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A Single-Crystal Neutron-Diffraction Study of Urea–Phosphoric Acid*

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The 1:1 compound of urea and phosphoric acid, $(NH_2)_2CO.H_3PO_4$, has been studied by single-crystal neutron-diffraction techniques. All atoms, including the hydrogen atoms, have been accurately located. The carbonyl oxygen atom of urea is linked to the phosphoric acid moiety by a short hydrogen bond in which an acid hydrogen atom is nearly centered. Each of the remaining hydrogen atoms participates in an ordinary hydrogen bond. The space group is orthorhombic *Pbca* with lattice parameters $a=17\cdot6492$ (6), $b=7\cdot4820$ (3), and $c=9\cdot0018$ (4) Å at 23°C.

Introduction

Urea and phosphoric acid form a 1:1 crystalline compound, $(NH_2)_2CO.H_3PO_4$, which has been examined by X-ray diffraction techniques by Sundera-Rao, Turley & Pepinsky (1957) and independently by Wolfram, Arutunian, Antishkina, & Porai-Koshits (1967). The symmetry is that of the orthorhombic space group *Pbca*, and there are eight formula units per cell.

Although the early studies did not reveal the hydrogen atom positions, they did show a short O···O distance between the two moieties. Sundera-Rao, Turley and Pepinsky suggested that this represents a strong hydrogen bond in which the hydrogen atom is nearly equidistant from the two oxygen atoms.

We report here a single-crystal neutron-diffraction study of this compound which accurately locates all the hydrogen atoms and clearly shows this centered hydrogen bond. After our work was completed we learned that Mootz & Albrand (1972) had made a precise X-ray diffraction study of this material which also located the hydrogen atoms. There are no major discrepancies between their results and ours.

Two other urea–acid compounds which have been studied recently also have hydrogen bonds between the acid and the carbonyl oxygen of urea. In the 1:1 compound formed from urea and nitric acid, Worsham & Busing (1969) found that the acid proton had transferred completely from the nitrate group to the carbonyl oxygen. This material should therefore be classed as a salt, uronium nitrate.

In the 2:1 compound of urea and oxalic acid (Harkema, Bats, Weyenberg & Feil, 1972) on the other hand, the acid proton remains as part of the oxalic acid molecule and forms an ordinary hydrogen bond. This material is therefore an addition compound.

The urea–phosphoric acid of the present study must be regarded as intermediate between these two extremes. It is clear that the differences between the structures of these compounds must be related qualitatively to the relative strengths of the acids involved, but we are not prepared to present a quantitative theory at this time.

Experimental

Sample crystals were obtained by slow evaporation of an aqueous solution containing equimolar quantities of urea and phosphoric acid. Lattice parameters were redetermined from X-ray diffractometer measurements of 12 high-angle reflections using the method of least-squares (Busing & Levy, 1967). At 23°C they are $a=17\cdot6492(6)$ $b=7\cdot4820$ (3), and $c=9\cdot0018$ (4) Å, based on a wavelength of 1·54051 Å for Cu $K\alpha_1$ radiation.

The sample for neutron diffraction weighed 69·3 mg and measured between 3 and 5 mm in each dimension. Integrated intensities of 1926 independent reflections were measured on the Oak Ridge automatic diffractometer (Busing, Smith, Peterson & Levy, 1964). These were put on an absolute scale by comparison with the intensity from a NaCl standard crystal of known weight. The temperature of measurement was 25°C, and the neutron wavelength was 1·077 Å.

Transmission factors which ranged from 0·45 to 0·59 were calculated (Busing & Levy, 1957) using a theoretical absorption coefficient $\mu=2\cdot23$ cm^{-1}. The measurements were reduced to structure factors in the usual way (Brown & Levy, 1964). The squares of these structure factors, which have been corrected for extinction as described below, are listed in Table 1 together with their standard errors.

Calculations

Approximate hydrogen positions were determined from a Fourier synthesis using observed structure-factor magnitudes with signs based on the heavy-atom coordinates reported by Sundera-Rao, Turley & Pepinsky (1957).

In the early stages of least-squares refinement it became apparent that the data suffer from the effects of

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severe extinction. Extinction corrections were therefore included in the refinement using the expression of Zachariasen (1967) in anisotropic form as described by Coppens & Hamilton (1970). The assumption of Type II extinction (dominated by mosaic block size) gave a slightly better fit than did Type I (dominated by angular mosaic spread).

The calculated extinction factors by which the squares of the observed structure factors have been divided are included in Table 1. Apparently extinction has

Table 1. *Observed squares of the structure factors for urea–phosphoric acid*

The columns headed H, OBS, SIG, and EXT contain, respectively, the index *h*, the observed F^2 (corrected for extinction) and its standard error in units of 10^{-25} cm^2, and the calculated extinction factor ($\times 10^3$). The original uncorrected value of F^2 and its standard error may be obtained by multiplying OBS and SIG by EXT. The indices *k* and *l* are given in the subheadings.

