were identified. (Hobbs & P.M. Co. have found COS but Tennessee have not). No acrolein present but it would have polymerized immediately. American Tobacco claim to have found acrolein and hence use charcoal in their filters for its removal.

Ashing of whole tar gave 2 ppm of Cr. and 20-30 ppm of As.

Fieser has found 2.5 - 5.0% of a sterol type compound in tobacco.

Most of Tennessee's work has been on the neutral tar fraction. They have also worked some on the Et Ac fraction but have found it pretty complicated. They have stopped using CCl₄. Their attention is now focussed on the benzene and hexane fractions.

Whole tar has been found to be far more active in animal studies than any of the individual fractions. If there is any action resulting from tars, it is likely to be from the whole tars rather than any one fraction. If benzpyrene is present it is there in very small amounts.

They are getting a small amount of fluorescence from tobacco itself when extracted with methanol and methylene chloride.

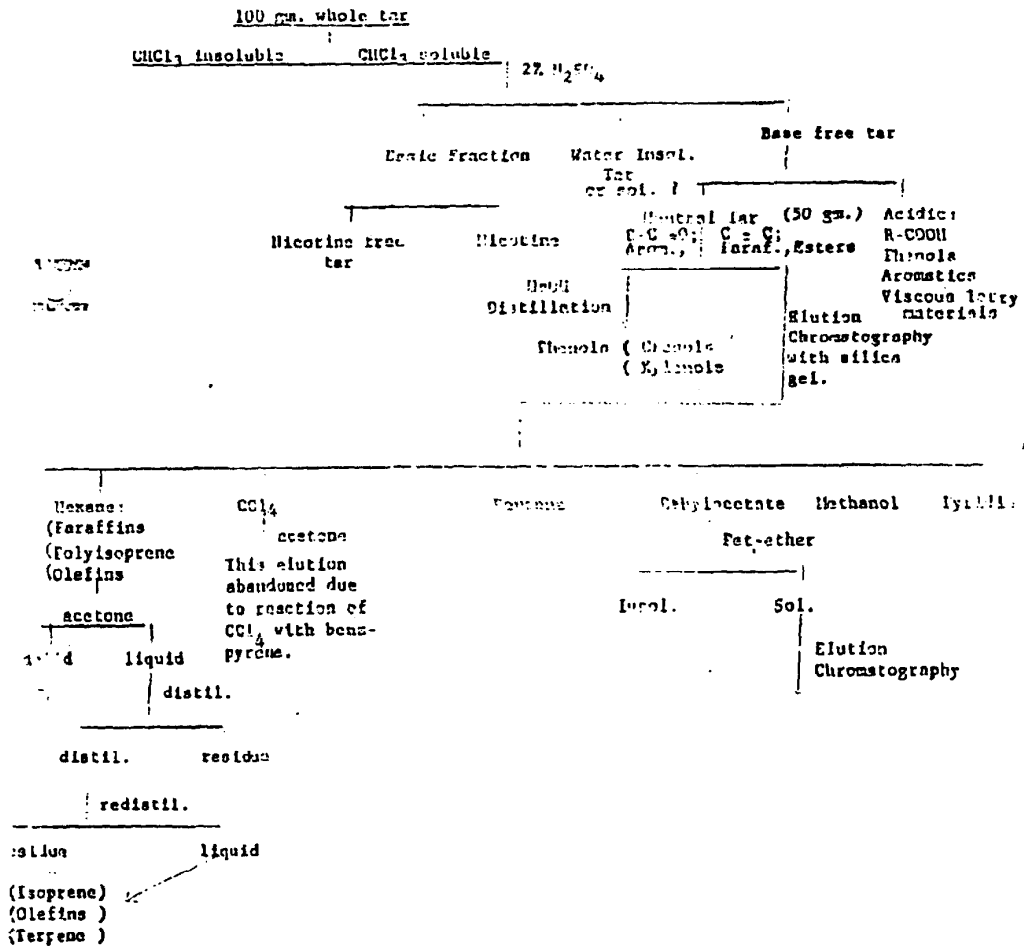
When tobacco was extracted to remove nicotine and this tobacco smoked after a few days standing nicotine was obtained in the smoke.

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Scheme for Separation of Tobacco Tars
Tennessee Eastman Company



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