

hour afterward. The amide was recrystallized from benzene-petroleum ether; m. p. 106-107°; yield 75%.

Anal. Calcd. for $C_{11}H_{15}ON$: C, 74.54; H, 8.53. Found: C, 74.72; H, 8.37.

2,2',4,4',6,6'-Hexaethylhydrobenzoin.—A 2-g. sample of 2,2',4,4',6,6'-hexaethylbenzil² was dissolved in 19 ml. of ethanol and hydrogenated at 135° and 2300 pounds pressure in the presence of 2 g. of copper chromite catalyst. The reduction was complete in five and one-half hours. After removal of the catalyst and solvent the hydrobenzoin was recrystallized from aqueous acetone. It separated in colorless crystals; m. p. 225-226°.

Anal. Calcd. for $C_{26}H_{38}O_2$: C, 81.62; H, 10.01. Found: C, 81.55; H, 10.02.

2,2',6,6'-Tetramethylhydroanisoin.—One gram of 2,2',6,6'-tetramethylanisil³ in 19 ml. of ethanol was hydrogenated at 130° and a pressure of 1800 pounds per sq. in. in the presence of 2 g. of copper chromite catalyst. The reduction was complete in five hours. After removal of the catalyst by filtration the filtrate was concentrated to 5 ml. The addition of 0.5 ml. of water caused the hydroanisoin to precipitate. It was recrystallized from a mixture of benzene and petroleum ether; m. p. 199-200°; yield 0.65 g.

Anal. Calcd. for $C_{20}H_{26}O_4$: C, 72.70; H, 7.93. Found: C, 72.84; H, 8.03.

(2) Fuson and Corse, *THIS JOURNAL*, **60**, 2063 (1938).

(3) Fuson, Corse and Welldon, *ibid.*, **63**, 2645 (1941).

DEPARTMENT OF CHEMISTRY
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REYNOLD C. FUSON
JOSEPH CORSE

RECEIVED AUGUST 13, 1945

1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol

Condensation of 2',4',6'-triisopropylchalcone with phenylmagnesium bromide was found to yield 1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propanone, the enol form of which—1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol—was stable in solution.

2',4',6'-Triisopropylchalcone.—Fifty grams of benzaldehyde was added, with stirring, to an ice cold mixture of 100 g. of 2,4,6-triisopropylacetophenone, 21 g. of sodium hydroxide and 500 ml. of 95% ethanol. The reaction mixture was stirred during the period of addition and for an additional twenty-seven hours. During the first three

hours of this time the temperature of the mixture was kept at 0° and afterward at room temperature. The chalcone crystallized from alcohol in pale yellow needles; m. p. 100-101°; yield 91%.

Anal. Calcd. for $C_{24}H_{30}O$: C, 86.17; H, 9.04. Found: C, 86.69; H, 9.32.

1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propanone.—To a solution of phenylmagnesium bromide made from 7.3 g. of magnesium, 47 g. of bromobenzene and 75 ml. of ether was added a solution of 33.4 g. of 2',4',6'-triisopropylchalcone in 150 ml. of ether. The mixture was heated for one hour under reflux and decomposed with cold ammonium chloride solution. The ether solution was divided into two equal parts. Evaporation of the solvent from one of them left the 1-(2,4,6-triisopropylphenyl)-3,3-diphenyl-1-propanone as a residue. It crystallized from ethanol in white needles; m. p. 117-118°; the yield was practically quantitative.

Anal. Calcd. for $C_{30}H_{38}O$: C, 87.33; H, 8.80. Found: C, 87.77; H, 8.68.

The ketone gave negative tests with ferric chloride solution, bromine in carbon tetrachloride and potassium permanganate in acetone.

1-(2,4,6-Triisopropylphenyl)-3,3-diphenyl-1-propen-1-ol Peroxide.—Half of the ether solution from the foregoing experiment was diluted with about five times its volume of low-boiling petroleum ether and washed twice with ice water. A rapid stream of oxygen was passed through the moist solution. The peroxide separated in white needles which were recrystallized from a mixture of ether and low-boiling petroleum ether. The yield was 11 g. The compound melted, with decomposition, at 156°. In contact with a flame it decomposed with a flash and then burned quietly.

Anal. Calcd. for $C_{30}H_{38}O_3$: C, 81.04; H, 8.17; Found: C, 80.73; H, 8.12.

A steady stream of oxygen was bubbled for twelve hours through a solution of 6 g. of the enol peroxide in 200 ml. of acetone. One gram of an acidic product was isolated and purified by sublimation; m. p. 144°. A mixed melting point determination showed the compound to be diphenylacetic acid.¹

(1) Dunlap, *THIS JOURNAL*, **19**, 645 (1897).

THE NOYES CHEMICAL LABORATORY
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REYNOLD C. FUSON
A. I. RACHLIN

RECEIVED SEPTEMBER 20, 1945

COMMUNICATIONS TO THE EDITOR

OBSERVATIONS ON THE ANGULAR SCATTERING OF LIGHT BY SULFUR SOLS

Sir:

Bidhubhusan Ray¹ observed that the light scattered by a sulfur sol produced by mixing dilute solutions of sodium thiosulfate and sulfuric acid exhibited different colors depending on the angle which the scattered light ray made with the incident beam. Ray correctly explained these "axial colors" as due to the asymmetrical distribution of the light scattered by the sulfur particles and to the strong dependence of the shape of the scattering patterns on the wave length of the incident light.

