Superior province into eastern North and South Dakota and Minnesota, by use of both outcrop and subsurface samples (*1*; *8–10*). Rocks of this same age have been found in the Wind River, Bighorn, Beartooth (*11, 12*), Casper (*13*), and Little Belt mountains (*14*) of western Wyoming and Montana; these have been interpreted as either a detached segment of the Superior province or a separate continental nucleus (*15*).

Much of the Precambrian basement lying between these two areas is not exposed and, until the present, only ages of 1.7 billion years or younger have been reported from the Black Hills (*2, 3*) and from drill core samples in eastern Montana and western North Dakota (*8, 10*). These data are compatible with the interpretation that subsequent geologic events obliterated evidence for Superior rocks in this region, if indeed they ever existed. The discovery of an ancient gneiss in the Black Hills, midway between the two areas of known Superior rocks, makes this interpretation obsolete. It also allows the prediction that additional discoveries of rocks of this age will be made by analyzing drill cores from basement rocks at other localities between the Bighorn Mountains and eastern South Dakota.

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Persistence of DDT in Soils of Heavily Sprayed Forest Stands

Abstract. *Soils from DDT-sprayed forest stands in New Brunswick and Maine contained DDT residues. The residues increased in successive samplings between 1958 and 1961, although no new spray was applied. The increase suggests that DDT residues may persist for several years in tree canopies but are ultimately carried into the soil.*

Persistence is an important characteristic contributing to the usefulness of DDT as an insecticide. Yet this very characteristic contributes significantly to the deleterious side effects of DDT applications, including the potential contamination of man's food chain, and raises important questions concerning the movement and storage of insecticide residues in nature. Such questions are especially pertinent in areas where DDT has been applied from airplanes during several years and residues may have accumulated. One of the most extensive aerial spraying operations ever undertaken has been directed toward the control of the spruce budworm (*Choristoneura fumiferana* Clem) in Maine and eastern Canada during the past decade (*1*). The study reported in this paper was an effort to determine the extent to which DDT residues have persisted in soils of forest stands heavily sprayed as a part of this program.

A total of four stands were sampled: three in northcentral New Brunswick and a fourth in northern Maine. The New Brunswick stands had been sprayed at various times since 1952; the Maine stand was sprayed experimentally for this study. All stands consisted of second growth fir, *Abies balsamea* (L) Mill., and spruce, *Picea rubens* Sarg. and *P. glauca* (Moench) Voss.

Several sampling points were selected along a line of travel through each stand. The line of travel was arranged to traverse gradients such as slopes and to avoid obvious heterogeneities in the vegetation and soil. Samples were taken from each soil horizon 10 meters in the cardinal directions from the point selected on the line of travel; thus each sample consisted of four subsamples. The organic layers, the A_{00} and the A_0 horizons, were sampled separately on the basis of area, 464.5 cm^2 (one-half of 1 ft^2) being obtained from each subsample point. Analyses from these organic horizons could then be expanded directly to kilograms of DDT per hectare. A composite sample of approximately 2 liters was obtained for each sampling point from each of the mineral horizons.

To calculate the total amount of DDT in the mineral horizons, a single soil pit was dug in each stand and a profile description was recorded. Three "undisturbed" samples were removed in 6.4-cm (2½-in.) diameter steel cylinders from each mineral horizon, and volume weight was determined from these. Determinations of DDT were expressed as DDT per unit weight of soil (air-dry) and were expanded to a land area basis by using the total weight-per-hectare of soil in each horizon. Benzene extraction and the Downing-Norton modification of the Schechter technique were used for the DDT analyses (*2*). Control samples were from unsprayed areas. Recovery of known quantities of DDT applied to these soils was 95 percent or higher. Standard errors were calculated for the means of samples from each horizon in each stand.

