acid, ketene dimer and traces of ketene. The statement regarding ketene dimer should be withdrawn for it has not been possible to obtain this substance from acetylphthalimide in subsequent experimentation. The volatile products found were acetic acid, acetic anhydride, acetone and a small quantity of ketene.

This aligns acetylphthalimide with propionyl-, *n*-butyryl- and caproylphthalimides (all $C_6H_4(CO)_2N—COCH_2R$), all of which give rise on pyrolysis to phthalimide, the acid RCH_2COOH or its anhydride, and the ketone $(RCH_2)_2CO$. In contrast, isobutyryl- and diphenylacetylphthalimides (both $C_6H_4(CO)_2N—COCHR_2$) pyrolyze satisfactorily to ketenes, $R_2C{=}C{=}O$.

For a time it was considered that the non-production of ketene dimer from acetylphthalimide (dried as before by the toluene distillation method) was due to moisture. The same results were produced, however, on acetylphthalimide which was dried in a desiccator over phosphorus pentoxide for ten months.

Ethyl acetate was found to be an excellent solvent from which to crystallize acetylphthalimide. After such a purification it was dried thoroughly as mentioned above and 50 g. of it subjected to pyrolysis. In the 6 g. of distillate which was collected during two hours there was found 1.0 g. of acetone (b. p. 56–60°, soluble in water and identified by converting to dibenzylideneacetone of m. p. 111–112°), 1.1 g. of acetic anhydride (analyzed by the method of Whitford[2] with oxalic acid and pyridine), and 3.4 g. of acetic acid. The total acidity (acetic acid + anhydride) was 4.5 g. The distillate reacted with aniline to produce acetanilide, m. p. 112–113°. If acetoacetanilide was present also its quantity was too small for identification. Evidence for ketene was found by condensing the liquid products at 0° and passing the vapors into aniline. The yield of acetanilide was 0.4–0.5 g.

The above experiment was representative of several which were performed. Variations included the manner of drying, the size of the run (up to 108 g.) and the manner of heating (free flame or salt-bath so that the temperature within the flask ascended gradually from 220 to 325°). The results were essentially the same in all.

Summary.—The volatile products from the pyrolysis of acetylphthalimide are acetic acid, acetic anhydride, acetone and a small quantity of ketene. No significant amount of ketene dimer was produced.

---

[2] Whitford, THIS JOURNAL, **47**, 2939 (1925).

CHEMICAL LABORATORY
NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS

RECEIVED FEBRUARY 28, 1935

COMMUNICATIONS TO THE EDITOR

COPPER SULFIDE-WATER CONTACT ANGLES

Sir:

Evidence has been obtained in this Laboratory which indicates that the presence of an adsorbed air film is the most probable cause of the contact angle of water with copper sulfide. Wark [Wark and Cox, *Am. Inst. Min. Met. Eng.*, Tech. Pub., No. 461 (1932)] has shown that a copper sulfide surface that has been ground under water exhibits no contact angle with water. If such a polished surface is exposed to air the contact angle with water assumes a definite value.

If a copper sulfide, covellite, surface is ground under water, such a surface has a zero contact angle with water; exposed to air this surface gives a definite contact angle with water. However, when such a specimen is put into a vessel in contact with water and the water is completely removed by evaporation at reduced pressure, the specimen being finally subjected to gentle warming while the container is evacuated, it is found that the surface again exhibits a zero contact angle with water. Further, when the water is again removed from the copper sulfide and air let into the vessel, the contact angle with water is again developed. Preliminary experiments with copper xanthate indicate a similar characteristic behavior.

Further work along these lines with liquids known to give contact angles with certain similar solids is planned.

DEPARTMENT OF CHEMISTRY
MICHIGAN COLLEGE OF
MINING AND TECHNOLOGY
HOUGHTON, MICHIGAN
C. C. DE WITT

RECEIVED APRIL 11, 1933

SURFACE PRESSURES AND POTENTIALS OF MONOMOLECULAR FILMS OF LONG MOLECULES: POLYMERS OF ω-HYDROXYDECANOIC ACID

Sir:

Remarkable relations are exhibited by films of linear polymers of the formula $HO[(CH_2)_9COO]_xH$ with molecular weights as high as 25,200 and values of *x* to 148 as obtained from Drs. W. H. Carothers, E. O. Kraemer and F. J. Van Natta [Kraemer and Van Natta, *J. Phys. Chem.*, **36**, 3175 (1932); Carothers and Van Natta, THIS JOURNAL, **55**, 4714 (1933)].