H	OBS	SIG	EXT
** H	0	0	********
2	57	3	355
4	123	7	342
6	6	3	915
8	174	10	424
10	4	4	980
12	0	4	980
14	382	18	397
16	676	28	333
E18	1506	58	238
20	203	14	564
22	-2	8	950
24	11	8	967
** H	1	0	******
2	8	2	836
E 4	2266	76	88
E 6	483	19	244
8	4	3	987
10	8	4	962
12	493	22	317
14	313	16	426
E16	1264	47	247
18	434	21	426
20	87	10	730
22	186	15	593
24	7	8	946
** H	2	0	******
E 0	2018	68	79
2	12	2	883
4	298	13	296
6	56	5	661
8	20	4	895
10	357	17	361
12	56	6	712
14	1	4	999
16	775	34	305
18	116	10	676
20	560	27	381
22	13	9	914
24	15	8	987
** H	3	0	******
2	125	7	446
4	344	15	306
6	38	5	731
8	86	7	620
10	107	8	600
E12	2220	78	165
14	172	11	562
16	253	16	498
18	9	7	916
20	1	8	941
22	294	20	498
24	135	13	647
** H	4	0	******
E 0	1718	62	126
2	5	3	975
E 4	1584	57	148
E 6	885	34	220
8	107	8	582
10	12	5	913
E12	1154	44	240
14	4	6	974
16	877	37	292
18	955	41	293
20	3	8	919
22	12	8	988
** H	5	0	******
2	502	23	262
4	346	17	344
6	222	12	436
8	472	21	329
10	124	9	574
12	114	11	580
14	1	6	983
16	3	7	970
18	80	11	712
20	33	10	867
22	629	31	372
** H	6	0	******
E 0	959	39	207
2	552	25	266
4	417	20	316
6	105	9	572
10	92	10	629
12	744	34	297
14	9	6	998
16	20	9	847
18	291	21	469
20	-6	8	999
** H	7	0	******
2	365	21	347
4	57	9	674
6	604	30	286
8	155	13	523
10	131	13	565
12	108	12	588
14	14	8	919
16	41	11	796
18	9	9	899
** H	8	0	******
0	804	37	249
2	136	12	515
4	1	6	998
6	247	18	423
8	553	30	305
10	1	8	964
12	11	7	993
14	100	13	639
16	-14	8	993
** H	9	0	******
2	32	9	842
4	103	13	583
6	16	10	747
8	0	8	999
10	140	17	506
12	23	9	908
14	138	15	554
** H	10	0	******
0	-2	8	988
2	23	8	916
4	11	9	588
6	361	27	365
8	12	10	807
10	-1	8	897
** H	1	1	******
1	146	6	318
2	28	2	676
3	54	4	587
4	-1	2	998
5	1	2	992
6	235	11	359
E 7	1015	38	172
8	16	4	843
9	272	13	343
10	390	18	343
11	65	6	706
12	19	5	688
13	458	21	355
14	620	26	322
15	16	5	895
16	8	6	972
17	22	7	892
18	136	11	655
19	64	10	735
20	5	7	998
21	13	8	905
22	-12	8	995
23	29	8	958
24	6	8	980
25	85	12	712
** H	2	1	******
0	10	2	856
1	43	3	634
2	269	11	299
E 3	804	29	174
4	5	2	965
5	10	3	914
6	23	4	611
7	30	4	771
H	2	1	********
E 8	907	34	216
9	0	4	999
E10	890	35	231
11	7	4	952
12	31	5	824
13	256	14	467
14	-4	5	987
15	437	20	380
17	125	11	640
18	51	8	776
19	9	7	908
20	235	15	550
21	316	20	477
22	7	8	998
23	11	8	965
24	9	8	955
25	59	10	772
** H	3	1	******
E 1	639	25	202
E 2	586	24	210
3	14	3	918
4	280	13	353
5	397	17	308
6	7	3	920
7	395	17	336
8	58	6	715
9	202	11	457
10	37	5	782
11	434	20	347
12	6	4	992
13	390	18	391
14	33	6	825
15	228	15	510
16	102	10	677
17	25	7	904
18	106	11	686
19	233	15	551
20	-26	8	985
21	48	10	766
22	22	9	911
23	-6	8	989
24	26	8	924
** H	4	1	******
0	88	7	546
1	157	9	464
2	236	12	383
3	10	4	936
4	3	3	997
5	240	12	404
6	350	16	353
7	34	5	819
8	87	7	627
9	170	10	514
10	47	6	769
11	3	4	999
12	161	10	556
13	79	8	687
14	32	7	857
15	11	6	919
16	6	6	948
17	4	7	984
18	23	8	897
19	1	8	983
20	3	8	960
21	57	11	759
22	37	10	834
23	127	13	677
** H	5	1	******
1	-2	4	994
2	60	6	668
3	145	9	505
4	387	19	318
5	3	4	996
6	70	7	672
7	15	5	901
8	56	6	731
E 9	2192	78	164
10	122	9	575
11	228	13	486
12	80	9	677
13	497	24	364
14	30	7	846
15	10	6	915
16	21	7	989
17	31	9	844
18	-9	7	999
19	20	9	893
20	7	8	997
21	6	9	953
22	115	13	646
** H	6	1	******
0	17	5	883
1	5	4	938
2	17	5	894
3	527	23	296
4	222	13	422
5	382	19	352
6	124	9	570
7	17	5	882
8	428	22	363
9	173	13	504
10	7	7	886
11	12	6	922
12	101	10	640
13	32	8	861
14	2	7	985
15	97	14	657
16	15	9	862
17	12	9	862
18	99	13	673
19	211	19	523
20	47	11	746
21	15	8	930
** H	7	1	******
1	614	35	280
2	69	8	692
3	-7	5	995
4	20	7	815
5	108	11	585
6	13	6	905
7	145	12	534
8	64	9	692
9	575	27	325
10	36	9	759
11	69	10	667
12	191	15	503
13	207	17	502
14	37	10	765
15	8	8	919
16	21	10	840
17	8	10	856
18	-2	8	976
19	249	19	487
** H	8	1	******
0	-1	6	978
1	7	6	973
2	6	6	925
3	-4	7	930
4	123	12	561
5	7	7	909
6	11	7	922
7	34	9	757
8	104	12	577
9	103	12	646
10	16	8	900
11	-12	8	916
12	12	8	916
13	109	14	614
14	7	8	997
15	190	19	499
16	111	14	609
17	55	12	713
** H	9	1	******
1	79	17	531
2	-1	8	887
3	423	27	343
4	129	16	491
5	-3	7	985
H	9	1	********
6	104	14	580
7	264	23	392
8	6	9	947
9	23	9	920
10	15	10	837
11	13	9	868
12	-1	9	856
13	-4	8	935
14	82	14	604
** H	10	1	******
0	401	27	376
1	77	13	645
2	72	13	624
3	51	12	685
4	51	11	744
5	15	9	890
6	0	9	875
7	57	11	746
8	2	8	953
9	10	9	876
10	46	10	744
** H	0	2	******
E 1	411	18	172
E 2	1169	43	103
3	72	5	499
4	119	7	445
5	136	8	425
6	54	5	659
E 7	907	35	185
8	124	8	510
9	33	5	769
E10	2023	72	148
11	602	26	288
12	188	11	502
13	534	23	340
14	156	10	580
15	188	11	557
16	686	31	329
17	736	33	321
18	838	36	315
19	56	9	817
20	72	9	786
21	58	9	825
22	331	21	465
24	26	8	925
25	2	8	960
** H	1	2	******
1	120	6	454
E 2	766	28	168
E 3	908	33	163
4	121	7	488
5	108	7	517
6	217	11	399
7	134	8	498
8	167	9	489
9	31	5	826
10	36	5	768
11	-15	4	994
12	355	17	402
13	44	6	776
E14	795	32	291
15	298	15	463
16	590	27	349
17	10	6	999
18	136	11	630
19	86	10	720
20	76	9	775
21	9	8	980
22	-5	8	998
23	95	12	756
24	48	10	831
25	4	8	977
** H	2	2	******
0	6	3	880
1	188	9	393
E 2	905	33	170
3	111	6	525
4	14	3	849
5	115	7	508
E 6	990	36	200
7	148	8	526
8	141	9	512
9	546	23	303
10	58	6	718
11	128	9	596
12	14	5	900
13	240	14	470
14	567	24	350
15	192	11	561
16	5	6	939
17	-7	6	954
18	488	23	411
19	79	10	764
20	44	9	860
21	9	8	935
22	43	9	878
23	125	14	664
24	14	8	972
25	-3	8	963
** H	3	2	******
1	73	5	609
2	6	3	999
3	87	6	607
4	158	9	474
5	7	4	905
E 6	537	22	282
7	110	7	599
8	23	5	818
9	28	5	833
10	147	9	573
11	18	5	897
12	511	23	340
13	273	14	464
14	267	14	479
15	264	16	490
16	42	8	822
17	28	7	840
18	104	12	663
19	253	20	518
20	94	14	634
21	180	15	613
22	5	8	971
23	17	8	932
24	-22	8	997
** H	4	2	******
0	2	3	981
1	404	18	309
E 2	1242	46	167
3	167	10	456
4	31	5	786
5	307	15	385
6	513	22	305
7	70	6	683
8	41	5	783
9	99	8	628
10	5	4	979
11	212	12	497
12	1	5	985
13	45	8	773
14	300	17	469
15	50	8	777
16	90	10	676
17	69	10	735
18	101	13	727
19	-4	7	994
20	39	9	823
21	11	9	928
22	6	8	999
23	51	10	779
** H	5	2	******
1	142	9	511
2	464	21	310
3	20	5	850
4	212	12	446
5	35	5	806
6	515	22	315
7	1	4	999
8	2	4	995
H	5	2	********
9	270	15	428
10	474	22	353
11	-1	6	999
12	239	15	465
13	-3	6	992
14	33	7	831
15	48	8	806
16	31	8	841
17	69	11	741
18	20	7	984
19	-18	8	995
20	99	12	702
21	32	10	844
22	57	11	746
** H	6	2	******
0	249	14	411
1	114	9	557
2	35	6	741
3	36	6	775
4	69	7	630
5	619	27	281
6	87	8	622
7	128	11	589
8	13	6	888
9	135	11	574
10	99	10	613
11	102	10	649
12	14	7	949
13	18	8	879
14	194	15	528
