Phase identification was done principally by means of X-ray patterns taken on a Norelco recording spectrometer using CuKα radiation (λ = 1.537 kX.). The following data of Schulze[1] were used as a standard:

d/n	Rel. int.	Indices
3.63	130	(101)
2.25	90	(112)
1.86	48	(211)
1.45	52	(114) or (213)

The quench results are most easily summarized in the form of tables. Table I gives the heat treatments and phase analyses of quenched samples of boron phosphate, while Table II gives the X-ray patterns of these same samples.

TABLE I

RESULTS OF QUENCH DATA ON BPO_4

Heat treatment Temp., °C.	Time, hr.	Phases present
Recd. from Victor	...	BPO_4
1005	7.5	BPO_4
1250	1	PBO_4
1355	1	BPO_4
1403	1	BPO_4
1452	5 min.	Sample partly vaporized, remainder BPO_4
1462	1	Sample completely vaporized

TABLE II

X-RAY DATA ON BPO_4

BPO_4 (as recd.)		BPO_4 (1005°, 1250°, 1355°, 1403°)		BPO_4 (1452°)	
d/n	Rel. int.	d/n	Rel. int.	d/n	Rel. int.
3.63	1.00	3.61	1.00	3.61	1.00
3.08	0.04	3.05	0.19	..	..
2.87	.03	2.88	.08	..	..
2.24	.32	2.25	.54	2.24	0.42
1.86	.08	1.86	.36	1.85	.18
1.81	.03	1.81	.25	1.81	.12
1.46	.06	1.45	.45	1.45	.18
1.31	.02	1.31	.20	..	..
1.19	.02	1.21	.08	..	..

From the quench data obtained, it is likely that BPO_4 vaporizes as such and does not decompose into the constituent oxides, B_2O_3 and P_2O_5, although there is no direct proof that this is the case. Phosphorus pentoxide has a very high vapor pressure at the temperatures used in the experiment,[3] and while anhydrous B_2O_3 does not vaporize as freely as P_2O_5 at these temperatures, in the presence of water vapor the hydrated forms vaporize readily.[4]

Using microscopic and X-ray methods on the partly vaporized sample quenched from 1452°, no free P_2O_5, B_2O_3 or H_3BO_3 was detected, indicating that the compound probably does not decompose into the component oxides. Further work would be necessary to conclusively prove this point.

Thermal expansion data were obtained on four-inch samples of sintered boron phosphate using a fused silica type dilatometer with dial gage reading to 0.0001″. The compound was sintered at 1130° for 1.5 hr. and at 1260° for 1 hr., and gave practically identical reversible thermal expansion curves after each heat treatment. The calculated coefficient of expansion in the range 25–1000° was 90×10^{-7} cm./cm./°C.

Conclusions.—Boron phosphate begins to vaporize in the neighborhood of 1450°, and will disappear completely at 1462° if held for one hour at this temperature. The coefficient of thermal expansion of BPO_4 sintered at 1260° is 90×10^{-7} cm./cm./°C. in the range 25–1000°.

Acknowledgment.—The authors are grateful to Mr. Howard Adler, Chief Chemist of the Victor Chemical Works, for supplying data on the compound BPO_4 and for the sample of BPO_4 used in the experimental work.

DIVISION OF CERAMICS
PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNA.

RECEIVED MAY 15, 1950

---

(3) W. L. Hill, G. T. Faust and S. B. Hendricks, THIS JOURNAL, **65**, 794 (1943).

(4) F. C. Kracek, G. W. Morey and H. E. Merwin, *Am. J. Sci.*, **35A**, 143 (1938).

Chemical Effects of the Cu^{63} (γ,n) Cu^{62} Reaction with Copper Salicylaldehyde-*o*-phenylenediimine

BY O. G. HOLMES AND K. J. MCCALLUM

Duffield and Calvin[1] have shown that when copper salicylaldehyde-*o*-phenylenediimine is bombarded with thermal neutrons, the Szilard–Chalmers effect permits a concentration of Cu^{64} with an inorganic carrier. In their work, the chelate complex was bombarded both in the solid state and in pyridine solution, and the resulting distributions of Cu^{64} between the complex and inorganic carrier were reported.

We have done similar experiments with the Cu^{63} (γ,n)Cu^{62} reaction on the same complex. The material was irradiated with γ-rays of 18 ± 0.5 Mev. peak energy in the betatron[2] at the University of Saskatchewan. The maximum recoil energy imparted to the Cu^{62} nucleus is approximately 0.1 Mev., since a threshold γ-ray energy of 11 Mev. has been reported for this reaction.[3]

Irradiation of the chelate complex was carried out with the material in the solid form and also in solution in pyridine. Carrier copper was separated from the complex,[1] precipitated as CuS, and the activities of the CuS precipitate and the residual complex were determined using a Geiger counter and scale-of-64.

The percentage of the total activity remaining with the chelate complex, or the retention, as found from duplicate experiments, is reported in Table I for irradiations under different conditions. The values for the retentions are corrected for self-absorption[4,5] and for decay of the 10.5-

---

(1) R. B. Duffield and M. Calvin, THIS JOURNAL, **68**, 1129 (1946).