Stand 1 was near the Budworm City Airport and was sprayed in 1952 with 0.89 kg of DDT per hectare (1 lb/acre) (*3*) and in the following years through 1958 with 0.45 kg per hectare. Total dosage during the 6-year period was 3.57 kg per hectare. Soils were sampled in 1958, 1960, and 1961. The samples collected in 1958 were from six sampling points, those collected in 1960 were from five points, and those in 1961 from two points. Total amounts of DDT in the soil profile increased annually in the samples of 1958 through 1961 (Table 1), although there was no application of DDT after the original sampling in 1958. In that year the soil profile contained about 0.45 kg of DDT per hectare. In 1960 the profile contained about 1.5 kg per hectare, approximately three times the amount of 1958;

Table 1. DDT recovered from the soil profile of stand 1. A total of 3.57 kg of DDT per hectare (4.0 lb/acre) was applied between 1952 and 1958. This contained 2.49 kg of *para-para* isomer and 1.08 kg of *ortho-para* isomer per hectare. Results expressed as kilograms per hectare.

Soil horizon	1958	1960	1961
		Total DDT	
A_{00}	0.169 ± .025	0.467 ± .080	0.628 ± .321
A_0	.089 ± .008	.782 ± .339	.544 ± .098
A_2	.032 ± .014	.086 ± .029	.316
B_{21}	.119 ± .075	.033 ± .018	.134
B_{22}		.128 ± .080	
C		0	
Total	.409 ± .080	1.496 ± .360	1.622
		p-p *Isomer*	
A_{00}	0.102 ± .024	0.230 ± .045	0.251 ± .139
A_0	.033 ± .004	.349 ± .102	.244 ± .046
A_2	.004 ± .003	0	0
B_{21}	0	0	.045
B_{22}		.037 ± .037	
C		0	
Total	.139 ± .023	.616 ± .154	.540
		o-p *Isomer*	
A_{00}	0.068 ± .022	0.237 ± .040	0.377 ± .257
A_0	.055 ± .006	.433 ± .091	.301 ± .055
A_2	.029 ± .014	.086 ± .029	.316
B_{21}	.119 ± .075	.033 ± .018	.089
B_{22}		.091 ± .055	
C		0	
Total	.271 ± .077	.880 ± .216	1.083

and in 1961 about 1.6 kg per hectare. In all years more than half of the total amount of DDT present was retained in the organic horizons; the principal increase from year to year was in these layers as well.

The two isomers of DDT, *para-para* and *ortho-para*, occur in technical grade DDT in the ratio of approximately 70 percent *para-para* to 30 percent *ortho-para*, and analysis of the sprays used in 1958 and subsequently in Maine confirmed the ratio for those sprays. Table 1 shows, however, that residues recovered from the soil contained in most instances more *ortho-para* isomer than *para-para*. The proportion of *ortho-para* was generally highest in the deeper horizons, lowest in surface organic layer (*4, 5*).

Stands 2 and 3 were replicates, one of the other, selected because their spraying history was known in detail and because they were representative of extensive sprayed stands in the Northeast. Both had received total deposits of 2.2 kg of DDT per hectare applied at the rate of 0.89 kg per hectare in 1952 and 0.45 kg per hectare in 1953, 1956, and 1957. Samples were obtained in 1960 and in 1961. The analyses reported in Table 2 are for two points sampled in 1960 and for five points sampled in 1961. In the samples collected from stand 2 in 1961, a faulty solution caused the loss of the analyses for the mineral horizons. Table 2, however, shows that in that year there was about 0.65 kg of DDT per hectare in the organic horizons, approximately the same amount as there was in the total horizon in the previous year. This suggests that the total in the soil profile was greater in 1961, the increase paralleling the increase in the other stands.

In stand 3 samples were also obtained from two points in 1960 and from five points in 1961. The total amount of DDT present in 1960 was approximately 0.22 kg per hectare and in 1961, more than five times that amount, about 1.15 kg per hectare.

Stand 4, a fir-spruce stand on the west bank of the Machias River at Russell's Crossing, was sprayed experimentally in June of 1960 with 3.57 kg of DDT per hectare. Soil samples were obtained from two sampling points in August of 1960 and the analyses appear in Table 3. There was at that time about 0.127 kg of DDT per hectare in the soil. This total was distributed between the organic horizons and the A_2 horizon. No DDT appeared in the B horizon.