15	259	19	472
16	-5	7	999
17	4	9	930
18	13	9	926
19	42	11	782
20	215	17	533
21	-7	8	999
** H	7	2	******
1	199	15	435
2	14	6	910
3	118	10	593
4	122	11	562
5	99	10	606
6	607	30	295
7	8	6	892
8	12	6	908
9	7	7	975
10	83	12	616
11	12	7	984
12	83	12	666
13	54	11	741
14	12	7	994
15	32	10	865
16	103	14	640
17	-7	8	995
18	14	9	975
19	78	12	691
** H	8	2	******
0	37	9	750
1	149	13	536
2	70	10	671
3	39	9	760
4	25	8	841
5	16	8	844
6	-3	6	978
7	35	8	803
8	26	8	965
9	-7	7	993
10	188	18	482
11	17	8	890
12	109	14	613
13	10	10	868
14	24	10	862
15	131	15	589
16	579	33	337
17	-16	8	995
** H	9	2	******
1	12	7	999
2	15	8	882
3	85	12	632
4	19	9	804
5	17	9	823
6	395	29	322
7	2	9	951
8	92	16	559
9	86	15	608
10	26	9	853
11	8	8	936
12	247	20	465
13	24	9	841
14	6	8	954
** H	10	2	******
0	151	16	554
1	105	15	564
2	27	10	830
3	3	8	953
4	24	11	757
5	4	8	987
6	6	9	905
7	6	7	987
8	0	9	855
9	41	11	694
** H	1	3	******
1	45	4	713
2	70	5	614
3	195	10	396
4	109	7	534
5	429	18	295
E 6	681	27	240
7	33	5	787
8	189	11	479
9	103	8	606
10	39	6	769
11	51	6	742
12	122	8	634
13	-7	4	997
14	50	7	766
15	495	24	373
16	124	11	632
17	-6	6	998
18	7	6	991
19	40	8	889
20	46	9	874
21	41	9	825
22	2	8	999
23	-8	8	999
24	38	10	829
** H	2	3	******
0	73	5	622
2	119	7	522
3	169	9	447
E 4	538	22	267
5	28	4	770
6	260	13	404
7	374	17	361
8	221	12	471
9	331	16	400
10	521	23	327
E11	791	32	270
12	243	13	484
13	409	19	408
14	53	7	739
15	64	9	750
16	250	15	510
17	23	7	910
18	10	7	972
19	25	8	893
20	3	8	999
21	27	8	912
22	20	9	896
23	107	13	681
24	154	15	597
** H	3	3	******
1	-1	3	999
E 2	588	24	253
3	465	20	291
4	4	4	938
E 5	557	21	282
H	3	3	********
6	473	20	321
7	72	6	682
8	216	12	475
9	79	7	711
10	53	6	744
11	228	13	490
12	468	21	371
13	179	11	553
14	198	13	552
15	370	19	435
16	7	6	999
17	20	8	851
18	35	9	799
19	67	9	853
20	-3	8	987
21	26	9	916
22	7	8	999
23	17	9	901
** H	4	3	******
0	24	4	806
1	480	21	295
2	83	6	643
3	208	11	453
4	15	4	869
5	101	8	593
6	101	8	618
7	213	12	472
8	620	26	300
9	33	6	812
10	59	7	733
11	31	6	839
12	699	31	310
13	62	9	721
14	19	7	849
15	9	6	983
16	18	7	957
17	35	8	856
18	45	9	838
19	3	7	994
20	25	10	831
21	113	13	690
22	91	13	685
23	66	11	731
** H	5	3	******
1	145	9	532
2	539	23	298
3	93	8	620
4	215	12	466
E 5	776	32	253
6	29	5	837
7	309	16	401
8	13	5	874
9	313	16	433
10	6	6	999
11	602	28	327
12	64	9	726
13	190	13	563
14	5	7	988
15	72	10	703
16	16	10	903
17	18	8	918
18	24	9	868
19	-3	8	983
20	-8	9	948
21	7	8	994
22	44	10	802
** H	6	3	******
0	258	14	428
1	154	10	512
2	21	5	823
3	9	4	980
4	3	4	951
5	153	10	551
6	178	13	515
7	650	30	299
8	568	27	333
9	3	6	983
10	-7	6	990
11	32	7	875
12	73	9	739
13	40	8	821
14	-3	9	986
15	10	8	912
16	86	12	661
17	54	11	771
18	84	13	665
19	62	12	729
20	112	13	654
** H	7	3	******
1	231	16	462
2	33	8	760
3	-1	6	986
4	318	19	397
5	45	8	713
6	269	17	445
7	159	12	548
8	7	7	997
9	738	35	294
10	149	14	549
11	559	33	345
12	20	8	943
13	4	7	993
14	15	8	915
15	46	10	823
16	96	14	647
17	17	8	957
18	3	9	891
** H	8	3	******
0	6	7	951
1	15	7	908
2	8	6	985
3	5	7	940
4	38	9	752
5	60	9	733
6	89	13	650
7	72	12	635
8	97	12	624
9	15	9	860
10	18	7	959
11	83	14	637
12	14	9	907
13	4	8	999
14	-7	8	992
15	-3	8	961
16	7	9	942
** H	9	3	******
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2	26	8	880
3	61	12	656
4	22	10	763
5	251	21	447
6	9	9	904
7	-2	8	979
8	156	17	505
9	6	8	984
10	59	12	705
11	93	13	634
12	9	8	921
13	125	14	583
** H	10	3	******
0	159	15	566
1	44	10	772
2	-3	8	963
3	17	9	869
4	37	12	684
5	88	15	555
6	33	11	734
7	16	9	845
8	50	11	714
** H	0	4	******
E 0	2007	70	115
1	95	7	517
E 2	592	26	208
E 3	765	32	195
E 4	573	25	227
E 5	1129	44	172
6	61	6	662
7	303	16	366
H	0	4	********
E 8	1084	42	205
E 9	1093	43	207
E10	1379	52	202
E11	832	34	263
12	128	9	591
13	437	20	401
14	58	7	746
15	28	7	856
E16	1058	42	286
17	3	6	968
18	193	14	599
19	3	7	999
20	70	11	742
21	174	14	625
22	-7	9	968
23	54	11	754
24	-19	8	969
** H	1	4	******
1	17	4	804
2	9	3	888
3	50	5	722
4	4	3	996
E 5	903	34	218
6	104	7	601
7	260	13	418
8	159	10	536
9	414	19	355
10	349	18	385
11	193	11	528
12	12	5	886
13	19	5	919
14	3	5	975
15	267	16	487
16	320	18	473
17	-10	6	999
18	477	24	425
19	113	13	703
20	24	8	922
21	99	13	705
22	33	9	920
23	2	8	970
24	33	9	861
** H	2	4	******
E 0	2007	76	163
1	50	5	699
E 2	480	20	289
3	173	9	481
4	78	6	639
E 5	717	28	253
6	110	8	601
7	55	6	757
E 8	728	29	277
E 9	757	30	283
10	22	5	893
11	640	27	318
12	528	23	358
13	56	7	784
14	-6	6	985
15	320	17	476
16	14	7	911
17	27	9	811
18	120	12	664
19	60	10	767
20	117	12	694
21	104	12	738
22	11	9	933
23	105	13	694
24	7	8	991
** H	3	4	******
1	450	19	315
2	4	4	953
3	22	4	860
4	2	4	996
5	82	7	657
6	145	9	532
7	362	17	380
8	120	8	618
9	186	11	593
10	22	5	925
11	10	5	987
12	86	8	705
13	132	11	644
14	462	23	402
15	8	6	948
16	8	6	990
17	74	10	721
18	0	8	961
19	-31	8	998
20	155	15	629
21	146	15	627
22	70	11	764
23	-4	8	984
** H	4	4	******
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1	21	4	865
2	40	5	779
E 3	752	30	259
E 4	847	33	245
5	253	13	432
6	681	28	289
7	471	21	343
8	58	6	748
9	69	7	701
10	27	6	823
11	439	22	349
12	243	16	477
13	419	23	383
14	236	15	530
15	173	14	575
16	-2	7	993
17	54	10	792
18	6	8	990
19	231	18	550
20	12	10	879
21	409	24	442
22	7	8	973
** H	5	4	******
1	420	19	359
2	12	5	884
3	339	16	389
4	32	5	829
5	34	5	826
6	31	6	819
7	169	11	540
9	107	11	606
10	-2	6	998
11	14	6	968
12	31	7	836
13	26	8	857
14	26	9	775
15	54	9	807
16	54	10	778
17	32	9	869
18	33	9	892
19	-10	8	993
20	-4	8	964
21	21	9	876
** H	6	4	******
0	596	26	299
1	22	5	837
2	51	8	717
3	218	14	487
4	128	11	571
5	8	6	909
6	48	8	771
7	512	25	340
8	2	6	964
9	17	7	869
10	32	7	854
11	5	7	980
12	9	7	950
13	32	11	799
14	11	8	910
15	11	8	903
16	19	10	900
17	30	11	783
18	120	14	641
19	23	9	856
H	6	4	********
20	43	9	822
** H	7	4	******
1	289	17	428
2	0	6	952
3	70	9	694
4	108	10	640
5	12	6	955
6	4	7	961
7	141	12	582
8	1	7	973
9	4	7	936
10	22	9	828
11	207	17	514
12	36	10	811
13	-5	7	994
14	88	12	705
15	-2	8	986
16	5	8	975
17	-5	8	990
18	20	9	860
** H	8	4	******
0	18	7	859
1	10	6	968
2	43	9	737
3	640	37	309
4	153	17	533
5	92	12	629
6	10	8	949
7	280	22	411
8	9	8	938
9	25	8	857
10	16	9	936
11	-5	8	994
12	60	11	730
13	51	12	674
14	-1	8	990
15	239	20	450
** H	9	4	******
1	317	25	399
2	87	13	650
3	5	9	949
4	12	9	869
5	21	9	868
6	3	9	893
7	12	8	951
8	31	9	824
9	48	10	814
10	-2	8	999
11	15	9	845
12	-8	9	998
** H	10	4	******
0	-13	8	967
1	10	8	959
2	3	8	963
3	93	12	675
4	160	17	508
5	82	11	426
6	-11	8	970
** H	1	5	******
1	133	8	559
2	3	4	965
3	34	5	799
4	419	19	346
5	55	6	710