The most prominent characteristics are listed with specific values which refer to condensed films under a pressure of 3 dynes per cm. unless otherwise stated.

1. Thickness of films from 4.0 to 5.0 Å.
2. Distance between centers of adjacent chains 4.0 to 4.8 Å.
3. Apparent vertical cross section of molecule 19.4 to 19.9 sq. Å., or nearly the same as the horizontal cross section (20.5) of molecules, such as stearic acid, oriented vertically.
4. The area (*a*) per molecule in condensed films is nearly proportional to the molecular weight at any given film pressure (*f*).

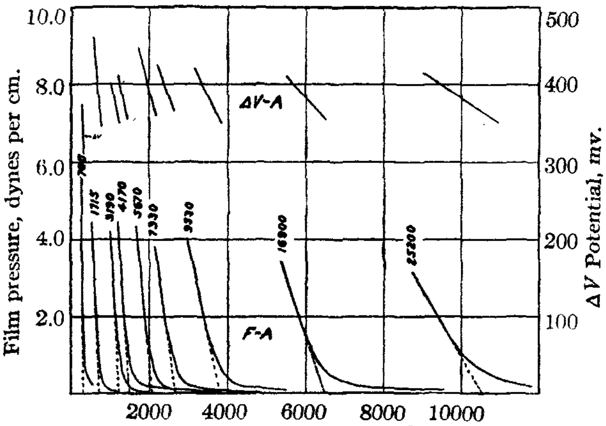

Fig. 1.—Film pressures and surface potentials for linear polymers of ω-hydroxydecanoic acid.

5. The compressibility is very large, since the value of $[(a_0 - a_1)/a_0]/(f_1 - f_0)$ lies between 0.040 and 0.053, and is almost independent of molecular size. Similar values for vertically oriented molecules are in general only one-tenth to one-sixtieth as large.

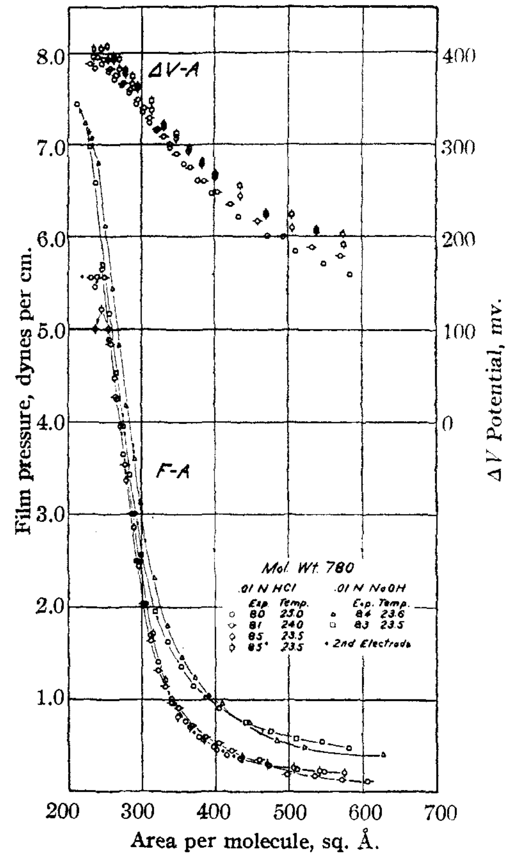

Fig. 2.—Values for polymer of molecular weight 780.

6. The pressure of collapse on 0.01 *M* hydrochloric acid increases from 3.2 to 5.7 dynes as the molecule becomes shorter, and after collapse the pressure decreases rapidly for a time. On 0.01 *M* sodium hydroxide the pressure of collapse is higher.
7. At pressures below 1.3 to 2 dynes the films are "expanded," with considerably higher molecular areas if spread on a base than if on an acid aqueous solution.
8. The surface potential (ΔV) of any one of the condensed films increases about 13% less rapidly than the surface density of the film. Thus the surface potential seems to increase with and nearly as the number of polar groups per unit area. However, the mutual effect of the dipoles is such as to decrease the potential.
9. At low pressures the expanded films become nonhomogeneous, since the surface potential varies with the location on the surface. This island effect is much more prominent for polymers of high than for those of low molecular weight.
10. The molecular area of the compound of molecular weight 25,200 is 10,400 sq. Å. for zero pressure, or 8800 sq. Å. at 3 dynes per cm. pressure. This is the largest area thus far found for any molecule.