(1) B. Ray, *Indian Assoc. Cultiv. Sci.*, **7**, 1 (1921).

LaMer and Barnes in some unpublished investigations, have shown that purer, more sharply defined colors and hence more uniform particle sizes can be obtained if the reacting solutions are very dilute (about 0.001 *M*). The growth of the liquid sulfur droplets is stopped by the addition of iodine and the sol stabilized.

We have found that although many bands of colors are exhibited, these sols are characterized experimentally most easily by the bands of reddish hue. The angular position and number of these red bands called "orders," are related theoretically to the size of the sulfur particles.

The position of the orders exhibited by such a sol at various times, *i. e.*, stages of growth, are

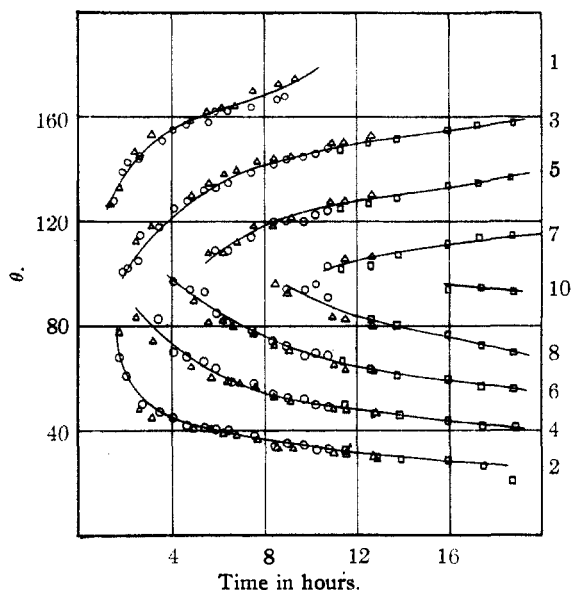


Fig. 1.—Position of orders in a growing sulfur sol as a function of time: O, run 5; Δ , run 7; \square , run 11.

shown in Fig. 1. In this figure, θ is the angle made with the negative direction of incident light propagation by the ray scattered to the observer. The curves are numbered according to the direction in which the orders move as the droplets grow. The θ values for odd numbered curves increase with time while the θ values decrease with time for even numbered curves. The orders corresponding to even-numbered curves are polarized in a horizontal direction (to the plane determined by the ray to the observer and the incident light ray), while for the odd-numbered orders the polarization is in a vertical direction.

These data were obtained at 25° . Exploratory runs at 40° indicate an approximate doubling of the rate of obtaining a given size for a 15° rise in temperature. A study is being made of the rate of growth of sulfur particles as a function of temperature and concentration.

These data can be correlated approximately at this time with unpublished data of Barnes and LaMer on the transmission of light by similar monodispersed sols. They show that after five hours the droplets have grown to 0.4μ radius and after nineteen hours to 0.6μ . Microscopical examination of a five-hour sol prepared similarly showed uniform particles of about 0.4μ .

A more accurate correlation between particle size and the position of orders is being investigated.

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RECEIVED OCTOBER 13, 1945

THE STRUCTURE OF CUBANITE, CuFe_2S_4 , AND THE COORDINATION OF FERROMAGNETIC IRON

Sir:

The structure of cubanite is of particular interest since this mineral is ferromagnetic. The solution of its structure has shown that the coordination of the iron atoms is unique.

Cubanite is orthorhombic, space group¹ $Pcmm$. The cell has the following dimensions, $a = 6.45$, $b = 11.095$, $c = 6.221 \text{ k}\text{\AA}$. This cell contains 4 CuFe_2S_4 . The structure has been uniquely determined with the aid of intensity data derived from Weissenberg photographs made with molybdenum radiation. The copper atoms and one-third of the sulfur atoms each occupy the equipoint $4c$ (reflection planes), while the iron atoms and the remaining sulfur atoms are in the general position, $8d$. The structure is defined by the following approximately determined parameters (origin at inversion center)

	(u)	(v)	(w)
	x	y	z
Cu	$7/12$	$(1/4)$	$1/8$
S_1	$11/12$	$(1/4)$	$1/4$
Fe	$1/12$	$1/12$	$1/8$
S_2	$5/12$	$1/12$	$1/4$

The metals are each surrounded by four sulfur atoms in almost undistorted tetrahedral coordination. The sulfur atoms are also surrounded by four metals in tetrahedral coordination. The environment of the S_1 's is an almost undistorted tetrahedron of two copper atoms and two iron atoms while the environment of the S_2 's is a distorted tetrahedron of three iron atoms and one copper atom. The structure may be regarded as made up of vertical slices of the wurtzite arrangement (containing and surrounding the symmetry plane of cubanite), joined to identical but inverted slices by means of the sharing of one of the edges of each iron coordination tetrahedron.

The structure is unusual in that one edge of the tetrahedral coordination of each iron atom is shared with that of another iron atom. As a consequence of this, the iron atoms are brought together in pairs across the shared edges. This strongly suggests that the iron atoms, in addition to being bonded to four sulfur atoms, are bonded to one another in pairs. This unusual bond arrangement is doubtless the cause of the ferromagnetism of the crystal.

A detailed account of the structure determination, together with refined parameters, will appear elsewhere.

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RECEIVED SEPTEMBER 20, 1945

(1) M. J. Buerger, *Amer. Mineral.*, **22**, 1117-1120 (1937).