

SOME PHYSICAL AND CHEMICAL PROPERTIES OF  
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The nature of this study and the plan of procedure is similar to that of Stout, Schutte and Fischer<sup>1</sup> in which the properties of rye oil as affected by the choice of menstruum is reported. The cocklebur is a plant common to most regions of South Dakota, and is found also widely scattered throughout most parts of the nation. Numerous varieties are reported in different localities. The burs selected for this study were gathered from the species known as *Xanthium commune* Britton. Interest in this problem was aroused by the prevalence of this plant over the state and the general knowledge that the burs contain a relatively high oil content. Eight representative solvents were used in these extractions because it was recognized that the kind and amount of oil and non-oil constituents removed would be somewhat dependent upon the solvent employed. The solvents used include three chlorinated hydrocarbons, one sulfur, and two oxygen containing compounds, a cyclic hydrocarbon, and petroleum ether. The information presented should be definitely regarded as merely a preliminary report, rather than a completed study. It is planned to continue this investigation, extending the type and number of physical and chemical properties studied, but limiting the solvent to those which have been found by experience to give the most satisfactory and consistent results.

## Experiments

Mature well-seasoned burs were ground and extracted with the eight solvents listed in Table I by use of the familiar Soxhlet extractor. All solvents employed were previously fractionated through an eight inch Widner column, retaining only those portions whose boiling points agreed with those generally accepted. The purification of the oil consisted of the filtration of the oil and solvent, followed by removal of the last traces of solvent by low-temperature distillation under re-

<sup>1</sup> Stout, Schutte, Fischer, J. Am. Chem. Soc., 56, 210 (1934).

duced pressure. The resulting oils varied noticeably in color and viscosity.

Table I

Solvent	B.P.	Pres. mm.	Oil Yield	d 25/4	Iodine No. (Hanus)	Index of Refr.	Color Yellow
Carbon tetrachloride -----	74.6 74.8	725.8	6.01	.9527	48.2	1.490	0.3
Chloroform -----	59.5 60.0	730.5	6.31	.9942	79.9	1.486	0.2
Ethylene dichloride -----	81.3 82.0	733.7	4.63	1.0088	81.4	1.4794	0.4
Carbon disulfide -----	45.0	730.5	7.14	.9655	103.0	1.4842	0.2
Acetone -----	54.7 55.0	722.4	5.86	.9095	44.6	1.499	0.3
Ethyl ether -----	34.0	732.0	4.99	.9186	89.4	1.4815	0.5
Benzene -----	78.0	720.7	7.45	.8222	83.9	1.484	0.0
Petroleum ether -----	29.0 40.00	738.0	5.47	.8938	87.9	1.4746	0.1

The iodine absorption number was determined by the Hanus method<sup>2</sup>. Acknowledgement is gratefully made to Homer W. Carhart of the University of South Dakota for determining the index of refraction. The index of refraction readings for the oil obtained by the acetone and carbontetrachloride extractions were of doubtful value. In the case of the acetone extract the readings varied from 1.478 to 1.499, and were indefinite, tending to increase upon standing until the higher value was reached. The same difficulty was encountered with carbontetrachloride, showing a range of 1.463 to 1.490. It will also be noted that the iodine number determined for these two oils from the same solvent was relatively low. As an explanation for this, three things may be possible. The resulting oil may have a large variety of products, or the last traces of solvent not have been removed, or a change may have occurred due to oxidation or polymerization. Acknowledge-

<sup>2</sup> Official and tentative methods of analysis of the Association of Official Agricultural Chemists, 319 (1930).

ment is also made to Emory D. Fisher of the University of Wisconsin for the determination of the degree of pigmentation of a ten per cent petroleum ether solution with a Hosenheim-Schuster colorimeter by means of which one arrives at the color in the Lovibond system.

### Results

On examination of the oil yield (Table I), it appears that benzene is the most efficient menstruum and ethylene dichloride the least efficient. Parallels cannot be drawn throughout the series as a whole, except perhaps in the case of a few pairs. For example, the density of oxygen containing solvents (ethyl ether and acetone) produced oils of nearly the same value and the three chlorinated hydrocarbons ranged in the heavier densities. Carbon disulfide gave the highest iodine number, one of the heavier densities, and was the second highest in oil yield.

Examination of the burs indicated an average moisture content of 8.80 percent and a total ash at 4.16 percent. A nitrogen content, based on the Kjeldahl determination, of 1.51 per cent which when converted to protein by the usual 6.25 factor was equal to 9.42 percent protein. A steam distillation proved to be of little value in extracting oil.

### Summary

As a result of this part of the investigation on the relation of eight different solvents to the properties of cocklebur oil, it is evident that from 4.63 to 7.45 percent oil can be obtained from ground cockleburs. With these various solvents there is a variation in pigmentation and also in the physical and chemical constants. It appears that the yields of oil that may be expected to assume the following in successively greater amounts: ethylene dichloride—ethyl ether—petroleum ether—acetone—carbon tetrachloride—chloroform—carbon disulfide—benzene. The following minima and maxima were noted:  $d_{25}^{25}$  .8938 and 1.0088;  $n_D^{20}$  1.4746 and 1.499; iodine number 44.6 and 103, color (yellow) 0.0 to 0.5.