The persistence of DDT residues in crop soils has been reported by several authors (*6*); it was also reported in a forest soil by Woodwell (*5*) for stand 1 at Budworm City. The data presented here supplement those published previously for the Budworm City stand and suggest that the residues were still increasing in total amount in these and other nearby soils in 1961, during the third year after the last spraying in the area.

The mechanisms responsible for the buildup of DDT residues in the soil for several years following cessation of spraying are not known. One of the most reasonable hypotheses is that residues persist in the tree tops for as long as several years and are carried to the soil by rain and by the fall of leaves and other organic matter. In the experimentally sprayed stand in Maine, 0.127 kg of DDT per hectare was recovered from the soil in August after a June spraying with 3.57 kg per hectare. An aluminum foil sheet exposed on the ground in the center of the stand during the spraying received 0.040 kg per hectare. Other foil sheets and dye-cards (*7*) placed in roads along two margins of the stand received about 3.57 kg per hectare, indicating that the planned dose was indeed applied. These data show that less than 5 percent of the applied spray penetrated the canopy of this closed-canopy fir-spruce stand during the first 2 months after aerial spraying. Yet in the Canadian stands sampled 3 years after completion of the last spraying, as much as 50 percent of the total DDT applied was recovered in the soil profile. Evidently, whatever the mechanics of the migration and accumulation in the soil, DDT residues do accumulate and persist for substantial periods, probably longer

Table 2. DDT in soils at stands 2 and 3 near Budworm City, New Brunswick. Samples taken in 1960 were from two points in each stand; those in 1961 from five points. Spray was applied in 1952 at 0.89 kg of DDT per hectare (1 lb/acre) and in 1953, 1956, and 1957 at 0.45 kg/hectare (½ lb/acre) making a total of 1.34 kg of DDT per hectare (2½ lb/acre). In 1961, mineral horizon soils were analyzed for a single sampling point in stand 3 only.

Soil horizon	1960	1961
	Stand 2	
A_{00}	0.115 ± .071	0.434 ± .030
A_0	.057 ± .005	.212 ± .094
A_2	.479 ± .309	
B_2	0	
Total	.651 ± .241	.646 ± .109 1.125*
	Stand 3	
A_{00}	0.089 ± .016	0.473 ± .087
A_0	.055 ± .021	.447 ± .074
A_2	.054 ± .054	.211
B_2	0	.093
C	0	
Total	.198 ± .091	1.224

* Corrected for estimated minimum in mineral horizons.

Table 3. DDT recovered in August 1960 from soil of experimental stand 4, Maine, sprayed in June 1960 with 3.57 kg of DDT per hectare (4 lb/acre). The stand had never been sprayed before June 1960.

Soil horizon	August 1960
A_{00}	0.034 ± .019
A_0	.013 ± .012
A_2	.080 ± .027
B_2	0
Total	.127 ± .037

than the 10 years previously estimated (*5*).

There is at present no evidence that the persistence of DDT residues in soils is affecting plant populations in any measurable way (*8*). There is abundant evidence, however, that food chains are contaminated in various ways, and the persistence of residues of DDT in soils for as much as a decade is ample reason to examine the cycling of this and other noxious products through ecological systems with great care.

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High-Pressure Polymorph of Iron

Abstract. *An x-ray diffraction study of iron under pressure has shown that alpha-iron (body-centered cubic) transforms to hexagonal-close-packing (designated epsilon-iron) at 130 kb, room temperature. The volume change for the transformation is − 0.20 ± 0.03 cm³/mole. The slope for the gamma-epsilon phase boundary has been calculated to be 2 ± 1°C/kb. The molar volume of iron decreases from 7.10 cm³/mole (density = 7.85 g/cm³) at room pressure to 6.10 ± 0.08 cm³/mole (density = 9.1 ± 0.1 g/cm³) at 200 kb and room temperature. The extrapolation of the gamma-epsilon phase boundary, consistent with shock wave data, suggests that it may have an influence on the properties of the earth's core.*