6	80	7	660
7	1	5	937
8	345	17	392
9	300	15	434
10	126	9	615
11	1	5	932
12	538	26	353
13	49	10	687
14	1	6	999
15	79	9	764
16	-4	6	990
17	99	11	693
18	42	9	785
19	1	6	995
20	70	10	788
21	9	8	988
22	97	13	697
23	8	9	883
** H	2	5	******
E 0	1857	64	159
1	11	4	958
2	116	8	588
3	321	15	384
E 4	607	25	286
5	385	17	369
6	124	9	581
7	6	4	987
E 8	761	31	285
9	59	6	773
10	759	31	294
11	262	14	473
12	27	6	859
13	153	12	612
14	52	8	817
15	13	8	859
16	279	18	494
18	316	22	463
19	179	14	620
20	26	10	871
21	10	9	956
22	73	11	785
23	27	9	857
** H	3	5	******
1	0	4	984
2	4	4	999
E 3	622	26	283
4	8	4	918
5	143	9	554
6	290	15	427
7	652	27	308
8	109	8	650
9	139	9	596
10	134	9	613
11	4	6	978
12	289	17	471
13	187	13	567
14	58	8	801
15	22	8	858
16	1	7	997
17	10	9	839
18	31	9	826
19	15	9	939
20	-3	9	976
21	30	9	921
22	48	10	794
** H	4	5	******
0	123	8	607
1	17	5	904
2	-4	6	997
3	525	22	328
4	99	8	658
5	7	5	933
6	12	5	894
7	44	6	770
8	579	25	327
9	180	12	569
10	753	33	305
11	-2	6	987
12	38	7	848
13	-3	6	975
14	4	7	999
15	88	12	633
16	-7	8	973
17	10	8	958
18	23	10	865
19	70	12	716
20	32	10	853
21	120	13	687
** H	5	5	******
1	605	25	316
2	150	10	584
3	721	29	291
4	604	26	308
5	59	7	733
H	5	5	********
6	54	8	753
7	91	10	650
8	53	8	785
9	4	6	999
10	8	6	956
11	92	10	670
12	22	7	910
13	41	8	836
14	115	13	682
15	42	10	796
16	5	8	967
17	-12	9	995
18	-5	9	925
19	401	24	449
20	33	9	866
** H	6	5	******
E 0	1699	65	199
1	404	21	379
2	9	6	973
3	4	6	964
4	108	10	622
5	20	6	936
6	216	15	484
7	3	6	924
8	8	6	978
9	8	7	903
10	18	7	966
11	18	7	898
12	210	18	497
13	124	12	649
14	70	11	730
15	314	21	471
16	24	9	950
17	-13	9	963
18	185	17	543
19	50	10	818
** H	7	5	******
1	156	13	555
2	67	10	675
3	772	36	266
4	38	9	749
5	9	7	948
6	0	6	998
7	164	15	506
8	9	8	950
9	-2	7	992
10	62	10	737
11	-5	8	896
12	6	9	970
13	116	15	598
14	91	14	636
15	14	9	903
16	24	9	913
17	62	11	740
** H	8	5	******
0	0	8	928
1	151	16	528
2	0	8	932
3	9	7	962
4	33	10	751
5	27	9	821
6	92	12	628
7	21	9	830
8	10	8	962
9	118	14	613
10	79	13	634
11	1	9	942
12	22	10	818
13	12	8	962
14	56	12	684
** H	9	5	******
1	12	9	864
2	-12	8	991
3	52	12	699
4	78	13	636
5	-5	8	999
6	17	8	924
7	-2	6	964
8	24	9	873
9	278	20	458
10	71	12	667
** H	10	5	******
0	225	19	471
1	-4	8	975
2	46	11	723
3	19	8	887
** H	0	6	******
E 0	849	34	227
1	152	11	495
2	196	12	464
3	166	11	469
E 4	1559	60	167
5	436	21	337
6	419	20	350
E 7	2767	98	140
8	1	5	988
9	105	9	631
10	0	5	983
11	1	5	978
12	622	30	335
E13	2646	95	184
14	5	7	935
15	32	7	872
16	581	28	386
17	-1	9	997
18	46	9	870
19	6	8	985
20	364	23	469
21	147	15	629
22	0	8	961
** H	1	6	******
1	40	5	788
2	106	8	615
3	492	22	327
4	12	5	904
5	28	6	821
6	344	17	418
7	130	9	621
8	304	16	449
E 9	979	39	252
10	32	6	822
11	539	24	356
12	170	13	590
13	30	8	823
14	167	13	592
15	142	12	631
16	93	10	743
17	37	9	860
18	10	8	972
19	93	11	727
20	7	9	980
21	-8	9	972
22	425	24	401
** H	2	6	******
E 0	870	33	264
1	43	5	825
2	7	4	921
3	412	18	363
E 4	870	33	259
5	21	5	868
6	143	10	561
7	702	29	305
8	448	20	388
9	337	16	446
10	5	5	996
11	1	6	973
12	342	18	459
13	74	9	742
14	15	7	956
15	-10	7	992
16	336	20	453
17	83	11	773
18	3	8	927
19	-7	9	999
20	19	10	874
21	64	11	786
22	6	8	955
H	3	6	********
1	9	4	978
2	88	7	689
3	199	12	489
4	37	6	777
5	55	7	741
6	438	20	364
7	113	9	632
8	22	6	811
9	441	23	380
10	373	19	437
11	-3	6	992
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14	55	9	769
15	14	8	922
16	11	8	929
17	7	9	935
18	238	20	502
19	53	12	755
20	-24	9	949
21	7	8	999
** H	4	6	******
E 0	1316	49	219
1	16	5	806
2	223	12	507
3	198	12	509
4	116	9	598
E 5	1122	43	240
6	24	6	849
7	-1	6	969
8	349	19	419
9	274	16	486
10	438	22	404
11	365	20	424
12	15	7	944
13	49	9	801
14	5	7	947
15	4	8	955
16	56	10	785
17	10	9	966
18	-9	9	987
19	8	8	993
20	3	8	967
** H	5	6	******
1	-2	6	967
2	307	17	434
3	37	7	795
4	45	8	780
5	105	10	655
6	-8	6	995
7	84	10	661
8	-3	6	979
9	378	21	408
10	71	10	696
11	167	13	580
12	-5	7	991
13	53	10	801
14	16	9	890
15	117	14	591
16	-9	9	992
17	77	11	774
18	71	12	731
19	72	11	735
** H	0	2	******
0	48	3	562
** H	6	6	******
0	19	7	868
1	45	7	802
2	3	6	996
3	10	6	917
4	1	6	977
5	117	11	624
6	3	7	962
7	224	17	481
8	0	7	948
9	156	13	575
10	-1	8	950
11	313	22	438
12	31	9	844
13	14	10	887
14	74	13	704
15	24	10	857
16	25	10	851
17	2	8	991
18	-8	8	987
** H	7	6	******
1	18	7	876
2	37	8	780
3	15	8	843
4	23	10	842
5	17	8	902
6	104	13	602
7	265	19	474
8	29	9	858
9	3	8	952
10	162	16	563
11	57	12	711
12	118	15	607
13	-1	8	997
14	22	10	821
15	0	8	971
** H	8	6	******
0	13	7	969
1	4	8	955
2	20	9	858
3	280	19	456
4	4	8	996
5	12	9	931
6	2	8	999
7	62	12	724
8	122	16	551
9	17	10	843
10	57	11	759
11	-1	8	966
12	14	8	999
13	44	10	789
** H	9	6	******
1	82	12	670
2	174	17	521
3	-6	9	904
4	14	9	841
5	15	8	972
6	8	8	981
7	4	8	970
8	16	8	893
** H	1	7	******
1	360	16	411
2	47	6	816
3	32	6	747
4	34	6	799
5	256	14	471
6	334	17	429
7	7	5	986
8	304	16	439
9	40	8	808
10	68	9	727
11	81	10	721
12	284	18	475
13	128	11	654
14	-5	7	982
15	76	9	788
16	13	8	987
17	5	8	983
18	7	9	975
19	267	19	541
20	30	9	912
21	18	8	947
** H	2	7	******
0	68	6	734
1	22	5	869
2	159	10	554
3	704	29	294
4	54	7	722
5	7	5	968
6	4	5	965
7	131	9	622
8	25	7	859
9	9	6	993
H	2	7	********
10	771	33	319
11	9	6	999
12	214	14	565
13	34	8	900
14	9	8	931
15	281	21	480
16	187	16	573
17	7	8	982
18	15	9	940
19	132	15	630
20	-8	8	959
21	6	8	964
** H	3	7	******
E 1	1193	44	239
2	9	5	952
3	188	11	535
4	3	4	999
5	30	6	842
E 6	1561	60	211
7	242	15	505
8	246	16	485
9	2	6	946
10	206	14	530
11	41	6	623
12	268	17	507
13	12	7	960
14	269	19	506
15	6	10	940
16	24	8	914
17	25	10	897
18	26	10	859
19	267	20	513
20	39	9	874
** H	4	7	******
0	14	6	689
1	69	8	732
2	38	7	794
3	15	6	884
4	46	8	782
5	25	7	671
6	174	11	600
7	14	7	872
8	10	6	957
9	10	6	979
10	245	17	483
11	82	11	691
12	1	7	980
13	16	9	902
14	2	8	998
15	25	9	861
16	-6	9	986
17	263	20	500
18	29	10	819
19	11	8	990
** H	5	7	******
1	120	11	611
2	90	10	671
3	557	26	344
4	97	10	669
5	43	7	848
6	22	7	855
7	100	11	656
8	-12	7	973
9	-4	7	987
10	21	10	867
11	34	10	826
12	109	13	653
13	284	20	475
14	71	13	717
15	492	30	380
16	18	9	932
17	149	15	621
18	-9	8	999
** H	6	7	******
0	1	7	943
1	23	7	937
2	16	8	828
3	200	15	534
4	-3	7	997
5	16	7	927
6	147	13	578
7	13	9	888
8	20	9	871
9	19	8	918
10	7	7	993
11	56	12	721
12	10	9	919
13	57	12	723
14	-6	8	999
15	37	10	827
16	314	23	441
** H	7	7	******
1	242	19	475
2	289	20	454
3	13	8	968
4	83	11	687
5	60	10	764
6	34	10	776
7	223	19	494
8	47	11	776
9	2	8	989
10	10	9	950
11	120	15	615
12	404	27	385
13	-10	8	989
