

Two greases were prepared as described above: one containing 20% lithium stearate, the other 20% commercial aluminum di-stearate, in a number 50 S.A.E. mineral oil. A lump of the lithium base grease retained its shape after two hours in an oven at 175°, while a similar sample of the aluminum base grease melted to a viscous liquid in less than twenty minutes. A small volume vacuum system containing a 14/35 ground glass joint lubricated with these greases and held thermostatically at a temperature of 170° showed a pressure increase of non-condensable gases from 10^{-6} mm. to 10^{-2} mm. in eighteen hours. Some oil distilled from the grease during the test, but, after cooling, the joint was easily taken apart. Although the aluminum base grease melts at a much lower temperature than that made with the lithium soap, the viscosity of the melted material is sufficient to give a good seal and it can also be used as a high temperature lubricant. It is noteworthy that the consistency of these greases does not increase greatly until cooled below the pour point of the oil used in their preparation.

These greases have been found to be very resistant to halogenation and have been used with success for about a year.

RECEIVED FEBRUARY 4, 1943

NEW COMPOUNDS

Xanthone 2,7-Dinitrile

This compound was required for investigation of its value as an insecticide.

Ten grams of 2,7-diaminoxanthone, prepared from xanthone by the method of Baeyer,¹ was dissolved in 25 g. of concentrated hydrochloric acid and 75 cc. of water, cooled in ice, a cold solution of 15 g. of sodium nitrite in 60 cc. of water slowly added, the reaction product diluted with 250 cc. of ice water and filtered. The filtrate was added gradually to a potassium cuprous cyanide solution prepared by mixing a solution of 25 g. of cupric sulfate in 100 cc. of water with a solution of 27.5 g. of potassium cyanide in 50 cc. of water and cooling in ice. The reaction product was allowed to stand overnight at room temperature, after which the precipitate was filtered off, washed thoroughly with water and dried at 100°. Sublimation *in vacuo* yielded 5.3 g., or 48%, of bright yellow crystals. The nitrile dissolved in concentrated sulfuric acid to give a colorless solution with a bright blue fluorescence. It was almost completely insoluble in all the common solvents. For analysis a portion was twice recrystallized from ben-

(1) Baeyer, *Ann.*, **372**, 138 (1910).

zonitrile, washed thoroughly with alcohol and dried at 100°. The recrystallized product was a cream-colored microcrystalline powder.

Anal. Calcd. for $C_{16}H_8O_2N_2$: N, 11.38. Found: N, 11.34, 11.27.

CONNECTICUT AGRICULTURAL EXPERIMENT STATION
NEW HAVEN, CONN. HARRY J. FISHER

RECEIVED MARCH 16, 1943

t-Butyl Benzoyldimethylacetate

This compound was prepared by a modification of the procedure for the synthesis of ethyl benzoyldimethylacetate.¹ *t*-Butyl isobutyrate² (0.158 mole) was added to an ether solution of sodium triphenylmethyl (0.158 mole), the mixture was shaken and allowed to stand at room temperature for forty minutes when the color had become light orange. Benzoyl chloride (0.158 mole) was then added with shaking. After standing for several hours, 10 cc. of acetic acid was added and the mixture was extracted with water, followed by sodium carbonate solution. After removing the solvent from the dried ether solution, the material boiling up to 160° at 15 mm. was collected. Refractionation of the material yielded *t*-butyl benzoyldimethylacetate boiling at 146–148° at 15 mm., and melting at 64–65° after recrystallization from methanol; yield, 50%.

Anal. Calcd. for $C_{18}H_{20}O_3$: C, 72.55; H, 8.12. Found: C, 72.89; H, 8.32.

(1) Hauser and Renfrow, "Organic Syntheses," **19**, 43 (1939).

(2) Abramovitch, Shivers, Hudson and Hauser, *THIS JOURNAL*, **65**, 986 (1943).

DEPARTMENT OF CHEMISTRY
DUKE UNIVERSITY
DURHAM, NORTH CAROLINA

J. C. SHIVERS
CHARLES R. HAUSER

RECEIVED MARCH 8, 1943

1-Phenylethylidenemalonitrile

This compound was prepared by a method analogous to that used by Cope¹ for ethyl 1-phenylethylidenecyanoacetate: 33 g. (0.5 mole) of malononitrile, 60 g. (0.5 mole) of acetophenone, 4 g. of ammonium acetate and 12 g. of acetic acid were refluxed with 100 cc. of benzene under a condenser equipped with a device for constant removal of water. After twenty hours the solution was cooled, washed with water and dried over anhydrous sodium sulfate. The benzene solution was warmed and diluted with technical hexane just short of turbidity. Upon cooling with ice water a large crop of orange crystals separated. Four recrystallizations from dilute ethanol gave 60 g. (56%) of colorless prisms, m. p. 94° (uncor.).

Anal. Calcd. for $C_{11}H_8N_2$: C, 78.5; H, 4.79; N, 16.65. Found: C, 78.27, 78.71; H, 5.03, 4.97; N, 16.40, 16.28.

(1) Cope, Hofmann, Wyckoff and Hardenbergh, *THIS JOURNAL*, **63**, 3452 (1941).

CENTRAL RESEARCH LABORATORIES
MONSANTO CHEMICAL COMPANY
DAYTON, OHIO

DAVID T. MOWRY

RECEIVED NOVEMBER 30, 1942