Three polymorphic phases of iron have been recognized: α-iron (body-centered cubic) is stable below 906°C at room pressure, γ-iron (face-centered cubic) is stable between 906°C and 1401°C, and δ-iron (body-centered cubic) is stable between 1401°C and 1530°C, the melting temperature of iron at room pressure. The effect of pressure on the phase boundary between α- and γ-iron has been investigated over a wide range of pressures by means of dilatometry by Birch (*1*), electrical resistivity by Kaufman *et al.* (*2*) and Clougherty and Kaufman (*3*), heat of transformation by Kennedy and Newton (*4*), thermal conductivity by Claussen (*5*), and shock wave propagation by Bancroft *et al.* (*6*) and Johnson *et al.* (*7*). The results of these studies have established that the temperature of the α–γ iron transformation decreases with increasing pressure. The electrical resistivity study of Balchan and Drickamer (*8*) has shown that there is a phase transformation at 130 kb at room temperature. It was considered that, because of the negative slope of the phase boundary, the γ-iron might become the stable phase above 130 kb at room temperature. On the other hand, Johnson *et al.* (*7*) showed that a sharp change occurs in the slope of the α–γ phase boundary at 110 kb and 502°C, and proposed that this inflection point represents a triple point for alpha, gamma and X phases. Jamieson and Lawson (*9*), and Jamieson (*10*) made an attempt to identify this high pressure phase by x-ray diffraction. Working with one diffraction line and the volume change for the phase transformation obtained by Bancroft *et al.* (*6*), they postulated that the high-pressure phase has a hexagonal-close-packed structure. However, the evidence was inconclusive. The purpose of the study reported here was to confirm their hypothesis.

A mixture of powdered iron (purity, 99.8 percent) and sodium chloride was placed between two flat faces (0.5 mm diameter) of diamond anvils in an instrument similar to the one designed by Van Valkenburg (*11*) for his optical work. A finely collimated x-ray beam (0.1 mm diameter, MoKα radiation) was directed to the specimen through the diamond anvil, and the diffracted x-rays were recorded on a flat photographic film. The sodium chloride serves two purposes, being a pressure-transmitting medium and an internal standard for pressure measurements. The pressure to which the specimen was subjected was calculated from the lattice dimensions of the sodium chloride and the available data on density obtained by Bridgman (*12*) for up to 40 kb and by Christian (*13*) for above 40 kb. A 20°C isothermal pressure-volume relation of sodium chloride was derived by Christian from his shock-experiment data by assuming the constancy of the heat capacity and $(\partial P/\partial T)_v$ over the pressure range of present interest. The uncertainty of the pressure-volume relations of Christian is ± 3 percent, and the overall uncertainty of the pressure values reported in this paper is ± 9 percent. Although Evdokimova and Vereshchagin (*14*) reported an extremely sluggish phase transformation of sodium chloride to a cesium chloride type structure below 18 kb, such a transformation was not observed by us.

The photographic records of x-ray diffraction obtained at room pressure,

Table 1. X-ray diffraction data of iron at 192 ± 18 kb and room temperature. MoKα radiation; zirconium filter. Unit cell dimensions were: $a = 2.45 \pm 0.01$ Å, $c = 3.93 \pm 0.01$ Å, $c/a = 1.61$. Molar volume = 6.16 ± 0.08 cm³/mole density = 9.1 ± 0.1 g/cm³. Compressibility $(\beta_{192}) = -(1/V_0)(\partial V/\partial P)_{20^\circ\mathrm{C}} = 4.94 \times 10^{-7}$ compressibility at 1 bar and 20°C $(\beta_0) = 5.94 \times 10^{-7}$.

I/I_0	d (Å)	(hkl)	Phase
	2.50	(200)	NaCl std.*
2	2.12	(100)	ε
8	1.97	(002)	ε
10	1.84	(101)	ε
1	1.44	(102)	ε
1	1.234	(110)	ε
1	1.113	(103)	ε

* NaCl internal standard.