14	33	10	793
** H	8	7	******
0	283	22	455
1	173	16	557
2	-5	8	992
3	16	8	999
4	14	10	856
5	-1	8	988
6	59	11	737
7	89	13	673
8	57	11	721
9	7	8	990
10	26	8	917
** H	9	7	******
1	4	9	877
2	-15	8	988
3	-7	8	956
4	64	11	726
5	198	18	475
** H	0	8	******
E 0	1787	65	185
1	46	7	755
2	565	27	310
3	53	7	756
4	184	12	521
5	74	10	715
E 6	1087	48	238
7	71	10	711
8	572	28	343
9	3	7	975
10	298	18	480
11	24	8	864
12	171	15	565
13	33	9	815
14	250	19	516
15	55	10	833
16	-3	8	994
17	5	9	969
18	27	10	860
19	20	9	897
20	343	22	470
** H	1	8	******
1	7	5	974
2	536	24	345
3	147	10	607
E 4	1404	56	219
5	210	15	520
6	7	7	952
7	128	12	617
8	276	17	473
9	31	6	844
10	38	8	740
11	59	10	754
H	1	8	********
12	28	8	919
13	45	9	766
14	-11	8	981
15	11	8	999
16	203	17	577
17	18	10	897
18	36	9	899
19	23	9	932
20	214	17	553
** H	2	8	******
0	156	11	604
1	168	12	580
2	347	19	424
3	-4	6	949
4	0	6	999
5	6	6	962
6	277	17	467
7	6	6	996
8	203	14	541
9	14	7	881
10	331	19	458
11	5	7	973
12	-6	7	992
13	2	8	970
14	533	29	343
15	23	9	877
16	118	15	662
17	7	9	993
18	88	13	710
19	-7	8	993
** H	3	8	******
1	281	16	485
2	329	18	449
3	40	7	842
4	43	8	806
5	7	6	999
6	151	12	568
7	8	6	969
8	92	10	658
9	22	8	848
10	367	21	439
11	10	7	994
12	115	13	665
13	5	8	998
14	113	13	654
15	14	10	897
16	14	9	927
17	58	11	776
18	54	11	777
19	26	9	873
** H	4	8	******
0	39	7	851
1	42	8	808
2	617	28	343
3	4	7	914
4	167	12	575
5	51	8	795
6	35	9	813
7	165	14	576
8	0	7	926
9	115	13	612
10	8	9	916
11	26	9	881
12	34	9	914
13	49	10	796
14	329	23	458
15	14	9	989
16	319	23	468
17	40	11	772
18	400	24	432
** H	5	8	******
1	93	10	693
2	279	18	470
3	5	7	918
4	221	15	528
5	67	9	749
6	2	7	976
7	28	9	822
8	10	8	980
9	222	17	515
10	48	10	761
11	22	10	904
12	62	12	728
13	7	9	961
14	519	32	368
15	1	9	949
16	137	15	616
** H	6	8	******
0	10	8	961
1	-3	8	969
2	314	22	426
3	18	9	876
4	-8	8	961
5	52	10	782
6	41	11	738
7	21	9	884
8	78	14	637
9	67	11	775
10	5	9	936
11	52	12	728
12	10	9	954
13	16	9	924
14	23	10	838
** H	7	8	******
1	106	13	665
2	12	8	998
3	2	9	960
4	-2	8	999
5	41	10	833
6	193	19	509
7	-3	8	999
8	36	10	844
9	49	10	830
10	-15	9	946
11	5	9	915
12	74	12	676
** H	8	8	********
0	43	13	691
1	21	9	918
2	166	17	536
3	47	11	733
4	146	14	616
5	3	8	999
6	65	11	725
7	23	9	874
** H	1	9	******
1	411	22	395
2	76	9	711
3	27	7	907
4	21	8	860
5	108	11	676
6	124	12	630
7	31	8	843
8	-18	7	994
9	49	11	759
10	5	7	979
11	30	10	809
12	-8	9	947
13	20	8	946
14	36	10	887
15	67	13	737
16	50	12	778
17	5	9	909
18	23	9	924
** H	2	9	******
0	23	7	903
1	27	7	881
2	556	26	357
3	504	25	366
4	118	11	648
5	20	7	894
6	136	12	591
7	94	11	713
8	12	8	923
9	7	8	960
10	18	9	906
11	8	10	858
12	202	17	555
H	2	9	********
13	19	9	905
14	72	12	761
15	93	13	731
16	137	15	627
17	69	13	705
18	99	13	695
** H	3	9	******
1	455	22	399
2	72	8	763
3	-4	7	999
4	10	7	957
5	68	10	716
6	83	10	709
7	93	11	672
8	11	8	920
9	28	9	904
10	-1	9	930
11	176	16	559
12	-1	8	956
13	60	11	809
14	16	9	961
15	18	8	992
16	2	9	946
17	162	16	582
** H	4	9	******
0	51	9	774
1	16	7	991
2	73	10	708
3	49	9	797
4	39	9	809
5	161	13	591
6	28	9	909
7	42	10	796
8	25	9	893
9	-8	8	994
10	1	9	916
11	3	9	989
12	16	9	974
13	103	13	701
14	46	12	753
15	107	14	660
16	12	9	934
** H	5	9	******
1	39	10	769
2	55	10	796
3	310	22	439
4	83	11	654
5	-9	8	992
6	2	8	977
7	23	9	873
8	22	11	618
9	114	15	609
10	3	9	989
11	3	9	929
12	58	12	731
13	2	9	992
14	71	12	692
** H	6	9	******
0	300	23	448
1	57	12	746
2	19	9	955
3	36	10	844
4	147	17	568
5	7	9	939
6	63	12	727
7	-9	9	989
8	4	10	839
9	36	10	823
10	6	8	961
11	-8	8	978
12	101	13	659
** H	7	9	******
1	3	8	992
2	10	9	964
3	53	11	766
4	286	22	459
5	273	21	461
6	5	9	976
7	28	10	845
8	10	8	991
9	6	8	933
** H	0	10	******
0	354	22	413
1	349	25	393
2	6	8	982
3	154	15	557
4	355	23	415
5	196	16	554
6	127	13	639
7	-6	9	994
8	-4	8	999
9	56	11	808
10	7	9	954
11	291	21	480
12	266	20	528
13	66	14	694
14	-2	9	997
15	30	11	812
16	635	33	374
** H	1	10	******
1	19	7	925
2	10	7	939
3	10	8	928
4	22	8	905
5	217	17	518
6	29	8	884
7	-3	9	979
8	68	11	758
9	150	16	586
10	102	13	659
11	-8	9	942
12	318	23	482
13	3	10	924
14	71	12	746
15	38	11	809
16	26	10	844
** H	2	10	******
0	488	25	396
1	9	7	972
2	137	13	593
3	13	8	905
4	42	9	782
5	1	10	936
6	73	12	722
7	164	16	561
8	39	10	846
9	7	8	998
10	162	18	575
11	6	10	976
12	275	21	505
13	6	10	918
14	13	9	967
15	3	9	964
16	59	12	741
** H	3	10	******
1	101	12	700
2	58	11	738
3	5	9	908
4	42	10	796
5	12	9	874
6	262	19	482
7	19	9	885
8	100	13	671
9	86	13	733
10	-5	9	998
11	149	18	559
12	-5	9	997
13	10	10	906
14	98	13	703
15	-3	8	999
** H	4	10	******
0	960	44	289
1	59	12	721
2	74	12	701
3	-4	8	992
4	9	8	967
5	58	11	740
6	-11	9	996
H	4	10	******
7	237	20	515
8	428	24	396
9	95	15	642
10	141	17	588
11	7	9	956
12	69	13	691
13	108	14	652
** H	5	10	******
1	73	12	734
2	40	10	887
3	0	9	958
4	9	9	943
5	-13	9	997
6	2	10	887
7	5	9	991
8	18	9	960
9	16	9	921
10	0	9	944
11	29	10	833
** H	6	10	******
0	354	25	428
1	47	11	814
2	20	9	955
3	74	13	713
4	184	18	528
5	-12	8	999
6	33	10	834
7	17	10	863
8	21	9	901
** H	7	10	******
1	4	8	998
2	7	9	894
** H	1	11	******
1	12	9	926
2	20	9	899
3	16	9	926
4	1	8	975
5	10	8	972
6	-3	9	963
7	-2	10	972
8	126	17	617
9	52	12	769
10	75	13	742
11	12	9	995
12	0	9	998
13	-1	9	975
** H	2	11	******
0	-16	9	967
1	124	13	653
2	106	14	639
3	223	20	473
4	6	9	973
5	229	20	535
6	75	13	746
7	4	10	991
8	9	9	981
9	6	9	995
10	27	10	950
11	27	12	806
12	47	12	795
13	13	10	844
** H	3	11	******
1	37	11	861
2	87	13	727
3	18	10	912
4	5	9	966
5	1	10	948
6	9	10	914
7	30	12	805
8	66	14	725
9	107	15	671
10	124	16	621
11	17	9	939
12	-5	9	994
** H	4	11	******
0	262	23	461
1	-5	9	967
2	6	11	844
3	43	13	729
4	39	11	795
5	27	12	803
6	6	10	953
7	96	15	651
8	-11	9	999
9	24	10	868
10	-1	10	929
** H	5	11	******
1	21	12	801
2	-6	9	980
3	83	15	622
4	209	20	502
5	9	10	864
6	64	12	724
7	57	14	676
** H	6	11	******
0	632	36	334
** H	0	12	******
0	324	24	440
1	64	13	751
2	30	11	894
3	206	20	550
4	17	11	928
5	164	19	569
6	152	17	619
7	35	11	867
8	35	13	757
9	166	16	614
10	164	16	606
** H	1	12	******
1	-8	10	986
2	113	17	605
3	142	19	564
4	117	18	599
5	53	14	746
6	35	10	940
7	1	11	926
8	-2	11	893
9	17	10	906
10	39	12	778
** H	2	12	******
0	57	13	757
1	-5	10	983
2	37	12	802
3	118	15	675
4	30	10	987
5	153	18	574
6	162	21	509
7	140	17	610
8	61	12	778
9	210	20	516
** H	3	12	******
1	50	13	756
2	34	12	803
3	153	19	547
4	-28	10	997
5	96	14	674
6	32	11	827
7	320	24	451
** H	4	12	******
0	115	14	677
1	11	9	950
2	184	19	543
3	103	14	667
4	-6	11	859
** H	1	13	******
1	256	23	484
2	63	13	765
3	8	11	907