(2) We wish to thank Professor R. N. H. Haslam for arranging the irradiations.

(3) G. C. Baldwin and H. W. Koch, *Phys. Rev.*, **63**, 59 (1943).

(4) C. V. Strain, *ibid.*, **54**, 1021 (1938).

(5) W. Libby, *Ind. Eng. Chem.*, *Anal. Ed.*, **19**, 3 (1947).

TABLE I

Expt.	Material irradiated	Method of sepn.	Retention from gamma irradiation, %	Retention from neutron bombardment,[1] %
1	1 g. solid complex	Complex washed with H_2O; 10 mg. copper as $Cu(Ac)_2 \cdot H_2O$ added to filtrate; pptd. as CuS	95 ± 1	97
2	1 g. solid complex	Complex dissolved in 50 ml. pyridine; soln. made 0.0032 M in $Cu(Ac)_2 \cdot 1Py$; complex pptd. by 10-fold addn. of 15% HAc; carrier pptd. as CuS	45 ± 2	48
3	50 ml. pyridine soln. 0.053 M in complex	Soln. made 0.0032 M in $Cu(Ac)_2 \cdot 1Py$ then as in expt. 2	18 ± 2	18[a]
4	50 ml. pyridine soln. 0.052 M in complex and 0.0032 M in $Cu(Ac)_2 \cdot 1Py$	As in expt. 3 but no more carrier added	15 ± 2	
5	50 ml. pyridine soln. 0.053 M in complex and 0.027 M in $Cu(Ac)_2 \cdot 1Py$	As in expt. 4	9 ± 2	3[b]

[a] Bombarded solution was 0.0152 M in complex. [b] Bombarded solution was 0.0152 M in complex and 0.0152 M in $Cu(Ac)_2 \cdot 1Py$.

minute half-life Cu^{62}. The uncertainties reported include an estimate of the uncertainty in the self-absorption correction, as well as the statistical counting error.

For comparison, the retentions resulting from neutron bombardment of this complex were calculated from the activities in the organic and carrier fractions as reported by Duffield and Calvin,[1] and are included in the table. Presumably the activities reported by them have not been corrected for self-absorption. It should be pointed out that, in order to obtain sufficient activity by irradiation in the betatron beam, it was necessary, in the work reported here, to use solutions of the complex approximately three and one-half times as concentrated as those used by Duffield and Calvin.

For those experiments in which the complex was irradiated in the solid state, there appears to be little difference between the retentions resulting from the (γ,n) and the (n,γ) reactions on Cu^{63}, although the recoil energies imparted to the resulting radioactive copper nuclei differ by several orders of magnitude.

Comparisons of the retentions resulting from nuclear reactions in the solutions are not so direct, since the concentrations of the chelate complex are not the same for the γ-ray and neutron activation experiments. It was found that the bombardment with nuetrons of a 0.015 molar solution of the complex in pyridine gives the same retention as does the irradiation with γ-rays of a 0.53 molar solution.

If carrier copper in the form of copper acetate is present during the irradiation with γ-rays, the retention is dependent upon the concentration of carrier copper. For solutions 0.053 molar in the chelate complex, retentions of 18, 15 and 9% were observed for solutions which were also 0, 0.0032 and 0.027 molar, respectively, in copper acetate. Due to the limited solubility of copper acetate in pyridine, it was not possible to prepare solutions in which the molarities of the chelate complex and carrier copper acetate were both equal to 0.053, but it appears probable from these results that in such a solution the retention would be less than 9%. In the neutron bombardment experiments, Duffield and Calvin's results indicate a retention of about 3% in a solution where the molarities of chelate complex and copper acetate were both equal to about 0.015.

We wish to thank the National Research Council of Canada for the award of a Bursary to one of us (O. G. H.) and for generous financial assistance.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF SASKETCHEWAN
SASKATOON, SASK., CANADA

RECEIVED MAY 22, 1950

Composition of W-6 Raney Nickel Catalyst[1]

BY V. N. IPATIEFF AND HERMAN PINES

In performing hydrogenation experiments, it was found necessary to make a precise determination of the composition of W-6 Raney nickel. The Raney nickel, which was prepared by the method of Adkins and Billica[2] was found to contain about 77% nickel, 21% alumina, 1.4% metallic aluminum and the remainder sodium aluminate. This is in contradiction with the data obtained by Adkins and Billica who reported the catalyst to be composed of about 11% of aluminum, the remainder being nickel.

The presence of alumina in W-6 Raney nickel can be easily confirmed by the following simple experiment: a small amount of the catalyst placed in a 9% aqueous solution of nitric acid all dissolves except for a white precipitate, which consists of powdered alumina. This alumina is not the oxide of any aluminum present originally in the catalyst and oxidized by the nitric acid, as may be seen from the fact that neither the aluminum–nickel alloy nor an aluminum powder will give any aluminum oxide precipitate on reacting with dilute nitric acid.

(1) This work was made possible through the financial assistance of the Universal Oil Products Company. The authors wish to thank the Analytical Laboratory of the Universal Oil Products Company for the analysis of the catalyst.

(2) H. Adkins and H. R. Billica, THIS JOURNAL, **70**, 695 (1948).