366. *Beryllium Benzoylacetonate.*

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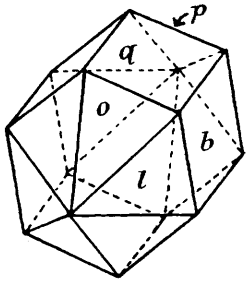
Although beryllium acetylacetonate has long been known, the *benzoylacetonate* has not yet been described. Mills and Gotts (J., 1926, 3121) have demonstrated the 4-covalency of beryllium in compounds of this type by the resolution of the benzoylpyruvic acid derivative. It was not possible to demonstrate optical activity in the benzoylacetonate, but it possesses other optical properties, notably a high dispersion and very strong double refraction, which are noteworthy.

Preparation.—Beryllium hydroxide was converted into the more reactive basic carbonate (compare Parsons and Robinson, *J. Amer. Chem. Soc.*, 1906, **28**, 557) as follows: A litre of water was saturated with solid ammonium carbonate at 25°. The temperature was then raised to 35°, and the solution shaken with an excess of beryllium hydroxide. After filtration, steam was passed through the solution for 2 hours and the precipitated basic carbonate filtered off, washed with hot water, and dried at 100°. 20 G. of benzoylacetone were dissolved in 200 c.c. of 98% alcohol, a suspension of 5 g. of the basic carbonate in 200 c.c. of alcohol added, and the mixture refluxed on a water-bath until carbon dioxide was no longer evolved (about 3 hours). When the clear solution so obtained was cooled, a white precipitate of *beryllium benzoylacetonate* suddenly appeared which would not redissolve on warming; it was recrystallised from toluene, a steam-jacketed filter being used [Found: Be, 2·82; C, 71·6; H, 5·5; *M*, cryoscopic in benzene (standardised against naphthalene), 328. $Be(C_{10}H_9O_2)_2$ requires Be, 2·73; C, 72·5; H, 5·4%; *M*, 331]. The beryllium content was determined by warming with concentrated sulphuric acid and subsequent ignition to oxide. The low carbon content may be due to carbon left in the combustion residue.



Properties.—Beryllium benzoylacetonate melts at 210°, is insoluble in water, dilute acids, and alkali, and is not appreciably attacked by these solvents even on boiling. It is almost insoluble in alcohol, but moderately soluble in benzene (2% at 15°) and more soluble in toluene.

Crystallographic Description (by H. M. Powell).—Colourless crystals (see fig.) were obtained from toluene solution by cooling. System: monoclinic; $a : b : c = 0·6236 : 1 : 0·5953$; $\beta = 95° 50'$. They were very soft and showed no distinct cleavage. Vicinal faces frequently developed on *l*-, *o*-, and *p*. Examination of a large crop revealed no sign of enantiomorphous forms. Etch pits produced by toluene do not indicate absence of a symmetry plane and no pyroelectric effect is observable by the liquid-air method. The crystals are presumed to be those of a racemic compound. In the following table are recorded the mean observed angles for three crystals measured on a two-circle goniometer. The angles in parentheses are to be added to give the calculated values.

	b (010).	*l* (120).	*q* (011).	*o* (111).	*p* ($\bar{1}11$).
ϕ	0° 0′	39° 16′ *	10° 0′ (−8′)	61° 3′*	304° 14′ (10′)
ρ	90 0	90 0	31 0 (−11)	50 31 *	46 7 (0)

The setting is in accordance with the Barker rules. Classification angles (calculated) are *cr* 40° 53′, *ra* 43° 17′, *am* 31° 27′, *bq* 59° 42′.

Optics. The double refraction is very strong, and the high dispersion gives the crystals the fiery appearance of diamond. The refractive index β which is for vibrations parallel to the *B* axis was determined by use of the natural prisms. Obs. β = 1·711 (Li), 1·732 (Na), 1·762 (Tl). The highest and lowest refractive indices observed for any vibrations were 1·795 (Na) and 1·633 (Na), but these are not necessarily γ and α. The lowest value observable by immersion methods was 1·633 (Na). In thin fragments one axis of a negative biaxial interference figure with highly coloured brushes was observed.

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[*Received, October 24th*, 1933.]