reduced the intensities of 382 of the 1926 measured reflections to less than half their ideal values. The 72 strong reflections with calculated extinction factors less than 0·289 were omitted from the refinement, and these are identified by the letter *E* in Table 1.

The 155 parameters adjusted in the final cycle of refinement include the scale factor, six anisotropic extinction coefficients, the coordinates and anisotropic temperature factor coefficients for each of 16 atoms, and the neutron-scattering factors for four kinds of atoms relative to that for oxygen which was held constant. The values of these parameters are listed in Table 2 together with their standard errors. The discrepancy factor based on F^2, $R=\sum|F^2_{\rm obs}-F^2_{\rm calc}|/\sum F^2_{\rm obs}$, was 0·092 for the observations used in the refinement and 0·114 with all observations included.

Interatomic distances and angles are listed in Tables 3, 4, and 5, with standard errors calculated from the full variance-covariance matrix of the least-squares refinement. Table 3 also includes values corrected for the effects of thermal motion for those distances for which the riding model (Busing & Levy, 1964) seems

Table 2. *Final parameters for urea–phosphoric acid*

The coordinates *x*, *y*, and *z* and the temperature factor coefficients B_{ij} have been multiplied by 10^4 and 10^2, respectively. The latter are defined by the expression for the temperature factor:

$$\exp\,[-(B_{11}h^2a^{*2}+\cdots\cdots+2B_{23}klb^*c^*)/4]\,.$$

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
P	3109 (1)	2783 (3)	3093 (2)	145 (8)	123 (9)	109 (8)	1 (6)	10 (5)	−12 (6)
O(1)	3391 (1)	907 (3)	3610 (3)	196 (8)	158 (9)	276 (9)	6 (6)	−33 (7)	20 (7)
O(2)	2775 (1)	3844 (3)	4356 (2)	200 (7)	173 (8)	104 (7)	2 (6)	14 (5)	−7 (5)
O(3)	2468 (1)	2508 (4)	1934 (2)	177 (7)	332 (11)	138 (7)	25 (7)	−42 (6)	−57 (7)
O(4)	3795 (1)	3662 (3)	2381 (2)	226 (8)	203 (9)	193 (8)	−62 (6)	78 (6)	−34 (6)
O(5)	4470 (1)	6340 (3)	3107 (2)	294 (9)	255 (10)	138 (8)	−111 (7)	19 (6)	−4 (6)
N(1)	5077 (1)	7830 (2)	4913 (2)	212 (6)	249 (8)	233 (6)	−78 (4)	4 (4)	−45 (4)
N(2)	3966 (1)	6363 (2)	5423 (2)	244 (6)	345 (9)	204 (6)	−97 (5)	81 (4)	−65 (5)
C	4500 (1)	6835 (2)	4469 (2)	159 (7)	146 (8)	143 (7)	−16 (5)	16 (5)	−14 (5)
H(1)	2977 (2)	90 (5)	3939 (5)	343 (17)	211 (15)	291 (16)	−32 (13)	−21 (13)	20 (12)
H(3)	2607 (2)	1981 (6)	951 (4)	291 (15)	300 (17)	193 (13)	20 (12)	−24 (11)	−40 (12)
H(4)	4088 (3)	5048 (6)	2815 (5)	344 (18)	374 (19)	240 (15)	44 (15)	70 (14)	45 (13)
H(11)	5497 (3)	8080 (7)	4193 (6)	282 (16)	390 (21)	361 (20)	−101 (14)	52 (15)	−22 (16)
H(12)	5120 (3)	8219 (7)	5973 (6)	404 (21)	430 (23)	306 (20)	110 (17)	−59 (16)	−106 (17)
H(21)	3518 (3)	5643 (8)	5092 (5)	307 (18)	482 (26)	335 (19)	−132 (18)	62 (15)	−61 (17)
H(22)	3987 (3)	6793 (9)	6475 (5)	470 (24)	613 (30)	242 (18)	−128 (21)	104 (16)	−129 (18)

Anisotropic extinction parameters†

W''_{11}	W''_{22}	W''_{33}	W''_{12}	W''_{13}	W''_{23}
17 (2)	9 (1)	19 (3)	7 (1)	2 (1)	3 (1)

Scattering amplitudes, ‡ cm × 10^{-15}

P	O	N	C	H
507 (7)	577	926 (6)	658 (7)	−370 (4)
[510]	[577]	[940]	[665]	[−372]

† These are the values of W'_{ij} (Coppens & Hamilton, 1970) multiplied by 10^4.

‡ Amplitudes of atoms of the same chemical element were constrained to be equal. That of oxygen was not varied. The figures in brackets are the values tabulated by the Neutron Diffraction Commission (1969).

Table 3. *Interatomic distances* (Å) *for bonded atoms and for atoms involved in hydrogen bonds*

Corrected distances are the estimated mean distances assuming the riding model for thermal motion.

Phosphoric acid	Uncorrected distance	Corrected distance	Urea	Uncorrected distance	Corrected distance
P—O(1)	1·560 (3)		C—O(5)	1·281 (3)	
P—O(2)	1·507 (3)		C—N(1)	1·323 (2)	
P—O(3)	1·553 (3)		C—N(2)	1·324 (2)	
P—O(4)	1·519 (3)		N(1)—H(11)	1·002 (5)	1·021 (5)
O(1)—H(1)	0·997 (5)	1·007 (5)	N(1)—H(12)	1·001 (5)	1·026 (5)
O(3)—H(3)	0·999 (4)	1·005 (5)	N(2)—H(21)	1·003 (5)	1·020 (5)
O(2)···H(1)′	1·664 (5)		N(2)—H(22)	1·000 (5)	1·032 (6)
O(2)···H(3)′	1·591 (4)		O(5)···H(4)	1·207 (6)	
O(2)···H(21)	1·992 (5)		O(5)···H(12)′	2·262 (6)	
O(4)···H(4)	1·223 (6)		O(5)···H(22)′	2·200 (6)	
O(4)···H(11)′	1·939 (5)				

appropriate. Table 6 lists the displacements of atoms of the urea moiety and of some surrounding atoms from the plane defined by N(1), N(2), and O(5). The standard errors of these quantities were calculated using the full variance-covariance matrix.

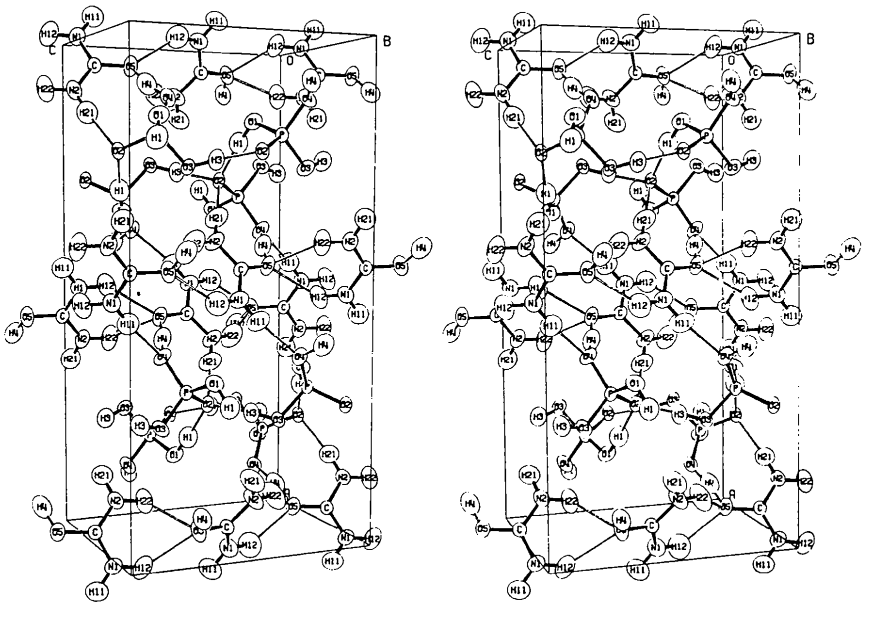
Fig. 1 is a stereoscopic drawing showing the packing of the molecules in a unit cell and the hydrogen bonds between them. Fig. 2, also a stereoscopic drawing, shows the basic asymmetric unit. The atoms are depicted as 50% probability ellipsoids (Johnson, 1965)

Table 4. *Bond angles and angles around atoms accepting hydrogen bonds* (°)

Phosphoric acid		Urea	
O(1)——P——O(2)	111·9 (2)	O(5)——C——N(1)	118·9 (2)
O(1)——P——O(3)	108·2 (2)	O(5)——C——N(2)	121·0 (2)
O(1)——P——O(4)	105·2 (2)	N(1)——C——N(2)	120·1 (2)
O(2)——P——O(3)	106·9 (2)	C——N(1)——H(11)	118·6 (3)
O(2)——P——O(4)	113·7 (2)	C——N(1)——H(12)	120·7 (3)
O(3)——P——O(4)	110·8 (2)	H(11)——N(1)——H(12)	120·4 (4)
P——O(1)——H(1)	114·0 (3)	C——N(2)——H(21)	120·8 (3)
P——O(3)——H(3)	117·9 (3)	C——N(2)——H(22)	120·2 (3)
P——O(2)——H(1)′	115·9 (2)	H(21)——N(2)——H(22)	118·9 (4)
P——O(2)——H(3)′	123·3 (2)	C——O(5)···H(4)	117·6 (3)
P——O(2)···H(21)	110·4 (2)	C——O(5)···H(12)′	138·5 (2)
H(1)′——O(2)···H(3)′	105·8 (2)	C——O(5)···H(22)′	118·2 (2)
H(1)′···O(2)···H(21)	102·8 (2)	H(4)····O(5)···H(12)′	102·4 (3)
H(3)′···O(2)···H(21)	94·9 (2)	H(4)····O(5)···H(22)′	98·4 (3)
P——O(4)···H(4)	124·7 (3)	H(12)···O(5)···H(22)′	62·4 (2)
P——O(4)···H(11)′	136·4 (2)		
H(4)···O(4)···H(11)′	98·7 (3)		

Table 5. *Hydrogen bonds. Uncorrected interatomic distances* (Å) *and angles* (°) *at the hydrogen atom*

X–H···O	X···O	X–H	H···O	∠ at H
O(1)——H(1)····O(2)′	2·658 (3)	0·997 (5)	1·664 (5)	173·7 (4)
O(3)——H(3)····O(2)′	2·589 (3)	0·999 (4)	1·591 (4)	176·4 (4)
O(4)···H(4)····O(5)	2·421 (3)	1·223 (6)	1·207 (6)	169·9 (4)
N(1)——H(11)···O(4)′	2·935 (3)	1·002 (5)	1·939 (5)	172·4 (5)
N(1)——H(12)···O(5)′	3·131 (3)	1·001 (5)	2·262 (6)	144·4 (4)
N(2)——H(21)···O(2)	2·982 (2)	1·003 (5)	1·992 (5)	168·9 (5)
N(2)——H(22)···O(5)′	3·095 (3)	1·000 (5)	2·200 (6)	148·3 (5)



UREA - PHOSPHORIC ACID UREA - PHOSPHORIC ACID

Fig. 1. Stereoscopic view showing the arrangement of the molecules in a unit cell and the hydrogen bonds between them.

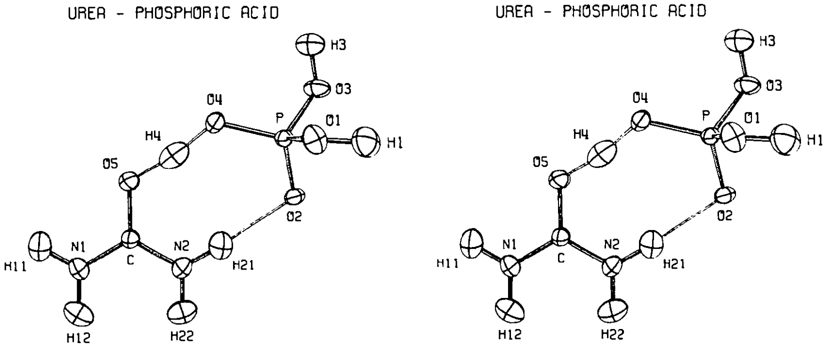


Fig. 2. Stereoscopic view of the basic asymmetric unit. The atoms are represented as 50% probability ellipsoids.

Table 6. *Displacements* (Å) *of atoms of the urea moiety and of some surrounding atoms from the plane defined by* N(1), N(2), *and* O(5)

Opposite the hydrogen atoms are listed the oxygen atoms which are acceptors for the corresponding hydrogen bonds. The displacement of atom C from this plane is 0·000 (2) Å.

Hydrogen atom		Acceptor atom	
H(4)	0·392 (6) Å	O(4)	0·894 (6) Å
H(11)	0·080 (6)	O(4)′	0·044 (5)
H(12)	0·016 (6)	O(5)′	−0·414 (5)
H(21)	−0·030 (6)	O(2)	0·256 (5)
H(22)	−0·032 (7)	O(5)′	−0·414 (5)

with ellipses showing the principal planes of thermal motion.

Computer programs used for these calculations included the following (see *World List of Crystallographic Computer Programs*, 1972);

least-squares refinement	*ORXFLS*3 ,
distances and angles	*ORFFE*3 ,
structure drawing	*ORTEP*2 .

Table 1 was prepared using program *EDIT* by G. M. Brown of this Laboratory.

Discussion

The most notable feature of the structure is the short hydrogen bond which links the urea and phosphoric acid moieties. The O(4)···O(5) separation for this bond is 2·421 Å (Table 5), and the acid proton H(4) is essentially centered at distances of 1·223 Å and 1·207 Å, respectively, from these atoms. The thermal motion of H(4), which is shown in Fig. 2, has its largest principal component within 11° of the O(4)···O(5) direction. The root-mean-square displacement in this direction is 0·23 Å.

The phosphate group is essentially tetrahedral, with O–P–O angles ranging from 105·2° to 113·7°, as shown in Table 4. The distances P–O(1) and P–O(3) involving the hydroxyl oxygen atoms are 1·560 Å and 1·553 Å, respectively (Table 3). These are considerably longer than the 1·507 Å length of P–O(2), which has more multiple-bond character because O(2) has no hydrogen atom of its own. Intermediate between these extremes is the P–O(4) distance of 1·519 Å, which seems reasonable because O(4) shares its hydrogen atom with O(5) of the urea molecule.

Worsham & Busing (1969) showed that the C–O and C–N distances in the uronium ion should be longer and shorter, respectively, than the corresponding distances in urea. The bond lengths found in the present study are, as would be expected, intermediate between those of urea (Pryor & Sanger, 1970) and those of uronium nitrate (Worsham & Busing, 1969). In urea, urea–phosphoric acid, and uronium nitrate, respectively, the C–O distances are 1·260, 1·281, and 1·298 Å, and the average C–N distances are 1·352, 1·324, and 1·314 Å.

The carbon atom of urea lies almost exactly in the plane defined by N(1), N(2), and O(5). Apparently the shared proton H(4) has little tendency to remain in the molecular plane, and it is displaced 0·392 Å to a position which makes the hydrogen bond nearly straight (Table 6). The deviations of the other urea hydrogen atoms from the plane are considerably smaller, and they do not seem to be related simply to the positions of the oxygen atoms to which they form hydrogen bonds.

Each hydrogen atom is involved in one hydrogen bond as described in Table 5, and the over-all bonding scheme is shown in Fig. 1. It is interesting to note that atoms O(1) and O(3), which have their own hydrogen atoms, accept no hydrogen bonds, but atom O(2), which has no hydrogen of its own, accepts three such bonds. Intermediate between these extremes is the atom O(4), which shares H(4) and accepts one other hydrogen bond, and the atom O(5), which also shares H(4) and accepts two rather long and bent hydrogen bonds.

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An X-ray Structure Redetermination of the Urea–Phosphoric Acid Compound*

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The crystal structure of the urea–phosphoric acid compound $CO(NH_2)_2.H_3PO_4$ has been redetermined ($Pbca$; $z=8$; $a=17{\cdot}6508$, $b=7{\cdot}4817$, $c=8{\cdot}9991$ Å). With a very short and nearly centred hydrogen bond from the acid to the oxygen atom of the base the structure is between that of a true adduct and that of an onium salt. All hydrogen atoms were located; with 1126 independent Cu $K\alpha$ counter data the final R value is 0·031.

As part of our program to study the structural behaviour of the acid proton in crystalline acid/base systems (*e.g.* Altenburg & Mootz, 1971; Mootz & Altenburg, 1971) we have carried out an accurate crystal structure determination of the urea–phosphoric acid compound. Earlier investigations by Sundera-Rao, Turley & Pepinsky (1957) and by Wolfram, Arutunian, Antishkina & Porai-Koshits (1967) revealed a very strong hydrogen bond between the acid and the oxygen atom of the urea moiety (O···O distance 2·414 Å and 2·45 Å respectively), but did not decide between nor comment on the structural alternatives of a true adduct and an onium salt. In order to study this aspect of the structure in detail, which requires a knowledge of the hydrogen atom positions as well as more accurate interatomic distances and angles of the heavy atoms, we decided to collect new X-ray data and repeat the analysis.

After we had finished the refinement we learned from the title programme of a meeting of a recent neutron diffraction study of the same compound by Busing & Kostansek (1971). This work has now been published in detail (Kostansek & Busing, 1972). Apart from being more accurate in hydrogen atom positions and thermal parameters, it is in good agreement with our own study, the presentation of which can therefore be kept short.

Experimental and crystallographic data

The urea–phosphoric acid compound crystallizes in space group $Pbca$ with eight formula units $CO(NH_2)_2.H_3PO_4$ per unit cell. A least-squares refinement of angular measurements on a diffractometer yielded the improved lattice parameters $a=17{\cdot}6508$ (9), $b=7{\cdot}4817$ (4) and $c=8{\cdot}9991$ (7) Å. The intensities of 1126 independent reflexions with $\theta<70°$ were measured with Ni-filtered Cu $K\alpha$ radiation on a tape-controlled Siemens automatic diffractometer in the $\theta/2\theta$ mode. No correction for absorption was applied ($\mu_{\mathrm{Cu}\,K\alpha}=38{\cdot}9$ cm^{-1}).

Refinement of the structure

Least-squares refinement was started from the heavy atom parameters of Sundera-Rao, Turley & Pepinsky (1957). The seven hydrogen atoms could be located in a difference Fourier map. After an empirical correction of the thirteen strongest intensities for extinction, anisotropic treatment of all atoms converged to an unweighted R value of 0·031. The observations were weighted according to $w=1$ for $|F_o|<K$ and $w=K^2/|F_o|^2$ for $|F_o|\geq K$ with $K=20$. The form factors used

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* Short version of a part of the dissertation of K.-R. Albrand, Techn. Universität Braunschweig, (1972).